

ict JOURNAL OF COATINGS TECHNOLOGY

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FEBRUARY 1983



Reactivity of Calcium Carbonate and Cuprous Oxide with Binder Acid Components in Antifouling Paints



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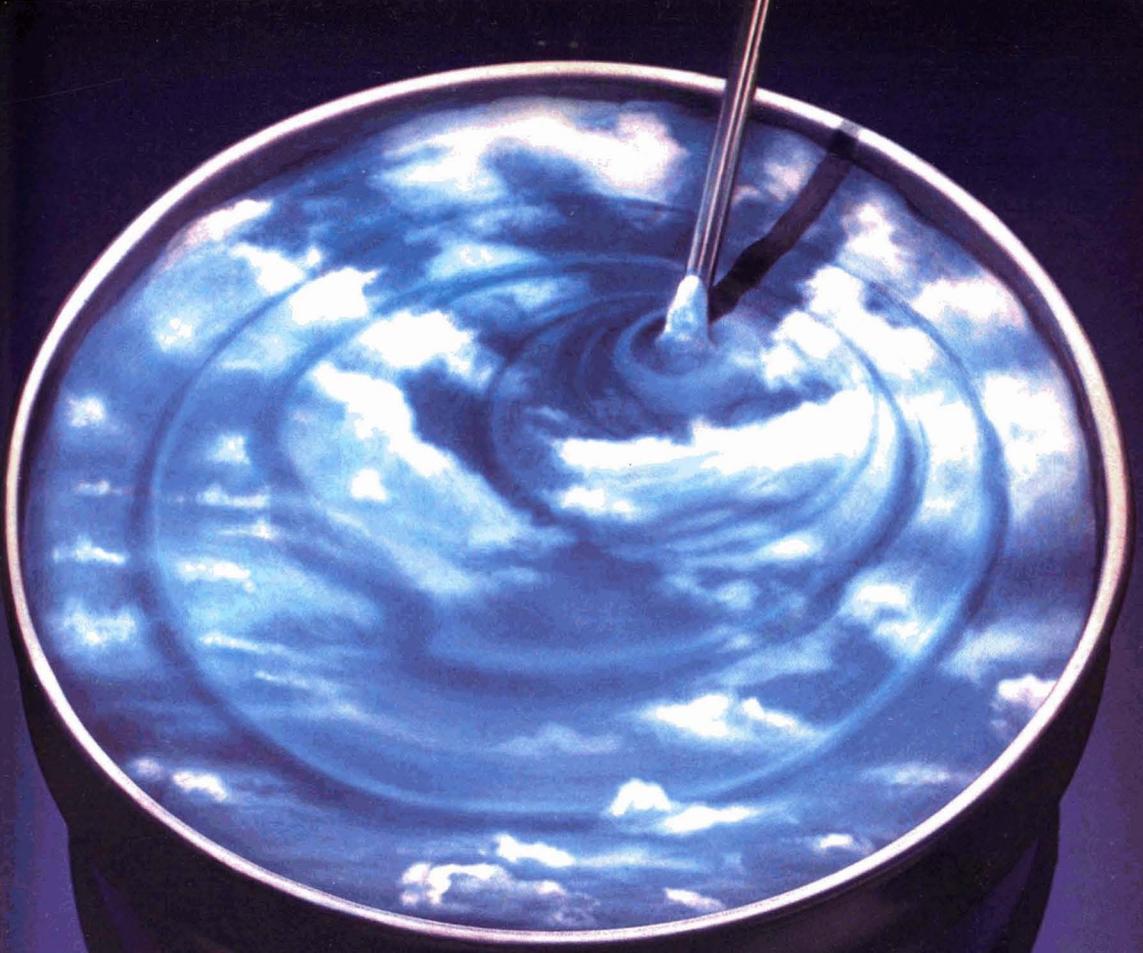
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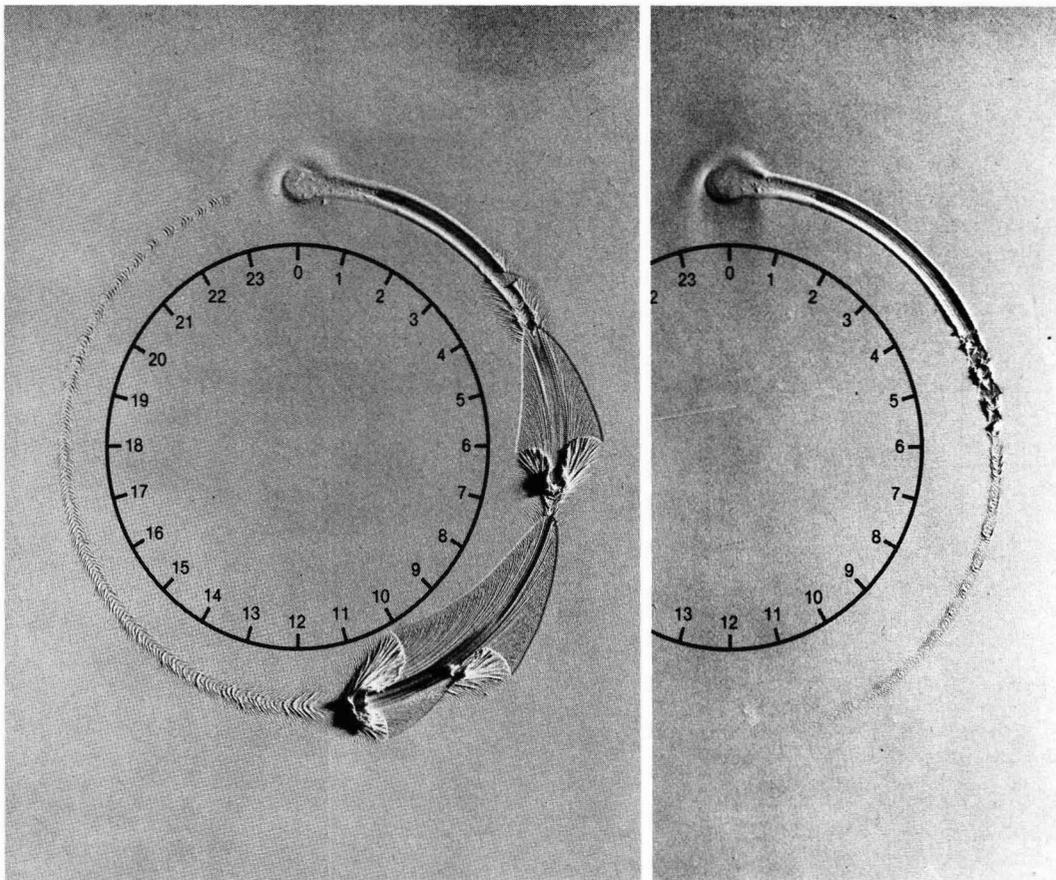
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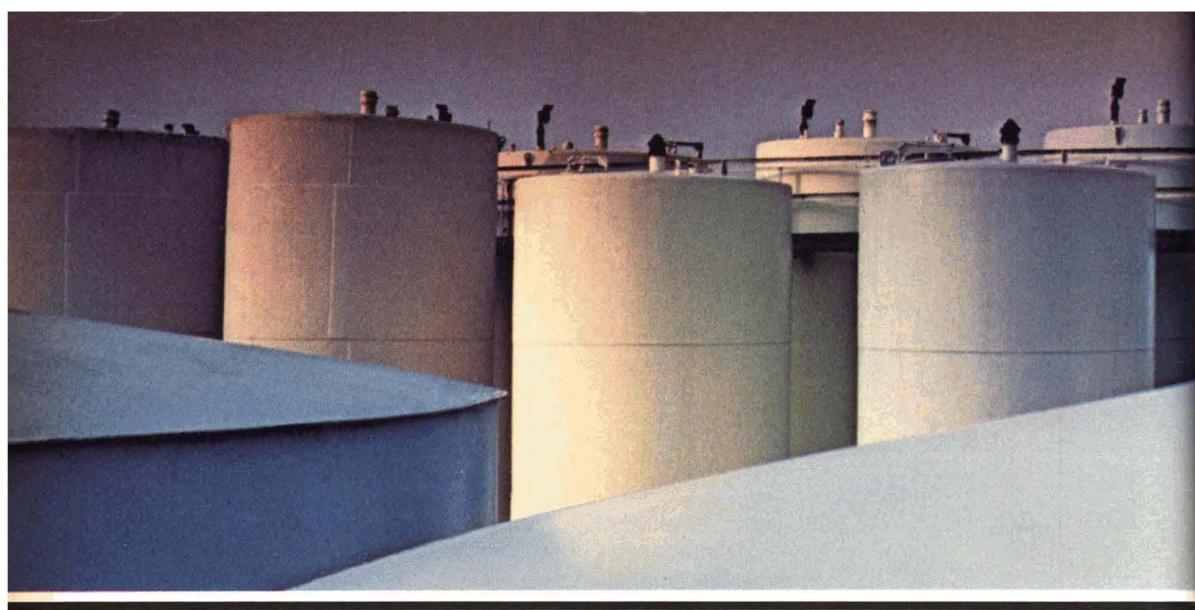
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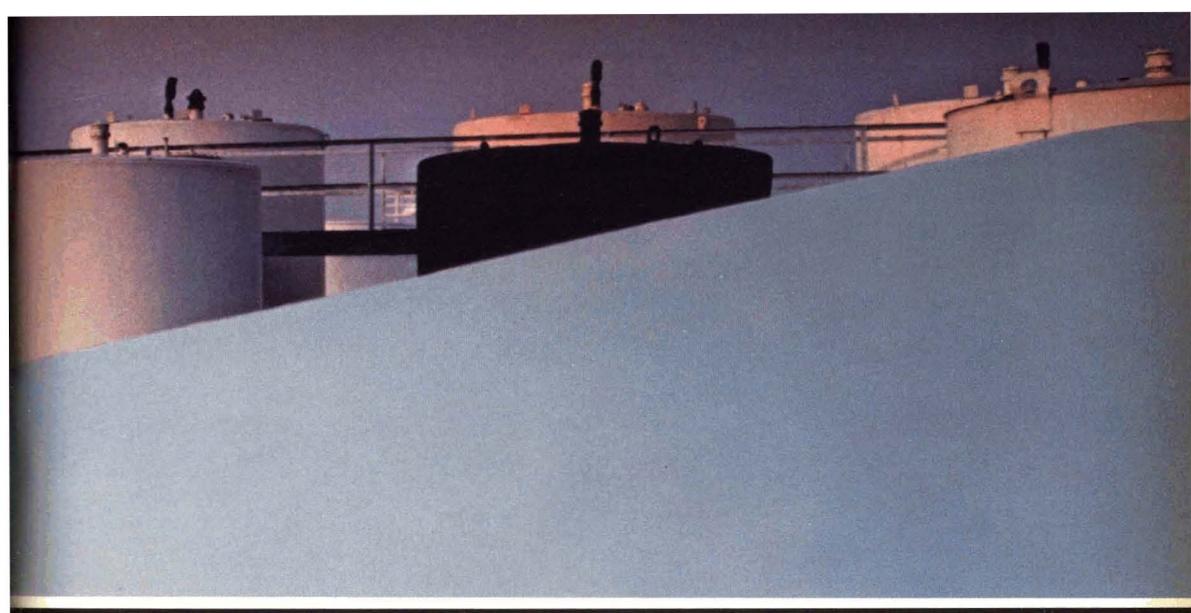


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Bisphenol-A based coating.

