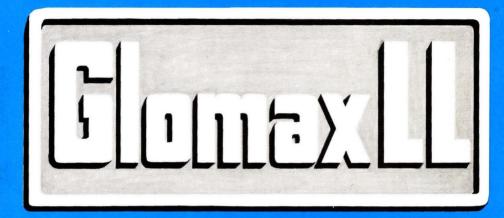


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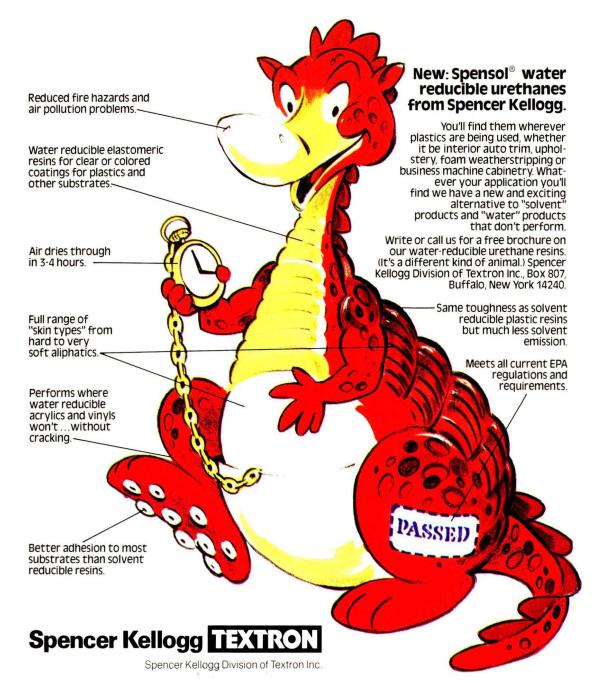
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# DECEMBER 1979 JOURNAL OF COATINGS TECHNOLOGY

Volume 51 Number 659

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# Comment

#### **The First Step**

A new organization—International Committee for Coordinating Activities of Technical Groups in the Coatings Industry—was formally established in Paris, France on June 23, 1979.

Attending that historic meeting were elected officers of the following groups, the charter members of the ICC:

Federation of Associations of Technicians in the Paint, Varnish, Lacquer and Printing Ink Industries of Continental Europe Federation of Societies for Coatings Technology Japan Society of Colour Material Oil and Colour Chemists' Association—Australia Oil and Colour Chemists' Association—United Kingdom Scandinavian Federation of Paint and Varnish Technologists

The purpose of the ICC is to promote international cooperation and understanding among members through exchange of communications, attendance and presentation of papers at each others' meetings, and sponsorship of joint technical programs.

The second meeting of the ICC was held during the Federation's Annual Meeting in St. Louis on October 4. After a review of organizational details, the committee set as its first cooperative effort a search for available information on "The Technical Uses of Computers in the Coatings Industry." The Federation, through its Technical Information Systems Committee, will lead this activity and the preparation of the bibliography.

This is a great first step and all best wishes to the ICC for a long life and continued progress.—FJB

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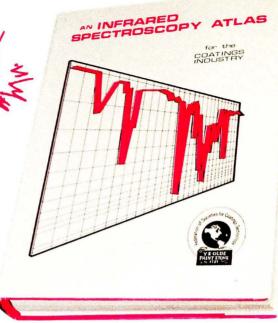
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# Abstracts of Papers in This Issue

COMPARISON OF METHODS TO DETERMINE WATER CONTENT OF EMULSION PAINTS—Golden Gate Society for Coatings Technology

Journal of Coatings Technology, 51, No. 659, 45 (Dec. 1979)

At the time the California Air Resources Board proposed stipulating the maximum amount of solvent permitted in a given volume of paint, there was no recognized method for determining the water content of an emulsion paint. In response to the evident need, two specific methods, using the gas-liquid chromatograph as the analytical tool, were proposed: One included a distillation step, the other did not. A third, unspecified method, based on Karl Fischer titration was also discussed. The Golden Gate Society compared these analytical methods by submitting emulsion paint samples to participating laboratories for water determination by one, or more, of these methods. The results of the comparison are discussed in this paper.

RESIDUAL STRESS IN A SOLVENTLESS AMINE-CURED EPOXY COATING—S.G. Croll

Journal of Coatings Technology, 51, No. 659, 49 (Dec. 1979)

Residual stress in a solventless, amine-cured epoxy coating has been measured and found to increase with coating thickness. The trend with thickness probably results from the competition between densification, produced by the curing reaction, and swelling by absorbed atmospheric moisture, which is prevalent in the thinner coatings. The exothermic curing reaction has no influence upon the internal stress nor upon curing rate of the coating because it does not produce a measurable temperature increase. A heat transfer calculation confirms that the temperature increase in a typical coating is very small.

SOLVENT LOSS FROM WATER SOLUBLE ACRYLIC COATINGS AND ITS EFFECT ON FLOW—L. B. Brandenburger and L. W. Hill

Journal of Coatings Technology, 51, No. 659, 57 (Dec. 1979)

Evaporation of water and several cosolvents during and up to 15 min after spray application was investigated using gas chromatography to determine residual volatile components. Statistical analysis was used to determine the independent effects of starting levels of water, secbutanol, and 2-butoxyethanol, as well as relative humidity and dry film thickness on water remaining in the coating. Independent effects were also determined for secbutanol and 2-butoxyethanol remaining. This information was used to prepare coatings to simulate compositions obtained during and following spray application. The low shear viscosity of the prepared compositions was correlated with visually observed sagging of the actual sprayed coatings.

LINSEED OIL-METAL ACETYLACETONATE SYSTEMS. V. THERMOGRAVIMETRY OF IRON-BASED PIG-MENTS—N. Indictor, C.J. Shahani, M.J.D. Low, J. Chan, and N.S. Baer

Journal of Coatings Technology, 51, No. 659, 67 (Dec. 1979)

Thermogravimetric data (weight change at constant temperature) are presented in the temperature range 60– 100°C in air for linseed oil, containing small quantities (2 mg/5 ml) of fifteen iron-based pigments. Inhibition periods, rate of increase in weight, maximum weight achieved, and subsequent weight decrease are reported. Temperature parameters are presented. These data are compared to data obtained for iron acetylacetonates. Iron pigments, at the concentrations used, produced thermogravimetric curves very little different from linseed oil alone.

BEHAVIOR OF AMINES IN WATER—DILUTABLE COATINGS AND PRINTING INKS—C. M. Hansen and K. B. Nielsen

Journal of Coatings Technology, 51, No. 659, 73 (Dec. 1979)

Legislation exists in Scandinavia restricting the "free amine" content of coatings to less than 0.5%. This legislation was originally intended for two component epoxy coatings where interpretation of what "free amine" is, is clear. Interpretation concerning the amine added to water-borne coatings has been less clear, i.e., is it "free" or not?

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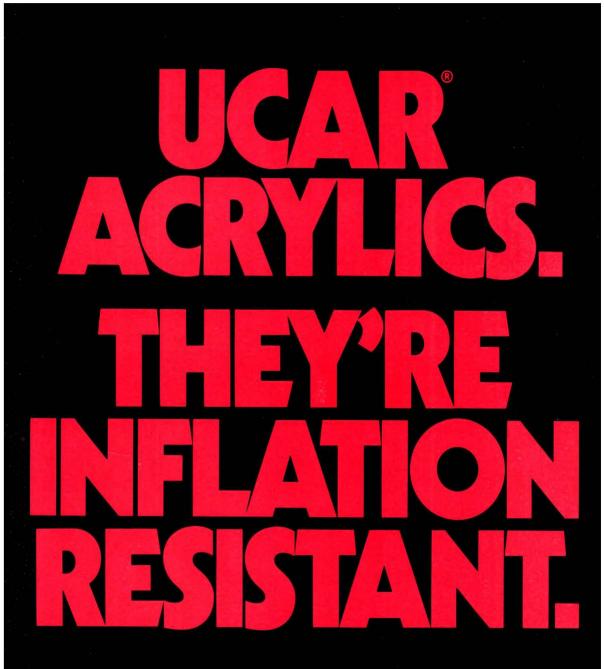
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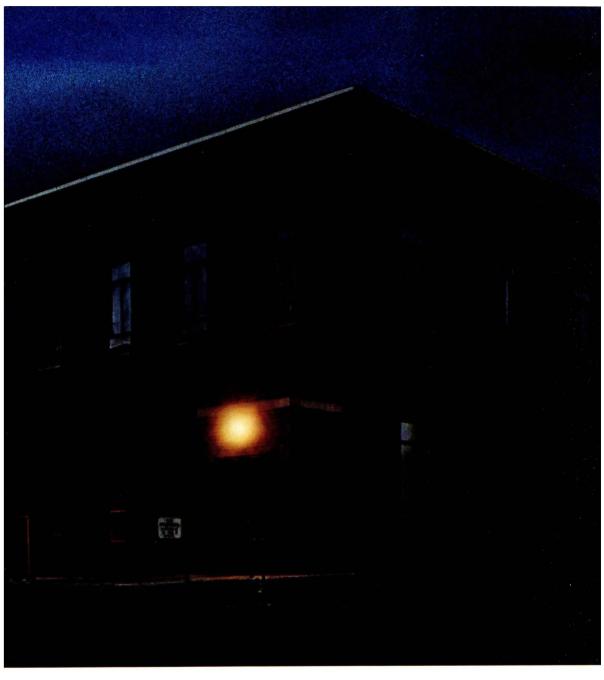
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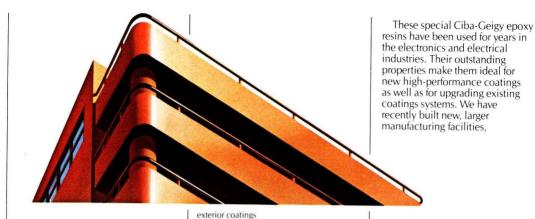
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# THE SECOND GENERATION EPOXY RESINS FROM CIBA-GEIGY.



tanker linings

Aracast® hydantoin resins provide exceptional weatherability Available in several forms: water soluble or insoluble, from low

viscosity liquids to solids.

Araldite® PT 810 resin crosslinks acrylic and polyester powders Exhibits excellent weatherability (gloss retention and color stability).

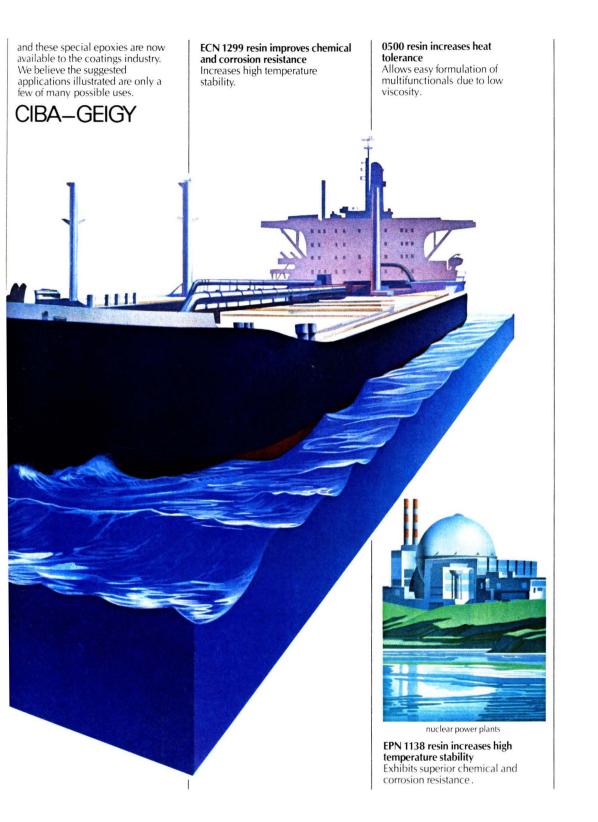


hardeners for vinyl plastisols

Hardeners HT 939 and HY 940 improve adhesion of epoxy modified vinyl plastisols Remain stable six months after

formulating – high latency. Cure rapidly – five minutes at 100°C.

FOR A NEW GENERATION OF



# **IGH-PERFORMANCE COATINGS.**

#### **Typical Product Family Profiles:**

	Form	Epoxy Functionality	Viscosity at 25°C/ melting point	Solution Viscosity*	Additional Features
0500	Liquid	3	3,000 cP	200 cP	high heat resistance
ECN's	Solid	2.7-5.4	35-99°C	3,000 cP	tight crosslink density, chemical resistance, hardnes
EPN's	Liquid/Semi - solid	- 2.2-3.6	1700-3500 cP at 52°C	3,000 cP	FDA-conforms to 175.300
Aracast® hydantoins	Liquid/Solid	2	500 cP/95℃	-	non-aromatic , weatherable non-crystallizing, very low viscosity
Araldite <sup>®</sup> PT 810	Solid	3	95°C	_	non-aromatic, weatherable
HT 939 HY 940	Solid Dispersion	_	105°C 600,000 cP	_	latent hardener/rapid cure latent hardener/rapid cure

\*80% solids in Cellosolve acetate

#### Suggested Applications:

	High Solids	Powders	Water- borne	Solvent- free	Solvent- based	Crosslink Polyesters & Acrylics
0500						
ECN's						
EPN's						
Aracast <sup>®</sup> hydantoins						
Araldite® PT 810						
HT 939/HY 940					(vinyl	plastisols)

.

Please send information on:       0500 ECN's EPN's Aracast® hydantoins Araldite® PT 810 HT 939/HY 940         Ciba-Geigy Corporation       Primers         Resins Department       Ardsley, New York 10502         Phone (800) 431-1874,       in New York (914) 478-3131         Name		
Ciba-Geigy Corporation         Resins Department         Ardsley, New York 10502         Phone (800) 431-1874,         in New York (914) 478-3131         Name         Company         Address         City         State       Zip	Please send information on:	JC
Resins Department   Ardsley, New York 10502   Phone (800) 431-1874,   in New York (914) 478-3131   Name   Company   Address   City   State   Zip	🗆 0500 🗈 ECN's 🗆 EPN's 🗆 Aracast® hydantoins 🗆 Araldite® PT 810 🗆 HT 939/HY 9	•40
Ardsley, New York 10502         Phone (800) 431-1874,         in New York (914) 478-3131         Name         Company         Address         City         State         Zip	Ciba-Geigy Corporation	
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		CIBA-GEIGY

FEDERATION OF SOCIETIES FOR COATINGS TECHNOLOGY

# 1979 ANNUAL MEETING REVIEW

# ST. LOUIS OCTOBER 3-5

## Over 4600 Registrants Attend Annual Meeting And Paint Show at St. Louis Convention Center

A total of 4691 registrants attended the Federation's 57th Annual Meeting and 45th Paint Industries' Show, held October 3-5 at the St. Louis Convention Center.

The three-day event provided those attending with a full slate of technical presentations keyed to the theme, "Progress Through Innovation," along with a record number of exhibits by supplier companies in the largest-ever Paint Show.

The Keynote Address by Howard K. Smith on opening day set the programming tempo as a standing-room only crowd turned out to hear the Newscaster/Commentator's comments on a wide range of current topics.

Chairman Morris Coffino and his Program Committee members had scheduled concurrent sessions all three days, and attendance was excellent throughout, particularly so for the presentations on Lab Management and Innovation Seminar, Ecological and High-Performance Coatings, and the PRI Seminar on Aqueous Coatings. And the free-wheeling questionand-answer sessions that marked many of the presentations underscored the high interest level of those in attendance. Meanwhile, traffic was heavy on the exhibit floor, where registrants viewed the displays of 139 exhibiting firms and talked shop with their top technical people. Exhibitors occupied a total of 337 booths, the largest in Paint Show history, and they generally expressed their satisfaction with both the number and quality of the registrants.

Again this year, the traditional banquet was replaced with a luncheon, on Friday, October 5. Business formalities were kept to a minimum, and following presentation of the Heckel Award, as well as the C. Homer Flynn Awards for outstanding exhibits in the Show (see Awards story), the more than 500 registrants in attendance were entertained by Dr. James H. Boren and his good-natured spoofing of bureaucracy in our modern society.

It was, all in all, a very successful event, and much credit for that happy state of affairs is due the many members of the Kansas City and St. Louis Societies who served on the Host Committee under the direction of Howard Jerome. The Federation is indebted to them and to all who helped to make the 1979 Annual Meeting such a success.



President James McCormick and wife, Elaine (right-center), opened 1979 Paint Show with traditional ribbon-cutting ceremony. Looking on are (left to right): Treasurer William H. Ellis; Treasurer-Elect Howard Jerome; Mrs. Ellis; Mrs. Elder Larson; President-Elect Elder C. Larson; Mrs. Frank J. Borrelle; and Executive Vice-President Borrelle

Stanley LeSota, Senior Chemist at Rohm and Haas Co., Spring House, Pa., Research Laboratories, was honored by the Federation of Societies for Coatings Technology with the 1979 George Baugh Heckel Award for his years of service to the Federation as Chairman of the Definitions Committee, whose efforts culminated in publication of the *Paint/ Coatings Dictionary*. The presentation was made at the FSCT Annual Meeting luncheon on October 5, in St. Louis, Mo.

The award plaque is presented each year to the individual whose contributions to the general advancement of the Federation's interest and prestige have been outstanding.

In addition to serving as Chairman of the Federation Definitions Committee, Mr. LeSota is also Chairman of the American Society for Testing and Materials D-I Subcommittee of Definitions, as well as Chairman of the Subcommittee on Pictorial Standards of Coatings Defects. He is also a member of the D-I Executive Committee.

A Past-President of the Philadelphia Society for Coatings Technology, he is a recipient of their Liberty Bell Award and has just completed a three-year term as Chairman of the Technical Committee.

Other awards presented during the Annual Meeting honored those who have made outstanding contributions to the Federation and the coatings industry.

#### Union Carbide Award

The 1979 Award, newly-established by Union Carbide Corp., was presented to the Philadelphia Society for Coatings Technology for its extraordinary contribution to the advancement of coatings technology in compiling the Paint / Coatings Dictionary. This work began in 1965 as a project of the Technical Committee of the Philadelphia Society. Originally conceived to compile a glossary of terms for the coatings industry, the project evolved into a more ambitious undertaking and the subcommittee formed for the enterprise became the Federation Definitions Committee, composed of members of the Philadelphia Society under the chairmanship of Stan LeSota. The 632-page dictionary, which contains more than 4500 coatings terms and their definitions, along with a thesaurus and extensive bibliography, was published in October 1978.

The Award (\$2000 in cash and a plaque) is given to an individual (or group of individuals) in recognition of an extraordinary contribution to: (1) the; advancement of coatings technology or; (2) the furtherance of research and education in the field of coating's technology.

#### Armin J. Bruning Award

Established in 1962 in honor of Armin "Joe" Bruning, pioneer in the application of color science to the paint industry, this award is for "the most outstanding contribution to the science of color in the field of coatings technology."

The 1979 award plaque was presented to Dr. Gunter Wyszecki, Head of the Optics Section of the Physics Division, National Research Council of Canada, in Ottawa, for his 25 years of leadership in the field of color through the direction of research, publication, and national and international professional society activities. He is the author of more than 80 publications, with major concentration on metamerism, color matching, heterochromatic brightness matching and color discrimination. Under his direction, the Optical Section of the National Research Council has become one of the world's leading groups in colorimetry, color vision, photometry, radiometry and related fields. In addition, Dr. Wyszecki is perhaps best known for his leadership

of the Colorimetry Committee of the International Commission on Illumination, which has developed many international standards in colorimetry.

#### **Roon Foundation Awards**

These awards, established by Leo Roon, well-known figure in the coatings industry and Director of the Roon Foundation, are for the best technical papers entered in the competition and submitted for presentation at a Federation Annual Meeting by individuals associated with the organic coating industry.

Winners in the 1979 competition were:

FIRSTPRIZE (\$900)—"Transfer and Leveling Process and Rheological Design in Water-Borne Reverse Roll Coating Systems"—Takehisa Matsuda and William H. Brendley, Jr., of Rohm and Haas Co., Spring House, Pa.

SECOND PRIZE (\$500)—"Plastic Pigment—A Novel Approach to Microvoid Hiding"—Alexander Ramig, Jr. and F. Louis Floyd, of Glidden Coatings & Resins Div., SCM Corp., Strongsville, Ohio.

THIRD PRIZE (\$300)—"Polyphospha-

#### Program Theme Announced and Papers Invited For FSCT 1980 Annual Meeting in Atlanta

The 1980 Annual Meeting of the Federation of Societies for Coatings Technology will have as its theme, "Three R's for the Eighties: Research, Resources, and Regulations," it was announced by Program Chairman Hugh W. Lowrey, of Indurall Coatings, Inc.

The Annual Meeting will be held in conjunction with the Paint Industries' Show at the Civic Center, Atlanta, Ga., on October 29, 30, and 31.

The theme recognizes that the coming decade looms as a time of stress and challenge. To remain an economically sound producer of increasingly valuable products, the coatings industry must utilize research and ingenuity to cope with the constraints of limited resources and restrictive regulations.

It is anticipated that among the demands of the Eighties will be alternative raw materials based on more plentiful resources, along with innovative recycling methods to minimize hazardous wastes. At the same time, the changing needs of the world must be served through coatings research. A prime consideration, for instance, will be to continue to augment the supply of dependable anti-corrosive coatings despite changing materials, methods, and surfaces.

Prospective speakers are invited to present original papers on the many and varied aspects of the theme, and are requested to submit abstracts for review to Hugh W. Lowrey, FSCT Program Chairman, c/o Indurall Coatings, Inc., P. O. Box 2371, Birmingham, Ala. 35201. *Deadline for receipt of abstracts is March 1*.

Assisting Chairman Lowrey in developing the program is a Steering Committee composed of: Thomas J. Miranda (Vice-Chairman), of Whirlpool Corp., Benton Harbor, Mich.; Umberto Ancona, of McCloskey Varnish Co., Philadelphia, Pa.; Fred M. Ball, of Eastman Chemical Products, Inc., Kingsport, Tenn.; Thomas Ginsberg, of Union Carbide Corp., Bound Brook, N. J.; Kenneth A. Kieselburg, of Valspar Corp., Rockford, Ill.; and William Mirick, of Battelle Memorial Institute, Columbus, Ohio.



Stanley LeSota (left) accepts the 1979 George Baugh Heckel Award from E.E. McSweeney, Chairman of the Heckel Award Committee



President McCormick presents Charles Kumins (left), the 1979 Mattiello Memorial Lecturer, with a certificate of appreciation for his presentation, "Physical Chemical Models for Organic Protective Coatings"

Umberto Ancona (center), Chairman of the 1979 Roon Awards Committee, presented checks to winners (left to right): S.H. Rose and A.K. Chattopadhyay (co-authors Third Prize); T. Matsuda and W.H. Brendley (coauthors First Prize); R.L. Hinrichs (co-author Third Prize); and A. Ramig and F.L. Floyd (co-authors Second Prize)





Union Carbide Award was presented to the Philadelphia Society for its efforts in compiling the Paint/Coatings Dictionary. Accepting the award from Dr. N.L. Zutty (right), Vice-President and General Manager, Coatings Materials Dept., Union Carbide Corp., are Stan LeSota (left), Chairman of the Definitions Committee and Carl Fuller, Society President



Dr. Gunter Wyszecki, of the National Research Council of Canada, was awarded the Armin J. Bruning Award for his "outstanding contribution to the science of color in the field of coatings technology." The award was presented by Ruth Johnston-Feller, Chairman of the Bruning Committee



Ruth Johnston-Feller and Dennis Osmer (center and right) were presented the 1979 Dry Color Manufacturer's Award for their paper, "Exposure Evaluation—Part II: Bronzing," (see March 1979 JCT, p 37) by Bob Marcus, of PPG Industries, Inc.

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Gus Leep (center), Chairman of the Trigg Awards Committee, presents checks for the best Society reports to Gordon Rook (left), accepting for Sharon Vadnais, Secretary of the Golden Gate Society (First Prize); and Sidney Rubin, Secretary of the New York Society (Second Prize)

zenes as New Coating Binders"—A. K. Chattopadhyay, R. L. Hinrichs, and S. H. Rose, of DeSoto, Inc., Des Plaines, Ill.

#### **DCMA** Award

This award is presented for the best paper prepared on any aspect of the science or usage of color, use of colorants or their behavior, which is presented to the Federation. The Award is presented by the Dry Color Manufacturers' Association.

The 1979 Award was won by Ruth Johnston-Feller, Consultant, Pittsburgh, Pa., and Dennis Osmer, of CIBA-GEIGY Corp., Ardsley, N.Y., for their paper, "Exposure Evaluation: Part II— Bronzing." This paper was published in the Federation's JOURNAL OF COATINGS TECHNOLOGY, in March 1979.

#### **Distinguished Service Award**

This award was presented to James A. McCormick, of the Baltimore Society for Coatings Technology, in grateful acknowledgment of his valuable contributions to the progress of the industry and the Federation while serving as President of the Federation in 1978-79. Mr. McCormick is Vice-President of Inland Leidy, Baltimore, Md.

#### Ernest T. Trigg Awards

Presented annually, these awards are for the two Secretaries of Constituent Societies of the Federation who furnish to the JOURNAL OF COATINGS TECHNO-LOGY the most interesting reports of Society meetings and discussions following the presentation of papers at those meetings. Consideration is given in the judging to general reporting and reader interest, reporting of technical papers and discussions, completeness of coverage and promptness.

Winners for 1979 were:

FIRST PRIZE (\$100)—Marvin Schnall (Troy Chemical Corp.), Secretary of the New York Society for Coatings Technology.

SECOND PRIZE (\$50)—Sharon Vadnais (E. T. Horn Co.), Secretary of the Golden Gate Society for Coatings Technology.

#### **MMA Awards**

Established in 1975 by Materials Marketing Associates, these \$300 cash awards are for notable achievements by Constituent Societies of the Federation for other than Society papers presented at the Federation Annual Meeting.

Winners in the 1979 competition were:

New York Society for the excellence of its educational programs.

Golden Gate Society for contributions to the coatings industry through its 1979 Western Coatings Societies' Symposium and Show.

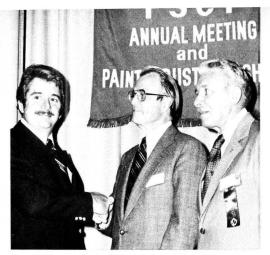
#### A.F. Voss/American Paint Journal Award

These cash awards are presented by the *American Paint & Coatings Journal* for the most constructive papers by Constituent Societies of the Federation, in connection with the research, development, manufacture, or application of the industry's products, or of the raw materials entering into their fabrication. The awards also commemorate the name of A. F. Voss, President of the American Paint Journal Co. from 1931 through 1969.

Winners in the 1979 competition were:



Horace Philipp (left), Chairman of the A.F. Voss/American Paint Journal Awards Committee, with winners: C.L. Aronson, accepting for the New England Society, and A.C. Boyce and P. Hiscocks, accepting for the Toronto Society



Program Awards for best presentation of Society papers were presented by Gordon Allison, Program Awards Committee Chairman, to Lowell Wood (center), accepting for Don Emch, of Northwestern Society, and A. Clark Boyce (right), accepting for S.K. Kambanis, of Toronto Society

FIRST PRIZE (\$200)—"Wet Adhesion—Fact and Fiction"—Toronto Society for Coatings Technology. (S. K. Kambanis, of Reichhold Chemicals Ltd.).

SECOND PRIZE (\$150)—"Evaluation of Inorganic Salts as Flash Rust Inhibitors in an Aqueous Acrylic Coating"—New England Society for Coatings Technology. (Robert Warren, of Cadilac Paint & Varnish Co., Chairman of the Technical Committee).

THIRD PRIZE (\$150)—"Critical Pigment Volume Concentration/Binder Index Relationship. Part I—Observations on the Experimental Determination of CPVC"—Toronto Society for Coatings Technology. (Peter A. Hiscocks, of Canadian Industries Ltd., Chairman of the Technical Committee).

#### **Program Committee Awards**

These awards are presented to individual members of Societies who present Society papers at the Annual Meeting in the best form and manner.

FIRST PRIZE (\$100)—Don Emch (Valspar Corp.), of the Northwestern Society.

SECOND PRIZE (\$50)—S. K. Kambanis (Reichhold Chemicals Ltd.), of the Toronto Society.

#### C. Homer Flynn Awards

Byk-Mallinckrodt Chem. Prod. GmbH, Elcometer, Inc., Morehouse Industries, Inc., O'Brien Industrial Equipment Co., Troy Chemical Corp., and Union Carbide Corp. were recipients of the C. Homer Flynn Awards at the 1979 Paint Industries' Show of the Federation of Societies for Coatings Technology, held October 3-5 at the Convention Center, St. Louis, Mo.

These annual awards are for outstanding exhibits in the Show on the basis of technical excellence, educational value, attractiveness, and novelty.

The prizes (engraved plaques) were awarded as follows:

#### Three Exhibitors Receive Special Citations At 1979 Paint Industries' Show in St. Louis.

Ashland Chemical Co., Neville Chemical Co., and Spencer-Kellogg Div. of Textron, Inc. were cited recently for having been exhibitors for 40 years in the Paint Industries' Show, which is sponsored annually by the Federation of Societies for Coatings Technology.

A plaque commemorating the occasion was presented on behalf of the Federation during the 1979 Paint Show

(the 44th) at the Convention Center, St. Louis, Mo., October 3-5.

SINGLE-BOOTH EXHIBITS: First Prize-

O'Brien Industrial Equipment Co., San

Francisco, Calif. (4 years in the Show);

Second Prize-Elcometer, Inc., Birming-

DOUBLE-BOOTH EXHIBITS: First

Prize-Troy Chemical Corp., Newark,

N. J. (26 years); Second Prize-

Morehouse Industries, Inc., Fullerton,

THREE-OR-MORE-BOOTH EXHIBITS:

First Prize-Union Carbide Corp., New

Byk-Mallinckrodt Chem. Prod. GmbH,

York, N. Y. (44 years); Second Prize-

Melville, N. Y. (15 years).

They bring to nine the number of exhibitors to be so honored by the Federation. In 1975, 40-year plaques were presented to Cities Service Co., Rohm and Haas Co., and Union Carbide Corp.; in 1976, plaques were presented to Reichhold Chemicals, Inc. and Tenneco Chemicals, Inc.; and in 1977, Hercules Incorporated received a plaque.

Deryk Pawsey (right), Chairman of the Paint Show Committee, presented C. Homer Flynn Awards to six companies for the best exhibits in the Paint Show. Shown left to right: Paint Show Manager Frank Borrelle; Roger Moss, of Elcometer, Inc.; William Funk, of O'Brien Industrial Equipment; William Hair, of Morehouse Industries; Elias Singer, of Troy Chemical Corp.; Wolfgang Zinnert, of Byk-Mallinckrodt; and Bill Mainella, of Union Carbide Corp.





Morehouse Industries, Inc.'s Golden Impeller Award is presented by Hugh Purcell to Fred Daniel (right), of Daniel Products Co. The Award recognizes Mr. Daniel's "outstanding service to the chemical processing industry for innovative designs and applications in the field of dispersion technology"

ham, Mich. (6 years).

Calif. (32 years).

#### Representatives From Six International Organizations Meet During Federation Annual Meeting in St. Louis

Representatives of the International Committee to Coordinate Activities of Technical Groups in the Coatings Industry met on October 4 during the Federation of Societies for Coatings Technology Annual Meeting and Paint Industries' Show in St. Louis.

The organizations are:

(1) Federation of Societies for Coatings Technology (FSCT).

(2) Federation of Associations of Technicians in the Paint, Varnish, Lacquer and Printing Ink Industries of Continental Europe (FATIPEC).

(3) Oil and Colour Chemists' Association - United Kingdom (OCCA-UK).(4) Federation of Scandinavian Paint

and Varnish Technologists (SLF).

(5) Japan Society of Colour Material (JSCM).

(6) Oil and Colour Chemists' Association - Australia.

The luncheon meeting was hosted by the FSCT and followed the June 1979 meeting in Paris, at which the ICC was formally established with a Board of Directors.

