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JOURNAL OF
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TECHNOLOGY

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SEPTEMBER 1979

Convention
Issue

Progress
thru
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The big coatings happening of the year takes place in a few weeks when the 1979 Federation Annual Meeting and Paint Industries' Show gets underway at the St. Louis Convention Center, October 3-5.

Theme for this year's event is "Progress Through Innovation," and a plenitude of offerings addressing that topic will be found in both the meeting room and exhibit halls.

Chairman Moe Coffino and his Program Committee members have put together three full days of "meat and potatoes" technical sessions that cover a variety of coatings subjects — from ecological and high performance coatings, waste management, and media milling, to photo-responsive coatings, lab management, and color and pigmentation. In these and other featured presentations, registrants will be provided with an update on what's happening on the coatings technical front by many of the top people in the industry.

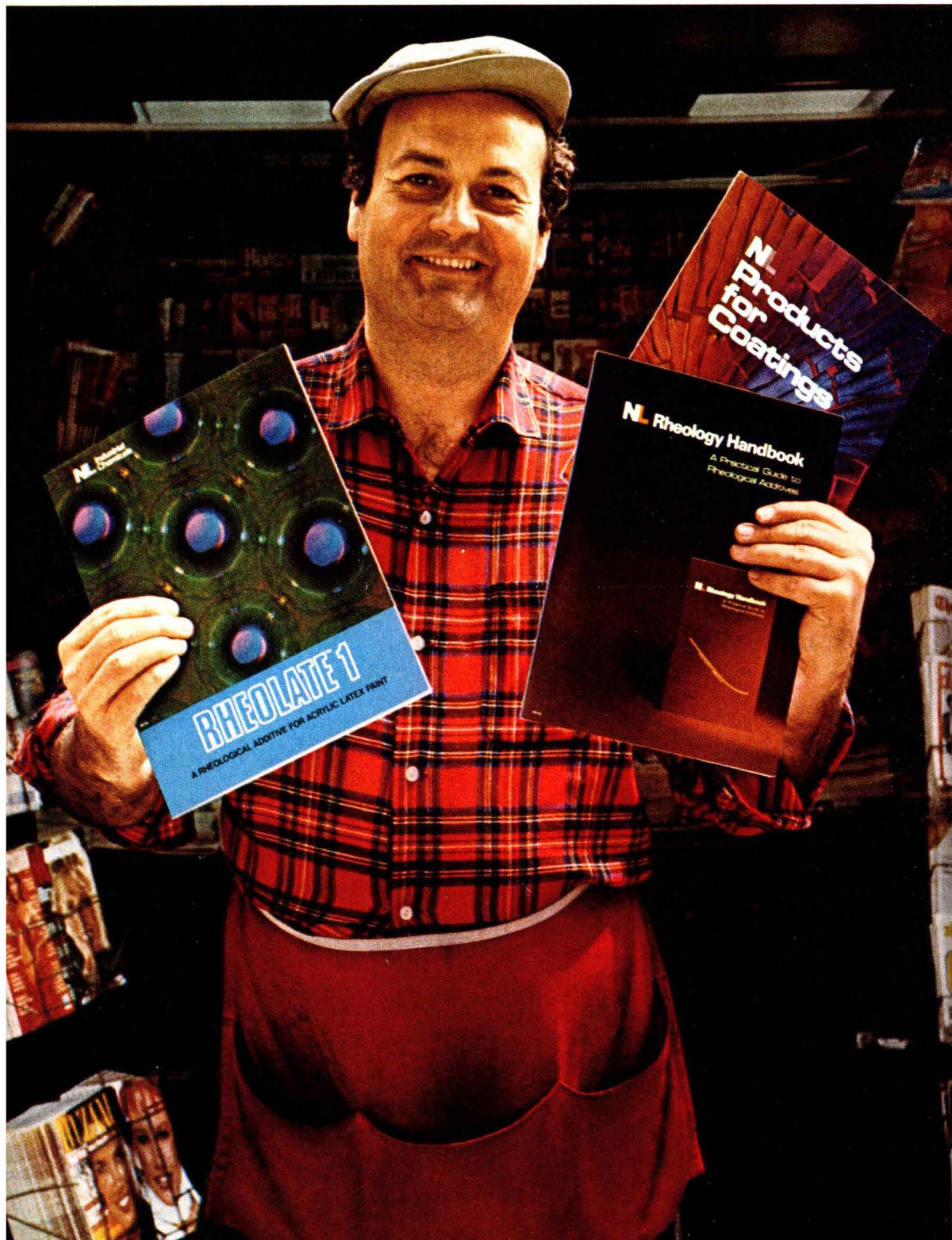
All this will be complemented by the biggest Paint Show ever. To be held concurrently with the Annual Meeting at the Convention Center, the Show will feature the exhibits of 149 supplier firms, who will display their latest developments and have their key technical people on hand for discussions.

Complete details on all the activities are included in the program section of this issue, beginning on page 21.

For the coatings man — or woman — interested in staying on top of fast-changing technical developments, the October 3-5 double feature is a "must see" event.

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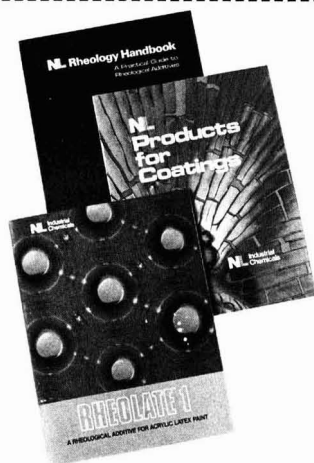
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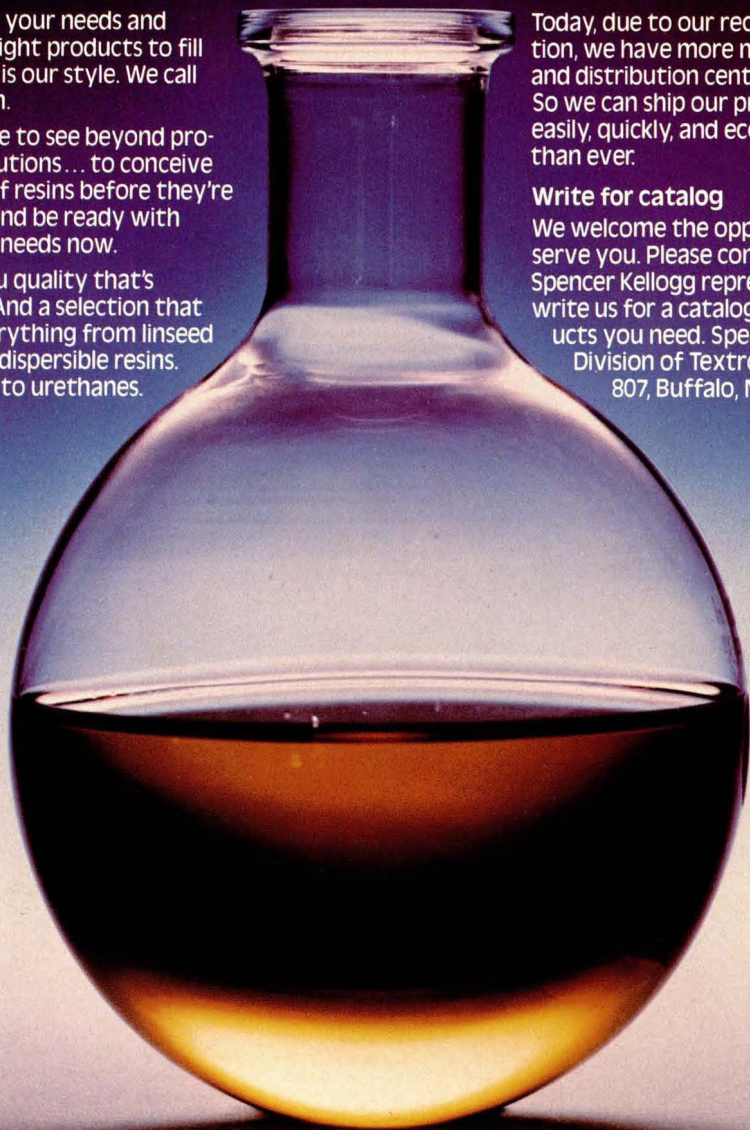
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Abstracts of Papers In This Issue

FACTORS INFLUENCING DRYING PROPERTIES OF A WATER-REDUCIBLE ALKYD—Kansas City Society for Coatings Technology

Journal of Coatings Technology, 51, No. 656, 49 (Sept. 1979)

The drying properties of water-reducible alkyd paints were compared with those of solvent-based paints made from the same resin. White, black, and clear formulas were used. Cobalt neodecanate was the drier. The resin in the water-reducible formulas hydrolyzed rapidly at elevated storage temperatures, but was quite stable at 24°C. The water-based formulas dried significantly slower than their solvent-based counterparts, summarized by $y=1.5x+1.0$, where y is the dry time in hours of the water-reducible and x is that of the solvent-base. Titanium dioxide had no significant effect on either dry times or hardness, while carbon black increased the drying time. The effect of carbon black was more pronounced in the water-reducible system.

REACTION MECHANISM OF MELAMINE RESINS—W.J. Blank

Journal of Coatings Technology, 51, No. 656, 61 (Sept. 1979)

The structure of melamine resins has a significant effect on the mechanism of catalysis. The analysis of the volatile reaction products of amino resins was used as a method to determine the reactions occurring during the crosslinking process. With fully alkylated melamine formaldehyde resins, specific acid catalysis was found to be the dominant crosslinking mechanism. With partially alkylated resins, demethylation and subsequent catalysis by a general acid catalysis mechanism was found to be the crosslinking mechanism. In each case methoxy methyl groups and not methylol groups were found to be the reactive site.

COLORIMETRIC ASSESSMENT OF THE DEGREE OF FLOCCULATION OF INORGANIC COLOR PIGMENTS—S. Keifer

Journal of Coatings Technology, 51, No. 656, 73 (Sept. 1979)

In the case of inorganic color pigments, flocculation causes characteristic shifts in the color effect as compared with the ideally dispersed pigment. The flocculates

are not regarded as coarser particles, but as local areas of higher pigment concentration. This principle makes it possible to establish by experiment a relationship between the pigment concentration—or between the distance apart of the pigment particles—and a selected color value. As a result, it is possible, conversely, to determine the mean concentration in the flocculates with the aid of an existing color value. The relative degree of flocculation derived from this enables—for the first time with inorganic color pigments—information to be obtained about the packing density in the flocculates.

A TANK CLEANING SYSTEM FOR THE SMALL PAINT PLANT—A.G. Morris

Journal of Coatings Technology, 51, No. 656, 81 (Sept. 1979)

Many of the smaller paint plants are cleaning their portable mixing tanks by hand with strong solvents. This practice is dangerous and unhealthy to the person doing the cleaning and the solvent costs are rising.

There is a need for a safe, effective and inexpensive means of cleaning these portable tanks and drums.

Such a system is described, a system that could be installed by most plants' own maintenance people.

FEDERAL SPECIFICATIONS: THE KEY TO GOVERNMENT REQUIREMENTS AND TESTING—R.F. Brady, Jr.

Journal of Coatings Technology, 51, No. 656, 87 (Sept. 1979)

There are approximately 600 federal specifications and standards for paints and coatings. These documents are the key to annual GSA procurements of about \$80 million. Test methods found in specifications are often unique and are not available from any other source. This paper describes the content and coverage of federal specifications, their importance to the coatings industry, and suggestions on how to obtain and use federal specifications. An invitation is extended to manufacturers to participate in the review of draft specifications.



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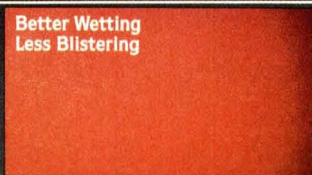
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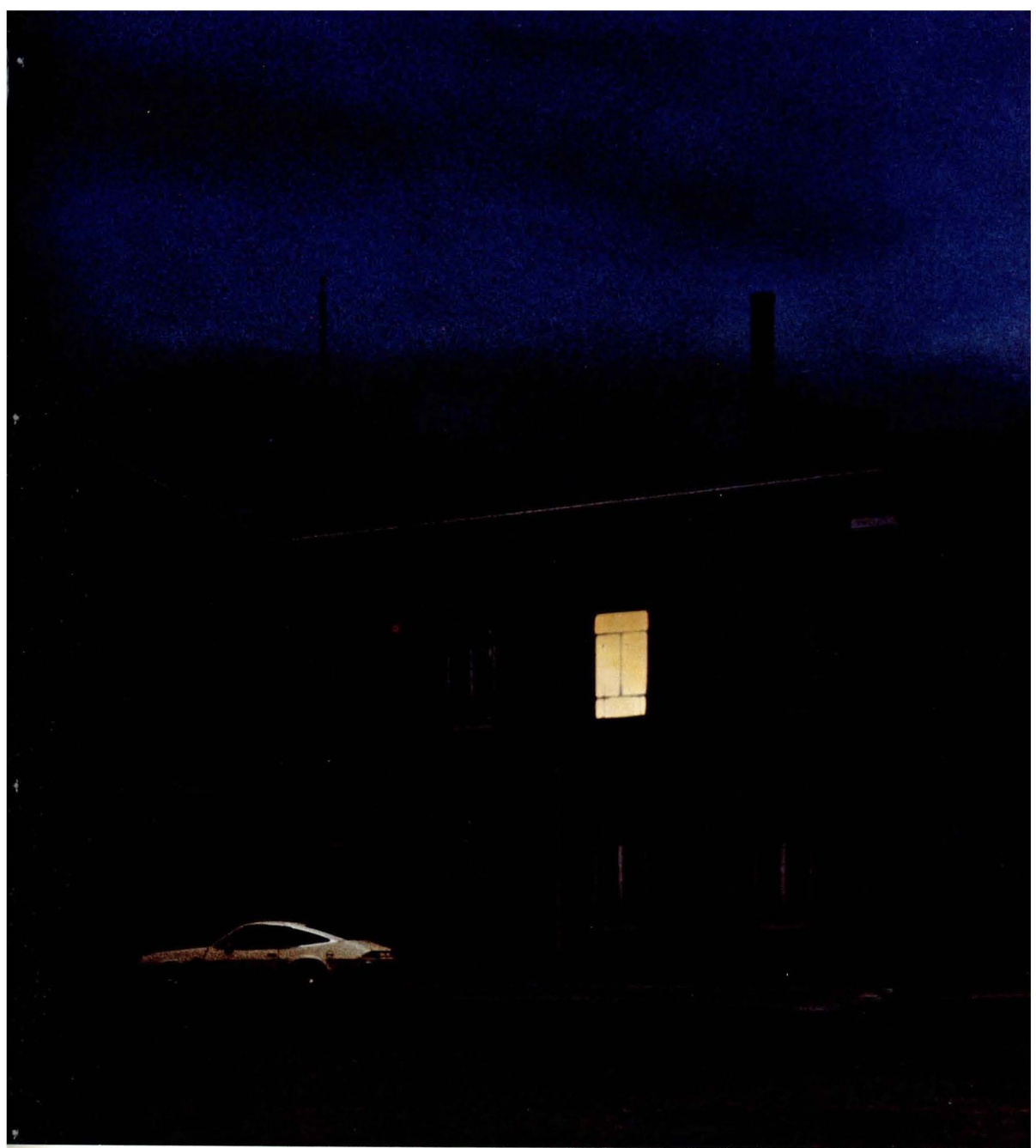
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FEDERATION OF SOCIETIES FOR COATINGS TECHNOLOGY

A large, stylized graphic of the St. Louis Arch, rendered in a light blue color, arches over the central text. The arch is composed of two parallel lines, one slightly offset from the other, creating a sense of depth and shadow.

PROGRAM

**1979 ANNUAL MEETING and
PAINT INDUSTRIES' SHOW**

**ST. LOUIS, MISSOURI
ST. LOUIS CONVENTION CENTER
OCTOBER 3-5**

“PROGRESS THROUGH INNOVATION”

Program

WEDNESDAY, OCTOBER 3

OPENING SESSION (10:00-10:30)

Fifty-seventh Annual Meeting of the Federation of Societies for Coatings Technology opened by President James A. McCormick

Invocation and In Memoriam: Carroll G. Scholle

Welcome: Herbert Rosenblatt, President of St. Louis Society for Coatings Technology

William A. Smith IV, President of Kansas City Society for Coatings Technology

Howard Jerome, Chairman of the Host Committee

Morris Coffino, Chairman of the Program Committee

Deryk R. Pawsey, Chairman of the Paint Industries' Show Committee

Introduction of Federation Officers

Introduction of Distinguished Guests

E. W. FASIG KEYNOTE ADDRESS (10:30-11:30)

Keynote Address by Newscaster and TV Commentator Howard K. Smith

LABORATORY MANAGEMENT AND INNOVATION (2:00-5:00)

INNOVATION: BARRIERS AND INCENTIVES—Milton Glaser, Consultant, Glencoe, Ill.

RESEARCH AND DEVELOPMENT TECHNICAL PROJECT SELECTION—J. M. Butler, of DeSoto, Inc., Des Plaines, Ill.

STRATEGY OF EXPERIMENTATION — Fred C. Chromey, of E.I. du Pont de Nemours & Co., Inc., Wilmington, Del.

IMPACT OF GOVERNMENTAL REGULATIONS ON PRODUCT SAFETY MANAGEMENT — Roy T. Gottesman, of Tenneco Chemicals, Inc., Saddle Brook, N. J.

EFFECTIVE R&D MANAGEMENT — E. Bruce Euchner, of Glidden Coatings and Resins Div., SCM Corp., Strongsville, Ohio

ECOLOGICAL AND HIGH-PERFORMANCE COATINGS (2:00-4:30)

WATER-REDUCIBLE CAN COATING—Peter V. Robinson, of Glidden Coatings & Resins Div., SCM Corp., Strongsville, Ohio

CONDENSATION POLYMER FORMULATION IN THE AGE OF OLIGOMERS — Lawrence H. Brown, of Dow Corning Corp., Midland, Mich.

REACTIVE SOLVENT-DILUENT FOR COATINGS SYSTEMS — W.D. Emmons, K. Nyi, P.R. Sperry, R.J. Wiersema, and J.P. Shelley, of Rohm and Haas Co., Spring House, Pa.

NEW ISOCYANATE FOR HIGH-PERFORMANCE HIGH-SOLIDS URETHANE COATINGS — K.C. Frisch, of University of Detroit, Detroit, Mich., and Gary L. Driscoll, of Sun Petroleum Products Co., Marcus Hook, Pa.

MULTIFUNCTIONAL RESIN COMES OF AGE — Marianne DiBenedetto, of CIBA-GEIGY Corp., Ardsley, N.Y.

Program at a Glance

Tuesday, Oct. 2 Registration (9:00 am to 5:00 pm)

Wednesday, Oct. 3 Registration (8:00 am to 5:00 pm)
Opening Session (10:00 am)
Keynote Address (10:30 am)
Technical Sessions (2:00 to 5:00 pm)

Paint Industries' Show (Noon to 5:30 pm)

Thursday, Oct. 4 Registration (8:00 am to 5:00 pm)
Technical Sessions (9:00 am to Noon) - (2:00 pm to 4:30 pm)
Paint Industries' Show (10:00 am to 5:30 pm)

Friday, Oct. 5

Registration (8:00 am to 3:30 pm)
Technical Sessions (9:00 am to 10:30 am)

Mattiello Lecture (10:30 am)
Technical Session (2:00 pm to 4:00 pm)

Annual Business Meeting (4:00 pm to 5:00 pm)

Paint Industries' Show (10:00 am to 4:00 pm)

Federation Luncheon will be held at 11:45 am on Friday, Oct. 5, at the Sheraton St. Louis Hotel.

Spouses' activities will begin with a Wine and Cheese Social on Wednesday afternoon, Oct. 3, at the Sheraton St. Louis Hotel. Registration for Spouses' program will begin on Tuesday.

PRI SEMINAR ON AQUEOUS COATINGS

(9:00-12:00)

Moderator — Raymond R. Myers, Research Director, Paint Research Institute, Kent, Ohio

OVERVIEW AND INDUSTRY PERSPECTIVE — James R. Erickson, of Glidden Coatings & Resins Div., SCM Corp., Strongsville, Ohio

SYNTHESIS AND CHARACTERIZATION — Loren W. Hill, of North Dakota State University, Polymer and Coatings Dept., Fargo, N. D.

FILM FORMATION PROPERTIES — Raymond R. Myers, PRI Research Director and University Professor, Kent State University, Kent, Ohio.

INTERFACIAL EFFECTS — Irvin M. Krieger, of Case-Western Reserve University, Cleveland, Ohio

SURFACE CHARACTERISTICS OF THE COATINGS — Henry P. Schreiber, of Ecole Polytechnique, Montreal, Quebec, Canada

WASTE MANAGEMENT IN PAINT MANUFACTURE

(9:00-10:30)

Panel discussion focusing on current regulations and how major producers are managing the problems of waste disposal.

Moderator — Gabriel Malkin, Consulting Engineer, Westfield, N. J.; Robert Nelson, of National Paint and Coatings Association, Washington, D. C.; Hugh Williams, of The Sherwin-Williams Co., Cleveland, Ohio; Laurence N. Streff, of PPG Industries, Inc., Springdale, Pa.; and John J. Oberle, of Benjamin Moore & Co., Newark, N. J.

SOLVENT RECOVERY

(10:30-11:00)

WASH SOLVENT RECOVERY WITH WIPED FILM EVAPORATION SYSTEM — Gerald V. DeLaney, of The Pfadler Co., Div. of Sybron Corp., Rochester, N.Y.

SOCIETY PAPERS

(9:30-11:30)

EVALUATION OF LEADING EMULSION ENAMELS AND A COMPARISON TO GOVERNMENT SPECIFICATIONS — Golden Gate Society for Coatings Technology. Presented by William Sawyer, of Sawyer Associates, Hayward, Calif.

CONTINUING STUDY OF NON-MERCURIAL MILDEWICIDES IN ACRYLIC HOUSE PAINTS — Louisville Society for Coatings Technology. Presented by Howard Ramsey, of DuPont Co., Inc., Louisville, Ky.

RELATIONSHIP OF BINDER INDEX TO CRITICAL PIGMENT VOLUME CONCENTRATION, PART I. OBSERVATIONS ON THE EXPERIMENTAL DETERMINATION OF CPVC — Toronto Society for Coatings Technology. Presented by Peter A. Hiscocks, of Canadian Industries, Ltd., Toronto, Ont., Canada

WET ADHESION — FACT AND FICTION — Toronto Society for Coatings Technology. Presented by S. K. Kam-banis, of Reichhold Chemicals Ltd., Weston, Ont., Canada

ROON PAPERS

(2:00-4:30)

POLYPHOSPHAZENES AS NEW COATING BINDERS — A. K. Chattopadhyay, R. L. Hinrichs, and S. H. Rose, of DeSoto, Inc., Des Plaines, Ill.

NATURAL OCCURRENCE AND VOLATILITY OF BENZENE, TOLUENE, AND XYLENE IN PETROLEUM THINNERS — W. H. Ellis and C. D. McLaughlin, of Chevron Research Co., El Segundo, Calif.

REACTIVITY OF ETHERIFIED AMINO RESINS — J. Owen Santer and George J. Anderson, of Monsanto Plastics and Resins Co., Springfield, Mass.

TRANSFER AND LEVELING PROCESSES AND RHEOLOGICAL DESIGN IN WATER-BORNE REVERSE ROLL COATING SYSTEMS — Takehisa Matsuda and William H. Brendley, Jr., of Rohm and Haas Co., Spring House, Pa.

PLASTIC PIGMENT — A NOVEL APPROACH TO MICROVOID HIDING — Alexander Ramig, Jr. and F. Louis Floyd, of Glidden Coatings & Resins Div., SCM Corp., Strongsville, Ohio

DISPERSION AND PHOTO-RESPONSIVE COATINGS

(2:00-4:00)

HYDROGEN BONDING — KEY TO DISPERSION? — Hilton G. Stephen, of Kemrez Chemicals, Cheltenham, Victoria, Australia. (Presented on behalf of the Oil and Colour Chemists' Association-Australia.)

DISPERSION OF TiO₂ IN WATER-BORNE INDUSTRIAL FINISHES — J. M. Rackham, of BTP Tioxide Limited, Stockton-on-Tees, Cleveland, England. (Presented on behalf of the Oil and Colour Chemists' Association — U.K.)

COATINGS FOR RELIEF PATTERNS — Ari Aviram, of IBM Corp., Thomas J. Watson Research Center, Yorktown Heights, N.Y.

APPLICATION OF PHOTOCHEMISTRY TO COATING PROBLEMS — Claude Loucheux, of Universite des Sciences et Techniques de Lille, Villeneuve D'Ascq, France. (Presented on behalf of FATIPEC: Federation of Associations of Technicians in the Paint, Varnish, Lacquer, and Printing Ink Industries of Continental Europe.)

SOCIETY PAPERS

(2:00-4:00)

A SURVEY OF VARIOUS FACTORS THAT AFFECT THE CORROSION PROTECTION OF COATINGS ON STEEL — New England Society for Coatings Technology. Presented by John E. Fitzwater, Jr., of Polyvinyl Chemical Industries, Wilmington, Mass.

AN EVALUATION OF INORGANIC SALTS AS FLASH RUST INHIBITORS IN AN AQUEOUS ACRYLIC COATING — New England Society for Coatings Technology. Presented by Robert Warren, of Cadillac Paint & Varnish Co., Boston, Mass.

AN EVALUATION OF WATER-BORNE, AIR-DRY, FORCE-DRY, LEAD- AND CHROMATE-FREE METAL PRIMER — Northwestern Society for Coatings Technology. Presented by Don Emch, of Valspar Corp., Minneapolis, Minn.

A MICROBIOLOGICAL AUDIT — Philadelphia Society for Coatings Technology. Presented by Michael McGroarty, of DeSoto, Inc., Pennsauken, N.J.

FRIDAY, OCTOBER 5

VALUE OF CONTINUING EDUCATION (9:00-10:30)

Panel discussion of the role of continuing education for coatings personnel from the viewpoint of industry and academia.

Moderator — John A. Gordon, of University of Missouri, Rolla, Chemistry Dept., Rolla, Mo.

Norma Fleming, of University of Missouri, Rolla, Extension Div., Rolla, Mo.

Carl Knauß, of Kent State University, Chemistry Dept., Kent, Ohio

Len Afremow, of DeSoto, Inc., Des Plaines, Ill.

COLOR AND PIGMENTATION (9:00-10:30)

COLOR VISION AND COLORIMETRY — Gunter Wysocki, of National Research Council of Canada, Ottawa, Ontario, Canada

USEFULNESS AND LIMITATIONS OF KUBELKA-MUNK THEORY FOR INCOMPLETE HIDING PAINTS — Ruth Johnston-Feller, of Center on the Materials of the Artist and Conservator, Mellon Institute of Research, Carnegie-Mellon University, Pittsburgh, Pa., and Dennis Osmer, of CIBA-GEIGY Corp., Ardsley, N.Y.

PIGMENT SYNERGISM AND PIGMENT ANTAGONISM IN AUTOMOTIVE PIGMENTATION — Sol Panush, of Celanese Polymer Specialties Co., Detroit, Mich.

MATTIELLO LECTURE (10:30-11:45)

PHYSICAL CHEMICAL MODELS FOR ORGANIC PROTECTIVE COATINGS — Charles Kumins, Director of Research and Development, Tremco Incorporated, Cleveland, Ohio

FEDERATION LUNCHEON (11:45)

Grand Ballroom, Sheraton St. Louis Hotel

MANUFACTURING COMMITTEE SEMINAR ON MEDIA MILLING (2:00-4:00)

Panel discussion on various types of milling equipment and their application to the coatings industry.

Moderator—Gene LeVea, of Pratt & Lambert, Inc., Buffalo, N.Y.

Roy Nelson, of Epworth Manufacturing Co., Inc., South Haven, Mich.

Myron Segal, of Premier Mill Corp., New York, N.Y.

Leo Dombrowski, of Chicago Boiler Co., Chicago, Ill.

ANNUAL BUSINESS MEETING (4:00-5:00)

Annual Business Meeting of the Federation

Presentation of these Awards: American Paint & Coatings Journal/A. F. Voss . . . Materials Marketing Associates . . . Program Committee . . . Armin J. Bruning . . . Dry Color Manufacturers' Association . . . Roon Foundation . . . Ernest T. Trigg.

Installation of Officers, 1979-80.



The Cervantes (St. Louis) Convention Center, shown above, is the site of the Federation's 1979 Annual Meeting and Paint Show



Mississippi River excursion is part of the Spouses' Program on Thursday in St. Louis. The "Admiral" (above), at 346 feet long, is the world's largest river excursion boat.

OTHER CONVENTION INFORMATION

PAINT INDUSTRIES' SHOW

The 44th Annual Paint Industries' Show will run concurrently with the Annual Meeting in the St. Louis Convention Center. The only national exposition of materials and equipment used in the formulation, testing, and manufacture of coatings, the Show will be open from 12:00 to 5:30 pm on Wednesday, October 3; 10:00 am to 5:30 pm on Thursday, October 4; and 10:00 am to 4:00 pm on Friday, October 5.

Participating supplier companies will have their top technical representatives on hand to discuss the latest developments with registrants at this year's event, the largest in Paint Show history.

REGISTRATION

Registration fees for the Annual Meeting and Paint Show are on a member and non-member basis:

	Member	Non-Member	Spouses
Advance*	\$35	\$50	\$20
In St. Louis	\$40	\$55	\$25
One-Day	\$20	\$30	—

* Special for retired members and their spouses only: Advance registration will be \$15 each.

Registration form has been mailed to all members.

Note: Purchase of luncheon tickets is optional for both advance and on-site registrations.

LUNCHEON

The Federation Luncheon will be held on Friday, October 5, at the Sheraton St. Louis Hotel.

Presentations will be made to recipients of the George Baugh Heckel Award (outstanding individual who has contributed to the advancement of the Federation) and the Flynn Awards (firms judged to have the best exhibit booths in the 1979 Paint Industries' Show).

Featured speaker will be Dr. James H. Boren, President of the International Association of Professional Bureaucrats (known as INATAPROBU) who is the author of the best-selling "When In Doubt, Mumble."

SPOUSES' PROGRAM

A schedule of activities has been planned each day for spouses attending the Annual Meeting, and a Hospitality Room will be maintained at the Sheraton St. Louis Hotel.

A get-acquainted Wine and Cheese Social is scheduled for Wednesday afternoon.

On Thursday, a continental breakfast will precede an all-day tour of the city.

Continental breakfast will be available again on Friday morning.

Registration fees for the Spouses' activities are \$20 in advance and \$25 on-site.

HEADQUARTERS HOTEL

The Sheraton St. Louis will be headquarters hotel, and Stouffer's Riverfront Towers will be co-headquarters. Other hotels with blocks of rooms set aside for the Annual Meeting are Bel Air Hilton, Breckenridge Pavilion, Holiday Inn

Riverfront, Holiday Inn Downtown, Lennox, Mayfair, Rodeway Inn, and St. Louisian.

Shuttle bus service will be provided between participating hotels and the Convention Center.

ROOM RESERVATIONS

All requests for rooms and suites must be sent to the Federation Housing Bureau on the official form provided by the Federation. These have been mailed to all members and additional forms are available from Federation headquarters.

GROUND TRANSPORTATION

Limousine transportation from the airport to downtown St. Louis is available on a regular basis. One-way fare is \$4.00; round-trip is \$7.00. Service is every 20 minutes.

Taxi fare from the airport to downtown (for two or more persons) is \$5-\$6 each.

BOARD MEETING

The Fall Board Meeting of the Federation will be held on Tuesday, October 2, at the Sheraton St. Louis Hotel.

SPEAKERS' BREAKFASTS

A breakfast and briefing for each day's program participants will be held in the Convention Center on Wednesday, Thursday, and Friday.

PUBLICATION OF PAPERS

The JOURNAL OF COATINGS TECHNOLOGY has prior rights to the publication of all papers presented at the Annual Meeting.

PROGRAM STEERING COMMITTEE

Chairman — Morris Coffino, of D. H. Litter Co., Inc., New York, N. Y.; *Vice-Chairman* — Hugh Lowrey, of Indurall Coatings, Inc., Birmingham, Ala.; Umberto Ancona, of McCloskey Varnish Co., Philadelphia, Pa.; Thomas Ginsberg, of Union Carbide Corp., Bound Brook, N. J.; William Mirick, of Battelle Memorial Institute, Columbus, Ohio; Colin D. Penny, of Hampton Paint Mfg. Co., Inc., Hampton, Va.; and Richard E. Wolf, of DeSoto, Inc., Des Plaines, Ill.

MEETINGS COMMITTEE

Members of the Kansas City and St. Louis Societies are serving on the Meetings Committee under General Chairman Howard Jerome, of Spatz Vane Calvert Paint Co. Chairing the various subcommittees are: Information Services—William Fitzpatrick, of Cook Paint & Varnish Co.; Operations—John Folkerts, of Plas-Chem Coatings; Luncheon—William Smith IV, of Conchemco, Inc.; Publicity—Herman J. Larson, of Poly Chem Resins, Inc.; and Busing—Floyd Thomas, Jr., of Thomas & English, Inc.

Mrs. Howard (Gene) Jerome is in charge of the Spouses' Program.

ABSTRACTS OF PAPERS

WATER-REDUCIBLE CAN COATING

Peter V. Robinson, of Glidden Coatings & Resins Div., SCM Corp.

A number of methods of synthesizing hydrolysis resistant water-reducible polymers are described. In particular, polymer grafting techniques are explored and the application of some of these techniques to the synthesis of water-reducible polymers for use in contact with food is described. It is shown that in order to obtain the total balance of properties necessary for container coatings, significant levels of grafting to a main polymer backbone are necessary.

NEW ISOCYANATE FOR HIGH-PERFORMANCE HIGH-SOLIDS URETHANE COATINGS

K.C. Frisch, of University of Detroit and G.L. Driscoll, of Sun Petroleum Products Co.

Energy costs and environmental restrictions are rapidly moving the coatings industry in the directions of high solids content and/or water-based vehicles. Polyurethane finishes based on aliphatic diisocyanates are one type of high-solids system noted for giving high-performance protective coatings. The diisocyanate (BDI) derived from 1,4-bis(amino-methyl)cyclohexane is being developed for use in these systems. This structure was chosen from considerations of expected coatings performance, raw materials availability, and cost-effectiveness. Data are presented to show that BDI is as reactive as expected and gives clear, tough, solvent-resistant, non-yellowing, corrosion-resistant coatings. Performance of representative formulations are described. Basic physical properties, including toxicity, necessary for proper formulation are described in detail.

REACTIVE SOLVENT-DILUENT FOR COATINGS SYSTEMS

W.D. Emmons, K. Nyi, P.R. Sperry, R.J. Wiersema, and J.P. Shelly, of Rohm and Haas Co.

A new reactive solvent diluent (dicyclopentenylloxyethyl methacrylate) has been developed which offers a number of approaches to new high solids coatings. The chemistry of this

monomer is described in detail and its utility with alkyds, polyesters, chlorinated rubber, and as a convertible latex coalescent is described.

CONDENSATION POLYMER FORMULATION IN THE AGE OF OLIGOMERS

Lawrence H. Brown, of Dow Corning Corp.

A review of the theoretical concepts of Carothers and Flory, applied by Kilb, Bernardo and Bruins, Patton, Finney, Earhart and others, shows that condensation polymers for solution coatings can be formulated according to incipient gelation criteria with reasonable assurance of success.

Today, with emphasis on oligomeric polymers for high solids, water-dispersible and powder coatings, the former concepts have less value.

For polyesters, Patton's constant ($K = Mo/Ea$) becomes less meaningful than theoretical \bar{M}_n and EW_{OH} (Weight of polymer per excess carbinol group). Reactions of the base polyesters with alkoxy-functional melamines, siloxanes or other modifiers during cure have changed the precepts on which formulations are based. This is true especially for the current rapid cures required for pre-finished metal.

Application of the new criteria is facilitated by equations which can be handled by programmable calculators or computers. Modification by silicone oligomers has been reduced to a mathematical treatment, and examples show the utility of the method.

THE MULTIFUNCTIONAL RESIN COMES OF AGE

Marianne DiBenedetto, of CIBA-GEIGY Corp.

Multifunctional resins, such as epoxy cresol and epoxy phenol novolacs and the tetraglycidylether of MDA, have long found utility in the molding, casting and electrical areas of application. In combination with anhydrides or aromatic amine curing agents, these resins were traditionally heat cured to form networks of extremely high crosslink density. This phenomenon was responsible for the high heat deflection temperatures and the excellent chemical resistance properties for which such systems were and still are known.

Today, however, the use of multifunctional resins need not be restricted to applications which require high gelation and post-cure temperatures. Through careful formulation, systems based on multifunctional resins show promise in room temperature cured coatings applications. Since they possess the same high crosslink density as their casting and molding counterparts, these r.t. cured coatings systems should afford excellence in the areas of chemical resistance and surface hardness.

Numerous application possibilities for such systems exist in the areas of maintenance and marine coatings.

INNOVATION - BARRIERS AND INCENTIVES

Milton Glaser, Consultant

Students of the R&D Innovation Process have learned much which can help increase the Innovative output of our laboratories. We can improve R&D efficiency both by removing the negative factors — the barriers, and by activating the positive factors — the incentives.

“Barriers” include such factors as: absence of an atmosphere conducive to innovation; perceived indifference of top management; imposition of avoidable manufacturing and sales problems on R&D; remuneration problems, failure to properly train R&D and Quality Control Managers; failure to adequately monitor the technical-marketing interface; failure to properly exercise the “champion” function, etc.

“Incentives” include such factors as: good communications of R&D with sales/marketing; removing causes of R&D employee hostility; improving the R&D/customer interface; understanding the important role of exploratory research; recognition and rewards for outstanding R&D achievements; removing the “barriers” cited above, etc.

All segments of a company, including the executives, the sales and marketing staff, the manufacturing and engineering personnel, the financial and accounting people and the production workers at all levels — can help or hinder the R&D innovation processes. Innovation audits by means of the “Glaser Innovation Index” model are helpful in evaluating commercially-oriented R&D projects before they are initiated (to be assured that they are viable) and periodically thereafter (to be assured that they are progressing satisfactorily).



H.K. Smith



P.V. Robinson



L.H. Brown



W.D. Emmons



G.L. Driscoll

RESEARCH AND DEVELOPMENT TECHNICAL PROJECT SELECTION

J. M. Butler, of DeSoto, Inc.

The division of management science defined as “research management” is, relative to its marketing, financial, and manufacturing counterparts, underdeveloped. Consequently, the selection of research and development projects may be the most difficult of all the managerial tasks. This difficulty is caused by the lack of initial “hard” data, such as knowledge of technical feasibility, marketing research forecasts, and availability of appropriate manufacturing capabilities.

Obtaining a clear definition of marketing and business goals for the company is the first step in project selection. Without these goals as guidelines, most projects will not ultimately contribute to additional corporate profitability. Projects may be developed through a top-to-bottom or a bottom-to-top approach. The former will produce more diversification technology while the latter will produce “pet” and highly directed projects. Every research project should satisfy one of the following corporate needs: production cost reduction, product utility enhancement; customer’s operating costs reduction; new product creation; and corporate technology enhancement. Research and development costs must be accurately monitored if the profitability of a given technology is to be determined.

Finally, project termination at the appropriate time is important because heavy financial losses may be incurred if unproductive efforts are allowed to continue indefinitely.



