

40

104°

35°

95°

30°

86°

25°

77°

20°

68°

15°

59°

10°

50°

5° C

41° F



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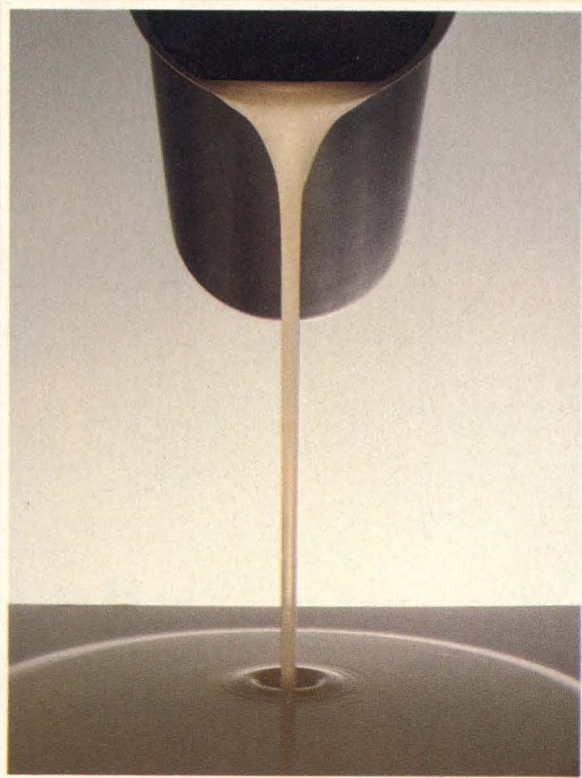
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MAY 1981



JOURNAL OF
COATINGS
TECHNOLOGY

Volume 53 Number 676

Features

- 31 DRYING OF AMINE-NEUTRALIZED POLY(ACRYLIC ACID) AND RELATED COPOLYMERS—K.S. Raju and R.R. Myers
- 40 DECEPTIVE CLOSED CUP FLASH POINTS OF SOLVENT MIXTURES CONTAINING CHLORINATED SOLVENTS—J. Rybicky and J.R. Stevens
- 45 USE OF MALEIMIDE ACCEPTORS AS A MEANS OF DEVELOPING CROSS-LINKING EMULSIONS—J.R. Grawe and B.G. Bufkin
- 57 PROCEEDINGS OF THE PAINT RESEARCH INSTITUTE NO. 1-139-1959 TO 1980

Federation Activities

- 24 1981 ANNUAL MEETING NEWS
- 26 WERNER BLANK—1981 MATTIELLO LECTURER
- 31 PROCEEDINGS OF THE PAINT RESEARCH INSTITUTE NO. 144

Departments

- | | | |
|---------------------------------|---|------------------|
| 7 Comment | 78 Society Meetings | 86 People |
| 12 Abstracts | 80 Elections | 88 Literature |
| 28 Government and Industry | 82 Technical Articles in Other Publications | 90 Book Review |
| 63 Report of ASTM Committee D-1 | 84 Meetings and Education | 91 Coming Events |

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National Paint Month

This month marks the debut of "National Paint Month," sponsored by the Consumer Products Division of the National Paint and Coatings Association.

Purpose of the campaign, entitled "Picture It Painted," is to increase the sales of paint products to the consumer.

The promotion got underway on April 6 in New York with a press conference for editors of industry, consumer, and shelter publications. Chief attraction was a large Victorian doll house filled with unique painting and decorating ideas created by students of the New York School of Interior Design.

During this Spring, paint-decorating features are running in "home" magazines such as *Woman's Day* and *Good Housekeeping*. And, displays of the colorful PIP logo are seen "all over town." You can even purchase PIP balloons, T-shirts, coffee cups, paint paddles, etc.

Trade sales paints have relinquished sales in recent years to other surface coverings. PIP aims to regain those sales by pointing out that paint is equally decorative as it is protective, and a lot less in cost than most of its competition.

We wish NPCA good luck with its sales-stimulating idea and hope it results in painting a rosier picture for the entire paint manufacturing industry.—FJB

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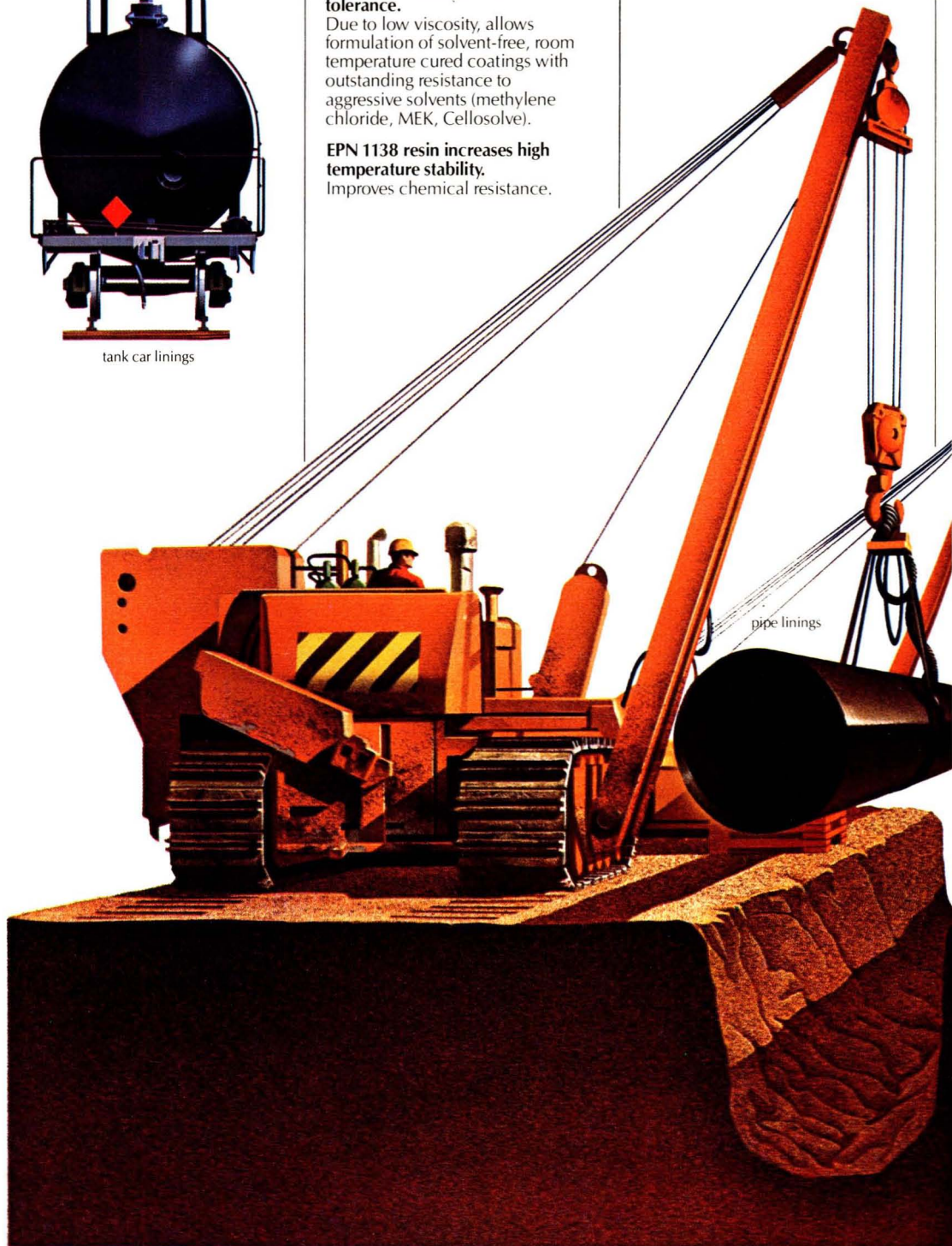
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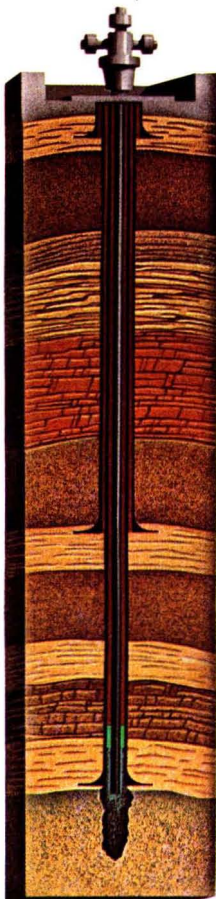
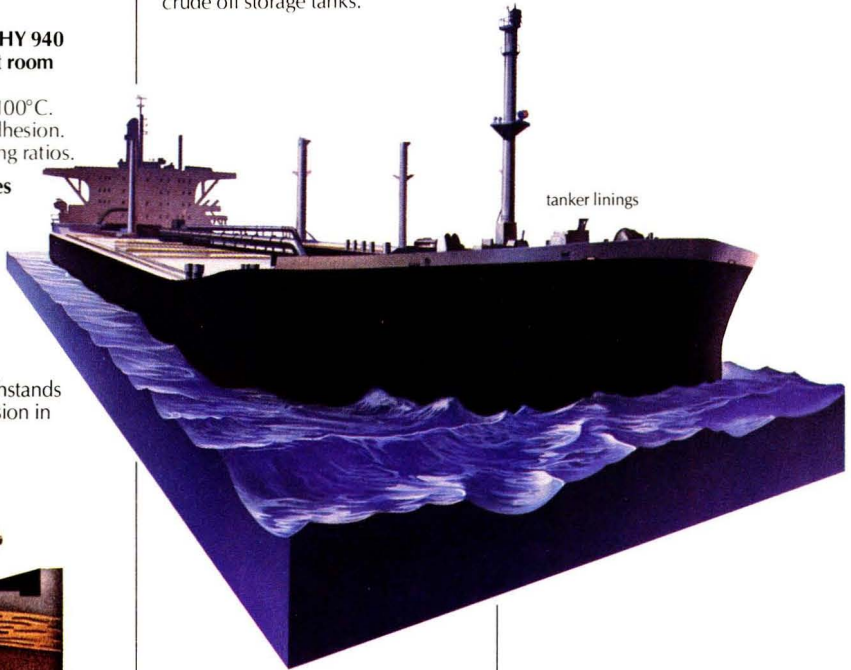
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Abstracts of Papers in This Issue

DRYING OF AMINE-NEUTRALIZED POLY(ACRYLIC ACID) AND RELATED COPOLYMERS—K.S. Raju and R.R. Myers

Journal of Coatings Technology, 53, No. 676, 31 (May 1981)

Amine-neutralized poly(acrylic acid) and acrylate copolymers were dried at various degrees of neutralization, α . Water retention by the dried film depended on α far more drastically in the case of the homopolymer than of the copolymer, supporting a hypothesis advanced earlier that water clusters around carboxylate groups that are in close proximity. Additional confirmation resulted from drying studies of maleic acid copolymer where the plasticizing effect of water clusters could not occur. This isomer of poly(acrylic acid) does not allow carboxylates to occur in clusters greater than two.

DECEPTIVE CLOSED CUP FLASH POINTS OF SOLVENT MIXTURES CONTAINING CHLORINATED SOLVENTS—J. Rybicky and J.R. Stevens

Journal of Coatings Technology, 53, No. 676, 40 (May 1981)

Mixtures of some chlorinated solvents with toluene are reported to display flash in a closed cup tester *only* within a narrow temperature range. Above this range, the flash ceases to appear. Caution is urged whenever the flammability of mixtures of chlorinated and nonchlorinated solvent is to be assessed by means of a closed cup flash point tester, the type of tester now most commonly favored. This anomalous flash point phenomenon is not encountered with an open cup flash point tester.

USE OF MALEIMIDE ACCEPTORS AS A MEANS OF DEVELOPING CROSSLINKING EMULSIONS—J.R. Grawe and B.G. Buffkin

Journal of Coatings Technology, 53, No. 676, 45 (May 1981)

The usefulness of employing maleimide-type acceptors to provide low temperature crosslinkable functionality was examined by determining the second order rate constant for the addition reaction with certain nucleophilic agents. The 3-chloromaleimido substrate was found to provide acceptable low temperature reactivity with nucleophiles while theoretically offering some degree of steric inhibition to free radical copolymerization (a trisubstituted olefin). Subsequently, to provide an acrylate-type polymer which could be post-reacted through the pendent maleimide unsaturation, 6-(3-chloromaleimido)hexyl acrylate (3-CMIHA) was synthesized using Searle's cyclodehydration method.

The model emulsions containing 3-CMIHA were prepared using a delayed, two-step addition technique whereby the total amount of 3-CMIHA was added in the last 50 weight percent of the total 60/40-ethyl acrylate (EA)/methyl methacrylate (MMA) monomer feed. However, in spite of the preventative measures used to reduce premature loss of the 3-chloromaleimido unsaturation, approximately 40% of the initial 3-chloromaleimido functionality was consumed during free radical polymerization.

Because of the effects of premature crosslinking, compositions containing incrementally higher concentrations of 3-CMIHA were found to possess network structures with greater crosslink densities and, consequently, reduced physical properties. However, compositions using 2 or 4 mole percent 3-CMIHA displayed significantly greater integrity when compared to a control which did not contain 3-CMIHA.

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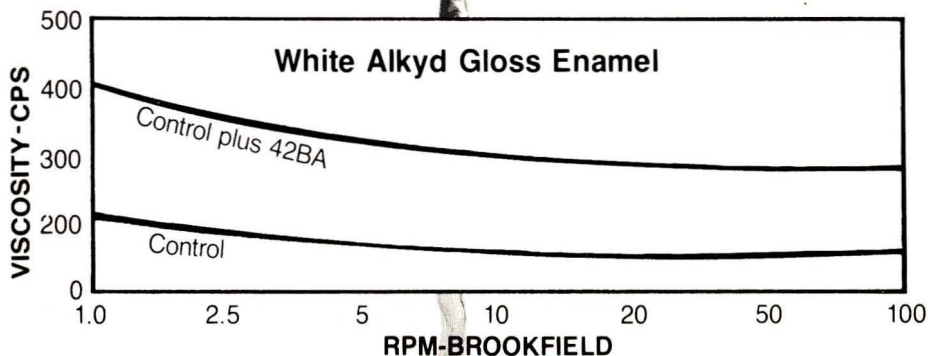
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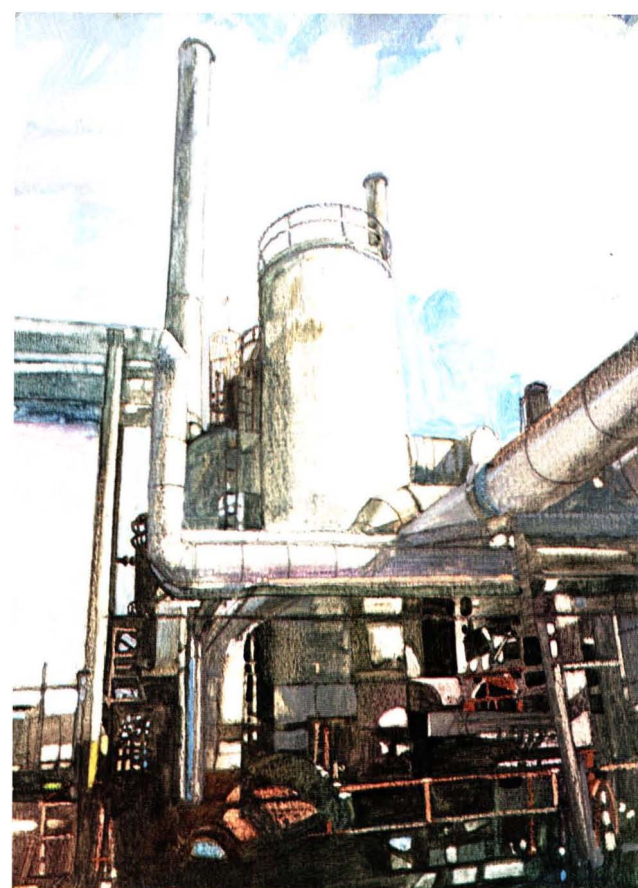
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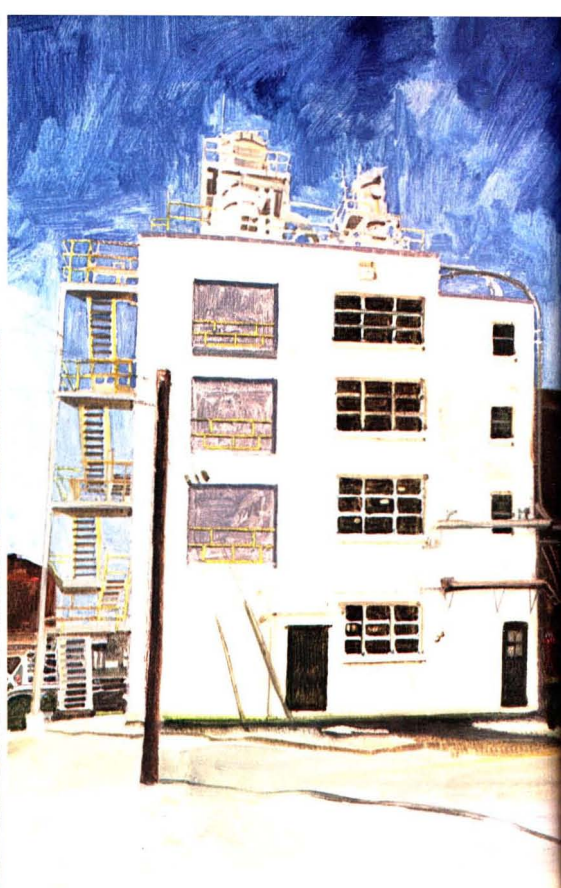
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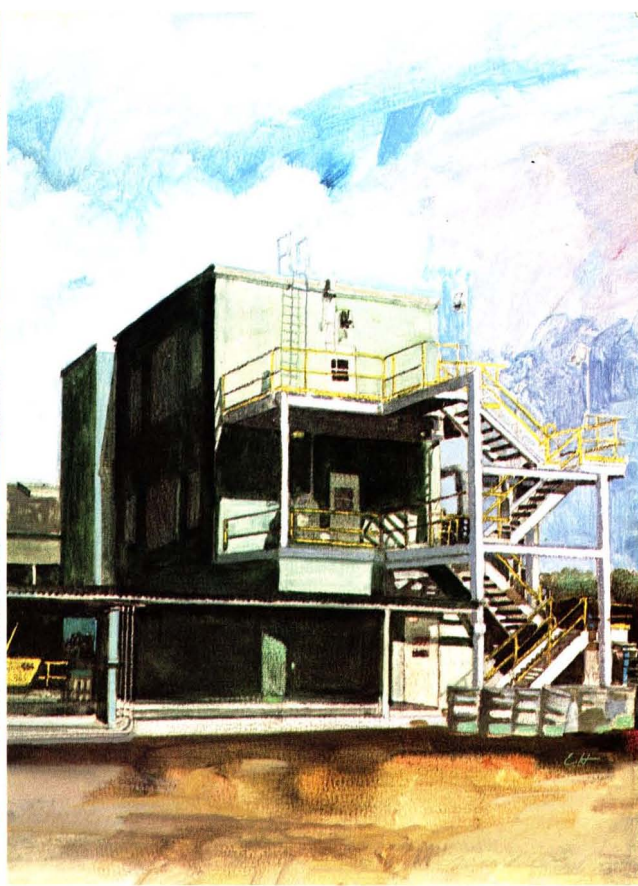
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FEDERATION OF SOCIETIES FOR COATINGS TECHNOLOGY
1981 ANNUAL MEETING AND PAINT INDUSTRIES' SHOW
COBO HALL, DETROIT, MICHIGAN
OCTOBER 28, 29, 30

THE ANNUAL MEETING

The Federation's Annual Meeting consists of three days of technical program sessions and three days of exhibits—running concurrently on Wednesday, Thursday, and Friday. These combined events were last held in Detroit in 1971.

"CHALLENGE, CHANGE, AND OPPORTUNITY"

The theme of the technical program—"Challenge, Change, and Opportunity"—reflects a positive viewpoint of the coatings industry future in the decade of the 80's. Program sessions will focus on such topics as innovations in research, meeting the changes of regulatory compliance and hazardous waste disposal, and analyzing and responding to causes of industrial and trade sales coatings failures (particularly, coatings, failures on steel and wood substrates).

PAINT INDUSTRIES' SHOW

The "Paint Show," as it is popularly known, is the only national exhibit of raw materials and equipment used in the formulation, testing, and manufacture of paints and related coatings. More than 150 well-known supplier companies to the coatings industry will participate in the Show—which will be the largest in Federation history.

Purpose of the show is to enable registrants to learn of the latest developments in raw materials and equipment and also provide the opportunity to discuss these developments with the top technical representatives of the exhibitor companies.

SPOUSES' ACTIVITIES

Included in the spouses' registration fee will be: a get-acquainted social on Wednesday afternoon; continental breakfast on Thursday and Friday; and a tour of Meadowbrook Estate, the home of Matilda Dodge Wilson, and lunch on Thursday. Spouses headquarters will be located in both the Detroit Plaza and Book Cadillac Hotels.

FEDERATION LUNCHEON

The Annual Federation Luncheon will be held on Friday. This event will feature a well-known personality as a speaker and a brief presentation of Federation Awards.

HOTELS AND RESERVATIONS

Eleven hotels in Detroit and Windsor, Canada have reserved blocks of rooms for the Federation 1981 Annual Meeting and Paint Show. The room and suite rates and a map showing the location of the hotels is included in this brochure. The Detroit Plaza and the Book Cadillac will serve as co-headquarters hotels.

All housing reservations must be processed through the Detroit Convention and Visitors Bureau using the form provided in this brochure. The Housing Bureau will send you an acknowledgment which will state the hotel to which you have been assigned. The confirmation of your reservation will come to you directly from the hotel, to whom you must direct all of your inquiries.

IMPORTANT: Reservations for the Detroit Plaza will be accepted for arrival beginning Wednesday, October 28, only. Any requests for Plaza accommodations prior to Wednesday will be assigned to another hotel.

NPCA CONVENTION

The National Paint and Coatings Association will hold its Annual Meeting also at the Detroit Plaza on Monday, Tuesday, and Wednesday, October 26-28. NPCA registration badges will be honored for admission to the Federation Annual Meeting and the Paint Show on Wednesday, October 28 (only).

PROGRAM COMMITTEE

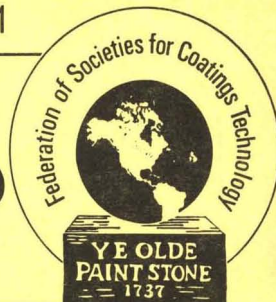
Following are the members of the Federation's 1981 Program Committee: Chairman—Thomas J. Miranda, of Whirlpool Corp., Benton Harbor, MI; Vice-Chairman—John C. Ballard, of Kurfees Coatings, Inc., Louisville, KY; Darlene Brezinski, of De-Soto, Inc., Des Plaines, IL; Peter Hiscocks, of C-I-L Paint Research Laboratory, Toronto, Ont., Canada; Percy E. Pierce, of PPG Industries, Inc., Allison Park, PA; Theodore Provder, of Glidden Coatings & Resins Div., SCM Corp., Strongsville, OH; and Walter Stuecken, of Grow Group, Inc., Troy, MI.

HOST COMMITTEE

The Detroit Society for Coatings Technology will serve as host organization. The members of the Host Committee are: Chairman—Jose Benavides, of Ford Motor Co., Mt. Clemens, MI; Bohdan Melnyk, of Chrysler Corp., Highland Park, MI; Taki Anagnostou, of Wyandotte Paint Products, Inc., Troy, MI; Jack Dentler, of Pfizer, Inc., Hoffman Estates, IL; Fred Boehle, of Boehle Chemicals, Inc., Southfield, MI; Walter Stuecken, of Grow Group, Inc., Troy, MI; Mrs. Fred F. Boehle; Mrs. Walter Stuecken; and Mrs. Jose Benavides.

FEDERATION

newsletter



EDUCATIONAL COMMITTEE PREPARES 1981 GUIDE TO COATINGS COURSES

The 1981 "Guide to Coatings Courses, Symposia, and Seminars," compiled by the Federation's Educational Committee, is now available.

Based on information supplied by the Federation's Constituent Societies, the Guide lists a variety of coatings educational offerings by geographic region and Society. The Guide is updated annually by the committee to reflect current programs, curricula, etc.

Free copies are available from the Federation office at 1315 Walnut St., Philadelphia, PA 19107.

BIRMINGHAM AND TORONTO PRODUCE NEW AUDIO/VISUAL PROGRAMS

Two new audio/visual programs have been produced by the Technical Committee of the Birmingham Club and the Manufacturing Committee of the Toronto Society:

"The Setaflash Tester"--by the Birmingham Club. The Setaflash Tester offers the capability to quickly ascertain the flash point of a volatile product. This presentation describes the equipment and explains the procedures for determining flash point in two temperature ranges (ambient to 110° C; 0° C. to ambient) by both the flash/no-flash method and the definitive method. (54 minutes running time, 134 slides, \$100.)

"Introduction to Resin Operations"--by the Toronto Society. The presentation has been developed to assist in the selection and training of resin plant operators, and focuses on basic concepts of manufacture and the role of a resin operator. (12 minutes running time, 58 slides, \$65.)

Both are available from the Federation office.

PRI ANNOUNCES AVAILABILITY OF HOWARD L. GERHART FELLOWSHIP

Chemists or chemical engineers who are doing coatings-related research at accredited universities in the U.S. are eligible for the Howard L. Gerhart Memorial Fellowship. The selected student will receive an award of \$5,000 for the applicable academic year.

To apply for the 1981-82 fellowship, a letter must be written describing the research problem and its bearing on coatings science. The letter must be signed by the student, endorsed by the professor, and sent before June 5 to: Dr. Raymond R. Myers, Research Director, Paint Research Institute, Kent State University, Kent, OH 44242.

The fellowship was established in 1978 by PPG Industries, Inc., in memory of Dr. Gerhart, who published extensively during his career as Vice-President of PPG Coatings R&D. The fellowship acknowledges the scholastic and professional achievements of Dr. Gerhart, who was a Trustee of PRI (which administers the fellowship).

NEWS BRIEFS FROM THE SOCIETIES

BALTIMORE--Because of the limited nature of the technical libraries of many companies, the Technical Steering Committee operates an "Intra-Society Coatings Library." Each participating company submits a list of any technical books and publications that they would be willing to make available for circulation. A master list is continually updated and distributed to participating organizations.....The new Virginia Section is off to a good start. Good attendance and high interest at the first three meetings.

BIRMINGHAM--Working on a new A/V program: "An Introduction to the Paint Industry."

CDIC--In the 1981 Federation Year Book, please make this correction to the Yenkin-Majestic Paint Co. listing: delete--Chester J. Wilkinson; replace with--Chester J. Stahl and George Wilkinson.

CLEVELAND--Calling for papers for the 25th Annual Technical Conference, "Advances in Coatings Technology," March 23-24, 1982, at Baldwin-Wallace College. Abstracts should be sent to: George R. Pilcher, Sherwin-Williams Co., Tech Center, 601 Canal Rd., Cleveland, OH 44113.

GOLDEN GATE--June 15 Manufacturing Conference will be devoted to "Safety and Government Regulations." Areas to be covered will be: the Workplace, Shipment of Material, and Disposal of Hazardous Waste. Speakers will be: M.J. Patterson, of SF Fire Dept.; Dr. Storm, of the Calif. Dept. of Health Services; H.M. Schneider, of Romac Corp.; J.W. Spivey, of the Dept. of Transportation; J. Aparton, of Clifton & Co.; N.S. Estrada, of Reichhold Chemicals, Inc.; and C. Erickson, of Calif. OSHA Consultants. Louie Sanguinetti is Chairman of Manufacturing Committee & Conference.

HOUSTON--Elder C. Larson, of Shell Development Co. and President of the Federation in 1979-80, was elected an Honorary Member. He is also a Past-President of both the Houston and Golden Gate Societies.

LOS ANGELES--Sponsored a Manufacturing Seminar on May 13. Subjects were: "Liquid and Solid Waste Disposal" and "Employee Motivation".....Patricia Shaw, former Chairperson of the Golden Gate Society Technical Committee, transferred to LA and will assume same responsibility with LA Society in 1981-82..... Joseph A. Cordero, of Act Container Co., has become a member of the distinguished

50-Year Membership Club and was so honored along with 25-Year members at the May meeting.....About 2,000 attended the successful Western Coatings Societies Symposium and Show held in March in Anaheim. The Show, with 67 paid exhibitors in 91 spaces, was the largest ever for the WCSSS.

LOUISVILLE--Served as gracious and cooperative hosts at the Symposium on Color Appearance and Instrumentation, held in Louisville, March 24-26.....Toured the Reliance Universal plant prior to the February meeting.

MONTREAL--Celebrated 50th Anniversary with dinner-dance on May 9. The commemorative booklet carried messages from the Premier of Quebec, Mayor of Montreal, and Federation President Bill Ellis. To further publicize the Golden Jubilee, members of the Society participated in radio programs to handle consumer queries and recommendations for paint applications and problems.

NEW ENGLAND--The April meeting in Auburn was tabbed, "the first west of Boston"... Plans to hold second "Expo" in May 1982.

NORTHWESTERN--Dr. Zeno Wicks and his 18 students at NDSU attended the April meeting.....The May meeting arranged jointly with the Winnipeg Section at NDSU.

PHILADELPHIA--April Technical Committee meeting was an "Application Workshop on New Generation Coatings," at Nordson Application Lab.....May Seminar entitled "Interfaces Revisited." Speakers: P. Becher, of Becher Assoc.; Dr. F.M. Fowkes, of Lehigh Univ.; Dr. G.D. Parfitt, of Carnegie-Mellon Univ.; Dr. F.J. Micale, of Lehigh; Dr. Harold Schonhorn, of Bell Telephone Labs.; and Dr. R.E. Baier, of Calspan Corp. Joe Lombardo is Chairman.

PIEDMONT--Special committee will compile furniture-finishing terms for inclusion in Federation's next "Paint/Coatings Dictionary".....Dr. Hurwitz to be Instructor of new coatings course at University of North Carolina, Greensboro.

SOUTHERN--255 attended annual meeting at Birmingham.....A Technical Subcommittee is working on a brochure to serve as a "Consumer Guide to Paint Quality."

ST. LOUIS--Will host annual joint meeting with Kansas City on June 6, at Lake of Ozarks. Theme is "Research and Development; the Key to Survival".....At the joint meeting, G.O. "Steve" Stephenson, former long-time Editor of the American Paint Journal, will receive the distinguished 50-Year Membership Pin.....Will have a paper for the 1982 Annual Meeting on a new method of determining the resistance properties of organic films.

TORONTO--Joining the 25-Year Membership Club are: R. Coates, J.G. Ritchie, D.C. Holland, C.H. Kaufman, C.N. Tomlenson, A. Buttivant, A. Cuyler, H. Baka, L. Prasow, B. Graham, W. Schmeisky, M. Ord, H. Zapfe, K.F. Chislett, B.V.E. Walton, and J.A. McDonald.

SEVENTEEN NEW EXHIBITOR COMPANIES TO BE SEEN AT 1981 PAINT SHOW

Seventeen new exhibitors will take part in the 1981 Paint Industries' Show at Cobo Hall in Detroit, October 28-30. They are: Capricorn Chemicals Corp.; CECOS

International, Inc.; CEM Corp.; Chemical & Engineering News; Continental Fibre Drum Co.; Dominion Colour Co. Ltd.; Eiger Engineering Ltd.; ISC Alloys Ltd.; Mearl Corp.; Mobay Chemical Corp.; Ottawa Silica Co.; Roper Plastics, Inc.; Sandoz, Inc.; SCA Chemical Services, Inc.; Sewell Plastics, Inc.; Standard Container Co.; and Waukesha Div., Abex Corp.

The increasing number of exhibitors each year is indicative of the effectiveness of the Paint Show and its reputation as one of the finest industry exhibits of its kind. This year's Show -- with 157 paid exhibitors in 36,550 net paid square feet of space -- will be the biggest.

COMING EVENTS

Coatings-Related Courses

Kent State University--"Adhesion Principles and Practice for Coatings and Polymer Scientist"(June 8-12); "Applied Rheology for Industrial Chemists" (June 15-19).

Univ. of Missouri-Rolla--"Intro to Composition of Paints and Coatings" (Aug. 10-14); "Basic Quality Control" (Aug. 17-21); "Intro to Paint Formulation" (Aug. 24-28); "Advanced Paint Formulation" (Aug. 31-Sept. 4); "New Concepts of Innovation in Coatings" (Sept. 8-9).

North Dakota State Univ.--"Basic Coatings Science" (June 1-12); "Advanced Coatings Science" (June 22-July 3).

Federation

May 14-15....Federation Spring Meetings. Society Officers on 14th; Board of Directors on 15th. Hilton Hotel, Denver.

June 5-6.....Joint meeting of St. Louis and Kansas City Societies. Lake of the Ozarks.

June 15.....Golden Gate Society Manufacturing Seminar on "Safety and Government Regulations." San Francisco.

Oct. 28-30...Federation Annual Meeting and Paint Show. Cobo Hall, Detroit.

1982

Mar. 10-12...Southern Society. Annual meeting. Savannah.

Mar. 23-24...Cleveland Society. Annual seminar on "Advances in Coatings Technology." Baldwin-Wallace College.

Apr. 22-24...Southwestern Paint Convention of Dallas and Houston Societies. Shamrock Hilton Hotel, Houston.

Apr. 29-30...Federation Spring Meetings. Society Officers on 29th; Board of Directors on 30th. Copley Plaza Hotel, Boston.

May 6-8.....Pacific Northwest Society. Annual symposium. Bayshore Inn, Vancouver, B.C.