The principal business of the St. Louis meeting was to review current activities of ICC members and to discuss possibilities for future cooperative efforts. One program the committee will undertake is the preparation of a bibliography of available literature on "Technical Uses of Computers in the Coatings Industry." The FSCT will initiate the work through its Technical Information Services Committee.

The next meeting of the ICC will be held in Amsterdam, Netherlands, June 1980, during the FATIPEC Congress.

The following were present at the meeting:

FSCT—James A. McCormick, President; Elder C. Larson, President-Elect; William H. Ellis, Treasurer; Howard Jerome, Treasurer-Elect; Frank J. Borrelle, Executive Vice-President; Milton A. Glaser, Chairman of Liaison Committee; Fred Daniel, Newell P. Beckwith, and John J. Oates, members of the Liaison Committee; and Andre LaMarre, a member of the Montreal Society who served as interpreter for Jacques Roire.

FATIPEC-Dr. Mart Kooistra, President; and Jacques Roire, Past President.

OCCA-UK—Francis M. Smith, President; and Donald J. Morris.

SLF-Borje Andersson, President.

JSCM—Tatsuo Sato (Painesville, Ohio).

OCCA-Australia-W. John Gutteridge.

#### Paint Research Institute

#### Dr. Orin Keplinger Re-Elected President of PRI for 1979-80

Dr. Orin C. Keplinger, of Valspar Corp., Minneapolis, Minn., has been reelected President of the Paint Research Institute of the Federation of Societies for Coatings Technology, 1979-80.

Other officers elected to serve are: Vice-President—Leonard C. Afremow, of DeSoto, Inc., Des Plaines, Ill.; Secretary—Charles Kumins, of Tremco Manufacturing Co., Cleveland, Ohio; and Treasurer—Howard Jerome, of Spatz/Vane-Calvert Paint Co., St. Louis, Mo.

The remaining Trustees are: Royal A. Brown, of National Paint and Coatings Association, Washington, D. C.; Dr. Philip Heiberger, of duPont Co., Wilmington, Del.; Ruth Johnston-Feller, Consultant, Pittsburgh, Pa.; Peter V. Robinson, of Glidden Coatings & Resins, Strongsville, Ohio; Garmond G. Schurr, of Sherwin-Williams Co., Chicago, Ill.; John Weinmann, of Reichhold Chemicals, Inc., Elizabeth, N. J.; and Marco Wismer, of PPG Industries, Inc., Allison Park, Pa.

Dr. Raymond R. Myers, University Professor at Kent State University, Kent, Ohio, continues as Research Director of PRI.



Executive Committee of the Federation, 1979-80. From left to right: Willy C.P. Busch, of PPG Industries, Inc., Houston, Tex.; Immediate Past-President James A. McCormick, of Inland Leidy, Baltimore, Md.; President Elder C. Larson, of Shell Development Co., Houston, Tex.; Treasurer Howard Jerome, of Spatz/Vane-Calvert Paint Co., St. Louis, Mo.; President-Elect William H. Ellis, of Chevron Research Co., El Segundo, Calif.; A. Clark Boyce, of Nacan Products Ltd., Toronto, Ontario; and William Mirick, of Battelle Memorial Institute, Columbus, Ohio

## **1979 Paint Show Exhibits**

The 1979 Paint Industries' Show of the Federation of Societies for Coatings Technology was held at the Cervantes Convention Center, St. Louis, Missouri, October 3-5. With 145 exhibitors in 329 exhibit spaces, it was the largest Show in Federation history.

As a continuing service to JCT readers, we present (in the following pages) a description of the products and services which highlighted the exhibits of exhibitor companies. These are reprinted exactly as published in the Federation's "Paint Show Program," which was given to all registrants at the convention.

Any requests for information from the exhibitor companies should be sent to the JCT office (1315 Walnut St., Philadelphia, Pa. 19107). We will forward all inquiries.—Ed.

#### ACETO CHEMICAL CO., INC. Flushing, N.Y. 11368

The company is exhibiting a variety of coatings raw materials. These include: organotin compounds; anti-skinning agents; electrostatic and aerosol spray paint additives; and UV photoinitiators.

#### AIR PRODUCTS AND CHEMICALS, INC. Allentown, Pa. 18105

The polymer chemicals section of the exhibit features the WAVE<sup>TM</sup> products, revolutionary emulsions with a distinctive new name. These emulsions offer significant cost savings to paint manufacturers. The acetylenics chemicals section of the booth deals with the benefits of Surfynol<sup>®</sup> nonionic surfactants. Live demonstrations show how these products improve coverage on oily surfaces and also defoam. Research scientists are on hand to discuss applications.

#### ALCAN INGOT AND POWDERS Elizabeth, N.J. 07207

On display are water-dispersible aluminum pigments which are easy to mix as needed. Also featured are nonleafing aluminum pigments for beautiful metallescent finishes and the company's bright-reflective aluminum pigments. Asphalt is a good roof coating and with the addition of these aluminum pigments, it becomes a great roof coating.

#### ALPINE AMERICAN CORP. Natick, Mass. 01760

The booth features the company's wide range of size reduction, air classification, and sieving equipment. Special emphasis is on the Mikroplex MPW wide chamber, with a cut point down to five microns, suited for precise classification of sticky pigments and those with a tendency to agglomerate. Mikroplex MPVI spiral air classifier, a selfcontained unit which separates coarse and fines internally with a cut point from 200 down to eight microns, and Model AJS-200 laboratory air jet sieve for sieving down to ten microns with precisely repeatable results are also displayed.

#### ALUMINUM CO. OF AMERICA Pittsburgh, Pa. 15219

The exhibit features aluminum pigmented roof coatings, maintenance paints, and automotive finishes. Specifications and product data is available for all of the company's powder and flake grades. A limited edition aluminum chess set will be on display and will be awarded to a visitor through a drawing at the conclusion of the show.

#### C. M. AMBROSE CO. Redmond, Wash. 98052

Equipment on display includes the Ambrose PFSP, a product fillerclincher for five gallon containers and the Model 310 filler-sealer used to package from quarter pints to gallons. The newest equipment available for display is the company's new 322-F, a four posted closer unit which can be supplied to seal both five-gallon plastic pails and metal lug lid containers.

#### AMERICAN FELT & FILTER CO., INC. Newburgh, N.Y. 12550

The company is exhibiting its complete line of filter products. The new model RBX-A, compact, highly efficient, filter bag system designed for use as an in-line liquid filter, is now available. IFC molded industrial filter cartridges offer savings of up to 80% with "Best by Test" data available on request. A complete line of pressure filters features the micron-rated, quick-change, Snap-Ring filter bag for effective filtration with greatly reduced downtime.

#### AMERICAN HOECHST CORP. Somerville, N.J. 08876

On display is a complete line of organic pigments, pigment dispersions, additives, and resins for automotive, industrial, and trade sales paint systems.

#### AMERICAN NEPHELINE CORP. Columbus, Ohio 43214

Both the Minex and the Lawson-United series of extender pigments are featured in a comparative exposure series versus competitive extenders. Special emphasis is placed on exterior resistance to frosting properties.

#### APPLIED COLOR SYSTEMS, INC. Princeton, N.J. 08540

The company is exhibiting its 600M Computer Color Control System with remote terminals and the LABCAL/COGIT Computer Program Package for paint and coatings formula design and inventory control. The 600M System features simultaneous multi-terminal operation and allows simultaneous operation of color programs and business programs, such as inventory control.

#### ASHLAND CHEMICAL CO. Columbus, Ohio 43216

On display is a complete line of solvents, exempt solvents, and specialty chemicals for paint formulation. Information about the company's computerized reformulation service for paint manufacturers is also provided.

#### ATLAS ELECTRIC DEVICES CO. Chicago, III. 60613

On display is the new Model Ci65-W xenon arc Weather-Ometer, the Uvcon-UV fluorescent/condensation screening device, the "all new" UV2, and an exhibit of our subsidiary, South Florida Test Service, Inc.

#### AUTOMATION INTERNATIONAL CORP. Norwalk, Conn. 06854

Films and photos of automatic rotary piston fillers and semiautomatic piston fillers for paint, varnishes, glue; carton packers; carton emptying machines; wrap-around case packers; palletizing equipment; and depalletizing equipment are on display. Experienced engineers are available to discuss requirements for either standard machinery or for specialized automation systems.

#### B.A.G. CORP. Dallas, Tex. 75228

The booth features disposable and reusable one ton containers for the handling of dry flowable solids and semi-bulk systems for pigments and fillers to the paint industry.

#### B.A.S.F. WYANDOTTE Holland, Mich. 49423

The main feature of this booth is a graphic demonstration of our new organic yellows for industrial, automotive, and trade sales coatings. In addition to a high degree of opacity, these items offer excellent chemical resistance, lightfastness, etc., properties. A second section presents the broad range of products handled by the company. The third section allows visitors to see a pictorial presentation of the Division's commitment to the coatings industry.

#### BELTRON CORP. Farmingdale, N.J. 07727

#### BENNETT'S COLORANT DIVISION Salt Lake City, Utah 84110

This exhibit features "Deltasperse," new high-strength, trade sales, universal in-plant tinting colorants that are volumetrically standardized. In addition, both the "Decorator" and "Dimensions in Color" programs are on display.

#### BLACKMER PUMP DIV. Dover Corp. Grand Rapids, Mich. 49505

Products exhibited include the company's XLW Abrasive Liquids pump. This pump has been manufactured with wear resistant parts and has been designed to handle abrasive liquids such as paints and inks. Other pumps for solvents, resins, and varnish are also on display.

#### BROOKFIELD ENGINEERING LABS., INC. Stoughton, Mass. 02072

The company's complete line of laboratory and process viscometers for the measurement and control of viscosity is shown, along with a cone and plate type viscometer for measurement of paints and paint products in the extremely low shear-rate range. This viscometer is capable of measuring viscosity products at shear rate less than 1 sec<sup>-1</sup>.

#### BUCKMAN LABORATORIES, INC. Memphis, Tenn. 38108

The exhibit features the multiple properties imparted to paint by Busan 11-M1, a modified barium metaborate monohydrate. These properties include both corrosion and mold resistance. Also featured are Busperse 47 and Busperse 55 (dispersants) and Busperse 53 and Busperse 48 (defoamers).

#### BYK-MALLINCKRODT CHEM. PROD. GmbH Melville, N.Y. 11747

Problem solutions in modern coatings systems such as high solids, water-reducible, and other coatings systems conforming to the new regulations are displayed using the company's current line of coating additives. The enlarged technical sales force is on hand to answer questions for specific applications of the additives. The display of instruments includes a Kesternick cabinet, in addition to the present line of Glossmeters, the Dynometer, and the Pendulum Hardness Tester.

CABOT CORP. Cab-O-Sil Div. Boston, Mass. 02110

On exhibit is a modular display featuring the use of fumed silica in the paint industry. A technical paper on dispersion is available.

#### CARGILL, INC. Minneapolis, Minn. 55440

The company's innovative computer answer center displays information on water-reducible, high solids, powder, and UV cure coating systems. The computer keyboard, TV information display, and picture projector are set up for attendee's selection. The new resins introduced are fast air-dry water-reducible systems, air-dry and bake high solids systems, and lower temperature cure powder coating systems. Also, new polyester powder coating resins are added to the present line. Spray and flow coating formulations, application, and exposure information is available.

#### CDI DISPERSIONS Newark, N.J. 07114

A complete line of color dispersions for coatings, inks, plastics, building material products, and paper is shown. Carbon black dispersions are a speciality, and custom dispersions are available.

#### CELANESE CHEMICAL CO., INC. Dallas, Tex. 75247

New products and application technology for ultra violet and electron-beam cured systems for adhesives, inks, and coatings are displayed. Emphasis is given to the use of multifunctional acrylates, monomers, and esters in high solids and water-borne systems as possible solutions to energy conservation and environmental problems.

#### CELANESE PLASTICS & SPECIALTIES CO. Chatham, N.J. 07928

While the exhibit features a broad line of coatings polymers, particular emphasis is given to water-reducible epoxies and curing agents for marine and industrial maintenance coatings, water-reducible acrylics for industrial baking coatings, acrylic and vinyl-acrylic emulsions for trade sales coatings and acrylated epoxies for radiation-curable coatings. Displays, highlighting specific performance characteristics of these various resins, are also featured.

#### CELLOMER AND CELLOFILM CORPS. Newark, N.J. 07105

Cellomer manufactures coating vehicles, principally alkyd resins, in Newark, N.J. Featured are zero-T flexibility polyesters for coil coatings, high solids resins, and water-reducible systems.

Cellofilm produces nitrocellulose and other thermoplastic resin solutions in Wood-Ridge, N.J. and in Chicago, III. Through the Fayette Division, both locations also offer uncut nitrocellulose. Featured is CV-150, a new modifying resin for fingernail lacquer formulations.

#### CHICAGO BOILER CO. Chicago, III. 60614

The exhibit features the horizontal dyno-mills and the "red head" small media mills. Various grinding medias used with these mills are also displayed.

#### CITIES SERVICE CO. Columbian Chemicals Div. Tulsa, Okla. 74102

The exhibit demonstrates the versatility of Mapico<sup>®</sup> synthetic iron oxides and Raven<sup>®</sup> carbon blacks in a wide range of industrial and trade sales applications as diverse as wood stains and automotive finishes. Highlighted are unique Mapico iron oxides/organic pigment blends, easy-dispersing yellows for energy and cost savings, and a complete line of high technology furnace carbon blacks.

#### COLOR CORP. OF AMERICA Div. of The Valspar Corp. Rockford, Ill. 61101

A completely new color system is being introduced. The new program contains 775 colors divided into 10 families with a further division into cool, warm, and neutral colors especially designed for easy decorating. A separate section contains 75 one-coat exterior colors. All colors are presented to the consumer with a molded plastic display in a concert-hall design that literally surrounds the customer with color. Other aids include a Color Album and Fan Deck plus a dominant Exterior Color Display featuring 58 giant-size, one-coat colors. Two new color cards, one featuring 58 exterior colors, the other containing 60 interior colors, supplement the new color offering.

#### COSAN CHEMICAL CORP. Carlstadt, N.J. 07072

C-FLOC-36, a new primary flocculent for latex wash water, is introduced. Also featured is COSAN PMA-100, a bactericide/fungicide; Cocures and Cotins, organo metallic catalysts for two component high solids coatings, adhesives, sealants, and caulks.

#### CUSTOM CHEMICALS CO. Elmwood Park, N.J. 07407

As a first time exhibitor, the company is highlighting Mikrolour® pigment dispersions. Available as micropulverized, nondusting powders and custom particle geometries, these dispersions are supplied at 15-80% pigment concentrations. Aqueous and industrial/solvent types are highlighted. Industry samples are displayed which demonstrate the unique quality obtainable with Mikrolour in a variety of transportation, industrial and household product finishes.

### CUSTOM SCIENTIFIC INSTRUMENTS, INC. Whippany, N.J. 07981

This booth displays two material testers: the two foot flame tunnel for measuring flame spread; and the fire test cabinet for fire retardant test by determining the weight loss and char volume.

#### DANIEL PRODUCTS CO. Jersey City, N.J. 07304

The exhibit introduces two new carbon black dispersions and three new Slip-Ayd Surface Conditioners. BB 1379 High Jetness Masstone Black in acrylic and AL 329 High Strength Tinting Black provide superior performance in most solvent systems. Slip-Ayd SL-404 and SL-425 are fine particle dispersions for solvent and water systems of an FDA approved polymeric wax. SL-300 is a high solids dispersion of an exceptionally hard polyethylene for use in aqueous systems. Also featured is a comprehensive line of pigment dispersions and chemical additives for all water thinned coatings.

#### DEGUSSA CORP. Teterboro, N.J. 07608

Featured is Special Black 6, a new color black for producing high jetness and superior blue undertone in coatings and plastics, along with Aerosil<sup>®</sup> 200 for pigment suspension; Aerosil R972 for corrosion resistant coatings; and Flatting Agent OK-412 for superior flatting effect.

#### DESERT MINERAL PRODUCTS Ontario, Calif. 91762

On display is talc which is mined and milled by the company for use in the paint and coatings industry.

#### DIAMOND SHAMROCK CORP. Process Chemicals Div. Morristown, N.J. 07960

The display spotlights two of the company's high performance products: Nopcosperse<sup>®</sup> 44 universal pigment dispersant and Nopcoside<sup>®</sup> N-96 fungicide. The recently introduced Nopcosperse 44 is gaining rapid market acceptance as a universal pigment dispersant because it delivers maximum efficiency over a wide range of formula variations. N-96, an industry standard, is a proven nonmercurial fungicide for both solvent and water-base paint systems. Also featured in the display is the Foamaster series of antifoaming agents. Additional material on the company's full line of cost-effective wetting agents, stabilizers, thickening agents, dispersants, and fungicides is also presented.

#### DIANO CORP. Woburn, Mass. 01801

On display is the Match-Mate 3000 Computer Color Matching System featuring the new Match-Scan Spectrophotometer, with its classic dual-beam optics, unique "zero reference" black light trap, constantbandwidth grating monochromator, and high sensitivity photomultiplier tube. The Match-Mate 3000 also features the company's Match-Pak II color matching software, with its advanced mathematical model and numerous user convenience features. Match-Pak II will produce the most accurate initial matches and most useable batch corrections possible.

#### D/L LABORATORIES New York, N.Y. 10003

"Consultants to the Industry" is the theme of the exhibit. The booth features examples of the services provided to the coatings, sealants, and plastics industry, including formulation, testing, evaluation, corrosion studies, inspection, industry and market surveys, market development preparation of specifications and manuals, personnel training, and legal assistance. Personnel are available to discuss your ideas or problems.

#### DRAISWERKE, INC. Northvale, N.J.

The company, a German manufacturer of wet milling and dispersing machinery, presents its newest additions to its product line. These additions include continuous agitation Bead Mills (Perl Mills) for heat sensitive and highly viscous products, as well as a Direct Dispersion Mill which completely eliminates all pre-mixing and pre-dispersing. The highlight of the exhibit is the unveiling of a newly designed Perl Mill which promises to increase versatility and simplicity of mill operation.

#### DRESSER INDUSTRIES, INC. Dresser Minerals Div. Houston, Texas 77005

Dress-tex UF, a paint filler, and other paint/coatings products are featured. On display is also a photo display of the company's plant.

#### DSET LABORATORIES, INC. Phoenix, Ariz. 85029

The company's operations, representing the largest and most modern outdoor weathering and solar testing facility in the world, are described. Featured are the various weathering techniques, programs, and services available to the industry.

#### EASTMAN CHEMICAL PRODUCTS, INC. Kingsport, Tenn. 37662

The exhibit features the company's line of solvents for water-borne and high solids coatings; coalescing aids for water borne coatings; refinish primers and base coat emulsions for automotive applications; glycols and intermediates for high solid appliance coatings, flexible coil coatings, primers, and water-borne coatings; cellulose acetate butyrate for wood finishes; cellulose acetate propionate for inks and overprints; and chlorinated polyolefin primers for polyethylene and polypropylene.

#### EBONEX CORP. Melvindale, Mich. 48122

The exhibit presents a series of low-priced, easy-to-disperse, low oil absorption black pigments, data sheets, brochures. Finished products manufactured from these pigments are available for inspection.

#### ELCOMETER INC. Birmingham, Mich. 48012

This exhibit features a new range of "pocket size" Digital Coating Thickness Gauges for both ferrous and nonferrous bases, also a new range of Analogue "pocket size" Thickness Gauges for increased stability, reliability, and ease of application for measurements on both of these bases. The well-proven Inspector and Elcometer Thickness Gauges, together with instruments for measuring adhesion of coating, pin holing, roughness of substrates, are also displayed. Practical demonstrations can be undertaken, so, if you have a problem which the company can solve, please bring samples for testing.

#### ENCAPSULAIR, INC. Wheeling, Ill. 60090

The booth features an exciting breakthrough in paint technology that permits raw material cost reductions of up to 20% in latex flat paints. The company, a division of the Insilco Corp., Meridan, Conn., is the exclusive U.S. distributor of Spindrift® beads, a licensed product of Dulux Australia Ltd. Spindrift® beads, used successfully in Australia for four years, are pigmented microvoid-containing beads that permit titanium dioxide (TiO<sub>2</sub>), latex, and extender pigment replacement for raw material cost reductions and improved product performance.

#### EPWORTH MFG. CO., INC. South Haven, Mich. 49090

A production size SWMill is on display, cross-sectioned to expose the features of the machine which disperses small and medium size batches of hard-to-grind pigments in an hour or less. Catalogs are available and experts are on hand to discuss a full line of ball mills, sand mills, mixing equipment, and various grinding media of all types and sizes.

#### FAWCETT CO., INC. Macedonia, Ohio 44056

On display are the company's air-driven stirrers and mixed flow impellers.

#### FEDERATED METALS CORP. Subsidiary of ASARCO, Inc. New York, N.Y. 10005

Information is available on the various grades of zinc dust produced by the company, including L-10 which combines a high zinc metal content with a very low micron particle size. L-10 is used extensively for coil coating applications.

#### FEDERATION OF SOCIETIES FOR COATINGS TECH. Philadelphia, Pa. 19107

Featured is the "Paint/Coatings Dictionary," compiled by the Federation Definitions Committee. The 632-page publication contains over 5500 coatings-related terms and includes a thesarurus and an extensive bibliography. Also featured is the new edition of "Pictorial Standards of Coatings Defects," along with available Federation slide/tape training programs. Publications on display also include: Journal of Coatings Technology, 27 units in the Federation Series on Coatings Technology; Infrared Spectroscopy; Fundamentals and Problems of Color; Mildew Defacement of Organic Coatings; and the 1979 Membership Directory (Year Book).

#### FILTER SPECIALISTS, INC. Michigan City, Ind. 46360

<sup>•</sup> Liquid bag-type filters are on display. Sizes range from miniature models to models with 1500 GPM capacity. Filter bags are available from 1 to 800 microns in a wide variety of shapes and sizes. Technical personnel are in attendance to help with any filtration applications or problems.

#### C. FRICKE CO. Granite Falls, Wash. 98252

A new semi-automatic binary paint filling and closing machine, capable of filling and closing one gallon through one-half pint containers, is on display, as well as a new automatic five gallon labeling machine which labels five gallon containers prior to or after filling.

# GAF CORP.

#### New York, N.Y. 10020

V-Pyrol, a radiation curing chemical is featured along with the firm's complete line of surfactants for the paint/coatings industry.

# GAIL INDUSTRIES

#### Cedar Rapids, Iowa 52406

The "Stripper," Gail's high pressure washer, makes fast work of washing and stripping paint from surfaces. Scraping and wire brushing will seem antiquated in comparison. The "Stripper" features a wet sandblast attachment for tough assignments and a down-stream injector with flow meter for applying chemicals. The exhibit includes eight models featuring both gasoline engines and electric motors. Demonstrations are held during the entire show.

#### GARDNER LABORATORY, INC. Bethesda, Md. 20014

A new era in colorimetry begins as the company displays its XL-805 Tristimulus Colorimeter utilizing advanced microprocessor technology, as well as their Glossgard II Glossmeter, completely portable and selfcontained, and their critical color matching Colorgard Light Booth. A wide selection of equipment for precise measurement and assessment of appearance and color properties as well as physical testing, viscosity, and laboratory apparatus is available.

#### GENERAL ELECTRIC CO. Silicone Products Dept. Waterford, N.Y. 12188

On display are silicone resins and additives for formulating protective coatings that provide high heat resistance; weather, ultraviolet, and hard radiation resistance; and flexibility and gloss retention.

#### GEORGIA KAOLIN CO. Elizabeth, N.J. 07207

This company offers the widest range of calcined, delaminated, and hydrated aluminum silicate extender pigments and information on their application in the paint industry. Information on the use of the slurry type is also available.

#### W. R. GRACE & CO. Davison Chemical Div. Baltimore, Md. 21203

The exhibit features the family of Syloid® micron-sized silica flatting agents for the Paint Industry.

# GRACO, INC.

Minneapolis, Minn. 55440

Featured is the Graco Auto Tint 2000 Color Processor, which automatically measures and dispenses paint colorant. This processor is recommended in high volume trade paint outlets for accurate, repeatable paint inting. With average productivity increases of 105% on multiple can orders, the Auto Tint frees personnel to perform other duties. The company's positive displacement metering pumps require no calibration. Color formulas are read with a light pen from standard bar codes or through pushbutton manual entry.

#### HALOX PIGMENTS DIV. Hammond Lead Products Pittsburgh, Pa. 15220

Formulating guidelines for paint producers using the company's pigments are presented. Also demonstrated is how these pigments perform as economical and effective corrosion inhibitors and in controlling tannin-stain bleed-through. For application in both solvent-thinnable paint systems and water-emulsifiable and water-soluble vehicles, these products offer the improved stability of finished paint as well as compatibility with a broader range of sensitive systems.

#### HARMON COLORS CORP. Haledon, N.J. 07506

Exhibited are 12 new pigments for architectural latex finishes, featuring new Yellow Shade Phthalo Greens and new Fanchon Yellows, Oranges, and Reds; and 14 new pigments developed for industrial finish applications, shown in deeptones, metallics, and tints. A demonstration of lead-free color matches for well-known industrial colors is also highlighted.

#### HARSHAW CHEMICAL CO. Cleveland, Ohio 44106

The exhibit features Aurasperse aqueous dispersions, Lancer thermoplastic dispersions, Sun and Meteor inorganic colorants, and the Color Concept Universal Colorant System including machine colors and professional tints. Visitors are invited to participate in a color survey.

#### HENKEL CORP. Minneapolis, Minn. 55435

The exhibit presents new developments in three basic coatings interest areas: high performance C-Cure<sup>®</sup> acrylic/urethane resin systems; Versamid<sup>®</sup> polyamide/epoxy resin systems for maintenance coatings; and the company's coating additives for both water-borne systems and solvent-containing paints.

#### HERCULES INCORPORATED Wilmington, Del. 19899

The exhibit feaures Hidracol<sup>®</sup> easy dispersing pigments to aqueous trade sales and industrial paint systems. Displayed are the broad spectrum of the company's products and services for the coatings industry, including high performance inorganic pigments; Natrosol B, a bacteria resistant thickener for latex paints; nitrocellulose; ethyl cellulose; Parlon; Hercoflat; pentaerythritol, and, new on the scene, plastic paint pails.

#### HILTON-DAVIS CHEMICAL CO. DIV. Cincinnati, Ohio 45237

The theme of the exhibit is "Cetting Ready for the Future." Displayed are the broad spectrum of the company's technical services and water-based colorants for the coatings industry, including new and innovative concepts for water-based finishes of the future. Highlighted are the environmentally compliant features of the colorant systems.

#### HOCKMEYER EQUIPMENT CORP. Harrison, N.J. 07027

On display is a Model HVR-25 heavy duty DiscPerser, a Model HV-15 standard duty DiscPerser, and a Hockmeyer Series 1000 Agitator Drive.

#### HOOKER CHEMICAL CORP. Niagara Falls, N.Y. 14302

Ferrophos<sup>®</sup>, an enhancer for zinc-rich protective coatings that provides improved weldability, while economically retaining full corrosion resistance, is featured. Ferrophos<sup>®</sup> enhancer was developed as a partial substitute for zinc-rich primers.

#### J. M. HUBER CORP. Havre de Grace, Md. 21078

New low cost silica products for flatting, plastic and thickening applications are displayed. Exhibits and data are available on new use for the functional spacer, Zeolex<sup>®</sup> 80. Water-borne formula containing Zeothix<sup>®</sup> 95 shows clarity, film smoothness, and good suspension. Information regarding new low cost silica for printing ink application and clay slurry handling systems is available.

#### HUNTER ASSOCIATES LABORATORY, INC. Fairfax, Va. 22031

The company exhibits their line of instrumentation which is directly related to the measurement of color and gloss of paints. This includes the D54P-5 Spectrophotometer, the D25-9 Microprocessor Colorimeter, and the D48 and D52 Glossmeters. The new D25-9 Colorimeter has proven to be a successful instrument with features which include automatic standardization, four color scales plus WI and YI indices, standard and product storage, programming capability, and a special option with batch correction designed especially for the paint industry.

#### IBM INSTRUMENT SYSTEMS White Plains, N.Y. 10601

The following units, which allow paint manufacturers to quickly analyze a wide spectrum of colors and color differences, are exhibited by the company: the IBM 7842 Color Analyzer II-a system which combines, in one integrated unit, advanced optical instrumentation and digital computing techniques; and the IBM 7410 Color Sensor Analyzer-a new pre-programmed microprocessor-based color analyzer.

#### INTERNATIONAL MINERALS & CHEM. CORP. McWhorter Resins Carpentersville, III. 60110

Come visit IMC's McWhorterville for a color-coded (and coated) tour of a typical modern village, showing the many end uses of the company's resins in high solids, water solubles, and emulsions throughout the community. You'll see why these resins, the problem solvers, have become your single, national source for all your resins needs.

#### INTERNATIONAL MINERALS & CHEM. CORP. NP and Industrial Chemicals Divisions Des Plaines, III. 60016

Want better protection for your solvent system from weather, water, and corrosion? NIPAR S-30 delivers it. Want outstanding performance from your latex and waterborne paints? AMP-95 provides it. Want superior film properties in your water-reducible alkyd resins? You'll get them with DMPA®. Want high gloss, gloss retention, and durability in your alkyd resins? Add TRIMET®. Want proof? Visit IMC's NP and Industrial Chemicals divisions in the company's booths.

#### INTERSTAB CHEMICALS, INC. New Brunswick, N.J. 08903

Visit the world of Interstab. A manufacturer of a complete line of additives for the paint and coatings industry since 1931, it is a company that commits itself to you, the consumer, from the very beginning. The company is a wholly owned subsidiary of Akzo Chemie, Amersfoort, The Netherlands. Interstab sales representatives are on hand for your convenience.

#### JOHNS-MANVILLE Denver, Colo. 80217

The company's Celite® functional fillers, Micro-Cel® functional extenders, and a complete line of yarn wrapped and fiber glass filter cartridges are on display.

#### KELCO DIV. Merck & Co., Inc. San Diego, Calif. 92123

This booth features ZANFLO and ZANFLO-10, biogum polymers which function as a hydrophilic colloid to thicken, suspend, and stabilize aqueous-based systems.

# KENRICH PETROCHEMICALS, INC. Bayonne, N.J. 07002

Exhibit features the use of Ken-React<sup>®</sup> Titanate Coupling Agents to replace metal chromates in polyamide cured epoxy, increase productivity, improve pigment dispersion, reduce viscosity, promote adhesion, lower bake temperatures, achieve high solids and solvent elimination, prevent corrosion, improve acid resistance, and increase scrubbability and prevent flash rusting in latex paint. Also shown are the non-mutagenic epoxy reactive diluents Ken Kem<sup>®</sup> CPE (cumylphenyl glycidyl ether) and Kenplast<sup>®</sup> ES-2 (cumylphenyl acetate).

LABELETTE CO. Forest Park, Ill. 60130

#### THE LENETA CO. Ho-Ho-Kus, N.J. 07423

As manufacturers and suppliers of paint test charts and test equipment, the company is distributing its current catalog and the exhibit includes many of the products described therein. Among the products on display are the recently developed Leneta Leveling Test Blade and Levelness Standards, the Leneta-EZ Paintr Certified Standard Paint Brush, a newly designed paint test chart of high utility and interest, wirewound drawdown bars, birch and wood veneer test panels, and various ink test sheets.

#### LIQUID CONTROLS CORP. North Chicago, III. 60064

The display features a series of positive displacement liquid meters. These include an M-7 NX 100 GPM, 150 PSI electric trade show demonstrator; an MS-7 100 GPM, 150 PSI spherical two-case steel case model; and an M-7-K flowing trade show demonstrator.

#### 3M CO. Chemical Resources Div. St. Paul, Minn. 55101

This booth features the company's epoxy curing agents and related products. These unique materials find utility in maintenance coatings, marine coatings, product finishing, and flooring. The primary features of the curing agents are generally low toxicity, rapid cure rates, the ability to cure at low temperatures, and the ability to cure in the presence of moisture. The curing agents can be employed in high solids and 100% solids coatings formulations.