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Chemical and solvent resistance of XU-252 based coatings							
Reagents	Months to date/test duration 8 months						
	1	2	3	4	5	6	7
Acetic acid (10%)							Failed
HCl (36%)							
NH ₄ OH (30%)							
Acetone							
Methylene chloride							
Methanol							
cure conditions: 10 hours @ 80°C (176°F)							

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Comment

Roon Awards—YOU Are Invited!

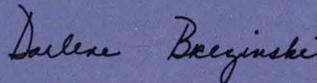
Each year authors are invited to participate in the Roon Awards Competition, and this year we are inviting YOU! The objective of the competition is to encourage and stimulate written contribution to the coatings industry which reflects the caliber of the scientific work being conducted.

The award competition is open to anyone involved in the study of or engaged in work related to the protective coatings industries, including manufacturers, raw material suppliers, research laboratories, and universities. The papers must describe original work and should reflect the advances which are being made in the field of protective coatings.

By entering the competition, authors take advantage of an excellent opportunity to make a scientific contribution, to derive professional prestige for themselves and their organizations, and also to share in the available prize money. The Roon Awards Committee has available \$4000 in prize money to award to authors of winning papers in the 1983 competition. Financing for these Awards, which are sponsored by the Federation's Paint Research Institute, has been provided since 1957 by the Leo Roon Foundation.

We encourage those who wish to participate to prepare now by submitting a letter of intent to compete, along with a suggested title, to Dr. Darlene Brezinski, DeSoto, Inc., 1700 S. Mt. Prospect Rd., Des Plaines, IL 60018.

We encourage YOU to make this year's competition an overwhelming success by sharing the knowledge that our research provides.



Darlene Brezinski
Chairman, Roon Awards Committee

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In fact, we think two-component coatings based on this Henkel resin will match or surpass any product on the market today. They provide twice the coating life of alkyds. Their adhesion and durability are equal to other leading urethanes. But these coatings cost less than a polyester/isocyanate system, because they require less of the high-cost isocyanate components.

But there's more to our acrylic family than just G-CURE 867. Our high-performance G-CURE 869 gives you an extremely fast tack-free dry time—almost lacquer-like drying. And our G-CURE 105-R70 is a lower molecular weight acrylic, designed to improve volume solids and reduce solvent vapor emissions by about 50 percent.

So if you need a long-life, high-mileage urethane formulation, contact Henkel. We have acrylic resins you'll take a shine to. For more information about G-CURE acrylic resins, write: Resins Division, Henkel Corporation, 4620 W. 77th Street, Dept. JCT 23, Minneapolis, MN 55435.

Resins Division



Committee Activities

DELEGATE TO SSPC

Report of Fall Meeting of Steel Structures Painting Council

The fall meeting of The Steel Structures Painting Council (SSPC) was held in Pittsburgh on September 28-30, 1982. A total of 130 suppliers, users and representatives of government agencies attended. About one third are members of the Federation.

The following minutes were submitted in time to report to the Federation:

SURFACE PREPARATION

Kenneth S. Trimber—Chairman & Acting Secretary

(1) *Power Tool Cleaning to Bright Metal* (J. Belisle)—A test panel study to compare power tool cleaning methods to achieve a "Near White" degree of cleaning will be carried out at Metalweld, Philadelphia, PA. The prepared panels will be incorporated in the PACE program. Guidelines for the use of power tools will also be prepared for review at the next meeting.

(2) *Abrasive Specifications and/or Guides* (W. Hitzrot)—The Maritime Administration is working on similar guidelines for abrasive evaluation. Therefore, Hitzrot will act as liaison with SSPC.

(3) *Wet Blast Cleaning* (T. Dowd)—Guides will be prepared for both water blast cleaning and wet blast cleaning. The guides will include inhibitors, flow rates, disposal of debris and safety.

(4) *Centrifugal Wheel Blast Cleaning* (D. Hale)—A guide will be prepared to supplement information on equipment and abrasives described in Volume I. The guide will include control of operating mix, adjusting wheel pattern, maintaining abrasive flow rate, ventilation and avoiding grease/oil contamination.

(5) *Organic Acid Cleaning* (H. Gewanter)—A specification will be developed including guidelines for cleaning methods and procedures.

(6) *Miscellaneous* (L. Schwab)—New cleaning methods will be investigated including a rust removal paste (PVP and EDTA in water), Xenon flash lamps, CO₂ pellets, and ultrasonic cleaning.

Surface Preparation for Maintenance Painting (B. Appleman)—The Maintenance Painting Committee needs guides for power tool and wet blast cleaning prior to repainting. Appleman will act as liaison to submit these anticipated guides for review by the committee.

SSPC Surface Preparation Manual (H. Hower)—SSPC is interested in preparation methods including the guides and specifications to be prepared by the Task Groups. The projected publication date is early Summer 1983.

BRIDGE PAINTING RESEARCH

Dr. Lloyd M. Smith—Chairman & Acting Secretary

The Federal Highway Administration is contemplating the development of a painting inspectors course. Suggestions are solicited as to format, content and length.

Reports on Improved Field Reliability of High Performance Coatings and Coatings for Non-Blast Cleaned Steel Surfaces will be available in a few months.

The following projects are underway at the present time:

(1) The National Bureau of Standard's short term evaluation

and analysis for predicting coating life. One method uses thermography for detecting underfilm corrosion.

(2) Florida Dept. of Transportation evaluation of and recommendations for the bridge corrosion cost model.

(3) The National Cooperative Highway Research Program to investigate the removal of lead-based bridge paint.

Federal Highway Administration research projects will be curtailed because of recent funding cutbacks.

PACE—EVALUATION OF PAINTS

Dr. Bernard R. Appleman—Chairman

Lloyd M. Smith—Secretary

The PACE program was started in 1974 in order to locate acceptable replacements for lead and chromate pigments, organic solvents and silica abrasives, all of which are being strictly regulated.

Phase I was sponsored by Penn. DOT, EPA, and several industry groups. Phase II, started in 1980, was sponsored by 25 state highway departments, FHWA, and EPA. The sponsorship is due to end in May 1984.

Pace I—This series includes the following:

	Paints	Panels	Exposed
(1) Alternate pigments in oil/alkyd	55	1200	1977
(2) Alternate pigments in vinyl	—	300	1977
(3) Alternate surface preparation	—	1000	1977
(4) Water-borne coatings	—	650	1978

PACE II—This series include the following:

	Paints	Panels	Exposed
(1) Alternate coatings	54	1700	1982
(2) Water-borne epoxies	—	—	1982
(3) Alternative surface preparation	—	200	1983
(4) Bridge exposure tests	9	500	1983

This series will be expanded to include the following variations:

(5) Alternate surface preparation method on rusted panels including steel grit, steel shot, non-metallic abrasives, citric acid pickling, water/sand/air blasting, power tool cleaning and hand cleaning.

(6) Five alternate coatings including an oil/alkyd control, an inorganic zinc, an organic zinc, a non-lead, non-chromate vinyl and a water-borne system (Cal. spec.).

(7) Six or seven coatings exposed in a marine site (Oregon), rural site (Kansas), heavy industrial site (Louisiana) and to deicing salts (Michigan), plus three accelerated tests.

PACE III—Proprietary Paints—It has been proposed that coating manufacturers and raw material suppliers absorb the major cost of this evaluation on their coatings since government funds have decreased considerably and will run out in 1984.

Task groups were formed to initiate a program for selecting and testing paints under PACE II₃, as well as the method of reporting the data obtained.

ONE COAT SHOP PAINTS

William C. Spangenberg—Chairman & Secretary

Draft No. 1 of SSPC—Paint XOCSPX-82P "One-Coat Shop Primer—With Alternate Inhibitors" had been reviewed

Due to a lack of space in the January 1983 issue, we were unable to publish the full text of Delegate Sidney Levinson's report to the Federation Board of Directors. We are pleased to do so now—Ed.

by the Committee. John Keane will be requested to review the corrected draft for editorial changes and then submit it for letter ballot as Draft No. 2.

VINYLS

Tom Ginsberg—Chairman & Acting Secretary

Efforts will be concentrated on the development of a high-build vinyl coating specification. This specification should be more detailed to more closely duplicate Military specifications. The first draft will be prepared in time for review at the January meeting.

It is anticipated that this document will be completed in time for inclusion in the second printing of Volume II in 1984.

WATER-BORNE EPOXIES

Dr. Edward Bozzi—Chairman

Arthur L. Cunningham—Secretary

The draft of the specification was mailed to the members of the Committee for review and ballot. The comments received were reviewed and the document revised where they were considered to be valid.

A revised draft will be submitted to the Committee members for letter ballot.

ZINC-RICH COATINGS

Kenneth Tator—Chairman

Jerry Everts—Secretary

J. Everts reported on the results of the evaluation of zinc level on performance. The data will be submitted at the January meeting. The formulations will also be circulated for comment.

The University of Texas has investigated "Faying Surfaces" with zinc-rich paints. Their report may become the basis of an ASTM standard. An abstract of their work will be distributed by J. Moore.

The careless use of chlorinated solvents with zinc-rich paints can be hazardous because of its reactivity. This is especially critical when using aluminum spray equipment. Dow Chemical Co. will be asked to supply recommendations for the proper use of their chlorinated solvents.

G. Tinklenberg and T. Neal will revise and update the "Guide to Topcoating Zinc Rich Primers" in time for the January meeting.

SSPC will be asked to distribute drafts of the "Guide to Topcoating Zinc Rich Primers" and the "Guide to Zinc Rich Coatings" to the members of the Committee.

PAINT FILM THICKNESS

Forrest M. Couch—Chairman

David Graham—Co-Chairman & Acting Secretary

The preliminary draft of the proposed specification for the "Measurement of Dry Paint Thickness by Destructive Means" was distributed by M. Schroeder. It will be reviewed by mail.

The "Measurement of Dry Film Thickness with Battery Powdered Digital Gauges" was discussed by J. O'Connor. He will investigate it further before preparing any document.

Excessive film thickness was recognized as a serious problem not adequately covered in present documents. The internal stresses caused at such thicknesses often result in loss of adhesion. Unfortunately, maximum film thickness varies with different coatings. The committee will review the existing comments in PA-2 concerning maximum film thickness and will develop a clearer warning of the potential problems.

MAINTENANCE PAINTING

Dr. Bernard R. Appleman—Co-Chairman

James R. Courtright—Co-Chairman and Acting Secretary

B. Appleman reviewed the "Guide to Maintenance Repainting with Oil Base or Alkyd Painting Systems"—SSPC-PA Guide 4—with particular emphasis on the tabular summary of paint system condition, rust grades, and cleaning and painting recommendations.

After considerable discussion, it was agreed that the objective of the committee should be to develop "Guidelines for Maintenance Painting" with priorities assigned as follows:

(1) Techniques for repainting—surface preparation (repair and feathering of edges) and application.

(2) Determination of when to repaint.

(3) Selecting the coating system—same or a new paint system.