M. Glaser



J.M. Butler



F.L. Chromeý



R.T. Gottesman



E.B. Euchner



L.W. Hill



R. Myers



G. Malkin



R.J. Nelson

STRATEGY OF EXPERIMENTATION Fred L. Chromey, of E.I. du Pont de Nemours & Co., Inc.

A brief description of the pedagogical approach and content of du Pont's popular 2½ day workshop/seminar.

The course is based upon the use of simulators to teach response surface methodology, the most efficient technique known for investigating a system's response to changes deliberately made in the controlled variables.

IMPACT OF GOVERNMENTAL REGULATIONS ON PRODUCT SAFETY MANAGEMENT Roy T. Gottesman, of Tenneco Chemicals, Inc.

The interactions necessary between Research and Development, Engineering, Manufacturing, Marketing, and the Health and Safety professionals during product development are described. The impacts of the Occupational Safety and Health Act as well as environmental regulations promulgated under the Clean Air Act of 1977, the amendments to the Clean Water Act, and those proposed under the Resource Conservation and Recovery Act on new product development and manufacturing are discussed. A novel approach developed for management of product safety which includes Material Safety Data Sheets, proper product labeling, and appropriate technical literature to minimize product liability exposure are discussed.



L.N. Streff



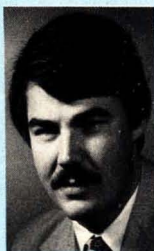
J.J. Oberle



W. Sawyer

INTRODUCTION TO PRI RESEARCH ON AQUEOUS COATINGS James R. Erickson, of Glidden Coatings and Resins, Div. of SCM Corp.

The technology of aqueous coatings has developed well beyond the point where most of the underlying scientific concepts and descriptions are accurately known. This situation tends to jeopardize the efficient advancement of the technology. To address this industry problem, the Paint Research Institute (PRI) currently is administering grants to perform fundamental research on the physical chemistry of water-soluble-dispersible (WSD) polymers and latex dispersions. In the WSD work, the individual academic researchers study standardized model systems which were identified with the help of the PRI Aqueous Coatings Committee of industrial scientists. The present talk describes some of the scientific problems faced in the aqueous coatings field, discusses the identification and selection of suitable model WSD polymers, and suggests how current PRI research may impact the development technology.



G.V. DeLaney



W.H. Ellis

VISCOSITY CONTROL IN WATER-SOLUBLE COATINGS Loren W. Hill, of North Dakota State University

Acrylic copolymers containing amine neutralized carboxyl groups exhibit viscosity plateaus or maxima when cosolvent rich solutions are diluted with water. Variations in dilution curves (log η vs. % polymer) with carboxyl content, amine selection and amount, copolymer molecular weight, and cosolvent content are presented. Changes in pH and viscosity during amine titration, shear rate dependence, and dilution



C.D. McLaughlin



W.H. Brendley



T. Matsuda

results are analyzed in terms of formation of polymer aggregates. Practical aspects of viscosity control such as extreme sensitivity to small changes in composition, volume solids at spray viscosity, and the effect of changes in cosolvent/water ratio on sagging tendency are discussed.

SEMINAR ON WASTE MANAGEMENT IN PAINT MANUFACTURE

Discussions focus on current regulations and how major producers are managing the problems of waste disposal.

Up-to-date information is provided on the regulations governing disposal of water waste, solvent wastes, and solid wastes. Then, panel of industry experts describes various waste treatment and disposal methods based on actual experience. Audience will have opportunity to discuss individual waste management problems.

WASH SOLVENT RECOVERY WITH WIPE FILM EVAPORATOR SYSTEM

Gerald V. DeLaney, of Pfaudler Co., Div. of Sybron Corp.

Wiped Film Evaporators (WFE) have been utilized by the food and chemical industries for over 20 years. This paper looks at the application of the Wiped Film Evaporator to the coatings industry. The paper describes what WFE's are and how they differ from conventional distillation techniques. The advantages and disadvantages of these differences for solvent recovery are discussed. Also discussed are results of the distillation of a variety of solvents.

EVALUATION OF LEADING EMULSION ENAMELS AND A COMPARISON TO GOVERNMENT SPECIFICATIONS

Golden Gate Society for Coatings Technology

Ten of the leading companies that supply the national market submitted samples of their best gloss and semi-gloss emulsion paints. The products were then tested using Federal Specification TT-P-1511A as the guide procedure. Members were urged to also evaluate these enamels by a series of procedures, not described in the specifications but which they felt could be of value in appraising the basic properties of these gloss finishes. Out of this mass of data, the committee submitted: (1) a list of test procedures for Spec TT-P-1511A that were to be accepted, modified, or deleted; (2) a series of tests that they recommended as guides for enamel evaluations.

The reasons for deletion and modifications for the Spec Procedures are thoroughly discussed and reasons for deletion or modification clearly stated. Where new procedures are adopted, these are carefully described in a step-by-step procedures report. Many procedures that were thought could be of value but proved to be of no merit are also included. This entire approach may set an entirely new method for approving new government specifications.

CONTINUING STUDY OF NON-MERCURIAL MILDEWCIDES IN ACRYLIC HOUSE PAINTS **Louisville Society for Coatings Technology**

The use of mercury-derived compounds as mildewcides was banned from solvent-base coatings in 1976. This action cast doubt on the continuing use of mercury in water-base coatings. Because of this, non-mercurial mildewcides have become a matter of intense interest, prompting a continuing exposure study of some of the non-mercurial mildewcides currently on the market. This is a study of acrylic latex house paint systems using a different non-mercurial with an alkyd primer and self-primer.

The non-mercurial mildewcides were compared to a mercury control and exposed under various climatic conditions. A cost comparison of the systems under study was made.

POLYPHOSPHAZENES AS NEW COATING BINDERS

A.K. Chattopadhyay, R.L. Hinrichs, and S.H. Rose, of DeSoto, Inc.

Many polymeric materials currently in use are extremely flammable and generate large amounts of toxic fumes during combustion. Although the incorporation of low molecular weight flame-retardant additives decreases the flammability hazards, they usually increase the generation of smoke and toxic products. A new family of polymers based on a phosphorous-nitrogen backbone, the polyphosphazenes, is being developed to replace existing polymers to alleviate flammability and toxicity hazards. The poly(aryloxyphosphazenes) have a high degree of flame-retardance and also have desirable polymeric properties for application as paint binders.

A series of poly(aryloxyphosphazenes) with limiting oxygen indexes between 27 and 35 were prepared and formulated into solvent-based paints. These paint formulations demonstrated very good coating performance characteristics. Adhesion, hardness, flexibility, and impact and abrasion resistance properties were comparable to a chlorinated alkyd paint. The limiting oxygen indexes of poly(aryloxyphosphazene) films were usually higher than the alkyd paint films.

NATURAL OCCURRENCE AND VOLATILITY OF BENZENE, TOLUENE, AND XYLENE IN PETROLEUM THINNERS

W. H. Ellis and C. D. McLaughlin, of Chevron Research Co.

Benzene, toluene, and xylene are of increasing concern to regulatory agencies because of potential acute and chronic health effects. Gas chromatographic analyses of fractions from laboratory fractional distillations showed that occurrence of an aromatic compound in a solvent depends upon its percentage in the original crude oil, the solvent distillation range, and distillation efficiency. Evaporation experiments and analyses of vapor and liquid in equilibrium showed that volatility is sometimes influenced by molecular interactions.

Special analytical techniques were required to determine benzene, toluene, and xylene concentrations in mixtures with other hydrocarbons.

REACTIVITY OF ETHERIFIED AMINOPLAST RESINS

J. Owen Santer and George J. Anderson, of Monsanto Plastics and Resins Co.

An important trend within the coatings industry is to use formulations which cure at lower temperatures and/or in shorter times. For aminoplast-crosslinked thermosetting formulations, improved cure response can be achieved by modifications of both the aminoplast and the primary film-former (vehicle). This paper discusses the effect of such modifications and describes experiments in which aminoplast resins were reacted with hydroxyl-functional model vehicles in order to gain insight into the effective functionality/reactivity of the various aminoplasts and to indicate routes towards more efficient low temperature cure.

TRANSFER AND LEVELING PROCESSES AND RHEOLOGICAL DESIGN IN WATER-BORNE REVERSE-ROLL COATING SYSTEMS

Takehisa Matsuda & William H. Brendley, of Rohm and Haas Co.

The entire coating dynamics (ribbing, transfer, and leveling processes) of water-borne reverse-roll coating systems were studied. The surface profilometric and high-speed photographic studies clearly demonstrated that the ultimate surface appearance is directly associated with the ribs (flow lines) which originate at the applicator and pick-up rolls. In viscoelastic coatings, the rib-number (or spacing of ribs) is primarily determined by the nip clearance between the applicator and pick-up rolls. The transfer characteristics of coatings determines the nip clearance required for a given film thickness. It was found that high-shear rate viscosity (HSV) at 10^4 sec^{-1} determines the transfer process and simultaneously the surface waviness. On the other hand, low-shear rate viscosity (LSV) controls primarily the leveling process. The ratio of HSV to LSV, defined here as a Rheology Index, was found to be a useful tool for predicting the ultimate surface appearance: a higher Rheology Index substantially provides better flow and leveling properties. The equation of coating transfer in reverse-roll coating systems which was empirically derived, is composed of two individual terms: operating variables (three roll-speeds and nip clearance) and material constants (HSV, surface tension and volume solids). This equation predicts well the roll-speeds dependence of flow and leveling properties. The anomalous ribbing phenomenon observed in viscoelastic coatings is also discussed.

PLASTIC PIGMENT — A NOVEL APPROACH TO MICROVOID HIDING

Alexander Ramig, Jr. and F. Louis Floyd, of Glidden Coatings Resins Div. of SCM Corp.

A novel approach for improving the cost-performance balance of latex paints has been developed. The new approach, referred to as "plastic pigment", involves the use of sub-micron size, non-film-forming polymer particles prepared by emulsion polymerization. Paint films containing plastic pigment have been examined using conventional techniques such as contrast ratio, enamel holdout and ink holdout; and by mercury penetration and scanning electron microscopy. Proper use of plastic pigment improves opacity without a corresponding degradation in holdout and other properties. Opacity is thought to be enhanced by the presence of micro-

voids in the resulting paint films, due to the packing of the non-film-forming particles.

Conventional latex paints exhibit an abrupt change in many properties vs. PVC as the critical region is encountered (CPVC). For opacity and porosity, the CPVC's are essentially coincident, within experimental error. Paints containing non-film-forming latex particles ("plastic pigment") exhibit widely diverging CPVC's for opacity and porosity, the latter being named the point of "Critical Surface Porosity" (CSP) to avoid confusion. This difference allows one to formulate a higher hiding paint at a given TiO_2 level than possible with conventional approaches, while retaining other properties. The net result is a 30% reduction in TiO_2 required to achieve the same opacity as a conventionally-formulated paint. The effects of binder particle size plus plastic pigment particle size, polydispersity, composition and hardness on performance are described. Additional comments on formulating parameters are offered, plus a prototype formula illustrating the plastic pigment concept.

HYDROGEN BONDING - KEY TO DISPERSION

Hilton G. Stephen, of Kemrez Chemicals

The Hansen, Burrell and Sorensen approaches are summarized and illustrated.

The effect of hydrogen bonding series on the relationship of pigment/binder/solvent is examined over a wide range of colors, particularly titanium dioxide.

The hypothesis is presented that the relationship between pigment and solvent may well be more important than that between pigment and resin, during the dispersion process, film formation, and retained can stability.

Attention is drawn to the need to develop the relevant correct solvent bases to allow maximum use of the pigment-binder relationship by avoidance of unnecessary flocculation.

DISPERSION OF TiO_2 IN WATER-BORNE INDUSTRIAL FINISHES

J. M. Rackham and T. Entwistle, of BTP Tioxide Ltd.

The dispersion stability of pigments in organic solvent-based paints is fairly well understood. The theory is relatively simple although the desired results may not always be fully obtained in practice. The attainment of dispersion stability in water-borne industrial paints is much more difficult and at present is not very well documented. This paper discusses some of the factors involved and shows how the proposed mechanisms can explain some of the experimental results obtained.

COATINGS FOR RELIEF PATTERNS

A. Aviram, of IBM T. J. Watson Research Center

Two materials that expand due to exposure to light up to 35% in linear dimensions were synthesized. These materials are polymeric, based on polyglutamic acid and polyacrylic acid.

The phenomenon stems from mechanochemistry in combination with principles of photochemistry. Basically, the primary event is an induced photoionization that triggers a deformation.

In free standing films the deformation is in three dimensions. However, when the films are chemically bound to solid surfaces, the surface dimensions are not free to deform. Therefore, exposure of such films to light patterns results in relief images.

APPLICATION OF PHOTOCHEMISTRY TO COATING PROBLEMS

Claude Loucheux, of Laboratoire de Chimie Macromoleculaire

The main principles which rule the uses of photochemistry in macromolecular chemistry are given. Then a discussion is proposed in order to consider the applications of photochemistry to coatings technology. This discussion is managed taking into account scientific, technological, and economic points of view.



A. Ramig



H.G. Stephen



J.M. Rackham

SURVEY OF VARIOUS FACTORS THAT AFFECT THE CORROSION PROTECTION OF COATINGS ON STEEL

New England Society for Coatings Technology

The key factors which influence the corrosion of coatings on steel are water permeability, oxygen permeability, ion permeability, the adhesion of the coating under wet conditions, and the resistance of the coating to alkali. Understanding these basic factors is important when designing aqueous coatings.

This paper is a survey of the factors that affect the corrosion resistance of coatings on steel and provides essential background information for a series of papers to be presented by the New England Society for Coatings Technology on "The Design of Aqueous Coatings for the Corrosion Protection of Steel Substrates."



A. Aviram



S.K. Kambanis



J.A. Gordon

AN EVALUATION OF INORGANIC SALTS AS FLASH RUST INHIBITORS IN AN AQUEOUS ACRYLIC COATING

New England Society for Coatings Technology

Inorganic salts are evaluated at different levels in an aqueous acrylic coating for flash rust resistance. Water immersion tests were performed on coated steel panels to determine the effect on water resistance. Salt spray resistance was also evaluated to determine performance. Many inorganic salts were found which improved the coating's resistance to flash rusting. Very few inorganic salts were found which contributed to flash rust resistance and did not adversely affect water resistance and salt spray resistance.



L. Afremow



G. Wyszecski



S. Panush

COLOR VISION AND COLORIMETRY

Gunter Wyszecski, of National Research Council of Canada

A brief review is given of the structure of the human eye and its mechanisms governing color vision. This is followed by a discussion of those characteristics of the visual mechanism which form the basis for colorimetry. In particular, the basic laws of matching additive mixtures of color stimuli are discussed and how valid they are when the conditions of observation vary, for example, with regard to field size and level of illumination. The restricted validity of the basic laws of color matching limits the usefulness of current colorimetric procedures in some practical situations.



R. Johnston-Feller



D. Osmer



C. Kumins

USEFULNESS AND LIMITATIONS OF KUBELKA-MUNK THEORY FOR INCOMPLETE HIDING PAINTS

**Ruth Johnston-Feller, of Carnegie-Mellon
University, and Dennis Osmer, of CIBA-GEIGY
Corp.**

The availability of incomplete-hiding computer programs in conjunction with the normal complete-hiding computer-color-matching programs suggests that it would be well to consider the status of the usefulness, as well as the limitations, of the application of Kubelka-Munk incomplete-hiding theory to paint systems. A review of the literature and of previous experience with the application of the theory to many other material systems, such as textiles, paper, and different plastics types, reveals that the theory works well - predicting color and spectral curves - under a variety of conditions of contrast ratio, substrate color, etc. In solvent-thinned paint systems, however, either those that dry by solvent evaporation or those that are thermally polymerized, accuracy of predicted results has been found to be dependent on contrast ratio - that is, on PVC and thickness - as well as on measurement and computation methods. The purpose of this paper is to review the past literature on hiding power determinations and incomplete-hiding color predictions, to point out

the lack of data on spectral curve shape predictions for both non-chromatic and chromatic paints, and to describe the parameters necessary for obtaining meaningful results with calculations based on incomplete-hiding Kubelka-Munk theory in solvent-containing paint systems.

PIGMENT SYNERGISM AND PIGMENT ANTAGONISM IN AUTOMOBILE PIGMENTATION

Sol Panush, of Celanese Polymer Specialties Co.

Synergism and antagonism are critical to automotive pigmentation where requirements are most stringent. Pigment Synergism: The phenomenon where the blend of weak pigments yields a strong acceptable system. Pigment Antagonism: The phenomenon where the blend of acceptable pigments yields a weak unacceptable system.

Since the most expensive and chemically sophisticated pigments are required to meet the ultimate performance requirements of automotive pigmentation, the understanding of the unusual phenomena of pigment synergism and pigment antagonism will yield rich rewards in both cost savings and aesthetic possibilities.

1979 PAINT INDUSTRIES' SHOW

EXHIBITORS

St. Louis Convention Center
St. Louis, Missouri
October 3, 4, 5

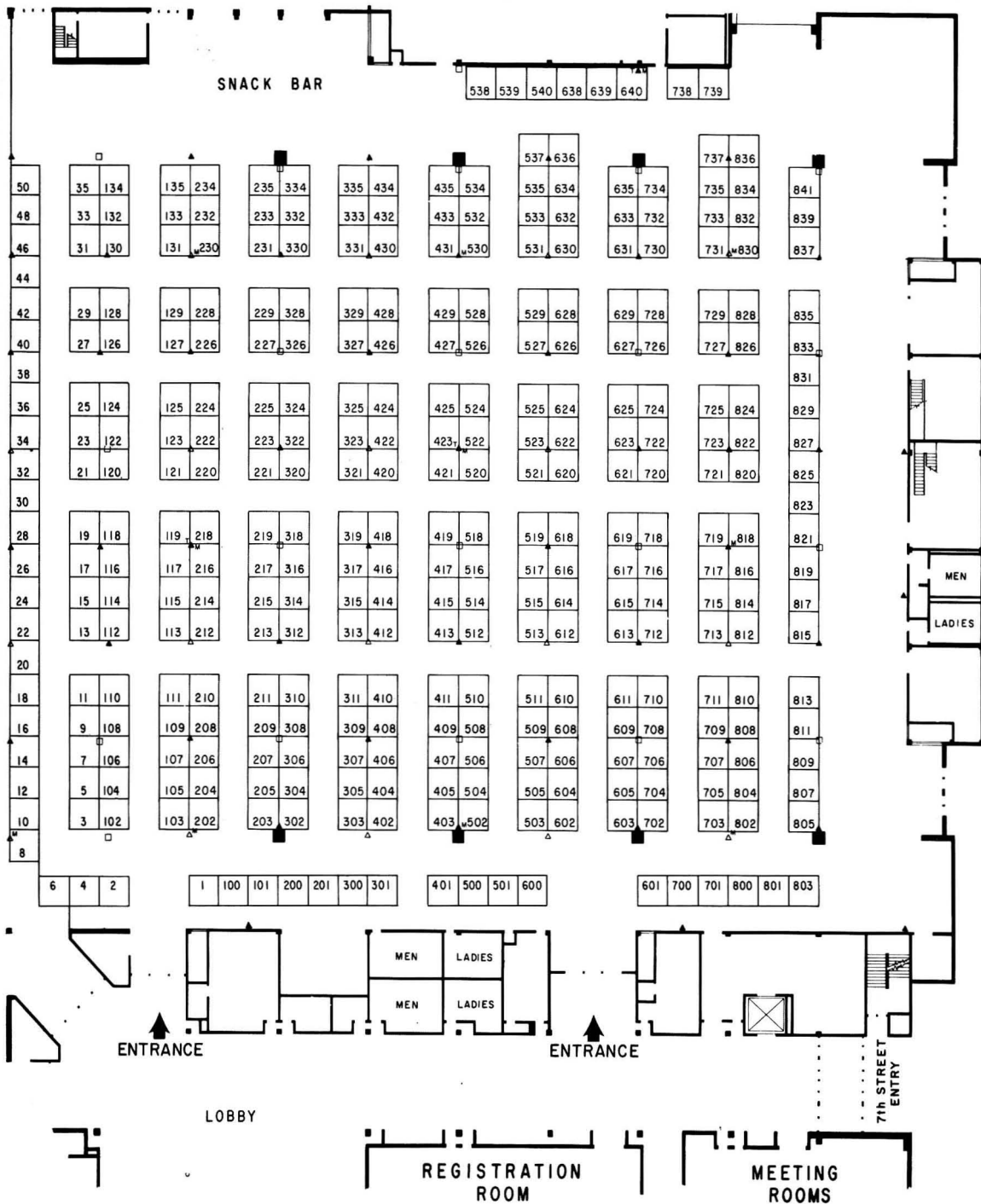
PAINT SHOW HOURS

Wednesday, October 3
Thursday, October 4
Friday, October 5

Noon to 5:30 pm
10:00 am to 5:30 pm
10:00 am to 4:00 pm

Exhibitors	Booth(s)
Aceto Chemical Co., Inc.	335
Air Products and Chemicals, Inc.	413-415-417-419
Alcan Ingot & Powders, Alcan Aluminum Corp.	713-715-812-814
Alpine American Corp.	635
Aluminum Co. of America	17-19
C.M. Ambrose Co.	109-111
American Felt & Filter Co.	8-10-12
American Hoechst Corp.	227-229
American Nepheline Corp.	107
Applied Color Systems, Inc.	816-818
Ashland Chemical Co.	613-615-617-619 712-714-716-718
Atlas Electric Devices Co.	608-610
Automation International Corp.	825
B.A.G. Corp.	835
BASF Wyandotte Corp.	430-432-434
Bennett's Colorant Div.	329
Blackmer Pump Div., Dover Corp.	330
Brookfield Engineering Labs., Inc.	401
Buckman Laboratories, Inc.	408-410
Byk-Mallinckrodt Chem. Prod. GmbH ...	527-529-626-628
Cabot Corp., Cab-O-Sil Div.	9-11
Cargill, Inc.	721-723-725 820-822-824
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Celanese Chemical Co.	113-115-117-119
Celanese Plastics & Specialties Co.	212-214-216-218

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Color Corp. of America	306
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Custom Chemical Co.	309-311
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Daniel Products Co.	303-305
Degussa Corp.	112-114
Diamond Shamrock Corp., Process Chems. Div.	2-4-6
Diano Corp.	127-129-226-228
D/L Laboratories	524
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Dresser Minerals Div., Dresser Ind., Inc.	36
DSET Laboratories, Inc.	32
Eastman Chemical Products, Inc.	621-623-625 720-722-724
Ebonex Corp.	807
Elcometer Instruments Ltd.	735
Encapsulair, Inc.	819-821
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Fed. of Societies for Coatings Technology	535-537
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C. Fricke Co.	327



1979 PAINT INDUSTRIES' SHOW



St. Louis Convention Center St. Louis, Missouri
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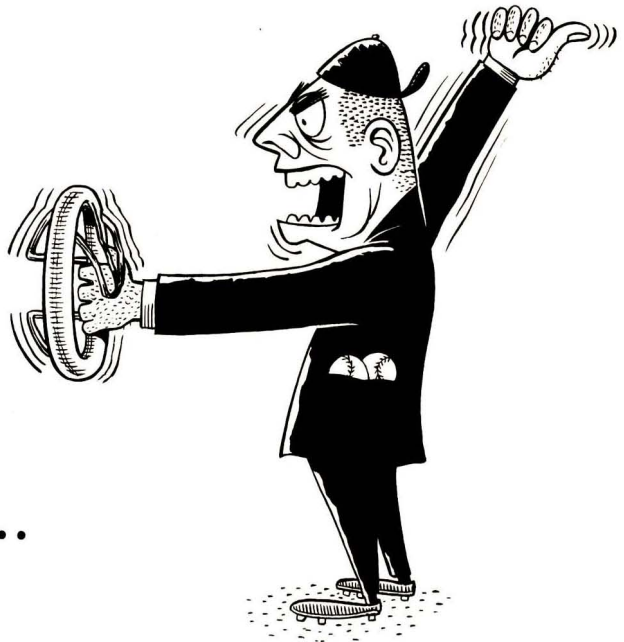
Exhibitors	Booth(s)
GAF Corp.	433
Gail Industries	27-29-126-128
Gardner Laboratory, Inc.	421-423-425
General Electric Co.	22-24
Georgia Kaolin Co.	13-15
W.R. Grace & Co., Davison Chemical Div.	121-123-125
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Halox Pigments Div., Hammond Lead Prods.	1-100
Harmon Colors Corp.	116-118
Harshaw Chemical Co.	321-323-325 420-422-424
Henkel Corp.	102-104
Hercules Incorporated	313-315-317-319 412-414-416-418
Hilton-Davis Chemical Co. Div.	427-429-526-528
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Johns-Manville	402-404
Kelco, Div. Merck and Co., Inc.	731-733
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Miller Paint Equipment, Inc.	133-135
MiniFibers, Inc.	101-200-201
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Morehouse Industries, Inc.	708-710
Mozel Chemical Products Co.	Message Center, Lobby
Myers Engineering, Inc.	630-632
National Assn. of Corrosion Engineers	831-833
Netzsch Brothers, Inc.	21-23
Neville Chemical Co.	512-514
New Way Packaging Machinery, Inc.	601-700
NL Industries, Inc., Ind. Chem. Div.	609-611
Nyco Div., Processed Minerals, Inc.	232-234
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Reichhold Chemicals, Inc.	521-523-525 620-622-624
Rohm and Haas Co.	513-515-517-519 612-614-616-618
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Shamrock Chemicals Corp.	28
Shell Chemical Co.	221-223-225 320-322-324
Silberline Manufacturing Co., Inc.	103-105
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Sun Chemical Corp.	403-405-407-409-411 502-504-506-508-510
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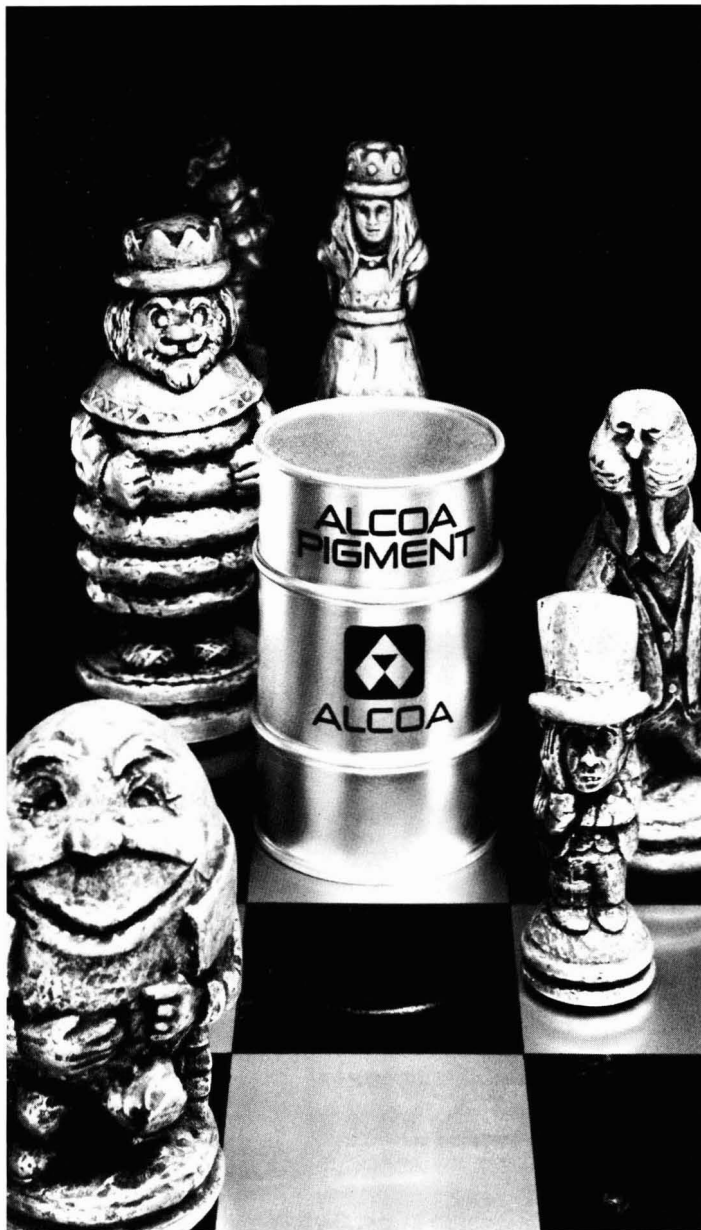
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Government / Industry

Chemical Manufacturers Association Urges New Regs for Old Dumpsites

The Chemical Manufacturers Association (formerly Manufacturing Chemists Association) has called for a new law to deal with old dumpsites, at the same time stressing that the program should focus on "orphan sites" which are dangerous and where no action is being taken to eliminate the danger.

Jackson B. Browning, director of health, safety and environmental affairs for Union Carbide Corp. spoke for his company and the Association before the Subcommittee on Environmental Pollution and Resource Protection of the Senate Committee on Environment and Public Works.

Mr. Browning said that the Carter Administration's "ultrafund" concept of legislation is inappropriate because it combines oil spills, hazardous substances spills and abandoned and inactive hazardous waste disposal sites into one liability and compensation fund. He said the three subjects are "different in nature, are addressed in differing degrees by societal and legal mechanisms already in place and must receive separate and distinct consideration."

Mr. Browning defined an orphan site as one that does not achieve interim Resource Conservation and Recovery Act standards, fails, and (because of that failure) causes health dangers. Also, no action is being taken at such sites because of the inability of the responsible party to pay, inability to locate the liable party or where a legal dispute would lead to a delay. It is essential, he said, that sites which present an imminent hazard be dealt with in a prompt and effective manner, and inaction or needless delay is not acceptable.

Funding for the program would come from regular Federal appropriations, from matching funds contributed by the states and from wrongful dumpers.

In addition to calling for a new law to take care of old dumpsites, Mr. Browning listed six CMA recommendations:

- (1) A state-by-state inventory of disposal sites to identify the location, number and potential dangers;
- (2) "Prioritization" of failing sites in each state with regard to potentials for health dangers;
- (3) A step-up of investigative and enforcement capacities of Federal and state governments on disposal site practices;
- (4) New and higher emphasis on solving the scientific and technological problems arising from neutralizing dangers caused by old dumpsites;
- (5) Prompt addressing of the problems of current and future siting of hazardous waste storage facilities; and
- (6) Full enforcement of disposal laws now and in the future.

Expanding on the last recommendation, Mr. Browning said wrongdoers must be punished and those legally responsible for dumpsite problems must bear the cost of cleanup. "Midnight dumping and the practice of racketeering in illegal waste disposal cannot be tolerated," he stressed.

Mr. Browning explained that enactment of a superfund that covers chemical spills is unnecessary because it "would overlap existing law, arbitrarily supplant existing legal remedies and is based on an acute shortage of reliable data." He added that it is "likely that the data used to justify a superfund for chemical spills are imprecise and exaggerated, both with regard to frequency and potential impact."

Mr. Browning concluded by noting that the Administration bill violated a number of Constitutional principles.

OIL & COLOUR CHEMISTS' ASSOCIATION



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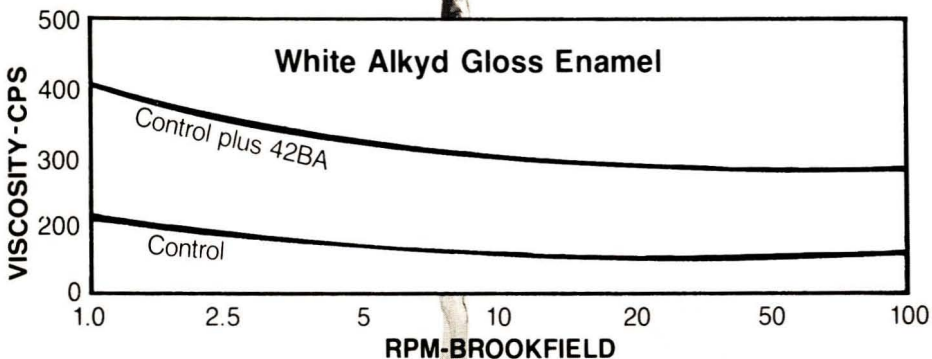
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Factors Influencing Drying Properties Of a Water-Reducible Alkyd

Kansas City Society for Coatings Technology
Technical Committee

The drying properties of water-reducible alkyd paints were compared with those of solvent-based paints made from the same resin. White, black, and clear formulas were used. Cobalt neodecanate was the drier. The resin in the water-reducible formulas hydrolyzed rapidly at elevated storage temperatures, but was quite stable at 24°C. The water-based formulas dried significantly slower than their solvent-based counterparts, summarized by $y=1.5x+1.0$, where y is the dry time in hours of the water-reducible and x is that of the solvent-base. Titanium dioxide had no significant effect on either dry times or hardness, while carbon black increased the drying time. The effect of carbon black was more pronounced in the water-reducible system.

INTRODUCTION

In recent years, industrial paints based on water-reducible alkyd resins have seen widespread use, especially due to increasing restrictions on organic solvent emissions. It has commonly been observed that such paints do not dry as fast as solvent-based paints, and are more subject to loss-of-dry upon storage. The purpose of this investigation was to study the problem of loss-of-dry by comparing water-reducible and solvent-based systems.

The point has been made that water-reducible alkyds do not have true solvent-based counterparts because of resin modifications required for water compatibility.¹ Other investigators² have identified resin type as a controlling factor in drying behavior. One cannot be certain whether the observed differences in drying properties are due more to the marked differences between conventional and water-reducible resins than to the water-based solvent system. The experimental approach, therefore, was to compare water- and solvent-based paints made from the same resin. A typical water-reducible alkyd resin was prepared, then half was dissolved in xylol, the other half in a suitable co-solvent blend. A medium oil length alkyd conforming to TT-R-266 Type III functioned as a control.

Temperature and humidity are known to affect the drying rates of alkyd and oleoresinous paints. It is also well known that such factors as type and amount of pigments, and storage time and temperature affect the drying time.³ Pigments tend to remove drier metals by adsorption, carbon black being one of the worst offenders in this regard. It has also been determined that such factors as solvents, drier metals, drier anions, and pigment type affect the rates at which driers are adsorbed on carbon blacks.⁴

To accommodate these factors, white, black, and clear formulas were prepared. They were aged at various temperatures, and dry time, hardness, pH, and viscosity were measured over a period of time.

RESIN FORMULATION

Initial work was with Amoco Chemical Co. formula WS-549⁵ short oil alkyd. This resin dried very fast (about 15-20 min) in both clear and white formulas. It was felt that the fast dry was determined mainly by rates of solvent evaporation, and that the short oil length would not afford much opportunity to study the effects of drier interaction with the unsaturated fatty acid moieties.

Drying times were measured with Gardner 24 hour circular Dry Time Recorders.⁹ This instrument measures hardness development rather than cure, and cannot distinguish differences in cure rate where much of the apparent cure is due to lacquer-type drying. Therefore, it was decided to continue work with a longer oil resin where significant hardness would develop only with crosslinking.

Amoco resin formulation 408⁶ is a medium oil alkyd with longer drying times than WS-549. It was prepared with Pamolyn[®] 200 linoleic acid substituted for tall oil fatty acids. This formula is listed in *Table 1* and is designated XZ-96. The resin was processed by the fusion method. All ingredients except TMA were charged into the reaction vessel and cooked to a low acid number at 250°C. The temperature was lowered to

¹ Presented by Steven D. Johnson at the 56th Annual Meeting of the Federation of Societies for Coatings Technology in Chicago, Ill., Nov. 2, 1978.

⁹ Pamolyn[®] is a registered trademark of Hercules, Incorporated.

Table 1—XZ-96 Alkyd Resin Formulation^a

Wt %	Material	
42.07	Pamolyn 200 Linoleic acid	
25.55	Trimethylol propane	
24.35	Isophthalic acid	
8.03	Trimelletic anhydride	
ALKYD PROPERTIES		
	XZ-96-A2	XZ-96-M
Acid number	55-65	55-65
Cure, sec at 200°C	42-46	42-46
Solvent	Xylene	50/50 Propylene glycol monomethyl ether/n-butanol
% Nonvolatile	70	70
Color (Gardner)	6	6
Viscosity	Z ₅ -Z ₆	U-W
Wt/Gallon	8.46	8.49

(a) Based on Amoco Resin Formulation 408^e

Table 2—Initial Screening Results of Cobalt Driers In Water-Reducible XZ-96-M Formula^a

Drier Anion	Neodecanate	Napththenate	Octanoate	Tallate
Dry time, hours:				
A. Aged 1 week at 21°C				
Set to touch	1	1	1	1
Tack free	5	4	9	8
Thru	24	24	24	24
B. Aged 1 week at 49°C				
Set to touch	1	1	1	3
Tack free	5	6	9	8
Thru	15.5	16.5	15	15.5

(a) The resin was neutralized with ammonia and reduced to 35% nonvolatile with deionized water. Driers are present at 0.1 cobalt metal on resin solids.

195°C and the TMA added and cooked in the region 190-195°C to an acid number of 55-60. The temperature was then lowered to approximately 60°C, at which it was filtered and split into two portions. Half the batch was reduced in xylol to 70% nonvolatile and designated XZ-96-A2. The other half was reduced to 70% non-

volatile in an equal parts mixture of n-butanol and propylene glycol monomethyl ether.* It was designated XZ-96-M. Viscosity reduction curves of these solutions are shown in Figure 1.

EVALUATION OF DRIER ANIONS

An initial screening series of cobalt driers was prepared with a clear water-based formula using XZ-96-M. This was not an exhaustive test, but was run to determine effectiveness and compatibility. Other investigators^{7,8} had identified the neodecanate as a superior drier in certain formulas, so it was tested. Three other cobalt driers were evaluated: naphthenate, octanoate, and tallate. The drier level was 0.1% cobalt metal on resin solids. Samples were neutralized with ammonia and reduced to 35% solids with deionized water, then aged for one week at 21°C and at 49°C. Drawdowns were made on cold rolled steel panels and dry times measured with Gardner 24 hour circular Dry Time Recorders. These are listed in Table 2. No substantial differences were observed between the driers in this system.

EXPERIMENTAL DESIGN

In order to keep the number of samples from becoming too large, it was decided to use cobalt neodecanate as the sole drier, and at one level, 0.08% metal on resin solids. To test the effects of pigments, both titanium dioxide and carbon black were used. Three storage temperatures were used, 24°C and two commonly tested elevated temperatures, 49°C and 60°C.

The basic experiment, then, was set up to contrast the resin in four areas of possible effect on dry properties: solvent system, pigments (or lack of), storage temperature, and storage time. For purposes of comparison, a parallel series of white, black, and clear formulas based on a medium oil soya-linseed alkyd

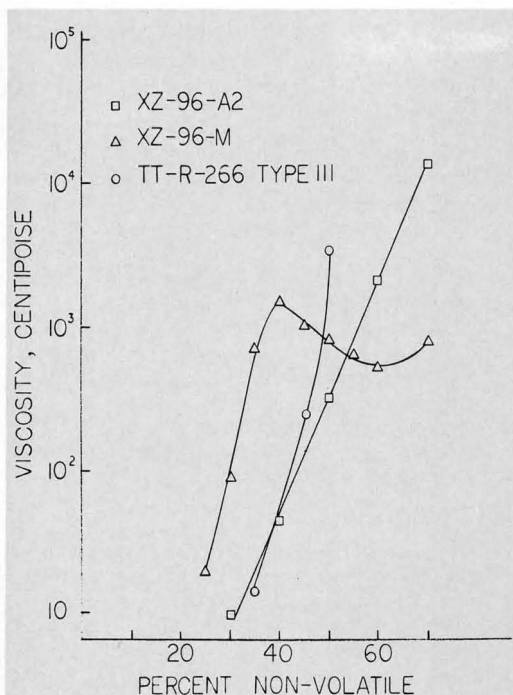


Figure 1—Viscosity reduction curves. XZ-96-A2 reduced with xylene. XZ-96-M neutralized (~80%) to pH 7.4 with ammonia and reduced with deionized water. TT-R-266 Type III at 50% solids in mineral spirits reduced with xylene

*Dowanol® PM, Dow Chemical USA.