MACBETH DIV. Kollmorgen Corp. Newburgh, N.Y. 12550

The company presents its MS-4045 On-Line System, specifically designed for coil coatings applications; its line of Building Block Products for color requirements, including the MS-2000 Spectrophotometer and MC-1010S Colorimeter; its Formulation/Batch Correction system; the new SPL-75B SpectraLight Color Matching Booth; and Munsell products, including the Book of Color, the Color Tree, and the Farnsworth-Munsell 100 Hue Color Vision Test.

#### MADISON INDUSTRIES, INC. Old Bridge, N.J. 08857

The exhibit features the company's Zinc Dust #5 and Zinc Dust #8, two products widely used by the paint industry to impart corrosion resistance to coatings. On display are samples of zinc dust, as well as numerous pictures of Madison's production and laboratory facilities.

#### MATEER-BURT CO. Wayne, Pa. 19087

Featured is the new 704 series Burt Roll-Through Labeler. The machine is capable of labeling all sizes of paint containers from 1/4 pint through imperial gallons. Bail orientation assures precise registration of paint can bail ears with die-cut labels. Also shown is a new pressurized pasting system for optimum label paste control and application.

#### MEADOWBROOK CORP. Subsidiary of T. L. Diamond & Co., Inc. New York, N.Y. 10020

On display are the high quality zinc dust pigments produced at the company's West Virginia smelter. Included is its low micron high metallic zinc dust.

#### MERCK CHEMICAL DIV. Rahway, N.J. 07065

Merck, the biocide innovator, presents a family of anti-microbials plus extensive experience and expertise to resolve your preservation and mildew problems. Products offered are MERBAC 35, METASOL TK-100, METASOL D3T, METASOL D3TA, and METASOL TK-100 Dispersion W. Consult with our technical experts at the Merck Biocide Information Center.

#### MILLER PAINT EQUIPMENT, INC. Addison, Ill. 60101

A high-speed tinting station is demonstrated. The new AccuTinter computerized colorant dispenser is shown to provide speed, accuracy, and ease of operation. Solid state, integrated circuitry allows for programming of formulas in permanent memory. Push-button operation for automatic tinting of fives, gallons and quarts. High-speed Cyromixer completes tinting/mixing operation within one minute.

#### MiniFIBERS, INC. Weber City, Va. 24251

The company reaffirms its position as an innovator in the uses of short cut synthetic fibers for their reinforcing, thixotropic and insulating properties in coatings, putties, sealants and allied products. Fibrillated polyethylene is demonstrated as an asbestos replacement. Other synthetic fibers, processed in various lengths and deniers, are exhibited for a multitude of end uses.

#### MODERN PAINT AND COATINGS Atlanta, Ga. 30328

Complimentary copies of the October Show Issue are being distributed at the booth. The Paint Red Book, the only directory in the coatings field, is on display, as are technical books of other publishers available from Palmetton Publishing Co.

#### MONTEDISON USA, INC. New York, N.Y. 10036

The booth features new pigment technology involving arylide and diarylide pigments reacted on the surface of  $TiO_2$  as a nucleus. In addition, new 1979 developments are demonstrated in the Color Index classes of Pigment Yellow 98 and Pigment Yellow 74. The company's pigment facilities are shown in large color photographs.

#### MOREHOUSE INDUSTRIES, INC. Fullerton, Calif. 92633

The display features a multi-vessel sandmill. Exhibit also includes new laboratory high-speed disperser with hydraulic lift, and a new, long wearing, plastic impeller.

#### MOZEL CHEMICAL PRODUCTS CO. St. Louis, Mo. 63110

Displayed is a replica of the river steamers that once plied the waterways. The Spirit of St. Louis is the place to meet your friends and business associates. You may leave or receive messages at the message board. Personnel are available to assist you in finding your way around St. Louis. Complimentary soft drinks, tables, and chairs provide a moment of relaxation between tours of the exhibition.

#### MYERS ENGINEERING Bell, Calif. 90201

On display is a large dual shaft disperser for high viscosity products. Also shown is a medium size single shaft disperser.

#### NATIONAL ASSOC. OF CORROSION ENGINEERS Houston, Tex. 77084

On display is technical literature concerning the protection of materials and equipment by use of coatings. Information is also available on membership services of the NACE.

#### NETZSCH BROTHERS, INC. Lionville, Pa. 19353

The exhibit includes a range of horizontal and vertical Agitator Bead Mills, featuring patented agitation and separation systems, which are suitable for all aspects of high quality paint manufacture. Also shown is a range of grinding media suitable for operation with the mills and, from the Netzsch Pump Div., Progressive Cavity Pumps for pumping pigment slurries, resins and aqueous materials.

#### NEVILLE CHEMICAL CO. Pittsburgh, Pa. 15225

Exhibit features the company's wide range of petrochemical derived hydrocarbon resins, Cumar® coumarone-indene resins, and Unichlor® chlorinated paraffins. Of particular interest is technical information on the utilization of the firm's resins and chlorinated paraffins in coating systems and in exempt solvents. Data on Unichlor 5722 and 5510 chlorinated paraffins, two nonfogging plasticizers, developed for application in polysulfide based sealants, are also available. Technical representatives are on hand to discuss Neville's capabilities and products for the coatings industry.

#### NEW WAY PACKAGING MACHINERY, INC. Hanover, Pa. 17331

On display is the Model EP wraparound labeler, set to handle gallon paint cans with bail ears, which includes necessary timers with Vansco lap plate glue system, and explosion proof motor, switch, and wiring. Also shown is the SENSE-Matic case sealer for top and bottom sealing. It includes such standard features as Vansco spray glue system, interlocked guarding, torque control on drive unit, spring loaded flight bars, dial-apattern glue system, explosion proof wiring, cantilevered design for easy access, and four-minute changeover.

NL INDUSTRIES, INC. NL Chemicals Hightstown, N.J. 08520

The exhibit includes the firm's broad line of rheological additives and pigments, supported by a seasoned staff of innovative technical experts committed to helping customers make better coatings. The exhibit features graphic evidence of the effectiveness of Rheolate 1, NL's new rheological additive for acrylic latex paint.

#### NYCO, DIV. Processed Minerals, Inc. Willsboro, N.Y. 12996

The company is exhibiting data on NYAD® wollastonite (calcium metasilicate) and Wollastokup® surface modified wollastonite. Technical data emphasizes wollastonite as pH buffer in acrylic and PVA systems; in zinc oxide containing exterior house paints to prevent gellation; and in water-borne metal coatings aiding blister prevention. Data is also available on surface modified wollastonite to aid corrosion resistance in industrial primers.

#### O'BRIEN INDUSTRIAL EQUIPMENT CO. San Francisco, Calif. 94124

A unique universal paint filler is displayed. A rental evaluation program is explained whereby a brand new machine can be tried out in your paint factory. The Level Tender is also displayed. This unit can control liquid levels in one or two tanks and prevent overflow by stopping a pump or closing a valve.

#### OMYA, INC. Proctor, Vt. 05765

Featured are the Hydrocarb® series of fine and ultra-fine wet ground calcium carbonates for application in gloss, semi-gloss and flat trade sales and industrial finishes; specially treated carbonates–WS and Albares; Omyacarb fine ground carbonates produced in California, and Vertal platy tales, from Vermont.

#### PAINT RESEARCH INSTITUTE Philadelphia, Pa. 19107

#### PENN COLOR, INC. Doylestown, Pa. 18901

Innovation, advancement, and technical service in pigment dispersion technology are highligted at the exhibit. Along with quality dispersion lines which currently service the coating, ink, and plastic industries, the company also features the latest advancements in water-borne and radiation curable pigment dispersions.

#### PENNSYLVANIA GLASS SAND CORP. Pittsburgh, Pa. 15235

The exhibit features information documenting the benefits of using both Min-U-Sil (micron-sized silica) and Supersil (custom ground silica) in both conventional and powder coatings.

#### THE PFAUDLER CO. Rochester, N.Y. 14603

Specialists are on hand to discuss the company's resin reactor systems, solvent recovery wiped film evaporator systems, and rotary piston paint fillers.

#### PFIZER, INC. MPM Div. New York, N.Y. 10017

A full line of color and extender pigments for the paint industry is exhibited, including easy dispersing, energy saving color oxides.

#### PHOTOMARKER CORP. Hawthorne, Calif. 90250

This exhibit introduces the PM 400 Color Analysis System, which combines advanced micro-processor technology and in-field portability. This system features autocalibration, an instant alpha-numeric printout in XYZ, CIELAB, and DELTA Lab; color averaging; and sample coding. Operated by a control panel or touch button on the hand-held sensor head which measures specimens as small as three-quarter inch, it records data in the micro-processor under all conditions. Also on display is the PM 300 featuring digital readout.

#### POLYSCIENCE CORP. Niles, Ill. 60648

The company, U.S. distributors for the Contraves line of Laboratory & Process Viscometer measuring and regulating equipment, features two laboratory Viscometers with one programming unit, and three types of process viscometers.

#### POLYVINYL CHEMICAL INDUSTRIES Wilmington, Mass. 01887

Featured are proven performance polymers: Neocryl A-622, which offers superior water and humidity resistance for metals and plastics; Neocryl B-1000, an extender that can replace 10 to 25% of nitrocellulose used in coatings; Neorez R-900 series water-borne urethanes offering high physical properties to both rigid and flexible substrates; and Neocryl A-634 a new water-borne polymer coating for finishing fine furniture and kitchen cabinets that is equal in performance characteristics to existing solvent systems.

#### PPG INDUSTRIES, INC. Pittsburgh, Pa. 15222

Lo-Vel® flatting agents for coil coatings, lacquers, clear finishes, textured finishes, vinyl, and furniture are featured. Also shown is Hi-Sil® 422 silica paint pigment for flat and semi-gloss latex interior paints, latex exterior paints, oil-based house paints, and traffic paints. Hi-Sil® T600 synthetic thickener and thixotrope, introduced within the last year, provides anti-sag action on vertical walls and keeps coarse particles in suspension in paints.

#### PREMIER MILL CORP. New York, N.Y. 10001

New "Supermil," a horizontal media mill with a unique system is displayed. Current models of vertical pressure sealed media mills are shown, as well as tank cleaning equipment and laboratory dispersers with all accessories.

#### PVO INTERNATIONAL, INC. Richmond, Calif. 94804

The company highlights its two English affiliates, Pacific Anchor Chemical Corp. (PAC) and Pacific Scott Bader (PSB). Ancamine@ cycloaliphatic, aromatic, and isolated adduct epoxy curing agents are featured in the PAC section, emphasizing their use in high solids and powder coatings. PSB features the dispersing medium Texigel@ 23-555 and its use in water-based trade and industrial finishes.

#### Q-PANEL CO. Cleveland, Ohio 44135

The exhibit features the QUV Accelerated Weathering Tester, a radically different approach to laboratory weathering. Rain and dew are simulated by a unique condensation mechanism. The effects of sunlight are simulated by fluorescent UV lamps. Advantages of the QUV tester include excellent reliability, low cost, and minimal maintenance. Also shown are Q-Panels, standard metal panels for paint tests.

#### REICHARD-COULSTON, INC. New York, N.Y. 10010

This our 25th year at the Paint Show and the company's 125th anniversary in business. On exhibit we feature four sections devoted to the following pigments: (1) A new product line of Super Strength High Opacity oxides; (2) Zinc Phosphate "317" a non-toxic anti-corrosive pigment; (3) "Your Metal Needs Color and Protection," which features oxides for use in coil coatings and (4) MIOX<sup>®</sup> "316" a non-toxic lamellar barrier type coating.

#### REICHHOLD CHEMICALS, INC. White Plains, N.Y. 10603

The booth features a conference center where Reichhold Chemicals representatives, and representatives from Reichhold Limited of Canada, are ready to discuss the latest developments in RCI's full line of chemical coatings, emulsions, and colors. The display features important additions in water-reducible resins and emulsions, epoxy resins and hardeners, and Varcum® metal coating resins.

#### ROHM AND HAAS CO. Philadelphia, Pa. 19105

This exhibit is of interest to those involved with industrial, trade sales, and maintenance coatings. Displays demonstrate the performance of high solids and aqueous vehicles for general product finishes which can be air dried or baked. Products for coil and can coatings are also available for examination. Rhoplex AC-64 acrylic emulsion for multipurpose use, as well as Rhoplex AC-417 for interior gloss and egg shell and Rhoplex MV-23 for stain blocking are shown. Exposure panels with paints at various RMC levels are used to compare 100% acrylic vehicles with other types.

#### RUSSELL FINEX, INC. Mt. Vernon, N.Y. 10550

The enclosed and fully automated strainer, together with the Finex 22 High Speed are featured in this exhibit. The Finex 22 High Speed can also be fitted with air sensors for automated straining.

# SEMI-BULK SYSTEMS, INC.

St. Louis, Mo. 63119

The exhibit introduces the Air-Pallet System, an effective new method of packaging, handling, and shipping such powdered products as pigments, clays, carbon blacks, calcium carbonate, resins, silicas, and other materials. The Air-Pallet is a reusable container comprising heavy-duty PVC coated fabric, and incorporating a unique pallet with built-in fluidizing floor and discharge port. Since filling and unloading operations are completely enclosed, highly effective dust control is achieved at point of origin and destination.

#### SEMLER INDUSTRIES INC. Franklin Park, III. 60131

The exhibit highlights new and established lines in the liquid handling equipment field featuring: custom engineered and designed systems; filtration and straining; humidifier control system; Owens-Corning tanks; paint waste treatment and high pressure wash system; and solvent recovery. Plant studies and new design surveys are also shown.

#### SHAMROCK CHEMICALS CORP. Newark, N.J. 07114

Technical personnel are available to discuss product applications and to take sample requests. A slide show based on photomicrographs is used to demonstrate the mechanism of abrasion resistance.

#### SHELL CHEMICAL CO. Houston, Tex. 77056

A Modern Newsroom is the theme that enables the company to present a video readout display highlighting Eponex®, a new weatherable resin. Also stressed are powder coatings and water-solvent blends for coatings, along with increased product capacity to support the growing coatings industry. On display are examples of research and development conducted by the company's Westhollow Research Center.

#### SILBERLINE MANUFACTURING CO., INC. Lansford, Pa. 18232

Eterna-Brite-the company's unprecedented breakthrough in leafing paste technology is featured. Panels and technical data illustrating the superior leafing, leafing stability, and reflectance of the three Eterna-Brite grades are on view. Comparative shelf life data in various ready-mixed aluminum paint formulations are available. The increasingly popular Sparkle Silver nonleafing grades are also on display.

# SPENCER KELLOGG DIV. Textron, Inc. Buffalo, N.Y. 14240

This modular type exhibit has four work areas and eight major features. Once again, a giant crystal ball visually introduces the at-tendees to "Resinvision," the theme of the exhibit. Personnel are available to discuss your ideas and problems. On display are coated items showing the company's expanded line of WD resins, WD urethane lacquers, 2 component systems, high solids resins et al. A Shaeffer pen and pencil desk set will be given away every two hours.

#### SUB-TROPICAL TESTING SERVICE, INC. Miami, Fla. 33156

A pictorial display of the facilities and services offered by the company, which is celebrating it's 50th year, are exhibited. Of particular interest will be examples of the variety and severity of failures which can occur on wood, plastic, fabric, and metal panels exposed to natural weathering in Southern Florida. Also featured is the company's drawingboard rack with its special features which can be adjusted to any angle and in any direction.

#### SUN CHEMICAL CORP. **Pigments Div.** Cincinnati, Ohio 45232

The booth features a display of the company's complete line of organic pigments for the coatings industry, and a review of the current environmental situation as it affects the organic pigments industry and pigments industry and the coatings industry and the coatings industry and big performance pigments. Sunfate<sup>®</sup> pigments for coatings, high solids presscakes, and fluidized dispersions are highlighted.

#### SWECO, INC. Los Angeles, Calif. 90051

The firm demonstrates its new high-speed separator, capable of screening solids from heavy, high viscosity liquids including latex coat-ings, and iron oxide slurries. A 30" diameter unit is shown in operation, and a 48" diameter unit is also available.

#### SYNRES CHEMICAL CORP. Anaheim, Calif. 92803

The booth features a display of the company's new developments of vehicles for water-borne and higher solids coatings.

#### TAMMSCO, INC. Cassopolis, Mich. 49031

Silica is one of the oldest most versatile pigment extenders available for the many types of paints and coatings being manufactured for today's consumer. Data and demonstrations are used to compare silica with other mineral extenders and to highlight the features of the various grades of silica produced by the company.

#### TENNECO CHEMICALS, INC. Piscataway, N.J. 08854

The exhibit features colorant products for industrial, trade sales, and on-the-job tinting, along with chemical additives for the coatings industry.

#### THIELE ENGINEERING CO Minneapolis, Minn. 55435

Three filling machines are on display: the semi-automatic (SAF), the twin-head automatic (AF-2), and the indexing/crimper (IC) machines. The SAF is now faster and easier to run with improved controls. The AF-2 is the industry favorite for automatic filling. The versatile and compact IC fills, moves, and seals one- through five-gallon sizes, and can be used as an SAF for all size filling.

#### TOKHEIM CORP. Ft. Wayne, Ind. 46801

The company manufactures positive displacement meters for measuring and flow control of resins, solvents, paints, and related paint products. Meters are available in 1" through 4" pipe sizes and flow rates from 0.2 GPM to 750 GPM. Accessories available include pulsers, flow rate indicators, temperature compensators, direct mounted or remote preset devices, and diaphragm flow control valves.

#### TORCO DIV. Toronto Coppersmithing International Ltd. Toronto, Ont. MIP 3A9

The exhibit features the company's Modular Grinder, an energy efficient, small media mill offering fume-free pressure chamber processing with fast changeover capability and modular design flexibility. Also on display are Shar High Speed Dispersers.

#### TROY CHEMICAL CORP. Newark, N.J. 07105

Biocides which are featured include: CMP Acetate; nonmercurial preservatives Troysan 14 and 192, and Troy's premium liquid nonmercurial fungicide, Polyphase Anti-Mildew. Also featured are problem solving additives including bodying agent 42-BA, a single component liquid post add for alkyd systems; Perma Dry, an effective lead free loss-of-dry inhibitor; and the company's full line of defoamers and anticratering agents for water based industrial and trade sales paints. Technical personnel are available to discuss specific problems.

#### UNION CAMP CORP. Wayne, N.J. 07470

The exhibit features UNI-REZ polyamide resins, tailor-made for high solids and for weatherable epoxy coatings. UNI-REZ polyamide resins are derived from trees-the renewable, natural resource grown in the U.S.A.

#### UNION CARBIDE CORP. New York, N.Y. 10017

The exhibit features materials for conventional coatings, industrial finishes, and trade paints. New opportunities for coil, wood, and product finishes are presented. Special emphasis is given to cost/performance opportunities of new UCAR acrylics for trade paints. The Captain's 14th Annual Putting Contest is being held-with golf pro Johnny Pott giving tips on Wednesday.

# UNION CHEMICALS DIV. Union Oil Co. of Calif. Schaumburg, Ill. 60196

Vinyl-acrylic latex polymers, solvents, and chemicals are displayed in a modern booth setting. Featured is AMSCO-Res 3079, a new vinylacrylic copolymer, which exhibits outstanding wet adhesion and semi-gloss properties. Dart contests are being held, with prizes offered to the winners,

#### UNION PROCESS INC. Akron, Ohio 44313

Exhibited are various types of equipment for dispersing and grinding; batch, continuous, and circulation processing. The ultimate grinding, dispersing, and mixing is obtained in attritors. Processing equipment is available for applications from laboratory experiments to large scale production.

#### UNIROYAL CHEMICAL CO. Naugatuck, Conn. 06770

The exhibit features a slide presentation on Polywet<sup>®</sup> dispersants and emulsifiers (surfactants) for the paint industry. A marketing development specialist is on hand to discuss the company's products.

#### UNITED CATALYSTS INC. Louisville, Ky. 40232

On display are clay based products that are used as anti-settling and thixotropic agents for the paint and grease industry. Literature and samples of products are available.

#### UNIVERSAL COLOR DISPERSIONS Lansing, III. 60438

A display of many water-reducible paint systems is featured, showing the difference in performance that colorants can make on your coating. Whichever use you may have, tinting or total pigmentation, the generic systems show how color affects your formulation.

#### UNIVERSITY OF DETROIT Detroit, Mich. 48221

#### UNIVERSITY OF MISSOURI-ROLLA Rolla, Mo. 65401

The booth features continuing education for persons interested in an increased knowledge of coatings, their application and testing, and the growing problems of government regulations and rapidly changing raw material supplies and pricing through coatings short courses. It also emphasizes the undergraduate and graduate student programs leading to BS, MS and PhD degrees in Chemistry with coatings emphasis.

#### UNIVERSITY, NORTH DAKOTA STATE Polymers and Coatings Dept. Fargo, N.D. 58105

Information on Coatings Program at NDSU, as well as descriptive literature is available. Faculty and other representatives are on hand to answer questions about undergraduate and graduate programs short courses, and symposia for industry technical personnel.

#### UNIVERSITY OF SOUTHERN MISSISSIPPI Dept. of Polymer Science Hattiesburg, Miss. 39401

The booth features a continuous slide presentation of the teaching, research, and industry service programs offered at USM. Information on the Seventh Water-Borne and Higher-Solids Coatings Symposium is available.

#### R. T. VANDERBILT CO., INC. Norwalk, Conn. 06855

The exhibit features a full line of products for the coatings industry –Drier accelerator Activ<sup>®</sup> 8; mold inhibitors, preservatives Vancide<sup>®</sup> PA, TH, 512; Dispersant: Darvan<sup>®</sup> No. 7; Air floated clay; Peerless; Pyrophyllite: Pyrax<sup>®</sup>; Talc: Nytal<sup>®</sup> and I.T. Wollastonite: Vansil<sup>®</sup> W; Thixotrope: Van-Gel<sup>®</sup>; Epoxy resins and curing agents: Vanoxy<sup>®</sup> and Van-Amid<sup>®</sup>; Flow control agent: Rheotol<sup>®</sup>.

#### VIKING PUMP DIV. Houdaille Industries, Inc. Cedar Falls, Iowa 50613

On display are cutaway models of internal and external gear pumps which show the company's total problem-solving approach for industry. The 624 series is especially developed for handling paints, inks, solvents, and abrasive liquids.

#### VORTI-SIV DIV. M & M Machine, Inc. Salem, Ohio 44460

On display is the recently introduced, enclosed and more efficient, gyratory Vorti-Siv, as well as two other gyratory screening and straining machines. These models have American standard nuts, bolts, and threads, and also can be produced in the metric system. Vorti-Siv has complete rebuilding and machine shop facilities, and a complete stock of parts for the older Lehmann Vorti-Siv.

#### THE WARREN RUPP CO. Mansfield, Ohio 44905

The booth contains displays of air-powered, double-diaphragm pumps featuring the VIP Model with Teflon diaphragms, ball valves, and gaskets for corrosive or volatile products which are self-priming and explosion-proof.

#### WELLCO PRODUCTS CO. Summit, III. 60501

On display is a new series of D.O.T. approved bulk liquid shipping tanks, along with tank washing equipment, rotating spray nozzles and specialized cleaning and stripping compounds. Working models of the spray nozzles are exhibited, along with several portable shipping tanks.

#### WILDEN PUMP & ENGINEERING CO. Colton, Calif. 92324

On exhibit are cut-away working models of unique air-operated, double diaphragm, positive displacement pumps. The four sizes of pumps on display are submersible, self-priming, and designed to handle very thick and abrasive, as well as highly shear-sensitive, products. They are available in optional alloys and elastomers to handle most erosive and corrosive applications. Also on display is the revolutionary Sentry 1 surge suppressor.

#### WITCO CHEMICAL CORP. Organics Div. New York, N.Y. 10017

The company's several lines of specialty materials for coatings are featured in the exhibit. These include Balab® Bubble Breaker® foam-control agents for water-based formulations, Witco® metallic stearates for lacquers and oleoresinous coatings, as well as driers, surfactants, and Keycide® X-10 anti-mildew additive.

## ZEKS AIR DRIER CORP.

Malvern, Pa.

The equipment display includes refrigerated air dryers which are used in compressed air systems. These units eliminate moisture in air power paint spray systems. Complete literature and drawings depicting mechanical operation are available. Personnel are present to describe accessories and applications.

# To keep your slurry tanks from running dry, we've added 72 cars to our rail fleet.

To assure you of dependable delivery of Du Pont TI-PURE<sup>®</sup> TiO<sub>2</sub> slurry, we added

72 cars last year to our already sizable rail fleet. We've also improved our order handling



system to serve you from 3 domestic plant locations. Now you can dial 800/441-9442 to place your next order.

If you haven't converted to slurry, consider these benefits. Many customers report savings of  $3 \notin$  to  $5 \notin$  a pound with TI-PURE® TiO<sub>2</sub> slurry versus dry. Bag unloading and disposal costs are eliminated, plus pigment losses from spillage and handling. And there are savings in improved housekeeping, safety and labor costs in general.

Increased production capacity and paint manufacturing efficiencies also add up...TiO<sub>2</sub> dispersion step is eliminated, faster TiO<sub>2</sub> charge time, optimized batch size, improved quality control and product uniformity.

All of these benefits and savings are possible with no loss in hiding power, tinting strength, gloss and durability.

As developers of TiO<sub>2</sub> slurry technology for coatings, we can advise you on the feasibility of a slurry system.

For further information, write to Du Pont Company, Room 27159, Wilmington, DE 19898.



# NPCA Presents Industry Statesman Awards at Annual Meeting

The National Paint and Coatings Association honored five men with long and illustrious careers in the U.S. paint and coatings industry at its recent annual meeting in San Francisco.

Receiving the Association's Industry Statesman Award for "long and unheralded service to the paint industry" were: John S. Ayres, President and Chief Executive Officer of Cook Paint and Varnish Co., Kansas City, Mo.; Richard G. Bull, retired Senior Vice-President of the Sherwin-Williams Co., Cleveland, Ohio; Dan K. Farstad, Senior Vice-President-Operations of the Spencer Kellogg Div. of Textron, Inc., Buffalo, N.Y.; Morris B. Lore, head of the Legal

# William A. Bours Is Elected President of NPCA

William A. Bours, III, Vice-President and General Manager of the Fabrics and Finishes Department of E.I. duPont de Nemours & Co., Inc., was elected President and Chief Executive Officer of the National Paint and Coatings Association at its 92nd annual meeting in San Francisco. Also elected were: Dr. C. Robert Hiles, of Lilly Industrial Coatings, Inc., Indianapolis, Ind., as Vice-President, and James H. Davis, of Porter Paint Co., Inc., Louisville, Ky., as Treasurer.

Regional Vice-Presidents elected were: New England Zone-Joseph S. Parker, of Sterling-Clark-Lurton Corp., Malden, Mass.; Eastern Zone-Stanley Eysmann, of Cellofilm Corp., Wood-Ridge, N.J.; East Central Zone-Merritt E. Marcus, of Edw. H. Marcus Paint Co., Louisville, Ky.: West Central Zone-Roland Gianneschi, of Standard T Chemical Co., Inc., Chicago Heights, Ill.; Southern Zone-Fred Schuenaman, of Davies Can Co., Tampa, Fla.; Southwestern Zone-Rex Hitt, of Standard T Chemical Co., Inc., Dallas, Tex.; and Western Zone-William B. Manuel, of Pacific Coast Chemicals Co., Berkeley, Calif.

Elected to the Board of Directors for a one-year term were: John G. Breen, of the Sherwin-Williams Co., Cleveland, Ohio; and Roland V. McPherson, of Armstrong Containers, Inc., Westchester, Ill.

Elected to the Board of Directors for a two-year term was Russell Banks, of Grow Group, Inc., New York, N.Y.

Elected to the Board of Directors for a three-year term were: C. A. Brethen, Jr., of Wyandotte Paint Products Co., Troy,

Department that advises E.I. duPont de Nemours, Inc. on labeling, Wilmington, Del.; and Frank H. Martin, retired Technical Director of PPG Industries, Torrance, Calif.

John Ayres, recently retired after 43 years of service with Cook Paint and Varnish Co., was named Manager there in 1940 and became Assistant Vice-President in 1953, was named Vice-President, Industrial Sales in 1959. He was elected to the Board of Directors that year and became President and Chief Executive Officer in 1960. He is active in the Kansas City Paint and Coatings Association, the Detroit Society for Paint Technology, and the FSCT.

Mich.; Robert M. Cox, Sr., of the Gilbert Spruance Co., Philadelphia, Pa.; Jerome J. Crowley, Jr., of the O'Brien Corp., South San Francisco, Calif.; P.J. Davis, of DAP, Inc., Dayton, Ohio; Charles J. Fisher, of Reliance Universal, Inc., Louisville, Ky.; Jay S. Hanna, of Hanna Chemical Corp., Columbus, Ohio: Clifton T. Hesness, of General Paint & Chemical Co., Div. of Cotter & Co., Cary, Ill.; R.J. Leahy, of Hercules, Inc., Wilmington, Del.; Heinz Machatzke, of Mobay Chemical Corp., Hawthorne, N.J.; M.R. Schimmenti, of Exxon Chemical Co. U.S.A., Houston, Tex.; and Thomas C. Sullivan, RPM, Inc., Medina, Ohio.

Richard Bull retired last February after 41 years in the coatings industry. Beginning in the Sherwin-Williams Company Technical Service Department, he subsequently held a variety of positions before becoming Vice-President and Corporate Director of Marketing, Group Vice-President-Coatings, Senior Vice-President-Marketing, Vice-Chairman of the Board, and Senior Vice-President. He was a Director of Sherwin-Williams and served on the board of the Sherwin-Williams Co. of Canada, Ltd.

Dan Farstad joined the Spencer Kellogg Div. of Textron, Inc. as a control chemist in the Edgewater, N.J. plant in 1940. He later became Manager of the Technical Service Department in Buffalo, N.Y. and in 1961 was promoted to Director of Technical Sales and Service. Later, he became General Sales Manager, Vice-President of Sales, and Senior Vice-President-Oil Seeds before assuming his present position as Senior Vice-President-Operations.

Morris Lore will retire in December after 42 years with E.I. duPont de Nemours, Inc. He joined the company as a research chemist and is now head of the Legal Department that advises duPont on labeling.

Mr. Lore has been an important member of NPCA's Labeling Committee since 1963. During his period of service, three editions of the Association *Labeling Guide* have been developed. He also has participated in over a dozen

# Mansel O. Wiley Is Winner of 1979 NPCA Heckel Award

Mansel O. Wiley, Vice President, Celanese Corp., Louisville, Ky., was named the 1979 winner of the George Baugh Heckel Award by the National Paint and Coatings Association (NPCA) at its 92nd annual meeting held recently in San Francisco.

The award recognizes outstanding personal contributions to the industry in the past year.

Mr. Wiley was Vice-President and General Manager of the Automotive Division of Jones-Dabney Company when it was acquired by Celanese Corp. in 1964. He had joined Jones-Dabney in 1948 as an industrial salesman. In 1950 he moved to the Automotive Division and later became Automotive Sales Manager in Detroit. In 1957 he was named VicePresident and General Manager, Automotive.

After Jones-Dabney was acquired by Celanese, he was named President of Celanese Automotive Marketing Company. Two years later he was appointed General Manager of three Celanese coatings divisions (automotive, industrial, and marine). In 1970 he became President of Celanese Polymer Specialties Company.

Mr. Wiley was named a Corporate Vice-President of Celanese Corp. in 1973. Last July, he also was named Vice-President of Agricultural Chemicals and Special Projects for the Corporation.

He is a member of NPCA's Executive Committee and Board of Directors and has also served terms as Treasurer, Vice-Chairman, and Chairman of the Board. labeling seminars conducted by the Association.

Frank Martin retired recently as Technical Director at PPG Industries, Torrance, Calif., after 39 years with that company. He began his career with PPG as a formulator chemist. Ten years later he took charge of technical activities for the Torrance plant of the Coatings and Resins Divisions—the position he held until he retired.

Mr. Martin further served the coatings industry by becoming involved with legislative and environmental affairs affecting the industry. He also served as a liaison between the California chapter of the Federation of Societies for Coatings Technology and the Southern California Coatings Association. He is a Past-President and former member of the Board of Directors for the Los Angeles Society for Coatings Technology.