Federation of Societies for Coatings Technology

DETROIT

HOUSING FORM

CHALLENGE

CHANGE

OPPORTUNITY

**ANNUAL MEETING
PAINT INDUSTRIES' SHOW
OCTOBER 28, 29, 30, 1981**

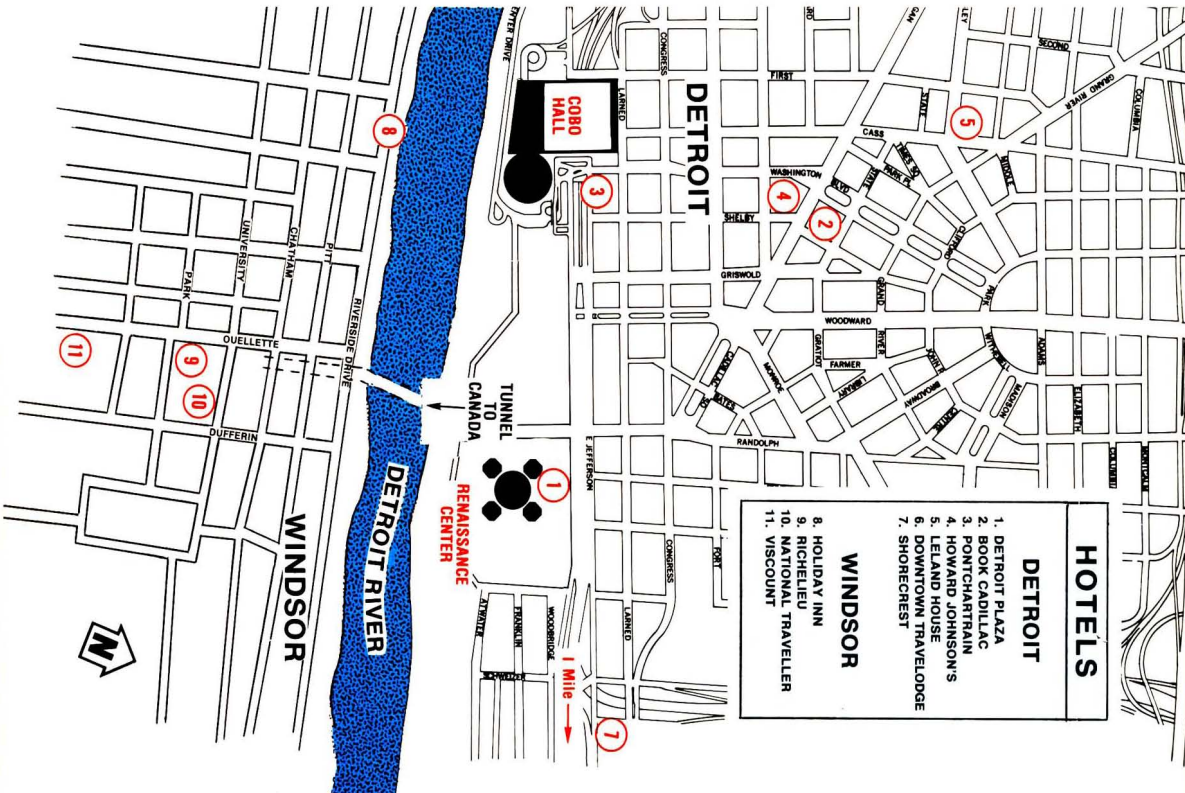
OCTOBER 26-30: COATINGS INDUSTRY WEEK IN DETROIT

HOTEL INFORMATION AND RATES

All room rates in Detroit are subject to an additional 4% charge—a Detroit tax. There is no hotel tax in Windsor.

Hotel	Singles	Doubles (D) Twins (T)	Parlor & 1 Bedroom	Parlor & 2 Bedrooms
Hotels in Detroit				
BOOK CADILLAC*	\$50	\$58 (D) 70 (T) (DD)	\$175/250	\$225/300
DETROIT PLAZA	59/69/79	71/81/91	170/190/280	240/270/365
DOWNTOWN TRAVELODGE	38	40 (D) 42 (T)	—	—
HOWARD JOHNSON'S	40	52 (DD) 50 (T)	—	—
LELAND HOUSE*	30 to 45	35 to 55 (D) 40 to 55 (T)	45 to 65	65 to 85
PONTCHARTRAIN	64/69/74/79	79/84/89/94	140 to 225	200 to 285
SHORECREST	25 to 34	27 to 38 (D) 33 to 44 (T) 50 (DD)	65 to 90	—
Hotels in Windsor, Ontario, Canada (All Rates in Canadian Dollars)				
HOLIDAY INN	49	59 (D) 63.50 (T)	—	—
NATIONAL TRAVELLER	36	41 (D)	—	—
RICHELIEU	46 to 52	52 to 60 (D)	—	—
VISCOUNT*	40/42	42/44 (D) 46/48 (T) 50 to 62 (DD)	—	—

* These hotels require a one night's room deposit.



FEDERATION OF SOCIETIES FOR COATINGS TECHNOLOGY
1981 ANNUAL MEETING AND PAINT INDUSTRIES' SHOW
COBO HALL, DETROIT, MICHIGAN
OCTOBER 28, 29, 30
(Wednesday, Thursday, Friday)

MAIL TO:	Coatings Federation Housing Bureau 100 Renaissance Center, Suite 1950 Detroit, MI 48243
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APPLICATION FOR ACCOMMODATIONS

Please indicate below the type of accommodations requested and your choice of hotels. Room reservations cannot be guaranteed unless this form is received by October 1. All reservation requests must be in writing and processed through the Housing Bureau. Phone requests will not be accepted.

TYPE OF ACCOMMODATION	NUMBER	RATE REQUESTED
Single (1 person)		
Double (2 persons)		
Twin (2 persons)		
Suite (parlor and 1 bedroom)		
Suite (parlor and 2 bedrooms)		

CHOICE OF HOTELS:
(see below)
1st
2nd
3rd
4th

Hotel assignments will be made in the order received, and in accordance with availability. The Housing Bureau will send you an acknowledgment which will state the hotel to which you have been assigned. The confirmation of your reservation will come to you directly from the hotel, to whom you must direct all of your inquiries.

Note: Reservations for the Detroit Plaza will be accepted for arrival beginning Wednesday, October 28, only. Any requests for Plaza accommodations prior to Wednesday will be assigned to another hotel.

Please fill in the names and addresses of all occupants of the rooms you have reserved. (Bracket those rooming together.) Incomplete information will delay assignment of rooms. Type additional names on reverse side of this sheet. Please give accurate arrival and departure dates.

Type of Room	Name	Address	Dates of	
			Arrival	Departure

SEND CONFIRMATION FOR ALL RESERVATIONS TO:

NAME _____

COMPANY _____

ADDRESS _____

CITY _____ STATE _____ ZIP _____

TELEPHONE _____

Please check here if there are also reservations on reverse side.

<i>(For Convention Bureau use only)</i>	
RESERVATION FOLLOW-THRU	DATE & INITIAL
Received at Housing Bureau	
Processed to Hotel/Motel	
Received at Hotel/Motel	
Confirmed to Guest	
Returned to Housing Bureau	

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years worked

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Pueblo, Colorado 81009

U.S. Department of Labor



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1981 Annual Meeting Highlights Detroit • Oct. 28-30

Program

The 59th Annual Meeting program will open Wednesday, October 28, at Cobo Hall in Detroit.

Program Chairman Thomas J. Miranda, of Whirlpool Corp., Benton Harbor, Mich., has announced the theme of "Challenge, Change, and Opportunity," and he and his Program Steering Committee are developing a schedule of presentations around this topic.

Featured presentations tentatively scheduled include:

- Keynote Address
- Mattiello Lecture by Werner Blank (see accompanying story)
- Paint Research Institute Seminar
- Manufacturing and Educational Seminars

Concurrent sessions will be held throughout the three-day meeting, but papers will be scheduled so as to avoid conflicting presentations.

Paint Industries' Show

To be held concurrently with the Annual Meeting, the 1981 Paint Show will be the largest in history. A total of 158 supplier companies have reserved booth space for displaying their products.

The Paint Show is the only national exhibit of raw materials and equipment used in the manufacture of paints and related coatings, and participating firms will have their top technical personnel on hand to discuss the latest developments.

Show hours will be 12:00 to 5:30 pm on Wednesday, October 28; 9:30 am to 5:00 pm on Thursday, October 29; and 9:30 am to 4:00 pm on Friday, October 30.

Headquarters Hotel

The Detroit Plaza will be headquarters hotel, and the Detroit Cadillac will be co-headquarters. Blocks of rooms have also been reserved at other downtown hotels convenient to Cobo Hall.

Room Reservations

All requests for rooms and suites must be on the official housing form furnished by the Federation. Housing will be processed by the Detroit Convention & Visitors Bureau, who will accept only those reservations which are on the official form.

Housing forms have been mailed to all Federation members, and a form is included in this issue (see pages 21-23). Additional forms are available from the Federation headquarters office.

NPCA Meets Same Week

The National Paint and Coatings Association will hold its Annual Meeting on October 26-28 at the Detroit Plaza Hotel.

The back-to-back scheduling of Association and Federation events provides coatings industry personnel with a full week of programming.

Registration Fees

Regular "on-site" registration fees will be \$45 for Federation members and \$60 for non-members. Advance registration will be available for \$40 for members/\$55 for non-members. Fee for spouses' activities will be \$30 on-site and \$25 in advance.

Once again there will be a special registration fee of \$20 each for retired members and their spouses.

Registration forms will be included in future issues of the JOURNAL OF COATINGS TECHNOLOGY, and will also be mailed to all members of the Federation in August.

Program Committee

Assisting Chairman Miranda on the Program Steering Committee are: John C. Ballard (Vice-Chairman), of Kurpees Coatings, Inc., Louisville, Ky.; Darlene Brezinski, of DeSoto, Inc., Des Plaines, Ill.; Percy E. Pierce, of PPG Industries, Inc., Allison Park, Pa.; Peter Hiscocks, of Canadian Industries Ltd., Toronto, Canada; Theodore Provder, of Glidden Coatings & Resins Div., SCM Corp., Strongsville, Ohio; and Walter Stuecken, of Grow Group, Inc., Troy, Mich.

Meetings Committee

Members of the Detroit Society are serving on the Meetings Committee under General Chairman Jose G. Benavides, of Ford Motor Co. Chairing the various subcommittees are: Information Services—Bohdan (Dan) Melnyk, of Chrysler Corp.; Program Operations—Taki Anagnostou, of Wyandotte Paint Products, Inc.; Publicity—Jack Dentler, of Pfizer, Inc., MPM Div.; Luncheon—Fred F. Boehle, of Boehle Chemicals, Inc.; Society President—Walter Stuecken, of Grow Group, Inc.

Co-chairing the Spouses' Activities Committee are Mrs. Fred (Rosemary) Boehle, Mrs. Walter (Lorraine) Stuecken, and Mrs. Jose (Lorraine) Benavides.

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Werner Blank, of American Cyanamid, Will Present 1981 Mattiello Lecture At Federation Annual Meeting in Detroit

The Federation of Societies for Coatings Technology is pleased to announce that Werner J. Blank, Manager of Resin Products at the Stamford (Conn.) Research Center of American Cyanamid Co., will present the 1981 Joseph J. Mattiello Memorial Lecture at the 59th Annual Meeting of the Federation, to be held October 28-30 at Cobo Hall in Detroit, Mich.

Mr. Blank will speak on "Amino Resins in High Solids Coatings," at the morning session on October 30.

Early Career

Mr. Blank, a native of Vienna, Austria, received his B.S. degree in Chemistry in Vienna, where he later attended Technical University. He left prior to graduation to pursue application, formulation, and technical service work on polyvinyl acetate homo and copolymer emulsions for trade sale applications at the Applied Research Laboratory of Farbwerke Hoechst AG, in Frankfurt, Germany. This work provided some insight into the complexity of formulating trade sale paints and into the interaction of the many additives of a commercial latex paint and the effect of the additives on application characteristics and film properties.

To further his understanding of water-borne coatings, Mr. Blank returned to Vienna to work on industrial water-soluble coatings at Peter Stoll Lackfabrik. Here he carried out development work of water-soluble spray, dip and flow coat primers and topcoats used primarily for automobile part and body applications.

Mr. Blank studied colloid and surface chemistry to gain a better understanding of the physical phenomena controlling the application characteristics of water-soluble resins, and his work at Vianova Graz (Austria) provided knowledge of the use of resin structures and how the application characteristics of water-borne paints could be controlled by specialty additives. This work led to a proprietary antifoam system for dip and flow coatings, which was commercially used in many automotive dip and flow-coat primers.

Further work resulted in the development of nonsilicone agents, adhesives, promoters, and additives to reduce solvent popping and blistering of water-reducible paints. His assignment at Stoll led to the development of anodic electrocoating primer formulations for the automotive industry.



Werner J. Blank

Research Work

After leaving Europe in 1965, Mr. Blank joined the American Cyanamid Company at its Research Laboratory in Stamford, Connecticut, where his research efforts concentrated on the development of amino resins for electrocoating applications. Through his study, he was able to demonstrate that amino resins could associate with the micellar electrocoating polymer backbone and codeposit at the same rate as the polymer due to their hydrophobic-hydrophilic balance. This research also developed a melamine and benzoguanamine crosslinker which permitted the design of nonoxiation curing electrocoating binders. This development opened areas for white and pastel colored single coat and appliance primer applications.

Mr. Blank then devoted his efforts to

the synthesis of electrocoating acrylic and epoxy primer resins. His work addressed the fractionation of polymers during the electrodeposition process and the effect of this fractionation on turnover stability. This polymer design work for electrocoating led to the design of polymers for other water-borne applications and to the development of nonionic polyether polyols for high solids water-borne applications.

Of prime interest to Mr. Blank was the design of amino resins and their structure performance relationship. Through research, he demonstrated that the cure behavior of all amino resins follows two curing mechanisms: general acid catalysis and specific acid catalysis. His work led to a new group of amino resins with reduced emission of formaldehyde and improved cure response and to new processes for amino resins. He also contributed with many others at Cyanamid to new processes for amino resins.

Mr. Blank's current position is Manager of the Resin Products Department in the Chemical Research Division of American Cyanamid Company. He is responsible for research and technical service in crosslinking agents and specialty polymers. His present research is concentrated on low temperature cure and the physical limitations in obtaining high crosslinking agents.

Author and Lecturer

Mr. Blank is author of several publications and U.S. and foreign patents on the subjects of electrocoating, water-reducible coatings, high solids systems, and crosslinking agents.

He has been a speaker for the Gordon Research Conferences, American Chemical Society meetings, and Federation meetings.

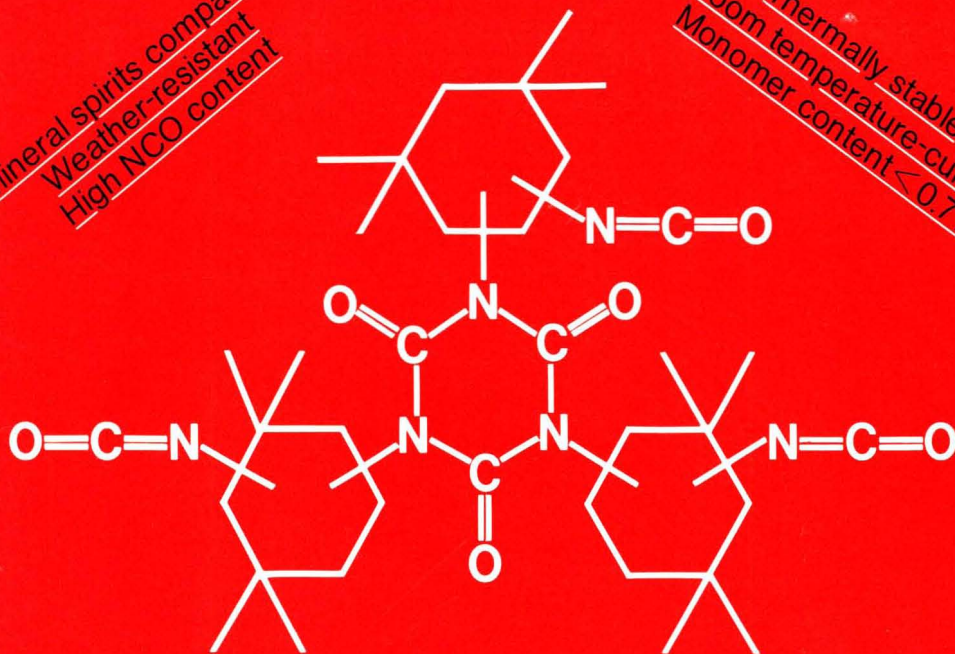
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**1981 ANNUAL MEETING
and
PAINT INDUSTRIES' SHOW**

see pages 21-23

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Government and Industry

Battelle Proposes Studying Plasma Polymerization For Use in Producing Colored Polymer Coatings

A study to help companies produce high-quality colored polymer coatings for a variety of products has been proposed by Battelle's Columbus Laboratories.

Such coatings—to be produced by plasma polymerization—could be used to protect surfaces from corrosion or chemicals, to improve adhesion, or for decoration.

The study will be conducted on a multi-faceted basis, with a number of companies sharing the costs and benefits. It will be of particular interest to companies involved with automotive products, packaging, appliance manufacturing, glass and glass products, medical instruments, and architectural products.

According to Battelle's Dr. Harry A. Beale, who will head the study team, thin, pinhole-free adherent colored polymeric coatings can be produced by plasma polymerization. With this technique—also known as glow discharge polymerization—an organic substance, or monomer, is introduced into a vacuum chamber. In the chamber it reacts under plasma conditions created by an applied electrical field—usually alternating at radio frequency—to form polymer material. Substances are placed in the pollution-free chamber, where they are then coated with the material.

Although known for more than 15 years, plasma polymerization has until now remained a laboratory curiosity and

is not widely used in industry, Dr. Beale said. Specialists at Battelle already have developed blue, gray, green, yellow, orange, and red coatings that have been applied to glass, wood, paper, metals, plastics, elastomers, and other substances.

To conduct the study, researchers initially will examine available data on the process, and then identify additional materials and concepts that might be useful in developing colored polymeric material.

This will be followed by in-depth studies aimed at determining the best

processing conditions and properties, and economic assessments of these promising concepts, Dr. Beale said.

Membership in the two-year study is open to companies for an investment of \$15,000 per year. As part of the study, sponsors will receive licenses to all patents in this field, whether resulting from the group study or from applicable research previously conducted by Battelle.

Additional information may be obtained from Dr. Harry A. Beale, Battelle's Columbus Laboratories, 505 King Ave., Columbus, OH 43201.

DOD Launches Program in Small Business Technology

The Department of Defense has established a Defense Small Business Advanced Technology Program to capitalize on the historic creative potential of small high technology firms.

The program is designed to promote innovative solutions to scientific and technical problems facing the defense community by increasing the participation of small high technology firms in the Department's research and development initiatives. Approximately 20 R & D project areas of particular interest to the Army, Navy, Air Force, and Defense Advanced Research Projects Agency (DARPA) will be identified for exploration under a three-phase program.

Phase I awards of up to \$50,000 each

are contemplated for preliminary research and development to demonstrate the feasibility of proposals deemed most likely to solve R & D problems identified by the Military Departments and DARPA. Contracts under Phase I will last for six months. Based on the results of Phase I, DoD plans to award advanced development contracts ranging from \$100,000 to \$500,000 each in Phase II for a two-year period for the projects judged most promising. Phase III will include follow-on DoD production awards, where appropriate, and/or commercial application of the research and development. Commercial application would be funded with private venture capital.

A key figure of the program is its streamlined procedure for reducing the small firm's initial investment in proposal writing. Phase I proposals are limited to 20 pages.

The Defense Small Business Advanced Technology Program is not a substitute for current unsolicited proposal mechanisms. It is designed to augment existing acquisition processes and to better inform DoD research offices of small business technological potential.

The Defense Small Business Advanced Technology Program Brochure is scheduled for distribution in April 1981, with proposals to be submitted to the respective Services and to DARPA by August 31, 1981. Awards are expected to be made in December 1981.

Program information may be obtained by writing to: Hal C. Felsner, Director, Small Business & Economic Utilization Policy, Office, Under Secretary of Defense for Research and Engineering (Acquisition Policy), Room 2A 340, Pentagon, Washington, D.C. 20301.



Kansas City Society Officers for 1980-1981. Left to right: Treasurer—Meryl Bertrand; Society Representative—Terry Johnson; President—Richard Warren; Secretary—Mike Bauer; Vice-President—Karl King

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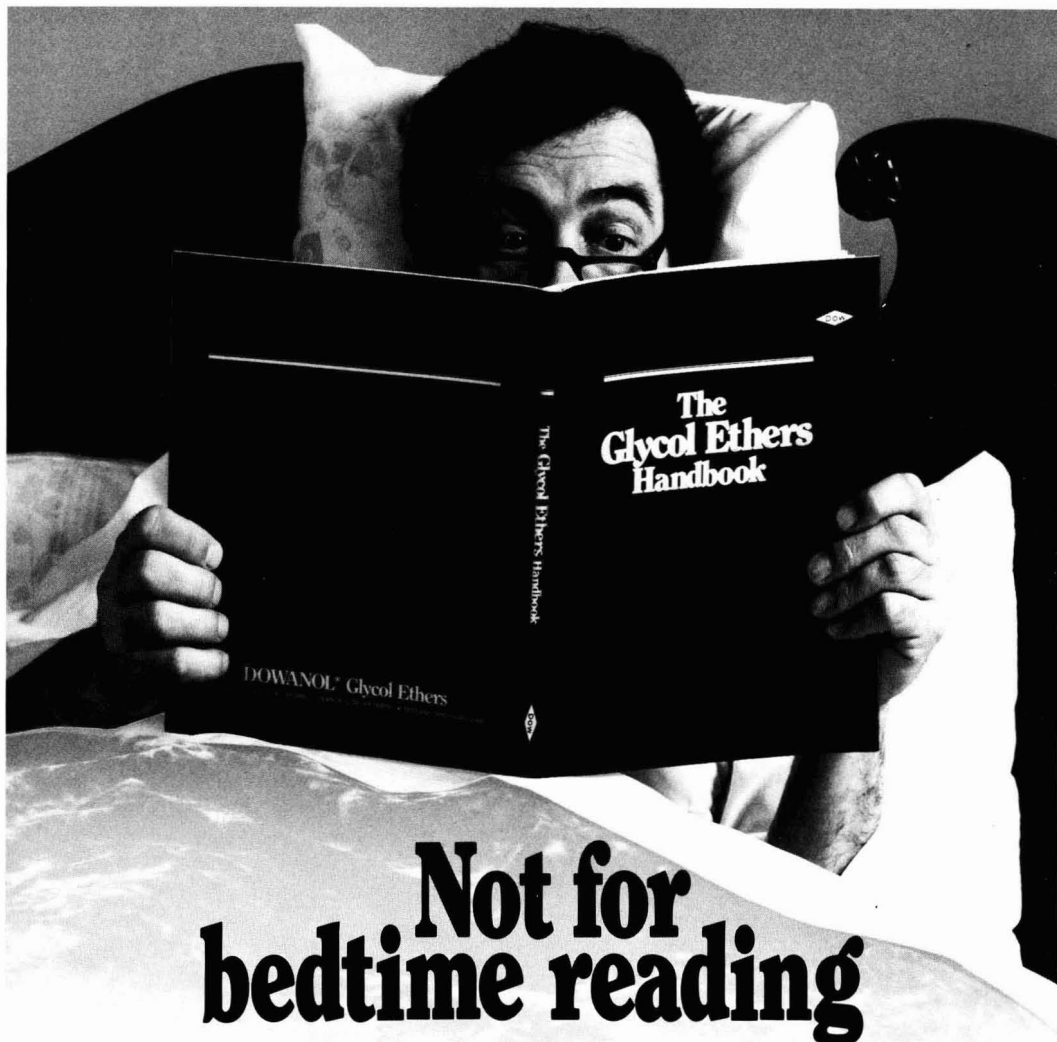
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Drying of Amine-Neutralized Poly(acrylic acid) and Related Copolymers

K.S. Raju[†] and R.R. Myers
Kent State University*

Amine-neutralized poly(acrylic acid) and acrylate copolymers were dried at various degrees of neutralization, α . Water retention by the dried film depended on α far more drastically in the case of the homopolymer than of the copolymer, supporting a hypothesis advanced earlier that water clusters around carboxylate groups that are in close proximity. Additional confirmation resulted from drying studies of maleic acid copolymer where the plasticizing effect of water clusters could not occur. This isomer of poly(acrylic acid) does not allow carboxylates to occur in clusters greater than two.

INTRODUCTION

Ion-containing polymers are among the versatile and useful materials currently under development as compliance coatings. The series of experiments (of which this is the second paper) began with a simple polymer of poly(acrylic acid) with a molecular weight of 80,000¹ neutralized to various degrees with alkali in an effort to learn how coil expansion on neutralization by a non-volatile agent under the simplest circumstances affected the film formation of the polymer cast on a substrate from aqueous solution.

Using a technique known as the attenuated reflection of shear pulses, one is able to observe the onset of rigidity

in a drying film and to use the progressive increase in rigidity beyond the gel formation stage as an indication of the rate and extent of drying. Simultaneous observations of the water lost from the drying system provided a true measure of the transition kinetics and permitted a relation to be found between rigidity and polymer content at any stage of drying.

Responses of these properties to systematic changes in degree of neutralization, α , shed insights into the conformation of the polymer. This conformation, in turn, affected the location of the gel point but not the rate of development of rigidity thereafter. Water contents of the dried films were ascertained and were related to the onset of secondary hydration** of the free carboxylate groups at α exceeding 0.5, where the groups began to act in clusters serving as nuclei for minipools of water. The effect of retained water of this magnitude was to lower the film's rigidity and to render it water sensitive.

In order to approach more practical conditions two extensions were made in the present study: the use of amines as the neutralizing agents, and the introduction of acid-bearing copolymers to the list of polymers studied.

Drying Studies

Practical applications of water-borne polymers require a knowledge of the rate of drying, of the factors that affect it, and of the various influences which one

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**See dashed line of Figure 12.

Table 1—Systems Studied

Amine	α range	Reason
Poly(acrylic acid)		
Ethanolamine	0.0-1.0	Standard neutralizer
Propanolamine	0.0-1.0	Extended homolog
2-Amino-1-propanol	0.0-1.0	Y-branched homolog
2-Amino-2-methyl-1-propanol	0.0-1.0	X-branched homolog
Benzylamine	0.0-1.0	Hydrophobic species
Diethanolamine	1.0	Progressive steric effect
Triethanolamine	1.0	Progressive steric effect
8% AA Copolymer		
Ethanolamine	1.0	Comparison with PAA; vary cosolvent
2-Amino-2-methyl-1-propanol	1.0	Comparison with PAA
Ethylene/Maleic Anhydride Copolymer		
Ethanolamine	0.0-1.0	Greatest hydration tendency
2-Amino-2-methyl-1-propanol	0.0-1.0	Least hydration tendency

Note: If E/MA copolymer were truly alternating it would be isomeric with PAA. Instead of alternating carboxyls the copolymer would have vicinal carboxyls separated by two carbon atoms.

has on the quality of the dried film. Most conventional rheological instruments are not suited to the measurement of successive changes accompanying a transition wrought by time. Instruments designed for studying coatings under conditions of drying must have one free surface, thereby compounding the difficulty. Drying time recorders measure only the time of drying and afford no insights into the structure or strength of the coating.

Attenuated reflection of pulses of parallel-polarized shear waves impinging on the film from inside the substrate had been used in successful studies of oleoresinous film drying² and was especially useful in elucidating the mechanism of latex drying³ before it was turned to the study of poly(acrylic acid) (PAA). That polymer required an ancillary measurement for rate studies in the form of weight loss measurements, and this combination was employed in the current research.

MATERIALS AND METHODS

Materials

Poly(acrylic acid), PAA, is a synthetic polymer with alternating carboxyl groups pendant on a polyethylene chain. It was obtained from Scientific Polymer Products at an \bar{M}_w of 2.5×10^5 and glass transition of 106°C . It is a polyelectrolyte, at least when neutralized. The ionic charges on the molecule affect its conformation and also the arrangements of counterions and water molecules around the functional groups.

Acrylic acid (AA)/butylmethacrylate copolymer was made as a 75% solution in t-butanol by Loren Hill, then of North Dakota State University. Its acid value was 63 (representing 8% AA). $\bar{M}_w = 3.1 \times 10^4$; $\bar{M}_n = 9.5 \times 10^3$ for a polydispersity of 2.2.

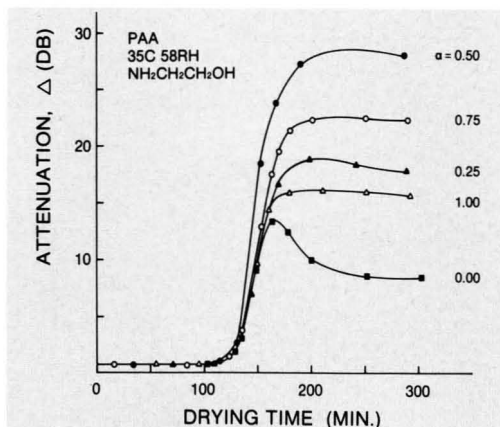


Figure 1—Drying of poly(acrylic acid) neutralized initially to various degrees by ethanolamine. Attenuation Δ in decibels DB increases after the gel point to levels determined by the hardness of the film. Peak in drying curve signifies adhesive loss resulting from residual stresses exceeding the local adhesive bond strength

Ethylene/maleic anhydride copolymer was supplied by Scientific Polymer Products at a mole ratio of ethylene to maleic anhydride of 1.4 to 1.0. Its melting point was 235°C . The acid value, 724, approximates that of PAA, 780.

All of the amines used were reagent grade and were used without purification. Their boiling points ranged from 170°C (the standard ethanolamine) to 277°C (triethanolamine).

Solution preparation was facilitated by making a 3.56 wt % unneutralized polymer (which was diluted to 2.85% with water or solvent mixture for film casting), and a 3.56% polymer to which the theoretical amount of amine was added to neutralize the polymer. This solution was diluted to 2.85% and then blended in the desired ratio with the base polymer to produce an initial neutralization ladder at intervals of $\alpha = 0.25$.

Table 1 lists the polymer/amine combinations and the range of α over which they were studied.

Methods

Development of rigidity on drying was followed by impedometry⁴ and weight loss.¹ Attenuation of shear pulses by the films cast $100\ \mu\text{m}$ (4 mil) thick on a quartz substrate was recorded as a function of time, simultaneously with the change in weight of an identical film cast on a glass slide attached to a single pan balance.

Film drying studies were conducted in a dry box maintained at a relative humidity of 58% (58RH) and a temperature of 35°C . Air flow was maintained at a slow steady rate by means of a small circulating fan.

RESULTS

Pulse attenuations, and thereby the moduli of the drying films, increased with time for the systems listed in Table 1. Typical plots of the liquid-to-solid transi-

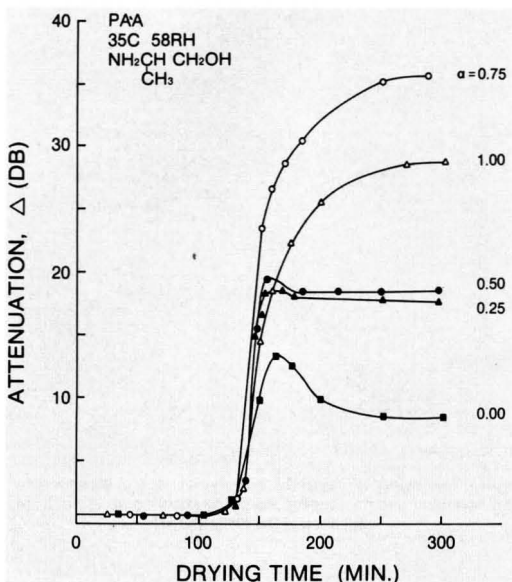


Figure 2—Drying of PAA neutralized initially by 2-amino-1-propanol

tion are given in Figures 1–5, whereas Figure 6 shows an atypical or anomalous behavior which has been shown repeatedly^{2,4,5,6} to accompany adhesive loss of the film. Moduli relate to attenuation Δ (in decibels, DB), according to the relation: $G' = \Delta^{1.73}$.

Poly(acrylic acid) Series

The amine standard (ethanolamine) in the poly(acrylic acid) system gave the drying curves of Figure 1. With α increasing gradually by $\alpha = 0.25$ increments, the family of curves displayed attenuation plateaus (Δ_{max}) whose level was highest at half neutralization ($\alpha = 0.50$). Only the unneutralized acid ($\alpha = 0.0$) showed a tendency to fail adhesively.

The conventional behavior and the decrease in Δ_{max} with α have been attributed in earlier papers^{1,8} to the balance between the uncoiling of the polymer molecule upon neutralization and the ability of the uncoiled molecule to retain water, particularly above the point of half neutralization. All other behavior types will be compared to this trend.

Figure 2 shows that if the neighboring homolog, 2-amino-1-propanol, is used as the neutralizer the moduli of the films are higher and the effect of neutralization is greatest at $\alpha = 0.75$. Continuing with the addition of pendant methyl groups the situation with 2-amino-2-methyl-1-propanol is shown in Figure 3. This time, Δ_{max} does not reach its highest level until $\alpha = 1.00$.

The trend toward higher moduli as the number of methyl groups is increased is shown in Figure 4, where the fully neutralized systems are compared. A general increase in plateau attenuation was observed, but no essential difference was noted in the rates of drying. All

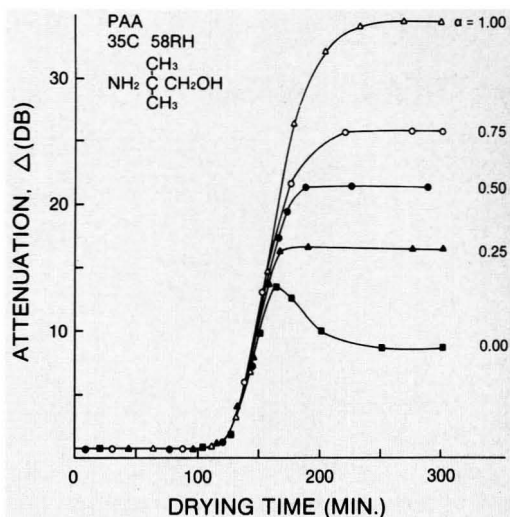


Figure 3—Drying of PAA neutralized initially by 2-amino-2-methyl-1-propanol

three curves started upward at slightly over two hours and they all followed the same pattern until the respective plateaus were approached.

In an attempt to ascertain if the methyl groups exerted a steric effect on the coiled polymer molecule, a comparison was made of three ethanolamines (mono-, di-, and tri-). By displaying branching at the functional nitrogen atom, this series should reveal the presence of a steric effect if the hydrophilic part of the molecule were held constant. Such utter simplicity was not possible in this case. Figure 5 shows that, apart from a slight increase in the drying rate on increasing the number of OH groups

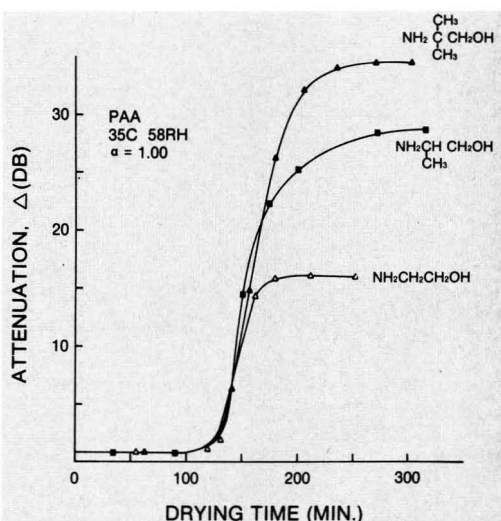


Figure 4—Homologous series trends at full initial neutralization showing less retention of plasticizing species by PAA as methyl groups are added

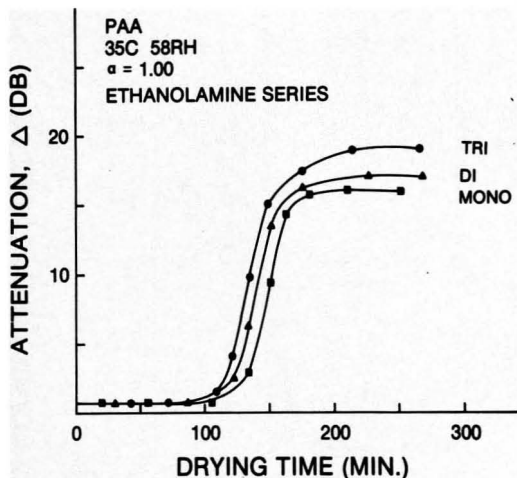


Figure 5—Drying of PAA in the presence of mono-, di-, and triethanolamine showing absence of an overriding steric effect

per molecule, there was no appreciable effect on the modulus of the dried film. All films were highly plasticized, as shown by the low levels of Δ_{max} ; either one hydroxy group plasticized as well as three, or a balance was struck between OH content and the steric effect of branching.