(4) Identifying the existing coating—its history and present condition.

(5) Inspection

Task Groups were then formed to investigate the first two points, namely: *Repainting Techniques*—Collect information on techniques used to prepare surfaces and apply generic types of coatings; and *When to Repaint*—Develop a decision tree to achieve a logical decision as to when to repaint.

LEGAL ASPECTS OF SPECIFICATIONS

Forrest M. Couch—Chairman

Charles Peshek—Secretary

The purpose of this meeting was to make recommendations for the formation of a select committee for the purpose of reviewing SSPC procedures for the development of specifications and guides in order to avoid any possibility of a legal or government action. These precautions should avoid any possibility of any action such as the Hydrolevel/ASME case.

The following recommendations were made based on procedures used by ASTM which has been very careful to avoid any such possibilities:

Structure of Committees:

(1) Classify all members as to their interest, e.g.:

(a) Producers—coatings manufacturers, raw material suppliers abrasives mfrs, equipment mfrs.

(b) Users—owners, contractors.

(c) General interest—government agencies (they may be users), consultants, testing laboratories.

(2) Establish guidelines as to how balloting should be done, e.g.:

(a) Who should vote—everyone, one per company?

(b) Balance between producers, users and general interest. Usually the latter two should be higher than the producers.

(c) Minimum number of returned ballots to make the balloting valid, e.g., 60%.

(d) Negative votes—an explanation in writing is mandatory.

(e) Resolution of negatives, e.g., 90% of those present.

(f) Record keeping of all balloting and resolution of negatives.

Evaluation of Proprietary Products: (a) Do so only under code; and (b) Require a written release by suppliers.

Certification or QPL Testing:—Avoid this. An associated developing specifications should not be testing them.

SSPC Disclaimer: It is satisfactory as presently used.

Liability Insurance: It is not necessary.

The ASTM procedure should be reviewed carefully in order to take advantage of their experience.

The following committee was formed to supervise this review: Forrest M. Couch; Sidney B. Levinson; Loren B. Odell; William C. Spangenberg; and William J. Wallace, Jr.

REGULATIONS

William C. Johnson—Co-chairman
Lee K. Schwab—Co-chairman
Helen Moore—Acting Secretary

Regulatory status and trends were reviewed.

Sandblasting requires an enclosure but regulation reinforcement is variable in different areas. SSPC Painting Manual, Vol. II provides guidance on blasting and on safety precautions.

Lead and *Chromates* are restricted both in paint formulations and as air contaminants during paint removal.

Volatile Organic Content (VOC) of paint formulations is undergoing hearings by the California Air Resources Board (CARB) with the possibility of assigning different maximum levels to light duty and heavy duty coatings. There also is a trend toward the use of high solids (85%) and water-based coatings for shop painting. The SSPC report on "Low Solvent Coatings" dated September 1, 1982 gives an industry consensus of current and developmental modes of compliance.

Urethane coating use on shipboard is being withheld in accordance with Navy publication 631.

TBTO, *TBTF* (Tributyl tin oxide and tributyl tin fluoride), used in Navy anti-fouling paints, have presented serious regional disposal problems. NAVSEA and Naval R&D Labs (Annapolis) were suggested as source of data on acceptable sanitary landfills.

Solvent-Borne paints are listed in Class I in some regions and cannot be buried in land-fills.

Methyl and Ethyl Cellulose solvents have been found to show bad reproductive effects in rats. A California OSHA alert has

been issued and maximum limits have been greatly reduced. NPCA has informed their members and recommends the replacement of these solvents.

Coal Tar contains carcinogenic polynuclear aromatics and should be avoided.

The committee will continue to act as a source for updating the status of government regulations to the extent that they effect members of SSPC. However, it was recommended that this be done automatically on a continuing basis rather than waiting until the committee meets.

NEW SSPC PUBLICATIONS

Harold Hower—Chairman and Acting Secretary

The following publications are either available now or within a month: *Painting Manual—Volumes I and II*; *Low Solvent Coatings for Structural Steel*; and *Survey of New and Existing Methods of Surface Preparation*.

SSPC plans to publish a *Surface Preparation Manual* plus a series of monographs. Suggestions for the monographs include: *Inspection of surface preparation*; and *Cleaning steel after abrasive air blasting*.

A standing publications committee to review manuscripts is recommended. The following are members who volunteered or were nominated to serve on the committee: Benjamin S. Fultz; Sidney Lauren; John S. Moore; Jack Oeschle; John W. Peart; and Victor H. Thompson, Jr.

SIDNEY B. LEVINSON,
Delegate

BY-LAWS COMMITTEE

Amendment to By-Laws and Standing Rules

The following amendment to the By-Laws was given first reading on November 2. It will be presented for adoption at the Board of Directors meeting of May 20, 1983.

Article IV—Nominations and Elections

A. (2) NOMINATIONS

WHEREAS the Baltimore Society has requested that nominations for Federation elective officers be permitted from the floor at the Spring Board of Directors meeting, be it

RESOLVED that By-Laws Article IV, Section A, Paragraph (2) be amended as follows:

"(2) The report of the Nominating Committee shall be announced at the Spring Board of Directors meeting. Nominations for any elective office may also be made from the floor by an Society Representative at the Spring meeting, or by a petition signed by 25 Active members and forwarded to the Federation Executive Vice-President in time for publication in the August JOURNAL OF COATINGS TECHNOLOGY, in which the slate of nominees shall be published. The Federation Executive Vice-President shall place such nominees-by-petition in nomination at the Fall meeting of the Federation Board.

"(3) Nominations for any elective office may also be made from the floor by any Society Representative at the Fall Board Meeting, prior to the election of Officers."

Comment: The By-Laws Committee recommends adoption.

Abstracts of Papers in This Issue

REACTIVITY OF CALCIUM CARBONATE AND COPROUS OXIDE WITH BINDER ACID COMPONENTS IN ANTIFOULING PAINTS—C.A. Giudice, et al.

Journal of Coatings Technology, 55, No. 697, 23 (Feb. 1983)

The acid components of WW rosin will react with the basic pigments during both manufacture and storage. The degree to which the mentioned reactions had advanced were determined by evaluating the residual acidity of the binder, which is due to the uncombined rosin.

The results obtained show that it is possible to prepare paints with matrices of the same solubility starting from different rosin/plasticizer ratios. Conversely, matrices of different solubility can be obtained from similar binder compositions. Type and content of pigment have a marked influence on the above mentioned behavior.

SOLUBILITY PARAMETER CONCEPT IN THE DESIGN OF POLYMERS FOR HIGH PERFORMANCE COATINGS—A.J. Tortorello and M.A. Kinsella

Journal of Coatings Technology, 55, No. 697, 29 (Feb. 1983)

The design of synthetic resins to function as vehicles for prospective aqueous aircraft coatings began with the characterization of the solubility parameter value of a number of test fluids. Using the method of atomic group contributions, urethane copolymers having the desired solubility parameter values were synthesized in organic medium. These solution polymers were then dispersed into aqueous medium as both anionic and cationic dispersions, formulated with film aids and additives, and evaluated for clear-film performance.

Performance was as expected for all test fluids except water and the onset of resistance to any test fluid was predicted.

ANTICORROSIVE EFFECT OF SOME β -DIKETONES IN POLYMERIC COATINGS ON LOW CARBON STEEL—T.T. Kam and P.K. Hon

Journal of Coatings Technology, 55, No. 697, 39 (Feb. 1983)

Corrosion protection of polymeric coatings on carbon steel using a methyl/n-butyl methacrylate copolymer resin, can be enhanced with Fe(III) β -diketonate complexes applied first to the low carbon steel surface. The iron β -diketonate complexes were formed by reaction of β -diketone complexing agents with iron at the surface. An

increase in corrosion resistance was demonstrated by several corrosion tests and the extent of the increase was found to be related to the type of complexes developed.

ACID CATALYZED CURING POLYESTER/MELAMINE RESINS WITH LATENT CATALYSTS—W.J. Mijs, W.J. Muizebelt, and J.B. Reesink

Journal of Coatings Technology, 55, No. 697, 45 (Feb. 1983)

In this paper, new latent catalysts (oximeesters of sulphonic acids) for the acid catalyzed curing of polyester/melamine resins are introduced. Mechanism and kinetics of the thermolysis reaction of these sulphonic acid esters, during the curing process have been elucidated.

The curing process was followed by monitoring changes in physical and chemical parameters of the resin system. Four methods were used: Fourier infrared spectroscopy recording emission and transmission spectra of the curing film; thermogravimetry; dynamic spring analysis determining Tg and loss tangent $\tan \delta$; and, finally, the "sag balance" viscosimeter.

Tests have been done to connect fundamental physical properties with end use properties of cured paint films to compare various catalysts. The test results indicate that crosslinking performance of the oxime-blocked sulphonic acid catalysts is similar to that of the usual amine/sulphonic acid salt catalysts. Low conductivity of the sulphonic acid oximeester resin systems, however, offers potential for electrostatic spraying. Furthermore, less absorption to some pigments was found with these catalysts compared to amine sulphonic acid salts.

INTERRELATIONSHIPS BETWEEN PIGMENT SURFACE ENERGIES AND PIGMENT DISPERSIONS IN POLYMER SOLUTIONS—G. Dale Cheever and J.C. Ulicny

Journal of Coatings Technology, 55, No. 697, 53 (Feb. 1983)

In the manufacture of paints, plastics, elastomers, and adhesives, good dispersion of pigments and other fillers is essential in producing a quality product. Although many theoretical examples exist in the literature describing the factors required for dispersion, a practical method is needed to measure these pigment and solution properties and relate them to dispersion.

The critical surface tension of wetting (γ_c) of 18 commercial pigments representing hiding, extender, colored, white, organic, and inorganic types were measured with a capillary flow technique. A relationship was found between pigment dispersion and the γ_c values of eight representative pigments and the surface tension of an aqueous polyvinyl alcohol solution.

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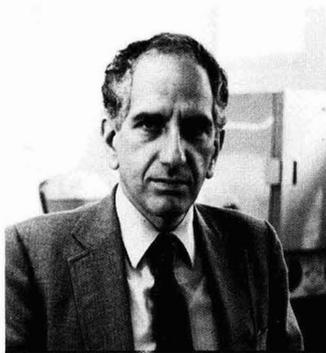


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Government and Industry

Battelle Indicates U.S. R&D Funding Will Surpass \$83 Billion This Year

Expenditures in calendar year 1983 for research and development in the United States are expected to reach \$83.6 billion, according to the annual forecast of Battelle's Columbus Division. This represents an increase of \$6.3 billion (8.2%) over the \$77.3 billion that the National Science Foundation estimates was to be actually spent for R&D in 1982.

While most of the increase will be absorbed by continued inflation (estimated at 4.5% for R&D in 1983), Battelle forecasts a real increase in R&D expenditures of 3.5%. This is slightly higher than the nine-year average rate of 3.3% in real R&D effort that has been experienced since 1973.

Industrial funding for R&D is projected to be more than government support. Industrial support is forecast to be \$41.4 billion, up 7.6% from 1982. This will account for 49.6% of the total R&D funding. Battelle sees an increase of 8.8% in federal support for R&D, with funding expected to be \$39.3 billion. This is 47.0% of the total R&D expenditures for 1983. Funding by academic institutions is expected to be \$1.8 billion (2.1% of the total), and other nonprofit organizations will provide \$1.1 billion (1.3%).