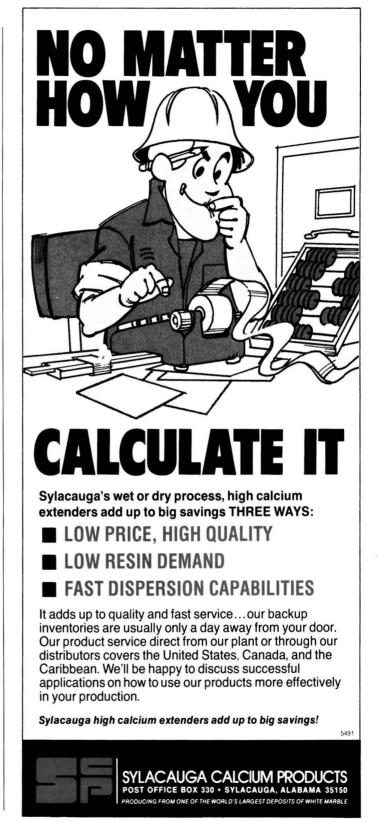
# CPSC Combustible Labeling Requirement in Effect As of October 6, 1979

Consumer paint products in the chain of distribution which have flashpoints "above 80°F to and including 150°F, tested by the Tagliabue Open Cup method" are now required to have the "combustible" statement of hazard on the front panel. The Consumer Product Safety Commission rule, originally published in the December 29, 1977 Federal Register, became effective on October 6.

Lone exception, according to a recent amendment to the rule, are pints and smaller containers which were *manufactured* prior to October 6. These are exempted from the front panel label requirement.

As originally published, all combustible paint products in the chain of distribution on October 6, 1979 were required to bear the front-panel statement. The two-year period from promulgation of the final rule was granted to industry to permit ample time to convert existing labels by moving the combustible statement from the back or side panel to the front panel.

After learning that many companies would have large inventories of combustible paint products in warehouses and on the effective date of the rule, NPCA petitioned CPSC on July 23, 1979 for emergency relief.

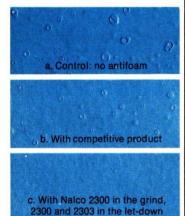


# **How to Select** the Right Antifoam for **Water-Based Paints**

# **Results are What Count**

Below is just one example of the results you get when Nalco tackles a foam problem. Take a good look. Results are what count and what Nalco is committed to.

"Vinyl Acrylic Terpolymer in an Interior Semi-gloss Paint"



Nalco has a unique approach to the selection of antifoams. And the results we achieve are proof of its effectiveness.

The Starting Point

Nalco starts from these basic premises.

What an Antifoam Should Do

- 1. It should prevent and destroy foam
- 2. It should be persistent.
- 3. It should not adversely affect color acceptance, gloss, water sensitivity, or intercoat adhesion.
- 4. It should not cause surface defects such as crawling, pinholes, or fisheves

Nalco is not satisfied with an antifoam selection until it meets all four requirements and achieves the results you are looking for.



# A Systematic Approach

Nalco uses a systematic screening procedure to determine antifoam performance. And before Nalco recommends an antifoam for your coating, we thoroughly analyze its effect in your total system. We:

Analyze Your Formulation The surfactants, co-solvents, flow control additives, pH, pigments, and resin type in your formula are studied since they affect foam stability and antifoam performance.

**Analyze Your Manufacturing** Procedures High sheer dispersion phases, transfer, and filling operations cause foam and may require several antifoams for effectiveness.

**Analyze Your Application Procedures** Techniques involving recirculation and high shear, such as spray and flow, can cause persistency problems and are considered when solving the foam problem.



Nalco's tests are designed to be as similar to actual usage as possible. Our recommendations may include products for the grind and others for the let-down. And to make sure the recommendation is right. Nalco checks results with the actual lab manufacture of your paint whenever possible.

# A Bank of Knowledge

Nalco works directly with new resin technology and may have already screened the resin you are using. And there probably is not a foam problem in manufacturing or application we have not seen before. We are old hands at licking foam problems. You gain the advantage of our experience.

A Broad Line of Products



From a broad selection of non-silicone and silicone types, you can choose the right defoamer for use in the grind and the let-down, for your trade sales coatings or industrial finishes. When used properly, it will give you effective foam control and minimize risk to film quality, color acceptance, and persistency.

# How to Get Started

If you are reformulating or developing a new water-based paint, give Nalco a call. Our paint experts will work with you from the start to develop the best foam control program possible. Just use our "hotline" number (312) 887-7500 ext. 1214. Or consult our literature. Write for bulletins K-8 and K-10

Nalco Chemical Company 2901 Butterfield Road Oak Brook, IL 60521 (312) 887-7500

# **Nalco Achieves Results**

50 Years of Leadership in Chemical Technology 1928-1978

# Comparison of Methods To Determine Water Content Of Emulsion Paints

Golden Gate Society for Coatings Technology Technical Committee

At the time the California Air Resources Board proposed stipulating the maximum amount of solvent permitted in a given volume of paint, there was no recognized method for determining the water content of an emulsion paint. In response to the evident need, two specific methods, using the gas-liquid chromatograph as the analytical tool, were proposed: One included a distillation step, the other did not. A third, unspecified method, based on Karl Fischer titration was also discussed. The Golden Gate Society compared these analytical methods by submitting emulsion paint samples to participating laboratories for water determination by one, or more, of these methods. The results of the comparison are discussed in this paper.

# INTRODUCTION

In March 1978, Regulation 9 (to become Regulation 8, Rule 3) of the Bay Area Air Pollution Control District was adopted. This rule for architectural coatings states that "no person shall sell or apply any architectural coating. . .which contains more than 250 grams volatile organic material per liter, as applied, excluding water." The adoption of a regulation that stipulated the amount of solvent permitted in a given volume of paint brought sharply into focus a problem that had been quietly ignored: A recognized analytical method for determining water in emulsion paint did not exist.

During September 1977, when the regulation on architectural coatings was first given general publicity by the California Air Resources Board (CARB), methods of determining water in emulsion paints were discussed with the paint industry. As a result of the discussions, two specific methods were proposed: /one, by CARB; the other, by the American Society for Testing and Materials (ASTM). Initial evaluation of these methods indicated that the CARB method would be procedurally difficult, and that the ASTM proposed method needed further development. In this time period other methods of water determination were also suggested. A modified Karl Fischer method generated particular interest.

With three methods to choose from, and none as yet required, the Technical Committee of the Golden Gate Society for Coatings Technology felt the paint industry should take the initiative in determining which was the most generally useful. The question was open because no one had much experience in determining the water content of emulsion paints. The California Air Resources Board had tested its method in its laboratory on a series of paints from one manufacturer; the American Society for Testing and Materials evaluated its method in four laboratories on a series of four paints. A standardized modified Karl Fischer method had not been proposed.

# PROCEDURE

#### Sample Selection

In the study seven paint products were chosen for testing. Three products were typical commercial emulsion paints taken off the store shelf. The remaining four products were prepared in the laboratory and were closely related to one another. One product contained an acrylic emulsion and water only; the second contained the emulsion, water, and propylene glycol; a third contained the emulsion, water, propylene glycol, wetting agents, anti-foam agents, thickeners, coalescing agents, and a can preservative; the fourth product contained every ingredient used in a typical emulsion paint including the pigment. This subset was prepared to examine constituents' effect on water determination. An eighth product was included after it was found that some laboratories particularly wanted to run the Karl Fischer determination on a sample containing an amine to test a suspected amine interference.

After the study was underway, seven ASTM round robin samples became available. These were also included where possible. This made a total of 15 samples for evaluation although not all laboratories had all 15 samples. *Table* 1 contains a fuller description of these products.

Presented by William Sawyer at the 56th Annual Meeting of the Federation of Societies for Coatings Technology in Chicago, Ill., Nov. 2, 1978

Sample number	Table 1—Description of Samples Coating description						
1	Laboratory prepared acrylic emulsion enamel	37.7					
2	Acrylic emulsion	54.9					
3	Low cost interior flat finish	71.0					
	Acrylic emulsion plus propylene glycol and additives	45.7					
5	High quality interior semi- gloss enamel	39.1					
6	Acrylic emulsion plus propylene glycol	45.0					
7	High quality exterior finish	39.0					
	Emulsion paint containing water soluble acrylic and amines	39.0					
Α	Polyvinyl acetate gloss finish	52					
B	Acrylic gloss finish	38					
	Acrylic flat finish	50					
Ε	Styrene butadiene flat finish	55					
	Acrylic flat finish	49					
	Acrylic semi-gloss enamel	48					
	Polyvinyl acetate flat enamel	50					

# **Description of Analytical Methods**

CALIFORNIA AIR RESOURCES BOARD METHOD:<sup>1</sup> A weighed amount of the emulsion paint (about 50 g) is distilled so that all the water is removed and captured. The distillate is weighed and the volume measured. The percent water in the distillate is determined using a gasliquid chromatograph and an external standard series. From these values the percent water per unit weight of paint can be calculated.

AMERICAN SOCIETY FOR TESTING AND MATERIALS PRO-POSED METHOD.<sup>2</sup> A weighed amount of the paint (about 0.6 g) is mixed with a weighed amount of moisture free isopropanol (about 0.2 g). This combination is added to 2 ml of dimethylformamide, mixed well, and directly injected into a gas-liquid chromatograph. The isopropanol acts as an internal standard. The values determined are the percent water per unit weight of paint.

MODIFIED KARL FISCHER METHOD: The regular Karl Fischer method is a recognized standard method for

determining small quantities of water in various organic solvents.<sup>3</sup> In the modified procedure, a small amount of emulsion paint is accurately weighed into a weighed amount of water-free, water compatible organic solvent combination. This new solution now resembles the standard Karl Fischer sample wherein there is a small amount of water in a large amount of organic solvent. Knowing the exact amount of emulsion and solvent used, the water content of the total emulsion can be calculated.

# **EVALUATION OF RESULTS**

Prior to discussing laboratory results, it might be well to review the criteria for selecting the most desirable analytical method. The requirement that the method be reliable and valid is primary. Secondary factors also need consideration such as: ease of performance, amount of laboratory time required, cost of equipment, safety, and ease of clean up. Precision and accuracy should never be sacrificed for these secondary factors. Should one method yield more reliable results, it should be the preferred method, even though it has such drawbacks as being time consuming and arduous.

# **Comparison of Procedural Judgements on Methods**

To better understand the following comparison, it is necessary to keep in mind that the distribution of the analyses and samples was not homogeneous throughout the study. That is, any one laboratory participating may have been given a complete or partial set of coating formulations, and may have used one, two, or all three of the methods.

Seven laboratories agreed at the outset to run samples using the CARB procedure. Five laboratories reported results which are tabulated in *Table 2*. The comments from the laboratories on CARB's method varied widely. Laboratory 3 completed the analysis on all samples provided, and gave no indication of any difficulty in following the procedure. Laboratory 2 noted that it encountered extreme difficulty in the distillation step and could not proceed with the method as described—it circumvented the problem by reducing the sample size and using vacuum distillation. Laboratory 12 did complete a series of seven samples, but noted that, because of difficulties in the distillation step, it had little

		Та	ble 2—	Percen	t Wate	r Deter	nined I	by CAF	B Met	hod					
Sample number Theoretical Value	1 37.7	2 54.9	3 71.0	4 45.7	5 39.1	6 45.0	7 47.5	8 39.0	A 52	B 38	C 50	E 55	F 49	G 48	Н 50
Laboratory 1	**	**	**	**	**	**	**	_		_			_	_	
Laboratory 2	35.4	53.4	74.2	41.2	40.3	41.8	45.1	_	49.8	38.2	50.1	_			
Laboratory 3	37.9	47.7	72.9	41.6	41.1	41.3	45.5		49.2	38.3	49.7			_	_
Laboratory 8		**	72.9	**	48.4	**	54.0	-	**	**	**	**	**	**	**
Laboratory 9	**	**	**	**	**	**	**	_	_	_	_	-			
Laboratory 11	**	**	66.6	**	36.1	**	35.6	39.2	47.3	**	**	52.3	**	**	50.9
Laboratory 12		32.8	57.3	41.0	32.5	24.5	37.6	—	—	-	_	—	—	_	_
Average Value	36.6	44.6	68.8	41.3	39.7	35.9	43.6	_	48.8	38.3	49.9	_	_	_	_

\*\* These samples were not run: in most cases because of excessive foaming in the distillation step.

Sample number	1	2	3	4	5	6	7	8	Α	в	С	E	F	G	н
Theoretical Value	37.7	54.9	71.0	45.7	39.1	45.0	47.5	39.0	52	38	50	55	49	48	50
Laboratory 1	35.0	52.7	73.5	42.1	37.0	43.3	41.4	_	_	_	_	_	_	_	_
Laboratory 2	37.3	46.2	76.4	39.8	35.8	36.6	45.1	_	50.3	41.6	53.7		_	-	_
Laboratory 3	_				_	_		_	49.9	35.5	53.8	_	_	_	_
Laboratory 8	**	**	74.2	**	37.5	**	48.2		54.3	35.0	50.0	55.3	48.3	47.8	52.0
Laboratory 9		56.4	77.5	45.5	39.9	45.2	48.6	_	50.3	38.0	51.1	54.9	46.8	47.8	51.
Laboratory 10					_	_	_	_	48.8	36.2	47.9	58.0	50.6	50.5	53.
Laboratory 11	39.8	52.3	70.6	44.2	37.7	42.6	48.9	38.7	51.2	37.0	50.8	52.6	45.3	46.7	49.
Laboratory 12	34.1	51.7	70.0	42.1	37.4	42.0	44.7	_	_			-	_		-
Laboratory 13			_	-	_	_	-	_	_	—	—	54.9	47.0	47.3	50.9
Laboratory 14			-	_	_		-		-			53.4	45.4	44.1	50.4
Laboratory 15			_	_	_	_	-	—	-		_	51.2	42.6	44.3	47.
Average Value	36.5	51.9	73.7	42.7	37.6	41.9	46.2	_	50.8	37.2	51.2	54.3	46.6	46.9	50.

Table 3—Percent Water Determined by	Proposed ASTM Method
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•• These samples were not run: The laboratory ran this series to compare ASTM with CARB values-it did not obtain CARB values.

confidence in the validity of the results. Laboratory 11 made every effort to obtain results using the specified distillation procedure but of 15 samples provided, only seven could be satisfactorily completed. Laboratory 8 ran three samples, but refused to attempt the others provided on the basis that there were too many sources of error in the method. Laboratories 1 and 9 reported, after several attempts to use the distillation process as outlined in the CARB method, that the process was not workable.

Eleven laboratories participated using the ASTM proposed method. All of the laboratories reported results which are tabulated in *Table 3*. None of the laboratories reported any technical complications that gave them great difficulty.

Four laboratories determined water content in the Golden Gate series of seven formulations using the modified Karl Fischer method. Their results are presented in *Table* 4. All four laboratories used an automatic titrating device and noted that the laboratory time required was considerably less than for either of the chromatographic methods.

# **Comparison of Quantitative Results**

The water content of the seven samples of the Golden Gate series was determined by all three methods. Our comparison of quantitative results will be confined to that seven. For each test the interlaboratory accuracy of the methods was determined by calculating the standard deviation of the interlaboratory reported values from the theoretical value of percent water in the samples (*Table* 5). The precision was determined by calculating the standard deviation of the interlaboratory values from the interlaboratory mean (*Table* 6).

Based on the series of seven samples, the modified Karl Fischer method has the best accuracy and precision of the three compared in the study. This result was unexpected because it was obtained in the face of the fact that a formal method was not described.

The ASTM proposed method, which was still being developed at the time of the Golden Gate study, exhibited less precision and accuracy than the modified Karl Fischer method. It has subsequently been improved, and accepted as an ASTM test method.

The CARB method showed the least promise of the three methods. The precision and accuracy obtainable by this method are very sample dependent. When the sample distills without excessive foaming or charring the results may be quite good—when it does not, the variation in results is substantial, e.g., samples 2 and 6.

# **Comparison of Qualitative Results**

The definitive qualitative result of the method comparison was that many laboratories either could not or would not use the CARB method on the variety of

Table 4—Percent Water Determined by Modified Karl Fischer Method															
Sample number	1	2	3	4	5	6	7	8	A	в	С	E	F	G	н
Theoretical Value	37.7	54.9	71.0	45.7	39.1	45.0	47.5	39.0	52	38	50	55	49	48	50
Laboratory 4	35.8	53.7	73.0	44.3	39.3	44.1	45.1	_	49.0	37.8	50.4	_	_		_
Laboratory 5	36.7	51.2	72.0	43.2	37.4	36.5	43.4	35.7	48.0	35.9	_	49.8	43.4	46.1	49.5
Laboratory 6	35.5	56.1		48.4	39.2	36.5	43.3	41.4	46.5	37.6	48.9	50.5	44.5	45.5	50.1
Laboratory 7	38.7	50.2	76.1	44.9	40.0	33.8	44.9	_	-	_		-	-	_	
Laboratory 9		_								_		53.0	46.7	48.3	48.7
Laboratory 13	—		_	_	_	_		_		-	—	55.3	48.4	48.0	55.0
Average Value	36.7	52.8	73.7	45.2	39.0	37.7	44.2	38.6	47.8	37.1	49.7	52.2	45.8	47.0	50.8

Table 5—Standard Deviation of Interlaboratory Results	
from Theoretical Percent Water Values	

		Standard Deviation						
Sample	Theoretical percent water	CARB Method	ASTM Proposed Method	Modified Karl Fischer Method				
1	37.7	1.5	2.3	1.6				
2	54.9	13.4	4.5	3.1				
3	71.0	6.7	3.9	3.2				
4	45.7	4.4	3.6	2.0				
5	39.1	5.4	2.0	1.0				
6	45.0	12.2	4.2	8.2				
7	47.5	7.6	3.0	3.4				

#### Table 6—Standard Deviation of Interlaboratory Results from Interlaboratory Mean

	CA Met		AS Prop Met	osed	Modified Karl Fischer Method		
Sample	Mean	Std. Dev.	Mean	Std. Dev.	Mean	Std. Dev	
1	. 36.6	1.0	36.5	2.0	36.7	1.3	
2		8.7	51.9	3.3	52.8	2.3	
3	. 68.8	6.3	73.7	2.7	73.7	1.7	
4	. 41.3	0.3	42.7	2.0	45.2	1.9	
5	. 39.7	5.3	37.6	1.2	39.0	1.0	
6	. 35.9	8.0	41.9	2.9	37.7	3.8	
7	43.6	6.5	46.2	2.7	44.2	0.8	

samples included in the project. Seventy samples were submitted to participating laboratories for determination by the CARB method, values were obtained on 37. In contrast, the 85 ASTM samples yielded 81 values; the 54 Karl Fischer samples, 54.

The modified Karl Fischer method was, by far, the least costly in terms of laboratory time and equipment. Equipment cost for the CARB and proposed ASTM methods is similar. Laboratory time cost is higher for the CARB method because it requires at least two hours for distillation of each sample before the chromatographic phase of the determination.

The study demonstrated that an analytical method that is suited to one laboratory and a limited variety of samples is not necessarily a universal method, and that an organized reporting system with a central coordinator, such as that used by ASTM, is essential to method development. The repeatability and reproducibility of the ASTM proposed method steadily improved because ASTM Committee D01.21 incorporated suggestions and comments of participating laboratories into each new draft of its method. The California Air Resources Board had no mechanism for either receiving or responding to comments on its method with the result that it stood as written even after comments and suggestions had been offered.

# SUMMARY

The results of the Golden Gate Society's comparison of methods for water determination in water-borne paints indicate that the modified Karl Fischer method is the best of the three compared. The proposed ASTM method is nearly equivalent to the modified Karl Fischer method in terms of quantitative results, but requires more expensive equipment and more laboratory time. The proposed CARB method gives more variable results (and is more

# GOLDEN GATE SOCIETY TECHNICAL COMMITTEE

William Sawyer, Chairman ......Sawyer Associates Patricia Shaw .....Esselte Pendaflex Corp.

time consuming) than either the modified Karl Fischer or proposed ASTM methods.

# POSTSCRIPT

ASTM's Task Group 21.54 chaired by Dr. W. Golton, of E. I. duPont de Nemours and Company, Inc., Philadelphia, Pa., is currently evaluating a standardized version of "Water Content of Paints by Karl Fischer Titration Method." ASTM's proposed method, "Determination of Water Content of Water Reducible Paints by Direct Injection into a Gas Chromatograph," has been modified under the direction of Richard Scott, Chairman of ASTM Committee D01.21. It has been accepted as an ASTM Test Method and has been tentatively included in the California Air Resources Board regulations.

# ACKNOWLEDGMENTS

The Golden Gate Coating Society thanks the following laboratories for participating in this evaluation and for their advice. They truly deserve recognition: Air and Industrial Hygiene Laboratory of California; Bay Area Air Pollution Control District Laboratory; Beckman Instruments Co.; California Ink Co.; Cal-Trans Laboratory; Chevron Research Co.; DeSoto, Inc.; Dymo Industries Technical Center; Napko Corp.; Sherwin Williams Co.; Tenneco Chemicals, Inc.; and the companies comprising ASTM Committee D01.21.

We are also greatly indebted to the following individuals who were helpful to us: Richard Scott, of the Sherwin Williams Co., Chicago, Ill., and Steven Balestrieri, of the Bay Area Air Pollution Control Board, San Francisco, Calif.

# References

- Bay Area Air Pollution Control District Method of Procedure 8-3-301, 302 6 March 79.
- (2) American Society for Testing and Materials Method D3792, "Test for Water Content of Waterborne Paints by Direct Injection into a Gas Chromatograph."
- (3) American Society for Testing and Materials Method D1364, "Test for Water in Volatile Solvents (Fischer Reagent Titration Method)."

# Residual Stress in a Solventless Amine-Cured Epoxy Coating

S.G. Croll National Research Council of Canada\*

Residual stress in a solventless, amine-cured epoxy coating has been measured and found to increase with coating thickness. The trend with thickness probably results from the competition between densification, produced by the curing reaction, and swelling by absorbed atmospheric moisture, which is prevalent in the thinner coatings. The exothermic curing reaction has no influence upon the internal stress nor upon curing rate of the coating because it does not produce a measurable temperature increase. A heat transfer calculation confirms that the temperature increase in a typical coating is very small.

# INTRODUCTION

Residual internal stresses may have an adverse effect on the adhesive and cohesive properties of an epoxy system.<sup>1</sup> Many such systems are solvent-borne and most of the previous work on residual stress has been performed on solvent-borne convertible coatings and adhesives.<sup>2,3</sup> Reference (2) is an extensive survey of Russian work in this field. Drying processes, whether by chemical change or solvent evaporation, involve a shrinkage of the coating or adhesive system. At some stage of drying, the coating solidifies and further shrinkage is constrained by adhesion to the substrate. Thus, internal stress arises in the now rigid polymer.

It was the purpose of this investigation to measure and become familiar with the appearance of internal stress without the complicating influence of solvent diffusion and evaporation or elevated curing temperatures. Information gleaned in this manner may help to clarify results gathered on more complicated formations and, thus, provide a means of reducing internal stresses.

### EXPERIMENTAL METHODS AND RESULTS

The data presented are concerned with a solventless, cold-curing formulation of Epon® 828 cured with 11 parts per hundred of reagent grade diethylenetriamine

(DETA). The mixture was made in 0.025 kg batches, each stirred at high speed for 1 min, then allowed to stand for 20 min before coating. All experiments were carried out in a controlled environment of  $23 \pm 0.5^{\circ}$  C and 50  $\pm 2\%$  relative humidity (RH).

#### **Measurement of Internal Stress**

TECHNIQUE: The apparatus and technique were exactly as described previously: a thin steel cantilever was coated on one side (underneath) with the liquid coating and its bending deflection measured as the coating dried and shrank.<sup>4</sup>

RESULTS: The measured residual internal stress,  $\sigma_{meas}$ , is plotted as a function of coating thickness in *Figure* 1, and as a function of time in *Figure* 2. The deflection of the cantilever proved to be quite sensitive to ambient humidity. If humidity increased, the deflection decreased, probably as a result of plasticization of the epoxy by absorbed water. Although the thicker coatings seemed to harden in about 4 hr., the data presented in *Figure* 1 were obtained after a period of one day when the stress appeared to reach its final equilibrium (residual) value.

ANALYSIS: As may be seen in *Figure* 1, the internal stress measured in this manner is a function of coating thickness. In conventional mathematical analysis, it is assumed that stress is constant through the coating and one might expect it to be independent of thickness. Thus, the analysis must be reviewed in order to assess the significance of the results.

In conventional analysis,<sup>5</sup> the stress,  $\sigma$ , through the coating is considered constant and the resultant bending moment per unit width, m, of coating is given by

$$m = \sigma \, \frac{c(t+c)}{2} \tag{1}$$

where t = thickness of metal shim c = coating thickness.

Deflection, d, at a distance,  $\ell$ , along the bent cantilever can also be expressed in terms of the bending moment, m,

$$m = \frac{d E t^{3}}{6\ell^{2} (1 - \nu)}$$
(2)

where E = Young's modulus of the metal shim and v = Poisson's ratio of the metal shim.

<sup>\*</sup>Building Materials Section, Division of Building Research, Ottawa, K1A 0R6, Canada. Epon is a registered trademark of Shell Chemical Co.

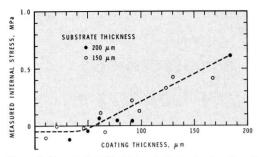


Figure 1—Measured residual internal stress as a function of coating thickness

Combining equations (1) and (2) results in the conventional expression for internal stress:

$$\sigma = \frac{d E t^3}{3c\ell^2 (t+c)(1-v)}$$
(3)

This equation is an approximation<sup>4</sup> that seems to be adequate for the experiment. In the more general case, where stress varies through the coating, the bending moment is given by

$$m = \int_{0}^{c} \sigma(y)(y + \frac{t}{2}) dy \qquad (4)$$

following the scheme of Kornum,<sup>6</sup> where  $(y + \frac{t}{2})$  is the distance from the neutral axis (*Figure 3*).

If internal stress varies through the coating, applying equation (3) will give a measure of the internal stress,  $\sigma_{\text{meas}}$ , equivalent in equation (1) to

$$m = \sigma_{meas} \frac{c(t+c)}{2} \qquad 1(a)$$

If equation (4) is combined with equation 1(a), then  $\sigma_{meas}$  represents

$$\sigma_{\text{meas}} \frac{c(t+c)}{2} = \int_0^c \sigma(y)(y+\frac{t}{2}) dy$$

or

$$\sigma_{\text{meas}} = \frac{1}{c} \int_{0}^{C} \sigma(y) \quad \left(\frac{2y+t}{c+t}\right) dy \tag{5}$$

The average stress through the coatings is given by

$$\overline{\sigma} = \frac{\int_{0}^{c} \sigma(y) \, dy}{\int_{0}^{c} dy}$$
$$= \frac{1}{c} \int_{0}^{c} \sigma(y) \, dy.$$
(6)

Provided t is much larger than c, which is normally true, then the measured internal stress,  $\sigma_{meas}$ , is very close to the average internal stress,  $\overline{\sigma}$ .

One can see, random experimental error permitting, that there should be a systematic change in the measured stress if different substrate thicknesses are used. The case where internal stress varies linearly within the coating has been analyzed in more detail,<sup>6,7</sup> and equations have been presented that permit the stress profile to be determined from the results on different substrate thicknesses. Such a procedure must be used cautiously, since the analysis is an approximation and the experimental error can easily be greater than 10%.

#### **Mechanical Properties**

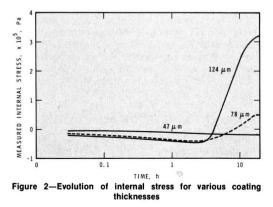
The modulus of the cured resin was found by compressing small cylinders 5 mm in diameter and 4 mm in length. Graphite was used to lubricate their end faces and to reduce the friction produced as the cross section changed under increasing load. After loading to a strain of  $5 \times 10^{-3}$ , deformation was maintained constant while stress was allowed to relax for two days, when it reached approximate equilibrium. The relaxed stress was used to calculate modulus because it more closely corresponds to the state in the coating at which internal stress has reached its final value.

The stress relaxation modulus was found to be 0.78 ( $\pm$ 0.05) GPa. Young's modulus (i.e., at short times) was found to be 2.8 GPa (comparable to quoted values<sup>8</sup>) and Poisson's ratio was assumed to be 0.34 GPa.

Cylinders used for these experiments were prepared by casting in appropriately shaped PTFE molds. Before use, they were conditioned at 23°C and 50% RH for at least four weeks. Modulus measurements were taken in compression because the cast resin proved to be very brittle in tension. Mechanical testing in compression did not open cracks and precipitate brittle failure. There seemed to be no other effect of sample geometry or testing conditions.

#### Weight Gain During Cure

It is known that epoxy resins and DETA absorb an appreciable amount of water. This could very well affect the magnitude of the internal stress. Consequently, the weight change of coatings of varying thickness was monitored as a function of time during the curing process. The coatings were about  $6 \times 10^{-3} \text{ m}^2$  in area and typically weighed 0.4 gm, weighed on a balance capable of measuring 0.1 mg changes. Results are plotted in *Figure* 4.



# Temperature Rise During Cure

THIN COATINGS: The curing of Epon 828 by DETA is an exothermic reaction that can produce considerable temperature rise in a bulk mixture. Temperature changes in typical coatings were measured to determine whether there is a significant temperature increase that would influence internal stress behavior. One thermistor was supported (Figure 5) in a blackened insulating shield a fraction of a millimeter above the surface of the coating. Another was placed in thermal contact (via heat conducting grease) with the substrate, 25  $\mu$ m brass shim. The substrate was thin so that it would conduct heat to its thermistor more easily. The shield was blackened so that any heat escaping from the top of the coating was, to some extent, retained in the medium surrounding the top thermistor and not reflected back. The thermistors were connected to a strip chart recorder via a YSI (Yellowstone Springs Instrument) Model 43 TD Telethermometer with an output of 2.4 mV/°C. Thus, 0.1°C temperature change would be easily detected.

After the normal 20 min wait before coating, the 25gram mixture was found to be at 40°C. After being transferred via a metal spoon and spread in a thin coating, however, the maximum temperature increase recorded by the thermistors was about 1°C, which disappeared in about 10 min. There was no further apparent temperature increase, even in coatings of 150  $\mu$ m thickness, although the coatings were still liquid and undergoing the curing reaction.

THICK COATINGS: The temperature rise in thick coatings, > 1 mm, was appreciable and was measured by incorporating a thermocouple (36 SWG) in the epoxy mixture. The test samples were cured in aluminum foil cups 50 mm in diameter; the thermocouple was placed as close as possible to the center of the area and thickness of the mixture. Increases of a few degrees were detected. *Figure* 6 shows the results as a function of thickness.

# DISCUSSION

The results in *Figure* 1 show, for Epon 828 and DETA at least, that residual internal stress is small at all coating thicknesses. One would not expect it to precipitate failure. It also shows that internal stress is a function of coating thickness, implying that it varies throughout the coating depth. Random experimental error masks any systematic trend there might be with substrate thickness.

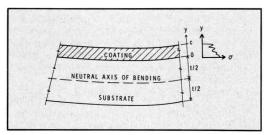


Figure 3—Schematic diagram of bending cantilever experiment

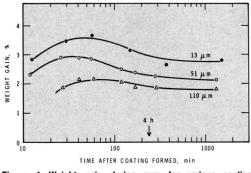


Figure 4—Weight gain during cure for various coating thicknesses

Stress varies rather slowly with thickness, so that one should expect the value obtained to be a good measure of average internal stress, although in thicker coatings the approximation will be less close.

## Thin Coatings

Below a coating thickness of 50 to 60  $\mu$ m, the stress appears to be negative, i.e., compressive, confirming Dannenberg's results.<sup>3</sup> The cantilever deflected upwards, possibly because of swelling of the coating or loss in weight. In fact, curing coatings actually gained weight (*Figure* 4), probably owing to water absorption. Thinner coatings gain proportionally more weight, because the water can diffuse through more of the coating before it cures and becomes comparatively impermeable. The compressive stress must be due to swelling by some means.