The drying behavior of propanolamine (3-amino-1-propanol), resembles that of ethanolamine. Here Δ_{max} reaches 28 decibels at $\alpha = 0.50$ and decreases as α is in-

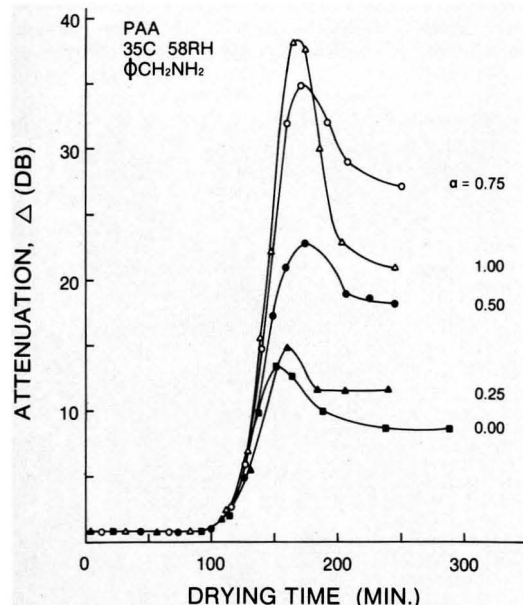


Figure 6—Drying of PAA in the presence of benzylamine showing adhesive failure at all degrees of neutralization and suggesting that water, unretained with this amine, is the plasticizing species

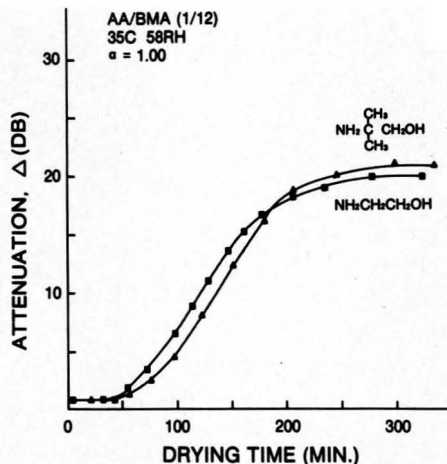


Figure 7—Drying of AA/BMA copolymer in the presence of ethanolamine and its dimethyl homolog showing no effect from water retention differences

creased thereafter. This behavior is in contrast with the hardening of PAA films by the 2-amino-1-propanol isomer (Figures 2 and 4).

Anomalous behavior upon drying resulted from the use of benzylamine as the neutralizer. Sharp peaks appeared in the curves, as shown in Figure 6, and the leveling off of Δ was significantly lower than at Δ_{max} . In turn, Δ_{max} has no quantitative significance when it peaks because its height is governed by how early in the drying process the onset of adhesive failure appeared. It is evident that this onset was determined by time and, therefore, by the water content of the film, rather than by the modulus directly; otherwise, all failures would have begun at the same level of Δ . Later considerations will show that strains after gel formation are more destructive than a high modulus.

Acrylic Acid Copolymer

Branched homologs resembled the standard in the case of drying of a 8% AA copolymer. The wider spacing of the carboxyl groups prevented ethanolamine and 2-amino-2-methol-1-propanol from displaying different behavior patterns, as shown in Figure 7. Compare this coincidence of plateau levels with the disparity of Δ_{max} in Figure 4. In both cases $\alpha = 1.00$ throughout the comparison.

The faint suggestion of a decrease in drying rate upon substituting the homolog for ethanolamine is difficult to explain. Evidently, the evaporation of water is not the only determinant of the rate of drying, for ethanolamine would be expected to retain water better than would the homolog. The reversal implicates the cosolvent.

To gain some insight into the role of the cosolvent in this regard the series of dryings shown in Figure 8 was made. In this series the t-butanol content of the solvent was varied from 20 to 40%. Drying rates increased appreciably in this interval and the levels of Δ_{max} responded slightly in the same direction.

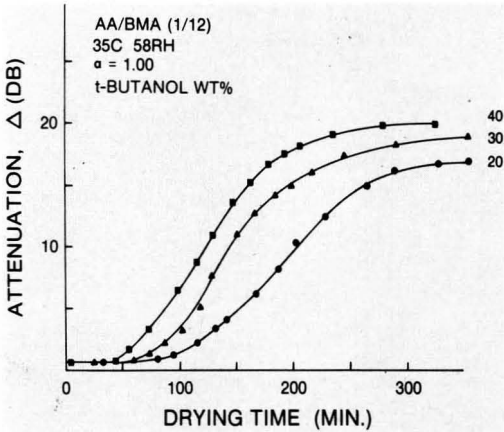


Figure 8—Drying of AA/BMA copolymer in the presence of varied percentages of t-butanol cosolvent showing that water decreases the drying rate and softens the dried film

Tertiary butanol evaporates more readily than water. This is not surprising in the light of its 83° C boiling point and lower energy of vaporization. A binary azeotrope exists at 88.3% t-butanol which boils at 80° C; therefore, the trend shown in Figure 8 should continue as t-butanol content is increased.

The composition of the dried film (or possibly its morphology) reflected the differences in cosolvent ratio as shown by annealing curves (wetting of the dried film, followed by softening and redrying). Attenuations dropped to below 5 decibels within 40 min, and when they returned to their plateau levels the Δ_{max} were positioned in the same manner as in Figure 8 and about 2 to 3 decibels lower. Had there been large residual stresses in the virgin films, the annealing would have produced larger Δ_{max} than the original.

Maleic Anhydride Copolymers

Residual stresses, as indicated by adhesive failure peaks in the curves, were especially pronounced in the case of drying copolymers of ethylene and maleic anhydride. This series was selected because two carboxyl groups appear adjacent to each other and, although they would exacerbate steric effects, there is even less chance for cations to congregate in clusters. The first publication in this series⁷ explains that clusters lead to secondary hydration above $\alpha = 0.5$; the use of maleic anhydride copolymer provides a means of preventing clusters beyond two vicinal carboxyls, and it also favors an alternating loss of protons on neutralization below $\alpha = 0.5$. The acid dissociation constant of the second carboxyl of each pair is lower than the first by at least a power of 10.

Maleic anhydride copolymers tended to be brittle, as shown by the peak in Δ at $\alpha = 0.5$ in Figure 9. There was an indication of incipient adhesive failure with the ethanolamine-containing film dried at $\alpha = 0.25$ and $\alpha = 0.50$. This effect is even more pronounced with 2-amino-2-methyl-1-propanol as shown in Figure 10. Here the high- α films are so little plasticized that they crack from

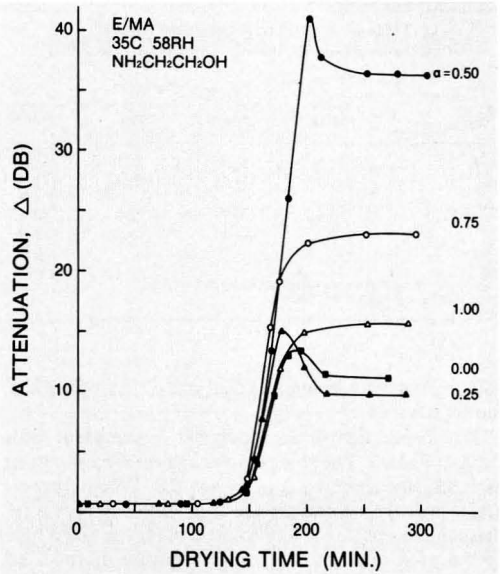


Figure 9—Drying of ethylene/maleic acid copolymer at various initial degrees of neutralization by ethanolamine showing high rigidity at half neutralization and a tendency toward film failure due to residual stresses

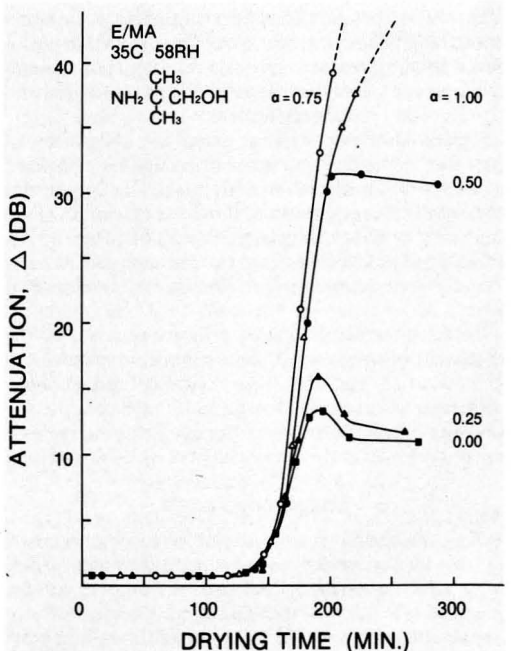


Figure 10—Drying of ethylene/maleic acid copolymer by 2-amino-2-methyl-1-propanol showing increased tendency toward film failure

Table 2—Maximum Attenuation (Δ_{\max}) Of Ethylene/Maleic Anhydride Copolymer (aq) Films

Amine	Δ_{\max} at $\alpha =$			
	0.25	0.50	0.75	1.00
EOA	16.6 (17.9)	39.5 (27.6)	22.0 (21.6)	14.6 (15.2)
2A2MIP	15.0 (16.0)	30.4 (20.4)	>40.0 (24.9)	>40.0 (33.7)

Numbers in parentheses are the corresponding values for PAA.
 $\Delta_{\max} = 12.4$ at $\alpha = 0$; 35°C ; RH 0.58

the surface during drying, and neither Δ_{\max} nor a plateau Δ can be read.

The maleic anhydride copolymer is compared with PAA in Table 2. The base polymer had the same modulus as PAA, as shown by Δ_{\max} at $\alpha = 0.0$. When ethanolamine was the neutralizer the Δ_{\max} behavior bore a remarkable resemblance to PAA except for the magnitude of the peak at $\alpha = 0.5$. This resemblance disappeared when 2-amino-2-methyl-1-propanol was employed, for Δ_{\max} attained unrecordable heights at high α . The combination of copolymer and homolog embrittled the film.

DISCUSSION

The gradual rise in Δ_{\max} and its subsidence after a particular α has been reached has been explained¹ in terms of the two competing effects of neutralizing a carboxy-containing polymer: extension of the molecule as manifested by the increase in intrinsic viscosity upon neutralizing, versus imbibition of water by the neutralized and open-coiled polymer molecule.

Figure 11 shows, without permitting calculation of $[\eta]$, that intrinsic viscosities of benzylamine-neutralized PAA respond rather drastically to α . This response is present at all concentrations; it reflects expansion of the molecule by electrostatic repulsion rather than by entrained water; and it becomes more pronounced as polymer concentrations approach the practical level studied here.

In short, the dilute solution behavior showed that the molecular dimensions of the polymer expanded on neutralization and that this expansion caused intermolecular interactions that increase on increasing the polymer concentration, as in drying. These interactions are responsible for the development of rigidity on drying.

Impedance Trends

Two major differences in drying behavior were noted: (1) not all Δ_{\max} values peaked at $\alpha = 0.5$ as was the case with alkali-neutralized PAA; and (2) random copolymers of low acid number displayed less sensitivity to amine structure than did PAA. In addition, it appears that the maleic acid copolymer, which differs from PAA in having vicinal carboxyl groups rather than alternating, is more difficult to plasticize than is PAA.

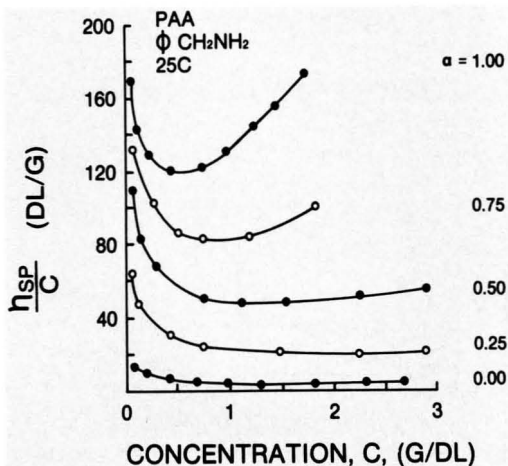


Figure 11—Inherent viscosity determinations of PAA neutralized to various initial degrees. Coil expansion indicated at high concentrations is shown to be electrostatic by concomitant increases in inherent viscosity at low concentrations

As the amine is made less hydrophilic by the insertion of methyl groups or by the removal of the hydroxy group, Δ_{\max} tends to peak at a higher α . This trend has been interpreted as a hydrophilic effect, by and large, but subsequent developments will show that the effect of water retention is more complex than plasticization of the film. When Δ_{\max} continues to increase with α above $\alpha = 0.5$ it is evident that entrained species are immobilized within the polymer folds. On a weight basis these species include more counterions than water.

Effect of Branching

Hydrophilicity differences do not fully explain the increase in modulus of PAA in the series represented by Figure 4. Despite the absence of a steric effect in the N-branched ethanolamines, it is likely that the plurality of hydroxy groups overrides any steric trends in Figure 5 and that branching is important when its effect is not swamped in this manner.

How the counterion interacts with the carboxy function on the polymer is revealed by Δ_{\max} trends. Larger, branched amine cations increase rigidity by forcing the PAA macromolecule to expand in order to accommodate them, but this effect is absent in the random copolymer.

Branching appears to exert a non-classical effect. That is, there is no steric hindrance to the formation of the cation nor to its occupying a coordination position around the carboxylate ion. The increased cross section of the branched amine caused it to lock more firmly into the polymer coils and to increase the rigidity of the molecule by converting flexible coils into rigid segments.

Indications are that an intercalation complex is formed. Intercalation is found in the interaction of drugs with nucleic acids, and it occurs in the well-known blue coloration of starch with iodine. Its effect on mechanical

Table 3—Water Retention of PAA After 220 Min

Amine	Water Retained at $\alpha =$			
	0.25	0.50	0.75	1.00
Ethanolamine				
% by weight	16.2	30.0	38.7	56.5
moles/mer	0.94	2.4	4.1	9.6
2-Amino-2-methyl-1-propanol				
% by weight	26.2 (25.4)	27.6 (27.8)	31.5 (30.0)	35.3 (33.8)
moles/mer	1.8	2.5	3.6	4.9

Water content at $\alpha = 0.0$ was 1.34% (0.62 moles/mer). Weight loss data used. Numbers in parentheses were obtained by Karl Fischer titration. Value at $\alpha = 0.0$ was 12.6%.

Table 4—Water Retention by PAA Films after 24 Hours

Amine	Water Retained at $\alpha =$			
	0.25	0.50	0.75	1.00
EOA ^a	12.9	14.7	17.3	19.9
POA ^a	11.1	12.2	15.0	16.5
2A2MIP ^b	10.2	11.6	14.5	15.1
BZA ^a	4.8	8.8	6.3	4.5
EOA ^b	0.7	0.98	1.37	1.63
POA ^b	0.6	0.84	1.26	1.61
2A2MIP ^b	0.6	0.85	1.31	1.60
BZA ^b	0.28	0.67	0.57	0.47

(a) Amount of water in weight %
(b) Amount of water in mole water/mer
Water retention at $\alpha = 0$ is 9.8% (0.44 moles/mer) (35° C RH 0.58)

properties is to rigidize the molecule, as shown by reported intrinsic viscosities of the starch iodine complex.⁹ The η_{sp}/C values in our work were limited to the range of 40 to 200 dl/g (implying considerable electrostatic repulsion of coil segments) but they were nowhere near the values reported by Katchalsky¹⁰ for poly(methacrylic acid) neutralized by alkali.

Water Content

Water retention is an ever-present malady in ion-containing polymers. Removal of water requires energy and time; it leaves the film with a porosity that may prevent it from providing a good barrier coating. There is no doubt that high- α coatings display greater water retention than do low- α coatings for reasons already published; this work suggests that one way to alleviate this situation is to use an amine of intermediate hydrophobicity.

Table 3 compares the water retention of PAA films 220 min after they were cast, comparing ethanolamine with its disubstituted homolog. Differences were noted only at high α (plus what appears to be a discrepancy at $\alpha = 0.25$).

Trends in water content after 24 hr reveal that no secondary waters of hydration exist in PAA films. Table 4, which compares ethanolamine with two homologs and with benzylamine, contains no entries in Section B above 2 moles of water per mer unit. It is clear that the drastic elevation of Δ_{max} by the branched homolog does not result from desiccation. A similar reduction in water content occurred with the linear homolog (propanolamine) which gave a low Δ_{max} peaking at $\alpha = 0.5$.

The effect of desiccation is shown with benzylamine. Not only is Δ_{max} increased by the exclusion of water from the polymer folds, but the increase in rigidity is accompanied by large strains in the film. The combination produces stresses large enough to cause failure while water is still present in the drying film. In this respect it appears that ion-containing polymers resemble latexes.

Higher homologs of hydroxy amines are suggested as practical ways of reducing water sensitivity. In practice,

one would not go so far as the nearly anhydrous situation represented by benzylamine because plasticization by either water or the amine appears to be a necessary condition for film integrity at this early stage in drying. The next stage of film formation, not covered here, involves curing by crosslinking; and if residual stresses have been minimized during the drying stage they may not build up to a disastrous level during the curing stage.

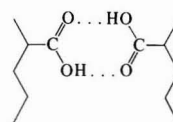
Sharp differences in trends were noted when the situation at 220 min was compared with that at 24 hr. At the beginning of the leveling-off of Δ the water content must exceed 0.5 moles/mer unit in order to avoid release from the substrate, for this level has been the common denominator in all instances of adhesive failure ($\alpha = 0.0$ and the benzylamine series). Only when hydrophilic amines are present do the drying curves proceed smoothly; to avoid a concomitant retention of secondary water or hydration, a limit must be set on the hydrophilicity.

On aging for 24 hr the secondary water (above 2 moles/mer) and about half of the primary water of hydration disappeared from the film as shown in Figure 12. Water retention of the amino alcohols was identical in magnitude and trend, and not as subject to the sudden increase above $\alpha = 0.5$ as that which appeared in the alkali-neutralized PAA (shown in Figure 12 as a dashed line). The knee at $\alpha = 0.5$ was vestigial at best.

Benzylamine, with no hydrophilic group other than the amine function, retained about half a mole of water per mer unit at all α .

Explanation of Role of Water

Aligned regions of the polymer (crystalline or intra-coil) can have carboxyl groups oriented in opposition as in a typical dimer acid;



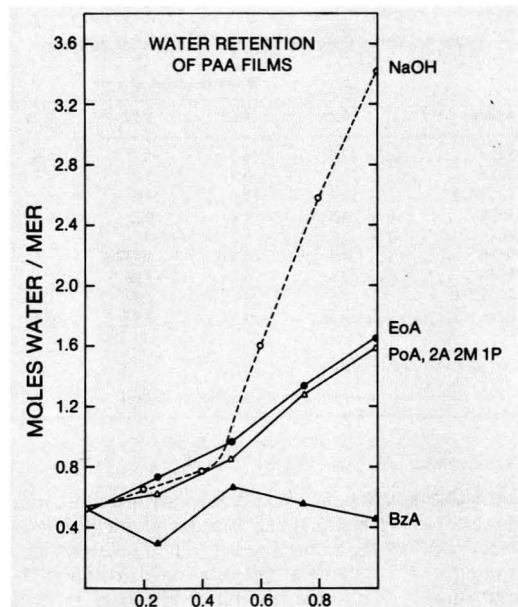
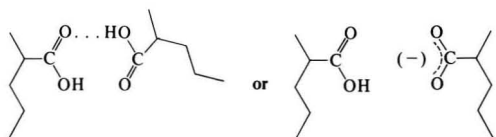


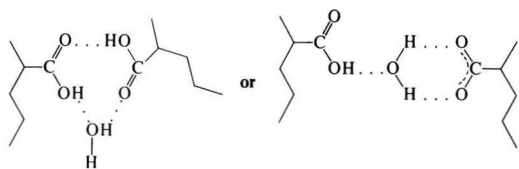
Figure 12—Water retention of PAA films at various initial degrees of neutralization, α . Primary hydration at $\alpha \leq 0.5$ is supplemented by secondary hydration attributed to clustering thereafter, to a lesser extent than with alkali, and to no extent with a hydrophobic amine

but amorphous or unmatched regions, and especially those segments in partially neutralized polymer which contain carboxylate groups would not have parallel alignment;



Somewhere in the vicinity of the carboxylate is the amine cation. Electrostatic repulsions and the incorporation of counterions cause the polymer coil to expand on neutralization, particularly beyond $\alpha = 0.5$.

The presence of water in the amorphous region serves, at first, to increase the crosslink density of the polymer, possibly via this sort of structure;



If the R tail of the amine contains hydroxyls, these groups also can H bond. The question which cannot be answered by this research is the relative positioning of the amine

Table 5— Δ_{\max} Trends with PAA Films^a

Amine	Δ_{\max} at $\alpha = 0.5$	Δ_{\max} at $\alpha = 1.0$	24-hr. Water Contents	
			$\alpha = 0.5$	$\alpha = 1.0$
EOA	27.6	15.2	14.7	19.9
DEOA	—	16.0		
TEOA	—	18.0		
POA	23.6	11.7	12.2	16.5
2A1P	18.5	27.7		
2A2M1P	20.4	33.7	11.6	15.1
BZA	21.7	37.2	8.8	4.5

(a) Arranged in decreasing order of expected hydrophilicity of the amine. Water contents in wt.%. These values are less than half that needed to satisfy primary hydration.

cation head and the hydroxy tail with respect to the carboxylate group.

A half mole of water should show strong evidence of H bonding, leading to rigidity, rather than plasticization. This evidence will be sought in ancillary measurements to be made in the course of extending the current work.

Amine Hygroscopicity

The Δ_{\max} trends among the amines lined up in accord with an intuitive rating of their hydrophilicity at $\alpha = 1.0$ (but not at $\alpha = 0.5$), giving the results shown in Table 5. Only propanolamine is out of line in this arrangement. Whether plasticization comes from hydroxy termination, from the lack of branching, or from the spacing to OH with respect to NH_2 groups is not known. The triethanolamine series, which also plasticized extensively, was hydroxy terminated but branched. One avoids in practice whatever situation leads to plasticization of this magnitude, and therefore its cause is worth further study.

That hydrophobic interactions actually play a role in film rigidity is suggested by the second column of Table 5. At $\alpha = 0.5$ there is no tendency for clustering and the concentration of counterions has not yet saturated the polymer. The low water contents encountered, especially at $\alpha > 0.5$, imply that the amine cations occupy primary coordination positions around the carboxyl. Under these conditions the compatibility of the R tail with the polymer backbone becomes a critical factor.

Exclusion of water reduces the tendency for ionic groups of the type $-\text{COO}^-$ and $-\text{NH}_3^+$ to form. Instead of dissociating, these ion pairs would form an adduct or undergo proton exchange, depending on the basicity of the amine. Dielectric relaxation studies are underway to test this hypothesis.

Copolymer Behavior

The use of PAA as a model for the copolymers that are used in water-borne coatings may not be as impractical as it appears from the wide differences in behavior trends. If PAA responds so vividly to systematic changes in the amine that rigidities vary over many decades, surely the PAA blocks that inevitably occur in a random copolymer display the same tendency on a molecular scale. There may be less of an effect observed rheologically, but the

blocks are present, nevertheless. They impart water sensitivity, and only a judicious choice of amine will relieve that sensitivity.

The most encouraging difference between random copolymer behavior and that of PAA is that branched homologs gave the same drying curves as ethanolamine. Water contents of the copolymer were too small to be measured accurately; nevertheless, whether the effect is steric or hydrophobic it is not prominent in an 8% AA polymer and the choice of amines is increased widely. Perhaps a return to amine basicity, which figured in the original selection of neutralizers, would answer unresolved questions involving the role of the cation. Additional work on the E/MA copolymer with vicinal carboxyl groups is needed. The fraction of amine remaining in the dried film needs study.

CONCLUSIONS

Both the hydrophilicity and the branching of amines used to neutralize acidic polymers control the film formation of poly(acrylic acid). These effects were absent in a copolymer containing only 8% acrylic acid and were exacerbated in a maleic acid copolymer with adjacent carboxyl groups.

Destructive residual stresses build up in PAA films that become desiccated during the drying process. Brittleness and adhesive failure result when water content falls to a half mole per repeating unit.

Branching effects are overridden by hydrophilicity. They appear to be more spatial than steric; in some respects they suggest that intercalation plays a role.

Branched amine homologs require a higher degree of neutralization to produce tough films than does ethanolamine. Water retention does not vary appreciably with the homolog, nor does its dependence on the degree of neutralization.

In contrast to alkali-neutralized polymer, films cast with amines do not contain secondary water of hydration at high degrees of neutralization.

ACKNOWLEDGMENTS

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Deceptive Closed Cup Flash Points Of Solvent Mixtures Containing Chlorinated Solvents

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Mixtures of some chlorinated solvents with toluene are reported to display flash in a closed cup tester *only* within a narrow temperature range. Above this range, the flash ceases to appear. Caution is urged whenever the flammability of mixtures of chlorinated and nonchlorinated solvent is to be assessed by means of a closed cup flash point tester, the type of tester now most commonly favored. This anomalous flash point phenomenon is not encountered with an open cup flash point tester.

INTRODUCTION

Some of the nontoxic chlorinated solvents have been suggested recently as a substitute for the conventional, flammable solvents in protective coatings.¹⁻³ Chlorinated hydrocarbons such as methylene chloride, trichloroethanes, trichloroethylene and others display no flash, and as such, they are considered to be fire retardants for liquids with a low flash point. A typical example is toluene with a flash point of 4.5°C; the possibility of suppressing its flammability by means of a nontoxic chlorinated solvent is very attractive.

With no attempt to deter anyone from considering the nontoxic chlorinated solvents as a substitute for flammable solvents, it is necessary to point out some unex-

pected irregularities in the behavior of certain solvent mixtures when they are tested for the flash point by the closed cup methods. The irregularities are of such nature that they pose a danger of overlooking a flash point and classifying a flammable solvent as a combustible or non-flammable liquid.**

FLASH POINT DETERMINATION

Three chlorinated solvents were mixed with toluene: methylene chloride, trichloroethylene and 1,1,1-trichloroethane (Chlorothene®, Dow Chemical). The flash points of the mixtures were investigated over the whole concentration range. The methods employed were Setaflash Closed Cup⁴ and Tag Open Cup⁵ Testers. The results of testings are summarized in *Tables 1-3*.

For each system, at least one particular composition was investigated in detail, as follows.

In *Table 1*, the composition with 33% trichloroethylene was tested for a flash in the closed cup at gradually higher temperatures. The first flash appeared at 8°C, and obviously this would be the flash point for the mixture. A flash also occurred at 11°C and 14°C, as expected. However, at 17°C a peculiarity was noticed; there no longer appeared any flash in the cup. Normally, one would expect the flash to become more violent as temperature of the test increases, but in this case the flash just did not appear. The testing became even more confusing when the test flame was applied inside the cup again, immediately after the first (or second) attempt; a flash was observed. A flash could also be achieved by a

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** Liquids are classified in the U.S.A. in reference to their T.O.C. flash points as extremely flammable (f.p. ≤ 20°F); flammable (20°F < f.p. ≤ 80°F); and combustible (f.p. > 80°F).

Table 1—Flash Points of Toluene/Trichloroethylene Mixtures

Trichloroethylene (%)	Flash Point (°C)	
	Close Cup	Open Cup
2.6	5.0	—
3.2	6.0	—
5.8	6.0	—
6.9	6.0	—
9	—	8
10	6.0	—
14	6.0	—
16	6.0	—
18	6.5	—
20	6.5	—
29	—	10
33	8,11,14,17 ^a ,23 ^a	11
40	11	—
60	11	—
74	—	30
90	none	—

(a) Refer to the discussion in the text for explanation.

slight blow of air against the cup opening. Apparently, in order for the gases to flash at 17°C, they required enrichment with air. The same behavior of the system was found at 23°C.

Further investigation revealed that some other chlorinated solvents, when mixed with toluene, displayed similar peculiarities during the closed cup flash point determination. Thus, for example, a mixture of 11% methylene chloride in toluene flashed at 7.5°C and 10°C, but it ceased to flash at and above 12°C unless, again, the cup was opened several times or a slight blow of air was directed against the cup opening at the moment of flame application. With Chlorothene/toluene mixtures, although it was often difficult to confirm or disprove a flash, the possibility of the temperature-dependent flash was strongly suggested.

What was observed during the closed cup flash point test is of utmost importance for the sake of safety:

Some mixtures of chlorinated and nonchlorinated solvents display flash in the closed cup tester *only* within

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a narrow temperature range. No flash occurs above this range.

CONCLUSION

When a solvent or a mixture of solvents is tested for a flash point according to ASTM D3278-73 or similar methods, the temperature of the solvent is gradually increased until a flash is observed when flame is introduced in the cup. It is tacitly assumed that vapors do not flash below a temperature at which they have been found nonflammable.

In the majority of cases this is true. The "unexpected" happens, however, when toluene is mixed with some chlorinated solvents. The following behavior of the solvent mixtures was observed when they were tested by the Setaflash Closed Cup Method:

For some compositions, a flash point was found only within a narrow temperature range. In other words, the flash ceased to appear at a higher temperature.

When a flash disappeared because the temperature of the test was increased, it reappeared when the cup was

Table 2—Flash Points of Toluene/Methylene Chloride Mixtures

Methylene Chloride (%)	Flash Point (°C)	
	Close Cup	Open Cup
10	6	—
11	7,5,10,12 ^a ,14 ^a	5
20	7	—
22 and up	none	—
23	—	17
45	—	18

(a) Refer to the discussion in the text for explanation.

Table 3—Flash Points of Toluene/Chlorothene Mixtures

Chlorothene (%)	Flash Point (°C)	
	Close Cup	Open Cup
20	6	—
28	—	13
30	8 (only) ^a	—
40	8	10
42	9,10(?),12(?) ^a	—
46	9 (only) ^a	—
50	none	—
70	—	26

(a) See the discussion in the text for explanation.

opened several times, or when a slight blow of air was directed towards the cup at the moment of opening the cup.

In practical situations, the following sequence of events could easily happen. The formulator, trying to suppress the flammability of a solvent-based coating, decides to use a mixture of toluene with methylene chloride, 89/11 by weight, instead of only toluene. He or she then determines the flash point of the new solvent by means of one of the closed cup testers. The testing is started at 12°C or above and continues, raising the temperature of the cup. Since no flash is observed, it is concluded that the solvent vapors are nonflammable, and the material is given the corresponding labels and hazard description. However, if one or both of the two following circumstances are met, the solvent vapors may be ignited; firstly, when a container with the material is open either wide enough or long enough to allow sufficient amount of air inside the container, or secondly, when the container is allowed to attain a temperature from 7.5° to 10° C, for this exemplified system. Obviously, the consequences can be disastrous.

The foregoing example should be generalized for any mixture of flammable liquids with chlorinated solvents unless an experimental evaluation proves otherwise.

Our observations also indicate that an open cup method could provide a definite flash point in cases when a closed cup method yields no flash point. As a matter of fact, the use of the open cup can eliminate all the problems, since once a temperature was reached at which the mixture flashed, it was found to flash at higher temperatures as well, as expected. However, there seems to be a general trend toward the use of the closed cups. The latter may be even required in the future by regulations for the transportation of dangerous commodities.⁶

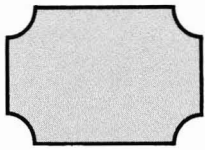
Everyone should be aware of the phenomenon described, whenever the flammability of solvent mixtures containing chlorinated solvents is to be experimentally evaluated.

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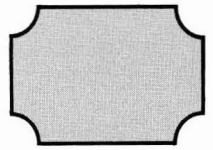
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Use of Maleimide Acceptors As a Means of Developing Crosslinking Emulsions

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The usefulness of employing maleimide-type acceptors to provide low temperature crosslinkable functionality was examined by determining the second order rate constant for the addition reaction with certain nucleophilic agents. The 3-chloromaleimido substrate was found to provide acceptable low temperature reactivity with nucleophiles while theoretically offering some degree of steric inhibition to free radical copolymerization (a tri-substituted olefin). Subsequently, to provide an acrylate-type polymer which could be post-reacted through the pendent maleimide unsaturation, 6-(3-chloromaleimido)hexyl acrylate (3-CMIHA) was synthesized using Searle's cyclodehydration method.

The model emulsions containing 3-CMIHA were prepared using a delayed, two-step addition technique whereby the total amount of 3-CMIHA was added in the last 50 weight percent of the total 60/40-ethyl acrylate (EA)/methyl methacrylate (MMA) monomer feed. However, in spite of the preventative measures used to reduce premature loss of the 3-chloromaleimido unsaturation, approximately 40% of the initial 3-chloromaleimido functionality was consumed during free radical polymerization.

Because of the effects of premature crosslinking, compositions containing incrementally higher concentrations of 3-CMIHA were found to possess network structures with greater crosslink densities and, consequently, reduced physical properties. However, compositions using 2 or 4 mole percent

3-CMIHA displayed significantly greater integrity when compared to a control which did not contain 3-CMIHA.

Of the various crosslinking agents investigated, coatings cured with *m*-phenylenediamine at ambient conditions provided the greatest increases in solvent resistance and tensile strength.

INTRODUCTION

Crosslinkable emulsions were first introduced into the coatings market in the early 1960's, and since their commercialization considerable effort has been devoted to improving the characteristics and acceptability of these products.¹ While the present generation of amino-plast-containing crosslinkable emulsions demonstrate many unique advantages over their predecessors, the marketability of these systems has been confined mainly to coil coating and wood finishing applications. The restricted end-use of these products arises, in part, because the crosslinking reaction between etherified methylol groups and carboxyl and/or hydroxyl functionality requires thermal treatment, i.e., 10 min at 400°F. Consequently, if emulsion systems are to find utility in typical on-site industrial applications, other methods of crosslinking must be employed which develop high integrity products that cure under ambient conditions.