The Battelle report indicates industry will remain as the dominant performer of R&D. In 1983, performance of R&D by industry is expected to rise to \$60.7 billion, or 72.5% of all research performed. This compares with \$10.8 billion (13.0%) for the federal government, \$9.7 billion (11.6%) for academic institutions, and \$2.4 billion (2.9%) for other nonprofit organizations.

The Battelle forecast notes that federal funding supports research performance in all four sectors. Currently, about one-fourth goes to support R&D conducted by the government itself; almost half goes to industry; approximately one-fifth goes to colleges and universities; and the rest, about one-twentieth, goes to other nonprofits.

Industry absorbs almost all of its own funds, either performing the R&D itself or contracting with other industrial performers. Its contracts and grants to nonprofit organizations slightly exceed those to colleges and universities. The nonprofit organizations finance both themselves and the academic institutions about equally, and colleges and universities use up all the funds they originate.

Four government agencies dominate the federal R&D scene and are expected

to account for 91.2% of total federal R&D funding in 1983. These are the Department of Defense, 56.6%; the National Aeronautics and Space Administration, 14.9%; the Department of Energy, 10.4% and Health and Human Services, 9.3%.

The forecast notes that increases in defense spending primarily is directed toward the acquisition of major weapons systems and the R&D that will be necessary to support them. Additionally, it is noted that budgets also increased for space and general science programs, despite appearances during 1982 that only defense appropriations would increase. Battelle foresees continued small-scale increases in these major fields, but declines in energy R&D.

The success of the space shuttle program and the potential use of the shuttle as an instrument for economical orbital insertion and repair of satellites is seen as justifying continued support for space R&D, the forecast says.

However, funding will continue to decline for energy research since there is little evidence that energy will be perceived as a national problem requiring massive, federal R&D support. Energy projects involving short-term or low-risk R&D likely will be financed by industry.

The report also anticipates that R&D dollars will continue to support the biological sciences areas. However, less emphasis will be placed on "soft" sciences, except those environmental, ecological, and socioeconomic impact investigations presently mandated by law.

Industrial support of research is growing in fields related to electronics, communications, advanced machinery, and in those fields most directly influenced by the need for more energy-efficient products and processes. R&D will be heavily self-funded in the manufacturing industries, where on the average, only 32.0% of the total will be supported by the federal government. The nonmanufacturing industries do relatively little R&D, and support for this activity will be divided almost equally between the federal government and industry.

As part of the forecast, Battelle estimated the industrial versus federal support for R&D performed by several broad industrial sectors. In 1983, Battelle expects the electrical machinery and communications industry to be the leader in total R&D, with funding of more than \$12.8 billion. Of that, 59.1% will be industrially funded.

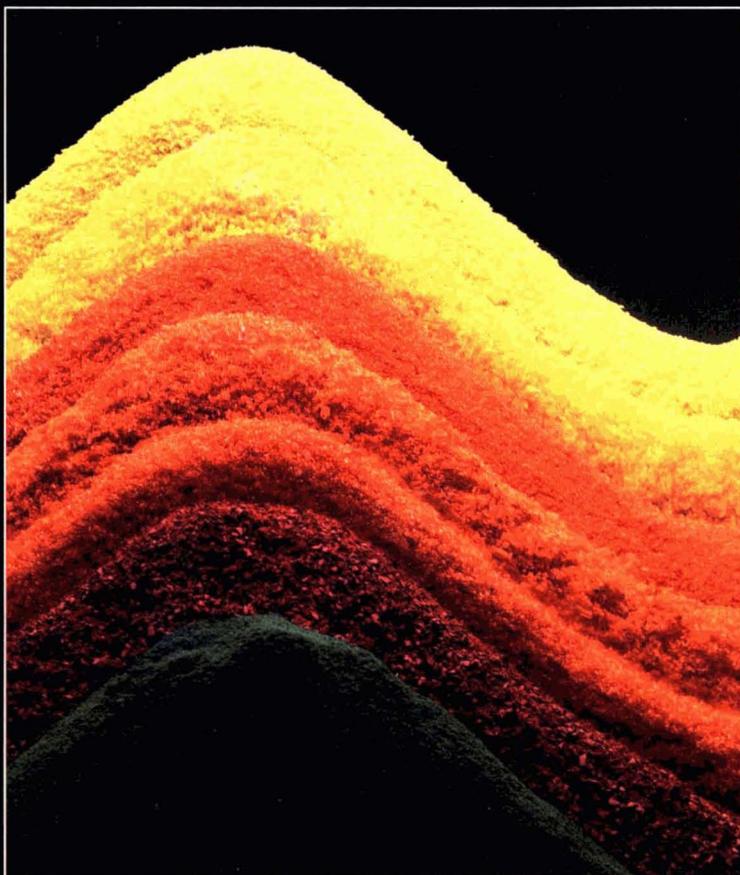
The aerospace industry is forecast to have the second largest total R&D support with \$11.8 billion. Of that, 20.3% will be industrially funded.

Other industrial sectors Battelle estimates will receive more than \$1 billion in R&D funds include: Machinery—\$9.1 billion, 83.3% of which will be industrially funded; Autos, truck and parts, and other transportation equipment—\$7.2 billion, 85.6% of which will be industrially funded; Chemicals—\$6.8 billion, 90.0% of which will be industrially funded; Professional and scientific instruments—\$3.4 billion, 93.6% of which will be industrially funded; and Petroleum products—\$2.5 billion, 87.5% of which will be industrially funded.

The Battelle forecast indicates that industry is taking over short-term R&D projects and is reacting to the growing pressure from foreign technological competition. However, it cautions that a depressed business environment may lead to reductions in future industrial R&D support. This is because corporate R&D decision-makers generally tend to lag R&D commitments about one-year behind cash flows. Thus, poor business conditions in the recent past may tend to offset real increases in funding.

The Battelle forecast also compares the four performing sectors in terms of their relative costs of R&D. During 1983, the overall cost increase for all R&D is estimated to be 4.5%. By sectors, the increases are estimated as government, 8.0%; industry, 4.2%; colleges and universities, 2.2%; and other nonprofits, 4.8%. From 1972-1983, costs of all R&D, as an average, are estimated to have risen by 111.14%. Increases in the individual performing sectors—over this same time period—are expected to be: federal government, 122.74%; industry, 108.12%; colleges and universities, 122.25%; and other nonprofits, 97.18%.

As part of the forecast, Battelle also examined R&D trends during the past and identified what impact they may have on the future. The report concludes that over the past few years, federal support has tended to shift toward more "development" and less basic and applied "research." If such trends continue, the long-range consequences may pose significant problems in terms of the science base upon which economic vitality grows. Such problems may, in part, be mitigated by a continuation of the recent resurgence of basic research support by industry.



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New England, Northwestern, Southern Societies And Birmingham Club Share MMA Awards for Achievements

The 1982 MMA Awards for notable achievements by Constituent Societies of the Federation were won by the Southern, New England, and Northwestern Societies, and the Birmingham Club. Presentation of the Awards was made at the recent Federation Annual Meeting in Washington, D.C.

Established in 1975 by Materials Marketing Associates, a national marketing group of manufacturer's representatives, the Awards recognize notable achievements by Constituent Societies (excluding those Society papers presented at the Federation Annual Meeting).

There are three categories of Awards, based on Society membership size.

Each winning Society receives \$350 in cash (divided equally in case of tie) plus a handsome plaque, appropriately engraved. New England and Northwestern tied for the Award for their category.

Consumer Guide To Trade Paint Quality

The Southern Society was cited for its work in producing the brochure, *Consumer Guide to Trade Paint Quality*, "Know Paint Quality Before You Buy." The guide, which is designed for point-of-purchase use to help the consumer understand what to look for in paints, focuses on latex interior flat paint and depicts the performance characteristics of low, average, and high-quality products.

Full-color reproductions illustrate the effects of paint quality features on stain-

ing and washability-durability, as well as spreading rate, application, and wet and dry hiding.

The brochure has been distributed to a large number of paint store dealers, and has received promotional support from the National Decorating Products Association which represents the dealers.

Industry Contributions Through Coatings Tech Expo, And Annual Symposium

The New England Society and Northwestern Society tied for the Award in their category; New England was recognized for its contribution to the coatings industry derived from the 1982 Coatings Tech Expo hosted by the Society; Northwestern won for the excellence of its Annual Symposium.

The New England event featured a technical program and exhibits of raw material suppliers, and attracted personnel from the rubber, plastics, and ink industries, as well as from coatings. Technical sessions featured discussions on dispersion, waste management, advances in formulation, and uses of computers.

The Northwestern symposium, whose topic was application equipment for compliance coatings, drew a record turnout of 127 persons, including attendees from outside the Society area, and featured technical papers and equipment exhibits. Speakers discussed electrostatic spray gun, atomizer and robot applications for coatings.

A/V Program Promoting Coatings Careers

The Birmingham Paint, Varnish, and Lacquer Club was cited for its production of an audio-visual program, "Introduction to the Paint Industry," which is aimed at encouraging students to consider coatings careers.

Club members have shown the program to student groups and school guidance counsellors, as well as at the recent Oil and Colour Chemists Exhibition. The program has also been used in conjunction with student visits to coatings manufacturing facilities, sponsored by the Club.

Principles Governing Awards

The MMA Awards are given in recognition of notable achievements in the field of education, manufacturing and training procedures, technology, public service, and other achievements deemed proper and desirable by the Awards Committee.

Not eligible are Society papers offered for presentation at the Federation Annual Meeting. Although the Awards are to be presented at the Annual Meeting, it is not mandatory that they be presented to any or all categories each year.

The President of any Society wishing to enter the competition must send a letter of intent, no later than March 31, to the MMA Awards Committee Chairman (Al Heitkamp, Cargill, Inc., P.O. Box 9300, Minneapolis, MN 55440).

A complete description of the Society activity to be considered in the 1983 competition must be submitted by the Society President to Chairman Heitkamp by July 31.

The members of Materials Marketing Associates who sponsored the 1982 MMA Awards were the following: Apco Industries Co., Ltd., Toronto, Ont.; George C. Brandt, Inc., Kansas City, KS and St. Paul, MN; Cordano Chemical Co., Inc., Portland, OR; E. T. Horn Co., Los Angeles, CA; Lukens Chemical Co., Boston, MA; Matteson-Ridolfi, Inc., Detroit, MI; McCullough & Benton, Inc., Atlanta, GA; McNalley & Webber Co., Cleveland, OH; Ribelin Distributors, Inc., Dallas, TX; Wm. B. Tabler Co., Inc., Louisville, KY; Van Horn, Metz & Co., Inc., Philadelphia, PA; Walsh & Associates, Inc., St. Louis, MO; and C. Withington Co., Pelham Manor, NY and Rio Piedras, Puerto Rico.

Theme Announced for 1983 Annual Meeting; Papers Invited for Event, To Be Held Oct. 12-14

The 1983 Annual Meeting of the Federation of Societies for Coatings Technology will have as its theme, "Knowledge Applied Profitably," it was announced by Program Chairman Peter Hiscocks, of C-I-L Paints, Inc., Toronto.