It was observed (*Figure* 2) that compressive stress developed quickly and was the precursor of a positive stress in thicker coatings. As the maximum rate of curing in epoxies occurs initially,<sup>9</sup> it might be supposed that compressive stress is due to thermal expansion of the epoxy or cantilever from the exothermic reaction. No such temperature increase was observed, however, and one would expect this stress to disappear upon cooling. In fact, the internal stress appears to be permanent and, so, a thermal origin for this stress is unlikely.

The swelling must be caused by water absorption. The thin coatings absorb about 3% of their weight by water, although it is not reflected in the magnitude of the stress. A value of  $5 \times 10^4$  Pa (a reasonable value for compressive stress) corresponds to a linear strain of  $4.2 \times 10^{-3}$ %, or a bulk strain (swelling) of  $1.3 \times 10^{-2}$ %. This is a very small amount of swelling. It would occur while most of the mixture is still liquid and one would not expect the internal stress to be equivalent to the total volume of water absorbed. Possibly, there may be a viscous contribution to the development of the stress, since the initial rate of water absorption is rather rapid and the crosslinking mixture becomes very viscous quite quickly. No attempt to calculate viscous stress will be made here, however.

#### **Thick Coatings**

As coating thickness increases from about 60  $\mu$ m, the value of residual stress is positive and increasing. One

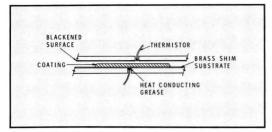


Figure 5—Detection of temperature increases in curing coating

might expect this to be an effect produced by differential thermal expansion between the epoxy and the cantilever substrate. A positive internal stress would be produced because the epoxy would heat while a liquid and cool after solidifying when the reaction was complete. The thicker the coating, the hotter it would become, because the heat could not escape so easily. Thus the increase in stress with thickness would be explained.

In order to calculate the temperature rise necessary to produce this stress, the linear expansion of the cured epoxy was measured in a duPont 941 thermomechanical analyzer and found to be  $5.4 \times 10^{-5}$ /°C at 23°C and 50% RH. For the steel substrate the value is  $1.1 \times 10^{-5}$ /°C. Assuming that the system hardens at an elevated temperature, the thermal strain,  $\epsilon$ , produced upon cooling is given by

$$\epsilon = \alpha \Delta T$$
 (7)

where 
$$\Delta T$$
 = rise in temperature above ambient  
 $\alpha$  = linear expansion coefficient for cured  
Epon 828.

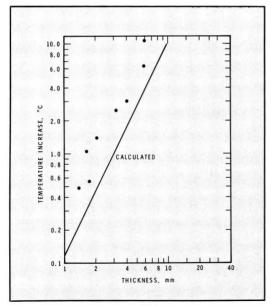


Figure 6—Temperature rise in thick epoxy films due to exothermic cure, experimental and calculated values

The strain can be calculated from the internal stress by using, in this plane stress situation,

$$\sigma_{\rm meas} = \frac{{\rm E} \ \epsilon}{1 - v} \tag{8}$$

where E = modulus of Epon 828v = Poisson's ratio of Epon 828.

Using the values of E,  $\nu$ , and  $\alpha$  results in temperature increases of a few degrees (*Figure 7*).

Although the temperature detection equipment was not sophisticated and no such temperature increases were noted, one of  $6^{\circ}$ C (~ 120  $\mu$ m) should have been detected. *Table* 1 gives the predicted temperature increase in these circumstances and confirms the experimental findings that temperature rise is negligible. The heat transfer analysis is dealt with in the next section.

One must conclude that residual stress in room temperature-cured Epon 828 has nothing to do with the exothermic nature of the curing.

Epoxy resins densify upon curing and residual stress would reflect the increase in density after solidification takes place. If water can diffuse only a finite distance into the coating before it cures completely, thicker coatings would show a greater average internal stress, because a smaller proportion would be swollen by water. This is confirmed by the plot of weight gained (water absorbed) during cure (*Figure 4*). Thicker coatings absorb proportionally less water. Competition between a finite waterswollen layer and the denser remainder of the coating produces the increase in stress with thickness.

Presumably, at coating thicknesses below ~50  $\mu$ m, water can completely penetrate the epoxy in the time before cure, resulting in a compressive stress that is independent of thickness. The residual internal stress results from the change in density from the solidification point to the completely cured state. For such small values of stress, the corresponding change in density is very small. For example, a positive residual stress of 0.5 MPa corresponds to a strain of  $4.23 \times 10^{-4}$ , representing a relative change in density of three times this value (bulk strain), i.e.,  $1.27 \times 10^{-3}$ , which is very small. Such a change in density would be very difficult to measure as a function of coating thickness.

#### Temperature Increase in Coatings

No measurable temperature increases resulting from exothermic curing were found experimentally, even in very thick coatings (~150  $\mu$ m). An approximate heat transfer calculation is presented here to show that this is what one might expect. The analysis is one dimensional because the heat lost through the edges of a typical coating will be negligible compared to that lost through the top and bottom surfaces.

The increase in temperature of the epoxy/unit time/unit volume

$$= C_p \rho \frac{\partial T}{\partial t}$$

where  $C_p$  = specific heat

- $\rho$  = density
- T = temperature
- t = time

The heat conducted away in the x direction

$$= \mathbf{K} \frac{\partial^2 \mathbf{T}}{\partial \mathbf{x}^2}$$

where K = thermal conductivity.

The conversion of Epon 828 by DETA is assumed here to be a first order reaction, i.e., the conversion is exponential in time. The detailed curing kinetics of such systems is discussed elsewhere,<sup>9</sup> but this assumption provides a reasonable approximation with which it is simple to deal.

The total heat produced, therefore, after a given time per unit volume

$$= \Delta H \rho (1 - e^{-\lambda t}).$$

Differentiating gives the rate of supply of heat per unit volume

= 
$$\Delta H \rho \lambda e^{-\lambda t}$$

where  $\Delta H$  = heat of reaction

 $\lambda$  = time constant governing the reaction.

Strictly,  $\lambda$  should increase with temperature for a thermally activated process, but, since the temperature rise is small, this complication is ignored. Thus, the increase in temperature is given by the equation

$$C_{p} \rho \frac{\partial T}{\partial t} = K \frac{\partial^{2} T}{\partial x^{2}} + \Delta H \rho \lambda e^{-\lambda t}$$
(9)

which is the normal heat diffusion equation, plus a term on the right side representing the internal heat source of the exothermic reaction. The solution of this equation is presented in Appendix A, together with the boundary conditions assumed.

The temperature rise, averaged over the thickness of the coating, is given as a function of time by

$$\Delta T = \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{e^{-\lambda t}}{(2n+1)^2} \frac{-\beta \lambda}{\lambda^1} \left( e^{\lambda^1 t} - 1 \right)$$
(10)

and the temperature rise in the middle of the coating by

$$\Delta T^{1} = \frac{4}{\pi} \sum_{\substack{n=0}}^{\infty} \frac{(-1)^{n} e^{-\lambda t}}{2n+1} \frac{\beta \lambda}{\lambda^{1}} \left( e^{\lambda^{1} t} - 1 \right)$$
(11)

where  $\beta$  and  $\lambda^1$  are combinations of the parameters used in equation (9) (Appendix A).

Figure 6 gives the rise in temperature necessary to explain the measured (average) residual stress in the coatings. Table 1 gives the highest predicted temperature increase as a function of coating thickness from equation (10). As one can see, the predictions are about three orders of magnitude lower than the data in Figure 6. This confirms the experiments, which could not detect any temperature rise, even up to  $150 \ \mu m$  thick coatings. This implies that there should be no acceleration in curing as a result of internal heating at these typical coating thicknesses.

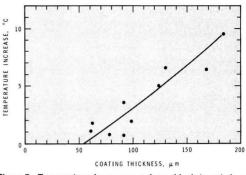


Figure 7—Temperature rise necessary for residual stress to have thermal origin

The temperature increases recorded in the thick (>1 mm) coatings can be used to check the accuracy of the analysis. Equation (11) can predict the highest temperature, during cure, in the center of the coating. The predictions are displayed in *Figure* 6 as the solid line. As may be seen, the prediction is a factor of two to three too small, but the disparity is acceptable considering the simplifications and boundary conditions assumed. Nevertheless, it still allows the analysis to confirm the experimental finding that temperature rise in coatings up to 150  $\mu$ m thick is negligible.

# CONCLUSIONS

The residual internal stress in this solventless, aminecured epoxy system depends on the thickness of the coating. Variation is so slight that the measured stress probably represents average stress even in thicker coatings. Below 55  $\mu$ m thickness, the stress is small and compressive and almost independent of thickness. Above this value it is tensile and increases gently with coating thickness. This variation is the result of two competing processes during cure: first, shrinkage due to structural change induced by conversion; and second, production of a slightly swollen layer due to absorption of water, etc., from the atmosphere. Indeed, the experimental results depended strongly on the ambient humidity, presumably owing to plasticization by absorbed water.

All of the recorded values of residual stress are quite small, particularly in thin coatings ( $<55 \,\mu$ m), and

Table 1—Predicted Temperature Increase as a Function Of Coating Thickness [Equation (10)]

ating Thio μm	kness,	Temperature Rise °C
10		$7.8 \times 10^{-6}$
20		
40		$1.2 \times 10^{-4}$
70		$3.8 \times 10^{-4}$
100		$.7.8 \times 10^{-4}$
150		$1.7 \times 10^{-3}$
200		$3.1 \times 10^{-3}$

#### S.G. CROLL

probably do not significantly influence the performance of this particular coating system, Epon 828 and DETA.

No temperature increase due to exothermic conversion could be detected in coatings up to 150  $\mu$ m thick, and this is confirmed by calculation, using a simple heat transfer analysis. Consequently, internal stress is not thermal in origin and neither will the rate of cure be accelerated by this means.

Temperature changes could be detected in systems greater than 1 mm thick and these were used to check the heat transfer calculation, which proved to be within a factor of two to three.

#### ACKNOWLEDGMENT

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# **APPENDIX A**

Solution of heat transfer equation (equation (9))

$$C_{p} \rho \frac{\partial T}{\partial t} = K \frac{\partial^{2} T}{\partial x^{2}} + \Delta H \rho \lambda e^{-\lambda t}$$

This equation does not admit to heat loss through mass transfer, i.e., convection. If some of the constants are combined such that

 $\alpha = \frac{K}{\rho C_p}$ , thermal diffusivity

$$3 = \frac{\Delta H}{C_{e}}$$
, temperature rise if there is no heat loss,

equation (9) can be rewritten as

$$\frac{\partial^2 T}{\partial x^2} = \frac{1}{\alpha} \frac{\partial T}{\partial t} - \lambda \beta e^{-\lambda t}$$
 (A 1)

To simplify, the temperature variable can be recast as the sum of a constant and a changing component

thus,  

$$\frac{\partial^2 \Delta T}{\partial x^2} = \frac{1}{\alpha} \frac{\partial \Delta T}{\partial t} - \lambda \beta e^{-\lambda t} \qquad (A 2)$$

Denoting the Laplace transform of  $\Delta T(t)$  by  $\tau(s)$ , equation (A 2) becomes, when transformed,

$$\frac{\partial^2 \tau}{\partial x^2} - \frac{s\tau}{\alpha} = \frac{-\lambda\beta}{\alpha(s+\lambda)}$$
(A 3)

Equation (A 3) is a simple differential equation with the solution

$$r = Ce^{-qx} + De^{+qx} + \frac{\lambda\beta}{s(s+\lambda)}$$

where  $q = (s/\alpha)^{\frac{1}{2}}$  and C and D must be determined from the boundary conditions.

#### **BOUNDARY CONDITIONS**

Choose x such that the middle of the coating is the origin (x = 0), then the top and bottom surfaces of the coating are at  $x = \pm h$ 

(i) Assume that heat loss is identical through the top and bottom faces so that at  $x = \pm h$ ,  $\Delta T = 0$ , i.e.,  $T = T_o$ , and similarly  $\tau = 0$  at  $x = \pm h$ . This choice probably lowers the temperature in the coating; one would expect a finite temperature gradient across the interface. It does, however, allow an algebraic solution to be obtained. In any case, it may compensate for the denial of heat loss by convection.

(ii) From (i), it follows by symmetry that at the middle of the

coating x = 0,  $\frac{\partial \Delta T}{\partial x}$  and, thus,  $\frac{\partial \tau}{\partial x}$  must be zero.

Using the two conditions, C and D can be found quite readily and the full solution is

$$\tau = -\frac{\lambda\beta}{s(s+\lambda)} \frac{\cosh qx}{\cosh qh} + \frac{\lambda\beta}{s(s+\lambda)}$$
(A 4)

When transformed back, knowing the Inverse Transform<sup>11</sup> of

$$\frac{1}{s+\lambda}$$
 and  $\frac{\cosh qx}{s \cosh qh}$ 

and using the convolution relation that

c t

$$\int_0^{\infty} F(t - u)G(u)du \text{ is the inverse Laplace Transform}$$

of  $f(s) \cdot g(s)$  if f(s) and g(s) are the Transforms of F(t) and G(t), respectively.

Thus,

$$\Delta T_{-} = \frac{4\beta\lambda}{\pi} e^{-\lambda t} \sum_{n=0}^{\infty} \frac{(-1)^{n}}{(2n+1)} \cos\left[\frac{(2n+1)\pi x}{2h}\right] \left(\frac{e^{\lambda^{1}t}-1}{\lambda^{1}}\right) \quad (A 5)$$

 $4h^2$ 

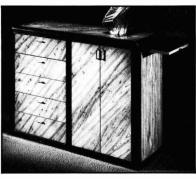
where

and equation (11) can be readily obtained by setting x = 0. The average temperature change over the coating thickness is given by

$$\Delta T = \frac{1}{h} - \int_{0}^{h} \Delta T(x) dx$$
  
Because  $\frac{1}{h} \int_{0}^{h} \cos \left[ \frac{(2n+1)\pi x}{2h} \right] dx = \frac{2(-1)^{n}}{(2n+1)\pi}$ 

the expression for the average temperature change over the coating thickness is obtained, i.e., equation (10).

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# Solvent Loss From Water Soluble Acrylic Coatings And Its Effect on Flow

Larry B. Brandenburger Inmont Corporation\* and Loren W. Hill North Dakota State University<sup>†</sup>

Evaporation of water and several cosolvents during and up to 15 min after spray application was investigated using gas chromatography to determine residual volatile components. Statistical analysis was used to determine the independent effects of starting levels of water, sec-butanol, and 2-butoxyethanol, as well as relative humidity and dry film thickness on water remaining in the coating. Independent effects were also determined for secbutanol and 2-butoxyethanol remaining. This information was used to prepare coatings to simulate compositions obtained during and following spray application. The low shear viscosity of the prepared compositions was correlated with visually observed sagging of the actual sprayed coatings.

## INTRODUCTION

Studies of water dilution of organic cosolvent rich solutions of water soluble binders show that viscosity depends on cosolvent/water ratio as well as on non-volatile content.<sup>1-4</sup> Therefore, when water soluble coatings are spray applied, it is logical to expect that the viscosity on the sprayed substrate will also depend on cosolvent/water ratio. During application the organic cosolvent and water are likely to evaporate at different rates, and the application properties, such as sagging, will depend on the cosolvent/water ratio which results from these different rates. Of course, the rate of water loss will depend on ambient relative humidity during spraying.

One purpose of this study was to elucidate the effects of initial composition of the volatile portion of the coating composition, relative humidity during application, and film thickness on solvent losses that occur during spray application. A second purpose was to determine the effect of such solvent losses on the rheological properties of coatings on sprayed panels over a time span from immediately after to 15 min after application. In order to obtain a basis for interpretation of the effect of solvent losses on viscosity, viscosity reduction behavior was compared for pure water dilutions and dilutions with a constant cosolvent/water ratio.

The approach to the solvent loss study consisted of using experimental data to obtain a computer model capable of calculating coating compositions at zero, 5, 10, and 15 min after spraying for any set of initial conditions within the ranges of variation used in obtaining the model. The effect of relative humidity on cosolvent/water ratio was analyzed in terms of the critical relative humidity (CRH) concept introduced by Dillon.<sup>5</sup> Visual observations of sagging of sprayouts under a given set of conditions were correlated with the compositions calculated by using the model for the same set of conditions. Samples having these compositions were prepared for viscosity determinations.

#### **EXPERIMENTAL**

# **Resin Preparation and Characterization**

The three copolymers used in this study have monomer ratios similar to resins used in thermosetting acrylic topcoats. Copolymers were prepared by free radical polymerization in 2-butoxyethanol. The temperature was maintained at  $100 \pm 2^{\circ}$  C. The initiator was azobisisobutyronitrile (AIBN). The initiator content was varied depending on the molecular weight desired. Two-thirds of the solvent was brought to  $100^{\circ}$  C in a 2 liter breakaway reaction flask, and a mixture containing initiator, monomer, and the remaining solvent was added dropwise over a three hour period. Temperature was maintained for one hour after the addition was complete. The monomer percentages, which were the same for all three copolymers, and resin characteristics are given in

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er			Mole %	(wt %)		
acid			12.6	( 8.5)		
Hydroxyethylmethacrylate						
			44.0	(40.4)		
			34.4	(40.4)		
Initiator <sup>a</sup>	M <sub>w</sub>	<b>M</b> _w/M_n		AN	NV	
(wt %)			(mg	KOH/g)	(wt %)	
3	41,000	5.0		51.0	84.8	
3	42,000	4.4	(	50.0	74.3	
5						
	kyethylmetha methacrylat crylate Initlator <sup>a</sup> (wt %) 3	acid cycethylmethacrylate methacrylate rylate Initiator <sup>a</sup> M <sub>w</sub> (wt %) 3 41,000	acid cycethylmethacrylate methacrylate rylate Initilator <sup>a</sup> M <sub>w</sub> M <sub>w</sub> /M <sub>n</sub> (wt %) 3 41,000 5.0	acid	acid         12.6         (8.5)           systethylmethacrylate         9.0         (10.7)           methacrylate         44.0         (40.4)           rrylate         34.4         (40.4)           Initilator <sup>a</sup> $\overline{M}_w$ $\overline{M}_w/\overline{M}_n$ AN           (wt %)         (mg KOH/g)         51.0	

Table 1—Copolymer Composition and Characteristics

(a) Azobisisobutyronitrile, wt % based on monomer.

Table 1. The acid number of resin A is considerably lower than that calculated from the acrylic acid content of the feed (AN = 64 mg KOH/g resin nonvolatile). This difference is attributed to esterification of carboxylic acid groups during solvent stripping at the boiling point of the 2-butoxyethanol solution. Resin A was prepared at 75% nonvolatile and stripped to reach the value reported in Table 1. Resins B and C were not stripped, and the experimental acid numbers are much closer to the calculated value which is probably slightly high due to neglect of initiator contribution to polymer weight and variation in the acid number of the acrylic acid monomer used.

Molecular weights were determined with a Waters 501 Chromatograph. The  $10^5$ ,  $10^4$ ,  $10^3$  and 500 Å Styragel columns were used. Tetrahydrofuran was used as the carrier solvent at a flow rate of 2 ml/min. Standardization was done using a series of 10 nearly monodisperse polystyrene standards supplied by Waters Associates.

Non-volatile content by weight was determined using the Federal Method 4041 schedule of three hours at 105° C in a convection oven. Sample sizes smaller than 2 g were used for coatings. A reproducibility of  $\overline{X} \pm 0.25\%$ was determined at the 99% confidence level.

#### Coating Formulation and Application

Resins were neutralized by addition of N,N-dimethyl-2-aminoethanol (DMAE) in an amount corresponding to 75% or 100% of the equivalents of carboxyl groups based on the experimental acid numbers. These values are called "extent neutralization" (EN) herein. Coatings were formulated to have 20% pigment volume concentration using TiPure<sup>®</sup> R-900-25. All formulations had an 80/20 acrylic/Cymel<sup>®</sup> 303 ratio by weight. Water and, in some cases, additional cosolvent were added to reach a spray viscosity of 1.4 to 1.6 poises as measured with a Wells-Brookfield cone and plate viscometer at a shear rate of 75 sec<sup>-1</sup>.

Coatings were applied, using an automatic spraying apparatus, to have a dry film thickness in the range of 19

to 30  $\mu$ m. The spraying apparatus was designed to move vertically mounted  $4 \times 8$  in. panels past the spray gun at a constant rate. After each horizontal pass, the spray gun mount indexed a pre-set distance upward or downward. A dry film thickness of about 25  $\mu$ m was obtained with a total of six passes; three while indexing upward and three while indexing downward. The spray gun was a vacuum feed type operated at 45 psi measured at the spray gun. The relative humidity (RH) was determined in the spray booth using a Bendix Psychron<sup>®</sup> before and after spraying. Since a controlled atmosphere room was not available, sprayouts were made at various ambient humidities determined by the weather conditions. Over a period of several months the range of relative humidities obtained by this realistic but primitive approach was from 29% to 69% RH. The airflow through the spray booth was  $0.32 \pm 0.05$  msec<sup>-1</sup> (62 fpm). The booth fan was turned off one minute after spray application was finished. Visual observations of sagging and leveling were recorded. One panel from each series was cured for 15 min at 175°C after a 15 min flash-off time and retained for film thickness measurements.

#### Sample Recovery and Analysis

Samples were collected using a spatula to scrape coatings off sprayed panels. The four times chosen for sample collection were: immediately after spray, 5, 10, and 15 minutes after spray. Sampling time averaged 40 sec and sample volumes ranged from about 2 ml (initial) to about 0.5 ml (15 min). Samples collected in this manner were analyzed for nonvolatile content by weight loss and for composition of the volatile fraction by gas chromatography. These values were combined in a calculation of grams of each volatile component remaining/100 g nonvolatile.

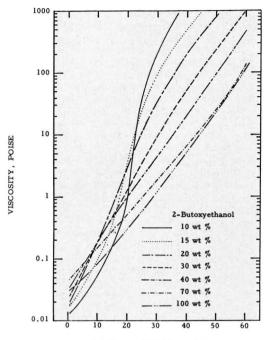
Gas chromatographic analyses were carried out using a Varian Aerograph Model 920 instrument which has a thermal conductivity detector. The recorder was a Barber-Coleman Model PR 25 with disc integrator. Samples were diluted with distilled, dried acetone to a viscosity low enough to permit loading of a microliter syringe. A disposable pyrex injection port liner was used to minimize contamination of the injection system. The following conditions were used: injection temperature, 245°C; column temperature, 100°C for one minute, programmed to 200°C; detector temperature, 245°C; helium flow rate, 55 ml/min; column, 6 ft aluminum packed with Chromosorb® 101 (Johns-Manville). Calculations were done according to the procedure given by Ettre<sup>6</sup> using relative response factors. A standard, which was a carefully prepared paint sample, was used to obtain new relative response factors on a day to day basis. Recalibration with every use helped eliminate the effect of slight variations in temperature and flow rate.

# **Viscosity Determinations**

A Ferranti-Shirley cone and plate viscometer was used to determine viscosities at various stages of dilution of pigment-free solutions (Resin A) and to determine the viscosity of paint compositions formulated to match

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WEIGHT PERCENT POLYMER

Figure 1—Variations in viscosity with dilution of resin A with different constant percentages of 2-butoxyethanol in water and with pure 2-butoxyethanol. EN = 100% with DMAE

compositions of samples recovered after spraying. Except where otherwise noted, measurements were made at  $25 \pm 0.05^{\circ}$  C and a shear rate of 814 sec<sup>-1</sup>. The viscometer was standardized using 1.01 poise, 10.6 poise, and 307 poise standards supplied by Brookfield Engineering Laboratories, Inc.

#### **Data Collection and Treatment**

Thirty-eight solvent loss experiments were carried out. Variables that were investigated (i.e., the independent variables) and the range of variation studied were as follows: initial water level, 161 to 181 g/100 g nonvolatile; initial 2-butoxyethanol level, 18 to 40 g/100 g nonvolatile; initial sec-butanol level 0 to 22 g/100 g nonvolatile; relative humidity, 29 to 69% RH; and dry film thickness, 19  $\mu$ m (0.75 mil) to 30  $\mu$ m (1.2 mil). The term "initial" as used above refers to the "reduced-to-spray" condition.

Variables which are known to or which are thought to affect solvent loss rates but which were held constant in this study include: resin type (resin B was used in all cases), pigment type and level, melamine type and concentration, spray viscosity (1.4 to 1.6 poises), air flow pattern (room ventilation conditions and spray booth on/off times were the same for each sprayout), and temperature ( $23.5 \pm 0.5^{\circ}$  C). Initially, the intent was to include extent neutralization as an independent variable at two levels, 75% and 100% EN; however, initial analysis indicated that this variation did not cause detectable differences in solvent loss rates. Therefore, extent neutralization was treated as a constant in subsequent data treatment.

A computer model for solvent loss was developed by analyzing data from the 38 sprayouts using the statistical analysis system (SAS 72) designed by A. J. Barr and J. H. Goodnight at North Carolina State University, Raleigh, N.C. The model permits calculation of grams of each volatile component remaining per 100 grams nonvolatile at each analysis time (0, 5, 10 and 15 min after spray) for any set of independent variables selected within the range of variation investigated. Details are given in the Appendix.

# **RESULTS AND DISCUSSION**

## **Viscosity Reductions**

The variation in viscosity of Resin A (*Table* 1) was determined at several fixed cosolvent/water ratios as a function of resin nonvolatile as shown in *Figure* 1. The 2-butoxyethanol curve is typical of the reduction of a solvent soluble resin; the logarithm of viscosity is nearly linear with decreasing nonvolatile content. The other curves show that as the ratio of 2-butoxyethanol is decreased, the dependence of viscosity on nonvolatile content becomes nonlinear. Comparison of the 10/90 (wt

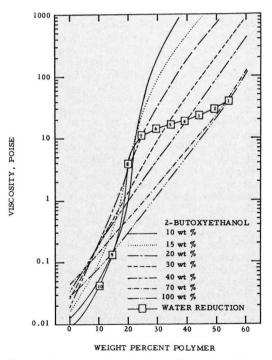


Figure 2—Comparison of mixed solvent and pure water dilutions of resin A. EN = 100% with DMAE. The weight percentages of 2butoxyethanol in water, which are reached on the water dilution curve, are indicated by the boxed numbers as follows: (1) 100%; (2) 57.1%; (3) 46.7%; (4) 38.1%; (5) 33.8%; (6) 24.5%; (7) 19.0%; (8) 14.3%; (9) 10.1%; (10) 6.3%.

#### Table 2—Solvent Loss During Spray Application at 23°C

Example 1—Sprayed at 45% RH. Dry film thickness, 20  $\mu\text{m}$ 

Grams Volatile Remaining Per 100 g Non-Volatile

	Reduced to Spray	Immediately after Spray	after	10 min after Spray	15 min after Spray
Water	169	119	102	88	76
2-butoxyethanol	40	35	33	32	31
Sec-butanol	0	0	0	0	0
Nonvolatile (wt %)	32	39	43	45	48

Example 2—Sprayed at 60% RH. Dry film thickness, 28 µm

Grams Volatile Remaining Per 100 g Non-Volatile

	Reduced to Spray	Immediately after Spray	5 min after Spray	10 min after Spray	15 min after Spray
Water	161	132	123	112	104
2-butoxyethanol	20	16	16	15	14
Sec-butanol	20	3	0.9	0.2	0.0
Nonvolatile (wt %)	33	40	42	44	46

%) 2-butoxyethanol/water dilution curve with that for dilution with pure 2-butoxyethanol shows that at 30 wt % polymer the viscosity of the former is 1000 times greater, at 15 wt % polymer the viscosities are equal, and below 15 wt % the viscosity of the 10/90 system is lower than that observed for pure cosolvent dilution. Similar results have been found using tert-butanol or 1-propoxypropanol as the organic cosolvent (curves not shown).

Viscosities of solutions represented in Figure 1 decreased with increasing shear rate. For example, for the 15% 2-butoxyethanol reduction at 30 wt % polymer, the following viscosities were obtained: 95 poises at 163 sec<sup>-1</sup>, 80 poises at 814 sec<sup>-1</sup> (plotted in Figure 1), 66 poises at 1628 sec<sup>-1</sup>, and 50 poises at 3256 sec<sup>-1</sup>. The nonlinear shape for low 2-butoxyethanol/water dilutions and the shear thinning behavior can be explained in terms of aggregate or micelle formation.<sup>1-3</sup> The aggregates consist of hydrophobic segments in the interior and ionic or polar groups on the periphery. Cosolvent absorption into the interior causes swelling and results in an increase in internal phase volume.<sup>4</sup> At high wt % polymer, crowding of swollen aggregates contributes strongly to viscosity. Shear rate dependence is considered to result from deformation or destruction of aggregates by shearing forces. As solvent mixture is added, crowding becomes less pronounced, and eventually the viscosity is determined mainly by the viscosity of the continuous phase.

For higher 2-butoxyethanol/water reductions, results suggest that aggregation is greatly reduced. Plots of viscosity versus nonvolatile content are nearly linear (*Figure* 1) and shear thinning is slight. For example, for the 30% 2-butoxyethanol reduction at 30 wt % polymer, the following viscosities were obtained: 9.2 poises at 163 sec<sup>-1</sup>, 8.9 poises at 814 sec<sup>-1</sup> (plotted), 8.6 poises at 1628 sec<sup>-1</sup>, and 8.3 poises at 3256 sec<sup>-1</sup>.

In practice, reduction of water soluble systems usually involves addition.of pure water to cosolvent rich polymer solutions. Such a reduction of resin A is shown in Figure 2 superimposed on the constant cosolvent/water curves from Figure 1. For the added curve the cosolvent/water ratio decreases throughout the reduction. Regardless of whether a point is reached with a decreasing cosolvent/ water ratio or a fixed cosolvent/water ratio the viscosity is the same for the same wt % polymer and cosolvent/water ratio. For example, when the water reduction curve reaches 15 wt % polymer (point 9), the 2butoxyethanol content has been reduced to 10.1% and. therefore, the curve intersects the 10% 2-butoxyethanol constant cosolvent/water reduction curve. Viscosity is a function of wt % polymer and cosolvent/water ratio. A shoulder or peak in the water dilution curve is now interpreted as the combined effects of moving to a lower nonvolatile content (decreases viscosity) and a lower cosolvent/water ratio (increases viscosity). These effects offset each other at higher wt % polymer levels which explains the observation of a plateau region as observed for the water reduction in Figure 2. Other studies of water reduction<sup>2,3,4</sup> indicate that in some cases a peak rather than a plateau results from these opposing effects. Below a certain wt % polymer viscosity begins to decrease rather than increase as the cosolvent/water ratio decreases. This combined with the decrease due to lowering of the wt % polymer causes a precipitous drop in viscosity with water dilution.

The observation that viscosity depends only on composition and not on the type of reduction (pure water or 2-butoxyethanol/water) used to reach the composition suggests that an equilibrium state of aggregation is reached. If equilibrium is reached, the equilibration time must be shorter than elapsed time between two successive viscosity determinations on the dilution curve. In this study the elapsed time was approximately 10 min.

#### Solvent Loss During Application

As indicated above the viscosity of water-reducible systems depends on cosolvent/water ratio as well as on nonvolatile content. During spray application of waterreducible coatings both of these variables will change. Furthermore, the rate of water loss will depend on ambient relative humidity. Two examples of solvent loss experiments, which illustrate these variations, are given in *Table 2*.

The first example indicates that at 45% RH, 30% of the water [100 (169-119)/169] evaporated during the spraying operation. The second example indicates that at 60% RH, only 18% of the water evaporated during spraying. This difference would result in a substantial difference in nonvolatile content on the sprayed panel. However, since the RH effect is easily anticipated, one might want to offset the lower water loss at 60% RH by using a faster evaporating cosolvent along with 2-butoxyethanol. In example 2, sec-butanol was used for this purpose, and it can be seen that with 20 g sec-butanol

the rapid sec-butanol loss did compensate for the lower water loss so that the nonvolatile content immediately after spray was 39% for example 1 and 40% for example 2.