Table 3—Paint Formulations^a

Raw Materials	Formula Number								
	1	2	3	4	5	6	7	8	9
XZ-96-A2	414.50	443.90	450.30	—	—	—	—	—	—
XZ-96-M	—	—	—	355.70	377.10	381.70	—	—	—
TT-R-266 Type III	—	—	—	—	—	—	558.30	595.80	603.90
Rutile TiO ₂	255.46	—	—	219.22	—	—	242.47	—	—
Carbon black	—	21.31	—	—	18.10	—	—	20.13	—
12% Cobalt neodecanate	1.94	2.07	2.10	1.66	1.76	1.78	1.86	1.98	2.01
10% NH ₃ (aq)	—	—	—	37.28	39.52	40.00	—	—	—
Xylol	310.88	332.93	337.73	—	—	—	139.58	148.95	150.98
Deionized water	—	—	—	385.12	408.28	413.27	—	—	—
Wt/Gal, lbs	9.83	8.00	7.90	9.99	8.45	8.37	9.42	7.67	7.57
% PVC	20	4	—	20	4	—	20	4	—
% Vehicle solids	40	40	40	32	32	32	40	40	40
% Cobalt on resin solids	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08

(a) Formulas 10 and 11 are the same as 2 and 5, respectively, but with twice the amount of drier, or 0.16% cobalt on resin solids.

conforming to TT-R-266 Type III was also prepared, but it was aged only at 49°C. For reasons described below, two additional black formulas were prepared: one each solvent- and water-based, but with twice the level of drier, i.e., 0.16% cobalt on resin solids.

At periodic intervals during the course of 16 weeks, the following were measured: pH and viscosity, as well as dry time with the Gardner 24 hour circular Dry Time Recorders, and Sward Rocker hardness at 24 hours and one week. Dry time samples were drawdowns of 0.5 mil dry film thickness on cold rolled steel Q-panels[®]. The low film thickness was used because cobalt is a "top drier," and we wished to minimize the effect from insufficient cure in the body of the film. In order to control the effects of temperature and humidity, test samples dried the first 24 hours in a Hotpack Corp. Model 434300 humidity chamber maintained at 27°C and 50% relative humidity. However, because of equipment restrictions, dry times at weeks 2, 10, and 16 were run on lab benches at 24°C with 45% RH at week 2; and 60-70% RH at weeks 10 and 16.

This additional variable evidently caused discrepancies in the drying time (see Figure 5), but its effect was minimized in the statistical calculations by the use of the pairing method (Appendix 2).

The molecular weight distributions of the resins were measured initially by gel permeation chromatography, and again at the end of the test period to note any shifts on storage.

Extracted vehicles were analyzed for remaining drier metal.

PAINT FORMULATION

The paint formulas are listed in Table 3. Those formulas pigmented with titanium dioxide were ground on a porcelain ball mill; the blacks were ground on a steel ball mill. All water-based formulas were ground in the

organic solvent resin solution prior to neutralization and water addition.

The water-based formulas were partially neutralized (about 80%) with aqueous ammonia after grinding, and prior to addition of the drier. The drier was added slowly while stirring and allowed to blend thoroughly before adding the balance of the water. The pH was then adjusted with ammonia to 7.8-8.0. The drier was found to incorporate much more smoothly in the solvent-based formulas if predissolved in a portion of the xylol before addition to the batch. Adding directly to the batch caused the formation of a gel structure (apparently due to the high acid number) that dissipated slowly with stirring.

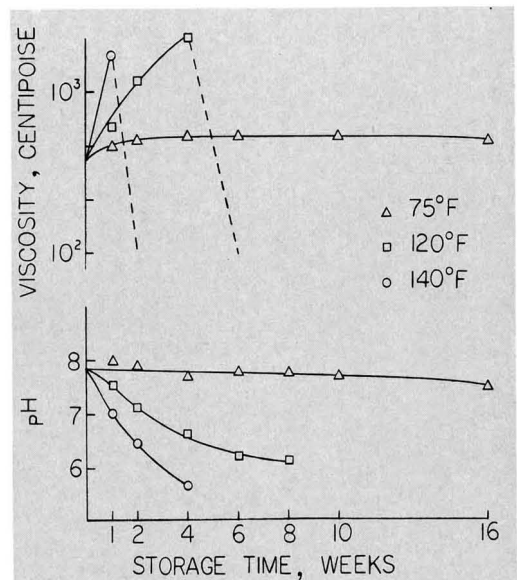
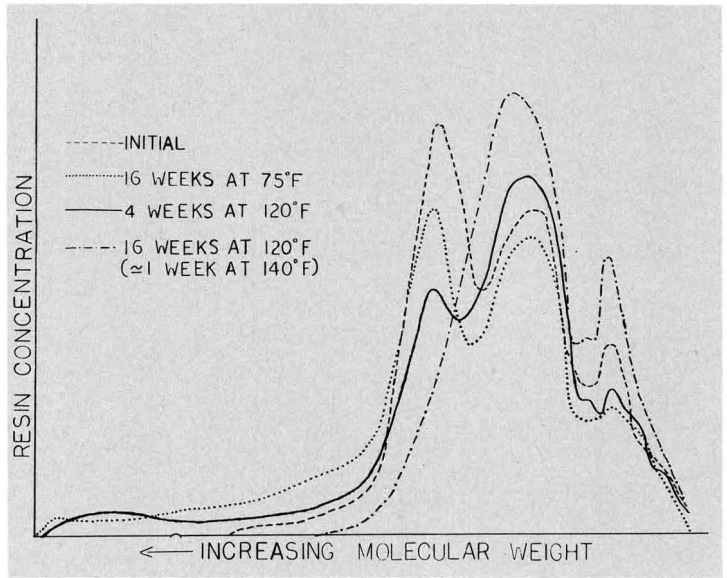


Figure 2—Viscosity and pH of water-reducible systems with storage time at the three storage temperatures

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Figure 3—Gel permeation chromatographs of water-reducible systems (formula 6)



All formulas were allowed to stand 24 hours before measuring the initial dry time. The black formulas numbers 2 and 5, respectively solvent and water, exhibited such slow initial dry times (see Appendix 1 and Figure 5) that it was feared they would disrupt the work schedule. Two further black formulas were prepared, numbered 10 and 11. These were identical to 2 and 5, respectively, except that twice the amount of drier was used, half being added to the grind paste.

Approximate quantities of two gallons per formula were made, then filled off in eight ounce cans, so that an undisturbed sample could be used at each interval.

RESULTS AND ANALYSIS OF DATA

The viscosities, pH, dry times, and hardness were recorded at 24 hours (initial) and at 1, 2, 4, 6, 8, 10, and 16 weeks.

One of the most obvious effects was the rapid drop in pH (Figure 2) and subsequent insolubility that occurred within one week at 60°C and four weeks at 49°C. As the pH dropped, the viscosity increased and then fell precipitously as the resin became insoluble, separating into two phases. This behavior typifies acid-functional alkyds in aqueous alkaline solvents,¹⁰ and is evidence for

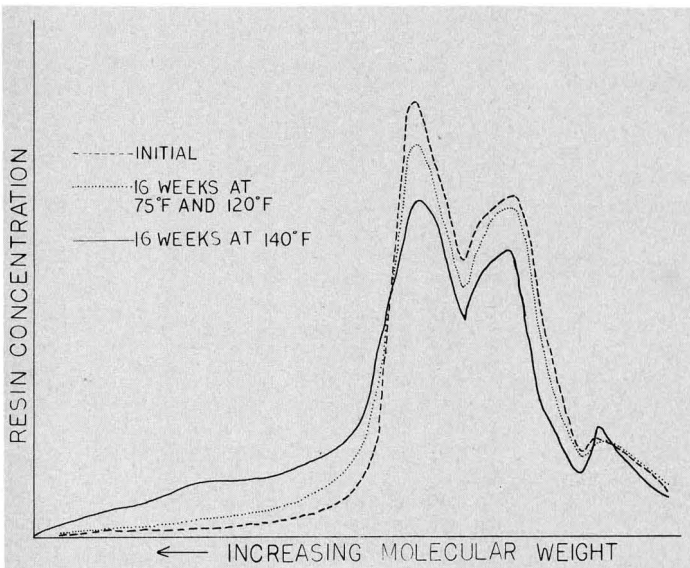


Figure 4—Gel permeation chromatographs of solvent-based systems (formula 3)

hydrolytic cleavage of ester linkages. Addition of ammonia did not redispersed the resin. Comparison of gel permeation chromatographs with those taken initially confirmed an increase in the lower molecular weight portions (Figure 3). As expected, the dry times increased in length and the hardness decreased as the alkyd degraded. No significant difference was noted in the effect at 60°C compared to 49°C; it simply occurred sooner.

In contrast, the pH of the water systems aged at 24°C remained quite stable. The GPC curve, however, does show evidence of a slight increase in the higher molecular weight portion due to bodying, with little evidence for hydrolysis.

In the xylol based formulas, the resin showed only small changes in molecular weight distribution at 24°C and 49°C, but showed evidence for bodying at 60°C (Figure 4). The TT-R-266 Type III alkyd was essentially unchanged at 49°C.

Because the changes in dry times and hardness were obviously affected by the degradation of the resin at the higher storage temperature, only the data acquired from samples aged at 24°C was used for further comparisons.

Figure 5 shows the variation in dry times with respect to storage time. The clear and white formulas show reasonably good stability, the black formulas being most subject to loss of dry. This parallels the performance of the TT-R-266 based formulas aged at 49°C. The "dips" in the curves at 2, 10, and 16 weeks correspond to dry times measured on the lab bench outside the humidity chamber. Although temperature and humidity

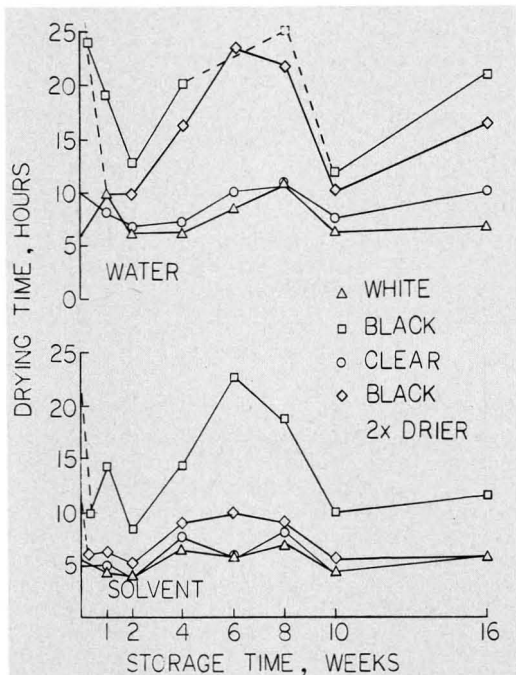


Figure 5—Dry time vs. storage time for systems stored at room temperatures

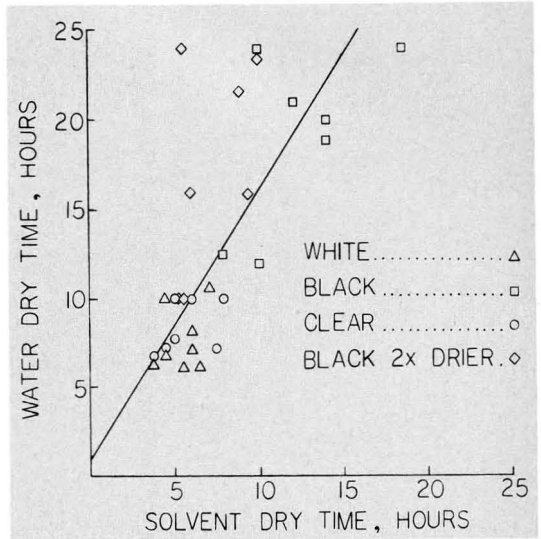


Figure 6—Dry time of water-reducible systems vs. solvent-based systems aged at room temperature. Data paired for formula type and storage time. Straight line $y=1.5x+1.0$ is best fit using least squares

were approximately the same as in the chamber, such factors as light and air movement could have contributed to the observed differences.

One marked difference between the TT-R-266 alkyd and XZ-96 resins was the unusually long time lag between drier addition and first evidence of activity in the XZ-96 blacks, both water- and solvent-based. Neither exhibited appreciable effectiveness until 48 hours after drier addition, and, even then, took much longer to dry than the white or clear formulas. In contrast, the black TT-R-266 alkyd dried as fast as the corresponding clear and white 24 hours after drier addition.

The water-reducible formulas dried significantly slower than their solvent-based counterparts. When the data were paired and fitted to a straight line using the least squares technique (Figure 6) a better fit was obtained if the initial dry times were ignored, with a correlation coefficient of .80 compared to .65 if the initial data was included. The resultant equation is:

$$y = 1.5x + 1.0$$

where x refers to the dry time in hours of the solvent-based formulas and y to those of the water-based. This may be due to a reduction in activity of the cobalt drier from a complex formed between ammonia and cobalt in the water-based formulas. The solvent-based XZ-96 formulas dried substantially slower than the corresponding TT-R-266 formulas. This may be due to both the higher molecular weight of the TT-R-266 and chelating effect from the higher acid number of the XZ-96.

Table 4 lists the results of testing for statistically significant differences due to the presence of pigments. Sets of data from pigmented formulas were compared with those of corresponding clears and were tested for significant difference at the 95% confidence level by

Table 4—Results of Testing for Statistically Significant Differences Due to Pigments, Using the t Test

Systems ^a	D/H ^b	Significant Difference ^c	t Calc.	t ₉₅ (one-side)
White (1) vs. Clear (3) solvent	D	no	-1.323	1.895
	H	no	-0.855	1.895
Black (2) vs. Clear (3) solvent	D	increase	4.497	1.895
	H	decrease	-3.248	1.895
White (4) vs. Clear (6) water	D	no ^d	-1.568	1.895
	H	no ^d	-1.789	1.895
Black (5) vs. Clear (6) water	D	increase	7.579	1.943
	H	decrease	-4.314	1.943
Black (2) solvent vs. Black (5) water, fractional increase over corresponding clear	D	no	-1.18	1.895
Black 2x drier (10) vs. clear (3) solvent	D	increase	2.820	1.895
	H	increase ^e	4.625	1.895
Black 2x drier (10) vs. Black (2) solvent	D	decrease	-4.752	1.895
Black 2x drier (11) vs. Clear (6) water	D	increase	4.265	1.895
	H	no ^e	-1.744	1.895
Black 2x drier (11) water vs. Black 2x drier (10) solvent, fractional increase over corresponding clear	D	increase	3.123	1.895
White (7) vs. Clear (3) TT-R-266 Type III	D	no	1.357	1.895
	H	decrease	-3.230	1.895
Black (8) vs. Clear (9) TT-R-266 Type III	D	increase	3.379	1.895
	H	decrease	-5.487	1.895
Black (2) solvent vs. Black (8) TT-R-266 Type III, fractional increase over corresponding clear (49°C)	D	no	0.623	1.895
	H	no	0.623	1.895

(a) Formula number in parentheses.

(b) D, dry time data; H, 24 hour Sward rocker hardness.

(c) Critical region for significance testing was chosen as 95% confidence level, but most differences shown are also significant at the 99% level.

(d) Significant decrease of both dry time and hardness at 90% level.

(e) Some 24 hour Sward hardness data on formulas 10 and 11 actually taken at 36 hours, so conclusions are suspect.

means of the t test (Appendix 2). Both dry times and 24-hour Sward rocker hardness were used. No significant differences were noted between dry times of the white paints and the corresponding clears, although a significant decrease in Sward hardness at 24 hours occurred in the TT-R-266 systems. (If the confidence level is dropped to 90%, the white water-reducible dry times are significantly lower than the clear.)

As expected, the black formulas dried significantly slower than the clears, even when twice the drier level was used. The 24-hour Sward hardness values showed a corresponding decrease, except with formulas 10 and 11, where some of the determinations were actually

made at 36 hours. The black dry times were arranged to show a fractional increase over those of the corresponding clears, then the solvent- and water-based formulas compared. At the original drier level of 0.08% Co, no significant difference was noted; but at 0.16% Co, the water-reducible formula showed a significantly greater loss-of-dry. Doubling the level of drier caused a significant improvement in dry times of both solvent and water-reducible formulas. No significant difference was noted between the loss-of-dry of the solvent-based XZ-96 and the TT-R-266 black at 49°C.

Other researchers¹¹ investigating the adsorption of cobalt octanoate on carbon black surfaces have found

the rate of adsorption to increase in the presence of polar solvents or moisture. This is consistent with the increased loss-of-dry noted above.

A further difference was noted in the Sward hardness values measured at one week. Those of the water-reducible formulas were significantly lower than those in solvent. The full significance of this point is not known, as the unusually high values are undoubtedly due to the effect of the substrate,¹² and differences in hardness may be due to slight differences in film thickness.

At the completion of the aging period, the vehicles were separated from pigmented formulas by centrifugation, then they and the clear formulas were analyzed for remaining cobalt. A chelometric titration based on the Lucchesi method¹³ was used. The results are shown in Table 5. For the solvent-based formulas, little or no loss of drier is indicated in the clears, or the white at room temperature. The blacks show a decrease in drier at room temperature and both black and white show decreases with increasing storage temperature, apparently due to drier adsorption on pigment surface. The results for the water-reducibles show unexpectedly high values. The value for the white TT-R-266 formula is usually low.

The titration yields values for total metal concentration. Since the samples were stored in unlined containers, there is a possibility that some tin or iron was extracted from the cans. To test this possibility, all samples were submitted for cobalt analysis by atomic absorption spectroscopy, but, as of this writing, results are not yet available.

Two further points were noted, but not investigated further. The water-reducible paints were found to dry much slower over iron phosphate treated panels than over untreated cold rolled steel. The clear formula (number 6) dried in nine hours on CRS, but required 18 hours over iron phosphate, at the same time and under the same conditions. No similar effect was found with the solvent-based formulas.

Dried films of water-reducible whites were also found to yellow noticeably after sitting several weeks on laboratory shelves. This was also apparent with the clear films. It was not evident as the coating dried, but only after several weeks' aging. The solvent-based counterpart showed no such tendency. This phenomenon may be due to ammonia residues in the film.

Table 5—Results of Cobalt Analysis by Chelometric Titration

Formula	% Co Initial	Storage Temp. °C	% Co remaining after (weeks)		
			1	4	16
White (1) Solvent	0.08	24	—	—	0.080
		49	—	—	0.066
		60	—	—	0.056
Black (2) Solvent	0.08	24	—	—	0.050
		49	—	—	0.047
		60	—	—	0.043
Clear (3) Solvent	0.08	24	—	—	0.084
		49	—	—	0.086
		60	—	—	0.084
White (4) Water	0.08	24	—	—	0.062
		49	—	0.084	—
		60	0.079	—	—
Black (5) Water	0.08	24	—	—	0.085
		49	—	nr	—
		60	nr	—	—
Clear (6) Water	0.08	24	—	—	0.090
		49	—	0.088	—
		60	0.089	—	—
White (7) TT-R-266	0.08	49	—	—	0.027
Black (8) TT-R-266	0.08	49	—	—	0.068
Clear (9) TT-R-266	0.08	49	—	—	0.084
Black 2x Drier (10) Solvent	0.16	24	—	—	0.140
		49	—	—	0.127
		60	—	—	0.081
Black 2x Drier (11) Water	0.16	24	—	—	0.167
		49	—	nr	—
		60	nr	—	—

SUMMARY

The drying properties of water-reducible paints were investigated in comparison with solvent-based paints made from the same resin. White, black, and clear formulas were used. Cobalt neodecanate was the sole drier at 0.08% metal on resin solids. Samples were aged at 24°C, 49°C, and 60°C. Similar formulas based on a solvent-reducible alkyd meeting TT-R-266 Type 3 were prepared and aged at 49°C. Drying times and hardness were measured at regular intervals during a 16 week period. Viscosity and pH were also measured.

Lack of hydrolytic stability was found to be a controlling factor at elevated temperatures; the water-reducibles degraded rapidly with a corresponding loss-of-dry and eventual insolubility. At the lower storage temperature where hydrolysis occurred to a very limited extent, drying times were found to be reasonably stable. No evidence was found for incompatibility of the drier with the resin although both water-reducible

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and solvent-based blacks required unusually long induction (storage) periods for drier effectiveness.

The water reducible formulas, in general, dried significantly slower than their solvent-based counterparts. This relation is summarized by the equation: $y = 1.5x + 1.0$, where the independent variable refers to the dry time in hours of the solvent-based paint, and the dependent variable to the corresponding water reducible.

Titanium dioxide was found to have no significant effect on the drying properties of either solvent or water-reducible formulas, while carbon black definitely retarded the drying speed. There is evidence that drier adsorption on carbon black occurs to a greater extent in the water-reducible formulas.

Iron phosphate treated metal panels were found to retard the drying rate of the water-reducible clear. This factor was possibly accentuated by the low film thicknesses used in the test. It is not known if this effect was due to the individual lot of panels or to the nature of the pretreatment itself. It was not investigated further.

Finally, dried white and clear water-reducible films were found to yellow noticeably several weeks after drying. This phenomenon also was not investigated further.

In view of these findings, it seems that much information could be obtained at mildly elevated storage temperatures of 32-38°C, where hydrolysis would occur more slowly and probably not cause such rapid degradations as occurred at the higher temperatures. It would also seem fruitful to investigate the effect of drier interactions at the vehicle/pigment interface, with other types of pigments, and with surface modifying agents such as certain titanate esters, or with metal chelating agents. It is not known whether the behavior of other

commonly used drier metals would parallel that of cobalt.

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APPENDIX I DRY TIME, hours

Formula	Temp. °C	Time (weeks)								
		Initial	1	2	4	6	8	10	16	
1. White Solvent-base	24	5.5	4.5	4	6.5	6	7	4.5	6	
	49	—	8	6	11	13	15	8.5	—	
	60	—	7	7.5	15	20	19	10	—	
2. Black Solvent-base	24	10 ^a	14	8	14	22.5	18.5	10	12	
	49	—	15.5	11	17.5	>24	>24	11	—	
	60	—	16.5	11.5	20	>24	>24	13	—	
3. Clear Solvent-base	24	5	5	4	7.5	6	8	4.5	6	
	49	—	8.5	4.5	11	12	12.5	5.5	—	
	60	—	10	5.5	14.5	16	15	6.5	—	
4. White Water-reducible	24	6	10	6	6	8	10.5	6.5	7	
	49	—	16	6.5	11	11	—	—	—	
	60	—	21	10.5	—	—	—	—	—	
5. Black Water-reducible	24	24 ^a	19	12.5	20	10	>24	12	21	
	49	—	18	15	>24	—	—	—	—	
	60	—	>24	18	—	—	—	—	—	

6. Clear Water-reducible	24	10	8	6.5	7	10	10	7	10
	49	—	8	8.5	15	—	—	—	—
	60	—	9.5	11	—	—	—	—	—
7. White TT-R-266	49	1	1	1.5	4	7.5	6	2.5	2
8. Black TT-R-266	49	1	2	2.5	7.5	13.5	10.5	5	6
9. Clear TT-R-266	49	1	1	2	3.5	6	5	2.5	2
10. Black 2x drier, Solvent	24	5.5 ^a	5.5	5	9.5	10	9	5.5	6
	49	—	6.5	6.5	11.5	16	16.5	—	—
	60	—	12	13	>24	—	—	—	—
11. Black 2x drier, Water	24	28 ^a	10	10	16	23.5	21.5	10	16
	49	—	12	13	>24	—	—	—	—
	60	—	17	12	—	—	—	—	—

(a) 48 hour dry times. At 24 hours, the following dry times were recorded: formula 2: 21 hr, formula 5: >48 hrs, formula 10: 8 hr, formula 11: 45 hr.

24 HOUR SWARD ROCKER HARDNESS

Formula	Temp. °C	Time (weeks)							
		Initial	1	2	4	6	8	10	16
1. White Solvent-base	24	40	48	53	47	47	50	37	45
	49	—	47	45	46	40	42	47	—
	60	—	39	38	38	34	21	43	—
2. Black Solvent-base	24	28	46	52	43	18	38	38	41
	49	—	38	47	43	0	0	43	—
	60	—	44	47	36	0	0	32	—
3. Clear Solvent-base	24	46	49	49	52	50	44	46	51
	49	—	59	50	42	46	40	46	—
	60	—	48	45	38	42	40	44	—
4. White Water-reducible	24	30	33	34	36	40	31	28	28
	49	—	24	24	20	22	—	—	—
	60	—	14	14	—	—	—	—	—
5. Black Water-reducible	24	12 ^a	11	30	24	41	0	24	20
	49	—	30	27	0	—	—	—	—
	60	—	6	14	—	—	—	—	—
6. Clear Water-reducible	24	40	40	31	40	37	30	35	32
	49	—	39	29	21	23	—	—	—
	60	—	30	16	—	—	—	—	—
7. White TT-R-266	49	34	40	53	41	42	43	26	32
8. Black TT-R-266	49	32	42	47	37	38	39	31	35
9. Clear TT-R-266	49	36	46	51	47	43	46	33	32
10. Black 2x drier, Solvent	24	48	45	63 ^b	62	53 ^b	50	54	49
	49	—	43	55 ^b	56	53 ^b	43	—	—
	60	—	42	56 ^b	35	47 ^b	16	—	—
11. Black 2x drier, Solvent	24	26 ^a	32	42 ^b	36	32 ^b	12	34	28
	49	—	28	36 ^b	0	—	—	—	—
	60	—	19	19 ^b	—	—	—	—	—

(a) 48 hour data

(b) 36 hour data

SWARD ROCKER HARDNESS, 1 WEEK

Formula	Temp. °C	Time (weeks)							
		Initial	1	2	4	6	8	10*	16
1. White Solvent-base	24	52	54	58	52	50	56	—	56
	49	—	55	58	56	54	54	—	—
	60	—	55	40	50	53	48	—	—
2. Black Solvent-base	24	50	57	61	61	57	48	—	52
	49	—	57	56	55	62	44	—	—
	60	—	57	48	49	54	42	—	—
3. Clear Solvent-base	24	62	56	59	58	59	58	—	61
	49	—	60	57	52	57	61	—	—
	60	—	59	59	53	57	55	—	—
4. White Water-reducible	24	44	42	54	44	43	48	—	39
	49	—	42	42	58	34	—	—	—
	60	—	31	24	—	—	—	—	—
5. Black Water-reducible	24	36	44	47	45	55	37	—	41
	49	—	49	43	30	—	—	—	—
	60	—	44	27	—	—	—	—	—
6. Clear Water-reducible	24	44	49	48	54	46	45	—	50
	49	—	48	47	32	34	—	—	—
	60	—	43	23	—	—	—	—	—
7. White TT-R-266	49	46	43	57	54	42	51	—	44
8. Black TT-R-266	49	44	42	51	50	44	52	—	44
9. Clear TT-R-266	49	57	44	52	48	46	50	—	48
10. Black 2x drier, Solvent	24	54	70	69	58	52	52	—	48
	49	—	64	71	54	55	61	—	—
	60	—	66	63	50	49	50	—	—
11. Black 2x drier, Water	24	45	55	54	48	47	45	—	50
	49	—	57	48	30	—	—	—	—
	60	—	47	23	—	—	—	—	—

(a) not run

APPENDIX 2

THE t TEST

Where sufficient random samples of size n are taken from a population of normal distribution with mean μ_0 and standard deviation σ , the mean of the set of sample means will be the population mean μ_0 . If μ_0 is the expected value of the mean x of a sample of size n, then the sample can be tested for significant difference from the population by comparing x with μ_0 .

Where the sample size n is relatively small, about 30 or less, and the population standard deviation σ is not known, the t distribution is used. The t value is given by

$$t = \frac{\bar{x} - \mu_0}{s_x / \sqrt{n}}$$

where \bar{x} is the sample mean, n is the number of items in the sample, and s_x is the sample standard deviation, given by

$$s_x = \sqrt{\frac{\sum(x-\bar{x})^2}{n-1}}$$

When comparing the means \bar{x} and \bar{y} of two samples the equation takes the form

$$t = \frac{(\bar{x}-\bar{y}) - \delta}{s} \bigg/ \left(\frac{1}{n_x} + \frac{1}{n_y} \right)^{1/2}$$

Where δ is the expected difference between the means \bar{x} and \bar{y} (zero where \bar{x} is expected to equal \bar{y}), n_x and n_y are the number of item in the samples, and s is a "pooled estimate" of the sample standard deviation where the variance s^2 is given by

$$s^2 = \left[\frac{(n_x-1)s_x^2 + (n_y-1)s_y^2}{(n_x+n_y-2)} \right]$$

where s_x and s_y are the standard deviations of the x and y samples.

Comparing the calculated t values with values published in statistical tables provides a quantitative basis for choosing between two hypotheses concerning the means of two sets of data. The null hypothesis $H_0: \bar{x} - \bar{y} = \delta$, where $\delta = 0$ is that both sets of data come from the same population, i.e. no statistically significant difference exists between the means. Three alternate hypotheses are possible: $\bar{x} - \bar{y} > \delta$, $\bar{x} - \bar{y} < \delta$, or $\bar{x} - \bar{y} \neq \delta$, which simultaneously tests the first two. The conditions for accepting or rejecting H_0 are given by

ALTERNATE HYPOTHESIS	REJECT H_0 IF
$\bar{x} - \bar{y} < \delta$	$t < -t_\alpha$
$\bar{x} - \bar{y} > \delta$	$t > t_\alpha$
$\bar{x} - \bar{y} \neq \delta$	$ t > t_{\alpha/2}$

Where t_α is obtained from published tables for level of certainty α and $(n-1)$ degrees of freedom.

A complete discussion of the above can be found in a good statistics text such as Miller and Freund.¹⁴

Macdonald¹⁵ describes a method for use of the t test where the items in two samples can be arranged in pairs. This method has the advantage of increasing the sensitivity of the test by reducing the magnitude of the standard deviation. It is applicable only if there is a physical basis for pairing, and cannot be used by pairing at random. In the present case, data from corresponding sets can be paired on the basis of storage time.

To illustrate, consider the dry times of the XZ-96 solvent-based white and the corresponding clear. We wish to test

whether or not the presence of titanium dioxide affects the dry time to a significant extent. The difference between each set of paints is taken, forming a third set of data:

Number of weeks:	initial	1	2	4	6	8	10	16
White (formula 1)	5.5	4.5	4	6.5	6	7	4.5	6
Clear (formula 2)	5	5	4	7.5	6	8	4.5	6
Difference	0.5	-0.5	0	-1.0	0	-1.0	0	0

If the titanium dioxide has no effect, the expected difference is zero, $H_0: \bar{x} = 0$ and $\mu_0 = 0$. The t value is given by

$$t = \frac{\bar{x}}{s_x/\sqrt{n}}$$

For the data listed, $\bar{x} = -0.25$, $s_x = .5345$, $n=8$ and $t = -1.323$. The published t value for $(n-1)$ or 7 degrees of freedom and 95 percent level of certainty for alternate hypothesis $\bar{x} \neq 0$ is 2.365. The absolute value of the calculated t is less than the published value.

$$|-1.323| < 2.365$$

So H_0 cannot be rejected. There is no significant difference in the dry times under the conditions tested.

Pairing the data minimizes the spread of the standard deviation due to drift of sample values with time, or where data was obtained under alternate conditions, such as at weeks 2, 10, and 16. Presumably, these factors would affect each of the points in much the same manner, and would be at least partially canceled by taking the difference.



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Reaction Mechanism Of Melamine Resins

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The structure of melamine resins has a significant effect on the mechanism of catalysis. The analysis of the volatile reaction products of amino resins was used as a method to determine the reactions occurring during the crosslinking process. With fully alkylated melamine formaldehyde resins, specific acid catalysis was found to be the dominant crosslinking mechanism. With partially alkylated resins, demethylation and subsequent catalysis by a general acid catalysis mechanism was found to be the crosslinking mechanism. In each case methoxy methyl groups and not methylol groups were found to be the reactive site.

INTRODUCTION

Commercially available alkylated amino-formaldehyde resins, such as used in coatings, are complex mixtures of monomers, dimers, and higher oligomers. Besides being different in molecular weight distribution, these products also vary in molar amounts of formaldehyde and alcohol combined. All these characteristics are interrelated when an amino resin is prepared by a commercial process.

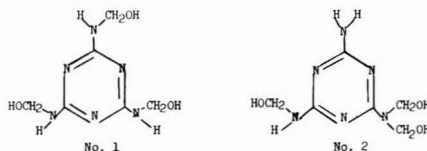
The performance of these amino resins in actual application is, therefore, dependent on a rather complex set of structural characteristics which, in the past, were not accessible to analytical probing. Furthermore, a clear understanding of the crosslinking mechanism was made difficult by the presence of catalyst from the manufacture of these resins. The assumption that partially alkylated amino-formaldehyde resins are faster reacting than fully alkylated resins, because of the higher reactivity of methylol groups compared to the alkoxyethyl groups, has been found erroneous. Also, the general belief that strong "acid" conditions are required for good cure has been found wrong.

Some of the conclusions of this work have been expressed in previous work,¹ although in this author's

opinion, no comprehensive understanding of the reactions involved emerged from that study.

COMPOSITION OF MELAMINE-FORMALDEHYDE RESINS

Instead of doing this work with model compounds which are extremely difficult to synthesize, it was decided to use commercially available resins whose structure was well defined by analysis. In reacting melamine with formaldehyde, random substitution of the $-NH_2$ groups is observed. Analysis of trimethylolmelamine by NMR technique² indicates that mainly the symmetric compound (No. 1) is formed and only small amounts of an asymmetric compound (No. 2):



We, therefore, can conclude that, in a highly monomeric melamine-formaldehyde resin with a melamine-formaldehyde ratio of > 3 , most of the unreacted hydrogen on the nitrogen are randomly distributed and either next to a methylol or alkoxy methyl group. Therefore, such a resin will have either $-N\begin{smallmatrix} \text{CH}_2\text{OR} \\ \text{H} \end{smallmatrix}$ or $-N\begin{smallmatrix} \text{CH}_2\text{OH} \\ \text{H} \end{smallmatrix}$ functional sites. The amount of these reactive sites should be equivalent to the $-NH$ content.

There is one exception: If the $-NH$ group is located next to a bridge in a polymeric unit of the melamine-formaldehyde resin ($-N\begin{smallmatrix} \text{CH}_2 \\ \text{H} \end{smallmatrix} - N-$).

Theoretically at least, the bridge unit could also be a methylene ether group. No analytical tools are available at the present time to prove or disprove the presence of such a unit, but other considerations to be

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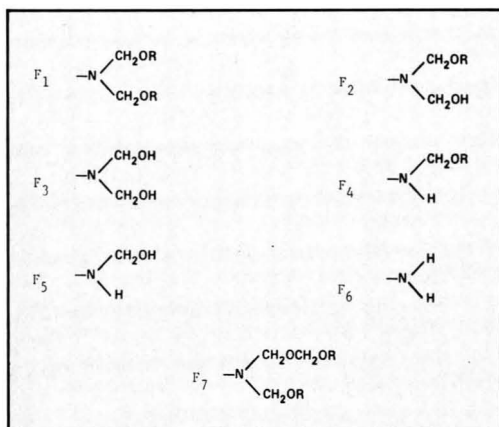


Figure 1—Functional groups in melamine resin

discussed later virtually exclude methylene ether and -HN groups in the same resin. The variety of different possible functional sites on a melamine resin is shown in Figure 1.

Commercial melamine-formaldehyde resins do not contain only one type of functional group, but a combination. The performance characteristics are, therefore, a function of the ratio of these functional groups in the amino resin. In a given amino resin, this probable distribution of functional groups is influenced by the process used to make the resin.

Technically, it is reasonably easy to prepare a monomeric hexakis(alkoxymethyl)melamine with only alkoxymethyl functional sites F_1 ; but it is not technically possible to prepare products with only F_2 , F_3 , F_4 , or F_5 (Figure 1) functionality. Products made by commercial processes contain a combination of these functional sites. Certain combinations are more frequent in an amino resin; others are, for practical purposes, non-existent. For example, a partially alkylated amino resin could contain F_1 , F_2 , and F_4 functionality, but no F_3 , F_5 , F_6 , or F_7 . In a highly alkylated commercial resin, F_1 , F_7 , and small levels of F_2 are normally present.

All commercial melamine-formaldehyde resins do contain variable levels of higher polymeric materials. All possible combinations of bridging units are shown in Figure 2. Again, as in the case of functional groups, the probability for the existence of a certain bridging unit will vary in different resin types. Methylene bridges (B_1 - B_6) are, for all practical purposes, non-reactive. Methylene ether groups (B_7 - B_{12}) should undergo the normal reaction of an ether group. Present analytical techniques cannot distinguish between bridging ether groups. (B_7 - B_{12}) and polyether side chains (Figure 1, F_7). Only circumstantial evidence can indicate the presence of either bridge ether or side chain polyether groups. From an application standpoint, it is not very important to be able to distinguish between either group. Only the presence or amount thereof is of significance.

For this study, three different types of methylated melamine-formaldehyde resins and one butylated resin were selected. The structure of the methylated resins was clearly established by analysis. More questions remain to be resolved in the case of the butylated resin. Table 1 shows the analysis of these amino resins. The analysis was performed by conventional wet techniques. The alkoxy content was determined by NMR.

Amino resin 1 is a commercial version of a highly methylated melamine-formaldehyde resin. The combined molar ratio of melamine-formaldehyde methanol ($MF_{6.1}Me_{5.2}$) is shown in the first line of the table. The combined amount of 6.1 moles of formaldehyde per melamine is not an error in the analysis, but an indication that polyether side chains and methylene ether bridging units are present in the resin. Methanol is combined in form of methoxymethyl groups ($>N-CH_2OCH_3$).

The subsequent number indicates the molar amount of methylol groups per triazine (0.3). The expression of $HCHO_F$ measures the amount of free-formaldehyde in moles of $HCHO$ /triazine (0.075). The amount of $-CH_2OCH_2-$ bridging units and polyether side chains per triazine is shown in the next line (0.5). This number is an approximation not obtained by analysis, but by calculation⁸ from molar ratios and degree of polymerization (\overline{DP}) information. All analytical errors can be magnified in this calculated "polyether" number; but as long as no reliable analytical tools are available to determine this amount of polyether groups, the calculated number offers important clues about the structure

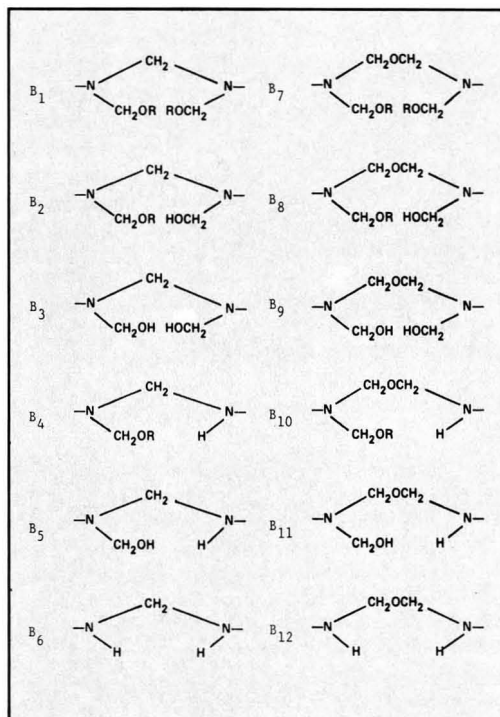


Figure 2—Functional groups in melamine resin bridges

of the resin. The degree of polymerization \overline{DP} was calculated from gel phase analysis of the resin. Gel phase gives a good separation of monomer, dimer, and trimer, but fails to give a good separation of higher oligomers. The distribution of higher oligomers can, therefore, be calculated from the ratio of monomer, dimer, and trimer, assuming a natural distribution of the oligomers. This procedure, although subject to some error, is sufficiently accurate for products high in monomer content, but is not acceptable for highly polymeric products.