The Annual Meeting will be held in conjunction with the Paint Industries' Show at Place Bonaventure, Montreal, Quebec, on October 12-14.

The theme recognizes that a major current challenge is gaining access to, and disseminating, the ever-growing store of

technical knowledge. The program will focus on addressing the issue of access and understanding of this information from the basics (no longer simplistic) to the cutting edge of technology.

Prospective speakers are invited to present original papers on the many and varied aspects of the theme, and are requested to submit abstracts for review to Program Chairman Peter Hiscocks, c/o Federation of Societies for Coatings Technology, 1315 Walnut St., Suite 832, Philadelphia, PA 19107. *Deadline for receipt of abstracts is March 1.*

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by March 1, 1983; manuscript copies must be submitted by June 1, 1983.

The Awards will be presented during the 1983 Federation Annual Meeting, October 12-14, in Montreal, Que., Canada.

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PAINT/COATINGS DICTIONARY

The technical terms and jargon of the coatings industry and its interfacing technologies are defined in this comprehensive work which contains more than 5500 entries compiled by the Definitions Committee of the Federation of Societies for Coatings Technology. Over 4500 terms are defined, and more than 1000 additional entries are synonyms cross-referenced to the defined terms.

The broad scope of terms included reflects the obvious usefulness of the dictionary to a wide audience, ranging from the layman, to artists and artisans, to

technicians in all the coatings-related fields.

A unique feature is classification of terms into one or more of 73 categories, which have been number coded and appear as superscripts following each definition. The terms are listed in their appropriate categories, making up a thesaurus which comprises the second section of the dictionary.

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Reactivity of Calcium Carbonate And Cuprous Oxide with Binder Acid Components in Antifouling Paints

C.A. Giudice, B. del Amo, V. Rascio, and R. Sanchez
CIDEPINT—Research and Development Center for Paint Technology*

Soluble matrix antifouling paints were formulated with sea water soluble resins such as WW rosin (gum rosin). The influence of the composition variables (rosin content, phenolic varnish used as plasticizer, cuprous oxide, and calcium carbonate levels), the preparation variables (dispersion time), and the period of storage were studied. The present research involved the preparation of 24 samples, which were tested in an experimental raft.

The acid components of WW rosin will react with the basic pigments during both manufacture and storage. The degree to which the mentioned reactions had advanced were determined by evaluating the residual acidity of the binder, which is due to the uncombined rosin.

The results obtained show that it is possible to prepare paints with matrices of the same solubility starting from different rosin/plasticizer ratios. Conversely, matrices of different solubility can be obtained from similar binder compositions. Type and content of pigment have a marked influence on the above mentioned behavior.

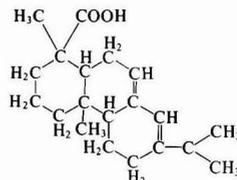
Raft trials served to establish the different bioactivity levels obtained with paints prepared with three and 12 hours of cuprous oxide grinding. Longer grinding time resulted in lesser toxicity. During storage, paints also showed some reduction in toxic action due to the reactions between binder and calcium and cupric ions present in the paint.

INTRODUCTION

Efficient antifouling paints have been formulated with toxicants which are leached from the film surface in contact with sea water. Thus, it is possible to control fouling attachment for various periods. In previous works^{1,2,3} satisfactory results were achieved in service trials during 12, 24, and 36 months.

In soluble matrix antifouling paints, the dissolution rate is the most important variable and has significant influence on film bioactivity. These paints are based on an acid resin which is soluble in sea water (pH 8.0–8.2) by reaction with sodium and potassium ions, forming alkaline resinates.

WW rosin has been used for binder preparation since the end of the last century. The main component of this resin is abietic acid, which has the following formula:



The molecule of abietic acid has two carbon-carbon (—C=C—) double bonds and one carboxylic group (—COOH). Its instability is due to the former, while the latter is responsible for the reaction with alkaline ions, giving the mentioned soluble compounds, or with calcium and magnesium ions, forming resinates of lesser solubility in sea water than the original rosin resin. As a consequence, film solubility decreases.

The mentioned reactions can be produced during paint manufacture when the binder is in contact with some toxicants such as cuprous oxide, or extenders, such as calcium carbonate. These reactions were studied in previous papers^{4,5,6} and it was concluded that it is very important to determine the degree of reaction during both grinding and in the final storage of the product and to establish its influence on the antifouling paint efficiency in service.

EXPERIMENTAL

Two series of 12 antifouling paints were prepared. The following variables were studied:

*52 entre 121 y 122, 1900 La Plata, Argentina.

Table 1—Composition of the Antifouling Paints Tested (g/100 g)

	Paint Identification											
	1	2	3	4	5	6	7	8	9	10	11	12
Cuprous oxide	46.0	27.0	14.0	46.0	27.0	14.0	46.0	27.0	14.0	46.0	27.0	14.0
Calcium carbonate	—	19.0	32.0	—	19.0	32.0	—	19.0	32.0	—	19.0	32.0
Additives	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0
WW rosin	18.0	18.0	18.0	19.8	19.8	19.8	21.6	21.6	21.6	24.0	24.0	24.0
Plasticizer	9.0	9.0	9.0	6.6	6.6	6.6	5.4	5.4	5.4	4.8	4.8	4.8
Solvents and thinners	23.0	23.0	23.0	23.6	23.6	23.6	23.0	23.0	23.0	21.2	21.2	21.2
WW rosin/plasticizer ratio (by weight)	2:1	2:1	2:1	3:1	3:1	3:1	4:1	4:1	4:1	5:1	5:1	5:1

Influence of the Formulation Variables

The resin used was WW rosin (gum rosin). Solubility of the matrix in sea water was regulated by the use of a plasticizer. A phenolic varnish was employed as co-binder with this objective: the film obtained had good flexibility and adhesion.

Four WW rosin/phenolic varnish ratios were used to achieve that purpose: 2:1, 3:1, 4:1, and 5:1 ratios by weight. Acidity of the binder and sea water solubility increased with increasing rosin content.

Calcium carbonate incorporation produced a reduction in binder acidity due to calcium resinate formation. This rosin soap is not as brittle as the rosin itself and has a lower dissolution rate in sea water.^{7,8,9} This reaction was evaluated by determining the acidity of the binder after the incorporation of different quantities of calcium carbonate in the samples (Table 1).

The toxicant employed (cuprous oxide) produces cupric ions by dismutation during the dispersion process. These ions (Cu²⁺) react with the acid components forming an insoluble copper resinate and modifying film dissolution rate.¹⁰

Table 1 shows that the paints were prepared with three different toxic pigment contents (46, 27, and 14% of cuprous oxide) for each rosin/phenolic varnish ratio.

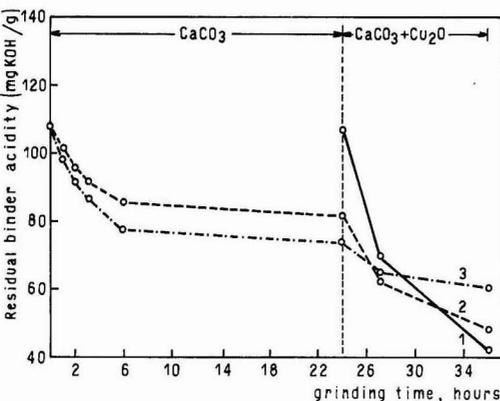


Figure 1—Residual binder acidity during pigment dispersion for paints 1, 2 and 3, WW rosin/plasticizer ratio 2:1 in weight

Influence of Paper Preparation Variables

Operative variables which affect pigment dispersion in ball mills of different capacity was another parameter studied previously.¹¹ In this work, porcelain ball mills of 3.3 litres capacity were used for pigment dispersion.

Paint was prepared by first incorporating calcium carbonate in the vehicle. This extender was milled for 24 hours and then cuprous oxide was added to the jar and dispersed for three hours in the first series of paints and for 12 hours in the second series.

In all cases, binder acidity was determined when milling was started and again after 1, 2, 3, 6, and 24 hours of calcium carbonate dispersion. Acidity was also evaluated after three and 12 hours of cuprous oxide incorporation.

To determine binder acidity, pigments and insoluble resins were removed by centrifugation. To neutralize resinic acids, an alcoholic potassium hydroxide solution was added in excess to a portion of the liquid. After 24 hours of reaction, the solution was back-titrated with hydrochloric acid, using phenolphthalein as an indicator.

Binder acidity determined during pigment dispersion is shown in Figures 1–4. The uncombined rosin content during the preparation process is indicated in Table 2, while Table 3 shows cupric and calcium resinsates, stoichiometrically calculated.

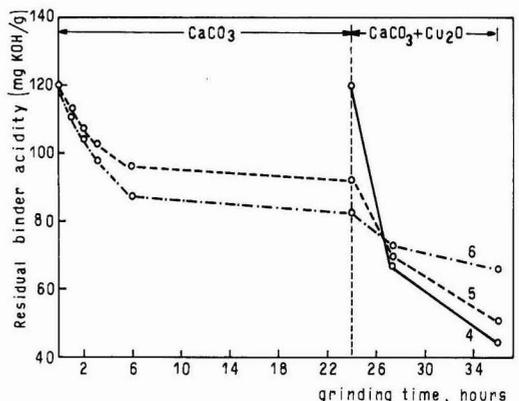


Figure 2—Residual binder acidity during pigment dispersion for paints 4, 5 and 6, WW rosin/plasticizer ratio 3:1 in weight

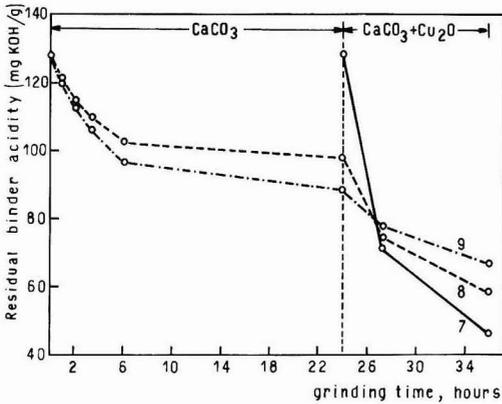


Figure 3—Residual binder acidity during pigment dispersion for paints 7, 8 and 9, WW rosin/plasticizer ratio 4:1 in weight

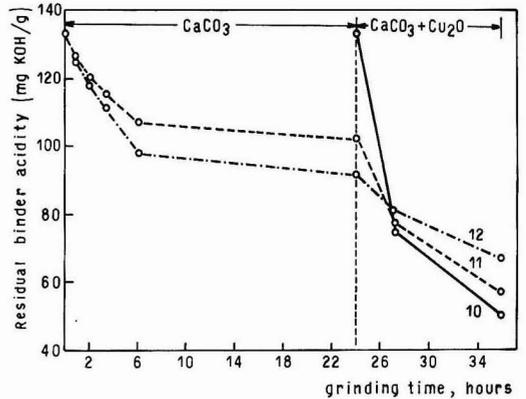


Figure 4—Residual binder acidity during pigment dispersion for paints 10, 11 and 12, WW rosin/plasticizer ratio 5:1 in weight

Influence of Storage Time

The above mentioned reactions can continue during paint storage. If these reactions occur to a significant extent, paint bioactivity could be modified, decreasing its toxic efficiency.