If viscosity depended only on nonvolatile content, similar viscosities would be expected on the panels after spray. In fact, example 1 had a greater sagging tendency even though it was sprayed at lower RH. This observation is consistent with the difference in cosolvent content. In order to make use of Figure 1, the compositions immediately after spraying as given in Table 2 can be recalculated to obtain wt % polymer excluding pigment. Using densities of 1.2 g/cm<sup>3</sup> for polymer, 4.24 g/cm<sup>3</sup> for TiO<sub>2</sub> and the PVC of 20%, one arrives at 25 wt % polymer in the cosolvent/water mixture for both examples. The volatiles contain 22.7 wt % cosolvent for example 1 and 12.6 wt % cosolvent for example 2. Interpolation on Figure 1 at 25 wt % polymer gives viscosities of pigment-free variations of examples 1 and 2 of about 8 poises and 35 poises respectively, both at a shear rate of 814 sec<sup>-1</sup>. At the much lower shear rates characteristic of sagging, the 35 poise value could easily increase sufficiently to prevent sagging in example 2 but even with a substantial increase in the 8 poise value prevention of sagging would be unlikely for example 1. Although numerous assumptions are made in this rough calculation, it serves to emphasize the importance of cosolvent/water ratio in sag control.

In general, leveling was not a problem in these sprayouts while sagging was frequently observed. Use of a faster evaporating cosolvent, such as sec-butanol, along with 2-butoxyethanol results in two effects that reduce sagging tendency. First, the amount used can be adjusted to compensate for slow water loss on humid days, and, second, the greater decrease in cosolvent/water ratio during spray results in a higher viscosity on the panel immediately after spray. These advantages must be balanced against a solvent popping tendency of water reducible thermosetting systems caused by late water release after cure has begun.<sup>7</sup> Retention of a slow evaporating solvent diminishes the popping tendency.

Amine (DMAE) loss is not included in *Table* 2 mainly because the amine peak was not well resolved in the gas chromatograms. The "reduced-to-spray" amine content was 4.4 g/100 g nonvolatile and integrations of amine shoulder on the g.c. peak after application gave amine contents of  $4.1 \pm 0.4$  g/100 g nonvolatile. The only conclusion this level of uncertainty permits is that DMAE loss is slow at ambient temperatures. The nonvolatile contents reported in *Table* 2 are calculated based on resin, water, and cosolvent contents with the small amount of amine excluded.

Table 2 illustrates an experimental difficulty associated with solvent loss studies. There are many variables, and our ability to select values for them is limited. For example, if we want to use different organic cosolvents and hold spray viscosity constant, the amount of water required to reach the selected viscosity depends on the cosolvent. Therefore, we cannot vary cosolvent composition and water content independently. Control of dry film thickness proved to be difficult, and as a result this variable was known only after experimental

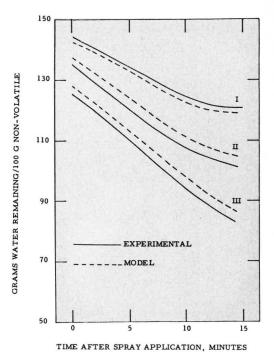


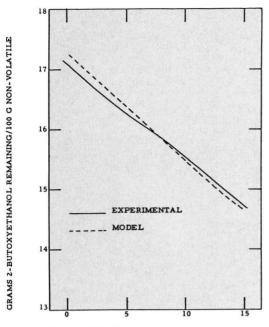
Figure 3—Water remaining for three resin B experiments (——) compared with values calculated from the model (----) for the following conditions:

	1	н	III
Initial water (g/100 g)	178	181	177
Initial sec-butanol (g/100 g)	0	0	0
Initial 2-butoxyethanol (g/100 g)	20.0	20.0	20.0
Relative humidity (%)	61	52	51
Film thickness (µm)		23	19

determination. The computer model described in the Appendix proved to be very useful for analyzing the factors controlling solvent loss under these conditions.

The grams of water remaining depended on all five independent variables (See Appendix). Dependence on initial 2-butoxyethanol and initial sec-butanol may be due to changes in the activity coefficient of water resulting from water-organic solvent interactions. The effects on solvent loss of activity coefficient variations are well documented for polymer-free systems.8,9 One contribution to non-ideal behavior may be surface active behavior of these solvents in water.<sup>10</sup> In contrast, grams of either cosolvent remaining depended only on its initial level and film thickness. Failure to detect dependence of cosolvent loss on water content may result from a relatively narrow range of variation in initial water level (161 to 181 g/100 g nonvolatile). The range of variation in initial water level was restricted by the necessity to work with sprayable paints.

Experimental results for grams of water remaining per 100 g nonvolatile are compared with the corresponding values calculated from the model in *Figure* 3. The variation of the experimental results from those calculated using the model are typical, and are considered



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Figure 4—Experimental results for 2-butoxyethanol remaining (—) compared with values calculated from the model (-----) for the following conditions: resin B; initial water, 177 g/100g NV; initial sec-butanol, 0 g/100g; initial 2-butoxyethanol, 20.2 g/100g; relative humidity, 38%; film thickness, 25 μm

to represent random variation associated with imperfect control of conditions (temperature, airflow, etc.) as well as analytical uncertainties. The relative positions of I, II, and III are readily understood in terms of the independent variables. In experiment I, the largest amount of water remains because the RH is highest and the film is the thickest. In experiment III, the smallest amount of water remains primarily because the film is the thinnest, and, to a lesser extent, because both the RH and the initial water level are slightly lower than for I and II.

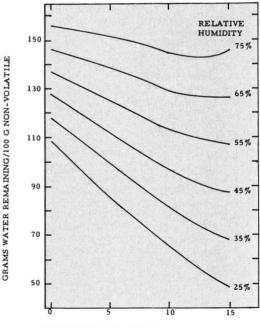
Experimental results for grams of 2-butoxyethanol remaining per 100 g nonvolatile are compared with the corresponding values calculated from the model in *Figure* 4. The agreement between the model and the experimental curve shown in this figure is typical of that obtained for other sprayouts carried out with the same initial 2-butoxyethanol content and dry film thickness.

Once an acceptable model is obtained, it can be used to calculate the effect of systematic variation in one of the independent variables while the other independent variables are held constant. In *Figure 5*, the effect of RH on water remaining is considered. The "zero time" point, corresponding to immediately after spray, permits calculation of the percent water lost during spraying. At 25% RH,  $[(177-109)/177] \times 100 = 38\%$  of the water is lost during spraying while at 75% RH only  $[(177-155)/177] \times 100 = 12\%$  of the water is lost during spraying. During the first 15 min after spray, RH continues to

dominate over water level in control of water loss rate. In other words, even though the 25% RH system contains the lowest amount of water after spray, it loses water fastest. At 15 min after spray, 72% of the initial 177g of water has been lost at 25% RH, while at 75% RH, only 20% of the water has been lost.

# **Critical Relative Humidity (CRH)**

Dillon<sup>5,11</sup> introduced the concept of critical relative humidity (CRH) to describe the effect of RH on the composition of unvaporized volatile components during evaporation. For a two-solvent system consisting of water and a cosolvent, volatiles are lost in proportion to their concentration when RH=CRH: thus the unvaporized volatiles are of constant composition at CRH. Systems consisting of water and two cosolvents have been analyzed by Rocklin.<sup>12</sup> Data for the two-solvent system represented in Figures 4 and 5 were recalculated to test the CRH concept for a pigment and polymer containing system. It is evident in Table 3 that the 2butoxyethanol concentration in the unvaporized volatile fraction remains nearly constant at 65% RH. Therefore, the CRH of this system is about 65%. Although computer simulation has been used to calculate CRH of polymer containing systems,<sup>11</sup> Table 3 represents the first report of determination of CRH for a polymer and pigment containing system.



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Figure 5—Variation in water remaining with time after spray application as calculated from the model for the following conditions: initial water, 177 g/100g NV; initial sec-butanol, 0 g/100g; initial 2-butoxyethanol, 20.2 g/100g; film thickness, 25  $\mu$ m; relative humidity, variable as indicated

Table 3—Weight Percent 2-Butoxyethanol in the Unvaporized
Volatile Portion as a Function of Relative Humidity

After Spray Minutes

	Time	Anter Sp	ray, wiii	lutes
Relative Humidity (%)		5	5 10	
75	10.0	9.75	9.78	9.12
65	10.6	10.6	10.7	10.4
55	11.3	11.6	12.1	12.0
45	12.0	12.8	13.7	14.3
35	12.8	14.3	16.0	17.7
25	13.8	16.1	19.1	23.1

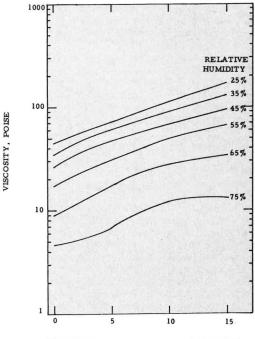
Dillon's calculations indicate that at 25° C and zero air flow rate, a polymer and pigment free system at 12 vol % 2-butoxyethanol has a CRH of about 80% (interpolation in Table 1, ref. 5). The 65% CRH reported here is obtained at 10.6 wt % or 11.6 vol %. Although conditions are not exactly identical, the paint system has a significantly lower CRH than that calculated for the solvent mixture. Assuming that the calculated value of 80% is correct for the solvent mixture, one can consider possible reasons for the lower value for the paint in terms of Dillon's analysis and in particular equations (12) and (21) in reference 5. Possible reasons include the effect of pigment, a simple dilution effect (colligative) by nonvolatile polymer, changes in activity coefficients (escaping tendencies), and partitioning of cosolvent and water between aggregates and continuous phase. At low pigment loadings, pigment is predicted to have a negligible effect on CRH.5 The colligative effect of polymer addition is proportional to mole fraction, and since the molecular weight of polymer ( $\overline{M}_n = 9500$ ) is so large compared to solvent molecular weights, changes in mole fractions of solvents are very slight so that the colligative effect on CRH is essentially negligible. The

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Figure 6—Variation in viscosity with time after spray application for resin B systems at selected relative humidities. (See Figures 4 and 5 for compositions)

activity coefficients are undoubtedly altered by polymer, and this effect is likely to be greater for cosolvent than for water because cosolvent concentration is much lower. If the activity coefficient of cosolvent, relative to the activity coefficient for water, were decreased by polymer, the CRH would increase, and if the activity coefficient of cosolvent were increased by polymer, the CRH would decrease as is observed. If cosolvent is preferentially absorbed in the aggregate interior, its effective concentration in the continuous phase would be decreased which could cause a decrease in CRH as is observed. The two effects which are in agreement with experiment would be an increase in activity coefficient of cosolvent and absorption of cosolvent by aggregates. Further study will be required to establish the dominant effect.

Table 3 indicates that over a 15 min period, the extent of enrichment or depletion of 2-butoxyethanol in the remaining volatile portion is small unless there is a large difference between RH and CRH. For instance at 75% RH, 2-butoxyethanol was only depleted from 10.0 to 9.1 wt %, and at 55% RH 2-butoxyethanol was enriched from 11.3 to 12.0 wt %. Although these changes in composition are not very great, *Figure* 1 indicates that at certain polymer concentrations their effect on viscosity could be substantial. Of course, when RH is far from CRH, a much larger change in composition of remaining volatiles occurs. The effect of these composition changes on viscosity of paints is considered in more detail in the next section. Table 4—Compositions at Spray Viscosity (1.5 poises) And Calculated Compositions Immediately after Spray At Various Relative Humidities for Resins B and C

### **Reduced to Spray**

	Grams Volatile Per 100 g Nonvolatile			
Component	Resin B	Resin C		
Water	177	174		
Sec-butanol	0	0		
2-butoxyethanol	20.2	14.4		
Non-Volatile (wt %)	33.6	34.6		

#### **Immediately After Spray**

	Grams Water Remaining Per 100 g Nonvolatile		
Relative Humidity (%)	Resin B	Resin C	
75	156	150	
65	147	141	
55	137	131	
45	128	122	
35	118	112	
25	109	103	
	Grams 2-butoxye per 100 g l		
	Resin B	Resin C	
All RH's	17.4	11.9	

#### Analysis of Sagging

As noted above sagging was observed in some of the sprayouts. In order to determine the viscosities corresponding to visual observation of sagging, 24 paints were formulated to match the compositions calculated from the model and plotted in Figures 4 and 5. These 24 formulations were prepared because six RH values were considered with four analysis times for each RH. The viscosities of these "matching" formulations are considered to be the same as the viscosities of the paint on the sprayed panels at corresponding RH and analysis times. Using this approach it is possible to plot the viscosity of the coating on the panel as a function of time after spray for various relative humidities as indicated in Figure 6. As expected, the lowest RH results in the greatest water loss during spray (Figure 5) and, therefore, the highest viscosity after spray application (Figure 6). Over the time interval evaluated, the viscosity increases with time after spray application for all RH values. Thus "delayed sagging," which results from changes in cosolvent/water ratio in some water soluble paint systems,<sup>1,3</sup> was not observed for this system within a 15 min period.

The effect of RH on the change in viscosity with time, as shown in *Figure* 6, can be explained in terms of nonvolatile content and 2-butoxyethanol/water ratio. Since 65% is the CRH, the 2-butoxyethanol/water ratio is constant, and the viscosity increase is attributed to the increase in nonvolatile content from 38% to 41.6%. On the basis of nonvolatile content, one would expect viscosity to increase by a larger factor for the 25% RH curve because the change in nonvolatile is much greater, 44% to 60.8% over 15 min. Since the observed proportional increase in viscosity at 25% RH is approximately the same as that at 65% RH, the off-setting effect of the increase in 2-butoxyethanol/water ratio must be substantial in pigmented systems as well as in pigmentfree systems (*Figure* 1).

Application properties were also determined for a higher molecular weight resin (resin C, see *Table* 1). Sprayouts of the resin C system were a little less subject to sagging than sprayouts of the resin B system. The resin C system did not sag unless the RH was above 60%, whereas the resin B system sagged when the RH was above 50%. *Table* 4 gives the compositions of the two systems in the "reduced-to-spray" condition and the amount of each volatile remaining immediately after spray at various relative humidities, as calculated from the model.

Table 4 provides an opportunity for analysis of factors that control nonvolatile content at spray viscosity. Despite the fact that the  $\overline{M}_w$  of resin C is about twice that of resin B, a spray viscosity of 1.5 poises was reached at nearly equal nonvolatile contents, 34.6% and 33.6%. This result is attributed to a weak dependence of viscosity on

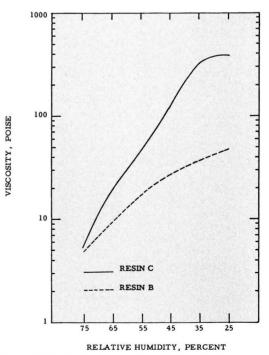


Figure 7—Variation in viscosity with relative humidity for resin B and resin C systems at zero time after spray application. (See Table 4 for compositions)

Table 5-	-Extrapolate	ed Low	Shear	Viscosit	ies at	Zero	Time
	After Spray	Applic	ation a	t Variou	s R.H	.'s	
	For Res	in B an	d Resi	n C Syst	ems		

	Viscosity in Poises at $\gamma = 1.0$ sec			
telative Iumidity (%)	Resin B	Resin C		
75	13	13		
65	40	50		
55	52	79		
45	100	167		
35	143	397		
25	190	563		

molecular weight in systems of this type<sup>4</sup> and to the lower 2-butoxyethanol/water ratio for the resin C system. Calculation of the weight percent polymer in the vehicle (pigment excluded) results in values close to 22 wt % polymer for both systems. The 2-butoxyethanol/water ratio is 8/92 for resin C and 10/90 for resin B. Reference to Figure 1 indicates that in these ranges of wt % polymer and 2-butoxyethanol/water ratio, reducing the ratio causes a decrease in viscosity. Thus, if the higher molecular weight of resin C tends to cause an increase in viscosity, this effect is off-set by the lower 2-butoxyethanol/water ratio.

In order to obtain viscosities corresponding to those of coatings on the panels immediately after spraving, six formulations were prepared to match the calculated compositions given for resin C in Table 4. Since each composition represented a different RH, it was then possible to plot viscosity at zero time after spray versus RH, as shown in Figure 7. The zero time points for resin B from Figure 6 are replotted here versus RH for comparison. At 75% RH the viscosity immediately after spraying is low for both systems, and sprayouts at RH values approaching 75% were observed to sag for both systems. For lower relative humidities, the viscosity of the resin C system immediately after spraying was substantially larger than for the resin B system. This difference is particularly important in the 50 to 60% RH range where the resin B sprayouts sagged, but the resin C sprayouts did not. Below 50% RH sprayouts did not sag for either system.

Although the higher viscosities for the resin C system, as shown in Figure 7, are consistent with the reduced sagging tendency compared to resin B, specification of the viscosity required to prevent sagging is not possible because the results in Figure 7 are for a shear rate of 814 sec<sup>-1</sup>, whereas the shear rate associated with sagging is very much lower. Therefore, the viscosities of both systems were determined as a function of shear rate. The results were analyzed using an equation from Patton<sup>13</sup> for non-Newtonian systems with no detectable yield value. The fit was determined to be satisfactory statistically, and the equation was used to calculate viscosities at 1 sec<sup>-1</sup> as indicated in Table 5. This represents an extrapolation because the instrumentation available did not permit direct measurement of viscosities at shear rates as low as 1.0 sec<sup>-1</sup>. The value of 1 sec<sup>-1</sup> is a compromise between actual sagging shear rates (which are below  $1 \sec^{-1}$ )<sup>14</sup> and the extent of extrapolation. Consideration of *Table* 5 along with the observation that resin B systems did not sag below 50% RH and resin C systems did not sag below 60% RH suggests that a viscosity of about 50 poise or greater at  $1 \sec^{-1}$  shear rate is required to prevent sagging with a wet film thickness of about 75  $\mu$ m in systems that have no detectable yield value. This 50 poise requirement is an approximate agreement with a rheological analysis by Wu<sup>14</sup> of sagging in high solids coatings.

# SUMMARY

The volatiles remaining in paint films immediately after spray application and at five minute intervals up to 15 min after spray have been analyzed. Paints were prepared using acrylic copolymers similar to those used in commercial thermosetting topcoats. N,N-dimethyl-2aminoethanol was used as the neutralizing amine, and either 2-butoxyethanol or a mixture of 2-butoxyethanol and sec-butanol was used as the organic cosolvent. Among the many conditions and composition variables that would be expected to affect the amount of volatiles remaining, those held constant or controlled in a reproducible manner were temperature, air flow pattern, spray viscosity, polymer type, pigment type and level, and crosslinker type and concentration. Two extents of neutralization (75% and 100% EN) were used, but for these EN values statistical analysis indicated no effect of EN on volatile composition. Variables investigated were initial (i.e., reduced-to-spray) water content, initial 2butoxyethanol content, initial sec-butanol content, relative humidity, and film thickness.

The effect of these five variables on the amount of volatile components remaining after spraying was determined by gas chromatography in 38 experimental sprayouts. The data were used to obtain a computer model which could be used to calculate the grams of each volatile component remaining for any selected set of the five independent variables. Statistical analyses showed that all five variables contributed significantly to accounting for the variation in "water remaining" (i.e., the grams of water remaining per 100 g nonvolatile). In contrast, the "2-butoxyethanol remaining" could be accounted for almost as well with two variables (initial 2butoxyethanol and film thickness) as with all five, and in a similar manner, "sec-butanol remaining" could be accounted for almost as well with two variables (initial sec-butanol and film thickness) as with all five variables.

The viscosity of polymer solutions was determined as a function of weight percent polymer for dilutions carried out with constant cosolvent/water mixtures and with pure water. The results of these determinations were used to facilitate interpretation of the effect of solvent losses on viscosity. Conclusions based on the dilution experiments are as follows:

(1) At high wt % polymer, increasing the organic cosolvent/water ratio causes a decrease in viscosity.

(2) At low wt % polymer, increasing the organic cosolvent/water ratio causes an increase in viscosity.

(3) The plateau observed at high wt % polymer during

water dilution of cosolvent rich solutions results from off-setting effects of a decrease in nonvolatile content (decreases viscosity) and a decrease in organic cosolvent/ water ratio (increases viscosity).

(4) After each solvent addition an equilibrium extent of polymer aggregation is apparently established in a relatively short period (< 10 min).

The model was used to study the effect of relative humidity on the amount of water lost during spray application, and on the cosolvent/water ratio in the unvaporized volatile portion. For example, with other variables held constant, 38% of the water initially present was lost during spraying at 25% RH while only 12% was lost at 75% RH. The critical relative humidity (CRH) of a system containing pigment and neutralized resin as well as 2-butoxyethanol and water was determined to be 65%. Above 65% RH evaporation resulted in depletion of 2butoxyethanol in the remaining volatile portion, below 65% RH evaporation resulted in enrichment of 2butoxyethanol in the remaining volatile portion, and at 65% RH evaporation caused no change in the composition of the remaining volatile portion. However, the extent of enrichment or depletion over a 15 min period was found to be small unless there was a large difference between RH and CRH.

An analysis of sagging tendency was carried out. The systems were shear thinning but did not have a detectable yield value. It was observed that a viscosity of approximately 50 poises at a shear rate of  $1.0 \sec^{-1}$  was required to prevent sagging with a wet film thickness of about 75  $\mu$ m. A resin having  $\overline{M_w} = 42,000$  in a representative thermoset formulation was observed to sag when the humidity during spraying was above 50% RH, and a resin having  $\overline{M_w} = 82,000$  was observed to sag when the humidity during spraying was above 60% RH. The model was used

in the sagging analysis to calculate the composition on sprayed panels so that matching compositions could be formulated for rheological characterization.

# ACKNOWLEDGMENTS

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# APPENDIX

The regression procedure which was used applies the principle of least squares in fitting a linear model to data. The regression estimates the parameters (the beta's) of an equation such as (1).

Water remaining after = beta<sub>0</sub> + beta<sub>1</sub> (Initial water) (1) spray application + beta<sub>2</sub> (Initial sec-butanol)

+ betas (RDFT)\*

"Water remaining after spray application" is the dependent or response variable and "initial water," "initial sec-butanol," etc., are the independent variables. The analysis includes the coefficient of determination ( $r^2$ ) and a t-statistic. The coefficient of determination is the fraction of total variation in the dependent variable which is accounted for by the chosen independent variables. The t-statistic is used to test whether a given beta improves the fit significantly or could about as well be taken as zero.

The coefficients of determination were: 96% or greater for "water remaining" at all analysis times; 93% or greater for "2butoxyethanol remaining" at all analysis times; and 97% immediately after spray, 86% 5 min after spray, and 66% 10 min after spray for "sec-butanol remaining." The decrease in coefficient of determination for sec-butanol is related to the very low levels remaining after 5 min.

Use of the t-statistic indicated that all five beta's had a high probability of being non-zero values for "water remaining." In contrast, for "2-butoxyethanol remaining" only the beta values associated with "initial 2-butoxyethanol" and "RDFT" had a high probability of being non-zero values. In a similar manner, "sec-butanol remaining" could be accounted for almost as well with just two independent variables ("initial sec-butanol" and RDFT) as with all five independent variables.

<sup>•</sup> RDFT is reciprocal dry film thickness. Use of the reciprocal resulted in a positive coefficient.

# Linseed Oil-Metal Acetylacetonate Systems

# V. Thermogravimetry **Of Iron-Based Pigments**

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and

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Thermogravimetric data (weight change at constant temperature) are presented in the temperature range 60-100°C in air for linseed oil, containing small quantities (2 mg/5 ml) of fifteen iron-based pigments. Inhibition periods, rate of increase in weight, maximum weight achieved, and subsequent weight decrease are reported. Temperature parameters are presented. These data are compared to data obtained for iron acetylacetonates. Iron pigments, at the concentrations used, produced thermogravimetric curves very little different from linseed oil alone.

# INTRODUCTION

The literature reporting the behavior of artists' pigments in drying oils makes frequent reference to significant variations in the rate of drying and character of films formed as a function of the pigment composition.<sup>1-4</sup> Typical results are reported in Table 1 for a range of ironbased pigments in linseed oil.

In a previous study,<sup>5</sup> the intention was to isolate the effects on the drying process of individual cations by using acetylacetonate as a common ligand. In this study, the authors have attempted to extend this work by varying the ligand while using a common metal species, iron. The general lack of major differences in behavior among the several iron-based pigments studied clearly demonstrates that the observations reported in Table 1 do not arise from homogeneous solution autooxidation phenomena.

In a recent communication, the thermogravimetric behavior (weight change at constant temperature) of

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linseed oil containing small amounts (10<sup>-3</sup> molar) of metal acetylacetonates was reported.<sup>5</sup> Two broad categories of behavior for the metal acetylacetonates were observed: one in which a weight gain occurred markedly faster (with lower temperature parameters) than linseed oil alone; and a second in which weight was gained little or no faster than was observed for linseed oil alone. Among the former were  $Co^{2^+}$ ,  $Co^{3^+}$ ,  $Fe^{2^+}$ ,  $Mn^{2^+}$ , and  $Mn^{3^+}$ . Included among the latter were  $Al^{3^+}$ ,  $Cr^{3^+}$ ,  $Cu^{2^+}$ ,  $Fe^{3^+}$ ,  $Ni^{2^+}$ ,  $TiO^{2^+}$ ,  $V^{3^+}$ ,  $VO^{2^+}$ ,  $Zn^{2^+}$ , and  $Zr^{4^+}$ .

In this study, the effect of iron-based pigments on the thermogravimetric drying of linseed oil has been studied under conditions similar to those used for metal acetylacetonates.<sup>6</sup> An effort is made in this study to establish comparisons between the behavior of real pigments and metal acetylacetonates used as pigment surrogates in previous studies.5-9

The pigments chosen for this study are contained in the Pigment Collection at the Institute of Fine Arts, New York University. Table 2 lists the pigments, iron analyses reported in the literature, and iron analyses obtained for samples used in this work.

#### **EXPERIMENTAL**

## **Reagents, and Solutions**

Metal acetylacetonates (McKenzie Chemical Corp.) were recrystallized from chloroform. Artists' refined linseed oil (Grumbacher 588) was used without further treatment. Pigments are described in Table 2.

For each of the pigments studied, a weighed amount was ground with an aliquot of linseed oil to obtain a fine and uniform suspension which contained 2 mg of pigment per 5 ml of linseed oil. Table 2 lists the calculated iron content in each linseed oil sample based on iron analyses of the pigments obtained from Schwartzkopf Microanalytical Laboratories.

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Table 1—The Drying Properties
And Film Characteristics of Iron Bearing Pigments <sup>1-4</sup>

	Film	
Drying Speed	Characteristics	
rapid	tough, flexible	
rapid	hard	
rapid	hard, fairly strong	
average	tough, fairly strong	
average	strong	
average	strong	
average	strong	
slow	fairly strong	
average-slow	usually brittle	
	rapid rapid rapid average average average average slow	

#### **Iron Analyses**

Iron analyses for the pigments by titration with dichromate were performed by Schwartzkopf Microanalytical Laboratories, Woodside, N. Y. 11377.

#### **Thermogravimetric Procedure**

The procedure for obtaining the thermogravimetric data has been described previously.<sup>5</sup>

# **RESULTS AND DISCUSSION**

Table 3 presents a summary of the thermogravimetric data at 60°, 80°, and 100°C for linseed oil samples containing iron-based pigments, together with data obtained for ferrous and ferric acetylacetonates, as well as data obtained in the absence of added pigment for comparison. Data include inhibition times, the maximum rate of increase in weight (%/hr), the maximum weight observed (as % of initial weight), the time (hr) at which the maximum weight gain is observed, and the rate of decrease in weight after the maximum weight gain has been achieved.

The general shape of all the curves (weight change pattern) is similar and consistent with those previously reported<sup>1</sup>: a steep rise after a short inhibition period,  $t_1$ , (generally shorter at elevated temperatures), a maximum followed by a downward curve of lesser slope than the initial rise preceding the maximum. Both the rates of weight gain,  $R^+$ , and weight loss,  $R^-$ , increase with temperature. The time of maximum weight gain,  $t_{max}$  or  $(t_{max}-t_1)$ , occurs sooner with increasing temperature. The value of the maximum weight gain,  $m_{max}$ , decreases with increasing temperature.

The  $Fe^{2+}$  and  $Fe^{3+}$  acetylacetonates appear to be distinguishable from each other. Ferrous acetylacetonate produces a larger effect; ferric acetylacetonate behaves in

Table 2—Description of Iron Containing Pigments							
Pigment	NBS No.ª	Iron Content %Fe	(Fe) × 10³ moles/liter at 2 mg/5 ml	Pigment	NBS No. <sup>a</sup>	Iron Content %Fe	(Fe) × 10³ moles/liter at 2 mg/5 m
Ferrous Acetylacetonate		20.69 <sup>b</sup>	1	Bright Red Oxide	310	67.77 <sup>b</sup> 65.26 <sup>c</sup> 68.54 <sup>a</sup>	4
Ferric Acetylacetonate	_	16.30 <sup>b</sup> 16.10 <sup>c</sup>	1	Black Iron Oxide	313	68.35 <sup>b</sup> 70.16 <sup>c</sup> 70.96 <sup>a</sup>	4
Raw Sienna	302	28.20 <sup>b</sup> 23.93 <sup>d</sup> 27.30 <sup>a</sup>	2	Yellow Iron Oxide, light lemon	314	59.91 <sup>b</sup> 60.85 <sup>a</sup>	4
Burnt Sienna	303	42.16 <sup>b</sup> 48.89 <sup>d</sup> 39.87 <sup>a</sup>	3	Yellow Iron Oxide lemon	315	60.49 <sup>h</sup> 60.85 <sup>a</sup>	4
Raw Umber	304	28.41 <sup>b</sup>	2	Yellow Iron Oxide, orange	316	60.33 <sup>b</sup> 60.15 <sup>a</sup>	4.3
Burnt Umber	305	27.30 <sup>a</sup> 42.26 <sup>b</sup>	3	Yellow Iron Oxide, dark orange	317	59.87 <sup>b</sup> 60.85 <sup>a</sup>	4.3
		37.87, 36.40 <sup>d</sup> 40.57 <sup>a</sup>		Iron Blue	325	35.06 <sup>b.f</sup>	2.5
Indian Red	308	68.26 <sup>b</sup> 69.08 <sup>c</sup>	4	French Yellow Ochre, medium	-	52.13 <sup>b</sup>	3.7
Mineral Red	309	68.54 <sup>a</sup> 57.76 <sup>b</sup> 60.51 <sup>c</sup> 51.35 <sup>a</sup>	4	French Yellow Ochre, light	_	17.20 <sup>b</sup> 13.19 <sup>d</sup>	1.2

(a) National Bureau of Standards, Standard Sample Certificates For Mass Color and

Tinting Strength.

(d) Reference 12.

(e) Reference 11.

(f) Complex potassium or ammonium ferriferro cyanide (No NBS analysis)

<sup>(</sup>b) This work.(c) Reference 10.