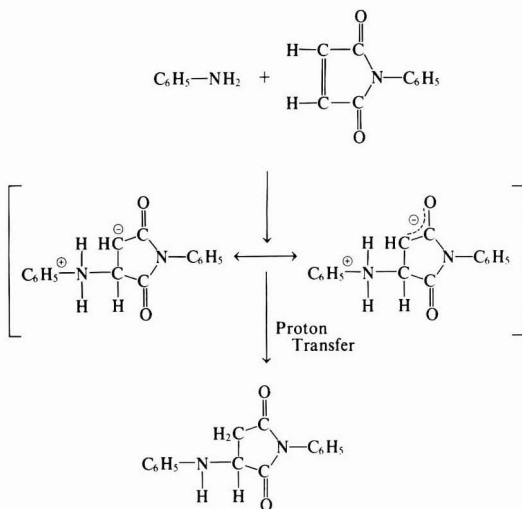
While few bimolecular reactions besides free radical coupling through autooxidation and oxirane addition by means of nucleophiles satisfy both criteria of (1) being compatible with the emulsion environment and (2) affording suitable reactivity at room temperature, several recent disclosures concerning Michael-type addition

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reactions reveal that polymer systems containing highly activated alpha, beta unsaturated acceptors such as maleimide or its chloro derivatives provide the characteristics required for the development of high integrity, low-temperature crosslinkable emulsions.

For example, besides offering such property advantages as improved impact^{2,3} and tensile strength,^{4,5} adhesion,^{6,7} and resistance to water,^{8,9} solvents,¹⁰ and thermal¹¹⁻¹³ or electrical stress,¹⁴ maleimide-containing systems are claimed to be highly reactive and capable of undergoing numerous bimolecular addition reactions at low temperatures.^{15,16} The examples in Table 1 illustrate the temperatures at which the maleimide unsaturation reacts with various nucleophiles.

The addition reaction between amine nucleophiles and the maleimide unsaturation has been found to follow second order kinetics, and the magnitude of the rate constant is governed in part by the polarity of the medium. The dependence between solvent polarity and the rate of reaction suggests that the addition of amine compounds to the maleimide unsaturation may involve the formation of a charged intermediate or transition state whose energy of activation is lowered through the dispersal of the incipient charge,²³ i.e.,



Because of the electron-deficient nature of the double bond, the maleimide substrate may undergo such bimolecular processes as dipolar cycloaddition reactions,²⁴⁻³² Diels-Alder additions,³³⁻³⁹ free radical copolymerizations,⁴⁰⁻⁴⁶ and nucleophilic additions,^{47,48} along with conventional Michael-type reactions.⁴⁹⁻⁵¹ Of these five reaction modes, perhaps Michael-type condensations and nucleophilic additions offer the greatest latitude for designing systems which can be tailored to meet specific end requirements since the crosslinking agent may be varied among such diverse compounds as ammonia,^{52,53} primary and secondary amines,⁵⁴⁻⁵⁷ thiourea,^{58,59} Schiff bases,^{60,61} dihydrazides,⁶² dioximes,^{63,64} phosphites,^{65,66} hydrogen sulfide,⁶⁷ dithiols,⁶⁸⁻⁷⁰ and pheno-

late anions.⁷¹ Thus, through the proper selection of reaction modes and respective curing agent, the conditions used to crosslink maleimide-containing systems together with the performance characteristics of the crosslinked product may be optimized to meet the numerous demands required by various end applications.

EXPERIMENTAL

The four maleimide model compounds described in Table 2 were prepared by means of the following sample procedure.

Synthesis of N-(n-heptyl) maleimide

STEP 1—PREPARATION OF N-(N-HEPTYL)MALEAMIC ACID: N-(n-heptyl)maleamic acid was prepared by reacting a solution consisting of 49.03 g (0.5 moles) of maleic anhydride dissolved in 50 g of distilled dimethylacetamide (DMAc) with a solution consisting of 58.19 g (0.5 moles) of n-heptylamine dissolved in 50 g of distilled DMAc. The reaction temperature never exceeded 10°C, and the total addition time for the amine/solvent mixture was two hours. At the end of the addition, the cold solution was allowed to equilibrate to ambient temperature, and the resulting N-(n-heptyl)maleamic acid solution appeared viscous and water white.

STEP 2—CYCLODEHYDRATION OF N-(N-HEPTYL)MALEAMIC ACID: After a two hour conversion period at ambient temperature, 25.34 g of fused sodium acetate and 306.3 g (3.0 moles) of distilled acetic anhydride were added to the N-(n-heptyl)maleamic acid solution in order to effect cyclodehydration. The resulting solution was heated to 65°C, and after a three hour conversion period at 65°C, the reaction mixture was cooled to 30°C and poured into distilled water to isolate the product. After repeated washings in water, the crude product was vacuum distilled and subsequently characterized by infrared, ultraviolet, and NMR spectroscopy. The results from the elemental analysis indicated good agreement between the experimental and theoretical values.⁷²

Preparation of 6-(3-chloromaleimido)hexyl acrylate (3-CMIHA)

STEP 1—PREPARATION OF N-(6-HYDROXYHEXYL)-3-CHLOROMALEAMIC ACID: N-(6-hydroxyhexyl)-3-chloromaleamic acid was prepared by reacting a solution consisting of 12.22 g (0.10 moles) of distilled chloromaleic anhydride (a 70% azeotrope with maleic anhydride) dissolved in 10.0 g DMAc with a solution consisting of 11.72 g (0.10 moles) of distilled 6-hydroxyhexylamine dissolved in 20.0 g of DMAc. The reaction temperature never exceeded 8°C, and the total addition time for the amine/solvent mixture was 1.5 hr. At the end of the addition, the cold solution was allowed to equilibrate to ambient temperature, and the resulting N-(6-hydroxyhexyl)-3-chloromaleamic acid solution appeared viscous and moderately amber.

STEP 2—ESTERIFICATION OF N-(6-HYDROXYHEXYL)-3-CHLOROMALEAMIC ACID WITH ACRYLIC ANHYDRIDE: After a two hour conversion period at ambient temperature,

75.69 g (0.6 moles) of distilled acrylic anhydride were added to the N-(6-hydroxyhexyl)-3-chloromaleamic acid solution in order to effect esterification of the hydroxyl groups. After an initial exotherm which raised the temperature to 48°C, the reaction mixture was heated to 60°C, and this temperature was maintained for a period of 1.5 hr.

STEP 3—CYCLODEHYDRATION OF N-(ACRYLOXYHEXYL)-3-CHLOROMALEAMIC ACID: At the end of the conversion period, the N-(6-acryloxyhexyl)-3-chloromaleamic acid solution appeared viscous and dark amber in color, and 3.78 g of fused sodium acetate were added to the reaction mixture to effect cyclodehydration. After a two-hour conversion period at 65°C, the reaction mixture was cooled to 30°C and poured into distilled water to isolate the product. After repeated washings in water, the crude product was vacuum distilled and subsequently characterized by infrared, ultraviolet, and NMR spectroscopy. The results from the elemental analysis indicated good agreement between the experimental and theoretical values.⁷²

The emulsions described in Tables 3 and 4 utilized the following sample procedure for their preparation.

Preparation of a 56.84/37.85/5.31-Ethyl Acrylate/Methyl Methacrylate/3-CMIHA Emulsion (2 mole percent 3-CMIHA)

A 500 ml flask was charged with 178 g deionized water which was subsequently deoxygenated by heating and sparging with nitrogen gas. After sparging for 5 min at 95°C, the water was cooled to 80°C, and 7.14 g (2% by weight of total monomer) of Triton® X-200 were added to the slowly agitated degassed water. After a 5 min equilibration period, 1.5 g of sodium bicarbonate buffer were added to the water/surfactant mixture; the temperature was adjusted to 75°C; and the rate of agitation was increased to 240 rpm. After a 5 min equilibration period, a solution consisting of 0.5 g of potassium persulfate dissolved in 10.0 g of deionized water was added to the water/surfactant/buffer mixture. Immediately following the addition of the initiator, a seed charge consisting of 28.42 g of distilled ethyl acrylate (EA) and 18.93 g of distilled methyl methacrylate (MMA) was added to the reaction mixture at such a rate that the

Table 1—Low Temperature Reactivity of Various Maleimide Compounds

Acceptor ^a	Donor	Reaction temp. (C)	Reference
PDM	Piperazine	-10	17
MDPBM	Piperazine	21.5	18
N-arylmaleimide	Piperidine	Room temp.	19
MDPBM	m- and p-xylylenediamine	15	20
MDPBM	Ethylene glycol bis (mercaptoacetate)	Room temp.	21
N-phenylmaleimide	Hydrogen sulfide	Room temp.	22

(a) PDM = N,N'-phenylenedimalcic acid
MDPBM = N,N'-(4,4'-methylenebis(phenylene))bis(maleimide).

temperature was maintained at 75°C. The total addition time for the seed mixture was approximately 40 min. Following a conversion period of 15 min at 75°C, an overlay charge consisting of 28.42 g of EA, 18.93 g of MMA, and 5.31 g of 3-CMIHA was added to the seed latex at such a rate that the temperature was maintained at 75°C. The total addition time for the overlay mixture was approximately 45 min. After a 15 min conversion period at 75°C, the temperature of the reaction mixture was increased to 92–94°C in order to drive the reaction to completion. When the reflux of the unreacted monomer was no longer detected, the emulsion was cooled to 30°C and filtered through cheese cloth.

Determination of the Second Order Rate Constants

The kinetic data were obtained using a Cary 17 ultraviolet spectrophotometer by following the loss of absorbance with time of certain characteristic wavelengths associated with the absorption of the maleimide unsaturation. Pseudo-first order conditions were employed whereby the concentration of the donor (nucleophile) exceeded that of the acceptor (maleimide) by a factor of one hundred. A variable temperature cell holder was employed, and the reaction temperature was controlled at 25°C with a Lauda® thermostat type K2.

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Table 2—Rate Constants for the Reaction of Maleimide-Type Acceptors With Various Nucleophilic Agents^a

Nucleophilic Agent	N-(n-heptyl) Maleimide	N-(n-heptyl) Citraconimide	N-(n-heptyl) 3-Chloromaleimide ^b	N-(n-heptyl)-3,4-Dichloromaleimide ^b
n-Butylamine	1.69×10^{-1}	1.91×10^{-4}	3.70×10^{-2}	3.45×10^{-2}
Piperidine	5.11×10^{-1}	1.89×10^{-4}	3.33×10^{-1}	6.95×10^{-1}
Diethylamine	7.49×10^{-3}	2.78×10^{-5}	5.55×10^{-3}	3.93×10^{-2}
n-Propylthiol	4.14×10^0	2.51×10^{-3}	8.92×10^{-1}	—

(a) The rate constants are expressed in liters/mole-seconds. All values were determined in ethanol at 25°C.

(b) Some deviation from second order kinetics occurred with the chlorinated derivatives.

Table 3—Effect of Delayed Addition Technique on the Degree of Divinyl-Type Premature Crosslinking

Amount of Total EA/MMA Monomer Feed Serving as a Seed Latex (%)	Amount of Total EA/MMA Monomer Feed Containing 3-CMIHA (%)	Yield (%)	Coagulum (%)	MEK Insolubles ^b (%)	Remaining 3-CMIHA Unsaturation ^c (%)
100	0	81.3	12.80 ^a	3.6	4.6
75	25	92.6	1.87	19.2	62.1
50	50	91.8	0.37	46.5	60.8
25	75	93.3	0.45	70.2	59.4
0	100	94.8	0.02	86.7	53.3

(a) Coagulum formed upon addition of 3-CMIHA.

(b) MEK = methyl ethyl ketone.

(c) The remaining unreacted 3-chloromaleimido functionality was determined by means of ultraviolet spectroscopy using 3-CMIHA as an internal standard.

Determination of the Remaining Unreacted 3-Chloromaleimido Unsaturation

The amount of 3-chloromaleimido unsaturation remaining after preparation of the emulsion was determined by means of ultraviolet spectroscopy. Basically, the absorbance of the 3-chloromaleimido unsaturation was measured both in the absence and presence of an internal standard (3-CMIHA), and the concentration of the remaining 3-chloromaleimido unsaturation was obtained by relating the concentration and percent absorbance of the internal standard with the percent absorbance of the unknown.

Determination of the Swelling Ratio

Swelling ratio measurements were determined on samples in latex form using the procedure established by Crews.⁷³

Determination of Oxirane Content

The oxirane content of the glycidyl methacrylate-containing emulsion was determined by means of the pyridinium chloride method.⁷⁴

TEST PROCEDURES

Sward rocker hardness, pencil hardness, impact resistance and solvent resistance were conducted according to standard procedures reported in the *Paint Testing Manual*, 15th ed., G. G. Sward, ed., and ASTM Technical Publication 500, 1972. Tensile strength and percent elongation were measured with an Instron tester, Model 1130, using a crosshead speed of 5 cm/min.

DISCUSSION AND RESULTS

Factors Influencing the Selection Of a Suitable Maleimide Derivative

Because of the tendency of the maleimide double bond to undergo free radical addition during preparation of the model emulsions, the experimental design of the

project included such preventative measures as (1) employing procedural techniques to reduce the residence time which the maleimide derivative experienced in the polymerization mixture and (2) optimizing those physical and chemical features of the maleimide derivative which control the susceptibility of the double bond to partake in free radical addition reactions.

The propensity of a double bond to undergo free radical addition is usually described in terms of the extent of resonance stabilization of the monomer and radical, the polarity of the double bond, and, perhaps less commonly, in terms of the steric factors governing the approach of the incoming radical.^{75,76} If the double bond of an olefinic monomer possesses sterically bulky substituents, the degrees of freedom available to the approaching radical become severely limited. Consequently, the susceptibility of the double bond to undergo free radical addition decreases since the thermodynamic factors associated with monomer reactivity (resonance and polarity) are outweighed, in this case, by the kinetic factors arising from sterically shielding the double bond. For instance, even though the double bond of methyl α -ethacrylate possesses similar resonance and polar characteristics to that of methyl α -methacrylate, the presence of the sterically larger α -ethyl group prevents methyl α -ethacrylate from achieving the facile degree of free radical polymerization characteristic of methyl methacrylate.⁷⁷

Therefore, in order to help minimize the loss of maleimide unsaturation during polymerization with conventional, vinyl-type monomers, several alpha (position 3) and alpha, beta (positions 3,4) substituted maleimides were selected as candidates for the ensuing investigation. These derivatives were tri- and tetra-substituted olefinic compounds, respectively, and conceivably provided greater steric hindrance to free radical addition than that provided by unsubstituted maleimide derivatives.

Syntheses of the Maleimide Model Compounds

The procedure used to synthesize the various maleimide model compounds was based upon the cyclo-dehydration method of Searle⁷⁸ as modified by Mallet and Darmory.⁷⁹

The modified Searle procedure was used to prepare

Table 4—Effect of Increasing the 3-CMIHA Concentration on the Physical and Functional Characteristics of the Emulsion

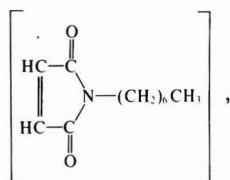
Amount of 3-CMIHA (Mole %)	Polymer Composition (Weight %)	Yield (%)	Coagulum (%)	MEK Insolubles (%)	Remaining 3-CMIHA Unsaturation ^a (%)	Swelling Ratio ^b
0	60/40-EA/MMA	98.9	< 0.10	0.0	—	∞
2	56.8/37.8/5.4-EA/MMA/3-CMIHA	95.4	0.26	44.2	60.1	32
4	53.8/35.9/10.3-EA/MMA/3-CMIHA	92.6	0.61	41.9	67.5	23
6	51.1/34.0/14.9-EA/MMA/3-CMIHA	90.3	1.22	44.1	63.1	12
8	48.4/32.3/19.3-EA/MMA/3-CMIHA	88.6	0.37	46.9	64.3	8
10	46.0/30.6/23.0-EA/MMA/3-CMIHA	87.1	0.39	48.2	61.3	9
12	43.6/29.1/27.3-EA/MMA/3-CMIHA	84.2	0.12 ^c	—	—	—

(a) The remaining unreacted 3-chloromaleimido functionality was determined by means of ultraviolet spectroscopy using 3-CMIHA as an internal standard.

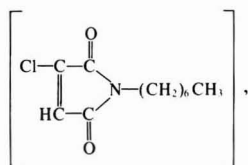
(b) Swelling ratio = volume of swollen polymer/volume unswollen polymer.

(c) Latex coagulated after aging six hours at ambient temperature.

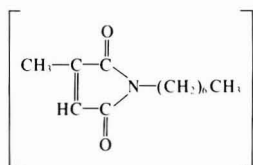
N-(n-heptyl) maleimide



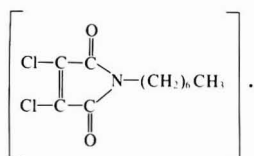
N-(n-heptyl)-3-chloromaleimide



N-(n-heptyl) citraconimide



and N-(n-heptyl)-3,4-dichloromaleimide



N-(n-heptyl)maleimide derivative since 3-chloromaleic anhydride was supplied as a 70% azeotrope with maleic anhydride. The n-heptyl derivative of each model compound was selected because the seven carbon alkyl chain provided the degree of water insolubility required for isolation of the maleimide compound from the reaction medium (acetic anhydride).

Factors Influencing the Susceptibility Of Alpha- and Alpha, Beta-Substituted Maleimide Compounds to Undergo Nucleophilic Addition Reactions

Maleimide compounds undergo nucleophilic addition reactions as a result of the electron-deficient character of the double bond.⁸⁰ Consequently, substituents which alter the electropositive nature of the maleimide double bond through an inductive effect also modify the overall reaction rate between the donor and acceptor. For example, substituents alpha or alpha, beta to the double bond which withdraw electrons, such as chlorine atoms, tend to increase the electropositive character of the maleimide double bond, thereby increasing the reaction potential with nucleophilic agents; whereas electron donating substituents, such as the methyl group, decrease the electropositive character of the double bond, thereby decreasing the reaction potential with nucleophilic agents.

Besides altering the electropositive nature of the double bond through an inductive effect, the presence of substituents alpha or alpha, beta to the double bond also influences the rate of reaction through a steric effect since the van der Waals radius of the substituent places spacial limitations on the direction of approach for the incoming nucleophile. Obviously, whether the steric effects are additive or diametrically opposed to the inductive effects depends upon the electronegative nature of the substituent. In light of these considerations, the three alpha- and alpha, beta-substituted maleimide derivatives were expected to exhibit markedly different

N-(n-heptyl)-3-chloromaleimide contained 30% of the

reactivities with a given nucleophile. For instance, N-(n-heptyl)citraconimide possesses the least electropositive double bond while providing the steric hindrance associated with one methyl group (van der Waals radius of 2.0 Å); N-(n-heptyl)-3-chloromaleimide possesses a double bond which displays more electropositive character than that displayed by unsubstituted N-(n-heptyl)-maleimide while also providing the steric hindrance associated with one chlorine atom (van der Waals radius of 1.80 Å); and N-(n-heptyl)-3, 4-dichloromaleimide possesses the most electropositive double bond while also providing the steric hindrance associated with two chlorine atoms.

Reactivity of Alpha- and Alpha, Beta-Substituted Maleimides with Various Nucleophilic Agents

Because the amalgamation of steric and inductive factors may have either a synergistic or opposing effect on the reactivity of the maleimide substrate, a donor-acceptor kinetic study was conducted in order to establish which substituted derivative provided the most suitable reactivity for ensuring low temperature cross-linking ability. To accomplish this goal, the second order rate constants (k_2) for the addition of various nucleophiles with the four maleimide model compounds were determined by means of ultraviolet spectroscopy. The kinetic data were obtained in the conventional manner by following the loss of absorbance with time of certain characteristic wavelengths associated with the absorption of the maleimide unsaturation. A portion of the more relevant data are presented in *Table 2*. In terms of the overall objectives of the investigation, the main conclusions from the kinetic study were as follows.

(1) If the various nucleophiles were ranked according to their reactivity with the four maleimide model compounds, the ratings, in general, would suggest that the addition reaction was markedly sensitive to steric factors and that the loss in reactivity resulting from steric hindrance (i.e., the reactivity of N-(n-heptyl)citraconimide was considerably less than the other maleimide model compounds) can be offset somewhat by the use of electron withdrawing groups such as chlorine atoms. For example, considering the kinetic data obtained with n-butylamine, the introduction of a methyl substituent onto the double bond of maleimide caused an approximate 880-fold reduction in the k_2 value while the introduction of one chlorine atom onto the double bond of maleimide resulted in only an approximate 4.5-fold reduction in the k_2 value. Furthermore, the introduction of two vinal chlorine atoms onto the double bond of maleimide, which greatly increases the degree of steric hindrance, resulted in only a slight decrease in reactivity as compared to N-(n-heptyl)-3-chloromaleimide.

Considering the kinetic data obtained with piperidine, N-(n-heptyl)-3, 4-dichloromaleimide displayed even greater reactivity than that of unsubstituted N-(n-heptyl)maleimide which suggests that the inductive effect may take precedence over the steric effect in controlling the rate of reaction.

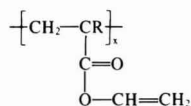
(2) Even though exceptions exist, the general order for decreasing reactivity of the various nucleophiles was: n-propylthiol > piperidine > n-butylamine > diethylamine.

Concordant with the previous conclusion, the order of reactivity for the various nucleophiles again reflects the importance of both the steric and inductive effects in controlling the rate of addition. For example, n-propylthiol was the most active addendum in the study which agrees with the claim that thiols are among the strongest nucleophiles in addition reactions with maleimide substrates.^{81,82} The greater reactivity of the secondary amine, piperidine, as compared with a primary amine, n-butylamine, also intimates that the rate of reaction is governed by the strength of the nucleophile. However, the marked difference in reactivity between the two secondary amines, piperidine and diethylamine, further suggests that the steric factors are balanced with the inductive effects.

In light of the results obtained from the kinetic study together with a consideration of the steric factors required for preventing premature loss of the maleimide double bond during free radical polymerization, N-(n-heptyl)-3-chloromaleimide was selected as the most likely candidate for ensuring both low temperature reactivity and a reduced tendency to undergo free radical polymerization. The 3,4-dichloromaleimido substrate was not selected because of the tendency of the chlorine atoms to undergo nucleophilic substitution.

Factors Involved in Designing A Maleimide-Functional Monomer

While various methods have been used to prepare vinyl-type polymers containing pendent maleimido unsaturation,^{83,84} perhaps the most common and convenient approach for synthesizing such polymers has been to replace the nonfunctional alkyl group of the N-alkyl maleimide with a vinyl-type substituent which is amenable to polymerization by means of conventional free radical techniques. The effectiveness of using such unsymmetrical divinyl monomers as a means of generating polymer with pendent unsaturated groups has been demonstrated for such difunctional monomers as vinyl methacrylate and vinyl acrylate which may be polymerized in such a manner that only one of the unsaturated groups preferentially reacts, leaving the other functionality pendent to the polymer chain,^{85,86} i.e.,



where R = H or CH₃

Unsymmetrical, divinyl monomers containing maleimido functionality (N-acryloxyethyl) maleimide, or more commonly called β -(maleimido)ethyl acrylate, have been used as crosslinking agents for specialty coatings,^{87,88} and such monomers are conveniently prepared under Searle's conditions as described by Yamado,

et al.⁸⁹ Through the application of this latter procedure, 6-(3-chloromaleimido)hexyl acrylate (3-CMIHA) was prepared in 23% yield.

The acrylate functionality of the 3-chloromaleimido derivative was chosen over the methacrylate functionality because the former possesses less affinity for copolymerizing with the maleimide double bond than does the latter. The affinity of these monomer types for the maleimide double bond is reflected in the Alfrey-Price e -values which are +1.35,³⁰ +0.60, and +0.40 for maleimide, ethyl acrylate, and methyl methacrylate, respectively. Ostensibly, because of the electropositive character of the acrylate double bond, the extent of interaction with the strongly electropositive maleimide double bond would be suppressed, and consequently the amount of maleimide unsaturation lost to intramolecular copolymerization would also be reduced.

Emulsion Techniques Used to Suppress Premature Reactivity Of the Maleimide Double Bond

Besides utilizing the more suitable inductive, steric, and polar factors to augment the desired reactivity of the maleimido-containing, difunctional monomer, the methodology used to prepare the crosslinkable emulsion was also optimized in order to further reduce the loss of maleimide unsaturation. Thus, to minimize the residence time that 3-CMIHA experienced in the polymerization mixture, a delayed, two-step monomer addition technique was investigated as a means of producing a viable system. The procedure involved dividing the monomer feed into two portions. The first portion to be polymerized contained only conventional, acrylate-type monomers [a 60/40 weight ratio of ethyl acrylate (EA) to methyl methacrylate (MMA)] and served as the "seed" latex for the second portion. The second portion of the divided feed contained the remainder of the acrylate-type monomer charge (also a 60/40 weight ratio of EA to MMA) comingled with the total 3-CMIHA charge. The effectiveness of the delayed, two-step monomer addition technique in reducing premature loss of the maleimide unsaturation was investigated by means of a compositional ladder study in which the amount of monomer feed containing 3-CMIHA was varied in 25% intervals from 0–100 weight percent of the total 60/40-EA/MMA monomer charge. Thus, by using the 0 to 100 weight percent compositional range in combination with the delayed, two-step addition technique, the residence time for 3-CMIHA in the polymerization mixture was extended over the entire minimum-maximum time spectrum. In the actual procedure, the portion of the feed containing 3-CMIHA was added 15 min after complete addition of the seed portion, and the composition of each polymer prepared in the series was maintained at 52.4/35.0/12.6-EA/MMA/3-CMIHA which represented 5 mole percent of 3-CMIHA. The results of the compositional ladder study are presented in *Table 3*.

In spite of the preventative measures taken, these data indicate that approximately 38–47% of the initial 3-chloromaleimido unsaturation was lost during free

radical polymerization. Moreover, the delayed addition technique was not significantly effective in preventing the copolymerization of the 3-chloromaleimido functionality since the addition of 3-CMIHA in the last 25% of the monomer feed resulted in only a 9.1% increase in the remaining unreacted 3-chloromaleimido functionality. Also, when 3-CMIHA was added during the later or last stage of the monomer addition, the amount of coagulum markedly increased which suggests that some degree of incompatibility existed between the stabilizing surfactant and a particle surface enriched with 3-CMIHA. Based upon these results, the split-feed technique which consisted of adding 3-CMIHA in the last 50 weight percent of the total 60/40-EA/MMA monomer charge was selected as a reasonable compromise between such factors as latex stability, premature loss of chloromaleimido functionality, and performance characteristics as governed by the thermoplastic polymer/crosslinkable polymer ratio. The split-feed technique of adding 3-CMIHA in the last 50 weight percent of the total 60/40-EA/MMA monomer charge was used as the procedure for synthesizing the remaining 3-CMIHA-containing emulsions described in this study.

Determination of the Optimum Level Of 3-CMIHA Needed For Suitable Performance Characteristics

Besides providing the properties associated with a network structure, the crosslinkable monomer also conveys to the polymer matrix those property advantages and disadvantages inherent in its own nature. Thus, the optimum concentration for physically and chemically different crosslinkable monomers may vary considerably as a result of balancing the properties emanating from the structural features of the monomer with those properties resulting from the formation of a crosslinked network.

Therefore, in order to determine the level of crosslinkable monomer which provided an acceptable balance of physical properties, a ladder study was conducted whereby the concentration of 3-CMIHA in the polymer composition was varied in increments of 2% from 0–12 mole percent of the total monomer composition. In each case, the noncrosslinkable monomer composition consisted of a 60/40 weight ratio of EA to MMA in order to obtain general application-type coatings properties,⁹¹ and 2%, based on weight of the monomer, of Triton X-200 (a sodium alkyl aryl polyether sulfonate) was used as the polymerization surfactant. The more salient characteristics of the emulsions produced in this study are presented in *Table 4*.

These data indicate that upon increasing the concentration of 3-CMIHA in the polymer, the percent conversion to polymer decreased, the crosslink density of the polymer fraction containing 3-CMIHA increased, and the percentage of 3-chloromaleimido functionality lost as a result of premature crosslinking remained approximately constant.

The independent relationship between the initial concentration of crosslinkable monomer and the amount

Table 5—Effect of Increasing the 3-CMIHA Concentration on the Physical Properties of Latex Films

Amount 3-CMIHA (Mole %)	Curing Agent	Impact Resist. Forward/Reverse (in-lbs)	Hardness		MEK Double Rubs/Mil	Elong. (%)	Tensile Strength (Kg/cm ²) After 6 hr Immersion in			
			Sward	Pencil			Initial	H ₂ O	NaOH(5%)	HOAc(5%)
0	None	160/160	10	3B	10	800	51	29	40	24
2	None	140/120	10	3B	46	610	67	37	48	30
2	1,6-hexanediamine	100/70	12	2B	92	520	80	41	51	36
4	None	110/35	12	2B	52	440	58	32	40	25
4	1,6-hexanediamine	90/24	12	2B	111	370	70	38	49	29
6	None	40/8	14	2B	67	210	36	21	23	17
6	1,6-hexanediamine	40/4	14	2B	100	170	43	25	27	20
8	None	40/5	10	3B	31	110	23	14	13	11
8	1,6-hexanediamine	30/2	12	2B	42	80	26	16	17	13
10	None	24/2	8	6B	20	60	15	9	9	5
10	1,6-hexanediamine	20/2	8	6B	25	55	17	11	10	6

of 3-chloromaleimido functionality lost as a result of premature crosslinking intimates that some restrictions are placed upon the factors controlling the addition of free radicals to the double bond of the 3-chloromaleimido moiety. Interestingly, chloromaleic anhydride is supplied as a 70% azeotrope with maleic anhydride, and therefore 30% of 3-CMIHA consists of N-(maleimido)hexyl acrylate which possesses an unsubstituted (unshielded) maleimide double bond. Conceivably, if all of the substituted maleimido functionality were consumed during polymerization, the remaining 10% loss could be that associated with the premature crosslinking of the 3-chloromaleimido functionality. In actuality, the loss may be more evenly partitioned between the two maleimido derivatives.

Also, because approximately 40% of the maleimido-type functionality undergoes premature crosslinking, an increase in the concentration of 3-CMIHA engenders a subsequent increase in the crosslink density of the gel portion of the polymer. Thus, the swelling ratio of these systems decreases upon increasing the concentration of 3-CMIHA.

A final characteristic which was not included in these data (Table 4) was the seemingly direct relationship which existed between the concentration of 3-CMIHA in the polymer and the instability of the emulsion. For example, the emulsion containing 12 mole percent 3-CMIHA coagulated after aging six hours at ambient conditions while the emulsion containing 10 mole percent 3-CMIHA coagulated after aging five days at ambient conditions. The latex containing 8 mole percent 3-CMIHA would coagulate when heated at 90°C for periods greater than 15 min (which was the condition used at the end of the polymerization to ensure high conversions of polymer).

Physical Evaluation Of the Compositional Ladder Series Of 3-CMIHA-Containing Emulsions

In order to determine the level of 3-CMIHA required for optimum performance characteristics, the ladder series of 3-CMIHA-containing emulsions was combined with the stoichiometric amount of 1,6-hexanediamine

curing agent (based on the remaining unreacted 3-chloromaleimido functionality), and films of the resultant mixture were evaluated for typical coating properties. Because of the occurrence of a substantial degree of premature crosslinking and in an effort to more clearly differentiate between the effects due to premature and postcrosslinking, the emulsions were evaluated both with and without the use of an external crosslinking agent. Also, to aid in film coalescence, 5% (by weight of latex solids) of butyl Cellosolve was added to each sample before application. The performance characteristics for this series of 3-CMIHA-containing emulsions are presented in Table 5.

While each member of the 3-CMIHA concentration series demonstrated some property improvement upon addition of the external crosslinking agent, in general these data indicate that an increase in the concentration of 3-CMIHA resulted in a subsequent decrease in impact resistance, elongation, and tensile strength of the system. This inverse relationship between physical properties and the concentration of 3-CMIHA suggests that the factors associated with the development of normal film integrity may be impaired by the effects associated with higher levels of premature crosslinking. For example, besides altering the properties associated with the microstructure of the bulk polymer [i.e., stress relaxation⁹²⁻⁹⁴ and the onset of brittleness⁹⁵], the formation of a network structure increases the modulus of the polymer making up the emulsion particle, thereby reducing the degree of flow and deformation needed for complete film coalescence. The loss of physical properties as a result of incomplete coalescence has been prevalent in other precrosslinked emulsion systems.⁹⁶

Contrasting the various disadvantages which arise from premature loss of the 3-chloromaleimido functionality, the data presented in Table 5 also illustrate the beneficial effects associated with using maleimide-type acceptors as a means of crosslinking emulsion systems. For example, as compared with the 60/40-EA/MMA control, films of the emulsion containing 2 mole percent 3-CMIHA demonstrated a 56.7% and 31% increase in tensile strength and a 35% and 23.7% reduction in elongation with and without the use of an external crosslinking agent, respectively; increased sol-

Table 6—Effect of Various Crosslinking Agents on the Physical Properties of 3-CMIHA-Containing Emulsions

Curing Agent	Impact Resist. Forward/Reverse (In-lbs)	Hardness		MEK Double Rubs/Mil	Elong. (%)	Tensile Strength (Kg/cm ²) After 6 hr Immersion In			
		Sward	Pencil			Initial	H ₂ O	NaOH(5%)	HOAc(5%)
None	140/120	10	3B	46	610	67	37	48	30
1,6-Hexanediamine	100/70	12	2B	92	520	80	41	51	36
1,2-Ethanediamine	80/30	14	2B	84	320	64	28	35	22
Jeffamine® D-230	100/80	14	2B	76	560	71	30	43	29
N-aminoethyl piperazine	50/12	12	3B	71	290	58	26	34	20
m-Phenylenediamine	40/6	16	2B	120	430	104	48	55	39
Oxalyl dihydrazide	32/5	16	2B	117	370	77	34	43	24
1,2-Ethanedithiol	80/25	14	2B	78	410	62	30	38	26

Jeffamine is a registered trademark of Jefferson Chemical Co.

vent resistance; and only moderate losses in impact resistance. Films of the emulsion containing 4 mole percent 3-CMIHA demonstrated a 35.5% and 13.3% increase in tensile strength and a 53.7% and 45% reduction in elongation with and without the use of an external crosslinking agent, respectively; increased solvent resistance; and moderately large losses in impact resistance. Emulsions containing 3-CMIHA in concentrations greater than 4 mole percent displayed properties that were inferior to those of the control. In light of these results, the emulsion containing 2 mole percent 3-CMIHA was selected as the most suitable candidate for further evaluations.

Use of Different Crosslinking Agents To Optimize Coating Properties

In order to investigate the effects that compositionally different crosslinking agents have on the performance characteristics of the system and to partially optimize the system for further evaluation, several chemically and structurally different curing agents were evaluated at the stoichiometric level (based upon the remaining unreacted 3-chloromaleimido functionality) using the emulsion containing 2 mole percent 3-CMIHA. The samples were processed in identical manner to those of the previous study (i.e., each sample contained 5% butyl Cellosolve based on weight of latex solids, films were dried at ambient for seven days, etc.), and the results of the evaluation are presented in Table 6.