Amino resin 2 was a typical commercial partially methylated melamine-formaldehyde resin.

Amino resin 3 was a higher methylated resin with high NH content. The -NH content was determined by NMR.

NH content < 1 per triazine can also be determined by potentiometric titration. Amino resin 1, as determined by titration, has virtually no NH groups and amino resin 2 has a small amount. Amino resin 1 is sold at 99+% solids. Amino resins 2 and 3 are sold as 80% solids solution in n-butanol.

Amino resin 4 was a conventional butylated melamine-formaldehyde resin, and it was used for comparative purposes. The analysis methods used for the methylated resins were not sufficiently reliable for this resin, therefore, only molar ratio ($MF_5Bu_{2.8}$) and GPC analysis are reported.

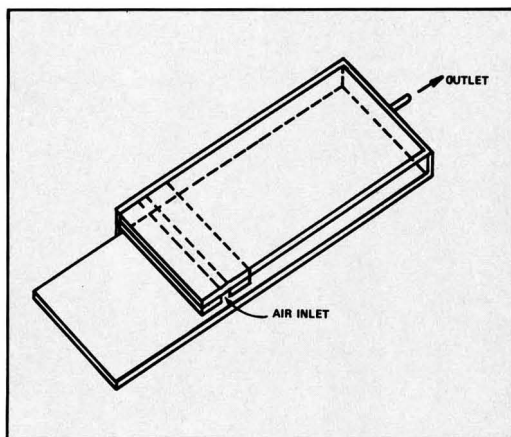


Figure 3—Flow cell design

As a backbone polymer for this study, a 100% solids polyether polyol was selected. This resin, a reaction product of bisphenol A with 6.5 moles of ethylene oxide, has only primary hydroxyl groups and a very narrow molecular weight distribution. Therefore, it was very well suited as a model compound.

CHARACTERISTICS OF DIOL

Molecular Weight506
Viscosity, Brookfield, 25°C, pascals	2.85
MEQ (OH/g)	3.95

This polymer has no residual acidity, therefore, an acid catalyst had to be used with all coatings formulations. The acid catalyst used in all experiments, if not otherwise stated, was dodecylbenzene sulfonic acid.

Table 1—Composition

AMINO RESIN 1	$MF_{6.1}Me_{5.2}$	
	-NCH ₂ OH	0.3
	HCHO _F	0.075
	-CH ₂ OCH ₂ -	0.5
	\overline{DP}	1.68
AMINO RESIN 2	$MF_{5.8}Me_{3.8}$	
	-NCH ₂ OH	1.0
	HCHO _F	0.19
	-CH ₂ OCH ₂ -	0.4
	-CH ₂ -	0.2
\overline{DP}	2.5	
AMINO RESIN 3	$MF_{4.0}Me_{3.4}$	
	-NCH ₂ OH	0.57
	HCHO _F	0.07
	-CH ₂ -	0.52
	-NH	1.48
\overline{DP}	2.1	
AMINO RESIN 4	$MF_5Bu_{2.8}$	
	GPC, MONOMER	16.5
	DIMER	11.9
	POLYMER	71.6

TEST CONDITIONS

Because of the difficulties in analyzing the composition of crosslinked films, it was decided to collect the reaction volatiles. From these volatiles, one can conclude possible reactions and mechanisms of reaction. For this purpose, coated panels were cured in a flow cell and all reaction volatiles were collected and absorbed in dry n-propanol and analyzed by gas chromatography.

The flow cell (Figure 3) was constructed of 5 mm window glass and cemented with a silicone rubber adhesive. The dimensions of the curing chamber are 105 × 100 × 5 mm.

Preheated air enters the mixing chamber at a flow rate of 300 ml/min. The air is withdrawn from the main chamber at a lower rate of 100 ml/min. A coated and predried panel is inserted into the flow cell. The chamber does not have to be closed air tight. Reaction volatiles are drawn from this chamber into the absorption liquid. The panels were either inserted into the flow cell at the baking temperature or at a lower temperature, and the temperature was raised to the baking temperature within 5-7 min. The heat-up rate of a panel in the flow cell and a panel in a forced air oven was about the same and required about 5 min to be within 5°C of the baking temperature.

Table 2—Reaction Volatiles

EQ 1	$>NCH_2OH \rightleftharpoons NH + HCHO$
EQ 2	$>NCH_2OH + >NH \longrightarrow >NCH_2N< + H_2O$
EQ 3	$>NCH_2OH + ROH \rightleftharpoons NCH_2OR + H_2O$
EQ 4	$>NCH_2OCH_3 + ROH \rightleftharpoons NCH_2OR + CH_3OH$
EQ 5	$2>NCH_2OCH_3 + H_2O \longrightarrow >NCH_2N< + HCHO + 2 CH_3OH$
EQ 6	$>NCH_2OCH_3 + >NH \longrightarrow >NCH_2N< + CH_3OH$
EQ 7	$2>NCH_2OCH_3 \rightleftharpoons >NCH_2N< + CH_3OCH_2OCH_3$
EQ 8	$2>NCH_2OH \rightleftharpoons >NCH_2OCH_2N< + H_2O$
EQ 9	$>NCH_2OCH_3 + >NCH_2OH \rightleftharpoons >NCH_2OCH_2N< + CH_3OH$
EQ 10	$>NCH_2OCH_3 + ROH \rightleftharpoons >NCH_2OR + >NCH_2OH$
EQ 11	$>NCH_2OCH_3 + HOH \rightleftharpoons >NCH_2OH + CH_3OH$

For these experiments, either molecular sieve dried (dry) air or air saturated at room temperature with moisture was used (wet).

The test panels used were 100 × 300 mm aluminum panels. These panels were dried and stored in a desiccator before use. Drawdowns were prepared in the open air, weighed immediately, and stored in the desiccator before use. When a solvent containing formulation was tested, the panels were dried at 45°C for 45 min in a forced air oven after drawdown. They were then weighed and stored in the desiccator. Even with these precautions, it was not possible to completely exclude moisture from the reaction chamber. Even the very short handling in the air was sufficient for small amounts of water to be absorbed in the coating. Therefore, analysis for water as a reaction product was abandoned. Accurate measurements of the amount of water

Table 3—Volatiles, Amino Resin 1

AMINO RESIN 1			
Composition:	$MF_{6.1} Me_{5.2} : NCH_2OH_{0.3} \cdot HCHO_{0.075} \cdot -CH_2OCH_2-_{0.51}$		
DP	1.68		
Baking Conditions:	20 Minutes @ 150°C. (302°F.)		
Film Thickness, μ m:	25		
Catalyst, %:	0.84		
POLYOL/AMINO RESIN 1	75/25	62.5/37.5	50/50
OH/TRIAZINE	4.48/1	2.49/1	1.49/1
MOL. VOLATILES/TRIAZINE:			
HCHO	0.2	0.52	0.33
MeOH	5.0	3.07	2.4
METHYLAL	--	--	0.09
H ₂ O	1.02	0.72	0.5

Table 4—Volatiles, Dry-Wet—Amino Resin 1

Composition:	$MF_{6.1} Me_{5.2} : NCH_2OH_{0.3} \cdot HCHO_{0.075} \cdot -CH_2OCH_2-_{0.51}$	
DP	1.68	
Baking Conditions:	20 min. 100 \longrightarrow 150°C (212 \longrightarrow 302°F)	
Film Thickness, μ m:	25	
Catalyst, %:	0.84	
Polyol/Amino Resin 1:	62.5/37.5	
OH/Triazine:	2.49/1	
AIR FLOW	DRY	WET
MOL. VOLATILES/TRIAZINE		
HCHO	0.35	0.83
MeOH	2.64	3.5

volatilized would have been of interest, but the required effort was not justified by the results expected from this work. Therefore, the efforts were mainly aimed at analyzing for formaldehyde, alcohol, and other volatile reaction products.

REACTION VOLATILES

Wohnsiedler⁴ has shown in his work on an alkyl melamine resin system that a variety of reactions can take place. Possible reaction schemes are shown in Table 2. He did not mention the influence of water on the self-condensation reaction (EQ₅) and he did not consider demethylation (EQ₁) as major reaction steps required for crosslinking of melamine resins.

In Table 3, the reaction volatiles of amino resin 1 with the polyol were studied at different ratios of polyol to amino resin, 75/25, 62.5/37.5, and 50/50 at a baking schedule of 20 min at 150°C. The reaction was catalyzed with 0.84% of dodecylbenzene sulfonic acid based on the total binder. The 100% solids resin blend was low enough in viscosity to be applied by drawdown without any dilution. Immediately after drawdown, the coating weight was determined. The dry film thickness of the coating was 25 μ m.

Assuming only reaction between the methoxy and the hydroxyl groups of the polymer (Table 2, EQ₄), the molar amount of methanol should be equivalent to the molar amount of hydroxyl groups in the polymer. Any excess of methanol has to come from self-condensation or hydrolysis of the melamine resin (Table 2, EQ_{5,11}).

IR analysis of the cured film did not show any evidence of increased HN< levels, therefore, self-condensation of melamine according to EQ₅ must be taking place. The level of formaldehyde found in the reaction volatiles also indicates a demethylation reaction. The resulting -NH groups, from the demethylation reaction, react either with some remaining methylol or methoxy groups. There is obviously no way to account for the amount of water collected in the trap. All reaction water has to be derived from the reaction of methylol groups (Table 2, EQ_{2,3,8}). Assuming that some

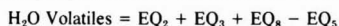
Table 5—Volatiles, Temperature—Amino Resin 1

Composition:	$MF_{6.1}M_{6.2} : NCH_2OH_{0.3} \cdot HCHO_{F 0.075} \cdot -CH_2OCH_2-_{0.51}$			
DP	1.68			
Film Thickness, μ m:	25			
Catalyst, %:	0.84			
Polyol/Amino Resin 1:	62.5/37.5			
OH/Triazine:	2.49/1			
BAKING TEMPERATURE 20 MIN., °C	125	150	175	200
MOL. VOLATILES/TRIAZINE				
HCHO	0.36	0.5	0.65	0.8
MeOH	2.6	3.1	3.6	4.3

Table 6—Volatiles, Catalyst Level—Amino Resin 1

Composition:	$MF_{6.1}M_{6.2} : NCH_2OH_{0.3} \cdot HCHO_{F 0.075} \cdot -CH_2OCH_2-_{0.5}$			
Film Thickness, μ m:	25			
Polyol/Amino Resin 1:	62.5/37.5			
OH/Triazine:	2.49/1			
Baking temperature, 20 min., °C	100 (212°F)			
		DRY (WET)		
CATALYST, %	0.88	1.75	2.625	3.5
MOL. VOLATILES/TRIAZINE				
HCHO	0.35 (1.19)	0.38 (0.48)	0.34 (0.39)	0.39 (0.54)
MeOH	2.44 (3.0)	2.5 (3.43)	2.35 (3.43)	2.50 (3.4)

of the water hydrolyzes the melamine resin, we arrive at the following equation for the reaction water:



The larger amount of water formed in the collector, therefore, has to come from water absorption of the uncured film and the panel. Therefore, in subsequent work, the amount of water collected was measured, but not reported.

Raising the level of amino resin 1 from 25% to 37.5% or a ratio of hydroxyl/triazine of 2.49/1 again produces higher than calculated levels of MeOH. At the 50/50 ratio of polyol/melamine resin, some traces of methylal were formed in the reaction mixture. This was the only occasion where the formation of methylal was ever observed. The overall reaction responsible for formation of methylal is shown in Table 2, EQ₇. Again, significantly higher levels of methanol volatiles compared to the theoretical level were observed. J. N. Koral and coworkers⁵ found in their work formation of methylal as self condensation product of hexa(methoxymethyl) melamine and concluded that methylal is the only reaction product of self condensation. Their experiments were done under dry nitrogen.

The fact that methanol is the reaction product of the self-condensation reaction and not methylal led to the concept that not only water in the film but possibly the humidity of the air could have an influence on hydrolysis and self-condensation reaction of a fully alkylated melamine resin.

Therefore, air was saturated at room temperature with moisture and introduced into the flow cell. This air will be identified in the experiments as "wet". Calculations show that at 150°C the relative humidity of such wet air is about 1%.

The experiments in Table 3 at the 62.5/37.5 polyol/melamine level were repeated with slight modifications. Panels were inserted into the oven at 100°C and heated within 8 min to 150°C. This slower heatup rate gave more reproducible results. The tests were further run with dried and wet air. The results are shown in Table 4. Surprisingly, a significant increase in HCHO (0.8 moles) and methanol (3.5 moles) volatiles was found, which shows that diffusion of moisture in the air into the

film can have a substantial impact on hydrolysis, demethylation, and self-condensation reactions.

In the presence of a sufficient excess of methoxymethyl groups, these groups will be hydrolyzed on extensive overbake; after loss of most of the methanol, hydrolysis of the melamine/polymer backbone linkages could take place. Indeed some evidence for this has been found in some coil coatings which can lose solvent resistance on overbake. Under most normal baking conditions, sufficient moisture is in the air to cause self-condensation of a hexa(alkoxymethyl) melamine resin. The release of formaldehyde from this reaction cannot be eliminated. Therefore, a reduction in residual methylol content of a hexa(methoxymethyl)melamine resin to reduce loss of HCHO by demethylation during cure is, under normal humid conditions, not sufficient.

In Table 5, the volatile loss at increased baking temperatures is shown. Indeed as expected, raising the temperature from 125° to 200°C increases the level of self-condensation reaction and about doubles the loss of HCHO.

Table 6 gives the volatiles as a function of catalyst level at dry and wet conditions. At 100°C cure temperature in a dry atmosphere, amino resin 1 does not self-condense even in the presence of large levels of acid

Table 7—Volatiles, Catalyst Level—Amino Resin 1

Composition:	$MF_{6.1}M_{6.2} \cdot NCH_2OH_{0.3} \cdot HCHO_{F 0.075} \cdot -CH_2OCH_2-_{0.51}$			
DP	1.68			
Film Thickness, μ m:	25			
Polyol/Amino Resin 1:	62.5/37.5			
OH/Triazine:	2.49/1			
Baking Temperature, 20 min., °C:	120 (248°F)			
		DRY (WET)		
CATALYST, %	0.88	1.75	2.625	3.5
MOL. VOLATILES/TRIAZINE				
HCHO	0.4 (.54)	0.45 (.56)	0.48 (0.7)	0.6 (0.84)
MeOH	2.56 (3.64)	2.85 (3.93)	2.77 (3.78)	2.93 (3.92)

Table 8—Volatiles, Dry-Wet—Amino Resin 2

Composition:	MF _{5.8} Me _{3.8} ; NCH ₂ OH ₁ , HCHO _{F 0.189} , -CH ₂ OCH ₂ - _{0.4}	
DP	2.5	
Film Thickness, μ m:	25	
Polyol/Amino Resin 2:	56.5/43.5	
OH/Triazine:	1.74/1	
Catalyst, %:	0.385	
Baking Conditions, 20 min., °C:	100 \rightarrow 125°C (212 \rightarrow 302°F)	
	<u>DRY</u>	<u>WET</u>
MOL. VOLATILES/TRIAZINE		
HCHO	0.85	0.96
MeOH	1.71	1.97

catalyst. Increased moisture in the air again significantly increases self-condensation. At a catalyst level higher than 1.75%, no further increase in self-condensation is found up to a level of 3.5%.

These data show that higher temperature and higher humidity in the air are mainly responsible for the self-condensation reaction of a fully alkylated melamine resin, but that increased acid catalyst levels are not. Therefore, decrease in performance of highly catalyzed coatings is probably not caused by a different extent of self-condensation reaction of the melamine resin, but rather by interaction of the acid catalyst with the substrate interface or by a change in migration rate of ion and water through the film.

Increasing the baking temperature from 100° to 120°C (Table 7) brings the expected increase in methanol and formaldehyde evolution, but otherwise, no change in the already observed pattern occurs.

In another set of experiments, the partially methylated melamine-formaldehyde resin, amino resin 2, was selected as a crosslinker (Table 8). This crosslinking agent has methylol and methoxy groups as potential functional sites. The films were predried for 45 min at 45°C to remove the n-butanol solvent after drawdown. Using a 0.38% catalyst level and a baking schedule of 20 min at 125°C, 0.85 mole of HCHO is released during

cure. This amount of HCHO is partially derived from free-formaldehyde (0.189) and partially from a demethylation reaction (0.66). This would indicate that, under the reaction conditions described, demethylation and formation of -NHCH₂OCH₃ groups (Figure 1, F₄) is a major reaction step. The molar amounts of methanol evolved are about equivalent to the moles of hydroxyl groups in the polyol used. This would indicate that methoxymethyl groups are the reactive site. Higher humidity also increases the HCHO and methanol volatiles. The difference between dry and wet conditions is smaller than for amino resin 1, although the absolute level of HCHO volatilized is higher. Therefore, demethylation is a major reaction step in the cure of a partially alkylated resin.

The rather high formaldehyde content in the volatiles of methylated melamine-formaldehyde resins is of concern, so a conventional solvent soluble butylated melamine-formaldehyde resin was used for comparison. This resin is typical of the majority of melamine resins used in coatings.

Amino resin 4, as shown in Table 1, is a polymeric resin. Approximate methylol content is 5-7% weight. This resin is supplied as a 75% solution in n-butanol. Most butylated resins are catalyzed with carboxylic acid. Therefore, 2% maleic acid was used as a catalyst.

Table 9 shows the volatile composition. The amount of formaldehyde released during cure is greater than that of the methylated resins tested. The n-butanol released is also of the same order as the hydroxyl groups in the blend. Increased humidity raises the formaldehyde to a very high level. There is some discrepancy in the amount of n-butanol released under wet conditions which remains to be resolved.

The reaction volatiles for all partially alkylated melamine-formaldehyde resins indicate that demethylation is a major reaction step (Table 2, EQ₁).

Therefore, if it is not the methylol but the $\text{-N}_\text{C}(\text{H})\text{CH}_2\text{OR}$ group which is the reaction site, significant reduction in formaldehyde could be achieved by preparing such a resin.

Amino resin 3, which is a higher alkylated, high NH resin, fits the description for such a product. Indeed,

Table 9—Volatiles, Amino Resin 4

Composition:	MF ₅ Bu _{2.8}	
Baking Conditions, 20 min. °C:	100 \rightarrow 150 (212 \rightarrow 302°F)	
Film Thickness, μ m:	25	
Catalyst, MA %:	2	
Polyol/Amino Resin 4:	56.5/43.5	
OH/Triazine:	2.14/1	
	<u>DRY</u>	<u>WET</u>
MOL. VOLATILES/TRIAZINE		
HCHO	0.96	1.43
n-BuOH	2.1	1.9

Table 10—Volatiles, Dry-Wet—Amino Resin 3

Composition:	MF ₄ Me _{3.4} ; NCH ₂ OH _{0.57} , HCHO _{F 0.07} , NH _{1.48}	
DP	2.1	
Film Thickness, μ m:	25	
Catalyst %:	0.385	
Polyol/Amino Resin 3:	56.5/43.5	
OH/Triazine:	1.46/1	
Baking Conditions, 20 min. °C:	100 \rightarrow 150°C (212 \rightarrow 302°F)	
	<u>DRY</u>	<u>WET</u>
MOL. VOLATILES/TRIAZINE		
HCHO	0.24	0.32
MeOH	1.88	1.91

this resin gives reduced formaldehyde emission under dry and wet conditions (Table 10). The increase in formaldehyde volatiles under wet cure conditions is still observable, but not nearly as pronounced as in a highly alkylated resin. (Amino Resin 1).

Using a weaker acid catalyst, such as a partial ester of pyrophosphoric acid, gives excellent cure with amino resin 3. Increased levels of catalyst at a baking temperature of 100°C produces a very low level of formaldehyde. The higher level of acid does not increase the formaldehyde loss (Table 11).

MECHANISM OF CROSSLINKING

Fully alkylated melamine-formaldehyde resins (amino resin 1) have been studied extensively by Koral, et al.⁵ The need for a strong acid catalyst, such as pTSA, is described. Weaker or undissociated acids do not effectively catalyze the reaction. Therefore, it can be assumed that specific acid catalysis is the catalyzing mechanism for fully alkylated melamine-formaldehyde resins.^{6,7} A proposed reaction pathway is shown in Figure 4. The formation of the carbonium ion determines the maximum rate which can be obtained. The carbonium ion can recombine with the split off alcohol or it can react with the functional groups on a polymer, such as hydroxyl, carboxyl, amide, or mercapto.

The overall rate of crosslinking will be determined by the reaction rates of all reaction steps. Indeed different rates of reaction have been observed for hydroxyl, carboxyl, and amide groups by Koral, et al.⁵ So, for example, an already formed crosslink between polymer and crosslinking agent can be reprotonated and form a carbonium ion which then in turn can react with the alcohol which has been split off from another alkoxymethyl group. Therefore, bonds are formed and broken continuously during the crosslinking process. The loss of the alcohol by diffusion and evaporation has, therefore, a significant influence on the reaction rate. Absolute rate measurements in a coating film are, therefore, virtually impossible. The rate of formation of the carbonium ion can be approximately measured if a mercaptan is used to scavenge the carbonium ion. The mercapto/melamine resin (NCH₂SR) linkage cannot be

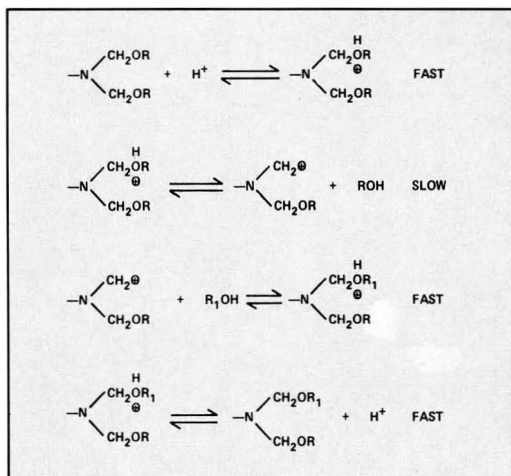


Figure 4—Reaction pathway (specific acid catalysis)

reprotonated and, therefore, is the end product of the reaction.

Most of the methanol collected as volatiles can be accounted for as a product of the crosslinking reaction. Assuming complete reaction of the functional groups of the backbone resin with the fully alkylated melamine-formaldehyde resin, for each mole of functional groups, one mole of methanol has been formed in the volatiles. Any excess methanol has to come from hydrolysis of the melamine resin (Table 2, EQ₅). This finding is also confirmed by the tests conducted under dry and wet conditions, which show significant increase in methanol and formaldehyde levels in the volatiles.

At the lower baking temperature especially, this appears to be the only mechanism which causes self-condensation of the fully alkylated melamine resin. It is rather surprising that water can, during the baking process, diffuse into the film and hydrolyze the amino resin. One would assume that the reaction volatiles such as methanol or formaldehyde would prevent any diffusion into the coating film. Calculations show that

Table 11—Volatiles, Catalyst Level—Amino Resin 3

Composition:	MF ₂ Me _{3.4} ; NCH ₂ OH _{0.57} ; HCHO _{F 0.07} ; NH _{1.48}			
DP	2.1			
Film Thickness, μm:	25			
Polyol/Amino Resin 3:	56.5/43.5			
OH/Triazine:	1.46/1			
Baking Conditions, 20 min., °C:	100 (212°F)			
CATALYST, DMAPP	1	1.5	2	2.5
MOL. VOLATILES/TRIAZINE				
HCHO	0.2	0.15	0.17	0.15
MeOH	1.35	1.3	1.3	1.36

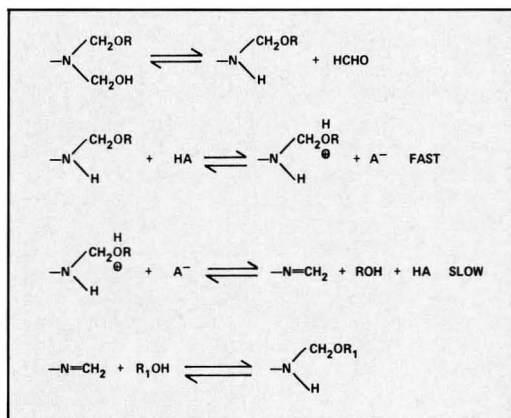


Figure 5—Reaction pathway (general acid catalysis)

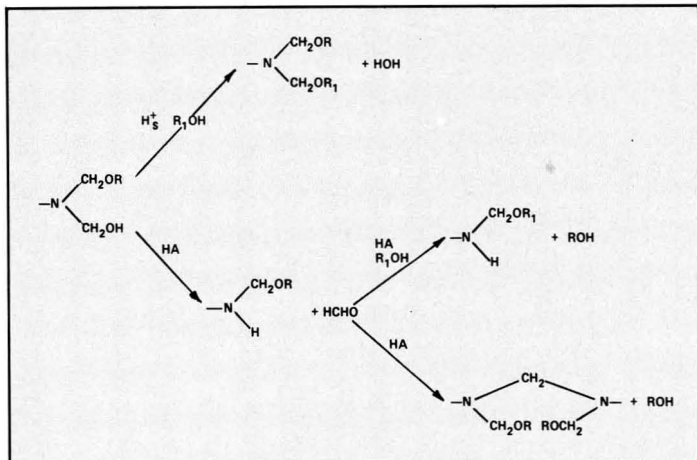


Figure 6—Reaction of partially alkylated melamine resin

about 10% of the water contained in the air flowing through the cell participates in the hydrolysis of the melamine resin.

The reaction of partially alkylated melamine-formaldehyde resins with hydroxyl functional polymers proceeds readily in absence of strong acid catalyst. The assumption that no catalyst is required is wrong. Completely neutral, salt-free systems will not react with a partially alkylated resin. For example, amino resins 2 and 3 will not react with the polyol used in this study at 150°C baking temperature. Kinetic hydrolysis studies of methylated trimethylol melamine and hexakis (methoxymethyl)melamine^{6,7} clearly show a different mechanism, indicating that general acid catalysis is involved in -NH CH₂OR functional amino resins. Therefore, any undissociated acid, salt, or weak acid should be a good catalyst.

These studies^{6,7} do not clarify the reaction mechanism of a partially alkylated melamine-formaldehyde

resin. Such a resin with $-N\begin{matrix} CH_2OR \\ CH_2OH \end{matrix}$ as a functional site

should react by a mechanism similar to a fully alkylated product; but the actual performance of a partially alkylated melamine-formaldehyde indicates a general acid catalysis mechanism.

Our work with a partially alkylated melamine-formaldehyde resin (amino resin 2) shows that demethylation (Table 2, EQ₁) is a major reaction step in a partially alkylated melamine-formaldehyde resin. Therefore, the behavior of a partially alkylated and a fully alkylated, high NH melamine-formaldehyde resin is very similar in a coating formulation. This is not surprising because, after demethylation of the partially alkylated melamine-formaldehyde resin, a fully alkylated, high NH melamine-formaldehyde resin is formed. Figure 5 shows the reaction path. In this mechanism, possibly a Schiff base intermediate is formed and not a carbonium ion. The resulting Schiff base is reactive with hydroxyl, amide, and other -NH groups of

the melamine, but not very reactive with carboxyl groups.

In most coating systems, one can, of course, observe an overlap of specific or general acid catalysis. Figure 6 gives the reaction products of a partially alkylated melamine-formaldehyde resin under specific and general acid catalysis conditions. Volatile tests have indeed shown that reduction in formaldehyde can be observed using increased levels of a strong acid catalyst (H⁺s).

With highly alkylated, high NH melamine-(amino resin 3) formaldehyde resin, the cure response is significantly better with a weaker acid than a stronger acid.

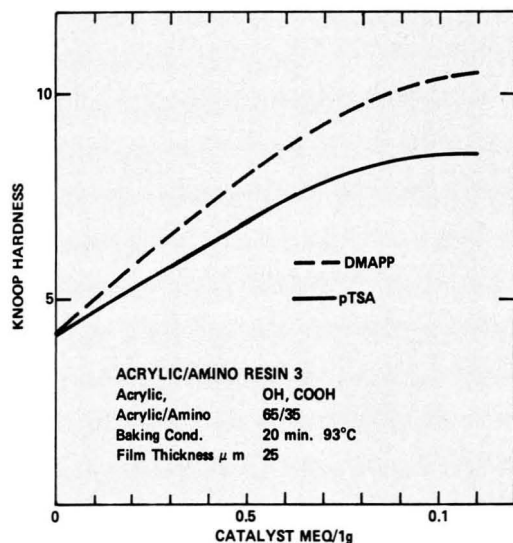


Figure 7—Comparison of acrylic/amino resin 3 using alkyl acid pyrophosphate and p-toluene sulfonic acid as catalysts

Therefore, only a weak acid is an acceptable catalyst. Figure 7 gives a comparison of a hydroxyl functional acrylic resin crosslinked with amino resin 3 and catalyzed with an alkyl acid pyrophosphate and *p*-toluene sulfonic acid catalyst. At equal MEQ of acid in the system, the stronger acid is a poorer catalyst. This effect can be explained by the increased basicity of the triazine ring of a highly alkylated, high NH melamine-formaldehyde resin leading to a protonation of the ring and reduction in reactivity.⁸

Comparing the cure response of amino resins 2 and 3 in a coating system (Table 12), it can be seen that the highly alkylated, high NH amino resin is faster reacting than the partially alkylated resin at lower cure temperatures (120°C). The demethylation step which is quite fast at higher temperatures slows considerably at temperatures below 120°C.

The difference in cure mechanism between fully alkylated and partially alkylated, fully alkylated, high NH melamine-formaldehyde resins have significant implications from a formulation standpoint.

Fully alkylated melamine-formaldehyde resins give good cure response in the presence of a strong acid catalyst, and the cure response can be significantly inhibited by amines and pigments with reactive surfaces. Contrary to the common opinion, fully alkylated melamine-formaldehyde resins are not slower curing than partially alkylated resins, if properly catalyzed.

Partially alkylated and fully alkylated, high NH melamine-formaldehyde resins are, with respect to catalysis, much more forgiving. Amines will reduce the cure response, but will not inhibit the reaction; pigments will have no or only a slight effect on the cure rate. Stability, on the other hand, is not as good as that of fully alkylated resins, even in absence of a strong acid catalyst.

In water-borne coatings, the amine salts of the carboxyl groups are an excellent catalyst for partially alkylated or high NH resins, regardless of the pH of the system. Therefore, stability of fully alkylated resins in water systems is superior to partially alkylated resins.

Table 12—Low Temperature Cure—White Gloss Enamels

Total Non-Volatile — 60%
Pigment — 44% TiO₂
Binder — Alkyd/Amino = 65/35; Alkyd.
Total Solvent — Xylene/Butanol = 75/25
Film Thickness, μ m 25

AMINO RESIN	2		3		4	
CATALYST DMAPP % OF TRS	—	0.8	—	0.8	—	0.8
CURE: 20 MINUTES AT 93°C.:						
HARDNESS, KNOOP, KHN ₂₅	1.0	5.0	2.5	9.0	2.0	6.0
HARDNESS, PENCIL	5B*	B-HB	B-2B	HB-F	5B*	B-HB
MEK RESISTANCE, DOUBLE RUBS	5	50	5	125	10	75
CURE: 20 MINUTES AT 121°C.:						
HARDNESS, KNOOP, KHN ₂₅	11.0	—	15.0	—	11.5	—
HARDNESS, PENCIL	H-2H	—	H-2H	—	H-2H	—
MEK RESISTANCE, DOUBLE RUBS	200*	—	200*	—	200*	—

Table 13—Low Temperature Cure of Combinations Of Amino Resins

Unpigmented Systems		Cure — 20 Minutes at 93°C.				
Substrate — Zinc Phosphate Treated CRS						
SYSTEM		1	2	3	4	5
ALKYD	SOLIDS, WEIGHT %	70	70	70	70	70
AMINO 1	SOLIDS, WEIGHT %	15	15	15	—	30
AMINO 3	SOLIDS, WEIGHT %	15	15	15	30	—
CATALYST	DMAPP, % ON TRS	0.5	—	1.0	1.0	—
CATALYST	DBSA, % ON TRS	0.84	1.68	—	—	1.68
DRY FILM THICKNESS, μ m		21	21	21	21	22
HARDNESS, KNOOP, KHN ₂₅		2.2	1.7	2.3	8.0	7.0
MEK RESISTANCE, DOUBLE RUBS		25	20	25	100	200+

The only way to improve stability in a water system would be to choose resins with very low acid number, thus reducing the level of catalyst in the system. This improved stability can indeed be observed in emulsions which give excellent stability with partially alkylated melamine resins.

It is also a fallacy to expect improved stability in an amine neutralized, water-borne coating if the strong acid catalyst, such as pTSA, is omitted from the formulation.

On baking, most of a strongly basic *t*-amine will have to leave the film before a fully alkylated melamine-formaldehyde resin will start to react. This temperature is usually above 135°C. Addition of larger levels of acid catalyst will not enhance the cure response at lower temperatures. A partially alkylated or a high NH melamine-formaldehyde resin will react in the presence of the amine, or at about 110-125°C; this can result in poorer water-resistance and increased blister tendency.

Crosslinking agents responding to two different mechanisms of catalysis should not be blended in a formulation if low temperature cure is desired (Table 13). The increased basicity of the partially alkylated or the high NH melamine resin is sufficient to neutralize portions of the strong acid catalyst and, therefore, reduce the acidity of the system. The weak acid catalyst on the other hand will not catalyze the fully alkylated resin. This effect of cure inhibition is only observable at lower cure temperature, but not at temperatures above 150°C.

CONCLUSION

Understanding the mechanism of catalysis of amino resins is essential to predict the behavior of a crosslinking agent in a complex paint system. General acid and specific acid mechanisms are responsible for catalysis of different amino resins. A. Berge¹ distinguishes between primary ($-\text{N}(\text{CH}_2\text{OR})$) and secondary

$-\text{N}(\text{CH}_2\text{OR})\text{CH}_2\text{OR}$ alkoxyethyl ether in melamine resins;

kinetic hydrolysis studies show that primary ether groups are catalyzed by a general acid catalysis mecha-

nism and secondary ether by a specific acid mechanism. Unfortunately, existing commercial partially alkylated amino resins, which are known by experience to be catalyzed by weak acidic catalyst or by a general acid catalysis mechanism, do not contain a sufficient amount of primary ether groups to account for such a mechanism. Our work has shown that demethylation of partially alkylated melamine formaldehyde resins and the formation of primary ether groups is a major reaction step. The thus formed primary ether functionality is then catalyzed by a general acid catalysis mechanism. Highly alkylated high NH resins low in methylol content also have shown to be reactive by the same weak acid catalyst, which confirms A. Berge's proposed mechanism.

Fully alkylated, only secondary alkoxy groups containing melamine resins require a strong acid catalyst for cure, which agrees with A. Berge's mechanism. In previous publications,⁵ formation of methylal was claimed as the reaction product of the self-condensation reaction. Under practical conditions, in the presence of small amounts of moisture, no formation of methylal was observed; however, it was found that hydrolysis and demethylation is the actual mechanism of self-condensation of hexa(methoxymethyl)melamine. This work also shows that the composition of the volatile reaction products depends to a large extent on the composition of the amino resin, the catalyst used, and the application conditions. Conventionally used, partially butylated and methylated melamine formaldehyde resins emit the largest level of formaldehyde during cure, fully methylated melamine formaldehyde resins

emit less, and highly methylated high NH resins do emit the least formaldehyde. Increased moisture in the baking oven has significant effect on increasing formaldehyde emission and self-condensation. A similar trend is seen with increased baking temperature. Higher catalyst levels surprisingly have only a small effect on the level of formaldehyde emitted and higher levels of catalyst combined with a reduction in baking temperature can actually reduce formaldehyde emission.

ACKNOWLEDGMENT

The author thanks Dr. I. H. Updegraff for his patience in conducting the volatile study and Dr. G. G. Parekh for his contribution in the mechanistic studies.

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Colorimetric Assessment Of the Degree of Flocculation Of Inorganic Color Pigments

Siegfried Keifer
BAYER AG*

In the case of inorganic color pigments, flocculation causes characteristic shifts in the color effect as compared with the ideally dispersed pigment. The flocculates are not regarded as coarser particles, but as local areas of higher pigment concentration. This principle makes it possible to establish by experiment a relationship between the pigment concentration—or between the distance apart of the pigment particles—and a selected color value. As a result, it is possible, conversely, to determine the mean concentration in the flocculates with the aid of an existing color value. The relative degree of flocculation derived from this enables—for the first time with inorganic color pigments—information to be obtained about the packing density in the flocculates.

INTRODUCTION

The coloring effect of an inorganic color pigment within a certain type of pigment is influenced, above all, by the particle size and the particle size distribution—factors which are fixed by the manufacturer during production. Because of the relatively high refractive index of inorganic pigments, the scattering power plays a major role in the color effect. As is known, color is the result of the counter-play of absorption and light scatter. Since the scattering power is dependent not only on the particle size but also on the distance between the particles, i.e., on the pigment concentration and on the uniformity of dispersion of the individual pigment particles in the medium (the state of flocculation), the said geometric parameters have a very considerable influence on the color effect.

The aim of this work was to investigate the extent to which particle distance, the distance apart of the individual pigment particles, influences the coloring of inorganic color pigments. Conversely, the extent to which the flocculation can be quantitatively determined from the amount in which the color in a given paint differs from a system in which the pigment is, for all practical purposes, ideally dispersed was also studied.

FLOCCULATION OF INORGANIC COLOR PIGMENTS

According to a definition given in DIN 53 206 (particle size analysis, basic terms), a flocculate is an agglomerate occurring in pigment/binder systems that can be broken up by low shear forces. This definition harbors the risk that, as a result of the use of the word agglomerate, flocculates are imagined to be something which they are only in exceptional cases. Agglomerates are instinctively thought of as globular, tightly packed accumulations of individual pigment particles. The level of flocculation is assessed through the size of these accumulations. However, with inorganic pigments, a picture such as this is very rare and usually occurs as the result of shock effects due to incorrect letting-down of mill pastes. Strictly speaking, these agglomerates are not flocculates any more, since, generally, they cannot be broken up again with the finger in the rub-out test or on recoating.

In an earlier paper, determination of the degree of flocculation of white pigments through the use of the scattering power was studied.¹ Flocculation was defined as local convergences of individual pigment particles to less than the expected mean distance on the basis of the nominal pigment concentration with equidistant distribution. This definition can easily be extended to inorganic color pigments. The flocculates are then regarded as areas of higher PVC than the nominal PVC expected from the formulation. This obviously means that there must also be areas in the film with lower pigment concentration or even without any pigment. These pigment-free areas are the most striking feature of electron micrographs taken of flocculated systems with inorganic pigments. The flocculated pigments surround these pigment-free areas in relatively loosely packed, multi-strand, three-dimensional chain and lattice structures. The more they are in evidence, the more pronounced is the flocculation.