Pigment	Temperature, (° C)	Inhibition Period (hr) t <sub>i</sub>	Rate of Gain in Mass (%/hr) R <sup>*</sup>	Maximum Gain in Mass (%) ∆m <sub>max</sub>	Reaction Time at Maximum Mass (hr) t <sup>*</sup> max	t <sub>max</sub> -t <sub>i</sub>	Rate of Loss in Mass (%/hr) R⁻
None	60	3.2	2.0	9.7	12.0	8.5	0.13
None	80	0.50	5.1	7.1	2.4	1.9	0.42
	100	0.11	9.0	4.5	1.0	0.89	0.83
Fe(acac) <sub>2</sub>	60	0.55	4.5	10.0	4.5	4.0	0.35
(uuu)2	80	0.20	6.9	6.7	2.0	1.8	0.63
	100	.0	13.0	3.9	0.50	0.50	1.5
Fe(acac)3	60	2.8	1.7	10.0	4.5	4.0	0.35
(uuu),	80	0.20	5.8	7.1	2.6	2.4	0.35
	100	.0	10.0	4.2	0.65	0.65	1.3
Raw Sienna	60	1.30	2.44	10.7	8.38	7.08	0.170
	80	1.06	4.76	7.04	3.60	2.54	0.487
	100	0.20	9.35	5.01	1.08	0.88	1.06
Burnt Sienna	60	1.55	2.39	10.9	9.6	8.05	0.225
	80	0.96	5.12	7.57	3.60	2.64	0.515
	100	0.233	9.65	6.10	1.43	1.17	0.755
Raw Umber	60	0.37	2.26	10.5	9.20	8.83	0.160
	80	0.83	5.55	6.93	3.20	2.37	0.544
	100	0.25	9.51	5.47	1.30	1.05	0.743
Burnt Umber	60	1.67	2.86	11.0	9.60	8.93	0.190
	80	0.90	4.92	7.44	3.50	2.60	0.345
	100	0.20	7.66	5.18	1.15	0.95	0.850
Indian Red	60	1.0	3.62	10.6	6.06	5.62	0.230
	80	0.04	6.28	7.59	2.66	2.62	0.352
	100	0	11.9	5.46	0.93	0.93	0.948
Mineral Red	60	1.35	2.98	11.0	8.80	6.45	0.174
Oxide	80	0.80	5.17	7.71	3.73	2.93	0.346
	100	0.25	9.69	5.52	1.35	1.10	0.620
Bright Red	60	1.50	2.57	10.7	8.67	7.17	0.202
Oxide	80	0.34	4.22	7.08	2.60	2.26	0.352
	100	0.25	8.50	6.00	1.55	1.30	0.711
Black Iron	60	0.78	2.32	11.0	9.50	8.72	0.164
Oxide	80	0.71	5.64	7.04	2.90	2.19	0.436
	100	0.18	9.65	5.76	1.24	1.06	0.710
Yellow Iron	60	1.55	2.32	10.2	9.36	7.81	0.146
Oxide,	80	0.46	5.98	6.56	2.40	1.94	0.387
light lemon	100	0.25	8.61	5.75	1.50	1.25	0.750
Yellow Iron	60	1.25	2.28	10.8	9.20	7.95	0.170
Oxide, lemon	80	0.870	5.70	6.80	3.0	2.13	0.574
	100	0.255	9.62	5.73	1.38	1.12	0.76
Yellow Iron	60	1.90	2.42	10.4	10.8	8.90	0.154
Oxide, orange	80	0.84	5.08	7.42	3.30	2.46	0.432
onido, orange	100	0.22	10.1	5.69	1.25	1.03	0.74
X 11 - I	(0	1.24	2.42	10.0	10.22	0.00	0.121
Yellow Iron Oxide, dark orange	60 80	1.34	2.42 4.82	10.9 7.93	10.32 3.84	8.98 2.82	0.131 0.416
Oxide, dark ofalige	100	0.06	12.1	5.64	0.75	0.69	1.30
Iron Blue	60	0.50	2.09	10.7	10.4	9.9	0.137
	80 100	0.83 0.06	6.20 13.3	6.66 5.78	2.67 1.02	1.84 0.96	0.517
	100	0.00	15.5	5.76	1.02	0.90	1.07
French Yellow	60	0.60	2.30	10.5	9.36	8.76	0.149
Ochre, medium	80	0.80	5.67	7.76	3.10	2.30	0.554
	100	0.066	11.6	5.62	1.00	0.93	1.18
French Yellow	60	0.50	2.05	10.5	9.10	8.60	0.129
Ochre, light	80	0.73	5.10	7.59	3.30	2.57	0.327
	100	0.060	13.9	5.99	0.91	0.85	1.20

# Table 3—Thermogravimetric Data of Iron Containing Pigments in Linseed Oil

Sample	Ea	R <sup>+</sup> <sub>20°</sub>	Ea	R_20°
Linseed oil control	10.0	0.25	10.0	0.018
Fe(acac) <sub>2</sub>	6.0	1.3	9.7	0.014
Fe(acac) <sub>3</sub>	9.6	0.29	12.0	0.036
Raw Sienna	8.53	0.424	11.7	0.0148
Burnt Sienna	8.66	0.405	8.18	0.0394
Raw Umber	9.16	0.365	9.85	0.0224
Burnt Umber	7.71	0.582	9.47	0.0238
Indian Red	7.54	0.773	8.92	0.0311
Mineral Red Oxide	7.70	0.595	8.08	0.0310
Bright Red Oxide	7.57	0.530	7.97	0.0355
Black Iron Oxide	9.09	0.379	8.94	0.0262
Yellow Iron Oxide, light lemon	8.39	0.456	10.4	0.0165
Yellow Iron Oxide, lemon	9.18	0.368	9.62	0.0251
Yellow Iron Oxide, orange	9.07	0.378	10.0	0.0196
Yellow Iron Oxide, dark orange	10.9	0.242	14.6	0.00584
Iron Blue	11.8	0.193	13.1	0.00923
French Yellow Ochre, light	12.1	0.166	14.1	0.00599
French Yellow Ochre, medium	10.3	0.285	13.2	0.00976

a manner somewhere between linseed oil alone and the  $Fe^{2+}$  augmented samples. Samples containing added pigment are similar to linseed oil alone, regardless of the composition of the added pigment. The inhibition periods at elevated temperatures (80°, 100°C) are not completely eliminated as they are with acetylacetonates.

Table 4 presents temperature parameters for the iron pigments together with data for the acetylacetonates for comparison. The temperature parameters are obtained from a least squares analysis of the slopes of the weight gain ( $\mathbb{R}^+$ ) and weight loss ( $\mathbb{R}^-$ ) portions of the thermogravimetric curves plotted against reciprocal temperature.  $\mathbb{E}_a^+$  and  $\mathbb{E}_a^-$  are, in effect, Arrhenius activation energy values in kcal/mole obtained, as previously described<sup>1</sup> to within ±2.5 kcal/mole. Rates of

Table	5-Decomposition of	Cumene	Hydroperoxide <sup>a</sup>
	by Iron	Salts <sup>b</sup>	

	% Decomposition				
Catalyst	½ hour	1 hour	2 hours		
Iron octoate	37.3	56.7	83.7		
Iron naphthenate	25.8	47.6	75.4		
Iron tallate	27.0	59.5	91.0		
Iron oleate	15.9	22.1	35.1		
Iron acetylacetonate	-	18.0	28.0		
Octanoic acid	_	2.7	2.7		
Oleic acid	_		6.3		
None		small			

(a) 0.2 M cumene hydroperoxide in xylene;  $13.9 \times 10^{-4}$  mole/liter catalyst;  $103^{\circ}$ C; N<sub>2</sub> atmosphere. (b) Reference 13.

(c) Reference 17. CHP decomposition has  $k_1 = 81 \times 10^{-4} \text{ min}^{-1}$  at 150°C in "white oil" (c) (kcal/mole) = 29.

weight gain and loss at  $20^{\circ}$  C,  $R_{20}^{+\circ}$ , and  $R_{20}^{-\circ}$ , have been obtained from the extrapolated Arrhenius activation energy curves and are also incorporated in *Table* 4. The temperature parameter for weight increase varies between 7.5 and 12.1 kcal and for weight decrease between 8.1 and 14.6 kcal for the various pigments. These values may represent the limits of experimental error in obtaining temperature parameters for linseed oil alone.

Accuracy of the data appears to be confounded by the effect of experimental sequence. The inhibition times for the samples taken first (80° runs, also some runs on freshly opened linseed oil at 60°) are higher and the rates of weight change are slightly lower than for replicate runs at 60°. Linseed oil samples taken later contain more peroxide. Peroxide initially present would reduce the inhibition period, increase the rate of oxygen absorption, and decrease the maximum weight gain by an amount dependent on the amount and nature of the initially present peroxide. A slightly narrower range of temperature parameters is calculated when the data obtained over a narrower time span are used. The extrapolated rates (to 20°C) imply that iron pigments at these concentrations have very little effect on the drying of linseed oil (cf. comparable extrapolated data for acetylacetonates, reference 5, Table 3).

The literature of autooxidation and peroxide decomposition has indicated that the metal and its oxidation state(s) are important factors influencing reactivity, but also that the associated ligands in some instances are enormously important. For example, cumene hydroperoxide decomposition is clearly affected by the associated ligand, as shown in Table 5. Many other studies of metal-catalyzed reactions have shown that the nature of the associated ligand or anion produces dramatic effects on reactivity.<sup>14-16</sup> The nature of the ligand either encourages or discourages complexation with oxygen or peroxide, influencing the activation energy of the chain-initiating process. The variously reported behaviors of iron pigments (see Table 1 and  $5)^{1-4,6,13}$  are inconsistent with the data of this study. Tables 1 and 5 suggest the wide variations that iron compounds are reported to have on autooxidation processes. Differences among the drying properties of ironcontaining pigments are clearly indicated in artists' paint manuals. The discrepancy between our observations and

# Table 6—Thermogravimetric Data For Different Pigment Levels In Linseed Oil at 100° C mg/5ml Linseed Pigment Oil t<sub>1</sub> R' △M<sub>max</sub> t<sub>max</sub> t<sub>max</sub>-t<sub>1</sub> ione 0 0.11 9.6 4.5 1.0 0.89

None	0	0.11	9.6	4.5	1.0	0.89	0.83
Iron Blue	0.2	0.28	9.79	5.81	1.28	1.00	1.02
	2.0	0.06	10.8	5.50	1.02	0.96	1.07
	200.0	0.08	10.0	5.20	0.96	0.92	1.28
Black Iron	0.2	0.38	9.66	6.01	1.50	1.12	0.95
Oxide	2.0	0.18	9.65	5.76	1.24	1.06	0.71
	200.0	0.25	10.5	6.17	1.35	1.10	1.21

R

the reported differences in drying properties of pigments may arise from (a) synergistic effects between the iron pigment and the ground\*, (b) surface interactions between the oil and the pigment; (c) dryers added to the pigment-oil system not indicated in recipes; (d) photochemical reactions excluded by our reaction conditions; or (e) differences in solubility or wettability of pigments in real systems. It must be pointed out that the amount of pigment used in these studies is far less than amounts used in oil paintings.4 However, runs done in the presence of larger and smaller amounts of iron blue and black iron oxide (Table 6) gave results substantially similar to linseed oil alone. It should be noted further that all but two of the pigments are principally Fe<sub>2</sub>O<sub>3</sub>; that one pigment, black iron oxide, contains some ferrous oxide, and that one pigment, iron blue, is a complex ferroferricyanide. The major compositional difference among the pigments is in the amount of H<sub>2</sub>O, bound and unbound, that is present, For a more detailed account of pigment composition, NBS reference sample certificates may be consulted.

#### Solubility of Iron Pigments

The pigments (2mg/5ml) were ground with linseed oil and permitted to stand for 60 hr, filtered and centrifuged. The colors of the filtered oil were observed visually and compared to linseed oil alone. Visually detectable color changes were apparent for Burnt Sienna, Burnt Umber, Indian Red, Mineral Red Oxide, Bright Red Oxide, Yellow Iron Oxide (Lemon and Light Lemon) and Iron Blue; other linseed oil pigment solutions were visually indistinguishable from linseed oil alone. Visual color changes are accompanied by limited solubility of the pigment but even dissolved pigment had no appreciable effect on the drying of linseed oil under the experimental conditions used.

#### CONCLUSIONS

Thermogravimetric analysis of linseed oil samples containing ferric and ferrous acetylacetonates show distinguishably different rates at 60, 80, and 100°C.

Thermogravimetric analysis of linseed oil samples containing ferric acetylacetonate shows  $Fe^{3+}$  to be much less active.

Thermogravimetric analysis of linseed oil samples containing 15 iron-based pigments gave results indistinguishable from linseed oil alone at pigment levels of 2mg/5ml.

Higher and lower levels (0.2mg/5ml and 200mg/5ml) of Iron Blue and Black Iron Oxide gave results indistinguishable from linseed oil alone. LINSEED OIL-METAL ACETYLACETONATE SYSTEMS V.

The 15 iron pigments in the absence of added dryer, ground, or canvas, at the pigment levels examined do not effect the drying of linseed oil as observed thermogravimetrically.

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ground: a term describing the layer of a painting which lies between the support layer and the design layer(s). The design of the picture is executed on the ground which most often consists of a white pigment in an adhesive binding medium.

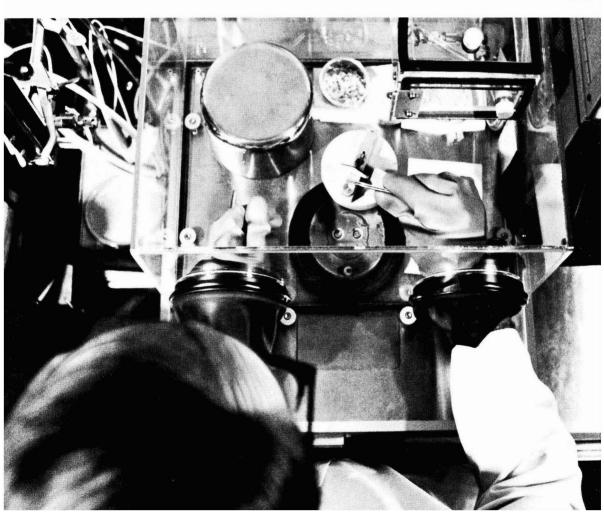
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### Behavior of Amines In Water-Dilutable Coatings And Printing Inks

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> and Kurt Bore Nielsen Bing and Grøndahl Porcelænsfabrik A/S<sup>†</sup>

Legislation exists in Scandinavia restricting the "free amine" content of coatings to less than 0.5%. This legislation was originally intended for two component epoxy coatings where interpretation of what "free amine" is, is clear. Interpretation concerning the amine added to water-borne coatings has been less clear, i.e., is it "free" or not?

This report shows that "free amine" could not be detected until all the acid groups in the water-borne systems studied were neutralized and, furthermore, that in practical systems applied at room temperature, the major part of the amine added is essentially permanently retained in the film.

#### INTRODUCTION

Developments within the coatings industry in recent years have been characterized by attempts to produce products with reduced solvent content. For many end uses, water-borne coatings have emerged as one means to achieve this goal. These products can still contain amines at a concentration which might be of environmental concern. Furthermore, legislation restricting the "free amine" content of coatings in general to less than 0.5% exists in Scandinavia.

This report discusses the behavior of amines in waterborne coatings before, during, and after application. Two aspects have been emphasized: (1) "Free amine" as a function of amine added; and (2) Amine evaporation during film formation at  $23^{\circ}$ C.

#### Free Amine in Water-borne Coatings

"Free Amine" in water-borne coatings is not a welldefined chemical (legal) concept. We have defined free amine in such systems as that amine which is not associated with the polymer phase in any way, but which can be found in the aqueous phase. It is this amine one might consider with regard to skin contact and which is presumed to be most readily available for evaporation.

Thus defined, free amine was measured indirectly by the head space method. A given volume of liquid sample is placed in a closed container and equilibrium is established. The amine content of the gas phase can be found by gas chromatography. This is then compared with the gas phase amine measured by the same technique over samples of various known concentrations of amine in pure water. Here, all the amine is presumed free. It is recognized that the amine may associate with itself, however, this is, in effect, ignored here.

Thus, the gas phase content of amine over a sample can be used to indirectly measure the amine content in the aqueous phase of a coating, assuming that this behaves similarly to water. This aqueous phase concentration is what is here called "free amine."

The head space technique requires a suitable large amine pressure from the sample, which, in turn, limits the method to the most volatile amines. Not surprisingly it was found that the water solubility of the amine was an important factor, with higher water solubility leading to lower equilibrium vapor pressure. Elementary principles often need repetition in ordinary language—especially in this industry.

#### EXPERIMENTAL RESULTS

Triethylamine (TEA) and diethyl amine (DEA) were analyzed over NeoCryl® BT-8 (40% solids acrylic emulsion from Polyvinyl Chemie), isostearic acid, dark shellac, Bevalkyd® AQ 7 (water reducible alkyd from Bergviks Hartsprodukt AB) and Soalkyd® 7710 (water reducible oil-free polyester from Berol Kemi AB). *Table* 1 shows some of the results of these analyses.

From these results it can be seen that there is no measurable free amine before all the acid groups are neutralized. For all the systems, with the exception of the model

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<sup>\*</sup>Agern Allé 3, 2970 Hørsholm, Copenhagen, Denmark. †Copenhagen, Denmark.

Systems	Amine Added (%)	pН	mol base mol acid	Free Amine (%)
20% Acrylic emulsion	1.7	7.6	0.76	< 0.05
+ TEA	2.7	9.2	1.20	~0.05
	3.7	10.7	1.64	0.75
20% Acrylic emulsion	0.95	7.5	0.58	< 0.05
+ DEA	1.95	10.1	1.20	0.07
	2.95	11.2	1.81	1.07
25% Shellac dispersion	3.0	8.1	1.08	< 0.05
+ TEA	4.0	10.3	1.43	0.36
	5.0	10.7	1.79	0.97
30% Bevalkyd + TEA	2.4	6.9	0.96	< 0.05
	3.4	9.6	1.36	0.14
	4.4	10.5	1.75	0.67
30 Soalkyd + TEA	1.8	7.9	1.01	< 0.05
	2.8	10.2	1.58	0.18
	3.8	10.5	2.14	0.67
20% Isostearic acid	7.0	9.3	0.98	< 0.05
dispersion + TEA	8.7	10.4	1.23	0.36
	10.6	10.5	1.46	0.82

system with isostearic acid, there is no measurable free amine before about 120% theoretical neutralization. This can be interpreted as a distribution of the amine between the polymer and water phases in a manner similar to that of solvents.<sup>2-4</sup> We feel this distribution affects the evaporation of both solvent and amine from practical coatings systems and, thus, explains in general terms why the solvent to water ratio is higher for evaporation from systems where polymer is present than where it is not, i.e., mixtures of solvent and water only.<sup>5</sup> A project to study these phenomena in greater detail is in progress at this institute.

Figure 1 shows plotted data for some of the systems reported as well as the method used to calculate the free amine content of a 20% acrylic emulsion with TEA as 0.75% where 3.7% total amine was added.

The role of pigment was found negligible for the pigments studied,<sup>1</sup> with the exception of lead chromate,

Table 2—Evaporation of Water and TEA From an Acrylic Emulsion

Time s.	% Sample Remaining	TEA g	Acrylic g	Waterg
0	100.0	5.7	20	74.3
120	99.4	5.3	20	74.1
240		5.0	20	73.9
480	97.6	5.0	20	72.6
960		5.0	20	71.0
1920	91.7	5.4	20	66.3
3840	88.1	4.6	20	63.5
7680	77.9	2.9	20	55.0
15180	53.0	2.8	20	30.2
28200	27.2	1.6	20	5.6
56700	21.4	1.2	20	0.2
86400	21.4	1.2	20	0.2

which reduced the free amine content presumably because of high surface activity. The pigments in addition to lead chromate were Lachrottoner®LCLL from Hoechst (not alkali resistant), Litholrubin®4581 D from BASF (alkali resistant), and Heliogen Blau®7081 D from BASF (alkali resistant). (See *Figure 2*.)

The following can be concluded from these studies:

(1) Free amine cannot be measured until all acid groups are neutralized.

(2) Pigments do not affect the free amine content with the possible exception of reducing it by selective adsorption.

(3) More extensive data on the systems studied showed that none of them had free amine at pH less than 8.5.<sup>1</sup> pH is dependent on base strength so neutralization should be based on acid content rather than pH, however.

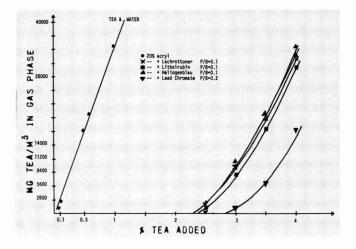
#### **EVAPORATION**

The evaporation of amines from water-reducible coatings differs from that of evaporation of water and solvent from such coatings. The amines form a water soluble salt

Britano P. 11200 

% AMINE ADDED

Figure 1—"Free amine" in water-reducible systems. Curves: (1) TEA in water; (2) DEA in water; (3) 20% ACRYLIC + DEA; (4) 20% ACRYLIC + TEA; (5) 30% BEVALKYD + TEA



#### Figure 2—"Free amine" in a TEA/acrylic emulsion system with additions of various pigments

(new material) having a much higher solubility parameter than either a low molecular weight acid or amine.<sup>6</sup> This means the bond between the polymer and amine is stronger that that between water or solvent and the polymer. This in turn results in a considerable (extra) energy barrier to evaporation of the amine from these systems. It appears that evaporation can even be stopped in some cases.

Amine evaporation was studied by weighing "coatings" in Petri dishes with analysis by gas chromatography or by the Kjeldahl method after suitable time intervals. The experiments were carried out in a hood in a climatized room  $(23^{\circ} \pm 1^{\circ}C, 50\% \pm 4\% \text{ RH})$ . Figure 3 shows the evaporation process from an acrylic system with TEA, DEA, and ammonia.

It can be seen that the amine evaporates when there is an excess over the mole base/mole acid ratio of 1, and that the final amount at apparent equilibrium is somewhat lower than this; however, it is still significant.

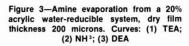
The presence of acid groups is not enough to retard totally the amine evaporation. The increase of viscosity accompanying film formation and significant diffusional resistance coupled with the energy of salt formation provide a total resistance, which for practical purposes stops further amine evaporation.

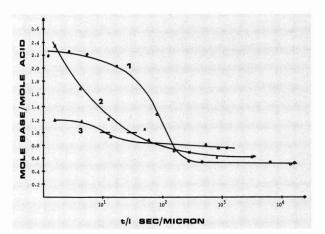
The three amines are not retained to the same extent, which may be explained by different base strengths and different molecular sizes. Further data is needed to clarify the situation, however.

TEA is the strongest base (pKa = 10.8) but is a larger molecule than DEA (pKa = 10.5). Larger molecules can be presumed to give greater steric hindrance to salt formation and this in turn weakens the strength of the bond. This would allow easier cleavage than might be otherwise expected from base strength alone.

Ammonia (pKa = 9.2) is such a small molecule that there is no significant steric hindrance, thus explaining the higher level of retention compared with TEA.

If one compares the evaporation rate of water ( $\approx 35$ ) and amine, it can be seen that, even with a large excess of amine, there is a significant retardation of the evaporation of the amine. Data are included in *Table 2* to show this effect for evaporation from an acrylic emulsion.<sup>1</sup>





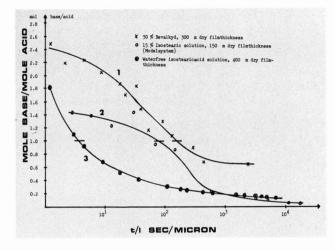


Figure 4—TEA evaporation from waterreducible alkyd and isostearic acid. Curves: (1) 30% Bevalkyd, L = 300 microns; (2) 15% isostearic acid, L = 150 microns; (3) Water free isostearic acid solution, L = 400 microns

Evaporation experiments with film thickness 4000 and 100  $\mu$ , respectively, of the acrylic/TEA mixture, yielded data identical that of Curve 1, *Figure* 3 when the base/acid ratio was plotted against time/dry film thickness. This confirms that the evaporation occurs with a boundary layer resistance control.<sup>7-8</sup> This again<sup>9</sup> confirms that evaporation from very thick "films" can be used to advantage, providing enough material for analysis, for example, to study evaporation from thin films. One only need modify the time scale by the film thickness as indicated.

Figure 4 shows TEA evaporation from Bevalkyd AQ7 and isostearic acid (model) systems. The same general picture as for the acrylic system was found, but the TEA level is slightly higher. This may be because of a larger energy of salt formation and/or different diffusion relations in the drying film.

Evaporation from isostearic acid gives a different picture since evaporation does not stop as long as amine is present. Isostearic acid is a liquid and, therefore, is not a film former as are the other systems. Thus, the diffusional component of the resistance is much less significant but the salt formation factor is still very significant.

The above experiments lead to the following conclusions:

Table 3—Initial Relative Evaporation R	ates
For Amines in Isostearic Acid and Paraff	in Oil

Evaporating Species	Alone	With Isostearic Acid	With Paraffin Oil
n-butylacetate	100 <sup>a</sup>	100 <sup>b</sup>	100 <sup>c</sup>
TEA	476	366	353
diethylamine	1810	707	882
dimethyl ethanol amine	21.4	12.2	35.3

Absolute rates: (a) 0.42 mg/min cm<sup>2</sup>

(b) 0.41 mg/min cm<sup>2</sup>

(c) 0.51 mg/min cm<sup>2</sup>

(1) Amine evaporation is strongly retarded by the presence of acid groups.

(2) Amine evaporation does not cease at a 1/1 mole ratio of base to acid, but slows markedly. For practical initial concentrations of amine (100-110% theoretical neutralization), the greater part of the amine is retained in the film.

(3) It can be estimated that this amine remains for a long time in the final film. The acrylic/TEA experiment yielded the same amine content after 1 day and 36 days.

Comparisons have also been made of the evaporation of pure amines, amines from isostearic acid, and amines from paraffin oil.<sup>10</sup> The initial relative evaporation rates (RER) were not too different for these three cases for TEA, DEA, and dimethylethanol amine, as can be seen in *Table* 3.

Relative evaporation rates evaluated over a few minutes show essentially the same general result. Retention evaluated at longer times shows clearly that the presence of acid groups causes retention of the amines for months in isostearic acid, while it evaporates in a matter of hours from paraffin oil.

These studies were carried out at room temperature. At higher temperatures evaporation of the amine is more complete.<sup>10</sup> This may be because of lower salt stability as well as reduced diffusional resistance.

#### CONCLUSION

The results of this room temperature study show that amines in water-based coatings are less of an environmental problem than might otherwise be expected from their bulk concentrations and physical properties as pure compounds.

Practical neutralization levels should not lead to significant levels of free amine. The amines studied here are among the most volatile. Less volatile amines should be retained to a greater extent with slower evaporation in the early stages of film formation.

The retarded and limited evaporation of amine means that the amine concentration in the air over the film is less than might be expected based on total amine concentra-

#### AMINES IN WATER-DILUTABLE COATINGS

tion in the coating. All volatile components contribute to the collective threshold limit value and, even though the amine concentration is low, its contribution must be added to that from other sources to evaluate whether or not established limits are exceeded.

#### ACKNOWLEDGMENT

The authors thank Karen Eng for considerable assistance in carrying out this report.

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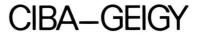
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### Society Meetings

#### Baltimore

Dr. Hugh Smith, of Sun Chemical Corp., spoke on "ENVIRONMENTAL UP-DATE."

Dr. Smith focused on the methods by which a pigment company can keep informed of the current environmental regulations. These regulations are evidenced in the increased surveillance by government agencies, environmental groups, and media; in the increased nonproductive expenditures which are environmentally related; and in the demands upon the industry for industrial hygiene and toxicological studies on its raw materials, products, work force, waste, etc.

To counter these demands, Dr. Smith suggested the need for leadership commitment, analytical expertise, trade association involvement, understanding government agencies, and quick response time to new demands.

Included in the discussion concerning current areas of pigment activity were several programs, such as the National Cancer Institute Bioassay Program and the National Center for Toxicological Research Program. In the future confrontation between environmentalists and pigment companies, Dr. Smith predicted lengthy court battles, inflationary and heavy non-productive company operating costs, and increased environmentalist action. With greater enforcement of regulations, he concluded, work situations will become healthier and safer.

MITCHELL DUDNIKOV, Secretary

#### C-D-I-C

Oct. 8

Dr. Raymond Myers, Director of PRI, Spoke on "CURRENT TRENDS IN THE COATINGS INDUSTRY."

Dr. Myers said that we are in the midst of a revolution in coatings technology. Where once there was a single type of coating for 30 markets, there are now six competing technologies for the same markets. The six he referred to are: conventional latex, high solids (including powder), water-reducible, UV-curable, and two-component coatings. Dr. Myers discussed how basic research in the areas of rheology, colloids, and polymers would provide a foundation for these competing technologies. He stated that the function of PRI, or any research institute, is to interface between basic research and industry.



Officers of the Baltimore Society for the year 1979-80. Left to right: Treasurer—Donald E. Keegan, of Conchemco, Inc.; President—Harry Schwartz, of Baltimore Paint & Chemical Co.; President-Elect—Gordon Allison, of McCormick Paint Works Co.; Secretary— Mitchell Dudnikov, of Harry T. Campbell Sons Co.; and Society Representative—Alex Chasan, of General Services Administration



C-D-I-C Society Executive Committee for the year 1979-80. Seated (left to right): President—Robert A. Broerman; Vice-President—William J. Frost; Secretary—Robert D. Thomas; and Treasure—Nelson W. Barnhill. Standing (left to right): Past-President— Raymond Schomaker; Past-President Lloyd J. Reindl; and FSCT Board Representative— William Mirick



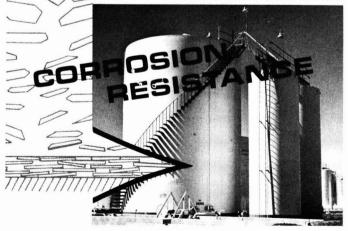
Officers of the Golden Gate Society for the year 1979-80. Left to right: Federation Board Representative—A. Gordon Rook, of Napko Corp.; Secretary—Don Mazzone, of Fuller O'Brien Corp.; President—Ted Favata, of Triangle Paint Co.; Vice-President—Sharon Vadnais, of E.T. Horn Co.; and Treasurer-Paul Christensen, of Morwear Paint Co.

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George E. Westwood, of Berger Paints Ltd., England, spoke on "THE COATINGS INDUSTRY IN ENGLAND."

In his presentation, Mr. Westwood mentioned some of the differences between the coatings industry in England and that in the United States. In the United Kingdom, the majority of automotive finishes are still nitrocellulose, 15% of their traffic coatings are paints, while 85% are thermoset coatings: and the retail market is dominated by a few major manufacturers, he said. Mr. Westwood's comments regarding the future of the industry included the areas of worldwide test methods, raw material shortages, escalating prices, energy shortages, toxicity, and regulations.

ROBERT D. THOMAS, Secretary

#### Chicago

Oct. 8

Dr. James Rieck, of Mobay Chemical Co., spoke on "THE CHEMISTRY AND APPLICATION OF URETHANE AQUEOUS DISPERSIONS," and Mr. George Erickson, a retiree of H.U.D., spoke on "H.U.D.'s PAINT ACTION PROGRAM." J. L. PETTY, Secretary

#### Detroit

Oct. 23

Gerald L. Schneberger, of General Motors Institute, spoke on "PAINT, POLARITY, AND PEOPLE.'

JOHN J. GENTILIA, Secretary

#### Kansas City

Oct. 11

President Bill Smith announced that the Society has presented \$500 to PRI.

John Bax, of Pacific Scott Bader, Inc., spoke on "NEW CONCEPTS IN THE FORMULATION OF GLOSS LATEX COAT-INGS."

To meet new government and industry performance standards with latex paints, problems arise in trying to get improved gloss, flow, and leveling together with lower glycol levels, said Mr. Bax. These problems can be solved by the use of alkali soluble acrylic (ASA) polymers in combination with the latex, he related. The ASA encapsulates the pigment and promotes stability against flocculation on drying which enhances gloss, flow, opacity, and color development. Mr. Bax explained that results of recent work on ASA's and their application in both trade sales and industrial finishes will be shown together with electron micrograph work on the distribution of pigment particles within the paint film.

RICHARD WARREN, Secretary

Dr. Robert Washburne spoke on "RECENT UPDATES IN LATEX BASED ALL-SURFACE PRIMERS."

Dr. Washburne explained that latex all-surface primers have become more interesting lately due to legislation initiated in California relative to solvent emissions. This interest is rapidly spreading across the country as we are being forced away from conventional solvents. Dr. Washburne reviewed some of the properties of all-surface primers and gave an update on two emulsion binders that appear to be suitable for various applications.

Recent advances in the design of latex polymers, he related, make it possible to formulate latex primers with good performance on problem surfaces, such as bare wood, galvanized metal, rusty steel, and chalky surfaces. These primers also show great promise in the areas of nailhead rust resistance, wood staining, knothole stain resistance, and alkaline hydrolysis resistance.

Q. Do the new primer formulations meet CARB regulations?

A. Yes. All this work was done with conformance to CARB regulations as one of the primary considerations.

Q. How high can PVC be raised and still maintain adequate stain and rust resistance?

A. The work in this area is not complete. PVC's as high as 40 have exhibited good wood stain resistance, but the exposures are not complete in the corrosion resistance area. There is an indication that the higher the PVC, the lower the resistance properties.