To establish the influence of this variable, four samples were stored (paints 2, 5, 8, and 11). These samples were formulated with the same toxicant content (27%) and four different rosin/phenolic varnish ratios (2:1, 3:1, 4:1, and 5:1 by weight, respectively).

Storage time was of 25 months for all the aforementioned samples, and this time was extended to 50 months in samples 5 and 11.

Binder acidity of the aged samples was evaluated and results were presented in Table 4. Uncombined rosin content and quantities of metallic resins formed in this stage were calculated as cupric resinate (Tables 5 and 6).

RESULTS

Laboratory Tests

A high uncombined rosin content in the binder leads to a high dissolution rate of the film.

A solution of WW rosin in a neutralized solvent mixture (toluene/white spirit 1:1) has an acid value of 160. The phenolic varnish used as plasticizer was previously neutralized with calcium oxide in a ball mill. In this form the phenolic varnish employed had a negligible acid value.

The different vehicle mixtures had the following values:

Rosin/phenolic varnish ratio, by weight	Acid value, mg KOH/g
2/1	106.8
3/1	120.1
4/1	128.1
5/1	133.4

These results are consistent with the WW rosin content in each mixture.

Table 2—Uncombined WW Rosin Content During Paint Manufacture (g/100 g)

Paint	Initial Value	24 Hours of Grinding (CaCO ₃)	3 Hours of Grinding (CaCO ₃ + Cu ₂ O)	12 Hours of Grinding (CaCO ₃ + Cu ₂ O)
1	18.0	—	10.2	7.0
2	18.0	13.8	10.5	8.1
3	18.0	12.4	10.9	10.2
4	19.8	—	11.1	7.4
5	19.8	15.2	11.5	8.3
6	19.8	13.6	12.0	10.9
7	21.6	—	11.9	7.8
8	21.6	16.6	12.5	9.9
9	21.6	14.9	13.1	11.3
10	24.0	—	13.5	9.0
11	24.0	18.4	13.9	10.3
12	24.0	16.5	14.6	12.1

Table 3—Formation of Calcium and Cupric Resinates During Paint Manufacture (g/100 g)

Paint	3 Hours of Cuprous Oxide Dispersion		12 Hours of Cuprous Oxide Dispersion	
	Calcium Resinate	Cupric Resinate	Calcium Resinate	Cupric Resinate
1	—	7.3	—	10.3
2	3.8	3.1	3.8	5.4
3	6.0	1.4	6.1	2.1
4	—	8.2	—	10.7
5	4.9	3.4	4.9	6.5
6	5.6	1.5	5.7	2.5
7	—	9.0	—	13.0
8	4.5	3.8	4.6	6.3
9	6.1	1.6	6.3	3.4
10	—	9.8	—	14.1
11	5.1	4.2	5.2	7.6
12	6.8	1.8	7.0	4.1

Table 4—Acidity of Binder in Aged Paints After Storage (mg KOH/g)

Paint	Initial Value	25 Months	50 Months
2	62.2	59.8	—
5	69.7	65.4	62.4
8	74.1	65.8	—
11	77.2	66.6	63.3

The above mentioned values and those obtained during pigment grinding are represented in *Figures 1 to 4*. Analysis of the data obtained shows that the addition of calcium carbonate produced a binder acidity reduction. This reduction occurred principally during the first hours of grinding; subsequently, no significant reduction was obtained, and values determined after 24 hours were not significantly lower than those obtained after six hours.

After the completion of the grinding process, a similar consumption of potassium hydroxide for different initial content of rosin in the binder was observed. This is due to the neutralization of the calcium carbonate incorporated by reaction with binder acid components. For example, in paints 2 and 6 (19 and 22% of calcium carbonate, respectively), with starting values of 106.8 and 120.1 mg/g, the final values were 81.8 and 82.4 mg/g.

At the end of the calcium carbonate dispersion, cuprous oxide was incorporated and acidity was evaluated after three and 12 hours of grinding.

Cuprous oxide contributes to establish clear differences in binder acidity, because the binder reacts with the cupric ions (Cu^{2+}) present in the medium, generated by dismutation and oxidation of cuprous ions.

Experimental values obtained for 24 hours of extender dispersion and the corresponding to 3 and 12 hours for cuprous oxide are indicated in *Table 7*.

As was observed during extender dispersion, increased cuprous oxide content in the paint led to increased reaction with the resinic acids. This is demonstrated by means of the determination of residual acidity. For example, in paints 10 and 12 (46 and 14% of cuprous oxide), the initial acidity is 133.3 mg KOH/g. After three hours of dispersion the values obtained were 74.8 and 81.1 mg KOH/g, respectively.

The increase of cuprous oxide grinding time seems to be a variable that is very important in the formation of cupric resinate. Paint film solubility diminishes by reduction of the quantity of uncombined rosin. In sample 2, with three and 12 hours of grinding, final values of 23.9 and 41.3 mg KOH/g were obtained.

Table 6—Formation of Metallic Resinates In Aged Paints After Storage (g cupric resinate/100 g)

Paint	Initial Value	25 Months	50 Months
2	0.0	0.4	—
5	0.0	0.8	1.3
8	0.0	1.8	—
11	0.0	2.7	3.6

Table 5—Content of Uncombined Rosin In Aged Paints After Storage (g/100 g)

Paint	Initial Value	25 Months	50 Months
2	10.5	10.1	—
5	11.5	10.8	10.3
8	12.5	11.1	—
11	13.9	12.0	11.4

The results show that products formulated with similar levels of rosin, but pigmented with different quantities of calcium carbonate and cuprous oxide, led to paints of different film solubility. In samples 7 and 9 (21.6% of initial rosin content) the final value for three hours of toxicant dispersion of free acid determined was 11.9 and 13.1 g/100 g, respectively (*Table 2*). Furthermore, a similar final rate can be achieved starting from binders of different resin content. It occurs in samples 3 and 11 (18 and 24% of initial resin content), where the values determined were 10.2 and 10.3%, respectively, for 12 hours of toxicant grinding.

Considering the aging produced during paint storage, the reduction of acid resin content in samples prepared with three hours of grinding of cuprous oxide did not have the same effect as that established during paint preparation (*Tables 4 and 5*). In sample 5, the decrease was 41.9% during manufacture and 10.4% after 50 months storage. In this case, the reduction of rosin content is also due to the formation of cupric and calcium resinates (*Table 6*).

RAFT TRIALS

In order to obtain a correlation between laboratory experimental tests and a paint's bioactivity in service, painted panels were placed in Puerto Belgrano raft (38°58' S, 62°06' W). Hydrological and biological characteristics of the area have been studied by Bastida and collaborators.^{12,13,14}

Sandblasted steel panels were employed and two coats of the antifouling paints (80 μm thickness) were applied over the anticorrosive system. Panels were immersed in sea water 24 hours after the application of the last antifouling coat.

Table 7—Mg KOH/g of Binder, for Different Periods of Dispersion

Paint	24 hours, CaCO_3	3 hours, Cu_2O	12 hours, Cu_2O
1	—	60.9	41.8
2	81.8	62.2	48.0
3	73.9	64.5	60.4
4	—	67.3	44.8
5	91.8	69.7	50.3
6	82.4	72.7	66.1
7	—	70.8	46.2
8	98.4	74.1	58.7
9	88.3	77.6	67.0
10	—	74.8	50.0
11	102.2	77.2	57.2
12	91.7	81.1	67.2

Table 8—Fouling Settlement^a

	Paint Identification											
	1	2	3	4	5	6	7	8	9	10	11	12
Samples prepared with 3 hours of cuprous oxide grinding:												
8 months	0	0	0	0-1	0	0-1	0-1	0-1	0	0	0	0
11 months	0-1	0-1	1-2	0-1	0-1	1-2	0-1	0-1	0-1	0	0	0
17 months	0-1	1	2-3	0-1	1	2	0-1	0-1	1	0	0	0
21 months	1	1	3	1	1	2-3	0-1	0-1	1-2	0	0	0
Samples prepared with 12 hours of cuprous oxide grinding:												
8 months	1-2	2-3	1	1	2	2	1-2	1	1	1-2	1-2	1-2
11 months	1-2	3	2	2-3	2-3	2-3	2	2	2	2	1-2	1-2
17 months	3	3-4	3-4	3-4	3	2-3	3	2-3	2	2-3	1-2	1-2
21 months	5	4-5	3-4	5	4-5	3	4-5	3	2-3	3	2-3	2-3
Samples prepared with 3 hours of cuprous oxide grinding and stored for 25 months:												
8 months	0-1				0			0-1			0	
11 months	1				0-1			0-1			0-1	
17 months	2				0-1			1			1	
21 months	2				1-2			1-2			1-2	
Samples prepared with 3 hours of cuprous oxide grinding and stored for 50 months:												
8 months					0						0	
11 months					0-1						0-1	
17 months					1						1-2	
21 months					1-2						2	

(a) Key to the table: 0 (panel without fouling); 0-1 (very rare or very little); 1 (rare or little); 2 (common); 3 (very common); 4 (abundant); 5 (completely fouled).

Panels were observed after 8, 11, 17, and 21 months of immersion and the records of fouling corresponding to those observations are presented in Table 8. A photographic control was made in order to standardize visual observations.

The scale of fouling settlement employed is mentioned in Table 8. The value 1 (rare or little) was established by the authors as an acceptable limit of performance for an antifouling paint of good bioactivity. Over this value, paints were discarded.

According to the records obtained, it is possible to establish a very important difference in bioactivity of the paints of the two series prepared with three and 12 hours of cuprous oxide dispersion.

In the observations corresponding to 8, 11, and 17 months, no significant differences were established in the behavior of the paints. After 21 months of immersion, including two periods of intense fouling (summer), only three of the samples prepared with three hours of cuprous oxide grinding exceeded level 1 of fouling (values 3, 2-3, and 1-2, respectively, for samples 3, 6, and 9). The fouling in the other paints was 0, 0-1, or 1, and in all cases fouling attachment decreases when matrix solubility increases. The paints elaborated with WW rosin/plasticizer, 5:1 ratio (greatest matrix solubility), had a fouling value of 0 (paints 10, 11, and 12).

After 21 months, samples prepared with 12 hours of cuprous oxide grinding had a fouling rating higher than 1 in all cases.

In samples stored for 25 and 50 months (Table 8), the antifouling efficiency has been reduced and fouling values 1-2 and 2 were observed. Aging time seems to have

significant influence on the bioactivity of the sample, since toxic action was reduced.

SUMMARY

Raft trials served to confirm the different behavior of samples of antifouling paints prepared with three and 12 hours of grinding, showing those with higher milling time to have lesser biocidal effect. This is due to the reactions between basic pigments (cuprous oxide and calcium carbonate) with the acid component of the binder (WW rosin) during dispersion and in the later storage.

Antifouling capacity is related to the toxicant leaching rate (which depends on its type and content in the paint) and with the matrix solubility. These two properties determine the effectiveness of the paint.