In this connection, it is helpful to refer to the scanning electron micrographs, published by Zorll,² of ionetched film cross-sections which illustrate the described phenomena in an almost picture-book form.

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*Technical Service Dept. for Pigments, Krefeld-Uerdingen, Germany.

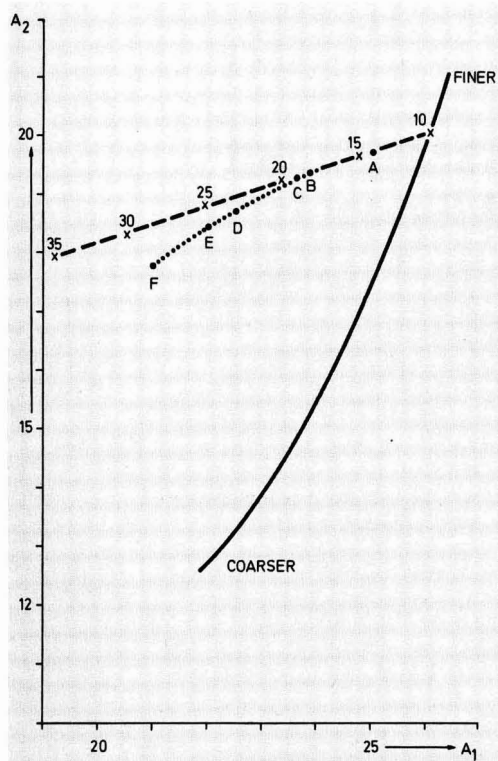


Figure 1—Shade of iron oxide red pigments as a function of particle size. Legend:
 function of particle size —————
 PVC 10-35 - - - - -
 degree of flocculation A-F ······

Particularly impressive are the pigment distributions or pigment-free areas in relation to the PVC, a subject which will be dealt with later.

Aside from the findings that can be derived from electron micrographs, there are other points which argue against describing the flocculates as coarser par-

ticles. The optical properties of flocculated systems, in particular, can be better described as differing concentration distributions in the film, which Balfour and Hird³ did, also using TiO₂ pigments. They were also able to show that the individual pigment particles in a flocculate are nearly resolved by visible light, meaning that the flocculate does not behave optically like a coarse particle.

The relationship between the change in shade and the particle size or the distance between the particles can be demonstrated very clearly with the aid of iron oxide red pigments. Figure 1, in a chromaticity plane of the AN system, shows the change in shade of iron oxide red as a function of particle size. The unbroken curve starts in the top right corner with the finest iron oxide red. The broken straight line details the change in the color coordinate of a fine-grain iron oxide red pigment with the PVC, i.e., with the particle distance in ideal dispersion. The points plotted with letters are the color coordinates of this pigment in binders of different wetting power, i.e., with different degrees of flocculation. With increasing PVC, or decreasing particle distance and increasing degree of flocculation, the shade changes only slightly towards blue. The saturation nevertheless decreases considerably. Up to medium degrees of flocculation, the color coordinates follow the PVC line. Only with heavy flocculation does the shade shift somewhat more noticeably towards blue. While the saturation declines slightly with increasing particle size, the shift in color is much more marked. The flocculates do not behave in the manner of coarser particles in this example either.

According to our definition, the wider the particle distance distribution curve, the greater the flocculation of a system. This is illustrated in the graph in Figure 2. The relatively tight distribution curve of the particle distance with ideal dispersion widens out during flocculation and the maximum shifts in the direction where the distance between the particles is shorter.³ While there is a large number of smaller particle distances in the flocculates, the low-pigmented areas can count only a small number of larger particle distances. This is the

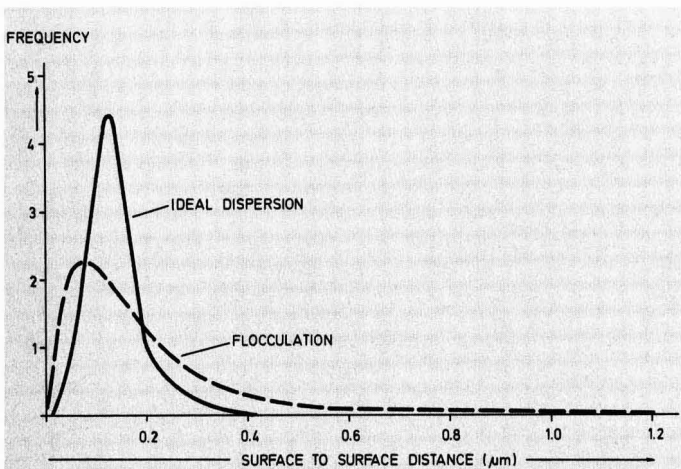


Figure 2—Particle distance distribution curves on ideal dispersion and on flocculation. Particle size: 0.28 µm; PVC: 15%

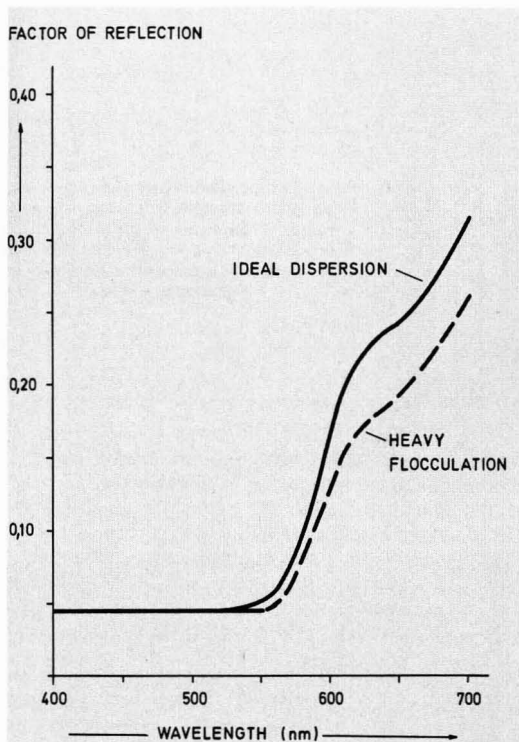


Figure 3—Reflection spectrum of iron oxide red (PVC 10%)

main reason for the decline in the scattering power in the flocculation of scattering pigments. The scattering power is dependent, first of all, on the number of scattering particles per unit volume. If the distances between the particles become shorter than half the wavelength of light, because of increasing pigment concentration or because of flocculation processes, there will be disruptions in the scattering processes. This can be explained simply by saying that light is no longer able to resolve two individual pigment particles whose distance apart is less than half of the wavelength of light, i.e., they act optically like one scattering particle. The number of particles capable of scattering decreases again below a certain particle distance. The resolving capacity for individual pigment particles by red light is comparably less than by blue light. On reducing the distance, the scattering power for the red light drops more appreciably than for the blue light, and, as a result, it is generally a shift to blue that occurs.

Until now, it has been assumed that the change in color that occurs with the flocculation of inorganic color pigments is based on the decline in the scattering power. On comparing a flocculated and a non-flocculated system of the same pigment, it is immediately evident that, irrespective of the color, the flocculated system always appears darker, less colorful, and somewhat bluer. This in itself suggests a decline in the scattering power. It is improbable that, because of flocculation, the absorbing power, i.e., the color strength, increases and, as a result, the sample becomes darker.

The decrease in saturation could also arise through a lower absorbing power. A comparison between the reflection spectra of a flocculated and a non-flocculated system (Figure 3) shows clearly that it is the reduction of scattering power that is primarily responsible for the decline in saturation. The difference in height between the absorption range and the maximum of the scattering range in the reflection spectra gives an indication of the colorfulness or saturation of the system.

RELATIONSHIP BETWEEN PARTICLE DISTANCE AND COLOR EFFECT OF INORGANIC COLOR PIGMENTS

According to the definition previously given, maximum flocculation exists when the entire pigment is flocculated up to the CPVC and the remaining volume of the film is pure binder. There is no flocculation if the nominal PVC is uniformly distributed over the entire film. The object is to establish the intermediate values via the level of color deviation so that the degree of flocculation can then be determined.

As a result of shortening the distance between the particles on flocculation, the color of an inorganic color pigment changes in a characteristic fashion compared with the same pigment in an ideal dispersion. If the change in color quality is linked with the particle distance, or more simply expressed, with the PVC—then it is possible, conversely, to obtain information about the mean PVC of the flocculates, defined here as the flocculation PVC, or about the degree of flocculation. A necessary condition for this is that the color values of the color pigment are determined in ideal dispersion as a function of the PVC.

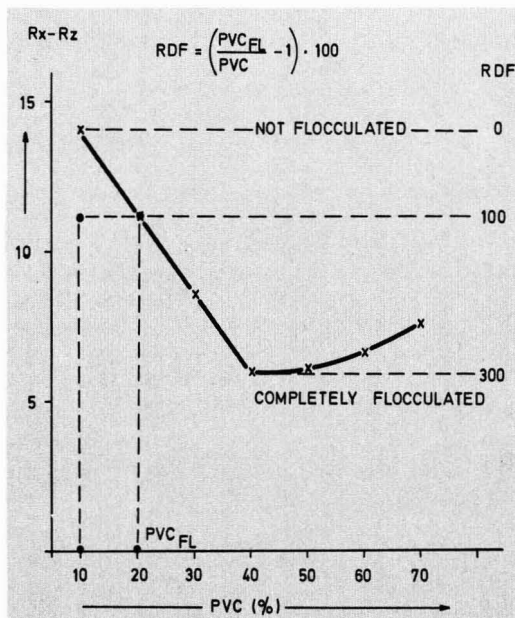


Figure 4— $R_x - R_z$ value of a fine-particle iron oxide red pigment as a function of PVC; determination of flocculation PVC (PVC_{FL})

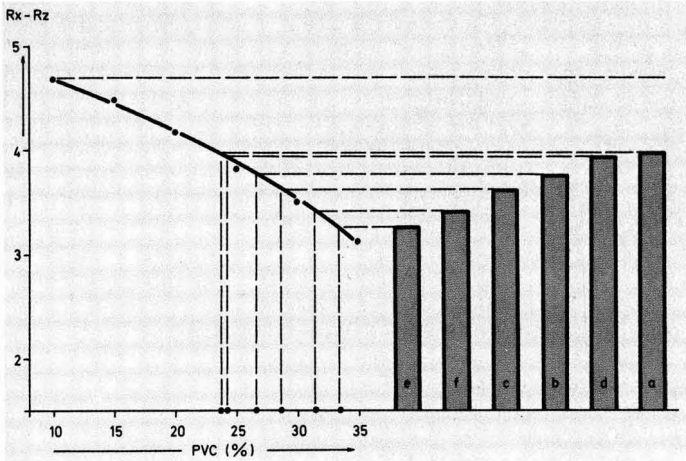


Figure 5—Flocculation behavior of a chrome oxide green pigment in various binders. Legend: a. Medium-oil Alkyd Resin; b. Short-oil DCO Alkyd Resin; c. Short-oil Fatty Acid Alkyd; d. Acrylic Resin; e. Epoxy Resin; f. Polyurethane Resin

Figure 6—Flocculation behavior of an iron oxide yellow pigment in various binders. Legend: (see Figure 5)

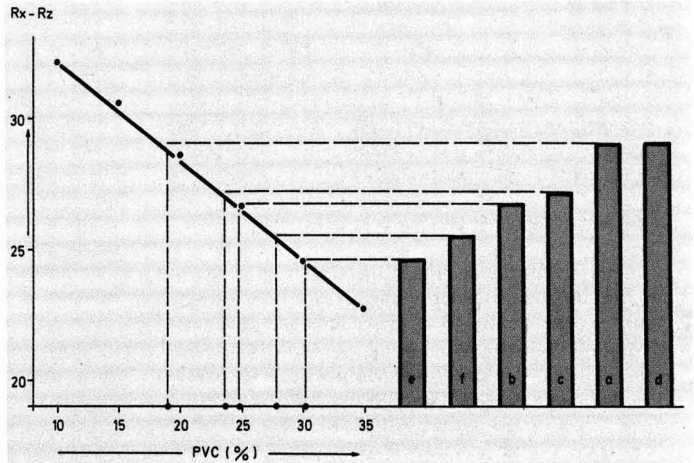


Figure 7—Flocculation of an iron oxide red pigment in various binders. Legend: (see Figure 5)

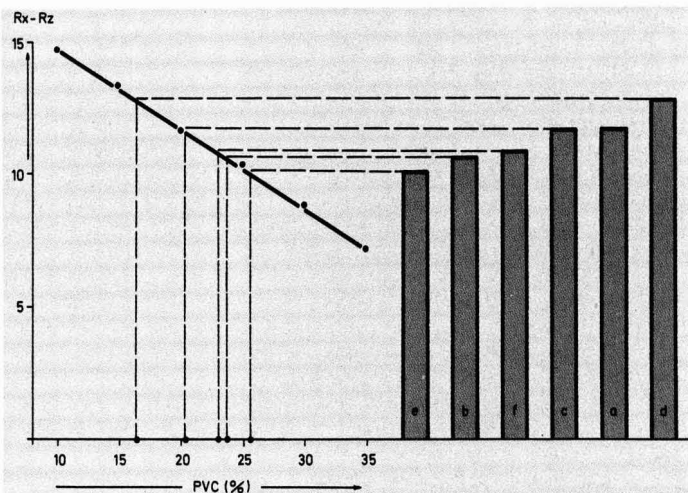


Table 1—Relative Degree of Flocculation Of Some Inorganic Color Pigments in Various Binders

	Chrome Oxide Green	Iron Oxide Yellow	Iron Oxide Red	\bar{M}_2
Medium-oil alkyd resin	138	90	104	111
Short-oil DCO alkyd resin	170	149	136	152
Short-oil fatty acid alkyd resin	188	137	104	143
Acrylic resin	144	90	64	99
Epoxy resin	234	202	156	197
Polyurethane resin	216	179	130	175
\bar{M}_1	182	141	116	—

In the familiar rub-out test, a kind of ideal dispersion state of the pigment is achieved by rubbing shortly before the drying starts. The obvious would be to produce, by rubbing, surfaces with an ideal dispersion state in a relatively well wetted system in which the risk of rapid reflocculation is small. Furthermore, if this is done in relation to the PVC, then all the experimental preconditions for drawing up a calibration curve are fulfilled. A disadvantage of this method is that the experimental uncertainties of the rub-out test are transferred to the calibration curve. In addition, the preparations for this method are rather complicated.

Paste methods are unsurpassable in their accuracy, reproducibility, and speed. Particularly good results have been achieved with Alkydal® L 64 (100%), which is also used in the DIN 53 165 for determining the relative scattering power. The pastes can be stabilized with the addition of, for example, up to 5% by volume Aerosil® 200, calculated on the binder. Color pastes of this kind do not exhibit any change in shade due to flocculation even after several hours. The color pastes are milled on an automatic muller at 4 × 25 revs, brushed onto a paste dish, and immediately measured according to DIN 53 236 process A, i.e., under the inclusion of gloss. Generally speaking, 6 to 8 PVC values are enough to plot a calibration curve.

The question arises as to which color value or color quality should be plotted against the PVC in order to obtain a calibration curve for determining the flocculation PVC. This depends on the individual color pigment and cannot be answered generally. Basically, though, the color value which changes most distinctly with the PVC or with flocculation should be chosen.

From the reflection spectra in Figure 3, it is evident that, for the example of iron oxide red, use could be made of the R_x or the R_y value or the corresponding color values A_2 or A_1 in the AN system and b^* or L^* in the CIELAB system. Particularly useful is a difference value like the $R_x - R_z$ value which covers the difference in height between the absorption range and the maximum of the scattering range of the reflection spectrum. This also correlates well with the saturation, which clearly changes on flocculation. Difference values have

the advantage in that they do not react quite as sensitively as, for example, the R_y value to influences exerted by the measuring apparatus or to the nature of the specimen surface. Wherever there was no reason against it, the $R_x - R_z$ value was preferred for plotting the calibration curves.

Figure 4 gives a somewhat more comprehensive calibration curve for a relatively fine-particle iron oxide red pigment. In this example, there is a linear relationship between $R_x - R_z$ value and the PVC to the CPVC. The determination of the flocculation PVC (PVC_{FL}) is easy to understand from the diagram. A coating pigmented at a PVC of 10% with this iron oxide red pigment gives a certain $R_x - R_z$ value. The PVC belonging to this value is the flocculation PVC, as in the example, 20%. Optically, this system behaves as if it were pigmented at PVC 20%. The flocculation PVC alone does not yet reveal enough, since there is a difference in whether a flocculation PVC of 20% is established for a coating formulated at a PVC of 5% or at one of 15%. To be correct, it must be related to the PVC given in the formulation. The relative degree of flocculation (RDF) is calculated as follows:

$$RDF = \left(\frac{PVC_{FL} - 1}{PVC} \right) \times 100$$

In the formula, PVC represents the nominal PVC and PVC_{FL} represents the determined flocculation PVC. The relative degree of flocculation of 100, calculated from the example in Figure 4, indicates that the pigment packing is on the average 100% denser than it should be for an ideal dispersion at this PVC of 10%.

EXAMPLES OF APPLICATION FOR THE RELATIVE DEGREE OF FLOCCULATION

Relative Degree of Flocculation Of Some Inorganic Color Pigments In Binders with Different Wetting Power

From the large number of tested binders and pigments, a few typical examples were selected as being

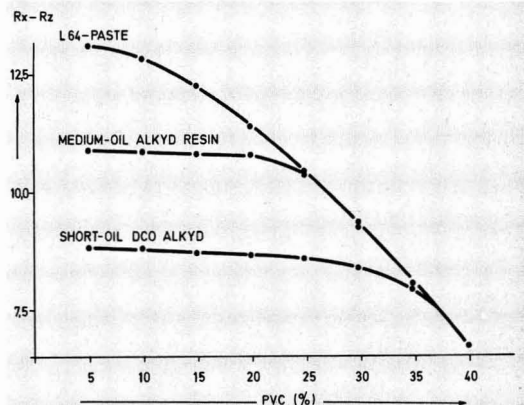


Figure 8— $R_x - R_z$ value of an iron oxide red pigment as a function of PVC

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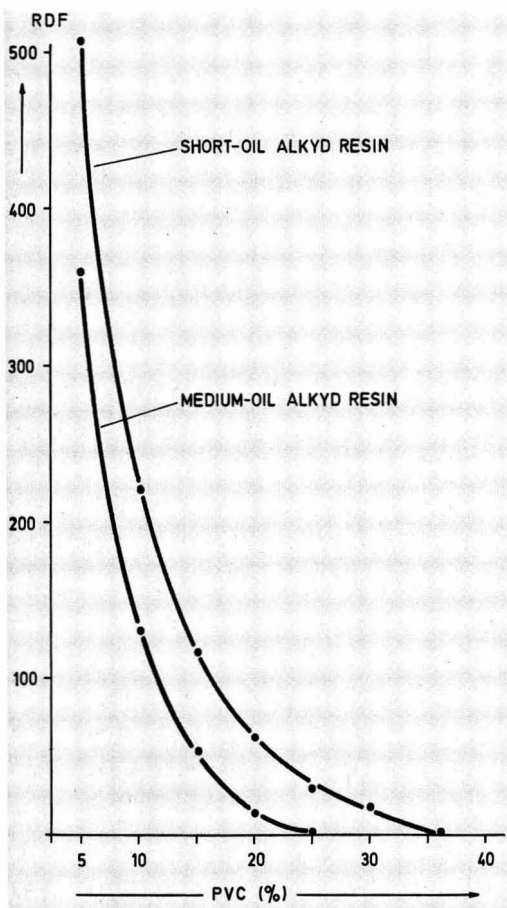


Figure 9—Relative degree of flocculation of an iron oxide red pigment as a function of PVC

representative. Of the binders, these were: medium-oil alkyd resin; short-oil DCO alkyd resin; short-oil fatty acid alkyd resin; acrylic resin; epoxy resin; and polyurethane resin. In these binders, the relative degree of flocculation at a PVC of 10% was determined for an iron oxide red, an iron oxide yellow, and a chrome oxide green pigment. Alkydal L 64 pastes were used for the calibration curves. The flocculation PVC was determined via the $R_x - R_z$ value. The flocculation behavior of the inorganic color pigments in the various binders is illustrated in Figures 5-7. The relative degrees of flocculation are summarized in Table 1. The mean values \bar{M}_1 allow more general information to be given about the flocculation behavior of the pigment, while the mean values \bar{M}_2 provide information about the wetting power of the binder for the three inorganic color pigments.

**Relative Degree of Flocculation
As a Function of the PVC**

With light-scattering color pigments, as already shown, the color effect is dependent on the pigment

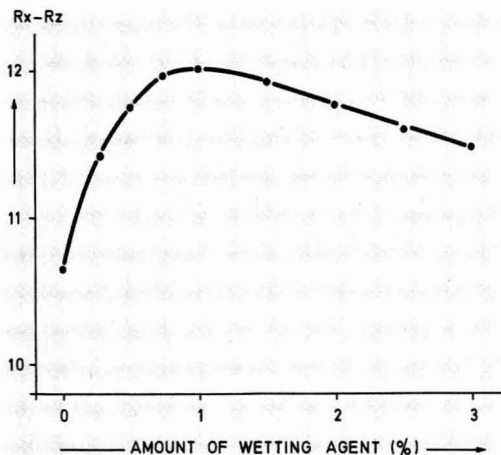


Figure 10— $R_x - R_z$ value of an iron oxide red pigment in a short-oil alkyd resin as a function of amount of wetting agent

concentration. Nevertheless, one often observes that, in relatively poorly wetted systems, the shade appears to be independent of the pigment concentration over wide ranges. An interesting example for this is shown in Figure 8. An iron oxide red pigment was incorporated into an L 64 paste, in a medium-oil, and into a short-oil alkyd resin in the PVC range of 5 to 40% and the $R_x - R_z$ value plotted against the PVC. As expected, an ideal dispersion resulted in the paste. In the medium-oil, and particularly in the short-oil alkyd, a certain flocculation PVC occurs over a wide PVC range depending on the

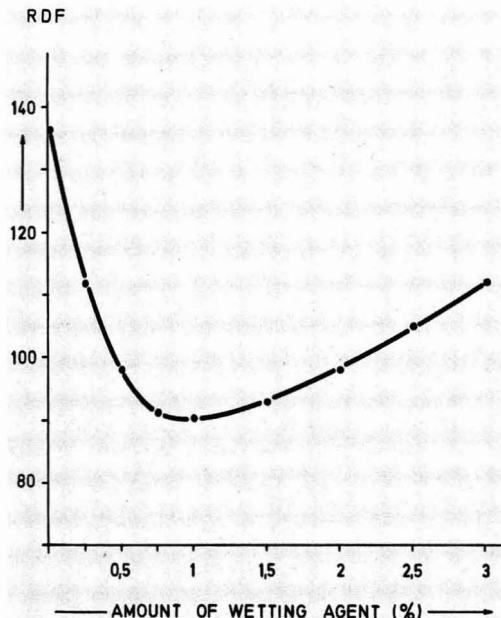


Figure 11—Relative degree of flocculation of an iron oxide red pigment as a function of amount of wetting agent

position of the wetting equilibrium. Raising the PVC does not evidently bring about any increase in the density of the pigment packing in this range. The pigment, which has accumulated locally up to the flocculation PVC, leaves behind pigment-free areas in the system. These are gradually filled with increasing pigment concentration. This process can be followed remarkably well with the scanning electron micrographs from Zorll² previously mentioned. This filling of the pigment-free regions continues without an increase in the density of the pigment packing and, thus, also without any major change in shade until the nominal PVC has reached the flocculation PVC. From this point, the sense of the definition of flocculation previously given is no longer applicable. Furthermore, no rub-out effect can be established.

Although the iron oxide red pigment becomes more compact from a wide PVC range up to a virtually constant flocculation PVC, the relative degree of flocculation decreases with increasing PVC because it is related to the formulated PVC. This is illustrated in *Figure 9*. The relative degree of flocculation declines relatively strongly with rising PVC and reaches zero when the PVC in the formulation has reached the flocculation PVC.

Influence of the Amount of Wetting Agent On the Relative Degree of Flocculation

When flocculation problems arise, help is often sought from some kind of wetting agent. Unfortunately, the most common wetting agents for preventing floating or sedimentation are flocking agents. The effectiveness of one of the rare wetting agents actually capable of reducing flocculation is shown in *Figure 10*. Increasing amounts of the wetting agent were added to the mill

base of a relatively heavily flocculating system, a fine-grain iron oxide red in a short-oil alkyd resin. The decline in flocculation can be followed from the increase in the $R_x - R_z$ value. The optimum addition in this case is approximately one percent wetting agent, calculated on the pigment. When higher quantities are added, flocculation increases again.

The relative degree of flocculation as a function of the proportion of wetting agent, shown in *Figure 11*, makes it possible to see clearly what can be achieved with this wetting agent and how far there is to go to reach the ideal target of complete deflocculation. In the given example, there is still quite a way to go.

CONCLUSION

In this study, it has been shown that the difference in a given color value of an inorganic color pigment in a flocculated and in an ideally dispersed system can be used to determine the degree of flocculation. A precondition for this was that the relationship between the packing density or PVC of the color pigment and a selected color value was determined by experiment. The flocculation PVC or the relative degree of flocculation calculated on the PVC in the formulation can be taken from this and permits, for the first time, quantitative information to be obtained about the mean pigment concentration, i.e., the packing density in the flocculates.

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A Tank Cleaning System For the Small Paint Plant

Alun G. Morris
L.V. Lomas Chemical Co.*

Many of the smaller paint plants are cleaning their portable mixing tanks by hand with strong solvents. This practice is dangerous and unhealthy to the person doing the cleaning and the solvent costs are rising.

There is a need for a safe, effective and inexpensive means of cleaning these portable tanks and drums.

Such a system is described, a system that could be installed by most plants' own maintenance people.

Introduction

There are many small paint plants' employees cleaning portable tanks by hand with solvents. This procedure is not only costly, with the increasing expense of solvents, but it has also been found to be extremely unhealthy and hazardous for the worker.

A simple, high pressure alkali cleaning system could alleviate this problem. The system need not be too complicated, nor should it have to be expensive. There are a number of commercial systems on the market and, if cost permits, it is suggested that these be employed. An adequate system, however, can be installed by factory maintenance personnel without too much expense. Such a system will be considered.

Basic Design

There are a number of choices possible when designing a cleaning system, both as to size and complexity. Many small companies may want to clean only portable tanks, others may want to clean larger fixed mixing tanks, as well

as five gallon pails and drums. (Figure 1).

The basic equipment would consist of:

(1) A storage tank to store and heat the cleaning solution.

(2) A high pressure centrifugal pump to circulate the hot cleaning solution under pressure to the tank cleaning area.

(3) A rotating jet (Figure 2) or nozzle (Figure 3) to spray the cleaning solution against the walls and bottom of the tanks being cleaned.

(4) A lid that would fit securely over the tanks while they are being sprayed. A good thickness of soft rubber should be vulcanized to the underside of the lid to permit a good seal to the top rim of the tanks. Attached to this lid would be the spray nozzle. The lid could be raised and lowered by an air cylinder.

(5) A low pressure return pump to return the cleaning solution to the storage tank, unless it can be returned by gravity.

(6) A means of straining the solution before it is returned to the storage tank.

(7) A means of rinsing the cleaned tank with clean hot water.

Operation

The actual operation can be carried out by one man. It should take ten to twenty minutes per tank, depending on how hard the paint has dried on the walls of the tank and the type of paint to be removed. If the tanks can be kept covered from the time they are emptied until they are cleaned, the cleaning job will be much easier.

Before putting a portable tank in place prior to spraying, it should be checked for contents. Any appreciable amount

of wet paint left in the tank should be removed, rather than allowed to contaminate the cleaning solution. The type of paint left in the tank and the degree to which it had been allowed to dry on the tank walls should be observed. This will help in estimating how long the spray system should be applied.

When the lid is closed and the spray system is turned on, the lip of the tank should be checked for leaks. It is possible that the top of some tanks may not be level enough to make a good seal with the lid. If the operator has to leave the area while the system is on, a suitable warning sign should be well displayed.

If the tank does not have a sloping bottom, it would be helpful if it was slightly tilted in the direction of the outlet, to permit reasonable draining. This allows for better spray cleaning of the bottom of the tank.

After an adequate length of time, determined by experience, etc., the spray system should be shut down, the lid raised, and the tank inspected. If the tank is clean enough, it should be rinsed with hot water. The rinse water should be fairly hot so that the heat absorbed by the tank walls will assist in drying traces of rinse water that may be left at the end of the rinse cycle.

Since very little rinse water should be necessary, a common household hot water tank should suffice, providing it has extra high wattage immersion heaters for quick heat recovery. It is unnecessary to have a hot water recovery system, since little hot water will be used.

The hot water rinse could be applied by a hand operated spray gun or incorporated into the line feeding the spray nozzle. If the latter system is employed, a suitable check valve would have to be

Presented by Mr. Morris on behalf of the Toronto Society for Coatings Technology at the Annual Meeting of the Federation of Societies for Coatings Technology, Nov. 1-3, in Chicago, Ill.

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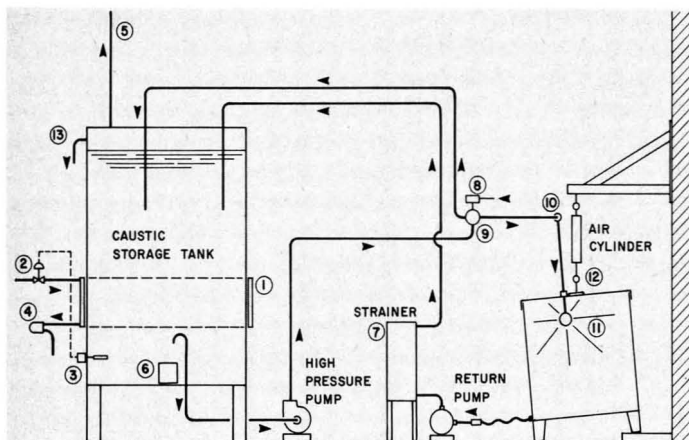


Figure 1—Kettle washing system

installed in the line to prevent hot caustic from getting into the hot water tank.

Tote Tanks

The same spray nozzle and tank cover could be used to clean tote tanks. However, modifications would have to be made to the lid to make it adaptable for this purpose. Since the opening of most tote tanks is 22 inches (0.56 m) in diameter, this system could also be used for cleaning open head drums.

Five Gallon Pails

With modifications, the system could be extended to include the spray washing of five gallon pails (20 litres) and

small gallonage mixing tubs. Only one high pressure delivery pump would be required for both systems.

The small container system would involve inverting the container to be cleaned over a spray nozzle. As the pail is placed over the nozzle, it should activate an air micro-switch that would open an air-operated valve, permitting the cleaning solution to enter the spray nozzle only when the pail is over the nozzle. Remote operation would be most desirable from a safety point of view. A drain pan should be suitably located underneath this pail cleaning station to catch the cleaning solution, allowing it to be pumped back to the cleaning solution holding tank.

Drums

A system for cleaning drums, both open and closed head, which is similar to that described for five gallon pails could be used. Spray nozzles, small enough to go through a standard two-inch (508 mm) bung opening, are commercially available. As with five gallon pails, inverting the drum over the spray nozzle permits constant draining of the cleaning solution.

The drain pan or trough should have the bottom sloping to an opening with a pipe connection. The drain piping should be so arranged as to permit the cleaning solution to be pumped back to the solution holding tank and the rinse water to be diverted to the sewer, if not re-cycled.

Stationary Mixing Tanks

Stationary mixing tanks can be included in the system, if required. (Figure 4). A larger pump would be needed if

many tanks are involved. Also, an additional return pump may be needed, unless the returns could be brought back by gravity flow.

The use of flexible hoses to permit moving the spray head from one tank to another is handy. This practice is, however, more dangerous than if solid piping were used throughout. If flexible hoses are used, they should be of the metal braided type with end connections welded to the braid. A hose should be selected with an inner liner that is able to withstand the temperature and chemical effects of the cleaning solution. Rotating jets are preferable for use on the larger tanks, but an adequate job can usually be done with spray balls if the tanks are not too large.

If spray nozzles or troll balls are used, openings on the tips of the tanks can be made to accept a three inch standard pipe nipple. These openings should not be too close to the tank agitator shaft, otherwise, the shaft could interfere with the spraying effect of the spray ball. A second opening on the opposite side of the agitator shaft would eliminate any dead spots created by the shaft.

Roto jet type spray nozzles are preferred for cleaning larger tanks. These can be inserted into the tank through the loading manway. A lid, with a suitable seal, would have to be made up to fit over the manway, with a means of clamping or bolting it down tightly.

Tanks being cleaned should be well vented. A six inch (0.15 m) vent on a 2500 gallon (10000L) tank is not too large. The air in a tank can expand sub-

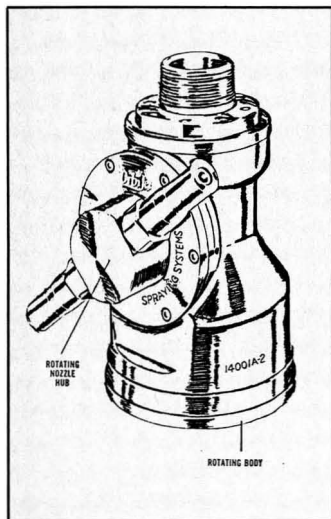


Figure 2—Typical rotating jet

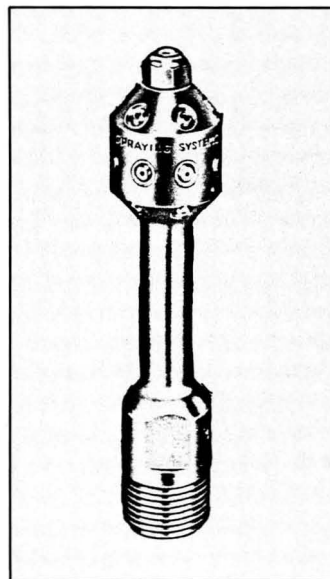


Figure 3—Spray nozzle

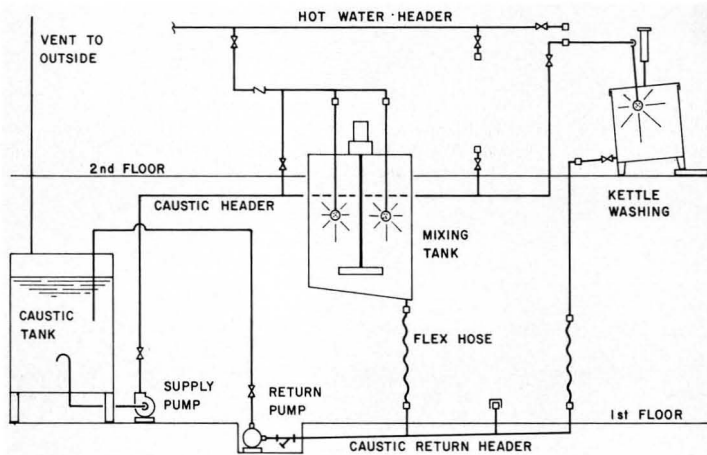


Figure 4—Hot caustic tank cleaning system

stantially when heated by a hot spray solution. Conversely, if one were to rinse the tank with cold water after a hot cleaning solution has been used, a partial vacuum could be created, sufficient to pull in the sides of the tank. Hot water should be used for rinsing.

Return Line

Draining the cleaning solution from the tank should not create a problem. As the liquid is not under pressure, standard paint hose should be adequate for draining purposes. It is important to label the tank bottom valve, and the hose to the effect that hot caustic is being used.

Safety

While a hot alkali cleaning system can be very effective and economical, it must be treated with the utmost regard for safety. When the system is in operation, signs to indicate this should be well displayed. Access to the area should be restricted. Adequate caution signs should be posted on all equipment and transfer lines containing caustic. Signs should be placed not only around any mixing tank that is being cleaned, but on any hose or other equipment containing cleaning solution as well.

Care must be taken not to use hot alkaline solutions to clean tanks containing aluminum paints or nitrocellulose coatings. The area where portable tanks are being cleaned should be well ventilated and well lit.

The operator must be supplied with proper safety equipment, such as goggles and face mask, rubber apron, gloves and boots. Emergency showers

and eye wash stations must be located in the immediate area. An operator training scheme should be established.

The Cleaning Solution

For purposes of cleaning, two major types of solvent-based paints are considered: conventional alkyd enamels, and epoxies and acrylic resins.

Cleaning solutions for alkyd type paints usually consist of alkali blends in a concentration weighing from 5 to 15%. Since a relatively high temperature is important, a minimum of 160°F to 200°F, (71°C to 93°C), and preferably between 180°F to 200°F, (82°C to 93°C) should be used.

There are many good cleaning solutions that are commercially available for

this purpose, as well as such additives as wetting agents, surfactants, and chelating agents that can be added to caustic solutions. In the interests of economy and ecology, it should be mentioned that these materials themselves are not necessarily highly toxic solutions, as are some paint removers.

Special, more complex additives are required for epoxies and acrylic resins. These, however, are also commercially available.

When cleaning solutions become contaminated, they can be decanted, separating them from the resins and pigments that have settled out. After the solution is allowed to cool overnight or over a weekend to about 80°F to 90°F, (27°C to 32°C), the resins will float to the surface and are able to be skimmed off. When the liquid is decanted off into drums, the heavy sludge that has settled to the bottom can be scraped out into properly marked drums for disposal. The liquid may then be returned to the tank and titrated to the recommended strength. In this way, it is possible to reclaim 75% of the solution.

Installation of the System

SUPPLY PUMP

The supply pump is the heart of the operation and will no doubt be the most expensive individual piece of equipment in the system. It should be a stainless steel pump. Although the cost is high, stainless steel will last longer. Many companies have found that black iron pumps are adequate and less expensive.

A centrifugal pump has an advantage over a rotary gear pump in that it will

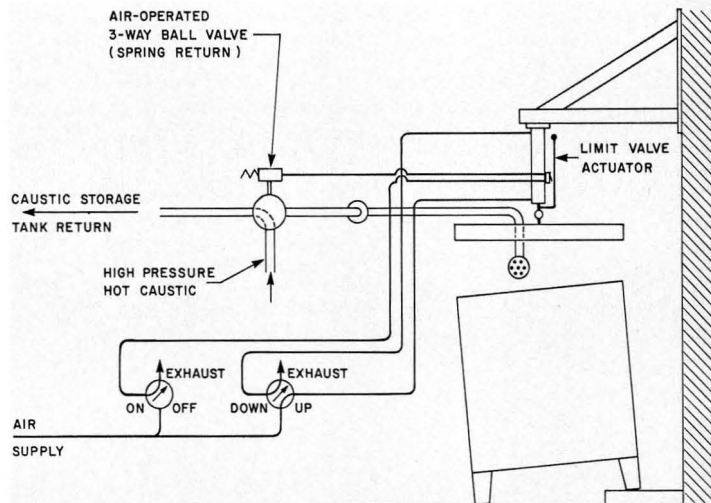


Figure 5—Kettle washing controls

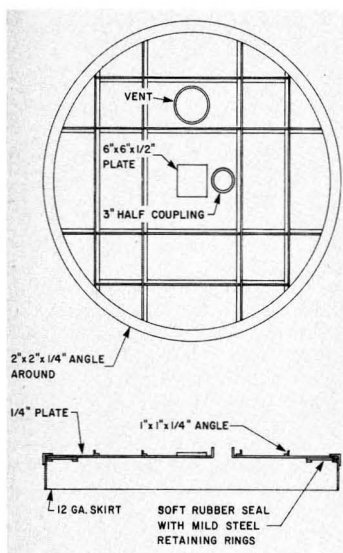


Figure 6—Kettle washer lid

continue to pump low viscosity material even if the blades are slightly worn. A direct drive 1800 r.p.m. pump should give less trouble than a 3600 r.p.m. pump.