Even though the concentration of each curing agent represents only a small portion of the total sample, unexpectedly the physical properties displayed by each polymer/curing agent combination varied considerably among the members of the series. These data indicate that as compared to coatings of the control (which was not crosslinked with an external curing agent), the emulsion coatings containing 1,6-hexanediamine, Jeffamine D-230 (a polyoxypropylenediamine with 2.6 oxypropylene units per molecule, total acetylatables of 8.75 meq/g, and a primary amine value of 8.30 meq/g), m-phenylenediamine, and oxalyl dihydrazide demonstrated a general, overall improvement in the performance charac-

teristics of the system while those coatings containing 1,2-ethanediamine, 1,2-ethanedithiol, and N-aminoethyl piperazine showed some losses in certain physical properties.

The antagonistic response displayed by this latter group of curing agents may be attributed to the occurrence of several deleterious modes of behavior. For instance, a reduction in certain physical properties may take place upon further crosslinking if the macroscopic, colloidal factors associated with film formation (i.e., the effect that the curing agent has at the interface between emulsion particles) outweigh the microscopic factors associated with the structure-property relationship between the crosslinking agent and the bulk polymer. Thus, if the rate of film formation is significantly less than the rate at which the curing agent combines with the functionality on the particle surface, the polymer in the outer shell areas of the emulsion particles may become crosslinked while the particles are still in suspended form. Such migrational behavior would increase the modulus of the polymer at the particle surface and subsequently reduce the flow and deformation required for complete coalescence.

Besides incomplete coalescence resulting from migration of the curing agent into the suspended emulsion particles, the loss of certain properties upon addition of the crosslinking agent could also take place if the curing agent displayed limited compatibility with the copolymer or if the resulting donor-acceptor adduct was hydrophilic in nature. In the former case, the curing agent could locate in the interstitial areas of the coalescing matrix because of a lack of diffusion, and much of the chloromaleimido functionality below the particle surface would remain unreacted. Under these circumstances, the excess curing agent could act as a plasticizer for the polymer and as a barrier to complete coalescence. In the latter case, water could become occluded in the interstitial areas of the latex particles because of association with the polar groups of the donor-acceptor adduct. Under those circumstances, water could then serve as a plasticizer for the system or act as an inhibitor to complete coalescence. Conceivably, the loss of physical properties upon addition of certain crosslinking agents may be a function of all three of the previously described modes of behavior.

If the various crosslinking agents were ranked in order of decreasing effectiveness as determined by the impact strength, solvent resistance, elongation and tensile strength of the system, the ratings would indicate that m-phenylenediamine was the most consistent and effective curing agent for providing those property changes which reflect the formation of a crosslinked network. Thus, after drying at ambient conditions for seven days, emulsion coatings containing m-phenylenediamine demonstrated a 93% reduction in reverse impact strength, a 160% increase in solvent resistance, a 30% reduction in elongation, and a 54% increase in tensile strength as compared with the control coatings which did not contain a crosslinking agent.

CONCLUSIONS

The suitability of using maleimide-type acceptors as a means of providing room temperature crosslinkable functionality was investigated by determining the second order rate constants (k_2) for the reaction of N-(n-heptyl)-maleimide, N-(n-heptyl)citraconimide, N-(n-heptyl)-3-chloromaleimide, and N-(n-heptyl)-3, 4-dichloromaleimide with conventional nucleophilic agents. While all of the maleimide model compounds displayed some degree of reactivity at room temperature (the k_2 values varied from a minimum of 414000×10^{-5} to a maximum of 2.78×10^{-5} l/mole sec), the 3-chloromaleimido moiety was selected as the most suitable candidate for the ensuing investigation since the chloro substituent provided a means of sterically protecting the double bond from excessive, premature loss during free radical polymerization.

In order to prepare an acrylate-type polymer containing pendent 3-chloromaleimido unsaturation, 6-(3-chloromaleimido)hexyl acrylate (3-CMIHA) was chosen as a reasonable candidate for the investigation. The emulsions were prepared using a delayed, two-step addition technique whereby the total amount of 3-CMIHA was added in the last 50 weight percent of the total 60/40-ethyl acrylate (EA)/methyl methacrylate (MMA) monomer feed. However, in spite of the methodological and steric measures used to prevent premature loss of the 3-chloromaleimido double bond, analysis for the remaining unsaturation indicated that approximately 40% of the initial 3-chloromaleimido functionality was consumed during free radical polymerization.

While films containing increasing amounts of 3-CMIHA (up to 12 mole percent) displayed reduced physical properties as a result of the premature crosslinking, coatings of the emulsion containing 2 mole percent (5.31 weight percent) 3-CMIHA demonstrated a 56.7% and 31% increase in tensile strength and 35% and 23.7% reduction in elongation with and without the use of an external crosslinking agent (1,6-hexanediamine), respectively; increased solvent resistance; and only moderate losses in impact resistance when compared to a 60/40-EA/MMA control which did not contain 3-CMIHA.

When the emulsion containing 2 mole percent 3-CMIHA was combined with various chemically and structurally different curing agents, the coatings cured with m-phenylenediamine displayed a 93% reduction in reverse impact strength, a 160% increase in solvent resistance, a 30% reduction in elongation, and a 54% increase in tensile strength when compared with identical coatings which did not contain a crosslinking agent.

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- No. 135—"Polymer Films Containing Chemically Bound Fungicides—An Introduction"—G.A. Stahl and C.U. Pittman, Jr., **Apr. 1978**, p 62.
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- No. 139—"Application of Hydrodynamic Volume and Swelling Theory to the Crosslinking of Latex Particles"—G.M. Crews, G.C. Wildman, J.R. Grawe, and G. Bufkin, **Oct. 1980**, p 33.

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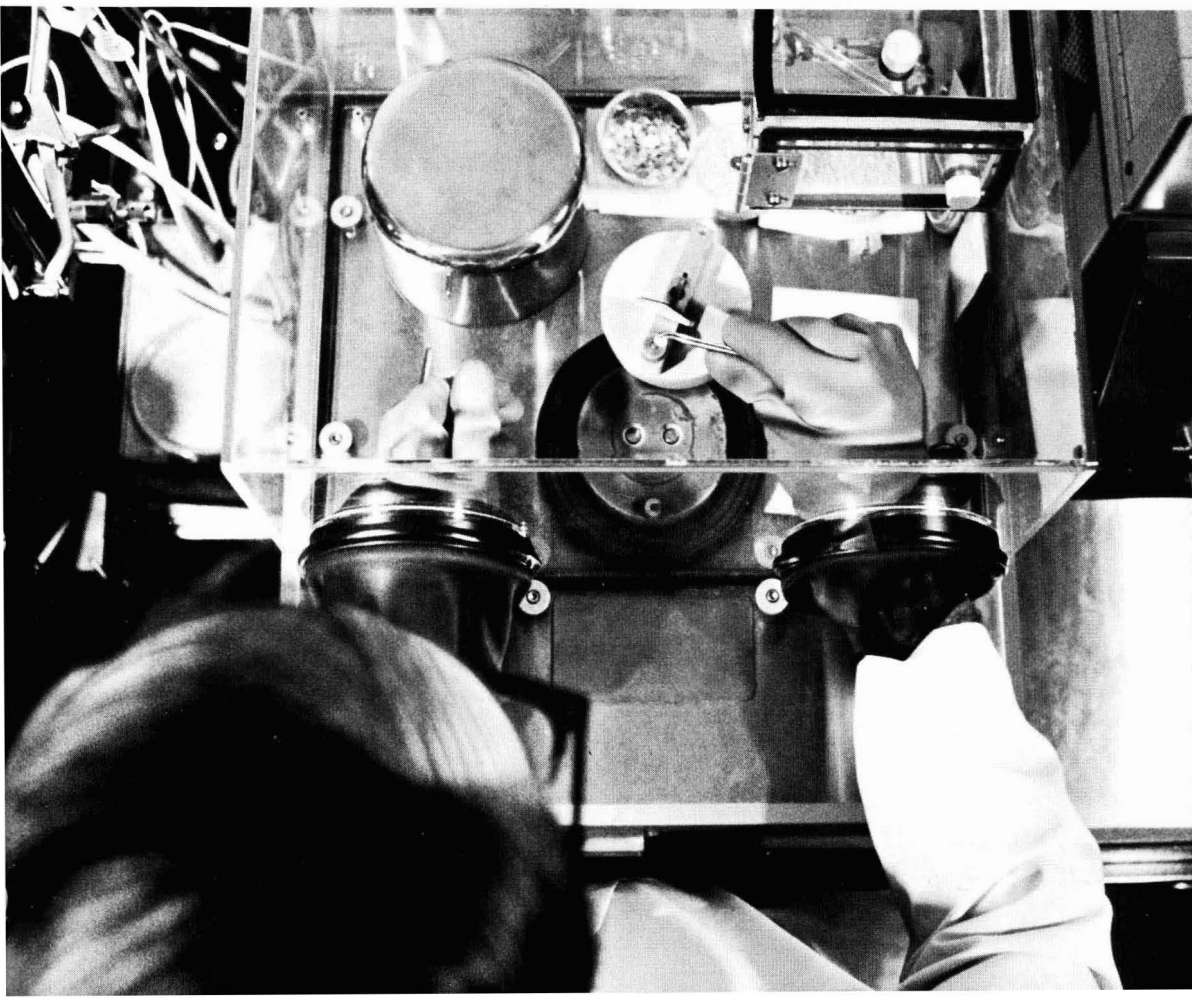
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These subcommittee reports are for the use of the membership of ASTM Committee D-1 in continuing its work and for the interest of readers in the activities of ASTM Committee D-1. The reports are not official actions of ASTM and may not be quoted as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

January 1981 Subcommittee Reports Of ASTM Committee D-1

The January 1981 meeting of ASTM Committee D-1 on Paint and Related Coatings and Materials was held on January 18-22 at the Dutch Inn Hotel in Buena Vista, Florida. In the four and one half days preceding the final report session and general meeting of Committee D-1, over 150 members and guests met in 137 scheduled meetings of D-1 subcommittees and working groups. The present membership of Committee D-1 is 531.

One day of the meeting was a joint symposium with Committee G-3 on Regimens for Prediction Performance of Decorative and Protective Surfaces. Two special sessions were held by Sub. D01.57 on Artists' Paints. The first session heard Mr. Marion Mecklenburg describe the results of a four and one half year study funded by the National Museum Act on the mechanical properties of paint films on the substrates used by artists. The second session was devoted to the reorganization and expansion of the task group on toxicity to deal with cautionary labeling for artists' paints and other related materials used by artists in response to pressure from Congress for a voluntary program to deal with labeling for chronic toxicity in these materials instead of further legislation.

Subcommittee Officers appointed by Chairman S.B. Levinson were: D01.90 Executive Subcommittee—R.F. Brady to serve out the unexpired term of R.C. Kissler; Sub. D01.33 on Varnish and Resins Including Shellac—A.C. Abbott to replace W.C. Golton as Chairman; Sub. D01.48 on Zinc Rich Coatings—M.D. Tellor as Chairman; Sub. D01.51 on Powder Coatings—C. Grenko to replace R.C. Kissler as Chairman; Sub. D01.94 on Awards and Memorials—H.K. Hammond III to replace R.C. Kissler as Chairman; Sub. D01.94.01 on Henry A. Gardner Award—H.M. Werner as Chairman; Sub. D01.94.02 on W.T. Pearce Award—S. Le Sota as Chairman; Sub. D01.94.03 on Certificates of Appreciation—W.C. Spangenberg as Chairman; Sub. D01.94.04 on Honorary D-1 and ASTM Membership—R.C. Marck as Chairman; and Sub. D01.94.05 on Award of Merit—H.A. Wray as Chairman. It was also announced that H.E. Ashton will continue to serve as Chairman of Sub. D01.18 on Editorial and Sub. D01.20 on Statistics and Sampling until replacements are announced.

Highlights

The following projects of major interest to the coatings industry represent areas of new or increased emphasis since the previous reports:

D01.07—considering a request from Military agency to convert a number of Federal and Military Specifications to ASTM documents.

D01.21.71—reactivated to evaluate a different method of sample preparation than is currently used in Method D 3335 for Determination of Low Levels of Lead, Cadmium and Cobalt in Coatings.

D01.22—proposed practice for disposing of harmful laboratory waste.

D01.23.11—a round robin to evaluate the Weftig wet film thickness gauge.

D01.24.25—a new task group which will survey surface tension methods and select one or more to write up as an ASTM method.

D01.26.23—a subgroup formed to evaluate portable instruments designed to test horizontal coatings for retroreflectance.

D01.27.02—will conduct a round robin to compare various water test methods and provide precision data.

D01.27.16—considering a proposed new chalking method incorporating procedures commonly used in the industry.

D01.29.01—a new task group formed to develop a practice for preparing sandblasted steel panels for coatings testing.

D01.31—is revising and updating a number of pigment specifications.

D01.33.25—is preparing a practice on methods of analysis of polyvinyl chloride resins and copolymers.

D01.33.14—will consider NMR methods to analyze alkyd resins.

D01.41.01—will continue round robin test program on latex flat wall paints.

D01.42.02—started development of a test method to evaluate practical stain removal.

D01.42.07—attempting to develop a method to evaluate sag resistance by the use of a draw down bar.

D01.42.17—is attempting to develop a method to measure practical opacity.

D01.44.01—new round robin on abrasion resistance, reflectance and viscosity of traffic paints.

D01.44.02—will review a proposed method for determining chlorinated rubber in highly pigmented alkyd containing chlorinated rubber finishes.

D01.44.06—a new group to study a method of measuring bead retention.

D01.48—a new subcommittee on zinc rich coatings. Several task groups are planned to develop methods on several properties.

D01.52.12—plans a round robin to investigate further the

performance and durability of the finish on textured paneling using standard dirt and the Gardner Washability machine.

D01.53.01—will begin work on painted aluminum and zinc coated substrates using a modification of the current biaxially stretched adhesion test to prepare methods for these substrates.

D01.53.03—a future round robin is planned for the development of a method to determine coating weights of zinc and iron phosphate on cold rolled steel.

Next Meetings

June 21–24, 1981—Providence, Rhode Island (Biltmore Plaza)

January 17–20, 1982—New Orleans (Monteleone)

SUBCOMMITTEE D01.92 MEMBERSHIP DEVELOPMENT

R.A. Brown, Chairman

Discussion at the meeting involved two general areas, the best sources for recruiting new members and the ways in which present members who are inactive could be encouraged to be more active. Past efforts at tapping sources such as the NPCA and the Federation were reviewed and more such attempts are indicated. With respect to the present membership, the letters to members and recognition documents to bosses which have recently been discussed were again reviewed. Such steps are still planned. It was pointed out that each subcommittee chairman will now be receiving a list each January of the voting and attendance records of each member for the previous two years. The circulation of such information could conceivably have some effect on inactive members. All should become aware that such records are being made and studied.

DIVISION 1 ADMINISTRATIVE

SUBCOMMITTEE D01.07 GOVERNMENT CONTACTS

R.F. Brady, Chairman

Copies of a booklet entitled "Guide to the Acquisition of Commercial Products" were distributed. This booklet is a guide to the commercial products acquisition methods which are coming into use. Attention was also directed to the procedure published in the *Federal Register*, Vol. 46, pp 1574–1587 (Jan. 6, 1981) which placed into effect OMB Circular A-119 on "Federal Participation in the Development and Use of Volun-

tary Standards." The procedures became effective on Feb. 5, 1981.

A letter was received from Naval Facilities Engineering Command asking that D-1 consider developing ASTM specifications to replace 55 Federal and 14 Military Specifications. It was recommended that the reply make the following points: (a) list the 69 documents in priority order; (b) ask for NAFAC participation in this effort with a technical person designated as a point of contact; (c) give a history of past D-1 efforts in this area pointing out that D-1 does not like to convert composition types; (d) one of the specifications on the list, TT-P-29 for interior latex paint, is receiving consideration in Sub. D01.41; (e) requirements for Qualified Products Lists will not be included in any ASTM specification; and (f) a meeting of D-1 and NAFAC personnel would be highly desirable to discuss the intent and problems associated with their proposal.

It was noted that Change Notice 1 to Federal Test Method Standard 141 has been published. It was pointed out that inspection would be helped if there was a precision statement in the weight-per-gallon test. It was felt that inclusion of the Index from this standard in either Part 27, 28, or 29 of the Book of Standards would facilitate cross referencing of ASTM and Federal Methods. Discussion with ASTM staff indicated this could be done.

SUBCOMMITTEE D01.13 CONSUMER AFFAIRS

E.T. Mooney, Chairman

Minutes of the D01.90 Executive Subcommittee meeting held in Philadelphia on Oct. 14, 1980 announced that a new scope had been approved for Sub. D01.13 and that both task groups D01.13.01 and D01.13.02 had been dissolved. The work of these groups has

been taken over by Sub. D01.41 on Criteria for the Purchase of Commercial Paints and Coatings. It was generally agreed that Sub. D01.13 should relate its interests and activities to the problems of individual paint users or consumers. Although a great deal of information has been made available to paint consumers from many sources, it is difficult to get them to read it and use it. It was suggested that Sub. D01.13 meetings could be used to talk with paint users, perhaps inviting wives of D-1 members to meetings.

A need was expressed for a practical and effective paint spreading rate test method which can be used to determine a practical spreading rate for specific paints and coatings. This need will be conveyed to Sub. D01.42 on Architectural Paints. It was voted to recommend to D01.90 Executive Subcommittee and Sub. D01.96 on Long Range Planning that Sub. D01.13 should continue as an administrative subcommittee for the purpose of monitoring the work on Consumerism done by other organizations such as the National Paint and Coatings Association and to be aware of activities in both industry and government which may affect the individual paint consumer.

SUBCOMMITTEE D01.16 DEFINITIONS

S. Le Sota, Chairman

Definitions for acid number, batch, edge-tracking, natural spreading rate, soil and stain were discussed. To protect against legal liability, it was recommended that the present definitions on industrial talc and industrial talc, non-asbestos type, be withdrawn from our list since the qualification on asbestos content is still open to misinterpretation. These definitions and the recommendation on withdrawal will be submitted for a Sub. D01.16 letter ballot.

DIVISION 20 RESEARCH AND GENERAL METHODS

SUBCOMMITTEE D01.20 STATISTICS AND SAMPLING

H.E. Ashton, Chairman

The revision to D 3925, "Practice for Sampling Paint," received no negative votes but several editorial comments, most of which were not related to the revision. Where appropriate, changes were made and the complete document will be sent to Society ballot.

The results of the subcommittee ballot on the "Practice for Interlaboratory Testing of Paint" were reported and reviewed. All of the changes proposed were, at the time of the meeting, acceptable with only minor changes. A request to change the design of experiments to allow lower replication when there are several samples was also agreed to. Suggestions received on the subcommittee and main committee ballots of January and June 1980 for other changes had been circulated for comment with the revisions. It was agreed to incorporate the "dot" diagram to illustrate a concise way of presenting results and to add blind replication as an alternate design. Because these proposals had been sent to subcommittee it was moved that these additional revisions be included on the D-1 ballot with those accepted above.

SUBCOMMITTEE D01.21 CHEMICAL ANALYSIS OF PAINT AND PAINT PRODUCTS

R.W. Scott, Chairman

The following actions were taken to resolve negatives received during recent letter ballots: On the revision of the new standard, "Test for Determination of Water in Paints and Paint Materials: Karl Fischer Method," one negative requested that the use of pyridine be avoided in the procedure. Since the voter did not provide an alternate reagent or procedure and the need for the method being balloted is immediate, it was voted to overrule the negative. A second negative was found persuasive and the suggested editorial changes will be included in the standard. On the revision of D 2369, "Test for Volatile Content of Coatings," one negative was overruled on the basis that all of the points raised had been considered previously by the task groups during development of the method. A full discussion of this negative

will be found in the minutes of the task group. A second negative was found persuasive and editorial changes were made to comply with his suggestions. A third negative was overruled because the points it raised had also been considered previously. On the negatives received on reapproval of D 1135, "Chemical Analysis of Blue Pigments;" reapproval of D 2621, "Infrared Identification of Vehicle Solids from Solvent Type Paints;" and reapproval of D 3271, "Practice for Direct Injection of Solvent Base Paints into a Gas Chromatograph for Solvent Analysis," all of the negatives were found persuasive and editorial corrections were made where required. In addition to the cited negatives, 27 comments were received concerning the 17 items balloted which are the responsibility of Sub. D01.21. These comments were all appropriate and the necessary editorial changes will be made. A Society ballot negative on D 3960, "Volatile Organic Content of Paints and Related Coatings, Practice for Determining," was withdrawn after the task group agreed to an editorial revision of the scope action.

Group 13, Coordination of VOC Standards and Information, J.C. Weaver, Chairman, addressed the resolution of a Society negative ballot on D 3960, "Practice for Determining Volatile Organic Content (VOC) of Paints and Related Coatings." This negative was based on the definition of VOC in D 3960 which does not include those solvents exempted as VOC by the EPA and state implementation programs. After a lengthy discussion, the voter agreed to withdraw his negative based on the insertion of section 1.3 under "Scope" which will read: Section 1.3: This method includes all organic materials volatile under the test conditions. When used for regulatory purposes, allowances may be made for organic materials which are exempt, i.e., as non-photoreactive material.

Group 22, Analysis of Electrocoat Bath Samples, A.G. Yeramyian, Chairman, acted on a negative vote on the method, "Nonvolatile Content and Pigment Content of E/C Bath." The voter stated the method was not necessary and can be replaced by D 3723 with the addition of a note and minor changes. The negative was ruled nonpersuasive since it was felt that sufficient differences existed between the two methods. The round robin results on acid and base equivalent values were reviewed and found to be acceptable. After editorial review the method will go to Sub. D01.21 letter ballot. pH and conductivity measurements will be the theme of future work.

Group 23, Analysis of Hot Melt Traffic Markings, D.R. Miller, Chairman, discussed the results of the current

round robin. Only three of the seven collaborators reported results. Major variances were noted in the determination of lead chromate. This determination will now be reviewed by task group D01.21.42.

Group 24, Test Method for Volatile Content of Coatings, A.C. Abbott, Chairman, reported that three negatives and one editorial comment were received on the balloting of the revision of D 2369, "Proposed Test Method for Volatile Content of Coatings." The first negative was found nonpersuasive on the basis that all of the objections had been considered during development of the method, establishment of precision and during round robin testing. The second negative was resolved by an editorial insertion in para 1.3. The third negative was also found nonpersuasive because the subject had been considered during method development and addressed in section 1.3. The task group voted to become inactive and will submit D2369 with revisions to Society ballot.

Group 25, Revision of D 2832, D.L. Campbell, Chairman, received reports from four working groups that had contacted other subcommittees and committees with jurisdiction of standards referenced in D 2832. A decision was made to prepare four practices based on D 2832. First drafts of these practices will be prepared and distributed to task group members. Work is proceeding at an excellent rate.

Group 26, Revision of D 2697, E.A. Praschan, Chairman, reported that preliminary study using aluminum tubes rather than the stainless steel discs in D 2697 indicated comparable results were obtained but the overall time needed was lower with the new approach. A round robin will be started within the next two months to compare both methods. Five representative paint samples will be tested by nine collaborators.

Group 42, AAS Analysis of Pigments Recovered from Whole Paints, W.V. Moseley, Chairman, reported that results obtained from determining TiO₂ using draft #8 showed the values were on the low side of the theoretical. However, this could be accounted for by the moisture and organics driven off in the low temperature ashing. After the data is analyzed for precision, the method will be submitted for a subcommittee letter ballot.

Group 46, X-ray Analysis of Pigments, H. Neubold, Chairman, discussed the results of the third round robin on the method to determine titanium dioxide using tetramethyl ammonium bromide as an internal standard. The precision was

considered quite poor. This was believed to be due to poor dispersion of the bromide, especially in the oil-based paints. New methods will be evaluated for incorporating the bromide compound. These will include the use of ceramic or steel beads while shaking the paint with the internal standard and a method of predissolving the bromide in a solvent compatible with both water-based and oil-based paints. The use of La_2O_3 as an internal standard will also be reevaluated. Another round robin will be run based on the findings of the exploratory work.

Group 52, Trace Levels of Benzene in Coatings, R. Domingo, Chairman, reported that the task group has been placed on an inactive status due to low interest.

Group 53, Trace Levels of Monomers in Paints, G. Cunningham, Chairman, discussed problems in obtaining suitable and stable reference standards. A round robin will be initiated around March 1, 1981. The modified method will be distributed to the collaborators. Residual butyl acrylate monomer in latex systems will be sought.

Group 54, Water Content of Water Reducible Paints by the Karl Fischer Method, W.C. Golton, Chairman, resolved the two negatives received as a result of the recent main committee letter ballot. The method will now go to Society ballot. The present method determines water in the 20-60% concentration range but does not address the low level or trace amounts (less than 2%) found in solvent type paints. The task group decided to take no action on this matter at this time and voted to go on inactive status pending results from the Society ballot.

Group 61, Revision of D 564, L. Bazarko, Chairman, has voted to go on inactive status since several methods have been completed and accepted as ASTM standards.

Group 72, Metals in Air Particulate Samples, L. Di Carlo, Chairman, has completed a round robin relating to the determination of lead and chromium. After the data are statistically evaluated and a suitable precision statement is generated, the method will be sent to a subcommittee letter ballot. A new round robin will be initiated on a limited basis to determine if a valid method is available for the determination of lead and chromium in lead silico chromate pigment dust samples.

Group 80, Exploratory Analytical Research, is currently inactive but will be reactivated when a new group leader is selected.

SUBCOMMITTEE D01.22 HEALTH AND SAFETY

H.A. Wray, Chairman

It was reported that the results of a round robin by five laboratories on determining closed cup flash points by two Seta methods, D 3278 and D3828, on relatively high viscosity materials indicate the methods compare favorably. Further testing is needed on higher viscosity materials, i.e., Z-5 to Z-10, and members were urged to submit samples. One member volunteered to furnish adhesive or mastic type materials to get these tests started. Another member also volunteered to try to get materials. On D 1310, "Flash Point by Tag Open Cup," a revision has been made to include the use of the method for determining fire point of liquids at the recommendation of the Coordinating Committee for Flash Points. The revised D 1310 will be submitted for subcommittee ballot. Under terminology, a new definition of flash point is given and under Description of Terms, a description of the term Fire Point is given.

The following actions were taken on disposition of negatives on Society ballot: On D 3934, negatives were received from two voters on this flash-no flash method. The first negative had two parts. One claimed the term P was not defined in the draft. Actually, the term P is defined in Section 10, Note 3. It was therefore ruled nongermane. The second part was on the lack of a precision and accuracy statement. This was ruled persuasive and a precision statement and accuracy statement will be added. The second negative involved seven sections of the draft. The first comment was that the draft was ambiguous and incomplete. This was ruled nongermane because it appeared the voter did not have the latest draft. The second comment was that true equilibrium conditions are not reached. This was ruled persuasive and a qualifying statement will be included in the next draft. The third comment was that Method D 3278 is not an equilibrium method as written. This was ruled non-persuasive on the basis that D 3278 flash-no flash method is an equilibrium method if D 3243 and D 3828 are, since they use the same procedure. The fourth comment stated the use of water baths in the method can be unsatisfactory since water vapor can cause inerting at temperatures starting at about 160°F. This was ruled persuasive. A statement covering the possible inerting effect will be added and the use of a covered water bath will be recommended. The fifth comment noted that the temperature should be adjusted to within some range $x^\circ\text{F}$ above the minimum flash point aim. As written, a tolerance above and below the mini-

mum corrected flash point specification is implied. This was ruled nonpersuasive. "Within" is defined by the dictionary as "not beyond quantity." This is done so that during the 10 minute hold the temperature may not go beyond the minimum temperature. Regulations indicate temperatures less than a specified degree. The sixth comment is editorial and states the flame appearing at the orifice while attempting a test will show the flash point is not always "blue." This was accepted by the subcommittee. The seventh comment stated that in view of the condition of the draft, editorial comments similar to those included on the sheet accompanying the draft have not been made. It was felt that for the most part comments have been considered. On motion it was voted to make the changes and resubmit D 3934 to subcommittee ballot. Also on D 3941, "Revision of Method-Definitive Method," a request to eliminate the use of the preliminary test will be honored and the revision submitted to subcommittee ballot.

On Fire Resistant Tests, it was reported that Committee E-5 has reviewed two tests, D 1360 "Cabinet Method," and D 3806 the "Two Foot Tunnel Test." Neither test met the 10 affirmatives required for E-5 approval. Since no interest was expressed by members present to work actively on these two fire resistant tests, it was suggested these methods be withdrawn. Before this is done, people who might be interested in retaining these methods will be contacted in an attempt to get the work done which will satisfy E-5.

It was reported that D 3630, "Standard Practice for Determining Constituents Classified as Hazardous Contained in Protective Coatings," was revised, balloted and reapproved and has been published in the December 1980 issue of the *Standardization News* for Society ballot. Regarding the proposed Hazardous Material Labeling Practice, the proposed practice prepared by the National Paint and Coatings Association was again discussed. This practice, titled HMIS (Hazardous Materials Identification System), suggests a label composed of four categories, i.e., Health, Flammability, Reactivity, and Personal Protection Equipment. The first three groups would contain a rating of 0, 1, 2, 3, 4 with 0 being the least hazardous. The Personal Protection Equipment scale is based on an alphabetized rating, i.e., A, B, C, . . . X with A requiring the minimum amount of protection. Since OSHA has just published rules on labeling in the *Federal Register* (Jan. 16, 1981) which the subcommittee members have not yet received, it was decided to postpone any active work on the writing of an ASTM standard until later. OSHA and EPA representatives will be invited to meet

with members of Sub. D01.22 on April 10 in Washington, D.C. following the regular Coordinating Committee for Flash Points meeting. The chairman advised of his letter comments to ASTM supporting the use of precautionary statements in ASTM standards. His suggested changes to the proposed statement was agreed to by the subcommittee. The biggest concern is that the caveat not be used in standards when no hazardous material or operation is involved. Copies of a "Standard Method for Disposing of Harmful Laboratory Wastes" which had been drafted were distributed. It was agreed that this draft should be balloted by the entire subcommittee membership.

On the status of flash point regulations, it was pointed out that differences exist in flammability and combustibility categories among the various regulatory bodies as published in various sections of the *Code of Federal Regulations*. For example, the Department of Transportation, in D.O.T. 49 C.F.R., uses closed cup flash points and rules that flash points under 100° F are in the flammable class and those 100° to 200° F are combustible. The Coast Guard, in 46 C.F.R., has cut-offs of less than 80° F, flammable and between 80° and 150° F, combustible. Likewise, OSHA, CPSC, and EPA have different cut-off points within their rulings. This information was gathered in an attempt to simplify and unify flash point regulations.

A number of items were discussed with regard to activities of the Coordinating Committee on Flash Point (CCFP). One is the use of the term "safety" solvent. This group objects to the use of this term in commerce. ASTM Committee E-34 on Occupational Safety and Health has been advised of our position that "safety solvents" listed by the Underwriters Laboratories be reviewed and that no solvents having a flash point be listed as "safety solvents." Military Specifications MIL-C-18718B reportedly contains a similar "safety solvent." The second item concerns a new flash point definition. The definition now reads: "Flash Point—the lowest temperature, corrected to a pressure of 760mm Hg (101.3 kPa, 1013 mbar), of a substance at which application of an ignition source causes the vapors of a specimen to ignite under specified conditions of test." CCFP has also worked on a definition of a "flammable solid." CCFP is also using the Seta Open Cup Flash as the basis of a combustibility test of mixtures. Volunteers are needed to help in a round robin test on this approach. CCFP has evaluated six calibrating liquids for flash point testing. These should be ready for publication in April. The CCFP is also trying to obtain a good method to use in their definition for a "solid material." It has also reviewed materials which flash after the

materials have boiled and has decided that if a material boils before it flashes, it should be considered not to have a flash point.

A proposal was made to develop a test procedure to determine the nonvolatile portion of drying oils by flash point. This test would be used as a reference in Method D 555, "Practice for Testing Drying Oils." It was agreed to submit this development for subcommittee ballot.

SUBCOMMITTEE D01.23 PHYSICAL PROPERTIES OF APPLIED PAINT FILMS

M.P. Morse, Chairman

The negative votes received in the recent main committee letter ballot were resolved as follows: (a) Negatives cast by one voter on items 19, 20, and 21 (methods for abrasion resistance) were found persuasive. The methods will be revised and resubmitted for a subcommittee and main committee letter ballot; (b) Negatives cast by three voters on item 22 (film thickness D 1186) were found persuasive. The suggested editorial changes will be made and the method will be submitted for Society ballot; (c) A negative cast on item 25 (impact test D 2794) was found persuasive. The method will be revised and resubmitted to subcommittee and main committee letter ballot; (d) A negative cast by another voter on item 24 (tensile properties D 2370) was also found persuasive. The method will be revised and resubmitted to a subcommittee and main committee letter ballot.

Group 10, Adhesion, H.E. Ashton, Chairman, is continuing its effort to develop a tensile pull-off test that will differentiate coatings in the good adhesion range. Additional tests have been conducted with the Instron Tester to find adhesives suitable for the pull-off test. The results show that two types of adhesives are suitable. All of the coatings tested failed by cohesion. Plans are being made to locate and test coatings that will fail by adhesion in this test. More results for measurements with the Tooke gauge were reported. Once again the Tooke ranked the coatings in reverse order of that obtained with the tape adhesion test. The availability of Bell Scrape Adhesion testers is being investigated to determine if a round robin can be conducted to determine the precision of the method.

Group 11, Wet Film Thickness, H.A. Ball, Chairman, discussed the notch type wet film thickness gauges on the market, e.g., the variation in their construction and the types of materials used, and decided to draft a standard practice for these gauges. This draft will be prepared and circulated to the group before the

next meeting. A new type of wet film thickness gauge, the Weftig, was presented. It was decided that a round robin would be conducted to evaluate this gauge.

Group 12, Dry Film Thickness, K.A. Trimble, Chairman, agreed that the three negatives received regarding method D 1186 were persuasive. The method is being revised to include the use of NBS standards for magnetic pull-off gauges and to make reference to SSPC method PA-2 for measuring film thickness in the field. Also, the two precision tables will be condensed into one table. Both this method and D 1400 will be submitted to Society ballot. The results obtained from the microscopic measurements of the cross sections of the coating films used in the round robin tests are being compiled. They will be distributed to the group for comments.

Group 14, Hardness, Mar Resistance, and Abrasion Resistance, M.P. Morse, Chairman, reported that as a result of negative votes received in the recent main committee letter ballot, the three methods for abrasion resistance (Taber, falling abrasive, and air blast abrasive) are being revised to increase the tolerances for specimen thickness to more realistic values. Some suggested editorial changes will be incorporated also. The three methods will be resubmitted to subcommittee and main committee letter ballot. The group plans to investigate methods for evaluating the mar resistance of coatings.

Group 15, Slip Resistance, G.D. Ernst, Chairman, reported that tests are being conducted to compare the slip resistance results produced by the James, Brungraber, and Liberty Mutual testers. Each of these instruments measure the coefficient of friction at the point and moment of contact of a shoe on a surface. More data are needed to compare results for surfaces having low coefficients of friction. Such coatings will be prepared and tested in a round robin test.