ROMER JOHNSON, Secretary

#### Louisville

#### Sept. 19

A moment of silence was observed in memory of Herb Griffin, Forest Alexander, and Austen Robinson, who have recently died.

Society officers for 1979-1980, installed for the first time, are: President— Gary W. Gardner, of Porter Paint Co.; President-Elect—John K. Menefee, of HyKlas Paints, Inc.; Treasurer—Phil W. Harbaugh, of Reliance Universal; Secretary—Fred Newhouse, of Reynolds Metals Co.; and Society Representative—Joseph A. Bauer, of Porter Paint Co.

Jim Hoeck, Chairman of the Education Committee, reported that the following courses are being sponsored at the University of Louisville this year: "Synthetic Resins" and "Trade Sales and Industrial Paints." D.A. Holtzen, of E.I. du Pont de Nemours & Co., Inc., spoke on "IM-PROVED HIDING POWDER MEASURE-MENTS BY MODIFIED DRAWDOWN TECH-NIQUES."

Mr. Holtzen indicated that nonuniformity of paint drawdown film thickness results in considerable error when determining scattering coefficients of prime hiding pigments. Simple modification of drawdown blades routinely used in paint laboratories results in improved uniformity and reduces thickness variation by approximately 30%, he said. These redesigned blades minimize pressure, viscosity, and rate effects.

F. E. NEWHOUSE, Secretary

#### Montreal

Oct. 10

Ken Lakin, of Manchem Ltd., England, spoke on "DRIER TECH-NOLOGY."

Mr. Lakin explained that driers have a functional role in the formulation and that their role is to give the paint film a resistance to deformation upon drying. This occurs as a result of an increase in molecular size and other chemical and physical changes. The type of equipment described by Mr. Lakin to determine drying time was a B.K. recorder, which records four stages of drying time.

Using a slide presentation to illustrate his findings, Mr. Lakin discussed the



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Los Angeles Society Officers for 1979-1980. (left to right): Society Representative— Gerald L. West, of Ameritone Paint Corp.; Treasurer—Jan Van Zelm, of Byk-Mallinckrodt; President—Albert Seneker, of Ameron R & D; Secretary—Romer E. Johnson, of Dorsett & Jackson, Inc.; and Vice-President—Donald I. Jordan, of Cargili, Inc.

value of partial replacement of cobalt driers. Driers which were considered as replacements were maganese, cerium, aluminum, and zirconium. He also emphasized the importance of using only the exact amount of driers necessary.

R. H. KUHNEN, Secretary

#### Northwestern

#### Sept. 11

Al Heitkamp, of Cargill, Inc., was formally installed as Society President for 1979-1980.

Larry Culver, of Eastman Chemicals, spoke on "MISCIBILITY CHARACTER-ISTICS OF ORGANIC SOLVENT-AQUEOUS COMPOSITION."

Designed to teach fundamental concepts in this area, Mr. Culver's presentation defined such terminology as phase boundary, lower critical solution temperature, classical phase diagram, and resin neutralization. In addition, the various functions of glycol ethers and alcohols in water-borne coatings, such as solubility, coupling, viscosity control, improving flow properties, and control of evaporation rate, were discussed.

Mr. Culver introduced the classical phase diagram as a tool to describe the water-soluble characteristics of a solvent/water blend at variable temperatures. A given water/solvent ratio exhibits phase separation as the temperature rises, called lower critical solution temperature. A plot of lower critical solution temperatures obtained by varying the water/solvent ratio forms the phase boundary, he continued.

The addition of a cosolvent, or a resin or varying the neutralization of the resin will change the solubility characteristics of a water/solvent blend. Mr. Culver illustrated these influences with some examples. He concluded his presentation with a discussion of the effects of phase separation on coatings properties, including lower gloss, difficulty in dispersing pigments, craters from poor wetting properties, haze, poor stability, and erratic rheological properties.

G. DALE ERNST, Secretary

#### Pacific Northwest Sept. 20

Robert Price, of Spencer Kellogg, Div. of Textron, spoke on "CONSERVATION ORIENTED COATING SYSTEMS."

ART BRAGG, Secretary

#### Rocky Mountain Sept. 10

President F. H. Meyers, of Kwal Paints, presented John Delmonico, of Old Western Paints, and Gerry Umbreit, of Kwal Paints, with their belated 25-year pins.

Robert Price, of Spencer-Kellogg, Div. of Textron, spoke on "CONSERVA-TION ORIENTED COATINGS SYSTEMS." During his discussion, Mr. Price presented medium- and high-solids resins in coatings systems which contribute such desirable characteristics as low or no solvent pollution, energy efficiency, minimized solvent waste, and mainteance of high quality.

STEPHEN CROUSE, Secretary

#### St. Louis

#### Sept. 18

Robert Rauch, of Tioxide of Canada Ltd., discussed the rheological assessment of flow and leveling using a Haake Rotovisco. Mr. Rauch showed how this instrument was used in evaluating the impact of titanium dioxide on flow and leveling, as well as on gloss. He also discussed some parameters of titanium dioxide, such as specific surface, inorganic and organic coatings, to show their importance on flow and leveling.

JOHN KEMPER, Secretary



Executive committee of the Mexico Society for the year 1979-80. Front row (left to right): Treasurer—Oscar Fernandez; Board Member—Jorge Calvillo; President—Antonio Guevara; Secretary—Juan Irazabal. Back row (left to right); Past-President—Emilio Santillan; Federation Board Representative—Antonio Pina; Board Member—Roberto Garcia; Board Member—Enrique Angon; Board Member—David Juarez

### **Future Society Meetings**

#### Baltimore

(Jan. 17)—FEDERATION OFFICERS NIGHT

(Mar. 20)—Manufacturing Committee Seminar on "WASTE MANAGE-MENT"

(Apr. 17)—Educational Committee program

(May 15)—Technical Steering Committee program on "WATER-BORNE COATINGS"

#### Chicago

(Jan. 7)—Discussion of Society project on "INFRARED SPECTROSCOPY"— John Vandeberg, of DeSoto, Inc.; "MOTIVATION OF PRODUCTION PER-SONNEL"—J. Richard Mathieson, of Dow Chemical.

(Feb. 4)—"EMULSION POLYMERIZA-TION TECHNIQUES"—speaker from Rohm and Haas Co.; "LATEX PAINT FORMULATION USING CPVC DETER-MINATIONS"—Ken Hesler, of DeSoto, Inc.

(Mar. 3)—Society Manufacturing Committee Program; "INTERFACIAL CHEMISTRY IN CORROSION-INDUCED PAINT ADHESION LOSS"—Ray Dickie, of Ford Motor Co.

(April 7)—Society Technical Committee Program; "EVAPORATION BE-HAVIOR OF ORGANIC CO-SOLVENTS IN WATER-BORNE COATINGS''—R. F. Eaton, of Union Carbide Corp.

#### C-D-I-C

(Jan. 14)—"WHAT IS HAPPENING TO THE COATINGS INDUSTRY?"—Howard Ellerhorst, of Chemical Marketing Services, Inc.

(Mar. 10)—Society Technical Committee Program.

#### Cleveland

(Jan. 15)—"FINANCES AND PROJECT SELECTION—E. Bruce Euchner, of Glidden Coatings and Resins, Div. of SCM Corp.; and Bosses Night.

(Feb. 19)—"WATER REDUCIBLES, COATINGS FOR CORROSION PROTECTION"—Speaker to be announced.

(April 15)—Plant Tour—Davies Can Co.

(Mar. 18)—"WASTE DISPOSAL AND THE ENVIRONMENT"—Speaker to be announced.

(May 20)—"PAINTING AND DECORAT-ING FROM THE FEMALE POINT OF VIEW"—Spouses' Night.

#### Detroit

(Jan. 22—"SALT FOG TESTING"—R. Van Doern, of NL Chemicals. (Feb. 26)—"CONVENTION OF 1981"

-F.J. Borrelle and T.A. Kocis.

(Mar. 25)-To be announced.

(Apr. 22)—"DEFOAMING AGENTS"

-Arlene Reich, of Air Products.

#### Louisville

(Jan. 16)—"AQUEOUS INDUSTRIAL COATINGS"—Nick Roman, of Rohm and Haas Co.

(Feb. 20)—"FOAM-RELATED AS-PECTS OF WATER-REDUCIBLE AL-KYDS"—Richard M. Thornton, of Nalco Chemical Co.

(Mar. 19)—"PLANT SAFETY"—Gil Kane, of Hercules Incorporated.

(Apr. 16)—FSCT SLIDE PRESENTA-TION—Federation Officers.

#### Montreal

(Jan. 9)—"TIO<sub>2</sub> GRADE RATIONALI-ZATION"—Joe Fiocco, of Canadian Titanium Pigments Ltd., and Robert Rauch, of Tioxide of Canada Ltd.

(Feb. 6)—"A SAFETY PROGRAM DE-SIGNED FOR THE PAINT PLANT"— Manufacturing Committee Presentation.

(Mar. 5)—JOINT MEETING with the Quebec Paint Industries Association.

(Apr. 2)-Speaker to be announced.

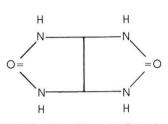
(May 7)—"GOVERNMENT SPECIFI-CATIONS IN THE PAINT INDUSTRY"—Joe Roberge, of B.N.Q., and Frank Eadie, of C.G.S.B.

#### Northwestern

(Jan. 8)— "AN EVALUATION OF WATER-BORNE, AIR DRY, FORCE DRY, LEAD AND CHROMATE FREE METAL PRIMERS"—Don Emch, of Valspar Corp.

(Feb. 5)—Federation Officers Visit. (March 4)—Annual Symposium.

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### **Technical Articles in Other Publications**

#### Compiled by the Technical Information Systems Committee-H. Skowronska, Chairman

#### **Organic Coatings and Plastics Preprints**

Papers presented before American Chemical Society Division of Organic Coatings and Plastics Chemistry, Washington, D.C. Vol. 41 No. 2 September 1979

Efforts and Problems in complying with Regulations on Polluting Coatings (11 papers)

Advances in Organometallic Polymers Vinyl polymerizations (6 papers) Phosphazenes (5 papers) Electrical conducting polymers (7 papers) Miscellaneous (7 papers)

Polymer Education (17 papers)

Binder Coatings in Textile Industries (5 papers)

New Concepts in Coatings and Plastics Chemistry Polymer synthesis and cure mechanisms (6 papers) Dynamic properties of polymers, using torsional analytical techniques (6 papers)

Transition studies above Tg and cure mechanisms (6 papers) Substrate pigment and filler interactions with polymers (5 papers) Polymer Containers (8 papers)

Stress cracking of Plastics (11 papers)

Macromolecular Secretariat — Water in Polymers (Abstracts only) Structure of water (4 abstracts) Association of water in synthetic polymers (7 abstracts) Water interactions with natural and synthetic fibers (6 abstracts) Natural and effect of water-polymer interactions (7 abstracts) Water - surface interactions (6 abstracts) Interaction of proteins with water (4 abstracts) Supplementary aspects of water - polymer interactions (8 abstracts)

Workshop on Current Trends in Surface Coatings (Papers presented at Honolulu Meeting, April 1979) (7 papers)

Note: Information regarding price and availability of this preprint volume may be obtained from Dr. M.J. Bowden, Bell Telephone Labs, 600 Mountain Ave., Murray Hill, N.J. 07974

Please consult the Union List of Series or New Series at your nearest public or college/university library for the location of listed periodicals in American libraries.



### Meetings/Education

### Symposium on Color and Appearance Instrumentation Scheduled for March 1981, in Louisville

A symposium on instrumentation for measuring color and appearance in the coatings industry will be held March 24-26, 1981, at the Executive West, Louisville, Ky. The 2- $\frac{1}{2}$  day meeting will be sponsored jointly by the Federation of Societies for Coatings Technology, the Manufacturers Council on Color and Appearance, and the Inter-Society Color Council.

The program will be divided between "hands-on" workshop sessions and general oral presentations about gloss and appearance, color differences and quality control, spectrophotometry, and colorant formulation. The format of the program is being designed to offer a "working meeting" environment, and registrants will be invited to bring samples with them.

General program sessions are under the direction of Dr. Robert T. Marcus, of PPG Industries, Inc., who chairs the Federation's Inter-Society Color Council Committee. Arrangements for the workshops and instrument displays are being handled by Charles Leete, Executive Director of the Manufac-

#### Pigment Dispersion Is Topic Of New England Seminar

A Technology Review Seminar on "Pigment Dispersion" will be sponsored by the New England Society for Coatings Technology on January 17 in Cambridge, Mass.

The half-day meeting, geared for those persons interested or involved in the theory and current practice of pigment dispersion, will feature the following presentations:

"Pigment Dispersion"—J.D. Easton, of Harshaw Chemical Co.

"Novel Oligomeric Polyelectrolytes"-Stephen Cantor, of Uniroyal, Inc.

"Pigments"—Al Singleton, of American Hoechst Corp.

"Sedimentation Analysis—An Early Determination of the Formation of Settlement in Coating Systems"—Frank Zurlo, of Byk Mallinckrodt.

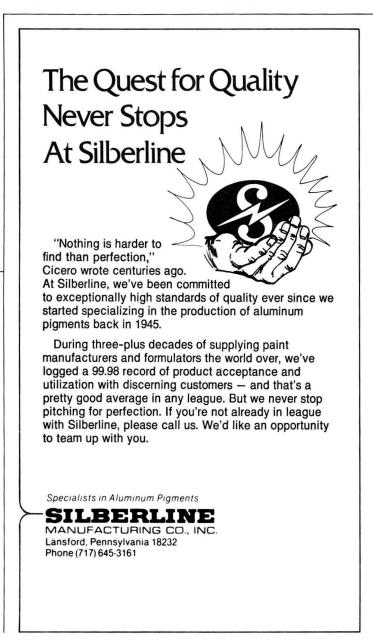
"High Speed Dispersers"-Gregory DeLong, of Shar, Inc.

"Media Grinding"—Jan Grodzinski, of Torco.

For additional information, or to register, contact John Fitzwater, Polyvinyl Chemical Industries, 730 Main St., Wilmington, Mass. 01887, or call (617) 658-6600. turers Council on Color and Appearance.

Complete details will be available shortly. Meanwhile, further information

may be obtained by contacting the Federation of Societies for Coatings Technology, 1315 Walnut St., Suite 832, Philadelphia, Pa. 19107.



### People

McCloskey Varnish Co. has appointed **Ronald Johnson** as Western Regional Sales Manager of their Industrial Div. In his new position, Mr. Johnson will assume responsibility for all of the company's West Coast marketing and sales operations. He is a member of the Los Angeles Society.

Samuel A. Wilson, Jr. has been appointed to the position of Product Manager, Chemicals, for the Petrochemical Group, Union Chemicals Div., Union Oil Co. of Calif. A member of the Southern Society, he will be headquartered in the company's Schaumburg, Ill. offices.

Rohm and Haas Co., Philadelphia, Pa., has appointed Sharon A. Ellis as Market Development Manager for its Polymers, Resins, & Monomers, North American business team. In her new assignment, Mrs. Ellis is responsible for managing market development programs in coatings, textile, paper, leather, and related areas.

George S. Weems has been named General Manager of the Organic Chemical Div. of the Richardson Co. Mr. Weems formerly served as Director of Business Development for the firm.

The American Hoechst Corp., Somerville, N.J., has elected **Dieter zur Loye** to the newly created position of Chief Operating Officer. He will also act as Senior Executive Vice-President and Deputy to the President.

In addition, the company has elected two new Directors to the Board: Harry R. Benz—Group Vice-President and Chief Financial Officer; and Dr. Hans F. Holzapfel—Group Vice-President, Law. Elected Corporate Vice-Presidents were: Harold J. Cavanaugh—Corporate Personnel; William L. Grabowski—Material Management; Emory Marton—General Counsel; and Raymond Smedley— Controller.

H. Claus Hamann, Vice-Chairman and Senior Executive Vice-President of the firm, will become Senior Advisor upon his retirement in January 1980.

Meanwhile, the company has announced the appointment of three divisional Vice-Presidents; Michael W. Nordhoff-Office of the President; Karlis V. Rutins-Information Systems; and Klaus Wieschenberg-Corporate Financial Planning.





M. Jay Kolaya has been named Com-

mercial Development Manager, Poly-

ester, by Interstab Chemicals, Inc., New

Brunswick, N.J. His new duties will in-

clude all aspects of product development

from laboratory to finished commercial

product with promotional and technical

service follow-up. Mr. Kolava is a mem-

Thomas C. Page has been appointed

Vice-President of Ford Motor Co., Di-

versified Products Operations. He suc-

ceeds Louis R. Ross. who has been

named Executive Vice-President of Car

Product Development for Ford North

Following a recent ownership change,

Cook Paint and Varnish Co., Kansas

City, has announced the restructuring of

its management organization. D. Patrick

Curran will serve as Chairman of the

Board, as well as President and Chief

Executive Officer of the firm and John S.

Ayres, President since 1960, will serve as

In addition, James Florence, as Group

Vice-President and Treasurer, will be

responsible for the Resources Group,

which is composed of the Finance Div.,

the Purchasing Div., and the Material

Management Div. Walt Belczak will be

responsible for the Marketing and

Research Group, including the Indus-

trial and National Accounts Sales Div.,

the Trade Sales Div., and the Research

Hercules Incorporated, Wilmington,

Del., has appointed Robert E. Boddorff

to the position of Product Director for

water-soluble polymers and coatings,

marketing. John L. Lawes will assume

Mr. Boddorff's prior responsibilities as

Director of Strategy, Cellulosics, World-

wide Water Soluble Polymers Business

and Quality Control Div.

Center.

Vice-President.

American Automotive Operations.

ber of the New York Society.





M. J. Kolaya

H. Vogl

Harry Vogl has been named Operations Manager for Bee Chemical Co.'s Western Chemical Div. In his new position, he will be responsible for all operations at the Gardena, Calif. plant. Mr. Vogl is a member of the Los Angeles Society.

Kazys Sekmakas has been promoted to the newly created position of Staff Scientist for DeSoto, Inc., Des Plaines, Ill. A member of the Chicago Society, Mr. Sekmakas will occupy the highest technical position at the firm.

Also announced by DeSoto, Inc. are the following promotions: Dennis A. Centofante—Manager of Operations, Chemical Coatings Div.; Terry Szesny— Elgin Plant Manager; Myron J. Bezdicek —Supervisor, Interior Sales Laboratory; Wayne E. Gyssler—Supervisor, Exterior Trade Sales Laboratory; and Raj Shah— Senior Research Chemist, Polymer Div. Messrs. Centofante, Szesny, Bezdicek, and Gyssler are active in the Chicago Society.

Meanwhile, John Krajewski and James Butler have been appointed to the positions of Manager, Polymer Development. Mr. Krajewski is also a member of the Chicago Society.

Philip R. Arvidson and Sam G. Houston have been elected Vice-Presidents of Inmont Corp., New York, N.Y. Mr. Arvidson, who will have responsibility of the firm's commercial development activities, has most recently served as Corporate Director of Planning. Mr. Houston will be responsible for business analysis and planning for Inmont.

In addition, the company has appointed **Barry B. Brodt** Manager of Environmental Liaison in the Dept. of Governmental and Environmental Affairs. In his new position, Mr. Brodt will relocate in Clifton, N.J. He is a member of the Los Angeles Society.

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Don Emch has been promoted to Technical Manager for Product Finishes in the Chemical Coatings Div. of Valspar Corp., Minneapolis, Minn. He will be responsible for the product finishes laboratories in Baltimore, Cleveland, Detroit, Fort Wayne, Chicago, Minneapolis, and Kansas City. Mr. Emch is active in the Northwestern Society.

Witco Chemical Corp., New York, N.Y. has appointed Charles A. Polacki —Group Vice-President, Chemicals; Lester J. Gilbert—Director of Safety, Health, and Environmental Affairs; and James F. Divito—Technical Sales Representative for urethane foam systems in Ohio, W.Va., western Pa., and western N.Y. Gary W. Winegardner has been named President of the Jotun/Baltimore Copper Paint Co., which is jointly owned by Glidden Coatings & Resins, SCM Corp., and A/S Jotungruppen, of Norway.

David N. Allard has been named a Chemist in the Water-Borne Coatings Section, Research and Development Div., Rust-Oleum Corp. He will participate in on-going programs relating to rust-inhibitive, water-based coatings at the company's Evanston, Ill., technical facility.

### Obituary

Frank M. O'Dea, former Vice-President of Swing Paints, Ltd, in Montreal, died recently. Mr. O'Dea served on the Federation's Board of Directors since 1964, and was Chairman of its Trigg Award, Manufacturing, and Membership Committees. A Past-President of the Montreal Society, he served as Council Representative for nine years.



Dr. Roy W.H. Tess, Consultant for Resins and Solvent Products of Shell Chemical Co., Houston, Tex., has retired

following more than 35 years of service to the company.

Dr. Tessjoined Shell as a Chemist in 1944 after receiving the Ph.D. Degree from the University of Minnesota and became Research Supervi-



sor in 1959. In 1962 he began an overseas assignment with Shell International Chemie Maatschappij B.V. Upon returning to the United States, and following a number of important supervisory positions, he transferred to the Solvents Business Center in Houston. In 1978 he became a Consultant to Shell, the highest attainable technical position within the firm.

Dr. Tess was the recipient of the 1978 George Baugh Heckel Award at the Annual Meeting of the Federation in Chicago, and has served the Federation as a member of its Board of Directors for three years. He also served as President of the Paint Research Institute for three years and was a Trustee of the PRI.

Dr. Tess and his wife, Marjorie, will reside in California. Their address is: P.O. Box 577, Fallbrook, CA 92028.



#### **Cost-Cutting Accelerator**

A new accelerator, designed to save costs up to 50% on the peroxide iniated curing of unsaturated polyester resins, is described in literature now available. Additional information can be obtained by contacting Interstab Chemicals, Inc., 500 Jersey Ave., New Brunswick, N.J. 08903.

### **Book Review**

#### HANDBOOK OF AEROSOL TECHNOLOGY Second Edition

Author Paul A. Sanders

Published by Van Nostrand Co. 1979 (526 pages) \$27.50

Reviewed by Gus W. Leep Seymour of Sycamore, Inc. Sycamore, III.

This book is an updating of Dr. Sanders original work Principles of Aerosol Technology, published by Van Nostrand Reinhold Company in 1969. Dr. Sanders is a recognized expert in the field, having recently retired from the DuPont Company after 38 years of service. For the last 26 years, he worked in the Freon Products Laboratory. His work has resulted in several patents, plus a general advancement of knowledge in aerosol science. Dr. Sanders has divided his book into three sections: homogeneous systems and their properties; emulsions, foams, and suspensions; and miscellaneous.

As aerosol paint is a homogeneous system, only section one carries information which will be helpful to an aerosol paint chemist. However, section three provides interesting background information on the development and evolution of the ozone depletion theory.

#### Dr. Sanders makes only the most fleeting reference to aerosol paint. Therefore, this book is not a "how-to" text for aerosol paint chemists; but, rather, a book which provides background information and deals with some very important chemistry concepts which must be understood in order for aerosol paint chemists to be successful in their work. The chemist who is a neophyte in aerosol paint formulation will find this book of more significance than the experienced aerosol paint chemist. Chapters on the general concept of the homogeneous system, containers, valves and actuators, filling methods, cans, vapor pressure, solvency, and flammability provide essential background. Of particular importance is the section on solvency. Here Dr. Sanders discusses the concept of solubility parameters as it pertains to gasket swelling and dip tube elongation. In the field of aerosol paint, the solubility parameter concept is of fundamental importance in establishing a compatible resin-solvent-propellant system. To be successful, an aerosol paint chemist must fully understand and use this concept.

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Laboratory, Inc., Div. of Pacific Scien-

tific Co., P.O. Box 5728, 5521 Landy

Lane, Bethesda, Md. 20014.

This reviewer was surprised that fluorocarbon propellants were given such prominence in this second edition, in view of the fact that Federal Regulations have severely restricted their use. Only in very few products can fluorocarbons now be used.

Nevertheless, although about half the book is not relevant to spray paint formulation, this book constitutes a good basic text which can be used effectively by aerosol paint chemists, particularly those just entering the field.

#### Glossmeter

A new hand-held, one-piece glossmeter designed for measurement of specular reflectance of non-metallic surfaces of paints, plastics, and other materials is described in recently released literature. For additional information, contact Hunterlab, Hunter Associates Laboratory, 9529 Lee Highway, Fairfax, Va. 22031.

#### Differential Scanning Calorimetry

Publication of a 44-page book, "Differential Scanning Calorimetry," written by J.L. McNaughton and C.T. Mortimer, has been announced. A concise document prepared for the practicing thermal analyst, this book details the operation of the differential scanning calorimetry thermal analysis instrument and explores many applications, including specific heats, purity determinations, phase diagrams, and heats of chemical reactions. For a free copy, ask for Order No. L-604, and write the Perkin-Elmer Corp., Instrument Div., Main Ave., Mail Station 12, Norwalk, Conn. 06856.

#### **Color Correction Program**

A new generation computer program for correcting off-shade batches in process is described in recently released literature. Features of the program include automatic incorporation of waste batches in new correction formulas, the ability to generate chromaticity plots after each adjustment, and a self-teaching file which provides explanations and operator prompting at any point in the program. For information, contact Applied Color Systems, P.O. Box 5800, Princeton, N.J. 08540.

#### Channel Oxygen Recorder Series

A six-page brochure which describes a new one/two channel oxygen recorder series has been issued. The brochure discusses product features and customer benefits; sensor elements; and sensor assembly types available for specific application requirements. For a free copy of Bulletin 4176, contact Beckman Instruments, Inc., Process Instruments Div., 2500 Harbor Blvd., P.O. Box 3100, Fullerton, Calif. 92634.

### **Coming Events**

#### FEDERATION MEETINGS

#### 1980

(May 15-17)—Federation Spring Meetings. Society Officers—15th; Executive Committee—16th; Board of Directors— 16th and 17th. North Star Inn, Minneapolis, Minn. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, Pa. 19107).

(Oct. 28)—Federation Board of Directors Meeting. Atlanta Hilton Hotel, Atlanta, Ga. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, Pa. 19107).

(Oct. 29-31)—58th Annual Meeting and 45th Paint Industries' Show. Atlanta Civic Center, Atlanta, Ga. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, Pa. 19107).

#### 1981

(Mar. 24-26)—"Symposium on Color and Appearance Instrumentation." Jointly sponsored by Federation of Societies for Coatings Technology, Manufacturers Council on Color and Appearance, and Inter-Society Color Council. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, Pa. 19107).

#### SPECIAL SOCIETY MEETINGS

#### 1980

(Jan. 17)—New England Society Technology Review Seminar on "Pigment Dispersion." Cambridge, Mass. (John Fitzwater, Polyvinyl Chemical Industries, 730 Main St., Wilmington, Mass. 01887).

(Mar. 10-12)—Seventh Annual Water-Borne and Higher-Solids Coatings Symposium. Hyatt Regency Hotel, New Orleans, La. Sponsored by Southern Society and University of Southern Mississippi. (Dr. George Bufkin, Dept. of Polymer Science, University of Southern Mississippi, Southern Station, Box 276, Hattiesburg, Miss. 39401).

(Mar. 12-14)—Southern Society. 44th Annual Meeting and Convention. Terrace Garden Inn, Atlanta, Ga.

(Mar. 25-26)—Chicago Society, Sympon '80, "Additives." Fountain Blue, Des Plaines, III. (Dennis Socha, U.S. Gypsum Co., 1000 E. Northwest Highway, Des Plaines, III. 60016).

(Apr. 14-15)—Louisville Society Spring Symposium, "Compliance with Government Regulations" and "Hazardous Materials Waste Disposal." Marriott Inn, Clarksville, Ind. (M. Joyce Specht, Porter Paint Co., Corporate Office, 400 South 13th St., P.O. Box 1439, Louisville, Ky. 40201).

(April 17-19)—Dallas and Houston Societies. Southwestern Paint Convention, Hilton Inn, Dallas, Tex.

(May 1)—Detroit Society FOCUS Seminar. "Environmental Challenges to Automotive Coatings." Michigan State Management Conference Center, Troy, Mich. (G. Sastry, Chrysler Corp., Chemicals Div., 5437 W. Jefferson, Trenton, Mich. 48183).

(May 1-3)—Pacific Northwest Society Annual Symposium. Marriott Hotel, Portland, Ore.

(Oct. 7)—Cleveland Society Manufacturing Committee Symposium, "Formulation for and Utilization of Pigment Dispersion Equipment." Cleveland Engineering and Scientific Center, Cleveland, Ohio. (G. Dubey, Cambridge Coatings, Inc., 5461 Dunham Rd., Cleveland, Ohio 44137).

#### 1981

(Mar. 4-6)-Western Coatings Societies Symposium and Show. Disneyland Hotel, Anaheim, Ca.



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#### OTHER ORGANIZATIONS

(Feb. 4-6)—Inter-Society Color Council. Williamsburg Conference, Williamsburg, Va. (Dr. Fred W. Billmeyer, Secretary, ISCC, Rensselaer Polytechnic Institute, Troy, N.Y. 12181).

(Feb. 25-Apr. 11)—Paint Short Courses at University of Missouri-Rolla. Painting Contractors and Paint Inspectors—Feb. 25-29; Composition of Paints and Coatings—Mar. 3-7; Quality Control—Mar. 10-14; Fundamentals of Paint Formulation—Mar. 31-Apr. 4; Chemical Coatings Workshop—Apr. 7-11. (Norma Fleming, Continuing Education, University of Missouri-Rolla, 501 West 11th St., Rolla, Mo.).

(Mar. 3-7)—"CORROSION/80," sponsored by National Association of Corrosion Engineers. Palmer House, Chicago, III. (Membership Services Dept., NACE Headquarters, P.O. Box 986, Katy, Tex. 77450).

(Mar. 4-6)—National Association of Corrosion Engineers. Materials Performance and Corrosion Show. Palmer House, Chicago, III. (NACE, P.O. Box 986, Katy, Tex. 77450).

(Mar. 23-28)—Div. of Organic Coatings and Plastics Chemistry Symposiums and Spring Meeting of American Chemical Society, Houston, Texas. (American Chemical Society, 1155 16th St., N.W., Washington, D.C. 20036).

(Mar. 25-27)—Society of Manufacturing Engineers Workshop on Radiation Curing in the Graphic Arts. Ramada O'Hare Inn, Chicago, III. (Susan E. Buhr, Technical Division, Society of Manufacturing Engineers, One SME Drive, P.O. Box 930, Dearborn, Mi. 48128).

(April 20-22)—Inter-Society Color Council. Annual meeting. Downtown Holiday Inn, Rochester, N.Y. (Dr. Fred Billmeyer, Secretary, ISCC, Rensselaer Polytechnic Institute, Troy, N.Y. 12181).

(May 5-8)—Society of Plastics Engineers, 38th Annual Technical Conference (ANTEC). New York Hilton, N.Y. (Rod Ellis, SPE, 656 W. Putnam Ave., Greenwich, Conn. 06830).

#### **CALL FOR PAPERS**

Environmental Challenges to Automotive Coatings

5TH ANNUAL

Detroit Society for Coatings Technology

May 1, 1980 Michigan Inn – Detroit, Michigan

The conference will FOCUS on the theme "Environmental Challenges to Automotive Coatings." The following challenges are only a beginning:

- · Limits on volatile organic components.
- Hazardous raw materials handling and substitution.
- Coatings for lighter weight substrates.
- Reduction in energy needs.
- Compliance with new corrosion demands.

All papers which fall within the scope of the theme will be considered with emphasis on original research contributions. Company and product commercials are inappropriate. Those wishing to participate are urged to submit a letter of intent including a tentative title of the paper as soon as possible and an abstract of about 200 words by February 15, 1980. Please forward all communications to Dr. G.M. Sastry, Chrysler Corporation Chemical Division, 5437 West Jefferson Avenue, Trenton, Michigan 48183. Telephone (313) 671-4753.

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