A good mechanical seal is suggested; preferably a double mechanical seal with a water flush between seals. Each seal should consist of a stationary carbon seal with a silica-free ceramic rotating, held by 316 stainless steel spring.

It is recommended that the pump have good flooded suction with minimal restrictions between the tank and the pump. The line from the storage tank to the pump should be somewhat larger than the entrance port of the pump. If there is a strainer ahead of the pump for protection, the strainer should be of a good size with reasonably large openings in the strainer basket in order that a minimum of restriction will be created. To prevent the build-up of excessively high pressure after the pump, a relief valve should be installed in the line. The piping should be so arranged that, if the pressure is relieved, it will go back to the storage tank.

STORAGE TANK

Any tank of reasonable size, e.g., 500 gallons (2000L), can be used for this purpose, provided it would not be affected by the hot cleaning solution. It would be preferable to have the heating coils, (Item 1 in *Figure 1*), on the outside, as it would be easier to clean out the tank, and any steam leaks would be more readily noticed. Also, there would be less chance of cleaning solution getting sucked into the steam system when the steam line is shut off and a vacuum is created as the steam in the line condenses. The tank should be well insulated.

The steam coil should be equipped with a steam regulating valve, (Item 2), actuated by a thermocouple, (Item 3), located in the tank, and a steam trap, (Item 4). The tank should have a vent, (Item 5), leading to the outside of the building. It should also have a reasonably sized clean out door, (Item 6). A pressure type filter, (Item 7), should be installed after the return pump.

An air-operated actuator, (Item 8), can control a three-way valve, (Item 9), that will divert the high pressure cleaning solution back to the storage tank when the spray ball is not in operation. *Figure 5* shows an air-operated valve being controlled by an air limit switch on the lid. This should be installed in such a way that the spray can only work when the lid is down on the kettle. A universal pipe joint, (Item 10 in *Figure 1*), is preferred to the use of a flexible hose.

A spray ball, (Item 11), that will spray in all directions, upward as well as downward, should be used, particularly if cleaning tote tanks is anticipated.

The air cylinder, (Item 12), should be well sized, permitting the washer lid to be lifted easily. An air cylinder supplier can give you assistance in choosing the adequate size, provided he is given information regarding the weight to be lifted and the variation in the heights of the tanks to be cleaned.

An overflow drain, (Item 13), should be provided, since there is a tendency for the caustic solution to saponify long oil alkyds. Any material that overflows should be caught in a drum or another suitable container.

KETTLE WASHER LID

The washer lid (*Figure 6*) should be so constructed that it will not flex when pressure is applied by the air cylinder. It is recommended that it be reinforced with one inch by one inch angle iron. (0.025 × 0.025 m). A soft rubber seal, a half-inch (0.0125 m) thick should be secured to the bottom of the lid around the outer perimeter. It should be wide enough to manage all the sizes of tanks which will be cleaned.

Besides the opening for the spray ball, it would be beneficial to have an opening for a vent, complete with a flexible vent hose. A heavy, half-inch (0.0125 m) plate should be used to secure the air cylinder piston.

A good skirt, at least six inches (0.12 m), must be provided around the lid. If there should be any leaking of the solution around the top of the tank, the skirt will cause it to run downward, rather than be sprayed outward.

Conclusion

Safety must be the first consideration when working with hot caustic. It is imperative that every safety measure previously mentioned be included in the operation. There is good reason to believe that the health hazards associated with the cleaning of portable tanks by hand with strong solvents are more serious than most people realize.

Also, many plants, if not most, do not really know how much solvent is being used for washing tanks, nor do they realize the costs of disposing of the dirty solvents.

The cleaning system just described cannot help but improve working conditions in the tank cleaning area, save considerable labor, and reduce cleaning material costs. This is a project that will benefit both workers and companies.

Acknowledgment

The author thanks the Spraying Systems Company, North Avenue, Wheaton, Illinois, 60187, U.S.A. for the pictures of the spray nozzles and the W.L. Avery Technical Services for the drawings.

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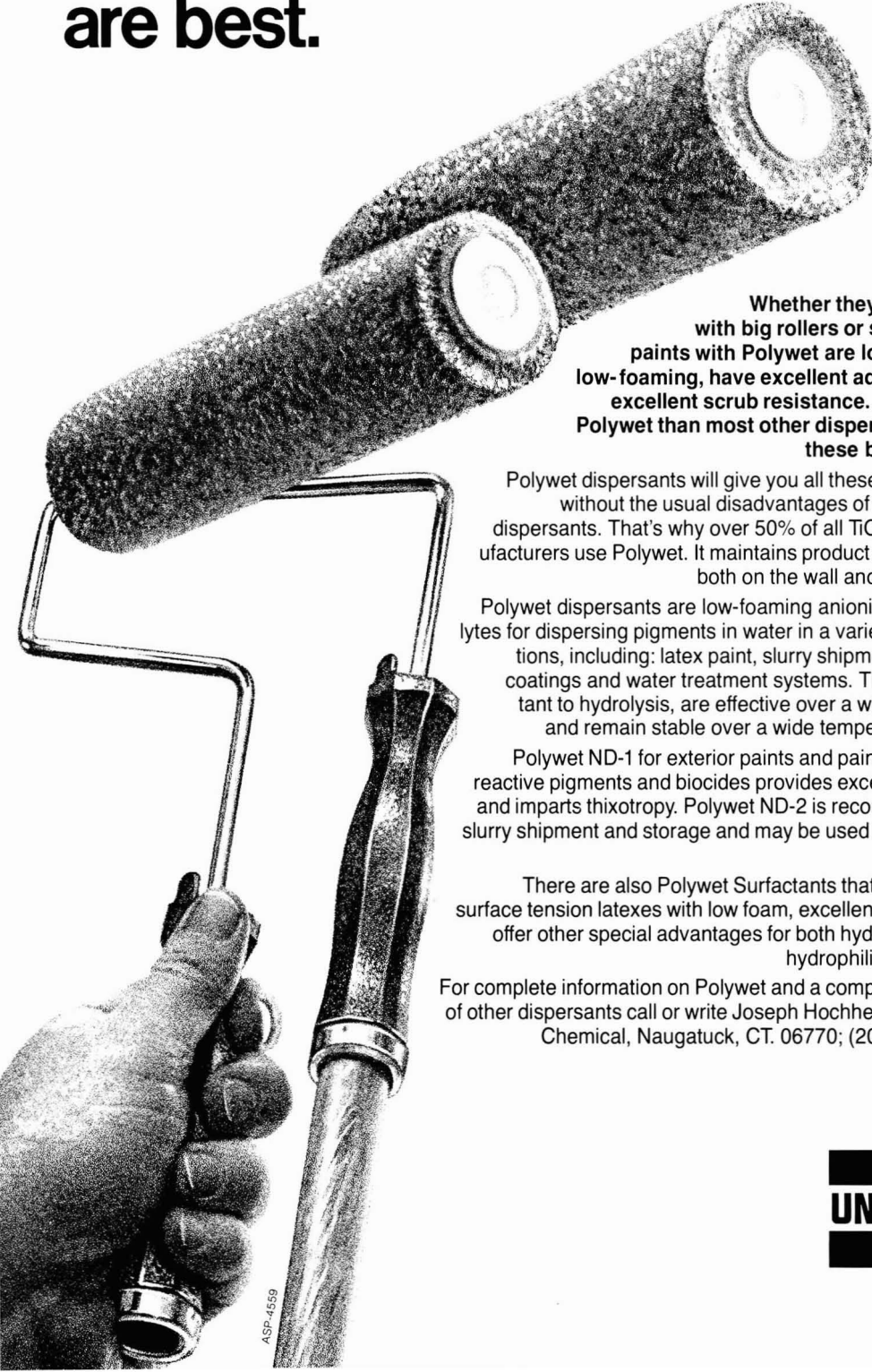
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Federal Specifications: The Key to Government Requirements and Testing

Robert F. Brady, Jr.
General Services Administration*

There are approximately 600 federal specifications and standards for paints and coatings. These documents are the key to annual GSA procurements of about \$80 million. Test methods found in specifications are often unique and are not available from any other source. This paper describes the content and coverage of federal specifications, their importance to the coatings industry, and suggestions on how to obtain and use federal specifications. An invitation is extended to manufacturers to participate in the review of draft specifications.

Introduction

Paints and coatings bought in the United States are usually manufactured to conform to an appropriate specification. This is particularly true for the Federal Supply Service (FSS), the division of the General Services Administration which buys much of the paints and coatings sold to the government. About 600 federal specifications and standards are used in FSS procurement.

A specification may be defined as a clear and accurate description of the technical requirements for a material, and the test methods by which it is demonstrated that the material meets the required properties. Thus, a specification is not a research and development document, nor can it be compared to a publication in a scientific or technical journal. A specification is a document for federal procurement, used to ensure that items purchased by the govern-

ment are suitable for the intended uses, and that maximum value is obtained for every dollar expended. Many FSS purchase contracts make reference to one of more federal specifications.

There are about 320 Federal specifications for paints, coatings, and related products. In addition, there are about 240 military specifications, and three major federal standards. Almost all of these are performance specifications, calling out the minimum end use requirements needed to meet the customer's needs. Very few are composition specifications, in which ingredients and the amounts to be used are specified. There are a few composition specifications left, but as part of a specification improvement program now being vigorously implemented by the Federal Supply Service, these are being changed into performance specifications.

Federal specifications are the responsibility of the Office of Standards and Quality Control of the Federal Supply Service, General Services Administration. Most Federal specifications are written in this office, but a small proportion is written by other federal agencies which have either specialized technical competence or a specialized use for the material. GSA, however, is responsible for the final acceptance and publication of the document. This office is responsible for all federal specifications for paints and coatings, and serves as a point of contact for information on military specifications on paints and coatings.

The federal government is the largest customer of the American paint and coatings industry, although our procurements account for only a small pro-

portion of total industry sales. To use rough figures, combined industrial and trade sales revenues were about 5.1 billion in 1975. The Federal Supply Service alone spent \$77 to 80 million on paints and coatings (1.6% of the total) and the sum of all government purchases was estimated at \$250-300 million (4 to 5% of industry sales). Furthermore, many state, county, and municipal governments, institutional and educational bodies, and commercial firms use federal specifications for their own purchases. The federal government interposes no objection to this practice, because federal specifications are public documents prepared at public expense. Thus, the impact of federal specifications for paints and coatings is not limited to federal procurements alone, but is multiplied many times as specifications are used over and over again in state and local procurements.

Content of a Specification

Following are six sections of a specification and the information that is contained in each, without going into too much detail. The six sections of a federal specification are: Scope, Applicable Documents, Requirements, Quality Assurance Provisions, Preparation for Delivery, and Notes. These sections are described out of order, for purposes of clarity.

SECTION 1, **Scope**, is a clear and concise abstract of the coverage of the specification. It summarizes the intent of the material, and gives some information on composition, properties such as color, and use.

* Presented at the 54th Annual Meeting of the Federation of Societies for Coatings Technology in Washington, D.C., October 29, 1976.

* Federal Supply Service, Code FMBP, Washington, D.C. 20406.

SECTION 3, Requirements, is the heart of the specification. The essential requirements applying to performance, and standards of quality and workmanship which the paint must meet to be acceptable are stipulated, and a definite basis is defined for rejection in those cases where the paint is unsuitable. The product is described in a manner which encourages competition and eliminates, insofar as possible, any restrictive features which might limit acceptable offers to the products of one or relatively few suppliers. Only the actual needs of the government are specified, and this is done in such a way that the product will usually be a product manufactured by the normal manufacturing operations of many potential suppliers.

SECTION 4, Quality Assurance Provisions, stipulates all the tests and methods of analysis that are to be performed to ensure that paint offered to the government conforms to all requirements listed in Sections 3 and 5. Methods for taking samples from lots of material, and for inspection for packing and marking are included by reference. Standard industry practices for testing and inspection are used. Whenever possible, such standard test methods as are found in the Gardner-Sward Paint Testing Manual, Federal Test Method Standard 141A, or in ASTM standards are cited. For all other cases, the complete test method is described in Section 4.

Many specifications contain in Section 4 unique and valuable test methods not found in other sources. If there is a need to demonstrate coating performance beyond the scope of ordinary tests, tried and proven methods are already available in federal specifications in many cases for this purpose.

SECTION 5, Preparation for Delivery, covers requirements for packaging, packing and marking for shipment.

SECTION 6, Notes, contains information of a general or explanatory nature, including a description of the intended

use of the paint, ordering data, and miscellaneous information.

SECTION 2, Applicable Documents, is a bibliography of specifications for materials and standard test methods which are referenced in the parent specification. In order to use the specification properly, and profit from all the information contained in it, it is important to have these reference documents on hand. Unfortunately, this has been a source of difficulty in the past because of what we call the "Christmas tree effect"—one paint specification will reference five other federal specifications; each of those five will reference five specifications; each of those 25 . . . and so on. Sometimes you can go full circle and come back to the original paint specification. As part of its specification improvement program, GSA is presently reducing the number of "first tier" documents—those cited in Section 2 of the parent specification. It will then be much easier to use a specification.

Recommended Reference Materials

To take advantage of all the information that is available in federal specifications, and also to use any federal document to its maximum potential, it is suggested that a library of federal paint specifications and reference documents relating to them be acquired and maintained. A reference collection of documents should include the following eight items:

(1) The "Index of Federal Specifications and Standards," which is issued annually by GSA. It lists federal specifications alphabetically and numerically, and specifies the cost of copies available for sale. The index also includes information concerning federal standards, federal qualified products lists, and related information. Cumulative supplements to the index are issued each month. The Index may be purchased on a subscription basis at \$10.75 per year from the Superintendent of Documents, U. S. Government Printing Office (GPO), Washington, DC 20401. (202-655-4000).

(2) A collection of all paint specifications, and also those specifications most frequently referred to in paint specifications. These specifications are available from the GPO, or better, from the Specification Distribution Branch, Code 3FRI, Building 197, Washington Navy Yard, General Services Administration, Washington, DC 20407. (202-

472-2205). The price of each specification is given in the Index of Federal Specifications and Standards.

(3) Federal Test Method Standard 141A, entitled "Paint, Varnish, Lacquer and Related Materials; Methods of Sampling, Inspection and Testing." This document is a compilation of some 160 standard test methods for paints and coatings, and is available from the specification distribution branch for \$3.75.

(4) Federal Color Standard 595A, which contains one-half by one-inch chips of the 499 colors procured by the Federal Supply Service. This is also available from the Specification distribution branch at a cost of \$2.75. A set of three by five-inch color cards is also available for \$33; individual color cards are seven cents each.

(5) The Department of Defense Index of Specifications and Standards (DODISS), issued annually with cumulative supplements. Part 1 is a straight alphabetical listing and Part 2 is a numerical listing. The numerical section separately identifies federal and military specifications, federal and military standards, and canceled documents by their individual numbers. This is available from the Government Printing Office, Washington, DC 20402; the current subscription rate is \$15 per year. Individual military specifications and standards are available from GPO, or better, from the Naval Publications and Forms Center, 5801 Tabor Ave., Philadelphia, Pa. 19120 (215-697-2000).

(6) A set of the latest ASTM Standards for paints and coatings, available from the American Society for Testing and Materials, 1916 Race St., Philadelphia, Pa. 19103 (215-569-4200). In 1979, these standards are contained in Volumes 27, 28, and 29 of the annual ASTM standards.

(7) The Gardner-Sward Paint Testing Manual, Thirteenth Edition, obtainable from the American Society for Testing and Materials, list price \$27.50.

(8) Reagent Chemicals (Fifth Edition), published by the American Chemical Society, 1155 Sixteenth St., NW, Washington, DC 20036, list price \$40. This is a collection of specifications for solvents and analytical reagents, many of which are routinely used in paint testing.

It is also useful to point out a few things that are *not* found in federal specifications. Rarely will there be found a formula for a particular product. There are no recommendations for raw materials, for brand names of equipment, nor for manufacturing techniques. Also, there is in federal speci-

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cations no reference to contractual matters.

Federal Standards

GSA is also responsible for three major federal standards relating to paints and coatings. There are different types of standards: one type has the objective of reducing the number of sizes and types of materials in a supply system. There is no such standard for paints, but there is for colors — Federal Color Standard 595A. From the infinite number of colors discernible by the human eye, 499 are contained in Federal Color Standard 595A, in order to reduce the number of colors in the federal supply system and in order to ensure that, for example, paint, furniture, and carpeting manufactured separately will harmonize when placed together in a federal office. The standard is described in this way because there is nothing "sacred" or "better" about our colors; these are simply the ones bought by the government.

A second type of standard establishes reference test methods, processes, or analyses that are used repetitively to ensure that procured products conform to the applicable specification. Federal Test Method Standard 141A, entitled "Paint, Varnish, Lacquer, and Related Materials; Methods of Inspection, Sampling, and Testing," is such a standard. When Revision A was published

in 1965, it contained approximately 290 standard test methods for paints and coatings. In 1972, the Army Coatings and Chemicals Laboratory, Aberdeen, Md., reported the results of a study on the equivalence of Federal Standard 141 methods of ASTM methods. Subsequently, in March 1974, by means of Change Notice 4 to Federal Standard 141A, about 130 ASTM methods were established as equal to a method in standard 141A. Federal Test Method Standard 141A is used throughout the paint and coatings industry.

Federal Test Method Standard 370, entitled "Instrumental Photometric Measurements of Retroreflective Materials and Retroreflective Devices," has recently been completed. This standard defines quantities used in, and establishes experimental procedures for, the measurement of light reflected from beaded traffic paint on runways or on highways, and from other retroreflective traffic signs and markings.

The wealth of information available in federal specifications, and how it may be used, has been described. It is also important to describe how this information gets into a specification. Each time a specification is written, revised, or amended, the proposed document is circulated to all government users of the material, and also to technical societies, trade associations, and manufacturers. Each party is asked to suggest changes that will improve the

specification. Time and other considerations permitting, industry comments and suggestions are not only welcomed, but are freely solicited and accorded full consideration. This process ensures that the documents reflect the latest developments of modern technology, and set forth requirements which can normally be satisfied by a regular commercial production. Clearly, specifications benefit from rigorous review by manufacturers in this proposed stage, and all manufacturers are invited to participate in this review of draft specifications. Copies of proposed specifications are sent to those manufacturers who express interest for their review. All comments which are submitted are studied and carefully evaluated.

Summary

A wide variety of federal and military specifications are used by the Federal Supply Service for procurement of paint and related products. These documents contain valuable information not available from other references. Complete directions for obtaining and using paint specifications are given. Proposed specifications are circulated for comment before issue, and responses are solicited and carefully reviewed. Participation by manufacturers is invited, and will ensure that the specification will describe a high-quality product.

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Journal of Coatings Technology

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.

June 1979 Subcommittee Reports Of ASTM Committee D-1

The June 1979 Meeting of ASTM Committee D-1 on Paint and Related Coatings and Materials was held on June 10-13, at the Shoreham-Americana Hotel in Washington, D.C. During the three and one-half days preceding the final general meeting of Committee D-1, approximately 200 registrants met in 147 scheduled meetings of D-1 subcommittees and working groups. Membership of Committee D-1 is now 582.

One of the highlights of the meeting was an open forum with representatives from four regulatory agencies of the U.S. Government. Alan Roberts, of DOT; Donald McCaulley and Alan Ehrlich, of CPSC; Virginia Steiner and Gary McAllister, of EPA; and Michael Moore, of OSHA, responded to a number of questions on current and pending regulations.

Award Presentations—Richard W. Scott, Chairman of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Products, was presented with the Henry A. Gardner Award for outstanding competence in the management of a D-1 group.

Subcommittee Officers appointed by Chairman S.B. Levinson were: Sub. D01.29, Substrate Preparation for Coating Testing—S.D. Ozenich, Chairman; Sub. D01.44, Traffic Paints—E.B. Countryman, Chairman replacing A.J. Zuick; Sub. 56, Printing Inks—J.M. Fetsko, Chairman replacing F.A. Falk; and Sub. D01.90, Executive Committee Member-at-Large—R.C. Marck to fill the unexpired term of R.R. Bennett.

Highlights

The following projects of major interest to the coatings industry represent new efforts since the previous reports in 1978 and January 1979:

D01.21—establishment of a new research group to explore feasibility of chemical and instrumental analysis applicable to the paint industry.

D01.21.13 and .24—work with the Environmental Protection Agency on the all-important definitions of Volatile Organic Content (VOC).

D01.23.11—development of a method for measuring wet film thickness in field applications using notched gauges.

D01.26—formation of a new task group to study visually perceived attributes of the appearance of materials.

D01.26.11—study of a recommended practice for visual intercomparison of gloss surfaces.

D01.27.06—survey to establish a new detergent formula representative of current products on the market.

D01.27.17—review of methods relating to evaluation of weathering effects.

D01.29—formation of a new subcommittee to develop methods of preparation of uniform metallic and non-metallic substrates used as a base for paint testing.

D01.33.12—study of a gas chromatography method for isophorone diisocyanate.

D01.33.14—conduct a round robin on a new gas chromatography method for orthophthalic acid and fatty acids in alkyd resins.

D01.33.26—development of methods for freeze-thaw, heat-age, and mechanical stability of latices including round robin tests.

D01.42.03—study to simplify method for evaluating leveling of paints by brushout.

D01.45.07—development of a new anti-fouling rating system.

D01.51.02—development of methods for melt viscosity of resins, ICI plate and cone viscometer; color of powder coating resin solutions; and color measurement of powder coatings using a tristimulus colorimeter.

D01.51.03—develop method on impact fusion.

D01.52.12—develop method for determining the durability of textured interior panels.

Group 52/55—develop a method for determining stability of UV curing coatings.

D01.55.07—develop a recommended practice for water-reducible industrial coatings excluding electrodeposition types.

SUBCOMMITTEE D01.92 MEMBERSHIP DEVELOPMENT

R.A. Brown, Chairman

Discussion centered around the problem of getting more of the 582 listed members of Committee D-1 to attend and participate in meetings. Less than half of the members attend any regularly scheduled meeting of the committee.

Suggestions were made to: (1) select members who are needed in specific work of one or more subcommittees and invite them to become active; (2) ask subcommittee chairmen to write letters to those members of his group who are not present, asking them to attend the next meeting; and (3) continue orientation meetings with emphasis on how new members may join in the work of a group or subcommittee.

The need for better test methods was again expressed and more technical people from the paint manufacturing industry are needed in the work of Committee D-1.

SUBCOMMITTEE D01.96 LONG RANGE PLANNING

H.M. Werner, Chairman

A review of the scopes of Sub. D01.07, D01.13, D01.41, and D01.42 was made to consider need for possible recommendations on revisions in light of current activities. It was revealed that Sub. D01.07 has decided to drop its specification task group. It will be recommended that the activity of conversion of government paint specifications to ASTM format be discontinued and that the Sub. D01.07 chairman request the Sub. D01.90 Executive Committee to review the continuing need for such activity by the department that originated the request. It was felt that the Sub. D01.07 scope should remain as is.

A review of the Sub. D01.13 scope indicated the need for a change. It was recommended that the "Institutional" task group be removed from Sub. D01.13 and that its activities be added to Sub. D01.41. Sub. D01.13's revised scope might include the "consumer sounding board" concept. These recommendations will be made to the Sub. D01.90 Executive Committee. Review of the scopes of Subs. D01.41 and D01.42 showed no need for change.

In the discussion of D-1 finances it was stated that a direct Society contribution to D-1 could not be made. It was revealed that Committees A-1, D-2 and others solicit their respective industries. It was suggested that D-1 should do the same, including NPCA

and other such organizations. It was felt that some would say they already support D-1 by funding individual member activities and attendance. It was suggested that a special letter should be sent out to companies of D-1 members, especially raw material suppliers. It was also recommended that consideration be given to increasing the suggested voluntary contribution to \$15. However, before any further solicitations are made, it was recommended that specific needs and a budget be developed.

DIVISION 1 ADMINISTRATIVE

SUBCOMMITTEE D01.05 INTERCOMMITTEE RELATIONS

J.C. Weaver, Chairman

This group attempts to coordinate the interests in paints in other technical committees, particularly the application of paint to the products in their scopes.

Committee A-5 on Metallic-Coated Iron and Steel Products has developed A 755-78 on "Standard Specification for General Requirements for Steel Sheet Zinc Coated (Galvanized) by the Hot-Dip Process and Coil-Coated for Roofing and Siding." This is coordinated by the chairman of Sub. D01.53. Committee A-5 is forming a new subcommittee on liquid coatings such as asphalt or laminated coverings of galvanized or black steel, large pipe for highway culverts.

Committee E-15 on Analysis and Testing of Industrial Chemicals has Board of Directors approval to expand its scope to include specifications in order to react to a Department of Defense blanket request on a long list of chemicals. This list, when available, needs review by Sub. D01.35 et al. for possible jurisdictional overlap.

Committee E-44 on Solar Energy Conversion is adopting Methods E 408 and E 434 on "Durability of Solar Absorptive Receiver Materials." (Cf Sub. D01.27)

Committee S-21 is a new coordinating committee for standard reference materials for particle metrology. (Cf Subs. D01.24, 31, 33, et al.)

About 100 returns from D-1 members to a letter inquiry from the Chairman lists their memberships in other Society Technical Committees. These will be formed into a table in alpha numeric

order for inclusion in the subcommittee reports to improve liaison.

The Baltimore Society of the Federation of Societies for Coatings Technology offered an extensive list of about 40 kinds of apparatus for D-1 response on possible formation of local round robin test groups. On April 9, 1979, a letter from the Vice-Chairman, Administration and Research, urged D-1 subcommittee chairmen to take early advantage of this general offer.

SUBCOMMITTEE D01.06 USA COORDINATING COMMITTEE FOR ISO/TC 35 ON PAINTS AND RELATED MATERIALS

J.C. Weaver, Chairman

Receipt in 1979 thus far of ISO/TC 35 documents included fire testing, salt spray, epoxy polymers, gloss, and their formation of a new Subcommittee 12 on Preparation of Steel Substrates before Coating. These documents are being referred to appropriate specialists and subcommittees.

A list of Committee D-1 specialists for each of the 18 working groups in TC 35/SC 9 on Paint Properties has been revised and will be distributed to the specialists, to ASTM headquarters and to the secretariats of TC 35 and SC 9. TC 35 plans to meet next in Israel in May 1980.

SUBCOMMITTEE D01.07 GOVERNMENT CONTACTS

L.S. Birnbaum, Chairman

It was reported that Federal Standard 141 B, on test methods, dated February 1, 1979, has been issued. It has a long list of ASTM replacements for obsolete federal methods and a short list of about 90 less used federal methods awaiting replacement methods from Committee D-1. Copies are for sale at the GSA Specifications Consumer Information Distribution Branch, Bldg. 197 (Washington Navy Yard), Washington, D.C. 20407. Federal Standard 595, on color standards, is being reprinted and should be available in the next few weeks. Cost of the book only with 1/2 x 1 in. color chips will be set at about \$5. Cost of a complete set of 499 3 x 5 in. color cards was established at \$70, with a cost of 15¢ each for an individual color card.

A revision to the draft of a proposed D-1 specification to replace Military Specification Mil-P-28582 was forwarded to the Executive Committee of

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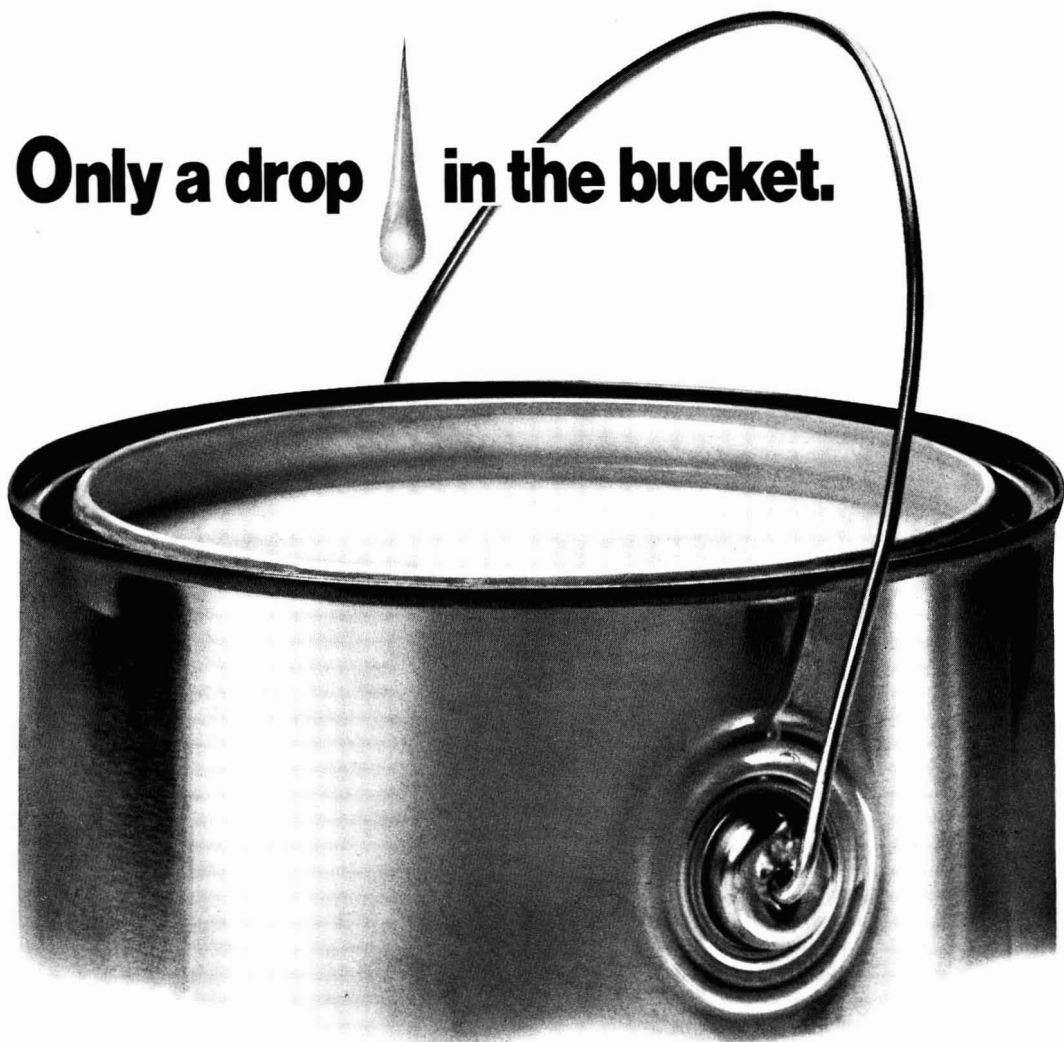
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D-1 for review and action. It was felt that the latest revision had taken care of the mandatory compositional requirements to which many members had objected. It is understood that the document will be assigned to an appropriate working subcommittee for further review and action.

GSA advised that they are developing commercial item descriptions to augment Federal Specifications for a number of paints and would keep this subcommittee informed of progress. This is to serve as an alternative for procurement of these type products.

SUBCOMMITTEE D01.13 CONSUMER AFFAIRS

**W.V. Moseley, Jr.,
Acting Chairman**

Group 1: State and Institutional Consumer Affairs, W.V. Moseley, Jr., Chairman. The working group reviewed the negative ballots received and resolved all of them. Draft #9 of the "Standard Recommended Guide for Purchasing State and Institutional Paint" will now be submitted to a main committee letter ballot. Details of the resolution of the negative ballot are as follows: (1) the objection to Section 3.1 of the guide was voted as nonpersuasive because of changes in Section 3.1 allowing either products offered to the general public or sold in accordance with applicable government specifications; (2) the objection concerning paragraph 4.2.1 was voted as nonpersuasive because there are specification products available to cover the requirements and there are also no reliable tests for bacterial or fungal attack; (3) the objection concerning Section 5 regarding subjectivity of tests by painters was voted nonpersuasive; and (4) the objection concerning section 5.7 was voted nongermane. The voter will be contacted regarding the actions of this group.

Concerning a request from the State of Pennsylvania for assistance from this subcommittee in writing contracts for procuring paint products and getting the most favorable prices, the subcommittee decided this is not a proper task for them or Committee D-1.

Group 2: Individual Consumer Affairs, J. Csernica, Chairman. R.A. Brown, of NPCA, reviewed the progress by this organization in developing a Consumer Awareness Manual for use by paint consumers. This is not yet completed but considerable progress has been made. This group will be kept informed of progress and the manual will be presented to them upon completion.

Several members offered to write suggested rewording of the scope which appears to be out of date. When the subcommittee is satisfied with the revision, it will be submitted to the Executive Committee of D-1 for approval.

SUBCOMMITTEE D01.15 LECTURES AND SYMPOSIA

H.A. Wray, Chairman

It was decided to proceed with the previous planned topic for the lecture program at the January 1980 meeting. Two suggested speakers will be contacted to determine if they will participate in the program on "The Impact of Enforced Technology."

At the request of the Executive Committee of D-1, work will begin on planning for a symposium for a future D-1 meeting or in conjunction with another group. Subject, time, and speakers were left to the discretion of this group.

SUBCOMMITTEE D01.16 DEFINITIONS

S. LeSota, Chairman

Five negative ballots and five affirmative suggestions on the 14 resistance properties and two fire-retardant definitions, which were balloted by Main Committee D-1, were discussed.

All negative ballots were voted nonpersuasive. One member withdrew his negative ballot. Another negative ballot suggested that the word "degradative" in the definition on "chalking resistance" be changed to "degrading." It was agreed that "degrading" is not usually used to describe physical and mechanical phenomena. Another negative ballot suggested that the phrase "when tested by _____ test procedure" be added to these definitions. It was considered nonpersuasive since these are definitions of coating defects and not part of any test procedure. This same reason was used to rule nonpersuasive a negative ballot to add "under defined conditions of test" to these definitions. However, it was agreed that the phrase "effectively protects" in the "intumescent coating" definition should be changed to "serve to protect." It was also agreed to delete the last sentence in the "chalking resistance" definition which is not really a definition, but rather an explanation. Some of the minor editorial changes suggested by others were incorporated into the definitions and these revised definitions will be submitted for a Main Committee ballot.

It was agreed that water-reducible is the preferred terminology for Committee D-1 Standards instead of water-based or water-borne; likewise solvent-reducible instead of solvent-based or solvent-borne; for the consumer, water-thinnable and solvent-thinnable.

SUBCOMMITTEE D01.17 RECORDS AND PUBLICATIONS

M.P. Morse, Chairman

This subcommittee is actively encouraging the technical subcommittees of D-1 to prepare research reports that document the tests performed in the development of test method procedures and the precision of these procedures. Subcommittee chairmen have been reminded that a documenting research report must accompany the submission of a test method to ASTM headquarters for a main committee letter ballot. Each research report is assigned a number and filed at ASTM headquarters.

During the past six months, three reports documenting analytical test procedures have been filed at ASTM headquarters. No proposed publication in journals has been received. Reports are being prepared documenting the interlaboratory test data obtained for measurements of (1) consistency by Stormer Viscometer, (2) viscosity by Brookfield Viscometer, and (3) viscosity by efflux cups.

DIVISION 20 RESEARCH AND GENERAL METHODS

SUBCOMMITTEE D01.20 STATISTICS AND SAMPLING

H.E. Ashton, Acting Chairman

The subcommittee reviewed the status of three documents that the group has been trying to prepare. The recommended practice for "Sampling" had been balloted to main committee at least once as had the recommended practice for "Standard Atmosphere." Changes suggested have been incorporated and they have been ready for Society ballot for two years. The subcommittee recommended that the latest versions be sent to Society ballot.

The recommended practice for "Interlaboratory Tests on Methods" had been submitted to both subcommittee

ballot and editorial review. Appropriate comments will be incorporated into the practice which will be submitted to concurrent subcommittee and main committee ballot.

SUBCOMMITTEE D01.21 CHEMICAL ANALYSIS OF PAINTS AND PAINT PRODUCTS

R.W. Scott, Chairman

Two new task groups have been formed as follows: D01.50 on Analysis of Solvents in Paints and a new research group to explore the feasibility of chemical and instrumental analysis applicable to the paint industry.

Group 13: Coordination of VOC Standard and Information, H. Fujimoto, Chairman. Minutes of the USEPA/ASTM meeting in Research Triangle, N.C. on April 17, 1979, were presented for consideration and approval.

The question of definition of VOC was raised, along with consideration of volatile determinations and methods for determining water content. The results of the letter ballot on the recommended practice for determining volatile organic content were reviewed. Three negatives and several editorial comments were received. The negative ballot regarding the mathematical symbolism was found nonpersuasive because the requester described a three letter symbol for VOC rather than a single symbol. The negative ballot regarding title change was considered editorial but was found to be nonpersuasive by the group. Also the negative ballot regarding the inclusion of a distillation step was considered nonpersuasive. The revised definition was accepted and the note changed to exclude any alternate definition. The practice will be revised to include the comments and corrections noted in the ballots.

The question of alternate approaches to VOC was discussed. It was noted that the EPA method, using a nondispersive infrared spectrometer after combustion of the volatiles, was an attempt to measure volatile organic content directly. It was also pointed out that other approaches would be welcomed for consideration by EPA. The formation of a new task group to work with EPA on alternate approaches to VOC was left to the discretion of the subgroup chairman.

Group 21: Analysis of Electrocoat Baths, A. Yeramyian, Chairman. Current round robin results on: (1) non-volatiles and pigment content and (2) acid value and solubilizer content, were

reviewed. Results indicated that major difficulties were caused by sampling. A sampling procedure will be issued. All test methods except acid value and solubilizer content are applicable to anionic and cationic electrocoat paint samples as well as their ultrafiltrates. This test method will be rewritten for general applicability to both anionic and cationic systems. Two different methods for solvent analysis by direct injection gas chromatography will be submitted for evaluation. A check will be made to determine interest, needs and know how in ionic constituent and contamination analysis and the findings reported at the next meeting.

Group 23: Analysis of Hot Melt Traffic Markings, W.V. Moseley, Jr., Acting Chairman. There was discussion on a method of analysis submitted recently along with results on one sample containing 1.50 mm glass beads. A second sample with 1.65 mm glass beads and silica filler will be sent out by July 1979.

Group 24: Test for VOC of Paints, A.C. Abbott, Chairman. Discussions were held concerning the first and second round robins. The input from round robin #1 gave data with results scattered with respect to variance. One laboratory varied in six cases. A large error was introduced with a sample based on a UV curable printing ink containing monomer. Statistical data omitting the laboratory in question did not result in any great improvement. When both the laboratory and sample in question were dropped, a great improvement was noted in the reproducibility of the method between laboratories. For round robin #2, 12 laboratories were involved and the coefficient of variation was somewhat improved. The data suggest the approach of treating all samples as per: (1) 20 min @ 110°C (D 2369) and (2) 1 hr @ 110°C (new approach). Fifteen samples will be distributed for round robin #3 with approximately 20 laboratories participating.

A summary statement was presented on the Karl Fischer water titration piggyback round robin. Data looks promising as was expected for Karl Fischer, i.e., $\pm 1\%$ at large concentrations. The problem is that an error of ± 1 or 2% is then introduced into the VOC calculation, thus causing substantial error. A further piggyback will be conducted. Discussion of the gas chromatography method for water suggests limiting the scope because the data may be questionable at low levels.