SUBCOMMITTEE D01.24 PHYSICAL PROPERTIES OF LIQUID PAINTS

C.K. Schoff, Chairman

Group 19, Viscosity by Efflux Cups, discussed the recent ballot on the proposed dip cup method. A negative relating to both editorial and procedural aspects of the method was ruled persuasive. The suggested changes will be made and the method reballoted. D 1200 on "Viscosity by Ford Cup" is up for reapproval. Some editorial changes will

be made emphasizing the lack of temperature control and the fact that Ford cups should only be used with Newtonian or near Newtonian liquids. The long-awaited precision statement will be completed and included in the method.

Group 20, Rotational Viscometers, D.W. Howard, Chairman, reviewed D 2196, "Test for Viscosity Measurements and Thixotropic Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer," which had just passed a subcommittee ballot. Comments from the Editorial Committee have been incorporated and the method now will be submitted for D-1 balloting. The group also discussed the possibility of writing a method for the Brookfield Small Sample Adapter which changes the normal Brookfield viscometer into a concentric cylinder viscometer with defined shear rate, good specimen temperature control, and significant reduction of waste material. Since the device is not yet widely used in the paint industry, it was decided not to consider a method for it at this time.

Group 21, Stormer Viscometer, M.P. Morse, Chairman, discussed the revised version of D 562, "Consistency of Paints by Stormer Viscometer," which was on the last subcommittee and D-1 ballots. A negative was withdrawn since most of the requested editorial changes had been made and a question on the precision statement had been cleared up. This method now is ready for Society ballot.

Group 23, High Shear Viscosity Measurements, considered the proposed method for "Determination of Viscosity of Paints and Varnishes at a High Rate of Shear by the ICI Cone/Plate Viscometer" which was on the last subcommittee ballot. One negative (which was due to the lack of a precision statement) was withdrawn on the understanding that the data for a statement was being collected and that a completed statement would be added to the method before D-1 balloting. Editorial changes suggested by others will be made in the method as well.

Group 25, Surface Tension, met for the first time. This task group has been organized for the purpose of surveying surface tension methods and choosing one or more to write up as ASTM methods for determining the surface tensions of paints and related materials. Several methods were discussed, namely the ring-pull method (DuNoüy ring), the Wilhelmy plate, the drop-weight method (as used and described by Patton), and Euerhard's bubble length method. These methods will be tested and compared with typical paints and the results reported at the June 1981 D-1 meeting.

Other items of discussion included

D 3539, "Evaporation Rates of Volatile Liquids," and D 1483, "Oil Absorption of Pigments by Gardner-Coleman Method." The former is up for reapproval. It will be submitted for concurrent subcommittee and D-1 ballots with only minor editorial changes (a change in the source for the Shell Thin Film Evaporometer and correction of typographical errors). The discussion of D 1483 centered around the confusing wording of the procedure. A member will rewrite the procedure to clarify the meaning.

SUBCOMMITTEE D01.26 OPTICAL PROPERTIES

C.J. Sherman, Chairman

Group 2, Color Measurement, J.G. Davidson, Chairman, reported that Method D1729, "Visual Evaluation of Color Differences of Opaque Materials," received no negative votes from the concurrent subcommittee and main committee letter ballot. However, the comments required revision of the method necessitating rebalancing by the subcommittee and main committee. The concurrent subcommittee and main committee ballot of Method D 3134, "Selecting and Defining Color and Gloss Tolerance of Opaque Surfaces and for Evaluating Conformance," received one negative vote at the subcommittee level. The negative was found persuasive and the method will be revised accordingly and submitted for concurrent subcommittee and main committee ballot. The latest draft of the proposed method, "Visual Evaluation of Metamerism," will be submitted for concurrent subcommittee and main committee letter ballot.

Group 6, Hiding Power, C.J. Sherman, Acting Chairman, did not hold a meeting at this session.

Group 11, Gloss and Goniophotometry, C.J. Sherman, Acting Chairman, reported that the proposed method, "Reflection Haze of High Gloss Surfaces," received only comments from the concurrent ballot. The comments were incorporated and the method will be sent to Society ballot. The proposed method, "Visual Evaluation of Gloss," received one negative from the subcommittee ballot. The method was revised to resolve the negative, a precision statement will be included and the method will be resubmitted to subcommittee ballot.

Group 19, Hemispherical Reflectance, K. Luyk, Chairman, reported that the latest revision of the proposed method, "Test for Total Luminous (Hemispherical) Reflectance," will include a precision

statement and be submitted for concurrent subcommittee and main committee letter ballot.

Group 23, Retroreflectance of Horizontal Coatings, N. Johnson, Chairman, reported that the proposed method, "Specific Luminance of Horizontal Coatings," will be submitted for concurrent subcommittee and main committee ballot. The possibility of publishing the geometry report by T. Bredahl in *Standardization News* is being investigated. A group was formed to evaluate portable instruments designed to test horizontal coatings for retroreflectance.

Group 24, Color and Strength of Color Pigments, C.J. Sherman, Acting Chairman, reported that Method D 387, "Color and Strength of Color Pigments," received only comments from the concurrent subcommittee and main committee ballot. The method was edited to include these editorial type comments and it was voted to send it to Society ballot.

General—A questionnaire to determine if there is interest and expertise in infrared reflecting and absorption coatings will be sent to D-1 members.

SUBCOMMITTEE D01.27 ACCELERATED TESTS FOR PROTECTIVE COATINGS

E.A. Praschan, Chairman

Negatives from the recent main committee letter ballot have been acted upon as follows: D 1540, "Effect of Staining Agents on Organic Finishes Used in the Transportation Industry"—the negative was deemed persuasive. It contained a number of editorial changes as well as suggestions for broadening the utility of the method beyond staining failures. It was agreed that D 1540 would be revised to include the suggested changes and then rebalanced. Comments from two other voters will also be considered in the revision. D 3361, "Practice on Operating Light and Water Exposure Apparatus (Unfiltered Carbon Arc Type) for Testing Paint, Varnish, Lacquer, and Related Products Using the Dew Cycle"—the negative was found persuasive. It was based on the fact that the unfiltered carbon arc produces light which does not exist at the earth's surface and that failure caused by this light may bear no relationship to failure in natural sunlight. The subcommittee agreed to add this precautionary statement to the significance section. The negative voter agreed with this action and when the change is made, the revised method will be placed on a main committee ballot. D 1150, "Single and Multiple Panel Forms for Recording Results of Exposure Tests of

Paints,"—a negative on the intent to withdraw D 1150 was received from the Committee on Standards. This negative was based on the fact that, although D-1 had ruled previous negatives from the main committee as nonpersuasive, the statement had been made that some other group might want to retain responsibility for this method. However, since the subcommittee could find no one who used this method nor who would assume responsibility, it was voted to continue withdrawal action.

The status of two methods were reviewed as follows: "Proposed Practice on Accelerated Outdoor Exposures" describes the use of black box, heated black box, and EMMAQUA for outdoor exposure was discussed. A second draft will be written incorporating comments and suggested changes and will be placed on subcommittee ballot prior to the June meeting. D 1006, "Conducting Exterior Exposure Tests of Paints on Wood" is overdue for reapproval. It has been reviewed and will be placed on concurrent subcommittee and main committee ballot for reapproval.

Group 2, Water Tests, G. Grossman, Chairman, will conduct a preliminary round robin to compare various water test methods as shown in D 870, "Immersion"; D 1735, "Water Fog"; and D 2247, "Relative Humidity." The results of the round robin will be used to provide precision data for the methods as well as to compare the procedures.

Group 4, Light and Water Exposure Apparatus, S. Totty, Chairman, presented preliminary findings of a group round robin comparing the standard cycle (102 min light/18 min light with spray) to a cycle of 4 hr light/4 hr dark with spray in a XW Weatherometer.® Using Florida black box panels (six and 12 months) as controls and comparing by Spearman Rank Coefficients, the standard cycle correlated better in gloss loss but poorer than the test cycle in color change. After some discussion on how best to interpret the data, it was agreed to calculate correlation coefficients to determine if these will offer additional practical data. A negative on subcommittee ballot for D 3361, "Practice for Operating Light and Water Exposure Apparatus (Unfiltered Carbon Arc Type)—Dew Cycle," was found persuasive and the significance section was changed to incorporate precautionary wording. The practice will go to main committee ballot.

Group 6, Detergent Testing of Organic Finishes, H. Leister, Chairman, reviewed comments received on the recent main committee ballot for D 2248, "Detergent Resistance of Organic Finishes." Most of these comments were editorial in nature

and were accepted. Two comments require further clarification and will be discussed with the persons who made them. Future action of the group will be directed toward evaluating the effect of procedural deviations (used by some who conduct detergent tests) to determine if the method should be revised to incorporate such changes.

Group 9, Evaluation of Corroded Specimens, R. Williamson, Chairman, discussed a proposed editorial revision of D 1654 and an agreement was reached on the changes to be made. The newer version will be submitted for reprint. The chairman will contact H.A. Hipwood regarding minor differences between the ISO method of salt spray and ASTM B 117 to determine if future work by this group is appropriate.

Group 10, Accelerated Outdoor Weathering, M.P. Morse, Chairman, discussed some of the data on accelerated outdoor exposure tests which was presented at the symposium on "Regimens for Predicting Performance of Decorative and Protective Surfaces" on Jan. 21, 1981. This data was obtained from exposures made by Group 10 in a number of locations and on various types of exposure racks. Initially it had been hoped that the data collected could be used for making a precision statement in a proposed method for accelerated outdoor weathering. However, there was such significant variations in results that could not be explained that no meaningful precision statement can be made. A first draft of a proposed practice for accelerated outdoor weathering was reviewed by the group. Suggested revisions will be incorporated and the revised version will be placed on subcommittee letter ballot.

Group 14, Filiform Corrosion, R. De Graaff, Chairman, reviewed Method D 2803 and discussed editorial changes. The possibility of including an ISO method (4623) for filiform was reviewed and the group decided to include this ISO method as an alternate method in a revision of D 2803. It will then be submitted for subcommittee ballot.

Group 16, Chalking, J. Robins, Chairman, presented an initial draft of a proposed new chalking method incorporating procedures commonly used in the industry. Comments were requested from the group and a draft will be submitted to the editorial subcommittee for comment before the proposed method is submitted for subcommittee ballot.

Group 17, Evaluation of Weathering Effects, D. Gleason, Chairman, reviewed several methods up for reapproval. It was agreed that the chairman will make edi-

torial revisions to methods D 660 (Checking), D 661 (Cracking), D 662 (Erosion), D 772 (Flaking) and submit them to ballot. These methods are all similar and contain pictorial standards depicting various degrees of failure. It was felt by the group that the methods (particularly the illustrations) are outdated and show failures limited to very specific finish systems. Contacts will be made with Sub. D01.25, Pictorial Standards, to determine if a cooperative effort can be established to update and improve these methods. Method D 1540, "Effect of Staining Agents on Organic Finishes Used in Transportation Industry," will be revised to incorporate the changes suggested in a negative vote. Other comments will also be considered and D 1540 will then be rebalotted. The negative vote on withdrawal of D 1150, "Report Forms for Exposure Results," was discussed. This negative came from a member of the Committee on Standards after Society ballot. After full consideration of alternate actions, the group felt that withdrawal plans should be pursued.

SUBCOMMITTEE D01.28 BIODETERIORATION

D.L. Campbell, Chairman

Group 1, Package Stability, W.B. Woods, Chairman, reported that an inoculum protocol based on the acclimatization of four bacterial isolates to an acrylic latex paint has produced the most consistent failure in unpreserved controls in a recent round robin study. To determine the applicability of this approach to other paint systems, additional paints (initially a PVA system) will be challenged by those laboratories participating in the next round robin. This acclimatization protocol will be followed, and it is expected that this study will be completed by the time of the next meeting of Sub. D01.28 in June 1981. Based on the results of that study, an expanded effort involving several paint systems will be considered. The development of a freeze-dried inoculum and the use of rapid biomass detection were discussed as possible future refinements of the method.

Group 2, Rapid Determination of Enzyme Presence, D. Massouda, Chairman, discussed a colorimetric quality control test for enzyme contamination. This test requires that a paint sample be centrifuged prior to the analysis of the supernatant liquid. The test requires three to four days to complete. The use of glucose detecting test strips is apparently inapplicable as the concentration of glucose in a paint is below the detection limits of the test strip. Had this test been

applicable, it would have offered the paint manufacturer a fast and simple means of determining the presence of enzymes as glucose is liberated as the cellulosic thickener decomposes. It was explained that the concentration of an enzyme in a paint may be as low as 100 ppb at the time an analysis is done on a contaminated paint. And, even if the chains in the cellulosic thickener are "cut" only once by an enzyme, a significant loss in viscosity is observed for the paint affected. At present, there is no direct method for determining the enzyme content of a paint. Only the indirect method of observing the effect of an enzyme on the viscosity of paints is available. While this method may not be useful in preventing or correcting a loss in viscosity for a plant batch, it is quite valuable in determining the cause of a viscosity loss in a given batch of paint. Therefore, it can be most useful in reducing future viscosity (and financial) losses due to enzyme activity. There are methods available to differentiate between redox and enzymatic viscosity losses in paints. A paint showing a viscosity loss can be added to a polyacrylate solution and to a cellulosic solution. If the problem is one of redox, both solutions will show a viscosity loss. But, only the cellulosic solution will show a viscosity loss if the problem was due to enzymes. Paints will be supplied for additional work with the described test method. The test method, as currently constituted, will be circulated to subcommittee members.

Group 3, Accelerated Tests, K.A. Haagensohn, Chairman, advised that the accelerated portion of the current study is complete, and the results of that study were reported previously. The secondary phase of the study was discussed. Panels painted with the coatings used in the accelerated portion of the study will be exposed in several states and Puerto Rico. Preliminary results from the exterior exposure study may be available for discussion at the next meeting in June 1981. As D 3273 is to be reviewed this year, it was agreed that a consolidation of the interior mold test method (D 3273) with that of a proposed exterior test method be attempted. A draft of this test method is to be available for discussion at the next meeting.

Group 4, Algae Resistance of Paint Films, W.B. Woods, Chairman, agreed to revise D 3274, "Evaluating Degree of Surface Disfigurement of Paint Films by Fungal Growth or Soil and Dirt Accumulation," to include photographs of algal defacement of coating films. These photographs should aid in differentiating between dirt accumulation and biological growth on coating films, particularly algae proliferate. Several photographs of

paint films disfigured by algae were examined by the group. Additional photographs are to be made from which publication quality standards can be selected. A revision of D 3274 text will be made to accommodate the algae photographs. The draft of this revision is to be circulated within the group in February or March, 1981. Accelerated tests for determining the susceptibility of paint to algal growth were discussed. An agar plate procedure will be circulated within the task group for review prior to the June 1981 meeting.

Group 5, Recoating Mildewed Surfaces, A.B. Juby, Chairman, discussed the first draft of the "Standard Guide for Determining the Presence of Mold and Removing Fungal Growth on Paint and Related Coatings." This draft is based on answers to a questionnaire submitted to D-1 members. The terminology, *Paint and Related Coatings*, is used in the appropriate locations in the draft instead of *Paint* in order to conform to the scope of D-1. A caution statement will be placed in the scope that will warn the user that removal procedures recommended may not be applicable to all coated surfaces. Three types of identification of the fungal growth were presented. Visual and bleaching procedures were accepted by the group. A procedure for subculturing the fungus or mold will be submitted. The method decided upon for the removal of fungal growth is as follows: Initial washing of the surface of the coating to be done with a solution consisting of 1 part 5% sodium hypochlorite (common household bleach) and 4 parts water. A second wash is to be done using an appropriate detergent solution suitable for use on a coating surface. Finally the surface of the coating is thoroughly rinsed with water to remove residual detergent. A caution statement will be included to indicate proper safety precautions required when using a bleach or detergent solution in this application. An appropriate detergent wash solution of 5% trisodium phosphate will be mentioned in a footnote. The question of a phosphate detergents' ability to function as a mold/fungal growth promoter was tabled. A second draft of the proposed guide will be submitted to subcommittee ballot. Results of the ballot will be discussed at the June 1981 meeting.

SUBCOMMITTEE D01.29 SUBSTRATE PREPARATION FOR COATING TESTING

S.D. Ozenich, Chairman

Two comments were received on D 2201, "Preparation of Hot Dipped Non-

passivated Galvanized Steel Panels for Testing Paint, Varnish, Lacquer and Related Products," which was submitted for reapproval. One comment was on the need to round off several items in the method. This was ruled persuasive. The other comment was that a document section should be included. A finalized copy will be submitted for inclusion on the next Society ballot. D 358, "Wood Panels Used for Weathering Tests of Paints and Varnishes," will be reviewed and discussed at the June 1981 meeting and submitted for concurrent subcommittee and main committee ballot for reapproval.

Group 1, Sandblasted Steel Panels, H.W. Leister, Chairman, was formed to develop a "Practice for Preparing Sandblasted Steel Panels for Coatings Testing." The purpose was discussed at length and the chairman will prepare an initial draft with title and scope for discussion at the June 1981 meeting. Some of the areas to be addressed in the establishment of the practice are: (a) type(s) of steel, (b) size and type of sand, (c) thickness of the panels, (d) precleaning necessary, (e) use and storage (how long), (f) profile measurement, and (g) inclusion of SSPC references. Members were asked to outline their thoughts on these points and the results would be sent to members of Sub. D01.23, D01.27, D01.29, and D01.46 for their comments.

DIVISION 30 PAINT MATERIALS

SUBCOMMITTEE D01.31 PIGMENT SPECIFICATIONS

C.W. Fuller, Chairman

The following actions were taken on negatives received on recent letter ballots: D 769, "Black Iron Oxide Pigment"—the negative was withdrawn because changes that the voter requested had already been made. It is now ready for Society ballot. D 963, "Copper Phthalocyanine Blue"—the negative contained nine parts. All were reviewed and, with the exception of the change in definition and the elimination of the pictorial illustrations of flocculation, were accepted. This standard lists an obsolete vehicle. A new short oil alkyd will be tried so this standard will be returned to the task group chairman. After revision, D 963 will be resubmitted for a concurrent subcommittee and main committee

ballot. D 153, "Specific Gravity of Pigments"—the negative referred to changes that were recommended as far back as 1975 and agreed upon according to prior minutes yet have not been incorporated. Since this negative was not received until this meeting, it was not possible to review the details of the comments. It will be reviewed later and resolved through further contacts with the voter. Editorial comments made by another voter were reviewed and incorporated into the method. As soon as the negative is resolved, this method will be submitted for Society ballot. D 1366, "Particle Size Characteristics of Pigments"—this negative contained seven parts. All parts were reviewed. Changing the term from "dispersion" to "scatter" was found to be confusing since this term has other meanings. The other parts were found to be persuasive and these changes will be made and the standard submitted for Society ballot. Terminology—A general editorial comment was made regarding addition of the word "Pigment" in the title of any standard that covers a pigment. It was agreed that this addition might facilitate computer retrieval and the word "pigment" will be added to the standard whenever it is appropriate.

The following specifications were reviewed in line with numerous editorial comments received during balloting: D 263, "Chrome Oxide Pigment"—a comment requesting changes in chemical and physical properties to comply with DIS 4621 was rejected. It is ready for Society ballot. D 267, "Gold Bronze Pigment"—changes suggested were made and the revised specification is ready for Society ballot. D 478, "Zinc Yellow (Zinc Chromate) Pigment"—editorial changes were made and the revised specification is ready for Society ballot. D 262, "Ultramarine Blue Pigment"—minor editorial changes were made and ISO-788 will be added under applicable documents. It is now ready for Society ballot. D 209, "Lampblack Pigment"—ready for Society ballot. D 85, "Ochre Pigments"—ready for Society ballot. D 656, "Pure Toluidine Toner"—paste in oil retained and D 1208 added. It is ready for Society ballot. D 602, "Barium Sulfate Pigments"—editorial comment regarding the present low pH as being indicative of low purity is not technically correct. Bleached barytes requires strong acids which after washing leaves material slightly acidic. Addition of alkaline materials to raise pH would lower purity. ISO-3262 will be added to applicable documents and the revised specification is ready for Society ballot. D 604, "Diatomaceous Silica Pigment"—editorial comments were reviewed. ISO-3262 will be added to applicable documents. The addition of Maritime 52M A-522 to this specification was referred to the task

group responsible for this specification and will be considered for inclusion on the next review. It is ready for Society ballot. D 607, "Wet Ground Mica Pigment"—Method D 3360 will be deleted. Request to add ISO-3262 was accepted. Comment to add 0.5% water solubles to properties table was referred to task group for review and possible inclusion on next review. It is ready for Society ballot. D 261, "Iron Blue Pigment"—ISO-2495 will be added to applicable documents and it is ready for Society ballot. D 1648, "Basic Lead Silico Chromate"—the safety statement included in the scope was modified to state this material may pose a safety or health hazard if improperly handled. This specification will be rebalotted concurrently by a subcommittee and main committee ballot. D 763, "Raw and Burnt Umber Pigment"—Society ballot completed and proof copy reviewed. D 768, "Yellow Iron Oxide"—Society ballot completed and minor editorial changes made on proof copy. D 867, "Pumice Pigment"—editorial change rejected and ready for Society ballot. D 210, "Bone Black Pigment"—the applicability of Method D 215 was verified and left in method.

Specifications up for review in 1981 are: D 561, "Carbon Black"; D 1649, "Strontium Chromate"; and D 3619, "Aluminum Silicate Pigments." These will be reviewed by appropriate task group chairman.

SUBCOMMITTEE D 01.33 VARNISH & RESINS, INCLUDING SHELLAC

W.C. Golton, Chairman

Group 1, Varnishes, H.A. Ball, Chairman, reviewed standards due for revision in 1981. D 479, "Test for Reactivity of Paint Liquids," was approved for withdrawal without any negatives. One negative was received on withdrawal of D 1643, "Test for Gas Checking and Draft Resistance of Varnishes." The negative was persuasive but remains unresolved pending another ballot. Editorial revisions will be made to D 154, "Testing of Varnishes"; D 1639, "Test for Acid Value of Organic Coating Materials"; and D 1647, "Test for Resistance to Water and Alkali of Dried Film." They will then be submitted for ballot.

Group 6, Drying Time, H.M. Werner, Chairman, reported that a poll of subcommittee members indicated little interest in developing methods for mechanical time determination. A proposed revision of D 1640, "Tests for Drying, Curing or Film Formation of Organic Coatings at Room Temperature," rela-

tive to determination of print free time will be balloted within the subcommittee.

Group 10, Film Properties, J.C. Weaver, Chairman, reported that D 1546, "Performance Tests of Clear Floor Sealers"; D 1641, "Test for Exterior Durability of Varnishes"; and D 1642, "Tests for Elasticity or Toughness of Varnishes"; passed both subcommittee and main committee ballots. They will now be submitted for Society ballot. It was recommended that control of D 1546 and D 1641 be transferred to Sub. D 01.42 on Architectural Paints and D 1642 referred for review of need to Sub. D 01.07 on Government Contacts.

Group 14, Alkyds, W.C. Golton, Acting Chairman, reported that four methods were reaproved. Editorial suggestions were incorporated and the edited documents will be sent for Society ballot. These methods were: D 1615, "Test for Glycerol, Ethylene Glycol and Pentaerythritol in Alkyd Resins"; D 2455, "Identification of Carboxylic Acids in Alkyd Resins"; D 2456, "Identification of Polyhydric Alcohols in Alkyd Resins"; and D 2998, "Determination of Polyhydric Alcohols in Alkyd Resins." Two companies will submit NMR methods on analysis of alkyds for consideration at the next meeting.

Group 15, Nonvolatile Determination, A.C. Abbott, Chairman, agreed to review D 1259, "Nonvolatile Content of Resin Solutions." If found necessary, a round robin will be conducted for both nonheat reactive and heat reactive resins. A round robin will be completed on D 1644, "Nonvolatile Content of Varnish," with the hope of incorporating this method into D 2369, "Volatile Content of Solvent Reducible Paint," by expanding the scope of D 2369 to include varnish.

Group 18, Liquid Properties of Resins, W.C. Golton, Acting Chairman, reported that D 1469, "Total Resin Acids Content of Coating Vehicles"; D 1542, "Qualitative Test for Rosin in Varnishes"; and D 2090, "Test for Clarity and Clearness of Paint Liquids," were reaproved with editorial revisions. A negative on D 1545, "Test Method for the Viscosity of Transparent Liquids—Bubble Time," was voted nonpersuasive on the grounds that however old, this method is used daily around the world and should be neither deleted or replaced. A negative on D 1725, "Viscosity of Resin Solutions," was found nonpersuasive because this method is a preparative step for the widely used D 1545. It was recommended that D 1545 and D 1725 be combined. The task group also recommended modernizing D 2090 and updating D 3132, "Solubility Range of Resins and Polymers," using a panel of experts.

Group 20, Shellac, W.C. Golton, Acting Chairman, reported the following results from recent subcommittee and main committee ballots: D 29, "Sampling and Testing Lacquer Resins," was approved with editorial changes; D 207, Specification for Dried Bleach Lacquer," was reapproved without comment; D 237, "Specification for Dried Bleach Lacquer," reapproved without comment; D 1650, "Sampling and Testing Shellac Varnish," was reapproved with editorial changes. It was recommended that the suggested editorial revisions be made and the edited documents be forwarded for Society ballot. D 360, "Specification for Shellac Varnishes," was supposed to have been revised and balloted but this still remains to be done.

Group 25, Poly(Vinyl Chloride) and Poly(Vinyl Butyral), J. Brezinshi, Chairman, reported that D 1396, "Chemical Analysis of Poly(Vinyl Butyral)" had passed both subcommittee and main committee ballots without negatives. A hazard warning statement will be added and the revised document forwarded for Society ballot. A negative received on D 1156, "Test for Total Chlorine in Poly(Vinyl Chloride) Polymers and Copolymers Used for Surface Coatings," was withdrawn. The task group will prepare a draft of a "Practice on Methods for Analysis of Poly(Vinyl Chloride) and Its Copolymers," and circulate it for comments.

Group 23, Epoxy and Phenolic Resins, P. Kamarchik, Chairman, reported that D 1312, "Test for Apparent Free Phenols in Synthetic Phenolics or Solutions Used for Coating Purposes," was reapproved. Editorial changes will be made and the revised document forwarded for Society ballot. A round robin is being conducted on determination of total chlorine in epoxy resins. Practice methods are being developed. A new method for "Acid Value Determination of Phenolic Resins" was proposed for total group consideration.

Group 24, Nitrogen Resins, J. Smith, Chairman, reported that D 1198, "Test for Solvent Tolerance of Amino Resins," and D 1597, "Test for Melamine Content of Nitrogen Resins," did not receive any negative votes on subcommittee and main committee ballots. Editorial changes will be made to D 1198. A precautionary statement will be added to D 1013, "Test for Total Nitrogen in Resins and Plastics," and it will be submitted for main committee ballot. A proposed practice for testing of nitrogen resins will be presented at the June meeting. Alternative procedures to D 1259, "Test for Nonvolatile Content of Resin Solutions," and D 1198, "Solvent Tolerance," are being prepared.

Group 26, Polymer Emulsions, O. Brown, Chairman, reported that the revised draft for the proposed method for nonvolatile content will be submitted for the subcommittee ballot. A proposed "Practice for Testing Latices" will also be submitted for subcommittee ballot. The variability of the round robin results of the new "Standard Test Method for Assessing Elevated Temperature and Freeze-Thaw Stability of Latices" appears to be caused by the inaccuracy of the viscosity measurement. This problem will be resolved before continuing round robin testing.

SUBCOMMITTEE D01.35 SOLVENTS, PLASTICIZERS AND CHEMICAL INTERMEDIATES

L.R. Thurman, Chairman

All negatives received on subcommittee letter ballot were resolved at the meeting and withdrawn. Eighteen standards were approved for main committee ballot as follows: D 329 on "Acetone"; D 331 on "2-Ethoxyethanol"; D 1363 on "Permanganate Time of Acetone and Methanol"; D 1399 on "Unsaponifiable Content of Tricresyl Phosphate"; D 1613 on "Acidity in Volatile Solvents and Chemical Intermediates"; D 1617 on "Ester Value of Solvents and Thinners"; D 1718 on "Isobutyl Acetate (95% grade)"; D 1719 on "Isobutyl Alcohol"; D 2634 on "Methyl Amyl Acetate"; D 2917 on "Methyl Isoamyl Ketone"; D 2999 on "Mono pentaerythritol in Commercial Pentaerythritol"; D 3130 on "n-Propyl Acetate (90 to 92% grade)"; D3540 on "Primary Amyl Acetate, Synthetic (98% grade)"; D 3541 on "2-Ethylhexyl Acrylate"; D 3545 on "Alcohol Content and Purity of Acetate Esters by Gas Chromatography"; D 3546 on "Formic Acid in Glacial Acetic Acid"; D 3547 on "n-Butyl Acrylate"; and D 3548 on "Ethyl Acrylate." One standard, D 554 on "Amyl Acetate made from Fusel Oil (85 to 88% grade)," is recommended for withdrawal because the product is no longer available.

Group 1, Hydrocarbon Solvents, S.A. Yuhas, Chairman, recommended a subcommittee ballot on a revision of D 235, "Mineral Spirits," which will incorporate D 484 on "Hydrocarbon Dry Cleaning Solvents" and subsequent withdrawal of D 484. Also the withdrawal of D 363 on "Industrial Grade Xylene" and D 1616 on "Copper Corrosion by Mineral Spirits" is recommended for subcommittee ballot. Round robin test data justifies the submission of a modified D 3606 test method for the determination of benzene in hydrocarbon solvents by gas chroma-

tography to subcommittee ballot. Four standards were issued for review.

Group 2, Oxygenated Solvents, T.H. Golson, Chairman, will continue study of low water analysis in urethane solvents by gas chromatography. Several negatives were received on the subcommittee ballot on the proposed standard for combining the ketone specifications. The proposed standard is being revised. Twelve standards were assigned for review as well as a proposed specification for methyl n-amylyl ketone. At the request of Committee D-1, Standard D 3128 on "Ethylene Glycol Monomethyl Ether" will be reviewed.

Group 3, Chemical Intermediates, J.M. Kauffman, Chairman, will continue study of a new proposed specification for glacial acrylic acid. A proposed specification for 2-ethylhexyl alcohol is being reviewed to determine if an assay method by gas chromatography should be included. A "sulfuric acid color test" proposed by Committee E-15 will be reviewed for addition to the 2-ethylhexyl alcohol document.

DIVISION 40 PAINT PRODUCTS APPLIED ON SITE

SUBCOMMITTEE D01.41 CRITERIA FOR THE PURCHASE OF COMMERCIAL PAINTS AND COATINGS

R.A. Brown, Chairman

Group 1, J. Csernica, Chairman, is presently concerned with a round robin test program which has been conducted on latex flat wall paints. This test program involved testing 12 paints by nine cooperating laboratories. The paints were tested for opacity, gloss, washability and scubbability. The samples included nine first line commercial paints plus the latex flat paints now purchased by Virginia, Florida, and GSA (TT-P-29). Three members had met to review the results of the test program. It was reported that contrast ratio (opacity) was generally satisfactory on all paints. The gloss was acceptable on all but three which were too high. Results reported by the cooperative laboratories on washability (D 3450) and scubbability (D 2486) varied widely. There was poor re-

producibility of test results among laboratories and apparently wide differences in the washability properties of the paints. Several laboratories reported considerable differences in reflectance values for the paints although all were marketed as "white." It was stated that GSA will probably specify an acceptable range of reflectance when a Commercial Item Description (CID) is written for white latex flat wall paints. Although more work is necessary before GSA can write a CID for the purchase of commercial latex flat wall paints, and the cooperating laboratories will be asked to run a few more tests, it was agreed that significant progress had been made toward this objective.

Group 2, W.V. Moseley, Chairman, reported that draft #10 of the "Standard Guide for State and Institutional Purchasing of Paint" has been fully approved and is being printed as an ASTM Standard with the designation D 3927. It was reported that this guide is now being used by the States of North Carolina, South Carolina, Georgia, and Virginia and will be considered for use in Florida soon. Questions were answered about how the guide is used in Virginia which is pleased with the results obtained to date.

SUBCOMMITTEE D01.42 ARCHITECTURAL PAINTS

R.S. Armstrong, Chairman

Group 2, Practical Stain Removal, has just started to develop a test method to evaluate this property. A round robin was conducted prior to the meeting in which an attempt was made to remove a number of different common household stains from several different paints with three different cleaning materials. The results of the test were discussed. One of the cleaning materials will be changed, different methods of applying the stains will be evaluated and some of the stains will be changed to get more representative stains. Another round robin is planned.

Group 3, Leveling, has submitted a test method to evaluate leveling by draw-down to subcommittee and main committee ballot. This group is now starting to develop a method to evaluate leveling when a paint is applied with a brush. An attempt will be made to correlate this method with the evaluation of leveling with a draw-down bar. A round robin is planned prior to the next meeting.

Group 7, Sag Resistance, is attempting to develop a method to evaluate sag resistance by the use of a draw-down bar. The group will also attempt to correlate

this method with evaluation of sag when paints are applied with brush or roller. Various devices and bars used by the paint industry to evaluate sag resistance will be evaluated. A round robin is planned.

Group 9, Color Compatibility, developed a method to evaluate color compatibility and submitted the method to Sub. D01.42 for vote. The method consisted of applying tinted test paint at two different shear rates and measuring any difference in reflectance between the two areas as an indication of color nonuniformity. Many members commented, however, that reflectance readings are not valid in measuring color differences and it was decided to use McAdam Color Difference Units, DE, instead. New precision data will need to be generated before this method can again be submitted for ballot since the previous data are no longer valid.

Group 13, Brushability, is starting to develop a method to evaluate brushability. An attempt will be made to correlate this method with the evaluation of high shear viscosity by means of an instrument, probably the ICI instrument.

Group 17, Roller Spatter, is attempting to develop a method to measure roller spatter with a special notched spool. Reproducibility within a lab and ranking of paints from lab to lab is satisfactory but correlation of the roller spatter pattern from one lab to another is not good. Paint was applied during the meeting according to this method by various members and differences in techniques were observed. Another round robin will be conducted to reduce the variables of the method.

Group 16, Opacity (Practical Methods), is attempting to develop a method to measure practical opacity. It was agreed that paint should be applied to a large area with a brush or roller but a suitable substrate has not been selected. Large paper charts with alternate white and gray stripes (of various reflectance) have been considered as a substrate but most group members feel that the charts are too expensive and that gray stripes of various reflectance be used without the white stripes between the gray ones. An attempt will be made to have this type of test surface prepared on roll paper to be used as test charts.

General: Three methods were submitted for subcommittee ballot. Two, D 2243, "Freeze-Thaw Resistance," and D 2354, "Minimum Film Formation Temperature," were revisions. D 2801 on "Leveling" was up for reapproval without change. A negative was cast on D 2354. In section 5.2 of this method, the rate of flow of the dry air or nitrogen

was specified but this was not included in the method as it was submitted for ballot. The comment with the negative stated that this rate of flow was critical and should again be included in the method. It was agreed that this comment was persuasive and the method will be revised accordingly. Since the deletion was not approved by the subcommittee and it could not be determined why the deletion was made, this portion will be added again as an editorial change. A negative vote was cast on D 2801, "Leveling." The comment with the vote was that the figure for the leveling blade was incorrect in a number of ways. It was agreed that this comment was persuasive. The correct figure with correct dimensions will be shown and the method will be resubmitted for subcommittee ballot. Comments were received on all three items. These comments were discussed and most of them will be incorporated into the methods.