The different bioactivity of the samples with three hours of grinding could be attributed to the fact that those paints with a low content of unreacted rosin and low cuprous oxide level, present a toxicant leaching rate lower than the lethal minimum value necessary to avoid fouling attachment. For the same binder, samples prepared with high and medium toxicant content have a matrix of low solubility but of adequate bioactivity. These paints, with higher content of free rosin or higher final acidity, can be formulated with low toxicant level. It is evident that in this case it is necessary to have a high film thickness in order to get a similar useful life.

The above mentioned points are demonstrated by paints 1 (10.2% of WW rosin and 46% of cuprous oxide) and 3 (10.9% of rosin and 14% of toxic material) which

showed, respectively, fouling rating of 1 (little or rare) and 3 (very common) after 21 months in the raft, while sample 12 (14.6% of WW rosin and 14% of toxicant) showed no fouling (0).

In the case of the samples made with 12 hours grinding, for each rosin/plasticizer ratio, the highest settlement value corresponds to the paints with greatest toxicant content. This is due to the reaction between toxicant and binder during the grinding operation and to the formation of cupric resinate. For this reason, a small final level of WW rosin is obtained.

Samples aged in the container did not show an important change in toxic efficiency such as that observed with an excessive grinding of cuprous oxide. No significant formation of calcium resinate and cupric resinate, due to the binder/pigment reaction, is produced during storage.

ACKNOWLEDGMENT

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Solubility Parameter Concept in the Design Of Polymers for High Performance Coatings

II. Urethane Copolymers

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Aircraft coatings are required to meet a variety of severe conditions. Among the various applied film requirements, the combination of fluid resistance and flexibility appears to be most challenging.

The design of synthetic resins to function as vehicles for prospective aqueous aircraft coatings began with the characterization of the solubility parameter value of a number of test fluids. Using the method of atomic group contributions, urethane copolymers having the desired solubility parameter values were synthesized in organic medium. These solution polymers were then dispersed into aqueous medium as both anionic and cationic dispersions, formulated with film aids and additives, and evaluated for clear-film performance.

Performance was as expected for all test fluids except water and the onset of resistance to any test fluid was predicted.

INTRODUCTION

The development of an aqueous polymer system to function as the pigment binding vehicle of a prospective aircraft topcoat or primer has been the object of a research effort recently sponsored by the U.S. Air Force.¹ This goal has been elusive primarily due to the unique requirements of this application. These high performance requirements have precluded the use of general purpose

water-based industrial polymers and have necessitated the development of a specialty polymer.

The previous article in this series² described the performance of a topcoat qualifying for designation as military specification C-83286. Basically, the specification requires resistance to a variety of corrosive fluids while maintaining flexibility over a broad temperature range. The article goes on to discuss the approach of employing the solubility parameter concept in the design of synthetically novel polymers for enhanced fluid resistance.

A series of anionic acrylic aqueous dispersions and a series of cationic acrylic aqueous dispersions were synthesized. Clear films (unpigmented) of these dispersions were evaluated and performance was interpreted as a function of solubility parameter variations. Observed performance was in close agreement with predicted performance and the onset of resistance to a particular fluid could be predicted.

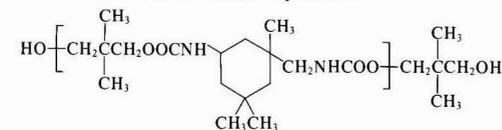
The following report represents a continuation of the study sponsored under Air Force contract F33615-78C-5096. The results present an extension from the class of synthetic acrylic resins to the class of synthetic polyurethane resins. And, as was the case for the study on acrylics, both cationic and anionic aqueous polyurethane dispersions will be discussed.

DISCUSSION

In reviewing the applied film properties of military specification C-83286, the combination of fluid resistance and flexibility provides the greatest challenge and constitutes the point of failure of most resin candidates. Since chemical resistance is usually achieved by crosslinking and since crosslinking typically results in embrit-

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Table 1—Solubility Parameter Calculation For NPG/IPDI Polyurethane

Group	Number	Δe (cal/mol)	Δe_1	Δv (cm ³ /mol)	Δv_1
CH ₃	5	1125	5625	33.5	167.5
CH ₂	6	1180	7080	16.1	96.6
CH	1	820	820	-1.0	-1.0
C	3	350	1050	-19.2	-57.6
NHCOO	2	6300	12600	18.5	37.0
6 atom ring chain atoms	1	250	250	16.0	16.0
	16				32.0
			27425		290.5

$\delta = (\Sigma \Delta e_i / \Sigma \Delta v_i)^{0.5} = (27425 / 290.5)^{0.5} = 9.7H$

tlement, the cause of failure appears obvious. Conversely, any polymer displaying optimum chemical resistance in the linear (thermoplastic) state should also be expected to display optimum film extensibility. The problem reduces to one of identifying a linear polymer displaying inherent chemical resistance.

In considering the relationship of polymer structure and chemical resistance, a literature search inevitably leads to the Flory-Huggins equation.³ Unfortunately, the

solvent-polymer interaction parameter (χ_1), described in this equation, is difficult to apply to the design of synthetically novel polymers. Alternately, the compatibility of a solvent and polymer has been expressed in terms of the solubility parameter concept.⁴

Solubility Parameter Concept In Resin Design

Hildebrand⁵ has shown that for a solution process to occur, the solubility parameter value of the solute and solvent must be nearly equal. In reverse, application of this theory to the case of solvents and polymers should provide a means of predicting solvent resistance. That is, solvent resistance should be expected for polymers and solvents of broadly distinct solubility parameter values.

Furthermore, the solubility parameter concept has been demonstrated to be useful in molecular design. Small,⁶ and later Rheineck and Lin,⁷ have estimated the solubility parameter of polymers by combining contributions of various atomic groups comprising the molecular structure. More recently, Fedors⁸ has provided an extension to the number of groups available.

Design of Novel Polyurethanes

Fedors' method for the calculation of the solubility parameter for acrylic copolymers has been discussed.² The case of polyurethanes employs similar considerations and begins with the definition of the solubility parameter as the square root of the cohesive energy density [equation (1)].

$$\delta = (\Delta E / V_m)^{0.5} \quad (1)$$

As described in equation (1), the cohesive energy density is defined as the ratio of energy of vaporization and molar volume.

Table 2—Energies of Vaporization And Molar Volumes of Some Polyurethanes

Isocyanate	Polyol	$\Sigma \Delta e_i$ (cal/mol)	$\Sigma \Delta v_i$ (cm ³ /mol)
Isophorone (IPDI)	Neopentyl (NPG)	27425	290.5
IPDI	Dimethylolpropionic (DMPA)	32900	285.5
DDI-1410	NPG	60040	696.6
DDI-1410	DMPA	65515	691.6
IPDI	2,2,4-Trimethyl-1,3-pentanediol (TMPD)	30135	339.4
Bis(4-cyclohexyl) methane (Des W)	TMPD	34670	338.8
Des W	DMPA	37435	284.9
Des W	2-Ethyl-1,3-hexanediol (EHD)	35610	339.3
IPDI	1,6-Hexanediol (DHH)	29545	313.1
IPDI	Cyclohexanedimethanol (CHDM)	31435	331.1
IPDI	Polymeg® Q650	27985	246.7
IPDI	Niax® PCP-0200	35825	313.0
Des W	Niax® PCP-0200	40360	344.4
IPDI	2,2'-Sulfonyl diethanol (SDE)	33425	262.4
Toluenediisocyanate (TDI)	DHH	28435	236.5
TDI	DMPA	31790	208.9
Diphenylmethane (MDI)	CHDM	38010	268.5
MDI	DMPA	39475	232.9
MDI	Niax® PCP-0200	42400	292.4
TDI	Niax® PCP-0200	34715	238.4

SOLUBILITY PARAMETER CALCULATION OF POLYURETHANES: Next, the structure of the smallest repeat unit is considered and the contribution of each atomic group to the total energy of vaporization and molar volume is summed over the molecular structure. The ratio is calculated as the cohesive energy density and the square root is taken as the solubility parameter value. The calculation is illustrated in *Table 1* using the homopolymer of neopentyl glycol (NPG) and isophorone diisocyanate (IPDI) as an example.

Thus, Fedors' method estimates that the polyurethane of neopentyl glycol and isophorone diisocyanate should have a solubility parameter value of 9.7 Hildebrand units.⁹ *Table 2* presents the values of the summation of energy of vaporization and molar volume terms for some hypothetical urethane homopolymers. These will be required for the calculation of the solubility parameter of copolymers.

SOLUBILITY PARAMETER CALCULATION OF URETHANE COPOLYMERS: The calculation is extended to the case of copolymers by considering the mole fraction of each homopolymer present in the copolymer repeat unit structure. The calculation is illustrated in *Table 3* using

the copolymer of isophorone diisocyanate with a diol mixture of 85 mol percent neopentyl glycol and 15 mol percent dimethylolpropionic acid.

Thus, the copolyurethane of isophorone diisocyanate with neopentyl glycol and dimethylolpropionic acid is estimated to have a solubility parameter value of 9.9H.

RESULTS

In order to examine the generality of the solubility parameter concept in the design of polymers for enhanced fluid resistance, two series of polyurethanes of differing functionality were considered. These polymers were synthesized in organic solvent, converted to either anionic or cationic aqueous dispersions and evaluated for clear-film fluid resistance.

Aircraft Test Fluids

Having demonstrated the method and capability of designing polymers of predetermined solubility parameter, attention is next focused on the fluid which the polymer must resist. Aircraft coatings are required to resist a variety of fluids covering a range of polarities. These fluids have been described and estimates of individual solubility parameters have been made.² The fluids are: Mil H5606 hydraulic fluid ($\delta \sim 7.0$ H); Mil TT-S-735 type III hydrocarbon ($\delta \sim 7.5$ H); diester lubricating oil ($\delta \sim 8.0$ H); Skydrol 500 B hydraulic fluid ($\delta \sim 11.0$ H); and deionized water ($\delta \sim 23.0$ H).

Construction of a spectrum of solubility parameters for these fluids reveals a distinct gap between Skydrol 500B and water. Consequently, any polymer required to resist the body of fluids should have a solubility

Table 3—Solubility Parameter Calculation For IPDI/NPG/DMPA Urethane Copolymer

Polymer unit	Mol. Frac. (X)	$\Sigma \Delta e_1$	$(\Sigma \Delta e_1) (X)$	$\Sigma \Delta v_1$	$(\Sigma \Delta v_1) (X)$
IPDI · NPG	0.85	27425	23311.25	290.5	246.9
IPDI · DMPA	0.15	32900	4935.00	285.5	42.8
			28246.25		289.7

$$\delta = [(\Sigma \Delta e_1) (X) / (\Sigma \Delta v_1) (X)]^{0.5} = (28246.25 / 289.7)^{0.5} = 9.9\text{H}$$

parameter value falling midway in this gap, as this point constitutes the value of greatest deviation from any singular fluid.