There was discussion about the translation of error into the final VOC calculation. It could result in an overall variation of as much as 15% from accumulated relative error. Some samples

should be saved from the third round robin for possible correlation to a future direct VOC method. It was agreed to hold the three temperature tier approach aside until the next round robin data is evaluated.

Ballots received on Method D2369-73 were discussed. One negative ballot was considered nonpersuasive. A number of comments were considered favorably and appropriate changes will be made. Discussion ensued concerning the title. It was voted to change the title to read: "Volatile Content of Solvent Reducible Paints." Discussion also occurred concerning a mention of a method for use with water reduced systems plus adding a footnote concerning the applicability of the method to water-reducible systems. It was decided to drop these suggestions because the scope in paragraph 1.1 says the method is for solvent-reducible systems.

Group 42: Atomic Absorption Spectrography, (AAS) Analysis of Pigments Extracted from Whole Paint, W.V. Moseley, Jr., Chairman. The group discussed the results of the last limited round robin and proposed another round robin with more samples and all interested laboratories involved. It was decided that the round robin should consist of three samples of whole paint for extraction and analysis of titanium dioxide and three samples of the same products already in solution and ready for the atomic absorption analysis. The method will be rewritten around "the methods of addition" and sent with the samples.

Group 44: Determination of Ferrous Iron in Synthetic Iron Oxides, K.R. Hancock, Chairman. One negative and two editorial comments were received on the main committee letter ballot. The editorial comments were reviewed and will be incorporated into the procedure where appropriate. The negative concerning the safety statement was accepted and the safety statement will be revised. Following editorial revision, the method will be submitted for Society letter ballot.

Group 45: Percent Solids in Titanium Dioxide Slurries, P.B. Mitton, Chairman. Two negatives and two affirmatives with comments were received on the proposed method of test for "Percent Solids in Titanium Dioxide Slurries" on the subcommittee ballot. One negative was resolved by accepting almost all of the suggested changes which were of an editorial nature. The second negative vote was ruled non-persuasive because the technique suggested had been tried before and found to produce a systematic differ-

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ence between the first and second replication. The editorially revised method, including a section on Applicable Documents, will be submitted for a main committee ballot.

Group 46: X-Ray Analysis of Pigments, H. Neubold, Chairman. Some preliminary results of titanium dioxide analysis performed on acrylic latex paints with titanium dioxide as the sole pigment and with titanium dioxide in various combinations of clay and calcium carbonate were discussed. The X-ray fluorescence technique used in these analyses had been developed by one of the members. Some apparent matrix effects were noted. It was explained that the point of this method was to eliminate these undesirable matrix effects. A round robin will be conducted using this method. There was some discussion of vehicle type and TiO₂ type to be used in the samples. It was pointed out that commercial TiO₂ is of variable composition because of surface treatment. A member agreed to supply information about certification of TiO₂ content. The vehicle should be something typically used in trade sales products. The samples should be sent out within the next few months.

Group 52: Trace Levels of Benzene in Paints, R. Domingo, Chairman. Four methods have been received for consideration by the task group. These include a capillary GC method using a UV detector. A second method for benzene in hydrocarbon solvents is a conventional packed column gas chromatographic technique. The detection limit is 500 ppm. A third method uses gas chromatography and conventional packed columns. The method has a lower detection limit of approximately 1000 ppm. The fourth method is based upon the De Soto method using GC with a Siponate® DS-10 liquid phase. The method employs an internal standard and is applicable to whole paints. It has been prepared in ASTM format.

Comments on the drafted method, "Proposed Test Method for Quantitative Determination of Benzene in Paint and Coatings by Gas Chromatography," were received from the floor for consideration and possible inclusion in the revised method. Appropriate changes will be made and the revised draft submitted for preliminary editorial and safety comments. A round robin will be started by September 1st. Details were discussed concerning samples for the round robin. Samples include an automotive paint, stain, appliance enamel, and an oil-base primer. A blank paint will be provided.

Group 53: Trace Levels of Monomers in Paint, G.L. Cunningham, Chairman. Generalized methods for free monomer analysis were discussed as they relate to specialized equipment: capillary GC, HPLC, and NPG detectors with a GC. It was pointed out that it is an ASTM practice to write methods using the most readily available equipment possible. It was agreed that a generalized method would be an appropriate starting point rather than trying to write methods for specific monomers. A proposed method employing a flame ionization detector with a 20 ft × 1/8 in. s.s. column packed with Carbowax® 20M will be evaluated with a round robin to begin by September 1979. The question of sample preparation was discussed. The problem of residual monomers already in the sample will be circumvented by spiking latices with compatible monomers but monomers not used in the latex.

Group 54: Water Content in Paints by Karl Fischer Titration, W.C. Golton, Chairman. It was reported that the recently developed gas chromatographic method for water content had passed Society ballot and been assigned the number D 3792. Upon reports that some members had experienced problems at low water levels, it was noted that the method had not been evaluated at low water content. It was decided to write in the scope that the method was limited to higher water concentrations.

The proposed catalyzed Karl Fischer method was reviewed. Preliminary data from the first round robin was presented. It was agreed that the data from four collaborators was encouraging and it was decided to conduct a full round robin in conjunction with an upcoming one on volatile content planned by task group D01.21.24. A revision of the water procedure which avoids the two burette approach will be written.

Group 61: Revision of D 564, "Testing of Liquid Driers", L. Bazarko, Chairman. The new method of test for iron in paint driers by EDTA method and the editorially revised methods for cobalt, lead, manganese, and calcium, or zinc (D 2373, D 2374, D 2375, and D 2613) have passed Society ballot and will be published in Part 29, 1980 edition of the *Annual Book of ASTM Standards*. The new method of test for cerium in paint driers by the ceric sulfate method has been balloted by the main committee. All pertinent comments will be incorporated and the method submitted for Society ballot.

Results from the round robin testing for vanadium and total rare earths in paint driers by EDTA methods have

been received and statistically analyzed by E 180. The methods will be submitted for subcommittee balloting. The method of test for zirconium in paint driers by EDTA method will be withheld from main committee voting pending atomic absorption verification of the hafnium-zirconium ratio in typical U.S. driers. Hafnium is allegedly present in many commercial zirconium compounds.

Specification D 600 on "Liquid Paint Driers" has been revised and forwarded to task group D01.35.03 on chemical intermediates for comments and subsequent concurrent subcommittee and main committee balloting. Method D 564, "Testing Liquid Paint Driers," has been revised. Pending further comments from subcommittee members, it will be submitted for subcommittee ballot. The group made a recommendation that Methods D 1953 and/or D 1640 on drying tests be revised to include mechanized (for example BK (Beck-Koller) drying recorder) methods for measuring drying time of drying oils and paints.

Group 62: AAS Analysis of Liquid Driers, H.D. Swafford, Chairman. The first draft of a method of test for the metal content of liquid metal driers by atomic absorption spectroscopy was used in a round robin test by one laboratory. This method uses as standard driers whose metal concentration has been ascertained by EDTA methods. Since, (1) an independent zirconium method is needed to determine hafnium presence in this drier, (2) some driers may not be soluble in MIBK, and (3) some instrumental laboratories are not equipped to run wet analytical methods, an alternate method using ashing and analysis in aqueous media was proposed using the appropriate metals dissolved in HF acid as standards. Recommendations on the draft will be reviewed and draft #2 written. Testing of samples per draft #1 will be suspended. Samples for draft #2 will be supplied to all new collaborators. Those who received samples for testing under draft #1 will continue to use the same ones for draft #2.

Group 71: Metals in Low Concentration, R.W. Scott, Acting Chairman. Preliminary results concerning an AOAC round robin to determine low levels of lead, cobalt, and cadmium indicated general agreement between D 3335 and the AOAC method. The go-no go method at the 0.5 and 0.06% lead level was discussed. Benzidine (once alleged toxic) which is used in the 0.06% lead level method can be used again in laboratories. Therefore, a review of the low lead method will be conducted and

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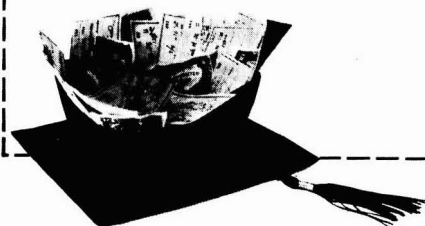
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if the data supports the method it will be submitted for letter ballot at the subcommittee level. At this time, there are no future plans for the analysis of heavy metals or trace elements. Alternate methods which should be considered are a flameless AA technique for lead which has advantages and XRF (X-ray fluorescence) where a PGT (Princeton Gamma Tech-100[®]) instrument may be cheaper than AA. It was decided the task group would remain active at least through January 1980.

Group 72: Metals in Air Particulate Samples, J.A. Devlin, Chairman. The organization of a second round robin on simulated dust of lead chromate type pigment in work place atmosphere was verified. The group highly favored sample preparation by deposition of a lead chromate dispersion rather than a dissolved lead solution on the membrane since the former was more apropos of its objective. Two round robins will be run. One, with 10 collaborators, will be a repeat of a former one at lower load levels of a regular primrose chrome yellow simulated dust; the second a simulated silica coated chrome yellow dust with eight collaborators. The third draft of a method for the determination of lead and chromium in the workplace pigments was distributed in preparation for the second round robin above. The procedure for the silica coated dust will be mailed within a month to the collaborators.

Backlog priorities are primarily: (1) electrothermal atomization of simulated dust for lead and chromium at low levels in chrome yellow type dusts; and (2) flame AA for lead and chromium in paints containing chrome yellow type pigments. Simplified x-ray fluorescence determination of lead and chromium on membranes of air samples of chrome yellow was discussed. Adaption of the PGT-100 Chemical Analyzer to membrane filters was noted.

SUBCOMMITTEE D01.22 HEALTH AND SAFETY

H.A. Wray, Chairman

Following a discussion on revision of withdrawal of Method D 1310, "Flash Point of Liquids by the Tag Open Cup," it was decided to make necessary revisions of the method and submit it to subcommittee ballot. The Open Cup method has become less important since the Consumer Products Safety Commission is working on a proposal to go to Closed Cup methods in their regulations. This is a result of an amendment to the Federal Hazardous Substance Act deleting the statutory requirement

for use of the Open Cup in the commissions regulations.

Since Method D 3278, "Test for Flash Point of Liquids by the Setaflash Tester," was revised to add an annex for testing high viscosity materials without a reproducibility statement, it was agreed to conduct a round robin. This will include materials with approximate D 1545 Gardner viscosities of Z-3, Z-6, Z-8, and, if possible, Z-10. Five members volunteered to conduct the test.

A representative of the Office of Hazardous Materials Regulations, DOT, gave a resume of the program underway in that agency to define a solid material. It was stated that a viscosity of 1000 stokes (Z-10) seems to be a good cut off point to differentiate between a solid and a liquid. DOT would be interested in testing the material used in the proposed round robin with a viscosity of 1000 stokes. Several members offered to look for materials in this 1000 stokes range.

Copies of revised equilibrium methods were distributed and discussed. The first method is a flash-no-flash method and the second a definitive flash point method. These methods provide for equilibrium temperature throughout the sample of viscous materials in the Tag or Pensky-Martens testers. These methods are similar to ISO Methods. EPA proposed regulations for toxic substances and hazardous wastes have provisions for this type of method.

A report was submitted on the activities of the Coordinating Committee on Flash Point and Related Properties and Interaction with Government Agencies on These Properties. These activities include a round robin to test the combustibility test for mixtures, the formation of a Waste Disposal Task Group, and the evaluation by the Calibrating Task Group of synthetic materials in place of pure hydrocarbons as calibrating liquids.

Two fire retardancy tests, D 1360, "Cabinet Method," and a new method, "Two Foot Tunnel Test," were in the June Society ballot. Precision statements are necessary and round robins will be required. However, there is a need to determine what woods should be used, who has the instruments, and who can participate in a round robin. NPCA's Committee on Fire Retardancy will be asked for their advice. The subcommittee will solicit help in conducting the round robins from the Fire Retardancy Group of NPCA, the Fire Technology Group of NBS, subcommittee members and others, such as the Baltimore Coatings Society.

The NPCA's *In Plant Hazardous Materials Identification System — Raw Materials Rating Manual* was presented to the subcommittee for their consideration. This system uses a 0-4

rating for Health, Flammability, and Reactivity properties of materials and an alphabetic classification of protective equipment required to handle safely hazardous materials. It was recommended that the NPCA document be reviewed by Sub. D01.22 and that in cooperation with NPCA, the subcommittee prepare an ASTM draft of the manual, probably one section at a time.

A copy of proposed hazard statements for inclusion in methods was circulated. After some discussion, it was proposed that these statements be prepared as a recommended practice and issued for subcommittee ballot. Method D 3630, "Standard Practice for Determining Constituents, Classified as Hazardous, Contained in Protective Coatings," and "Bibliography of Information on Toxic Materials" are in need of review. The subcommittee desires support from additional personnel who have an interest in these items.

As the result of a request by a representative of the Office of Hazardous Materials Regulations, DOT, to update the standards referenced in U.S. Government Regulations, the subcommittee agreed to recommend that the Executive Committee of D-1, through the Society, petition DOT to update the standards for flash point referenced in government regulations.

SUBCOMMITTEE D01.23 PHYSICAL PROPERTIES OF APPLIED PAINT FILMS

M.P. Morse, Chairman

Group 10: Adhesion, H.E. Ashton, Chairman. The group is investigating the measurement of adhesion of coatings by a pull-off technique using tensile testers. Details for a round robin test by this procedure are being developed. Information has been developed regarding satisfactory adhesives to use for coatings. Information is being developed also regarding devices for insuring proper application of load to the specimens. One laboratory has used parts of the Elcometer portable adhesion tester for this purpose. A representative of Gardner Laboratory will provide information on cost and availability of Elcometer parts and test cylinders for the proposed test procedure. For a round robin test of the pull-off method, coated panels differing in adhesion will be prepared by using different metal surface treatments and by using coatings known to differ in adhesion. A questionnaire will be prepared asking D-1 members of their interest in method of A of D 2197 which measures adhesion by the balanced beam scrape adhesion tester. The

extent of interest received will determine whether interlaboratory tests will be made to establish a precision statement.

Group 11: Wet Film Thickness Measurement, H.A. Ball, Chairman. Improved procedures for measuring wet film thickness with the Interchemical Gauge and the Pfund Gauge were developed. The method, D 1212, has been approved by Committee D-1 and has been submitted to Society ballot. A report documenting the interlaboratory data obtained with the improved procedures has been filed at ASTM headquarters. The group is now undertaking the development of a method for measuring wet film thickness in field applications using notched gauges. Some interlaboratory test results have been obtained with the Nordson gauge and a preliminary draft of a test procedure will be prepared for circulation to the group members.

Group 12: Measurement of Dry Film Thickness, K.A. Trimmer, Chairman. Nearly completed is an extensive round robin test to determine the accuracy and precision of various types of commercially available instruments for measuring dry film thickness of coatings. Drafts of revisions of Methods D 1400 and D 1186 have been prepared including those instruments found to have given satisfactory precision of film thickness measurements of coatings on magnetic and non-magnetic substrates. The accuracy of the instrumental measurements of the thickness of the specimens used in the round robin test are being determined by means of microscopic measurements and photographs of film cross sections and by slit microscope measurements.

Group 14: Hardness, Mar and Abrasion Resistance, M.P. Morse, Chairman. A round robin test is being conducted to determine the precision of Method D 968, "Abrasion of Coatings by Falling Sand," and of D 658, "Abrasion of Coatings by Air Blast Abrasive." D 968 is being revised to include the use of falling silicon carbide. The round robin test also includes measurements with the Taber Abraser. Results received to date show wide variations in the results obtained by different laboratories for each of the falling abrasive procedures. The reasons for this are being sought. Wide variations were obtained with the air blast abrasive test also, but differences in flow rates and air pressures used account for this. This method is being revised to improve the calibration procedures. The Taber Abraser results re-

ceived to date have shown good precision when abrasion is expressed as cycles per mil of coating removed. A draft of a method using this procedure will be prepared.

Group 15: Slip Resistance, G.D. Ernst, Chairman. Various methods are being investigated for measuring the slip resistance of coatings. It has been decided to first evaluate a method based on the use of an inclined plane to determine the coefficient of friction of a coated panel under a sliding leather "shoe." Drawings of the simple apparatus needed for this test will be distributed to members who will participate in the evaluation of the proposed procedure.

General: The recommended revisions resulting from Committee D-1 letter ballot have been made to Methods D 522, D 1005, D 1212, D 1474, D 1653, D 1737, and D 2197. The revised drafts have been submitted to Society letter ballot. Technical revisions are being made to two methods which received negative votes in Committee D-1 ballots. These methods are D 823, "Preparation of Uniform Films of Coatings," and D 2370, "Elongation and Tensile Strength of Free Films." Method A 1653, "Moisture Vapor Permeability of Organic Coating Films," is being reexamined to determine its need in view of the existence of Method E 96 which is similar and more extensive in application.

SUBCOMMITTEE D01.24 PHYSICAL PROPERTIES OF LIQUID PAINTS AND PAINT MATERIALS

C.K. Schoff, Chairman

Group 18: Dispersion Phenomena, V.R. Waltz, Chairman. Discussed were several methods including D 1210, "Fineness of Dispersion of Pigment-Vehicle Systems," the precision statement of which had received two committee ballot negatives. These had been withdrawn after editorial changes, including identification of units. It was voted to send the method for Society ballot with the editorial changes. Method D 281, "Oil Absorption of Pigments by Spatula Rub-Out," received a negative ballot on Main Committee vote because it was not in modern ASTM format. The negative was judged non-persuasive on the grounds that no criticism of the test method itself had been made. However, it was agreed that the method is not in the correct format and that it should be revised before the next approval date. Method D 1483, "Oil

Absorption of Pigments by Gardner-Coleman Method," also up for reapproval, received several comments including one on a misprint that has been in the method for many years. Most of the editorial changes suggested will be made. It was voted to send both D 281 and D 1483 for Society ballot with the editorial changes.

Group 19: Viscosity by Efflux Cups, C.K. Schoff, Chairman. The precision statement for Method D 1200, "Viscosity by Ford Cup," was discussed. It was pointed out that the round robin used mainly non-Newtonian (shear thinning) paints, but the method is designed for Newtonian and near-Newtonian fluids. It was decided to repeat the round robin with the latter materials. Precision may not be any better, but we have to determine what it is with the correct type paints.

The draft of the proposed dip cup method was discussed in detail. The method was considered acceptable on most counts, but objections were raised over the lack of cup specifications. This brought out the fact that Zahn cups are not made to a given specification. Each manufacturer uses different dimen-

sions. The next draft of the method will contain mention of this and the difficulties it presents when efflux time comparisons are attempted. The question of whether an ASTM cup specification should be established and what the dimensions should be was not resolved. Another matter that came up was the problem of temperature control during calibration with standard mineral oils and the considerable temperature dependence of the viscosity of these oils. The same problems are found with all viscosity methods. It appears that castor oil and linseed oil may be better for calibration of viscometers since their viscosities are less temperature dependent.

Group 20: Viscosity of Non-Newtonian Materials by Rotational Viscometer, P.C. Underhill, Chairman. Discussed was the revision of Method D 2196, "Viscosity Measurements and Thixotropic Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer." A number of comments were made and a new draft will be written.

Group 21: Stormer Viscometer, M.P. Morse, Chairman. Discussed was the

revision of Method D 562, "Consistency by Stormer Viscometer." The difficulty of calibration and inherent problems with standard mineral oils was raised here as well. A previous round robin involving both oils and paints was discussed. It was concluded that a viscometer would be considered "in calibration" as long as it gave a value within plus or minus 15% of the expected load for 200 rpm for the given oil and within plus or minus 5% of the consistency in Krebs units. A new draft of the method will be written and submitted for subcommittee letter ballot.

General: Method D 1475, "Density of Paint, Varnish, Lacquer, and Related Products," was discussed. Reapproval of this method had received a negative ballot on the grounds of the lack of hazard statement concerning the chromic acid recommended for cleaning the apparatus. It was considered persuasive and a suitable hazard statement will be written. There were a number of comments which were deemed editorial as well. The subcommittee agreed to make the editorial changes and then send D 1475 on to Society ballot. The existence of other density methods and instruments, including the Mettler/Paar de-

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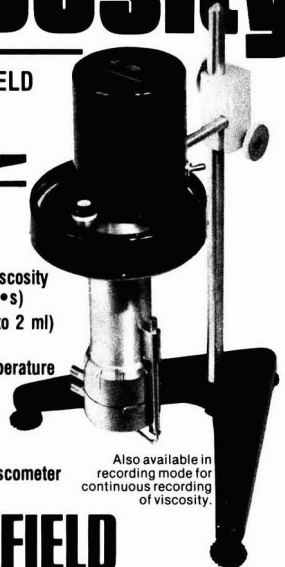


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vice was brought up. A survey of density methods will be made before the next meeting and an additional working group will be set up for the consideration of new ASTM methods in this area.

SUBCOMMITTEE D01.25 PICTORIAL STANDARDS OF COATINGS DEFECTS

S. LeSota, Chairman

A revision of the "Pictorial Standards of Coating Defects," a joint effort with the Federation of Societies for Coatings Technology, has been published and is now available from the Federation at 1315 Walnut Street, Suite 830, Philadelphia, Pa. 19107 at a cost of \$75 a copy plus 5% to cover handling and postage.

Other appropriate subcommittees are encouraged to obtain pictorial standards of failures more typical of modern latex and oleoresinous coatings where the mode of failure differs from that of existing pictorial standards.

SUBCOMMITTEE D01.26 OPTICAL PROPERTIES

P.B. Mitton, Chairman

Group 2: Color Measurement. J.G. Davidson, Chairman. The revised draft of a standard practice for the visual evaluation of metamerism will be submitted to subcommittee vote. Three methods which need reapproval were reviewed. The previously assigned task groups will make various changes and then the revised methods will be voted upon as follows: D 3134, "Color and Gloss Tolerances of Opaque Materials," by subcommittee only; D 1535, "Munsell Specification," by both subcommittee and main committee; and D 1729, "Visual Color Difference of Opaque Materials," by subcommittee only. A task force was established to study "Visually Perceived Attributes of the Appearance of Materials."

Group 6: Hiding Power. P.B. Mitton, Chairman. A round robin is in process to test a proposed new hiding power method. It was voted to submit Method D 2805, "Hiding Power of Paints," to simultaneous subcommittee and main committee ballot for reapproval. A new source of supply for black glass used in the method will be added as a footnote.

Group 11: Gloss and Goniophotometry. R.C. Kissler, Chairman. The proposed editorial and precision changes in Method D 523, "Test for Specular Gloss," will be submitted to simultane-

ous subcommittee and main committee ballot. A new task force was formed to consider a recommended practice for visual intercomparison of gloss surfaces.

Group 19: Hemispherical Reflectance. K. Luyk, Chairman. A round robin to establish the precision of a proposed method is nearly finished.

Group 22: Sample Preparation for Determination of Optical Properties. H. Hammond III, Chairman. After discussion and modification of a fourth draft, it was voted to submit it to a subcommittee ballot.

Group 23: Retroreflectance of Horizontal Coatings. N. Johnson, Chairman. Final details of a round robin to test "Specific Luminance of Horizontal Coatings" were discussed. The round robin should start this summer.

Group 24: Color and Strength of Color Pigments. R. Zellers, Chairman. Specific vehicles were decided upon to test a new draft of the method for "Mass Color and Tinting Strength of Color Pigments." The round robin will start soon. It was voted to submit two methods for reapproval to simultaneous subcommittee and main committee ballot. These methods are D 332, "Tinting Strength of White Pigments," which is a visual method, and D 2745, "Instrumental Tinting Strength of White Pigments."

SUBCOMMITTEE D01.27 ACCELERATED TESTS FOR PROTECTIVE COATINGS

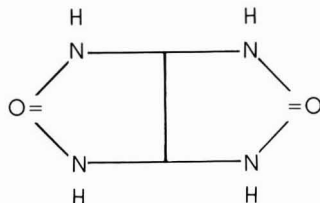
E.A. Praschan, Chairman

Group 2: Water Tests. G.W. Grossman, Chairman. A new format for the four water test methods was discussed and approval given to proceed with formal write-ups.

Group 4: Light and Water Exposure Apparatus. S.M. Totty, Chairman. A negative vote on withdrawal of Standard Practice D 3361 (unfiltered carbon arc: dew cycle) was ruled persuasive so this practice will be revised and balloted later. An alternate test cycle of four hours light followed by four hours dark with spray has been selected for D 822 test. The round robin will consist of automotive panels from ASTM series 22895.

Group 6: Detergent Test for Organic Finishes. H.M. Leister, Chairman. A survey to establish a new detergent for-

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mula for Method D 2248 is being initiated since the existing formula does not represent the current products on the market. Round robin testing will follow.

Group 9: Evaluation of Corroded Specimens, R. Williamson, Chairman. Four negatives and four comments were received from balloting on Method D 1654. Three of the negatives were persuasive and fourth was ruled nonpersuasive because the tape method it advocated was determined by earlier work to be less reproducible than the current air blow-off and spatula methods.

Group 10: Accelerated Outdoor Weathering, M.P. Morse, Chairman. Visual observations of exposed panels supported the instrumental gloss and color differences previously reported on panels exposed at four different locations in the Miami area as well as Arizona and Puerto Rico. Efforts are being made to explain the differences encountered between exposure sites in the Miami area. Since rank order ratings of the different coatings were consistent, it was agreed to draft two recommended practices, one utilizing the Black Box and the other the heated Black Box, to produce accelerated outdoor exposure affects.

Group 16: Chalking, J.S. Robbins, Chairman. A recent subcommittee ballot brought three negative votes: two were persuasive and one was nonpersuasive because it was judged to be unrelated to D 659, but will be considered in the new method being developed. Other comments were either agreed upon or satisfactorily resolved. Method D 659 will be resubmitted for a simultaneous subcommittee and main committee ballot.

Group 17: Evaluation of Weathering Effects, M. Mollman, Chairman. This new group was formed to review and act upon the seven methods in Sub. D01.27 which relate to the evaluation of weathering effects. Appointments were made for review of those methods requiring reapproval action in 1979 and 1980.

General: Two negatives from a concurrent ballot on a proposed method for "Preparation of Glass Panels for Testing Paint, Varnish, Lacquer and Related Products," were deemed persuasive and added to the method. Comments on other areas, however, dictated further clarification was required and these will be resolved prior to the January 1980 meeting.

SUBCOMMITTEE D01.28 BIODETERIORATION

R.T. Ross, Chairman

Group 1: Package Stability, W. Woods, Chairman. As part of the development of a test to replace Method D 2574, four collaborating laboratories evaluated the susceptibility of unpreserved emulsion paints to bacterial spoilage. The results were mixed, one collaborator obtaining good sustained growth in the paints, two, limited growth, and one, minimal to no growth. It was agreed to review in detail the methodology used by the successful participant. Based on the results of this review, recommendations will be made regarding modifications of the present inoculation protocol. Several emulsion samples will be screened to determine the ease with which bacteria can be grown in them. An additional round robin will be run when a quantity of satisfactory unpreserved resin has been obtained for sample preparation.

Group 3: Accelerated Tests, K. Haagenson, Chairman. Round robins continue in an effort to determine whether Method D 3273 can be modified to make the method acceptable for determining the fungus resistance of exterior paints. Results of evaluation of seven fungicides by five laboratories using UV exposure, leaching, and a combination of UV exposure and leaching followed by four weeks in the tropical chamber were discussed. Correlation between laboratories was poor. Only one sample was rated similarly by all participants. This sample and one which showed the poorest correlation will be repeated using the fungicides at the same level tested previously and at 50% and 150% of that level. A control with no fungicide will also be run. A new protocol will be written by the chairman with additional controls to improve correlation.

SUBCOMMITTEE D01.29 SUBSTRATE PREPARATION FOR COATINGS TESTING

**E.A. Praschan,
Chairman Pro Tem**

The chairman outlined the objectives of the study group which had been appointed by the Executive Committee. There was considerable discussion about the need for a separate D-1 subcommittee to deal with methods relating to paint substrates and whether or not the scope should include substrate

preparation procedures for painting as well as for paint testing. The study group finally agreed that a separate subcommittee is needed but that the scope should be limited to test panel preparation at the present time to avoid conflicts with other subcommittees. The proposed title selected was: Substrate Preparation for Coatings Testing. The proposed scope agreed upon was as follows: This subcommittee is responsible for methods of preparation of uniform metallic and non-metallic substrates used as a base for testing coatings. This subcommittee will coordinate with other subcommittees in D-1 and other committees in ASTM on matters concerning surface preparation.

Six existing methods were identified as falling under the jurisdiction of the new subcommittee. These methods, dealing with test panel preparation, are as follows: D 358, on Wood; D 609, on Steel; D 1733, on Aluminum; D 1734, on Concrete and Masonry; D 1911, on Asbestos-Cement Shingles; and D 2201, on Galvanized Steel.

The above action was proposed to the D-1 Executive Committee and approved with the designation of D01.29 as the number of the new subcommittee. S. Ozenich has agreed to chair the new subcommittee and his appointment was announced by S.B. Levinson, Chairman of Committee D-1. An organizational meeting will be conducted at the January 1980 meeting of Committee D-1.

DIVISION 30 PAINT MATERIALS

SUBCOMMITTEE D01.31 PIGMENT SPECIFICATIONS

C.W. Fuller, Chairman

The subcommittee acted on fifteen specifications that are up for reapproval in 1979. Each specification was reviewed prior to the meeting by the various task force chairmen. Their comments and those raised during the meeting were considered in arriving at proposed courses of action. Three specifications: D 80, on Leaded Zinc Oxide; D 82, on Basic Sulfate White Lead; and D 405, on Blue Lead; Basic Sulfate were recommended for concurrent subcommittee and main committee ballot for withdrawal. To the knowledge of the subcommittee, these pigments are no longer manufactured.

Specifications D 79, on Zinc Oxide; D 81, on Basic Carbonate White Lead; D

209, on Lampblack; D 210, on Boneblack; D 212, on Pure Chrome Green; D 763, on Raw and Burnt Umber; D 765, on Raw and Burnt Sienna; D 768, on Yellow Iron Oxide-Hydrated; D 769, on Black Iron Oxide; D 1199, on Calcium Carbonate Pigments; and D 1648, on Basic Lead Silico Chromate are up to 35 years old and are in essentially the original format. Some of the tests and specification values are obsolete. Applicable ASTM documents need to be referenced. All of the above specifications will be revised editorially and table adjustments will be made in D 79, D 768, and D 1199 before they are submitted for balloting.

Specification D 3360, "Particle Size Distribution of White Extender Pigments," was approved with the addition of "By Hydrometer" to the title. A negative was received on Specification D 962, "Aluminum Pigments, Powder and Paste," regarding shipping containers. The negative was reviewed and considered nonpersuasive in that it should be part of D 480 which covers the sampling and testing of aluminum powder and paste rather than D 962. An editorial change will be made in Specification D 963, "Copper Phthalocyanine Pigments," to insert Aroplaz[®] resin 1444-X-50 in place of Glyptal[®] resin ZA-262, which is no longer available.

SUBCOMMITTEE D01.32 DRYING OILS

P.C. Stievater, Chairman

Twenty-five methods from the April 1979 subcommittee ballot were discussed, including one revision and 24 reapprovals. The revision of Specification D 960, "Raw Castor Oil," received only one minor editorial comment and will be submitted for Society ballot. Two negatives were received on Specification D 260, "Boiled Linseed Oil." One negative was found to be persuasive. This negative requested an increase in the loss on heating from 0.3% to 0.4% and 0.6% for the two types of boiled linseed oil, the reason being that the driers now being used are only available in mineral spirits rather than as solids, and this increases the loss on heating. The other negative was withdrawn prior to the meeting with the understanding that the necessary safety statement on the flammability of isooctane be included in the revision. The revised specification will be submitted for subcommittee ballot.

Negatives on 10 other methods were all withdrawn prior to the meeting with the understanding that these methods would have the necessary hazard/safety

statements included before they are submitted for Society ballot. A negative on Specification D 1537, "Distilled Soybean Fatty Acids," requested that this specification be cancelled since there is only one grade of soybean fatty acids listed in the NPCA Index and it does not meet the requirements shown for any of the types. The negative was found to be nonpersuasive on the basis that the NPCA Index is not a complete listing of all products commercially available, is not a consensus standard and may not even be correct.

Twelve methods received only comments which will be included before the methods go to Society ballot. One comment on Method D 1963, "Test for Specific Gravity of Drying Oils, Varnishes, Resins, etc.," requested that since the use of a pycrometer is very time consuming, a note be added to allow the Paar Digital Density Meter to be used. It was decided that this would best be handled by considering a new method under Sub. D01.24 because of the probable need for round robin testing. The cost of the equipment is about \$5,000. Prior to the replacement of D 1953 by D 1640 as requested in a previous negative vote, it was decided that D 1640 will be reviewed for suitability of use by Sub. D01.32 and revised if necessary.

SUBCOMMITTEE D01.33 VARNISH AND RESINS, INCLUDING SHELLAC

W.C. Golton, Chairman

Group 12: Urethanes, R.A. Foss, Chairman. It was decided to suspend activity on development of a liquid chromatography method for free isocyanates until a written method which is reproducible is available. The chairman will draft a gas chromatography method for isophorone diisocyanate and set up a round robin to test the method. Several actions taken at the January meeting will be executed in time for the next ballot.

Group 14: Alkyds, R. Malek, Chairman. Several actions agreed upon at the last meeting will be taken care of in time for the next ballot. A round robin will be organized on a new gas chromatography method for orthophthalic acid and fatty acids in alkyd resins.

Group 26: Emulsions, O. Brown, Chairman. Upon completion of statistical work and editorial revision, the new method for "Nonvolatile Content of

Latices" will be submitted for subcommittee letter ballot. Methods will be drafted and a round robin will be organized to establish standard methods on freeze-thaw, heat-age, and mechanical stability of latices. A recommended practice for testing latices will be drafted for consideration at the next meeting. Test methods for minimum film temperature and seed content are being sought.

It was voted to consider nonpersuasive a negative vote cast on the revision of D 1544, "Color of Transparent Liquids," during the last D-1 letter ballot. It was felt that the revision permitted use of a less precise method of view. The subcommittee held that this concern had been dealt with at the time of the letter ballot by the subcommittee by inserting a note that the precision statement applied only to the instrument the voter preferred.

General: The status of nine balloted items with negatives was reviewed. All negatives have been resolved by agreeing to make the changes requested. The status of some 20 items due for action in 1979 was reviewed. Volunteers were obtained to review most of them. The chairman will request a one-year extension pending their resolution. It was also voted to request reassignment of several physical property tests to other subcommittees. The chairman will also ask the Executive Committee to transfer jurisdiction of specifications of certain non-pigmented coatings to other subcommittees.

SUBCOMMITTEE D01.35 SOLVENTS, PLASTICIZERS AND CHEMICAL INTERMEDIATES

L.R. Thurman, Chairman

A negative vote was received on the April 1979 Society ballot. This was discussed by the subcommittee and the voter will be asked to withdraw his negative on editorial corrections. It was intended to have changed the term heterogeneity to precipitation. The word heterogeneity was incorrectly left in the proposed standard.

On the main committee letter ballot, the proposed "Standard Gas Chromatography Method of Test for Determining the Purity of Methyl Butyl Ketone, Methyl Amyl Ketone and Methyl Isoamyl Ketone" received two negative votes. One negative was withdrawn on editorial revision at this meeting. A second negative was considered editorial in nature and the voter will be contacted to see if he will also withdraw his negative.

D 2086, D 2190, D 2193, D 3362, and a proposed specification for glacial methacrylic acid received only editorial comments and will be included in the September 1979 Society ballot.

The following specifications, D 302, D 303, D 304, D 319, D 363, D 608, D 770, and D 1007 were approved on a subcommittee ballot and will be submitted for a main committee ballot. Specification D 343, "2-Ethoxyethyl Acetate (95% Grade)," and D 966, "Secondary Butyl Acetate (85%-88% Grade)," were approved for withdrawal on the basis that the products are no

longer available. They will be forwarded for a main committee ballot.

The following work is in progress:

Group 1: Hydrocarbon Solvents. S.A. Yuhas, Jr., Chairman. Method of analysis for benzene at the parts per million level in hydrocarbon solvents.

Group 2: Oxygenated Solvents. T.H. Golson, Chairman. Discussion of revision of Method D 268, "Sampling and Testing Volatile Solvents for Use in Paint, Varnish, Lacquer and Related

Products," is continuing. Discussion of a proposed specification for ketones. Determination of water in urethane solvents in parts per million levels. Assignment of 15 standards for review.

Group 3: Chemical Intermediated. J.M. Kauffman, Chairman. Review of specification and test methods for glacial acrylic acid.

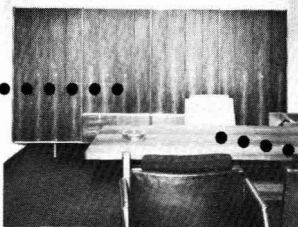
General: At the request of Sub. D01.21 in conjunction with their revision of D 564, Sub. D01.35 will review D 600, "Liquid Paint Driers." The proposed changes will be balloted concurrently with a main committee ballot. Committee D-2, on Petroleum Products, has requested transfer of jurisdiction for D 484, on Hydrocarbon Dry Cleaning Solvents, and D 1836, on Commercial Hexanes, to Committee D-1. Sub. D01.35 sees no problems in accepting these standards.

Sub. D01.21.54 is preparing a new standard for water in whole paints. Members of Sub. D01.35 will participate in the round robin tests and evaluate their proposed method in view of the need for low levels of water in urethane solvents by Method D 1364, "Water in Volatile Solvents." Efforts will continue with other subcommittees through ASTM staff to get government personnel to specify the proper ASTM standards instead of government standards.

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DIVISION 40 PAINT PRODUCTS APPLIED ON SITE

SUBCOMMITTEE D01.41 CRITERIA FOR PROCUREMENT OF COMMERCIAL PAINT AND COATINGS

R.A. Brown, Chairman

Group 1: J. Csernica, Chairman. This group is examining Federal Specification TT-P-29 for latex flat wall paint and ASTM Recommended Practice D 2931 for Latex Flat Wall Paint. In a meeting of this group following the Sub. D01.41 meeting, some excellent progress was made. The group agreed that a minimum number of properties can probably be used to describe the quality of a latex flat wall paint. These could be hiding, porosity, cleanability-scrubbability,

and uniformity of appearance. Quality control by volume solids as supplied and weight per gallon in the field are tests to be included. Government personnel felt that both a first quality and a maintenance grade quality would be necessary. It was felt that standard colors are needed by government users and that 12 colors for flat wall paint could suffice. GSA will supply 12 colors subject to change based on current market preference. Suitable test methods to judge quality of the above properties will be selected. The most difficult part may be to set levels of quality.

Group 2: W.V. Moseley, Chairman. It was reported that Draft 9 of the "Standard Recommended Guide for Purchasing State and Institutional Paint" was being revised editorially and would be submitted to D-1 letter ballot this year. A discussion of how this document might be used to buy commercial paints by Federal Government Agencies followed. Federal Government employees felt the system, as it is now written, would be more applicable to State purchasing than Federal. It was also felt mandatory that quality criteria be used in purchasing paint products by the government. This is based on the belief that it will be necessary to do some quality control testing to ensure an adequate level of quality. It was also indicated that it is likely that the work of Sub. D01.13.01 would be transferred to Sub. D01.41.