SUBCOMMITTEE D01.44 TRAFFIC COATINGS

W.V. Moseley, Chairman

Group 1, Thermoplastics, D.R. Miller, Chairman, reported that round robins are underway on bond strength, reflectance, and binder, and bead content. Two new round robins were initiated on abrasion resistance, reflectance, and viscosity.

Group 2, Traffic Paints, E.B. Countryman, Chairman, reported that round robins will be initiated for D 711, "Dry To No Pick-Up Time," and another set of tests will be run to determine the reproducibility of D 1309 and D 869 on settling properties. A round robin will be conducted on D 868 and D 969 on bleeding to develop a precision statement.

Group 3, Night Visibility, J. Ritter, Chairman, received a report on progress of research of mobility systems for measuring retroreflectance of horizontal traffic markings. A number of questions to be answered in evaluating the reflectometer were discussed. Round robins will be initiated on glass bead roundness, crushing strength, and gradation to develop precision statements.

Group 4, Catalyzed Coatings for Pavement Markings, D.R. Miller, Chairman, reviewed 15 test methods discussed at the previous meeting. It was the consensus of the group to continue this investigation and to see if any methods presently being used for other pavement markings would be applicable.

Group 5, Water-Borne Coatings for Pavement Markings, E.B. Countryman,

Chairman, will contact representatives from GSA and California, who have done the most work to date on water-borne coatings, to see if they want this task group to work on any specific tests.

Group 6, Bead Retention of Traffic Marking Compounds, E.B. Kelch, Chairman, was formed to initially study a method of measuring bead retention from Virginia Department of Highways and Transportation.

General: A new sub task group under D01.44.02 was formed to work on a proposed method for determination of chlorinated rubber in highly pigmented alkyd-containing chlorinated rubber finishes. C.M. Winchester will chair this task group. Because of late receipt of negatives and comments on all concurrent letter ballots from one voter, action could not be completed at this meeting. Resolution will be deferred until the June 1981 meeting. It was also decided that the June 1981 meeting will be held with Committee D-1 and Not D-4 as previously planned.

SUBCOMMITTEE D01.45 MARINE COATINGS

L.S. Birnbaum, Chairman

The chairman reported on a number of actions since the last meeting: (a) a letter soliciting the support of subcommittee members brought a positive result; (b) a letter was sent to the Naval Ship Research and Development Center at Annapolis, Md., soliciting their cooperation and participation in the program to revise the antifouling rating system prescribed by Method D 3628. NSRDC agreed to supply samples of the MIL-P 24441 epoxy anticorrosive paints required for panel preparation in the program; (c) a letter was sent to the Naval Sea Systems Command suggesting that they arrange for exposure of panels at their Pearl Harbor test site and that they also consider rating of antifouling paints which they would test for qualification under MIL-P-0015931D in accordance with the proposed ASTM scheme and make results available (without identifying suppliers) to ASTM for statistical analysis. NAVSEA advised that this could probably be worked out and that they would initiate action accordingly; (d) a number of paint suppliers were contacted and agreed to provide paint samples of MIL-P-0015931D formula 121 vinyl antifouling paint required for preparation of exposure panels to be used to develop the statistical data for precision and reliability of the proposed new rating scheme; (e) the proposed new antifouling system was circulated to permit review and comment prior to the January meeting.

Group 5, Algae Control, C.D. Stevens, Chairman, discussed a proposed program and it was agreed that a description of suggested approaches and existing techniques should be forwarded to the secretary. Responses will be circulated prior to the next meeting.

Group 6, Dynamic Testing, A. Freeman, Chairman, was formed in 1977 to evaluate the merits of existing dynamic tests for screening candidate coatings for underwater applications. Following a review of past efforts, it was agreed that this objective was still valid and the next step should be the test of two paint systems on at least two rotating drums. Miami Marine Research and Battelle Florida Marine Research Facility in Daytona Beach offered to accommodate two systems on their apparatus. Naval Ship Research and Development Center offered to look into the possibility of evaluating these same systems at the Pearl Harbor facility. Accordingly, a total of 12 drum panels (four per set) for each system would be required. It was decided that one of the cuprous oxide vinyl AF systems would be selected from the Group 7 project for this purpose and panels would be prepared for all three sites. It was further decided that the second system should be a chlorinated rubber organo-tin AF system to be selected from three candidate proprietary AF systems. Twelve panels will be prepared for testing.

Group 7, Antifouling Rating, C. Perez, Chairman, reviewed the Miami Marine Research "Explanation of Behavior Report" proposed as a substitute for the current scheme. It was decided that there should be an observation made on bacterial slime and/or silt as well as on algae spores. Further discussion was deferred in order to discuss the actions required for preparation of panels for exposure. It was agreed that Miami Marine Research would prepare the epoxy AC/vinyl AF system panels and that Hercules would prepare the chlorinated rubber AC/vinyl AF panels for static exposure. In addition, Hercules agreed to prepare chlorinated rubber AC/chlorinated rubber AF panels for static exposure as an additional control for the dynamic exposures. Battelle agreed to check out the use of their facilities as a third site for static exposure of these systems. Because of the number of panels that would probably be required for each system and the burden on exposure site members, it was decided to use only the standard formula 121 (non-exempt solvent version) to be obtained from six suppliers. It was agreed that the Chairman would prepare a proposed matrix of paint systems, exposure sites, and panel replication to be sent to co-

operators for concurrence with a target date sometime in March for preparation of panels.

SUBCOMMITTEE D01.46 INDUSTRIAL PROTECTIVE PAINTING

D.M. Berger, Chairman

At the October meeting of the Executive Subcommittee, it was agreed to set up an Ad Hoc Committee on Zinc Rich Coatings to act on the request to transfer Group 9, Zinc Rich, into a new subcommittee. The ad hoc group met to consider actions required for approval of this request. It was decided the title should remain as is and the scope would cover development of standards, guides, and methods of test for zinc rich and other galvanic protective coatings. Task groups proposed were as follows: Group 2 on Determination of Cure; Group 3 on Laboratory Evaluation; Group 4 on Film Integrity and Solids by Volume; and Group 5 on Top Coating. Officers proposed were M. Tellor as Chairman, S. Mark as Vice Chairman, and E. Horvick as Secretary. These actions were approved by the Executive Sub. D01.90 and Chairman S.B. Levinson confirmed the appointment of M. Tellor as chairman. The first meeting of this group as Sub. D01.48 is scheduled for June 1981 in Providence, RI.

Group 2, Surface Preparation, reported that three standards on surface preparation for aluminum and magnesium, D 1730, D 1731, and D 1732 were sent to other subcommittees for editing.

Group 3, Field Applied Coatings, D. Nixon, Chairman, intends to develop a standard method for field identification of coating films. This field test method covers procedures and test equipment to be used for generic identification of dry surface coatings. The method will be written around a test kit developed by the Navy. Samples will be sent to six volunteers to evaluate the test method. The group will also develop a standard practice for selecting new coatings which are compatible and can be applied over existing coated surfaces. This practice will include generic identification, compatibility chart, integrity of existing coating, and evaluations using test patches.

Group 4, Pull-Off Adhesion Test, A. Cunningham, Acting Chairman, discussed the questionnaire sent to members. It covered the use of the Elcometer Adhesion Tester and other pull-off adhesion testers. A standard pull-off adhesion test method has been distributed for comment. A round robin test is to be conducted and the validity of scoring

through the coating around the test dolly evaluated.

Group 7, Inspection, revised D 3276, Guide for Paint Inspectors. All comments will be reviewed and the revised document submitted for ballot. Work has been initiated on the preparation of a standard practice for field measurement of "Surface Profile of Blast Cleaned Steel." This practice covers the description of various techniques used for measuring the profile of abrasive blast cleaned steel surfaces. This practice is intended as a guide for use in the field and/or shop and will not describe laboratory procedures. The techniques have been categorized as follows: *Comparative*—Keane-Tator Comparator, Clemtext Coupons, ISO and DIN; *Replica*—Testex and Casting Technique; *Stylus*—BSRA Gage, ANSI Method, Monotester, Bendix, and Gould; *Other*—Roughsector, Depth Micrometer, Microscope, (SSPC Method) and (Control). The standard practice will provide detail on the use of the Keane-Tator Comparator, Clemtext Coupons, Testex Tape, and Depth Micrometer. The SSPC microscope method will be described and used as a reference standard.

Group 10, Painting Galvanized Steel, E. Horvick, Chairman, discussed the development of a guide. A document has been written and will be passed around for review. Identification of the various types of galvanized surfaces and treatments will be included.

General: Method D 1014, "Conducting Exterior Exposure Tests of Paints on Steel," is being revised. The revision will include measurement of dry film thickness, blast cleaning, and the use of backed panels or the black box per ASTM G-7.

DIVISION 50 PAINTS FOR FACTORY APPLICATION

SUBCOMMITTEE D01.51 POWDER COATINGS

C. Grenko, Chairman

There were no negative votes requiring action. D 3451, "Standard Practice for Testing Polymeric Powders and Powder Coatings," has been reapproved in main committee ballot. A proposed method for resin color determination by the platinum cobalt method has been drafted and

is ready for subcommittee ballot. A proposed method for color determination of base resins using a Gardner tristimulus colorimeter has been drafted and is ready for subcommittee ballot. Melt viscosity determination of base resins using an ICI cone and plate apparatus is under study. Work has not progressed enough to draft a specification.

A questionnaire will be sent to subcommittee members seeking their opinions about future meetings.

SUBCOMMITTEE D01.52 FACTORY-COATED WOOD PRODUCTS

R.C. Marck, Chairman

Negative votes from the recent main committee ballot were resolved as follows: D 2830, "Test for Durability and Compatibility of Factory Primed Wood Products with Representative Finish Coats," received a negative vote, pointing out two omissions in the report section. These were corrected and judged editorial. D 2691, "Standard Methods for Microscopical Measurement of Dry Film Thickness of Coatings on Wood Products," received a negative concerning several editorial changes which were resolved. It also concerned errors in the precision statement which will require much time and effort to correct. It was agreed that the standard would be submitted for Society ballot with the corrected editorial changes and that the revised precision statement will be submitted for a later revision with the approval of the negative voter. D 2336, "Specifying Properties of Coatings from the Liquid Through the Curing Stage for Factory Applied Coatings on Wood Products," received editorial changes which were resolved as suggested.

Group B, Hardboard, J.J. Medica, Chairman, has established a new task force to study problems that are occurring to hardboard siding as a result of the use of certain new and low cost coatings. This task force will convene a special working session in early spring.

Group 11, Dirt Collection, J.J. Medica, Chairman, has completed work on D 3719, "Test for Dirt Collection on Coated Exterior Panels." With recent Society approval of the standard, the task group has been put on an inactive status.

Group 12, Textured Paneling, R.C. Marck, Chairman, has organized a round robin to further investigate the performance and durability of the finish on textured paneling using standard dirt and the Gardner Washability machine. Volunteers are solicited.

Group 52/55, UV Curing Coatings, J.M. Behrle, Acting Chairman, discussed a proposed method for "Determining Package Stability of Coatings for Ultraviolet Curing" which had been rewritten after the June 1980 meeting and submitted for letter ballot to both subcommittees. There were no negatives but seven of the affirmatives had comments. All were considered and most incorporated in the method. All were judged to be editorial in nature. The revised method will be submitted to concurrent subcommittee and main committee letter ballot.

SUBCOMMITTEE D01.53 FACTORY-COATED STRIP METAL

E.E. Haney, Chairman

Group 1, Formability, W.H. Gunn, Chairman, discussed the replies received from four voters on the second draft of the proposed test method for "Formability Characteristics." The changes and recommendations were reviewed and incorporated into the latest draft. A photographic standard was also added along with additional comments by subcommittee members. This latest revised draft will be submitted for subcommittee ballot. This task group will begin work on painted aluminum and zinc coated substrates using a modification of this biaxially stretched adhesion test to prepare methods for these substrates.

Group 2, Cure, K.E. Luyk, Chairman, discussed the results of the first round robin on the finger rub test method. The correlation indicated several problems, including the end point determination. Suggestions were made to modify the method and the round robin will be repeated.

Group 3, Pretreatments, R.L. Williamson, Chairman, discussed the results of the phosphate coating weight determinations on zinc surfaces. This method is now ready to be put in final form for subcommittee ballot. Chrome determination by the Portaspex method was discussed. Contact will be made to determine if liaison is needed with other ASTM committees concerning this method. A future round robin is scheduled for the development of a method to determine coating weights of zinc and iron phosphates on cold rolled steel.

Group 5, T-Bend Flexibility, E.E. Haney, Acting Chairman, reported on the status of the "Method for T-Bend Flexibility on Aluminum." At the last meeting suggestions were made that will be incorporated and the revised method will be submitted for subcommittee ballot.

Group 6, Drawdowns, R.W. Reinsel, Chairman, reported on the status of the most recent draft on the drawdown method. The need for a precision statement was discussed. After checking with the editorial subcommittee and considerable discussion, it was decided not feasible and not required for a standard practice. The draft will be corrected to include the editorial changes and submitted for subcommittee ballot.

General—Editorial comments were submitted by four voters on D 3003, "Pressure Mottling and Blocking Resistance of Organic Coatings on Metal Substrates," during a recent main committee ballot. All of the comments were considered and those that were persuasive were incorporated into the method. It was reported that Sub. D01.46 is releasing three methods for preparing test panels for painting on various substrates. It was felt that these methods were more in line with the activities of Sub. D01.29 and should be given to that subcommittee. Suggestions from the Executive Sub. D01.90 regarding attendance are being considered.

SUBCOMMITTEE D01.55 FACTORY COATED PREFORMED PRODUCTS

F.J. Steslow, Chairman

Six methods which had been recently balloted were discussed. These included D 333 on "Testing Lacquers," D 1308 on "Effect of Household Cleaners," D 2091 on "Print Resistance of Lacquers," D 2571 on "Practice for Wood Lacquers," D 2620 on "Light Stability of Clear Coatings," and D 3459 on "Humid-Dry Cycling for Coatings on Wood." One negative ballot was received on D 3459. This was judged nonpersuasive because the improved editorial style changed the test sequence and was expected to alter test results. Editorial comments from two voters were also discussed. They were considered constructive and will be incorporated.

Group 1, Coatings for Wood Products, M.E. Mull, Chairman, reviewed D 3002 and D 3023. On D 3002, "Practice for Evaluation of Coatings for Plastics," para 2.1 was modified to read: "Unless directed otherwise, performance tests shall be conducted on coatings of specified thickness applied to a normally molded sample of the designated plastic. Applied coatings cannot correct surface defects from improper molding procedures." Para 2.2, 9.1, 10.2, 11.2, 14.1, 15.1, and 16.1 were changed to replace the phrase "by the purchaser and the seller" to "by the producer and user."

Subnote (1) was changed to show that D 3002 was the responsibility of Sub. D01.55 not Sub. D01.53. On D3023, "Resistance of Factory-Applied Coatings on Wood Products to Stains and Reagents," under para 2 Reagents, etc. add, "V-H-L refers to product and use. See Paragraph (7)." In para 6.2 change 18 h to "18h exposure or a time agreeable to producer and user." In 6.3, change "After 18h exposure," to "after 18h exposure, or other time agreed to by producer and user," . . . etc. After subnote (2a) insert the following comment prior to subnote (3): "If any of the following products are not available, equivalent products agreeable to both producer and user, can be used." Standards up for reapproval in 1981 are D 2199 on "Plasticizer Migration from Vinyl Fabrics to Lacquers" and D 3732 on "Practice for Cure Times of Ultraviolet Cured Coatings."

Group 7, Water-Borne Industrial Coatings, F.J. Steslow, Acting Chairman, discussed in detail the first draft of the "Practice for Testing Industrial Water Reducible Coatings" and the 13 sections put together by various members. The entire draft was reviewed and the second draft will be updated by the end of February and distributed to subcommittee members for comments. After receipt and review of the comments, a new draft will be ready for distribution prior to the June 1981 meeting.

SUBCOMMITTEE D01.56 PRINTING INK

J.M. Fetsko, Chairman

Group 1, Viscosity by Falling Rod Viscometer, J.M. Fetsko, Chairman, reported during concurrent subcommittee and main committee balloting. One negative was withdrawn after discussion at the meeting. The second negative, having been submitted without any reason, was considered nonpersuasive. The third negative will be withdrawn if one paragraph is relocated; other comments from the same voter were considered nonpersuasive. Each point will be discussed with this voter and he will be requested to withdraw his negative. Editorial comments from two other voters suggested that Sections 5 and 6 be reorganized into one section entitled "Terminology" and that the precision statement contain additional information. Other comments involving definitions and language were considered. When all of the editorial changes have been made and the method reviewed by the subcommittees on editorial and definitions, it will be submitted for Society ballot.

Group 6, Ink Tack, C. Shepard, Chairman, reviewed the draft of the Inkometer

method along with suggested changes submitted by the task group and by attendees at a pre-meeting. As a result, the title will be changed from Relative Tack to Apparent Tack. Other technical and editorial revisions will also be made. Results of a previous round robin and of in-house studies will be used as the basis for the required precision statement. After a new draft is ready, efforts should be made to bring the draft of the Tack-O-Scope method into agreement insofar as possible. The two methods are to be balloted by the subcommittee before the June meeting.

Group 7, Smoke Test—Heat Set Inks, J. Coleman, Chairman, reported results of a questionnaire sent to 17 ink manufacturers inquiring about smoke test procedures in current use. Through the questionnaire and group contacts, seven companies making smoke tests were identified. Recognizing that the test procedures varied, a decision was made to supply four coded inks to all companies willing to evaluate them. The results of the round robin are expected to indicate which, if any, of the procedures in current use can be the basis for an ASTM standard.

Group 8, Tinting Strength, A.N. Scarlatti, Chairman, used Method D 387 as a guide for a new method applicable to printing inks. Copies showing suggested changes were circulated and discussed. Table 1 is to be updated and suggested changes by members will be incorporated in a second draft. It was learned that Group 24 of Sub. D01.26 has responsibility for D 387 and it was decided that representatives from this group would attend their meeting to determine whether a separate method is needed. The consensus is that different methods may be needed. At the same time, an instrumental method for evaluating results will be explored. Three members agreed to review Method D 3424, "Standard Method of Evaluating Light Fastness of Printed Matter," and report at the next meeting.

Other Business—Method D 2369 on "Volatile Organic Content" was reviewed by one of the members. It was his opinion that the procedure was not applicable to printing inks and that the method should include a statement to that effect. (Group D01.21.24 agreed at a subsequent meeting.) He will review available methods and report at the next meeting.

The problem of specifications for water emulsification based on arbitrary methods was discussed. It was suggested that workers in the field be invited to the next meeting to discuss test procedures and the correlation found between results and actual press performance.

SUBCOMMITTEE D01.57 ARTIST PAINTS AND RELATED MATERIALS

J.T. Luke, Chairman

A special subcommittee session was held to hear Mr. Marion Mecklenburg describe the results of a four and one half year study funded by the National Museum Act on the mechanical properties of paint films on the substrates used by artists. His conclusions relate to the subcommittee's effort to write test methods for flexibility, distensibility, and adhesion.

Group 1, Preparation of Samples for Colorimetric Determination, H.W. Levison, Chairman, reviewed the first draft of the standard test method for "Preparation of Films of Artists' Paste Paints for Colorimetric Determination." Discussion and explanation followed. Minor corrections were made. This proposed standard is now ready for editorial comment and letter ballot by the subcommittee.

Group 2, Lightfastness of Pigments, H.W. Levison, Chairman, reviewed an interim report outlining the status of the four lightfastness tests presently being conducted. The two outdoor exposure tests have been completed and results are being compiled. The indoor fluorescent tests are 40% complete. The Xenon Arc accelerated tests are 60% complete. The ISO Blue Wool and AATCC Blue Wool standards were examined after 50,000 rangeleys of exposure. Work has begun in preparing samples from eight different hue ranges which can be used to establish visually equal steps at the lightfastness level of the samples used in the lightfastness tests. Seven color chips from each

hue will be prepared with each chip varying by one delta E. Permanency ratings will be developed using this visual spacing to judge the amount of fading in the exposed paint samples.

Group 3, Tinting Strength of Pigments, I. Shack, Chairman, reviewed a draft of a proposed method for tinting strength determination of artists' colored paints. This test method compares artists' acrylic paints for tinting strength using Y measurements converted to Munsell value notation after the paints have been reduced by volume with a standard titanium dioxide white paint and compared with the Munsell measurements of a known paint made with the same pigment and reduced with the same standard titanium dioxide white paint. The method has been used to compare three sets of paints each group made from the same pigment. Additional colors will be examined and comments on the proposed method were requested.

Group 4, Text of the Specification for Artists' Paints, A.L. Spizzo, Chairman, discussed the revised draft of the specification. Minor changes were made. This document will be held until the approved pigment list to be included is complete.

Group 6, Definition of Terms, T. Vonderbrink, Chairman, reported the terms that had previously circulated within the subcommittee will be sent to the "Paint/Coatings Dictionary." All new and revised documents of the subcommittee will be reviewed for terms which should be defined.

Group 7, Physical Test Method, H.W. Levison, Chairman, reviewed a number of drafts on proposed test methods. A draft of a standard test method for "Yellowing of Paint Vehicles in Indoor

Environment" was discussed. Several minor changes were suggested and will be incorporated into the next draft, then the method will be sent to the editorial subcommittee for review. A draft of a standard test method for "Adhesion Between Artists' Paint Films Having Dissimilar Vehicles" was discussed. Some objections raised about the method were resolved. A section on humidity/temperature will be added. Additional comments will be sought. A test method for "Distensibility of Artists' Paint Vehicles" was circulated for comments. Additional comments will be sought. A draft of a standard test method for "Flexibility of Artists' Picture Varnishes" was also circulated for comments. Additional comments and the results of other tests will be obtained.

Group 8, Toxicity, held a special meeting to reorganize and expand the task group to deal with cautionary labeling for artists' paints and other related materials used by artists in response to Congressional pressure for a voluntary program to deal with labeling for chronic toxicity in these materials instead of further legislation. Dr. Woodhall Stopford was elected chairman. Two study groups were organized. One group is to study the Federal Hazardous Substances Act to determine how it relates to labeling for chronic hazards and how specific limits could be set on which substances and products that should carry warnings for chronic toxicity. The second study group is to examine the warning labeling requirements by the European Community, the labeling suggested by the National Paint and Coating Association, and the current labeling being used by the art material industry.

Society Meetings

Baltimore

Virginia Section

Feb. 25

Nick Rounds, of Rohm and Haas Co., gave a slide presentation featuring "RADIATION CURABLE COATINGS." Emphasizing the differences between conventional and radiation curable coatings, Mr. Rounds stressed that future growth of this field would be dependent on the development of new applications. However, given the advantages of hardness, high solids, no waste, fast process times, and excellent film properties, the use of radiation curable coatings is likely to increase, according to Mr. Rounds.

CARL B. MINCHEW, *Secretary*

Birmingham

Jan. 8

W.K.H. Lakin, of Manchem Ltd., spoke on "DRIERS."

Mr. Lakin began by defining "drying" as the evaporation of volatiles and

interaction of nonvolatiles to give a surface with an acceptable level of resistance to deformation. He described the various stages of drying as (1) wet edge, (2) dust free, (3) touch dry, and (4) hard dry.

According to Mr. Lakin, driers could be divided into several distinct groups depending on their mode of action. For example, cobalt, lead, and manganese assist oxidation of the film, while calcium acts as an acid acceptor. Zirconium and aluminum improve drying because of their ability to form coordination bonds.

A graph depicted the effect on percentage drying time of varying lead drier concentration in a cobalt-calcium-lead system. One effect was clearly seen: that their was an optimum level of lead above or below when the drying was slower. Excess lead could reduce hardness and durability besides slowing curing, explained Mr. Lakin. Therefore, it is important to determine the optimum level for any given drier, taking into

account that the level may vary depending on the other driers used.

Although cobalt and manganese perform similar functions, manganese has in the past been discounted due to its tendency to discolor films, said Mr. Lakin. Improvements in manufacturing techniques have eliminated this problem and tests showed that a mixture of the two materials could be used to achieve drying times similar to those for cobalt alone.

Mr. Lakin described how basic calcium driers work well in conjunction with zirconium. For example, a system having fixed optimum quantities of cobalt and zirconium cured faster with an addition of basic calcium than with an equivalent quantity of neutral calcium.

The results obtained from testing a wide range of double and triple drier systems were described. In particular, Mr. Lakin described how a mixture of aluminum and cobalt would give films with good durability, hardness and resistance to chalking and water-immersion.

Q. Would coordination driers assist in formulating paints which would dry when made and after several months in the can?

A. Drier adsorption is not fully understood. There is no loss of catalytic action when they are adsorbed into pigments. It is thought that the liberation of acid into the system nullifies the drier. The remedy is to use basic materials. (e.g., basic calcium drier).

Q. What is the length of time between addition of driers and their reaching optimum performance?

A. Lead achieves optimum with 24 hours, zirconium within 3 to 5 days. Test results discussed took this into account. In practice, an artificial standard may be necessary to speed up quality control testing.

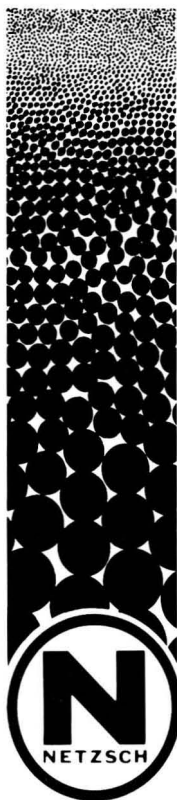
Q. How does aluminum drier affect flexibility?

A. Crosslinking induced by aluminum is superimposed on oxidation crosslinking. This could lead to embrittlement in the long term. This is not affected by the oxidative materials.

Q. What effect will aluminum drier have on paint stability?

A. If an alkyd has more than one carboxyl group per molecule, gellation will take place.

BRIAN ADDENBROOKE, *Secretary*



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Birmingham

Feb. 5

Dr. R.J. Loveland, H.M. Senior Electrical Inspector of Factories, spoke on "STATIC ELECTRICITY."

According to Dr. Loveland, between 20 and 30 incidents relating to electrostatic discharge are annually reported by industry. The combination of a high energy spark and a flammable atmosphere, as might be encountered when loading a ceramic-lined ball mill with toluene or xylene, can badly injure or kill operators.

After investigating the causes of many incidents, nine simple rules to follow were formulated to remove the electrostatic hazards, according to Dr. Loveland. They include: (1) all equipment which may become charged should be grounded, (2) antistatic footwear should be worn, (3) workshop floors should be constructed to allow any charge to leak to earth, (4) floors and footwear should be kept clean to maintain good electrical contact, (5) avoid use of insulating plastics, (6) electrostatic eliminators should be used where films or sheet of plastics are involved, (7) avoid the manual loading of powders or low conductivity liquids into vessels containing flammable vapors, (8) modify hazardous liquids by the addition of antistatic additives, and (9) vessels containing an easily ignited powder cloud should be fitted with explosion relief venting to a safe place.

Dr. Loveland emphasized the importance of the safety rules and cited examples of actual incidents. He stressed that these rules could not be applied in all cases, but they should be considered so that each potentially hazardous situation could be handled in the safest possible way.

BRIAN ADDENBROOKE, *Secretary*

Houston

Jan. 14

The Technical Symposium entitled "ENVIRONMENTAL REGULATIONS AND THE COATINGS INDUSTRY" was featured.

Starting the symposium was an update of government regulations affecting the coatings industry, presented by Art McDermott, of Nalco Chemical Co., and Don Montgomery, of Napko-O'Brien. Their presentation featured a survey of the coatings manufacturers in the southern U.S. This survey introduced several regulatory affects experienced by industry. The regulation having the most influence is the Resources Conservation and Recovery Act (R.C.R.A.) regardless of whether it was water-based coatings, solvent-based coatings, or effluent/discharge control, according to the speakers.

The second part of the symposium dealt with "Approaches to Meeting the Regulations While Producing



Southern Society Officers for 1980-81. Left to right: Society Representative—Bobby D. Moore; Secretary-Treasurer—William G. Early; Vice-President—Dan M. Dixon; President—Thad T. Broome; President-Elect—Frank D. Rector

Quality Coatings," presented by R. Pierrehumbert, of Union Carbide Corp. Mr. Pierrehumbert explained the difference in the number of phases present in latex and oil-based paints. Oil-based paints contain only two phases; the solid phase (pigments, extenders, etc.) and the liquid phase (binder and solvent). Latex paint contains three phases; water, latex particles, and pigments. Mr. Pierrehumbert then explained the effects of latex paint composition on exterior exposure.

J. Larson, of Cargill, Inc., presented the third session entitled "Smorgasbord Approach to High Solids Coatings." He compared four types of coatings; conventional—35% solids; high solids—65%; high solids—75%; and very high solids—90%. Solids determination to high solids paints is involved and specific procedures must be used, according to

Mr. Larson. Viscosity, temperature, and surface tension are factors affecting solids determination. Film properties, salt fog resistance, detergent resistance, and outdoor durability of all of the paints were discussed. Mr. Larson stressed that cost comparisons can be misleading.

The fourth speaker was Carl Hoffman, of Abcor, Inc., who discussed "Recycling Waste Streams with Ultrafiltration." Mr. Hoffman explained the two uses of ultrafiltration; reduction of pollution and recovering chemicals for recycle. Many industries use ultrafiltration in a diversity of applications, according to Mr. Hoffman. Ultrafiltration is adaptable to a continuous process by collecting the permeate while the waste is continually swept away. In regard to other separation processes, UF becomes more efficient as the velocity of stream increases.

K.D. JACOBSON, *Secretary*



Pacific Northwest Executive Committee for 1980-81. Seated left to right: Past-President—Walter B. Clyde; President—Curtis P. Bailey; President-Elect—Richard P. Stewart; Secretary—Bob Miller; Past-President—John A. J. Filchak. Standing: Administrative Secretary and Past-President—William A. Shackelford; Past-President—Michael Griffen; Treasurer—Robert S. Hogg; Society Representative and Past-President—Deryk R. Pawsey. Not shown: Art Bragg.

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 KAFFEN, SHARON L.—Glidden Coatings & Resins, Strongsville.
 LIAO, C.W.—Master Builders, Beachwood, OH.
 OKIN, SUSAN P.—PPG Industries, Inc., Cleveland.

PONYIK, CHARLES A., JR.—Cook Paint & Varnish, Cleveland.
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 USCHEEK, DAVID—Body Bros., Inc., Bedford, OH.
 WALLICK, TOM—Kiser Paint Co., New Philadelphia, OH.
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 NICHOLSON, ALBRIGHT M.—Ferro Corp., Bedford, OH.

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Journal of the Oil and Colour Chemists' Association

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Vol. 63 No. 11

November 1980

Badran, B.M., El-Anwar, I.M., and Ibrahim, M.S.—“Surface Coating Based on Dehydrated Castor Oil, Part 2: Dielectric Properties”; 427-432.

Bishop, R.R. and Winnett, M.A.—“Evaluation of Protective Coatings on Rusty Steel With the Use of Electrical Resistance Probes as Substrates”; 433-437.

Carr, W., Patterson D., and Tonge, M.—“Effect of Pigmentation Level on the Efficiency of Triple Roll Milling”; 438-445.

Staddon, A.W.E.—“Industrial Nitrocellulose”; 446-451.

Vol. 63 No. 12

December 1980

De la Court, F.H.—“Value of Tributyltinfluoride as a Toxicant in Antifouling Formulations”; 465-473.

Krishnamurti, N., Shirsalkar, M.M., and Sivasamban, M.A.—“Partial Allyl Ethers of Polyhydric Alcohols as Intermediates for Water Soluble Vehicles”; 474-481.

Schenck, H.U. and Stoelting, J.—“Electrodeposition: Anodic Versus Cathodic”; 482-491.

Gooch, C.—“Applied Finishes for Timber in Exterior Situations in New Zealand”; 492-495.

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Vol. 9 No. 6

June 1980

Andrew, R.T.—“Engineering Solutions to Resin Plant Problems”; 4-7.

Pearson, A.W.—“Pumping and Metering of Solvents”; 8-9.

Anon.—“Coatings Update: A Look at Economics and Statistics” (literature review); 10-12.

Chandra, S. and Batra, K.—“Polybisphenol Phthalate Alkyds”; 13-15.

Vol. 9 No. 7

July 1980

Stoye, D. and Dorffel, J.—“High-Solids One-Component Paints Based on Saturated Polyester Resins. Part 1”; 4-7, 16.

Paterson, A.J.—“Use of Brookfield Synchro-Lectric Viscometer in the Surface Coating Industry”; 8-9, 11.

Vol. 9 No. 8

August 1980

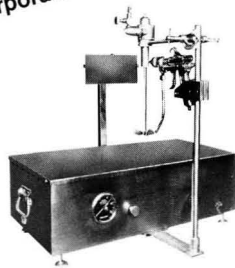
Kaluza, U.—“Flocculation—Which Factors Influence It? Part 1”; 4-7. (Translation of article published in *Defazet*).

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Ellis, R.A.—“Novel Use of Gel Permeation Chromatography in the Determination of Composition of Resin Blends”; 11-15.

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Vol. 9 No. 9

September 1980

- Kaluza, U.—“Flocculation—Which Factors Influence It? Part 2”;
4-7. (Translation of article published in *Defazet*).
Smith, C.A.—“Pollution-Free Printing Inks”;
11-12.
Blakey, R.R.—“Optical Properties of White Printing Inks”;
14,16, 18-21.

Vol. 9 No. 10

October 1980

- Kaluza, U.—“Flocculation—Which Factors Influence It? Part 3”;
4-7,10. (Translation of article published in *Defazet*).
King, S.—“Organotin Antifouling Coatings: Today and Tomorrow”;
8-10.
Lower, E.S.—“Lauroyl Peroxides as Polymer Catalysts and Other
Uses”;
11-12.
Smith, C.A.—“Pollution-Free Printing Inks. Part 2”;
13-15.
Anon.—“Coatings Update: Scientific Studies” (literature review);
16-19.

Vol. 9 No. 11

November 1980

- English, R.W.—“Automatic Production in the Paint Industry”;
5-7.
Smith, E.A. and Pemberton, E.W.—“Simple Method for Assessing
Fineness and Stability of Pigment and Dye Dispersions”;
8-9.
Anon.—“Coatings Update: Scientific Studies” (literature review);
17-20.

Vol. 9 No. 12

December 1980

- Kaluza, U.—“Flocculation—Which Factors Influence It? Part 4”;
3-9. (Translation of article published in *Defazet*).
Lower, E.S.—“Commercial Applications for Dibutyltin Dilaurate”;
10-11.
Walton, A.J.—“Applications of Gas Chromatography in the Paint
and Allied Industries. Part I: Raw materials”;
15-19,25.

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Vol. 8 No. 1

1980

- Sato, K.—“Hardness of Coating Films”;
1-18.
Nakamichi, T.—“Behavior of Thermosetting Powder Coatings During
Curing Process”;
19-46.
Zosel, A.—“Mechanical Behaviour of Coating Films”;
47-79.
Kamarchik, P. and Cunningham, G.P.—“Applications of X-ray
Techniques to Coatings Analysis”;
81-107.

Vol. 8 No. 2

1980

- Ramsbotham, J.—“Solvent Formulation for Surface Coatings”;
113-141.
Sato, K.—“Internal Stress of Coating Films”;
143-160.
Hill, L.W. and Wicks, Z.W., Jr.—“Amine Solubilizers for Water-
Soluble Acrylic Baking Enamels”;
161-182.
Perera, D.Y.—“Hygric Aspects of Coated Porous Building Materials”;
183-206.

Vol. 8 No. 3

1980

- Christensen, G.—“Analysis of Functional Groups in Amino Resins”;
211-239.
Bender, H.S., Cheever, G.D., and Wojtkowiak, J.J.—“Zinc Phos-
phate Treatment of Metals”;
241-274.
Kornum, L.O. and Raaschou Nielsen, H.K.—“Surface Defects in
Drying Paint Films”;
275-324.

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Vol. 86 No. 11

November 1980

- Zorll, U.—“Interlayer Adhesive Strength of a New Type of Paint
Finish”;
956-959.
Muller, F.—“Viscosity Measurements in Practice”;
959-965.

Lincke, G.—“Stuart Spherical Sections as an Aid in Studying Crystal
Structure of Linear Trans-Quinacridone Pigment (γ -phase)”;
966-972.

Peigner, M., Lasnet de Lanty, P., and Zimmermann, R.—“Isopropyl
Acetate—a Solvent for the Future”;
973-975.

Brushwell, W.—“Results of Technical Research into Lacquers”;
976-982.

Vol. 86 No. 12

December 1980

Broustin, P.—“Value Analysis—A Path for Optimising Profits”;
1045-1046.

Volz, H.G., Kampf, G., and Klaeren, A.—“Experimental Techniques
for Determining the Internal Degradation and Protective Effect of
TiO₂ Pigments in Paints During Weathering”;
1047-1055.

Lauterbach, H.—“Aqueous Powder Suspensions (APS)—Applica-
tions and Limitations”;
1056-1058.

Biermann, M.—Theory of the Tear-Off Test, (3) Axi- and Plani-
Symmetric Problem of the Extension of a Linearly Elastic Com-
pound Body with a Thin Joint”;
1058-1066.

Gross, H.—“Effect of Various Salt Solution in Salt Spray Test (DIN
53 167)”;
1066-1068.

Brushwell, W.—“Review of Raw Materials Available for Manufac-
turing Paints and Lacquers”;
1069-1076.

Vol. 87 No. 1

January 1981

Bagda, E., Renz, W., and Weissert, A.—“Resistance to Moisture of
Facade Coatings”;
8-12.

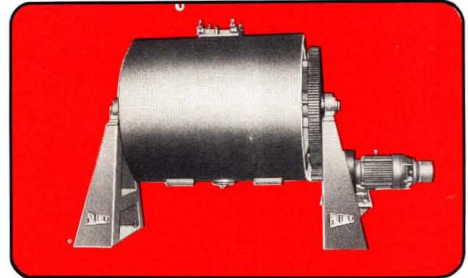
Volmer, R.—“Error Propagation in the Calculation of Absorption
and Scattering”;
12-15.

Schmelzer, H.—“Approximation Solutions for the Theory of Trans-
parent Films on a Scattering Substrate”;
15-18.

Engels, K.—“Outlet and Sealing Problems of Enclosed Pearl Sand
Mills”;
18-25.

Brushwell, W.—“Data for Forecasting in the Paint Industry”;
26-29.

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Special Topics Courses Scheduled for Kent State in June

The Rheology and Coatings Laboratory at Kent State University will present two special topics courses entitled, "Adhesion Principles and Practice for Coatings and Polymer Scientists," June 8-12, and "Applied Rheology for Industrial Chemists," June 15-19. Designed for those involved in research, development, and production who have need for knowledge of improved adhesion of coatings, inks, adhesives, and related materials, the programs will be presented by industrial and university authorities.

Program Chairman Carl J. Knauss announced the following presentations:

Adhesion Principles and Practice for Coatings and Polymer Scientists

JUNE 8

"Principles of Bonding I"—Dr. R.R. Myers, Kent State University.

"General Rheology and Its Application to Adhesion"—Dr. D.L. Hunston, National Bureau of Standards.

"Principles of Bonding II"—Dr. R.R. Myers.

"Practical Application of Silane Adhesion Promoters."—Dr. E. Pluddeman, Dow Corning Corp.

JUNE 9

"Introduction to Stress Analysis of Adhesive Bonded Joints"—Dr. W.B. Jones, Air Force Materials Laboratory.

"Structure and Properties of Organosilane Primers for Adhesive Bonding of Metals"—Dr. F.J. Boerio, University of Cincinnati.

"Newer Approaches to Surface Tension Measurements and Adhesion Enhancement"—Dr. H.P. Schreiber, Ecole Polytechnique.

"Interfacial Energies and Interaction at the Interface"—Dr. R.J. Good, State University of New York at Buffalo.

JUNE 10

"Interfacial Chemistry of Corrosion

Induces Adhesion Loss"—Dr. R.A. Dickie, Ford Motor Co.

"Overview of Surface Chemical Analysis Techniques"—Dr. T.W. Rusch, Perkin-Elmer Physical Electronics Div.

"Etching of Polymer Surfaces for Adhesive Bonding"—Dr. J. Koutsky, University of Wisconsin.

"Spectroscopic Characterization of Polymer/Metal Adhesion"—Dr. J.P. Wightman, Virginia Polytechnic Institute and State University.

JUNE 11

"Introduction to Pressure Sensitive Adhesive"—J.A. Miller and P.J. Houck, Morgan Adhesives Co.

"Chemical Physical Properties and Durability of Structural Adhesives"—Dr. A. Pocius, Minnesota Mining & Manufacturing Co.

"Principles of Adhesion Measurements"—Dr. W.K. Asbeck, Consultant.

"Matrix-Fiber Adhesion in Composites"—Dr. L.T. Drzal.

JUNE 12

"Effect of Water on Adhesive Bonding"—Dr. R.L. Patrick, Rockwood Systems Corp.

"Industrial Uses of Primers, Rubber-to-Metal Bonding"—D.P. Nick, Lord Corp.

Applied Rheology for Industrial Chemists

JUNE 15

"Basic Concepts of Rheology I"—Dr. H. Markovitz, Carnegie-Mellon University.

"Basic Concepts of Rheology II"—Dr. H. Markovitz.

"Measuring Techniques for Viscoelastic Properties of Liquids"—Dr. R.R. Myers.

"Applied Rheology of Elastomers"—Dr. E.A. Collins, Diamond Shamrock Corp.

JUNE 16

"Kinetic Rheology Applied to Liquid-to-Solid Conversion"—Dr. R.R. Myers.

"Application of Rheology to Process Problems"—Dr. J.M. Starita, Rheometrics, Inc.

"Application of Rheology to End Use Performance Problems"—Dr. J.M. Starita.

TECHNICAL COMPUTER APPLICATIONS IN THE COATINGS INDUSTRY A Bibliography



This 11-page bibliography consists of references grouped into the following sections:

- Applications—General • Analytical Methods • Color and Optical Properties • Evaluation; Performance • Formulation • Manufacture and Plant • Pretreatment; Applications; Finishing • Printing Inks • Polymers/Resins • Testing

Most of the references were located by searching the subject indexes of Chemical Abstracts and/or Review of Current Literature/World Surface Coatings Abstracts. Produced by the FSCT Technical Information Systems Committee (in cooperation with the International Coordinating Committee) this work should be of great value in our industry. Price \$2.00.

Order from:

Federation of Societies for Coatings Technology
1315 Walnut Street
Philadelphia, PA 19107

Continued on page 85

Southern Society Hosts 45th Annual Meeting

An attendance of 255 (212 men, 43 women) was recorded at the 1981 annual meeting of the Southern Society, in Birmingham, AL, March 11-13.

The theme of the meeting was "Coatings Technology—the End Users' Perspective."

Presentations

The Keynote Address—"We Need to Help Our Products Go First Class"—was presented by Charles W. Finegan, President of Havco Paints, Inc. [see the April JCT—Ed]

Other presentations were as follows:

"Waterborne Coatings Practical Problems"—a panel discussion moderated by Fred Shahade, of PPG Industries, Inc. Members were: Tom McCraney, of Mobile Paint Mfg. Co.; Craig Larson, of Wyandotte Paint Products, Inc.; Arvid C. Walberg, of A.C. Walberg Co.; and

Tom Graves, of the National Paint and Coatings Association.

"Trends in Metal Finishing"—Joseph E. Ziegeweid, consultant.

"High Solids Solvent-Based Coatings"—Vic Ginsler, of Freeman Chemical Corp.

"Role of Organic Co-Solvents in Waterborne Coatings"—Dr. Robert Eaton, of Union Carbide Corp.

"Paint Quality—the Producer"—Thad T. Broome, of Precision Paint Co. (and President of the Southern Society this year).

"Paint Quality—the User"—a panel discussion moderated by Bill Early. Members were: Leonard Fulghum of Furgeson-Fulghum, Inc.; Guy A. Walters, Jr., of Broyhill Furniture Co.; Bob Kane, of International Harvester Co.; and Paul Oliver, of U.S. Steel Corp.

"Paint Quality—the Consumer"—Joseph Csernica, of Consumers Union of the United States.

Twenty-five year membership pins were presented to: William Timme, of Crowe Industrial Coatings; Harry J. Poth, of Burk-Hall Co.; Richard F. Hirt, of Burk-Hall Co.; and Billie Williams, of

American Lacquer & Solvents Co.

Southern Society officers for 1981-82 were elected as follows: President—Frank Rector; President-Elect—Dan M. Dixon; Vice-President—Bill Early; Secretary-Treasurer—Jim Geiger, of Sun Coatings, Inc.; and Society Representative—Bobby D. Moore.

Special Award to "Colonel Billy Hood"

At the luncheon, a special award of appreciation for long-time service to industry was presented to William E. (Colonel Billy) Hood, Chairman of the Board of Indurall Paints, Inc. "Colonel Billy"—who introduced his famous questions and answer sessions on paint production at Federation Annual Meetings during the 1950's—was President of the Southern Society in 1950. He served as President of the NPCA in 1963 and was honored as an Industry Senior Statesman by NPCA in 1980.

The 1982 annual meeting will be held at the Hyatt Regency Hotel in Savannah, GA, March 10-12.

Continued from page 84

"Use of Rheology in Industrial Applications"—Dr. D. Bigg, Battelle-Columbus Laboratories.

JUNE 17

"Rheological Control for High-Solids Coatings"—Dr. C. Schoff, PPG Industries, Inc.

"Chemorheology of Thermosets"—Dr. R.R. Elley, Glidden Coatings & Resins Div., SCM Corp.

"Rheology of Dispersions"—Dr. E. Bagley, U.S. Dept. of Agriculture.

"Rheology of Gels"—Dr. E. Bagley.

JUNE 18

"Brush Application of Coatings"—Dr. J.E. Glass, North Dakota State University.

"Roll Application of Coatings"—Dr. J.E. Glass.

"Correlation of Film Morphology with Drying Behavior"—Dr. J.W. Vanderhoff, Emulsion Polymers Institute, Lehigh University.

"Flow Fundamentals in Fluid Systems of High Molecular Weight Additives"—Dr. D.L. Hunston.

JUNE 19

"Fracture Behavior of Brittle Materials"—Dr. D.L. Hunston.

"Fiber-Reinforced High Performance Engineering Thermoplastics"—Dr. A.F. Lewis, Lord Corp.

Additional details can be obtained from Carl J. Knauss, Kent State University, Chemistry Dept., Kent, Ohio 44242.

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Edward S. Demers has joined International Minerals & Chemical Corp. as Senior Product Manager for Derivatives in the firm's NP Division. Also, **Dennis T. Leu** has been named Product Manager for Specialty Chemicals in the company's NP Division. Mr. Leu is a Chicago Society member.

Chemcentral Corp. has announced the following appointments. **A.M. Schulten** has been named Regional Manager of the firm's west coast region. **K.M. Jagers** was appointed Vice-President and General Manager at the company's Los Angeles office. Mr. Jagers is a Los Angeles Society member. Named to the position of Technical Sales Representative was **Eric C. Lynd**.

Wallace P. Giordano has been named Allied Chemical Corporation's Polyethylene Western Regional Sales Manager. Headquartered in Schaumburg, IL, Mr. Giordano will be responsible for sales activity in the upper Ohio Valley, Midwest, Southwest, and the West Coast.



E.S. Demers



D.T. Leu



R.E. Perkins



D.H. Gels

Richard E. Perkins has joined Fuller-O'Brien Paints in the newly created position of Technical Director, Midwestern Region. He will be located at the firm's technical and manufacturing facilities in South Bend, IN. Mr. Perkins is a Chicago Society member.

Whittaker Corp. announced the election of **Gregory T. Parkos** as Vice-President and Group Executive, responsible for the firm's coatings and chemicals operations.

Donald H. Gels has been appointed President and Chief Executive Officer of the Red Devil Paint Co., Div. of Insilco, Mt. Vernon, NY.

Edward R. Alkins has been appointed Senior Sales Representative, Southeast District, for Polyvinyl Chemical Industries.

James Moe has been selected to manage the BPS Division of the Enterprise Companies (an Insilco company).

Glidden Coatings & Resins has appointed **F. Duane Smith** as Manager—Standardization at their research center in Strongsville, OH. Named to Field Sales Manager—Chemical Coatings, Central Region, was **Charles E. Newhouse**.

NL Chemicals/NL Industries, Inc. has announced several staff appointments for its Eastern Sales Region. **Michael J. Kenny** has been named Eastern Regional Sales Manager; **George W. Bovenizer**, District Sales Manager; and **William R. VanBeusichem**, Sales Representative for New England.

Dr. Henry W. George, Director of Resource Development for Trail Chemical Corp., has retired after 16 years of service. Dr. George is a Los Angeles Society member.

John E. Grigsby, Jr., has been named Manager of Plant Engineering for the paint manufacturing facility of Inmont Corporation's Automotive Group, Detroit, MI.

Jones-Blair Co. announced the promotion of **A.H. "Dusty" Hundt** to Manager of Technical Services. Also, **John Ostrowski** has joined the firm in Dallas as Laboratory Manager of Special Products. Mr. Ostrowski is a Pittsburgh Society member.

Media Formulation/ Process Development Engineer/Chemist

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William E. (Colonel Billy) Hood received a special award of appreciation for his longtime service to the industry during the annual meeting of the Southern Society, March 12, in his hometown of Birmingham, AL. He served as President of the Southern Society in 1950 and a few years later introduced his forever-famous "Colonel-Billy" question and answer sessions on production at Annual Meetings of the Federation. Active also in the NPCA, he served as its President in 1962-63 and in 1980 was recognized by NPCA as an Industry Senior Statesman. Shortly after graduation in 1931 from the University of Alabama, "Colonel Billy" joined the paint industry and the Alabama Paint Company. Along with Louis Prosch, he formed Indurall Coatings, Inc. in 1947 and serves now as Chairman of the Board.

Kevin B. Murphy has been named General Manager of Sico Coatings Corp. of Braintree and Taunton, MA. He will hold a similar position with NuBrite Chemical Co., a subsidiary of Sico.

Bob Mackey has been promoted to Branch Manager of the Houston distribution center of Thompson-Hayward Chemical Co. He will be responsible for the sales and administration of the industrial, textile maintenance and pest control divisions. Mr. Mackey is a member of the Houston Society.

Mooney Chemicals, Inc. has appointed **Mark H. Schmidt**, Midwest Sales Representative, in Chicago.

PPG Industries, Coatings and Resins Division, announces the following appointments in their Research and Development Department at the R&D laboratory facilities located in Allison Park and Springdale, PA.

Dr. Marvis E. Hartman was named to the position of Senior Research Associate. **Dr. Gregory J. McCollum** and **Dr. Robert M. Piccirilli** were appointed Research Associates.

Hercules Incorporated has announced that its Water-Soluble Polymers and Coatings Business Center has been restructured into two distinct elements. **Dr. Frank H. Wetzel** will continue as Director of the Worldwide Water-Soluble Polymers Business Center, and **Edwin S. Moler**, formerly President, Hercules Canada Ltd., has been named Director of the newly formed Coatings Business Center. **Walter W. Schultz**, former Director of Operations, Hercules Canada, has been appointed President, Hercules Canada Ltd.

DeSoto, Inc. has promoted **Don Fieder** to Research Chemist in their Polymer Development Department. Mr. Fieder is a member of the Chicago Society. Also announced by the firm was the appointment of **Carol J. Pinson** to Senior Chemist, Research Services Department.

Russell Koch has joined Universal Color Dispersions as Technical Director, responsible for laboratory and technical service activities.

Melvin L. Penn has joined the Pigment Division of Kerr-McGee Chemical Corp. as a Sales Representative in the Southern Region Office located in Decatur, GA.

Kraft Chemical Co. has announced the appointment of **Elizabeth Shepherd** as Sales Representative. Headquartered in Melrose Park, IL, Ms. Shepherd will concentrate her sales efforts in the coating, cosmetic, and specialty chemical industries.

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Microprocessor Colorimeter

Literature is available featuring a microprocessor colorimeter, which provides permanent storage for instrument and product standard values. For more information, write Hunter Associates Laboratory, Inc., 11495 Sunset Hills Rd., Reston, VA 22090.

Solvents

A new brochure providing data on a new line of solvents designed to meet the demands of the market has recently been published. For a copy of the brochure, contact the Specialty Chemicals Div., Ashland Chemical Co., Dept. SC, P.O. Box 2219, Columbus, OH 43216.

Thermal Processing Systems

New literature has been published on a series of thermal processing systems and their application in the study of volatile and nonvolatile organics in liquid or solid sample matrixes. For more information, contact Chemical Data Systems, Inc., RD 2, Box 74, Oxford, PA 19363.

Guide to Vinyl Coatings

A 20-page booklet on the use of vinyl maintenance paints for corrosion protection is now available. The coatings described are single-package systems that can be formulated from solution vinyl resins to comply with current air pollution control regulations. Copies of "Vinyl Coatings for Corrosion Control," F-46055A, can be obtained from Union Carbide Corp., Coatings Materials Div., Dept. K3442, Danbury, CT 06817.

Thickness Measurement Gauge

A gauge for nondestructive thickness measurements of paint and other nonmagnetic coatings on ferrous substrates is the subject of recent literature. A two-page catalog sheet, including specifications can be obtained by writing Elcometer, Inc., P.O. Box 1203, Birmingham, MI 48012.

Acrylic Emulsion

A 32-page booklet featuring a general-purpose emulsion for trade paints has been published. Properties, applications, and formulating guidelines are discussed. Copies of the booklet, "UCAR Acrylic 500—General Purpose Acrylic Emulsion," F-48216, are available from Union Carbide Corp., Coatings Materials Div., Dept. K3442, Danbury, CT 06817.

Solvent

New literature featuring a solvent used to reformulate coatings for greater economy with no reduction in performance is now available. This folder includes a Hansen resin and solvent map along with detailed specifications and physical and chemical data for the solvent. Write DuPont Co., Room X38526, Wilmington, DE 19898.

Hi-Drum Racks

Hi-drum storage racks, making vertical storage of 55 gallon drums a safe and efficient space saver, are the subject of recent literature. For more information, write Jarke Corp., Sales Dept., 6333 W. Howard St., Niles, IL 60648.

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Radiation Cured Coatings Monograph

A monograph developed to address the safe industrial use of radiation cured coatings has been published by the National Paint & Coatings Association and is now available. Developed by a Special Subcommittee on Radiation Cured Coatings, "Safe Handling and Use of Ultraviolet/Electron Beam Curable Coatings," serves as a safety primer for the use of UV/EB cured coatings, as well as a refresher for users and manufacturers. Copies of the monograph are available to NPCA members for \$5, to nonmembers at \$10 per copy. Write NPCA, Bob Nelson, 1500 Rhode Island Ave., N.W., Washington, DC 20005.

Stainless Steel Tubing

The advantages and applications of welded stainless steel tubing and pipe are featured in a new, illustrated 20-page brochure. Topics include the classification, processing, finishing, and nondestructive testing of stainless tubing and pipe. Tolerances and standard sizes are presented in chart form, as well as the physical characteristics of square, rectangular, and special shape stainless tubing. Copies of the brochure are available from Republic Steel Corp., P.O. Box 6778, Cleveland, OH 44101.

Paint Pigment

A bulletin describing a new, corrosion-inhibiting paint pigment is now available. The pigment containing zinc molybdate for corrosion inhibition, is formulated to provide adhesion to rusted steel substrates. To obtain a copy of Technical Bulletin 363, write Sherwin Williams Chemicals, Publications Dept., 10909 S. Cottage Grove Ave., Chicago, IL 60628.

Health and Safety Handbook

The second edition of "Health and Safety, Environmental Pollution and the Paint Industry," is now available. It features a survey, listing all potentially hazardous materials and a checklist of their effects on man and the environment. Replacements for the more dangerous materials, conditions defined for the safe use of others, means for monitoring control, and voluntary restrictions adopted by industry in advance of legislative control are discussed. This standard health and safety handbook for the coatings industry is available from the Paint Research Association, Caroline Veitch, Publications Coordinator, Waldegrave Rd., Teddington Middlesex, TW11 8LD.

Liquid Alkaline Cleaner

A bulletin describing a liquid alkaline cleaner designed to remove latex and similar chemicals from inside tanks in minutes is now available. This spray tank cleaner is a nonsilicated concentrated heavy-duty liquid cleaner designed for use in tank cleaning operations. For more information on Bulletin 1480, write the Specialty Chemicals Div., Ashland Chemical Co., Dept. SC, P.O. Box 2219, Columbus, OH 43216

Batch and Extended Control Systems

Literature featuring a new batch and extended control system is now available. The new system provides computerized monitoring and regulation of production elements for petroleum refining, power generation, chemicals, steel and pulp and paper industries. For more information, write Honeywell, Process Management Systems Div., 16404 N. Black Canyon Hwy., Phoenix, AZ 85023.

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Book Review

WATERBORNE COATINGS: EMULSION AND WATER-SOLUBLE PAINTS

Author

Charles R. Martens

Published by

Van Nostrand Reinhold

New York, NY

1980, 336 pgs., \$22.50

Reviewed by

Leon Kutik

Dutch Boy, Inc.

Baltimore, MD

"Waterborne Coatings: Emulsion and Water-Soluble Paints" is a substantially revised and much more comprehensive treatment of this technology as compared with the author's earlier work on this subject entitled "Emulsion and Water-Soluble Paints and Coatings," which appeared in 1964.

In this recent work, the author presents a somewhat elementary, but well organized, introduction to the coatings industry, with particular emphasis on air pollution, in order to set the stage for a general discussion on coatings technology, and as an introduction to water-

borne and organic binder chemistry. Although basic concepts are not ignored, most of the emphasis is on a practical elementary level particularly aimed at the student or entry level technician or Junior Chemist.

The chapters on organic binders, surfactants, protective colloids and thickeners, pigments and extender pigments, biocides and miscellaneous ingredients present an excellent review of what is available from raw material suppliers with, once again, only a brief reference to theoretical considerations.

Similarly, the chapters on emulsion formation and manufacture and handling are again quite basic, and serve rather simply in explaining and introducing these aspects of water-based technology to the reader.

A significant section of the book is devoted to formulation of water-based trade sales paints, maintenance paints, industrial coatings, cement and silicate coatings and miscellaneous products, such as caulks. Here the author has borrowed heavily, if not exclusively, from raw material suppliers with all the inherent risks associated with this level of technology. Nevertheless, this will be of value to some readers, particularly in the number and variety of formulations presented.

In an effort to be all inclusive, the author has not neglected color theory,

raw material testing, surface testing and application as well as government regulations and coating calculations. This is commendable, but should only serve as an introduction to these complex subjects.

In summary, Charles R. Martens, in his book, has put together an excellent introduction and review to coatings technology, in general, and water-based technology, in particular. This should be of great value to those entering the industry in marketing, production or a support function. The more experienced practitioner will appreciate the scope of this work and may use it as a teaching tool.

SIZE EXCLUSION CHROMATOGRAPHY

Edited by

Theodore Provder

Glidden Coatings and Resins

Published by

American Chemical Society

Washington, DC

1980, 312 pgs., \$30.75

Reviewed by

John Wuepper

Whirlpool Corp.

Benton Harbor, MI

This book, like others in the ACS Symposium Services, is comprised of collected presentations from a symposium. The manuscripts are published as they are received from the different authors. This particular symposium took place in September 1979, and dealt with size exclusion chromatography, otherwise known to many workers who followed its development as gel permeation chromatography or GPC.

The seventeen "chapter" book includes papers on the use of GPC for particle size analysis, polymer viscosity characterization, polymer chain branching and copolymer composition determination as a function of molecular weight. Topics on the effect of solute shape in size exclusion chromatography, HPGPC analysis of oligomers and reversed micellar systems and kinetic modeling of polymerization of reactions are also presented. The book ends with chapters on aqueous GPC and the use of Sephadex and Spherogel-TSK gels.



Executive Committee of the Montreal Society for 1980-81. Seated (left to right): Luigi Catroni, Robert Kuhnen, John Flack, Al Marchetti, Raye Fraser, Robert Rauch, and John Erskine. Standing (left to right): Bill Reid, Gilles Bernicky, Eric Templeton, Robert Snyder, Andre Lamarre, Vaughn Pedersen, H. Philipp, Art Hagopian, Yvan Maillette, Gordon Simpson, and Robert Cabral

Coming Events

FEDERATION MEETINGS

(Oct. 28-30)—59th Annual Meeting and 46th Paint Industries' Show. Cobo Hall, Detroit, MI. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, PA 19107).

1982

(Apr. 29-30)—Spring Meetings. Society Officers on 29th; Board of Directors on 30th. Boston, MA. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, PA 19107).

(Nov. 3-5)—60th Annual Meeting and 47th Paint Industries' Show. Sheraton Washington Hotel, Washington, D.C. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, PA 19107).

SPECIAL SOCIETY MEETINGS

(June 15)—Golden Gate Society Seminar, "Safety and Government Regulations." (Louie Sanguinetti, Jasco Chemical Corp., P.O. Drawer J, Mountain View, CA 94042).

(Sept. 23-24)—Montreal and Toronto Societies Joint Symposium on "Coatings Directions for the 80's."

1982

(Mar. 10-12)—Southern Society Annual Meeting. Hyatt Regency, Savannah, Ga. (Dan Dixon, Freeport Kaolin Co., P.O. Box 337, Gordon, GA 31031).

(Mar. 23-24)—25th Annual Technical Conference of the Cleveland Society for Coatings Technology. Baldwin-Wallace College, Berea, OH.

(Apr. 22-24)—Southwestern Paint Convention. Shamrock Hilton Hotel, Houston, TX.

(May 6-8)—Pacific Northwest Society. Annual Symposium. Vancouver, B.C.

1983

(Feb. 23-25)—16th Biennial Western Coatings Societies' Symposium and Show. Hyatt Regency, San Francisco, CA.

OTHER ORGANIZATIONS

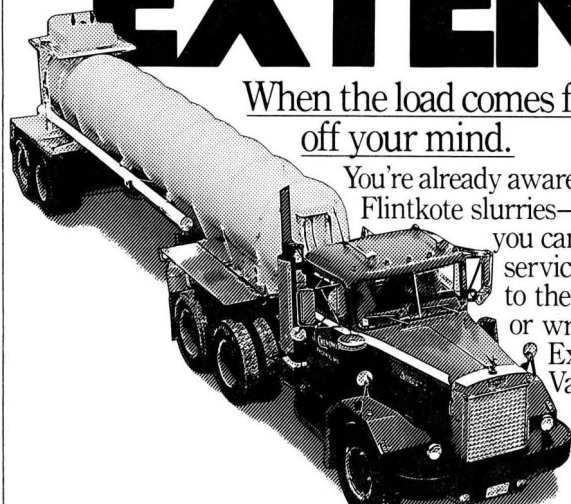
(May 31-June 5)—"Polymer Blends and IPN's" Short Course. Lehigh University, Bethlehem, PA (Jone Svirzofsky, Lehigh University, Bethlehem, PA 18015).

(June 1-12)—"Basic Coatings Science" course. North Dakota State University, Fargo, ND. (Dr. R. Chenoweth, Div. of Continuing Studies, NDSU, Fargo, ND 58105).

(June 8-12)—"Adhesion Principles and Practice for Coatings and Polymer Scientist" Short Course. Kent State University, Kent, OH. (Carl J. Knauss, Chemistry Dept., Kent State University, Kent, OH 44242).

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(June 8-12)—"Advances in Emulsion Polymerization and Latex Technology" Short Course. Lehigh University, Bethlehem, PA. (Dr. Mohamed S. El-Aasser, Dept. of Chemical Engineering, Whitaker Lab. #5, Lehigh University, Bethlehem, PA 18015).

(June 14-17)—Dry Color Manufacturers' Association Annual Meeting. The Greenbriar, White Sulphur Springs, WV. (P.L. Lehr, DCMA, Suite 100, 1117 North 19th St., Arlington, (Rosslyn) VA 22209.)

(June 15-19)—"Applied Rheology for Industrial Chemists" Short Course. Kent State University, Kent, OH. (Carl J. Knauss, Chemistry Dept., Kent State University, Kent, OH 44242).

(June 17-20)—Oil and Colour Chemists' Association Conference, "Alternative Technologies in Coatings." Beaufort Hotel, Bath, England. (The Director and Secretary, OCCA, Priory House, 967 Harrow Rd., Wembley, Middlesex HA0 2SF, England.)

(June 21-26)—Air Pollution Control Association 74th Annual Meeting & Exhibition. Philadelphia Civic Center, Philadelphia, PA (Public Relations Dept., Air Pollution Control Association, P.O. Box 2861, Pittsburgh, PA 15230).

(June 21-24)—American Society for Testing and Materials Committee D-1 on Paint and Related Coatings and Materials Meeting. Biltmore Plaza, Providence, RI. (Jane R. Turner, ASTM, 1916 Race St., Phila., PA 19103).

(June 22-July 3)—"Advanced Coatings Science" course. North Dakota State University, Fargo, ND. (Dr. R. Chenoweth, Div. of Continuing Studies, NDSU, Fargo, ND 58105).

(June 29-July 3)—SURTEC (International Congress and Exhibition on Surface Technology). International Congress Center, Berlin, West Germany. (Dieter von Schramm, AMK Berlin, 1735 Eye St. N.W., Washington, D.C. 20006).

(July 5-10)—Fourth International Conference on "Surface and Colloid Science." Jerusalem, Israel. (The Secretariat, Fourth International Conference on Surface and Colloid Science, c/o Conventions (Kopel Tours) Ltd., P.O. Box 3054, Tel Aviv 61030, Israel).

(Aug. 10-14)—"Introductory Short Course on Composition of Coatings." University of Missouri-Rolla, Rolla, MO (Norma Fleming, Coordinator, Arts & Sciences Continuing Education, University of Missouri-Rolla, Rolla, MO 65401).

(Aug. 31-Sept. 4)—"Introduction to Paint Formulation" Short Course. University of Missouri-Rolla, Rolla, MO (Norma Fleming, Coordinator, Arts & Sciences Continuing Education, University of Missouri-Rolla, Rolla, MO 65401).

(Sept. 7-11)—"Advanced Paint Formulation" Short Course. University of Missouri-Rolla, Rolla, MO (Norma Fleming, Coordinator, Arts & Sciences Continuing Education, University of Missouri-Rolla, MO 55401).

(Sept. 13-16)—Canadian Paint Manufacturers Association Annual Meeting. Four Seasons Hotel, Vancouver, B.C. (Lydia Palazzi, Canadian Paint Manufacturers Assn., 515 St. Catherine St. W., Montreal, Que., Canada H3B 1B4).

(Sept. 14-16)—Second International Conference on "The Durability of Building Materials and Components." Gaithersburg, MD. (Dr. Geoffrey Frohnsdorff, B348, Bldg. 226, Center for Building Technology, National Bureau of Standards, Washington, D.C. 20234).

(Sept. 20-25)—4th Congress of the Association Internationale de la Couleur, "COLOR 81." International Congress Centre (ICC), Berlin (West), Germany. (Prof. Dr. Heinz Terstiege, (AIC COLOR 81), Bundesanstalt für Materialprüfung (BAM), Unter den Eichen 87, D-1000 Berlin 45, Federal Republic of Germany).

(Sept. 21-22)—"Techniques and Mechanics of Marketing Specialty Chemicals" Short Course. Hilton Plaza Inn, Kansas City, MO (Norma Fleming, Coordinator, Arts & Sciences Continuing Education, University of Missouri-Rolla, Rolla, MO 65401).

(Sept. 23-24)—"Managing for Innovation in Coatings" Short Course. Hilton Plaza Inn, Kansas City, MO (Norma Fleming, Coordinator, Arts & Sciences Continuing Education, University of Missouri-Rolla, Rolla, MO 65401).

(Sept. 28-29)—Golden Jubilee of Colour in the CIE. The Colour Group (Great Britain). Imperial College, London, England. (Ms. M.B. Halstead, Thorn Lighting Ltd., Great Cambridge Rd., Enfield, Middlesex EN1 1UL, England).

PAUL O. ABBE, INC.	83
ASARCO	43
ATLAS ELECTRIC DEVICES CO.	82
BUCKMAN LABORATORIES, INC.	Cover 4
CHEMISCH WERKE HULS AG	27
CIBA-GEIGY CORP.	10-11
COSAN CHEMICAL CORP.	56
DOW CORNING CORP.	30
ECLIPSE SYSTEMS, INC.	82
FLINTKOTE STONE PRODUCTS CO.	91
GEORGIA KAOLIN CO.	Cover 2
GOODRICH	Cover 3
W.R. GRACE, DAVISON CHEMICAL DIV.	29
HERCULES INCORPORATED	16A,B,C,D
J.M. HUBER CORP.	81
ILLINOIS MINERALS CO.	85
NETZSCH, INC.	78
NL CHEMICALS/NL INDUSTRIES, INC.	1
C.J. OSBORN CHEMICALS, INC.	80
PETROLITE CORP., BARECO DIV.	61
ROHM AND HAAS CO.	8-9,14-15
SCHOLD MACHINE CO.	87
SILBERLINE MANUFACTURING CO., INC.	89
SOUTH FLORIDA TEST SERVICE, INC.	82
SPENCER KELLOGG DIV., TEXTRON, INC.	2
TENNECO CHEMICALS CO.	13
TROY CHEMICAL CORP.	16
UNION CARBIDE CORP.	4-5, 25

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