SUBCOMMITTEE D01.42 ARCHITECTURAL PAINTS

R.S. Armstrong, Chairman

On subcommittee letter ballot, Methods D 1736, "Efflorescence of Interior Wall Paints," D 2486, "Scrub Resistance of Interior Latex Flat Wall Paints," and D 2932, "Exterior Solvent-Based House and Trim Coatings," had one or more negative ballots casted against them on voting for reapproval. These negatives have been resolved and withdrawn. Also receiving a negative ballot was a new proposed standard on "Uniformity of Gloss and Sheen." This negative has also been withdrawn.

On main committee letter ballot, Methods D 2931, "Latex Flat Wall Paints," D 2932, "Exterior Solvent Based House and Trim Coatings," and D 3383, "Solvent-Thinned Floor Paints," up for reapproval received one or more negative ballots which were subsequently withdrawn. The new proposed method on Gloss and Sheen Uniformity received a negative vote which was ruled persuasive.

Task group reports are as follows:

Group 1: Scrub Resistance, M.S. Abriss, Chairman. Method D 2486, "Scrub Resistance of Interior Latex Flat Wall Paints," was revised as a result of both subcommittee and main committee letter ballots and is now ready for Society ballot. A new proposed standard for scrub resistance has performed well in round robin testing and is being rewritten for comment by the Editorial and Safety Subcommittees prior to its being balloted by the subcommittee.

Group 3: Leveling, C.W. Vanderslice, Chairman. Draft #6 of a proposed new standard for "Leveling of Paints by Drawdown" was reviewed by the group and will be submitted for subcommittee ballot before the next meeting. The group will next consider simplifying another recently drafted method for evaluating "Leveling of Paints by Brushout."

Group 11: Gloss and Sheen Uniformity, R.S. Armstrong, Chairman. A negative ballot and comments received as a result of recent main committee voting on the group's method for "Gloss and Sheen Uniformity" were discussed. The negative ballot was ruled persuasive.

Group 16: Opacity, Practical Method, J. Csernica, Chairman. The group has had difficulty in locating a suitable, inexpensive paper for performing opacity tests. Consequently, the chairman recommended that the group be disbanded. This was rejected by the subcommittee with the understanding that alternate substrates will be suggested or an ad hoc group formed to study the overall problem.

Group 17: Roller Spatter, J.F. Price, Chairman. Procedures using conventional roller covers and a new notched-spool roller as used and proposed in the first and second round robins respectively were discussed. Spatter charts using these tools were shown. The encouraging results with the new roller seen in the E Z Paint Co. laboratories will be tested in the forthcoming round robin to be conducted prior to the next meeting in January 1980.

SUBCOMMITTEE D01.44 TRAFFIC PAINT

E.B. Countryman, Chairman

Group 1: Hot Melt Traffic Markings, W. Hildebrand, Temporary Chairman.

The proposed specification on thermoplastics was discussed. It was pointed out that no provision was made for yellow thermoplastics. Other comments will be reviewed by the task force chairman. A report was received on the work being done in Sub. D01.21 on chemical analysis of thermoplastics.

Group 2: Traffic Paint, E. Countryman, Chairman. The status of two active round robins were reviewed in detail. One on Method D 711, on Dry Time, should be completed before the January 1980 meeting while the other, D 1309/D 869, will be in progress for at least one year. Another round robin on the bleeding of traffic paint per D 869/D 969 was proposed and an effort will be made to get GSA participation in this program. It was suggested that another task group be formed on Water Borne Traffic Paints since it was noted that GSA is currently purchasing a substantial quantity of such products for airfield markings.

Six test methods which are due for action in 1979 and the seven test methods which are due for review in 1979 were discussed in detail.

Group 3: Night Visibility, E. Countryman, Temporary Chairman. Three test methods which are up for reapproval were discussed. There was also a lengthy discussion on the results of the night visibility readings on the Pennsylvania DOT road test utilizing three different retroreflectometers. A detailed report on this test program should be available at the January 1980 meeting.

SUBCOMMITTEE D01.45 MARINE COATINGS

L.S. Birnbaum, Acting Chairman

Group 2: Shallow Submergence, L.S. Birnbaum, Chairman. Reviewed comments on the revision of Method D 3623, "Testing Anti-fouling Panels in Shallow Submergence." A statement regarding the number of test panels in each system being evaluated will be balloted for inclusion in the method.

Group 3: Intermittent Submergence, A. Ticker, Chairman. Discussed was a previous proposal to restrict its work to coating systems for ship bottom exposure. The group voted to adopt a plan to study coatings for all types of intermittent exposure as listed in the minutes of July 1977.

Group 5: Algae Control, C.D. Stevens, Chairman. Reviewed was a sum-

mary of possible test methods. An *in-vitro* method will be pursued by surveying producers of anti-fouling paint for test methods currently in use. Proposed methods for *in-situ* tests will be written by several members for future consideration.

Group 7: Anti-fouling Rating, L.S. Birnbaum, Chairman. Discussed was a new anti-fouling rating system that will require a new round robin and ultimately will obsolete Method D 3623-78a. Producers of anti-fouling paints will be contacted to enlist their support as cooperators. The cooperation of the U.S. Navy in providing a west coast or Pearl Harbor test site will be pursued. The control systems will be the epoxy

anti-corrosion coating, Mil-P-2441, with the 121 and/or 1020A Mare Island anti-fouling coatings.

DIVISION 50 PAINTS FOR FACTORY APPLICATION

SUBCOMMITTEE D01.51 POWDER COATINGS

R.C. Kissler, Chairman

Group 2: Physical Properties of Powder Coating Materials, J. Huddleston,

Chairman. Three proposed methods for review were received. These were: "Melt Viscosity of Resins, ICI Plate and Cone Viscometer"; "Method for Color of Powder Coating Resin Solutions"; and "Color Measurement of Powder Coating Resins Using a Tristimulus Colorimeter." After discussion, it was decided to send the three methods to Sub. D01.51 members for comments and possible collaborators for round robin testing. The assistance of the Baltimore Society for Coatings Technology will be sought in the round robin on the method for color determination.

Group 3: Application and Film Formation, J. Huddleston, Chairman. Work will be discontinued on particle charge capacity because of the need for basic research in this area. On powder compatibility, a rough draft of a recommended practice will be mailed to members of Sub. D01.51 for review and comments. Work completed on a test method for impact fusion was presented and discussed. Powder samples were requested and promised for additional testing.

Group 4: Properties of Powder Coating Films, R.C. Kissler, Chairman. Received was a report on results using a profilometer from Elcometer in England. They concluded the instrument was not sensitive enough to produce the results desired. Samples of two proposed hiding power test panels were distributed. The substrates were metallized cardboard and cold rolled steel, each coated with black and white squares. These were specially prepared for powder coatings. Volunteers took the samples and promised to report back on their results by the next meeting.

Group 5: Safety and Handling, C. Grenko, Chairman. A questionnaire on "Practice in Storage, Handling and Use of Powder Coatings as They Relate to Safety" was presented. After review and discussion, it was agreed that it should be sent to members of Sub. D01.51 for further comment.

SUBCOMMITTEE D01.52 FACTORY-COATED WOOD PRODUCTS

R.C. Marck, Chairman

Group B: Hardboard, J.J. Medica, Chairman. Test data was reviewed from the second round robin on accelerated tests for hardboard siding. It was pointed out that this evaluation would have been of greater value if variable

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quality substrates had been utilized as well as variable quality finishes. The test fences will be evaluated this fall after three years' exposure. If the need for variable quality substrates is still apparent, a new round robin will be undertaken in January 1980.

Group 11: Dirt Collection, J.J. Medica, Chairman. The results of D-1 balloting on the proposed revision of Method D 3719-78, "Test for Dirt Collection on Exterior White Coated Panels," were reviewed. Two negative ballots and several editorial comments were resolved. These changes will be incorporated into a revised test method for discussion at the winter meeting.

Group 12: Textured Panels, R.C. Marck, Chairman. Test methods were evaluated for determining the durability of textured interior panels. A round robin utilizing the Gardner Straight Line Rub Tester and ASTM standard abrasive cleaner has been completed and was discussed. It is believed that this test method provides the best results obtained to date so a recommended practice will be prepared for the next meeting.

General: Methods D 2793 and D 2839 were reviewed and will be submitted to Sub. D01.52 and Committee D-1 balloting. Method D 2921, "Qualitative Tests for the Presence of Water Repellants and Preservatives in Wood Products," is considered to be beyond the scope of Committee D-1 and will be recommended for transfer to Committee D-7 on Wood during its fall meeting.

**SUBCOMMITTEE D01.53
FACTORY-COATED
STRIP METAL**

E.E. Haney, Chairman

Group 1: Formability, E.E. Haney, Acting Chairman. Test results from Bethlehem Steel, Diamond-Shamrock, and Armco concerning the proposed Ball Punch Test were discussed. The results of the Parker study were also presented. Due to the differences in the results obtained by these investigators, it was agreed that more tests are needed on one or possibly two substrates under specified conditions and should be completed by mid-July. These results are needed to determine the feasibility of future round robin testing. It was agreed that temperature of conducting the test is important and that it will be

recorded at the time of test. The panels and equipment should be allowed to equilibrate prior to testing. All the test data will be forwarded for tabulation and the results and test panels will be brought to the next meeting for discussion and comparison of the ratings.

Group 2: Cure, E.E. Haney, Acting Chairman. A meeting will be held in Pittsburgh in late fall to finalize the program for the solvent rub method for determining cure. Test samples have been prepared and at this meeting the actual methods the cooperators use will be worked out. If everything goes according to the present plan, data could be available for discussion at the next meeting. Future effort will concentrate on the development of other methods for determining cure. Two that were discussed are Evaporative Rate Analysis (ERA) and Infrared Analysis (IR).

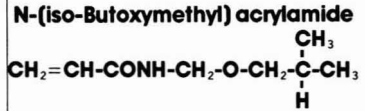
Group 3: Pretreatments, R.L. Williamson, Chairman. Four methods were presented on coating weight determinations on zinc, aluminum, and steel. A round robin will be conducted on minimized spangle galvanized steel to determine coating weights by stripping in an ammonium dichromate solution. Samples of low to high ranges of coating and samples of production metal will be obtained and distributed for test. The x-ray spectrographic method will also be studied to get some information on this procedure.

Group 5: T-Bend Flexibility, R.J. Leipertz, Chairman. A preliminary test method for T-bend flexibility, using E 290, Semi-Guided Bend Test for Ductility as a guide was presented. After discussion it was agreed to use the simple bend method based on making the bend with a vise or a brake press. A round robin will be conducted using seven participants and one metal substrate. A good and a poor flexibility coating will be used. The tests will be conducted so the results may be used for the precision statement.

Group 6: Drawdown Method, R.W. Reinsel, Chairman. A rough draft for the recommended practice on "Applying Coatings by the Drawdown Method" was discussed. Three sections needed to be added on significance, safety, and sampling. Several other editorial changes were made and the revised method will be submitted to Sub. D01.53 members for review.

General: Method D 3363, "Pencil Hardness," was reviewed by several members. The comments were dis-

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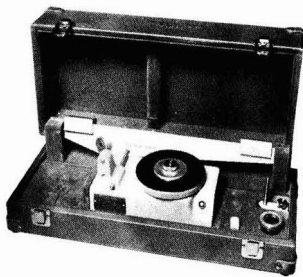
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cussed and a report was made on the status of the National Coil Coaters Association method. It was agreed to submit the method as written to Society ballot for reapproval.

SUBCOMMITTEE D01.55 FACTORY APPLIED COATINGS ON PREFORMED PRODUCTS

J.M. Behrle, Acting Chairman

Group 1: Wood Coatings. R. Wint, Acting Chairman. Reviewed Method D 1308, "Chemical Resistance Using Covered Spot Test," was reviewed and modified to include 15 minute, one hour, or times agreed upon by the buyer and seller in the requirements. The revised method will be submitted for a concurrent Sub. D01.55 and Main Committee ballot.

D01.52/55 UV Cured Coatings: Since the last meeting, a round robin has been started to check a proposed method for determining stability of UV curing coatings. Six cooperators are involved and three different materials are being used, two clears and one pigmented filler. The tests at 60°C are complete. The room temperature tests are planned to run until November 12, 1979. Agreement on the filler test at 60°C was very good as all cooperators reported gelation on the eighth day. With the clear plus inhibitor, three laboratories reported gelation by the 32nd day, one laboratory reported gelation by the 16th day, and one laboratory reported the sample still liquid after 32 days. With the clear without inhibitor, one laboratory reported gelation on the fourth day, three on the eighth day, and one on the 16th day. Discussion centered on the frequency of examination of the clears and whether the tipping of the samples during examination affects the stability. Some additional testing will be done to see if there is such an effect. It was agreed also that it would be good to check all clears additionally on the 24th day.

General: The subcommittee voted to establish a new working group known as Group 7 to develop a recommended practice for water-reducible industrial coatings excluding electrodeposition coatings. A task force was established to prepare a draft practice.

Status of Methods: Method D 1211 was reviewed and recommended for a

concurrent Sub. D01.55 and Main Committee ballot without revision; Method D 3170 was reviewed and recommended for a concurrent Sub. D01.55 and Main Committee ballot with editorial changes; and Method D 1308 was reviewed and recommended for a concurrent Sub. D01.55 and Main Committee ballot with revision to clarify time periods for spot test exposure.

Methods D 3281, D 3023, D 2337, D 2198, and D 365 were balloted by Main Committee in 1978. All comments received were incorporated. Two negative ballots were withdrawn after consultation and editorial changes. All five methods were forwarded for Society ballot.

SUBCOMMITTEE D01.56 PRINTING INKS

F.A. Falk, Chairman

Group 1: Viscosity. J.M. Fetsko, Chairman. A procedure based on the Falling Rod Viscometer, which had been presented at the Atlanta meeting and since rewritten in ASTM format, was reviewed. Time did not allow a complete review so it was decided that the method would be circulated to the members of Sub. D01.56 with the minutes prior to the January meeting. Action for approval is planned for 1980.

Group 6: Tack. W. Gerlach, Chairman. Two suggested methods of test for measuring printing ink tack using the Inkometer were received. The draft prepared by Claudia Shepard was reviewed paragraph by paragraph to provide technical guidance for a rewrite which will be circulated with the minutes prior to the January meeting. Subcommittee approval of the method is anticipated at that time.

General: Method D 3424 is due for review in 1979 and action in 1980. A task group will be selected to review the method and recommend appropriate action at the January 1980 meeting.

SUBCOMMITTEE D01.57 ARTIST PAINTS AND RELATED MATERIALS

J.T. Luke, Chairman

A special all day meeting of the D01.57 Task Groups was held on June

10, 1979 followed by the regularly scheduled meetings on June 11, 1979. The purpose of these meetings was to come to an agreement on package labeling requirements for the proposed standard for artists' paints. Six motions which were passed at the two special task group meetings will be circulated with the minutes in a letter ballot for approval by the subcommittee as a whole.

Group 1: Preparation of Samples for Colorimetric Determination, H.W. Levison, Chairman. All tests have been completed and confidence levels are being determined.

Group 2: Lightfastness of Pigments, H.W. Levison, Chairman. There will be retesting of pigments which did well in the first test, extending exposures from 25,000 langley's to 50,000 langley's. Tests will be in both acrylic emulsion and oil vehicles under fluorescent illumination, outside under glass, and in a xenon-arc fadeometer. A new method of reducing the pigment with white will be used whereby the pigments are reduced to 40% reflectance at the point of maximum absorption.

Group 3: Tinting Strength of Pigments, H.W. Levison, Chairman. A report was given covering the work completed in an attempt to develop a tinting strength standard for artists' paints. Results indicate that the method used in U.S.N.B. Standard CS98-62 was not adequate. A new chairman was appointed to work on the development of another method.

Group 4: Investigation of Test Methods Applicable to this Subcommittee, A. Spizzo, Chairman. Methods D 1210 and D 1640 could possibly be used to check properties of artists' paints. Additional test methods will be developed including consideration of freeze-thaw and long term stability test. Several members were asked to review the suggested methods and give their comments.

Group 5: Pigment Identification, T. Pamer, Chairman. A minor adjustment was made in the reported results of the use of Method D 3256 in identifying phthalocyanine pigments in artists' paints. Copper-free phthalocyanine blue pigments will now be analyzed.

Group 6: Definition of Terms, T. Vonderbrink, Chairman. More terms

suggested by artists were added to the terms given in the report circulated in January. Several individuals noted they have suggestions and corrections to the list and will contact the chairman about them.

Group 7A: Vehicles, Lightfastness, and Distensibility, H.W. Levison, Chairman. Information was presented on the proposed tests for flexibility, deterioration, and yellowing of painting mediums and vehicles. Various products will be tested under conditions of fluorescent illumination as well as sun exposure. Adhesion and distensibility tests will also be conducted on various combinations of acrylic emulsion paints on different painting supports.

Group 10: Toxicity. This group is in need of a qualified person to serve as chairman. Dr. John Cromer, of Duke University, will serve as toxicologist and Mr. David Pomeroy, of the U.S. Consumer Product Safety Commission, is interested in participating. A list of pigments used in artists' paints were submitted to Dr. Cromer for his comments.

Technical Articles in Other Publications

Compiled by the Technical Information Systems Committee—H. Skowronka, Chairman

Double Liaison - Chimie des Peintures (in French)

Published by EREC, 68 rue Jean-Jaures, 92800 Puteaux, France

Vol. 26 No. 284

April 1979

Deighton, G.M. - "Durability and Temperature"; 23-29.

Pion, G. - "Protection of Concrete Floor. Paints and Supports"; 31-33.

Dauvilliers, J. - "Research on Oil Soluble Formophenolic Resin Reactions"; 35-41.

Sickfeld, J. and Heinze, B. - "Application of Thermoanalysis for the Investigation of Coating Deficiencies"; 366-369.

Kaiser, H. - "Economy Heating of High-Temperature Reaction-Vessels for Production of Synthetic Resins"; 369-375.

Häntzschel, W. - "Applicability of Spindlemill 192 C in the Paint Industry"; 376.

Brushwell, W. - "New Developments in Paints and Coatings" (literature review); 377.

Anon. - "Cubic Cans in the Paint Industry"; 406-409.

Farbe und Lack (in German)

Published by Curt R. Vincentz Verlag, Schiffgraben 41-43, 3000 Hannover, Postfach 6247, Germany

Vol. 85 No. 5

May 1979

Anon. - "New Picture in Paint Cans"; 355.

Ahmad, H. and Yaseen, M. - "Solubility Parameter Values for Polyethylene Sulfonates and Polyethylene Sulfonyl Halides Determined by Chemical Group Contribution Technique"; 356-361.

Weinmann, K. - "Emulsion Paints and Paints Based on Silica for Old and New Constructions. A Critical Comparison of Properties and Applications"; 361-365.

Plaste und Kautschuk mit Fachtteil Anstrichstoffe (in German)

Published by VEB Deutscher Verlag für Grundstoffindustrie, 27 Karl Heine Strasse, 7031 Leipzig.

Vol. 26 No.3

March 1979

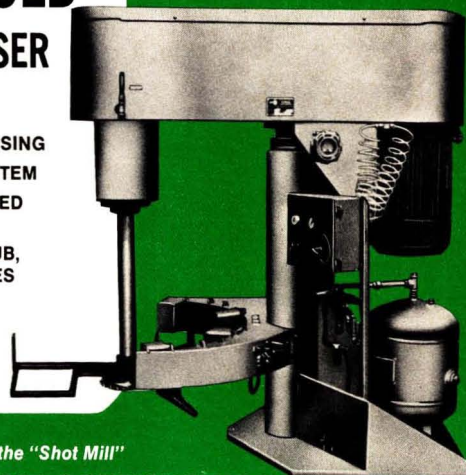
Scheler, H., Ronsch, E., Schneider, W., and Schulz, W.-D. - "Present Status of Development of Silicate-Based Paint Systems with Zinc and Other Metallic Pigments"; 163-164.

Huttmann, E., Millow, S., Korn, E., and Killiches, N. - "Studies of Drying Behaviour of Alkyd Resin/Melamine Resin-Based Paints With and Without Acid Catalyst Under the Influence of Heat and Ultraviolet Radiation"; 165-167.

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- ALLEN, JOHN F. — Zany Eminatons, San Francisco, Calif.
BRILL, BERNARD M. — Union City Chemicals, Union City, Calif.
DANIEL JR., WILLIAM G. — International Paint Co. Calif., Inc., South San Francisco, Calif.
HUFFMAN, JOHN W. — California Ink, Berkeley, Calif.

Associate

- BOARD JR., CHARLES L. — E.I. duPont de Nemours & Co., Inc., Foster City, Calif.

Retired

- CALLIS, DALE E. — San Carlos, Calif.

HOUSTON

Active

- CHMIEL, RICHARD P.—Reliance Universal, Houston, Tex.
GOMEZ, VICTOR—Reliance Universal, Houston.
HALL, RUSSELL M.—Ferra-Lox Coatings, Channel View, Tex.
PHILLIPS, GARY M.—Reliance Universal, Houston.

Associate

- DOUGAN, DICK—Mobay Chemical Corp., Spring, Tex.
McDERMOTT, ARTHUR R.—Nalco Chemical Co., Sugar Land, Tex.

NEW ENGLAND

Active

- CONTI, MICHAEL — Cadillac Paint & Varnish, Sharon, Mass.
EISELLER, RICHARD C. — Davidson Rubber Co., Dover, N.H.
GAY, EDWARD T. — Sterling-Clark-Lurton Corp., Malden, Mass.
JOHNSON, RICHARD E. — Polyvinyl Chemical Ind., Wilmington, Mass.
KARP, DONALD F. — Cadillac Paint & Varnish, Boston, Mass.
KELTZ, GARY — Polyvinyl Chemical Ind., Wilmington.

KIEZULAS, MARGARET P. — Polyvinyl Chemical Ind., Wilmington.

LEVINE, LEON — Loctite Corp., Newington, Ct.

SHEPHERD, LEE — M & M Paint Mfg. Co., Pawtucket, R.I.

TAYLOR, PAUL — Shipley Co., Newton Lower Falls, Mass.

WERTZ, HARRISON G. — Schenectady Chemicals, Schenectady, N.Y.

Associate

DENNIS JR., DANIEL J. — Intl. Minerals & Chemicals, Litchfield, N.H.

WILLIS, DARRYL — E.I. duPont de Nemours & Co., Inc., Holliston, Mass.

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H.B. Fuller Co., St. Paul, Minn., has named **Hans Dirks** to the position of Plant Manager for their industrial coatings department. He will be responsible for plant management and for the production of powder coatings and water-based industrial finishes. Mr. Dirks is a Past-President of the Northwestern Society.

Ulla Walsler has been named a Technical Sales Representative for Packaging Products at DeSoto, Inc., Des Plaines, Ill. Ms. Walsler is a member of the Chicago Society for Coatings Technology.

Meanwhile, **Ken J. Matyska** has also been appointed a Sales Representative for the DeSotos Packaging Products.

Thomas Stasiak has been appointed Director of Marketing of Applied Color Systems, Inc., Princeton, N.J. He will be responsible for new product development and for the promotion of the firm's color control systems.



H. Dirks



U. Walsler



L. Burns



J.E. Rademan

Larry Burns has been appointed Central District Manager for Universal Color Dispersions, Lansing, Ill. He is a member of the Chicago Society.

Jerry E. Rademan has joined the Additives Dept. of CIBA-GEIGY Corp. in Ardsley, N.Y., as a Technical Representative. Mr. Rademan is involved in the department's coatings, inks, and photographic business, working in the industrial and trade sales coatings markets.

Francis X. McDermott has been appointed to the position of Vice-President of Kelco, San Diego, Calif. He will be responsible for marketing and sales, as well as research and development, and production.

Meanwhile, the company has named **Donald W. Krech** Director of Production.

Harri T. Itameri has been named Marketing Manager of Latin America for Sylvachem Corp.

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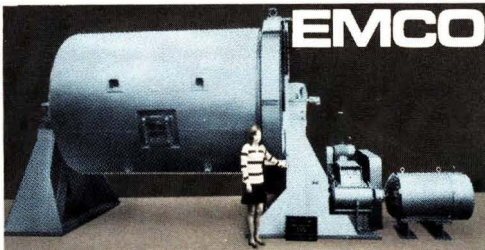
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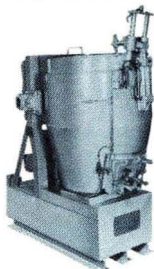
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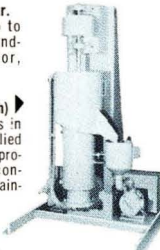
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Applied Color Systems, Princeton, N.J., has appointed **David Malthouse** Regional Manager for the Midwestern states. His responsibilities will include technical sales assistance to the company's representatives and customers in his region.

In a series of managerial appointments, the Petrochemical Group, Union Chemicals Div., of Union Oil Co., Calif. has named **William A. Oudshorn** to the newly created position of Manager of Distributor Sales and Marketing Administration; **William Murphy** to General Manager of the group's Midwestern region; and **Robert C. Hoffman** to Manager of Marketing Planning and Sales Service.

In addition, **Dr. Evord F. Knights** has been appointed to the position of Manager of Quality Control and Manufacturing Planning and **Ted S. Hallen** to Plant Manager for the Petrochemical Group's manufacturing plant in La Mirada, Calif.

The DuPont Company has named **H.S. Wellman** Manager of its Troy Laboratory. He succeeds **F.M. Gavin** who will manage research and development for the company's Specialty Products Div. in Wilmington, Del. Mr. Gavin is a member of the Detroit Society for Coatings Technology.

At DuPont's Marshall Lab. in Philadelphia, **Q.E. Lengel** has been named Technical Manager with responsibility for overseas technical programs. He will be assisted by **C.J.A. Peters** at the Marshall Lab. and **D.C. Gleason** at Troy.

Obituary

Fred Apfel, 57, Plant Manager of International Paint Co., San Francisco, Calif., and President-Elect of the Golden Gate Society, died May 21, of cancer. He is survived by his wife, Herta, a daughter, Ruth, and a grandson.

William R. Holmes, 72, recently retired Vice-President of D.H. Litter Co., Inc., died July 9, in Boston, following a long illness. Mr. Holmes was a Past-President and Honorary Member of both the New England Society for Coatings Technology and the New England Coatings Association. He also served two terms as Regional Vice-President of National Paint and Coatings Association.

E.I. du Pont de Nemours & Co. has named **Jeffrey M. Lipton** to the position of Director of the Colored Pigment Products Div. in the Chemicals, Dyes, and Pigments department. He formerly served as Director of the Development Div. in the company's Central Research and Development department. Mr. Lipton succeeds **Dr. Edward C. Broge**, who has been appointed Director of the White Pigment and Mineral Products Div.

Donald R. Bushell has been appointed a Business Unit Manager of the Printing Ink Group of Inmont Corp., Clifton, N.J. In his new position, Mr. Bushell will be responsible for the nationwide marketing of many of the company's ink products.

Morgan C. Locke, Jr. has been appointed Vice-President of Chemical Manufacturing operations for Kerr-McGee Chemical, Oklahoma City, Okla. In his new position, Mr. Locke will be responsible for the ten manufacturing operation of the company.

Meanwhile, **Ray C. Green** has been named Manager of Kerr-McGee's Searles Valley operations.

Gary L. Mossman has been appointed General Manager of the newly formed Specialty Chemicals Div. of Thiokol Corp. This new operation was created by the consolidation of the company's Chemical Div. in Trenton, N.J. and Southwest Specialty Chemicals, Inc., Houston, Tex. Mr. Mossman, formerly President of SSCI, will be headquartered in Trenton.

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Coatings System

Literature is now available which describes a new system of coatings and paints that cleans easily with water, promises cleaner air, and has the potential for saving large amounts of petroleum. For additional information, write The Flecto Company, Inc., 1000 45th St., Oakland, Calif. 94608.

Self-Cleaning Vibrotron

Literature which describes a self-cleaning electrostatic precipitator that collects hazardous "respirable fraction" dust and fume particles is now available. Information on this unit may be obtained by contacting Eltron Manufacturing, Inc., Interstate Industrial Park, Bellmawr, N.J. 08031.

Glycol Ethers

A 24-page booklet which details the use of glycol ethers in the coatings industry has been prepared. These glycol ethers, through the combination of an alcohol and an ether functional group in the same molecule, provide solvency characteristics appropriate for the individual systems for which they are designed. For copies of booklet F-47184, write Union Carbide Corp., Chemicals and Plastics, Dept. MLH, 19th floor, 270 Park Ave., New York, N.Y. 10017.

Coatings Newsletters

Two monthly reports, "Powder Coatings Bulletin" and "Waterborne & High Solids Coatings," are now available. Both newsletters provide an updating service on the respective aspects of the environmentally friendly coatings field. Such topics as raw materials, new products, environmental news, standards, health and safety, market surveys, and company news are covered. The subscription rate for each bulletin is \$110 for one year; \$200 for two years; and \$264 for three years. Combined subscription rates are also available. For order payments and fulfillment enquiries, write Elvesier International Bulletins, 52 Vanderbilt Ave., New York, N.Y. 10017.

Diatomite Filteraids

Techniques for obtaining the best results in vacuum filtration systems using diatomaceous earth filteraids are described in a nine-step instructional bulletin. For a copy of Bulletin K-117, contact Witco Chemical Corporation, Inorganic Specialties Div., 277 Park Ave., New York, N.Y. 10017.

ASTM Standards

New and revised standards dealing with the production, purchasing, application, and inspection of paints and related coatings are contained in the recently released *Parts 27, 28, and 29 of the Annual Book of ASTM Standards*. The publications contain over 612 standards, covering such topics as paint tests for formulated products and applied coatings; paint pigments; and resins and polymers. Additional information may be obtained by writing Pamela R. Bateman, ASTM, 1916 Race St., Philadelphia, Pa. 19103.

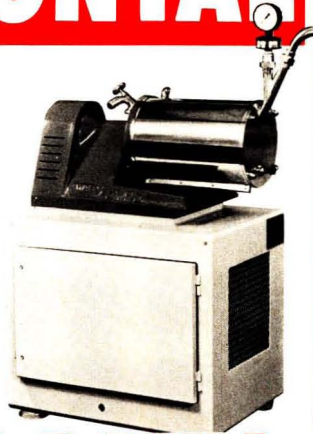
Process Equipment Series

A book series, providing information on the basic processes and equipment used in the chemical and related industries, is to begin in September with the publication of Volume I "Solids Separation and Mixing." This book will contain 291 pages and will be available for \$35 from Technomic Publishing Company, Inc., 265 Post Road, West, Westport, Conn. 06880.

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Coming Events

FEDERATION MEETINGS

(Oct. 2)—Federation Board of Directors Meeting. Sheraton St. Louis Hotel, St. Louis, Mo. (FSCT, Suite 832, 1315 Walnut St., Philadelphia, Pa. 19107).

(Oct. 3-5)—57th Annual Meeting and 44th Paint Industries' Show. St. Louis Convention Center, St. Louis, Mo. (FSCT, Suite 830, 1315 Walnut St., Philadelphia, Pa. 19107).

SPECIAL SOCIETY MEETINGS

(Sept. 19-20)—Montreal and Toronto Societies Joint Symposium. Toronto—19th; Montreal—20th. "Coating Technology Economics—Can Know-how Make Money?" (Montreal—R.A. Fraser, CNR Technical Research Center, 3950 Hickman Ave., Montreal, Quebec H4T 1K2; Toronto—Paul Cooper, Hoechst Dyestuffs, 100 Temple Ave., Willowdale, Ontario M2H 2N8).

(Oct. 18)—Philadelphia Society. "Sludge '79" - Liquid/Solid Waste Seminar. Valle's Steak House, Philadelphia, Pa. (Wayne A. Kraus, Lawrence-McFadden, 7430 State Rd., Philadelphia, Pa. 19136).

1980

(Mar. 10-12)—Seventh Annual Water-Borne and Higher-Solids Coatings Symposium. Hyatt Regency Hotel, New Orleans, La. Sponsored by Southern Society and University of Southern Mississippi. (Dr. George Bufkin, Dept. of Polymer Science, University of Southern Mississippi, Southern Station, Box 276, Hattiesburg, Miss. 39401).

(April 14-16)—Dallas and Houston Societies. Southwestern Paint Convention, Hilton Inn, Dallas, Tex.

(May 1-3)—Pacific Northwest Society Annual Symposium. Marriott Hotel, Portland, Ore.

OTHER ORGANIZATIONS

(Oct. 1-4)—"Progress in Corrosion Control," Canadian Regional Meeting, National Association of Corrosion Engineers. Skyline Hotel, Ottawa, Ontario, Canada. (Membership Services Dept., NACE Headquarters, P.O. Box 986, Katy, Tex. 77450).

(Oct. 9-11)—Western Regional Meeting, National Association of Corrosion Engineers. Jack Tar Hotel, San Francisco, Calif. (Membership Services Dept., NACE Headquarters, P.O. Box 986, Katy, Tex. 77450).

(Oct. 10-11)—"Cathodic Protection," North Central Regional Meeting, National Association of Corrosion Engineers. Alameda Plaza Hotel, Kansas City, Mo. (Membership Services Dept., NACE Headquarters, P.O. Box 986, Katy, Tex. 77450).

(Oct. 10-12)—Fifth International Photopolymer Conference: Principles, Processes, and Materials, sponsored by the Mid-Hudson Section, Society of Plastics Engineers. Nevele Country Club, Ellenville, N.Y. (Dr. Maung Htoo, 10 Rabbit Trail Rd., Poughkeepsie, N.Y. 12603).

(Oct. 10-12)—"Polymer—Polymer Composites: Blends, Grafts, and Blocks," State University of New York. (Dr. Angelo V. Patsis, Chairman, Department of Chemistry, State University of New York, New Paltz, N.Y. 12562).

(Oct. 15-17)—9th Congress of the Federation of Scandinavian Paint and Varnish Technologists. Stockholm, Sweden.

(Oct. 15-19)—Institute of Applied Technology, Training Course, "Marine Coating Procedures." Phila., Pa. (Institute of Applied Technology, Suite 600, 1776 K St., N.W., Washington, D.C. 20006).

(Oct. 15-19)—"Scanning Electron Microscopy and X-Ray Microanalysis: Theory and Practice in Materials Science, Biology, and Medicine," State University of New York. (Dr. Angelo V. Patsis, Chairman, Department of Chemistry, State University of New York, New Paltz, N.Y. 12562).

(Oct. 15-Nov. 9)—Paint Short Courses at University of Missouri—Rolla. Paint Formulation Course—Oct. 15-19; Advanced Coatings Workshop—Oct. 22-26; For Painting Contractors and Maintenance Engineers—Nov. 5-9. (Norma Fleming, Extension Div., University of Missouri—Rolla, 501 W. 11th St., Rolla, Mo. 65401).

(Oct. 16-17)—Society of Plastics Engineers National Technical Conference, "Coloring of Plastics." RETEC sponsored by the Philadelphia Section and the Color and Appearance Div., Sheraton Poste Inn, Cherry Hill, N.J. (Robert C. Foley, Society of Plastics Engineers, 656 W. Putnam Ave., Greenwich, Conn. 06803).

(Oct. 16-18)—Sixth Annual UMR-DNR Conference and Exposition on Energy. Conducted by the University of Missouri—Rolla and the Missouri Dept. of Natural Resources. (Dr. J. Derald Morgan, Conf. Dir., 108 Electrical Engineering Dept., University of Missouri—Rolla, Rolla, Mo. 65401).

(Oct. 16-18)—Northeast Regional Meeting, National Association of Corrosion Engineers. Sheraton Hotel, 7th Ave. and 56th St., New York, N.Y. (Membership Services Dept., NACE Headquarters, P.O. Box 986, Katy, Tex. 77450).

(Oct. 21-24)—Southeast Regional Meeting, National Association of Corrosion Engineers. Landmark Hotel, Myrtle Beach, S.C. (Membership Services Dept., NACE Headquarters, P.O. Box 986, Katy, Tex. 77450).

(Oct. 22-24)—"Fundamentals of Adhesion: Theory, Practice, and Applications" State University of New York. (Dr. Angelos V. Patsis, Chairman, Department of Chemistry, State University of New York, New Paltz, N.Y. 12562).

(Oct. 23-25)—"Finishing '79," sponsored by the Association for Finishing Processes of SME. Convention Center, Cincinnati, Ohio. (Edward Cheryholmes, AFP/SME, One SME Dr., P.O. Box 930, Dearborn, Mich. 48128).

(Oct. 29-31)—National Paint and Coatings Association Annual Meeting. San Francisco Hilton Hotel, San Francisco, Calif. (Georgene Savickas, NPCA, 1500 Rhode Island Ave., N.W., Washington, D.C. 20005).

(Oct. 30-31)—Institute of Applied Technology, Mini Course, "Designing for Protective Coatings." Boston, Mass. (Institute of Applied Technology, Suite 600, 1776 K St., N.W., Washington, D.C. 20006).

(Nov. 6-7)—Institute of Applied Technology, Mini Course, "The Specification Document: Key to Quality Coating Work." Dallas, Tex. (Institute of Applied Technology, Suite 600, 1776 K St., N.W., Washington, D.C. 20006).

(Nov. 6-8)—Society of Plastics Engineers, National Technical Conference, "Plastics: The Answer to Transportation in the 80's." Detroit Plaza Hotel, Detroit, Mich. (Eugene E. Wilson, SPE, 656 W. Putnam Ave., Greenwich, Conn. 06830).

(Nov. 7-8)—2nd Resins and Pigments Exhibition. Royal Hotel, Copenhagen, Denmark. (Mike McIntyre, International Symposia & Exhibitions Ltd., Queensway House, 2 Queensway, Redhill, Surrey RH1 1QS, England).

(Nov. 16-18)—Annual National Decorating Products Association Convention and Show. McCormick Place, Chicago, Ill. (NDPA, 9334 Dielman Industrial Dr., St. Louis, Mo. 63132).

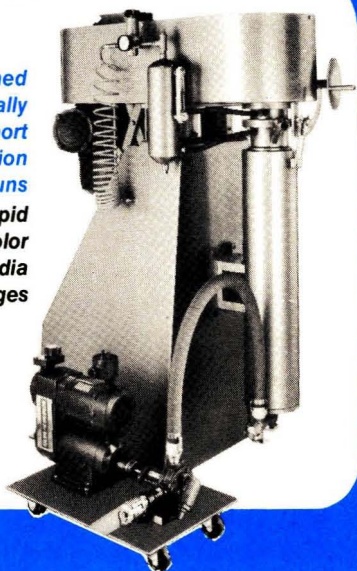
(Nov. 20-21)—Institute of Applied Technology, Mini Course, "Estimating, Planning, and Scheduling Painting." Chicago, Ill. (Institute of Applied Technology, Suite 600, 1776 K St., N.W., Washington, D.C. 20006).

(Dec. 3-7)—Institute of Applied Technology, Training Course, "Nuclear Quality-Assured Coating Work." Miami, Fla. (Institute of Applied Technology, Suite 600, 1776 K St., N.W., Washington D.C. 20006).

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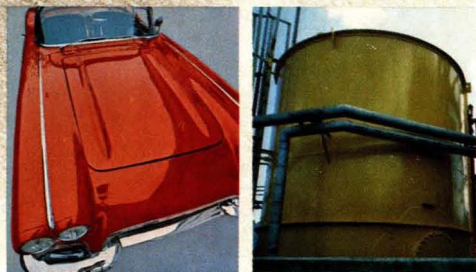
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Comparison of Busan® 11-M1 (middle) with zinc phospho-oxide (left) and calcium borosilicate (right) in latex emulsion primer. Each primer topcoated with two coats of latex emulsion paint without inhibitor. Panels exposed 200 hours in salt fog cabinet. Panels shown before and after paint was removed.

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