Anionic Polyurethane Aqueous Dispersions

A series of urethane solution polymers varying solely in solubility parameter was designed and synthesized. Since these polymers were to be dispersed into aqueous medium, part of the design included use of dimethylolpropionic acid to introduce water-compatibilizing carboxyl functionality. When reacted with volatile amines and mixed with water, these carboxy functional polymers

Table 4—Anionic Polyurethane Formulation Properties

	2524-130	2722-177	2722-166	2722-171	2722-187	2722-162
Composition ^a	IPDI/DHH	MDI/EHD	DesW/DANT ^b	TDI/DHB ^c	TDI/DANT ^b /DHB ^c	TDI/DANT ^b
Solubility param. (δ)	9.9	10.8	11.2	11.5	12.2	12.8
Acid value	25.9	25.9	24.6	24.0	24.5	23.7
Aziridine resin ^d	XAMA-7	XAMA-7	XAMA-7	XAMA-7	XAMA-7	XAMA-7
Solids, percent	24.3	37.6	25.6	40.9	35.3	28.2

(a) All polymers prepared with dimethylolpropionic acid, DMPA.

(b) Dantocol DHE, bis(2-hydroxyethyl)dimethylhydantoin, Glyco Chem. Co.

(c) I,4-Dihydroxybutane.

(d) Cordova Chem. Co.

Table 5—Fluid Resistance of Anionic Polyurethane Coatings

Formulation (δ)	Film Thickness, mil	Original Hardness	Lub. Oil ($\delta, 8$)	Water ($\delta, 23$)	H5606 ($\delta, 7$)	Skydrol 500B ($\delta, 11$)	TT-S-735 ($\delta, 7.5$)
2524-130 (9.9)	1.0-1.9	B, 2B	HB	<4B	HB	<4B	2B
2722-177 (10.8)	0.8-1.3	HB	<4B	<4B	HB, B	DF ^a	HB, B
2722-166 (11.2)	0.5-0.7	B	F	<4B	HB	<4B	F, HB
2722-171 (11.5)	0.7-1.0	HB	F	<4B	HB	DF ^a	HB, B
2722-187 (12.2)	0.7-1.1	B	F	<4B	2B	DF	HB
2722-162 (12.8)	0.5-0.8	HB	F, H	<4B	HB	4B	HB

(a) Dissolved Film

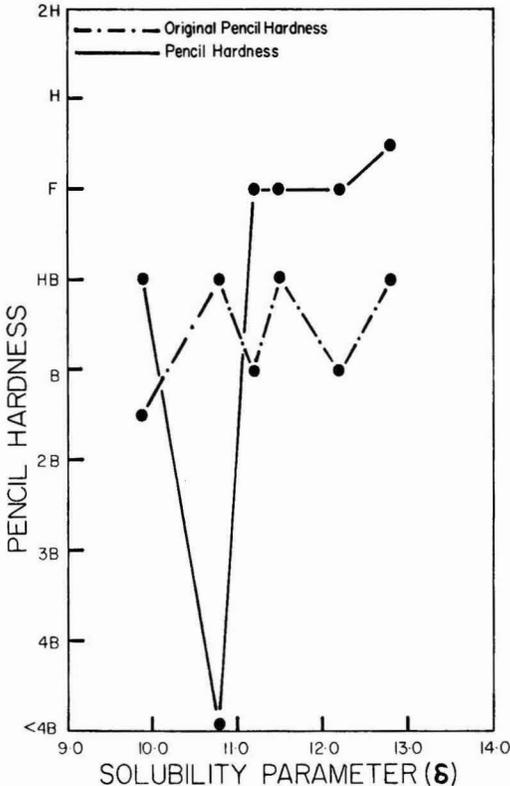


Figure 3—Effect of anionic polyurethane solubility parameter on diester lubricating oil ($\delta \sim 8H$) resistance

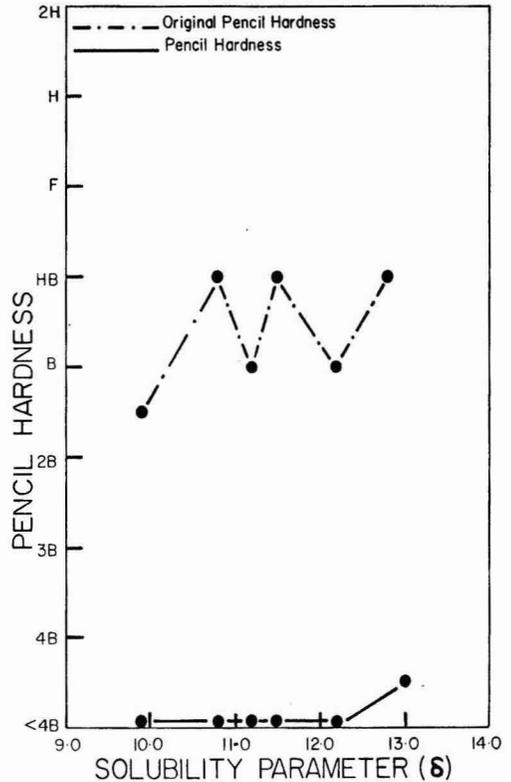


Figure 4—Effect of anionic urethane solubility parameter on Skydrol 500B ($\delta \sim 11H$) resistance

can thereby be minimized leaving polymeric solubility parameter as the significant variable.

The solution polymers were next reacted with triethylamine dispersed into aqueous medium and formulated with aziridine resins and, in some cases, film formation aids. Table 4 summarizes the properties of these clear-film aqueous polyurethane formulations.

The identity of the component materials can be found as abbreviated in Table 2 except where described in footnotes. As indicated, all polymers contained dimethylolpropionic acid for carboxyl functionality. And all formulations were prepared with stoichiometric equivalence of aziridine resins to insure consumption of the carboxyl functionality.

FLUID RESISTANCE PERFORMANCE: The formulations were spray applied to aluminum substrate, allowed to dry for seven days at constant temperature (25°C) and humidity (50%) and immersed in each test fluid. Fluid resistance was measured in terms of pencil hardness rating. Each coating was rated before and after immersion and a decrease was indicative of attack.

Table 5 describes the results of the fluid resistance tests. Experiments were performed in duplicate and both values are reported where different. All formulations

exhibit the expected trends. As the polymeric solubility parameter diverges from that of the fluid, resistance is achieved.

In the case of Skydrol 500B, achievement of resistance appears to be just beginning with the last member of the series.

All coatings were severely attacked by water. This fluid appears to constitute a special case. Incomplete reaction between the aziridine and carboxyl functionalities would leave water sensitive sites within the film and result in water softening. The likelihood of this occurrence improves under ambient conditions and may be the cause of this unexpected attack in all cases.

PREDICTING FLUID RESISTANCE: The fluid resistance results presented in Table 5 can be analyzed in graphic detail for each fluid. Such analysis enables the prediction of the solubility parameter at which the onset of resistance can be expected.

Figures 1—5 depict the results of Table 5 data. Each figure represents the assigned pencil hardness rating after immersion in the indicated test fluid. Also included as the reference is the curve of pencil hardness rating before immersion.

Figure 1 displays the resistance of the series of anionic

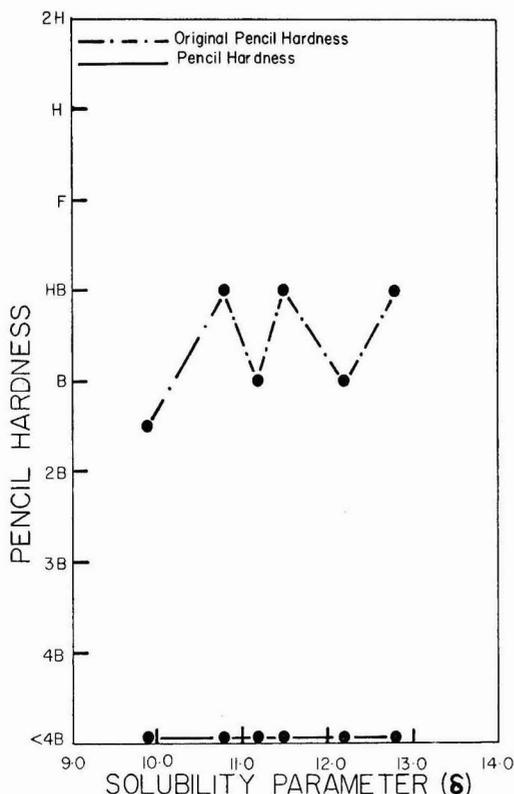


Figure 5—Effect of anionic urethane solubility parameter on resistance to deionized water ($\delta \sim 23\text{H}$)

polyurethanes to Mil H5606 hydraulic fluid ($\delta, 7\text{H}$). The curve relates that all members of the series resist this fluid and predicts that anionic polyurethanes of solubility parameter about 10H and greater should not be appreciably attacked. There appears to be some slight softening around $\delta, 12\text{H}$, but the attack is not very severe.

Figure 2 indicates resistance to TT-S-735 type III hydrocarbon ($\delta, 7.5\text{H}$). Some slight softening is apparent around 10H, but, in general, resistance over the entire series is good.

Figure 3 representing resistance to diester lubricating oil ($\delta, 8\text{H}$) indicates that resistance can be predicted for anionic urethanes of solubility parameter greater than 11H.

Figures 4 and 5 detail that both Skydrol 500B and deionized water severely attack all the polymers of the series. Figure 4 relates that some resistance to Skydrol 500B appears to be developing around $\delta, 13\text{H}$ and that greater values may provide the anticipated resistance. Figure 5 shows that water devastates the entire series. But as described previously, this performance may be more an artifact of incomplete carboxyl reaction than the true effect of polymeric solubility parameter.

Cationic Polyurethane Aqueous Dispersions

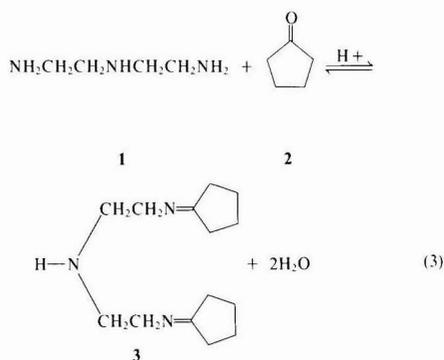
Having demonstrated the applicability of the solubility parameter concept to the design of fluid resistant anionic polyurethanes, examination of an alternate cure mechanism was in order. Given the restriction of reaction at ambient temperature, the epoxy-amine reaction was selected as the appropriate cure mechanism. Hence, a series of polyurethanes of varying solubility parameter with pendant amine functionality was required.

When reacted with volatile acids and mixed with water, the amine-functional polymers result in colloidal dispersions stabilized by electrostatic repulsion attributed to the ammonium cation. The dispersion is then to be blended with epoxy resins and applied to the substrate. Upon film formation, the acid evaporates leaving the amine functionality for reaction with the epoxy resin.

INTRODUCTION OF AMINE FUNCTIONALITY: Since amines are more reactive with isocyanates than are alcohols, the preparation of amine-functional cationic dispersions is not as straightforward as the analogous anionic dispersions. Incorporation of amine functionality into a polyurethane backbone must utilize an indirect method.

Literature techniques¹⁰ have reported addition of a polyamine in the presence of methyl ethyl ketone as a solvent to an isocyanate terminated prepolymer. Gellation is presumably prevented by formation of the intermediate ketimine. Since the polymers of the proposed series are of sufficient solubility parameter to prevent solution in ketone solvent, direct adaptation of this technique is not possible.

A similar technique involving isolation of the ketimine was employed. The ketimine (3) of diethylenetriamine (1) and cyclopentanone (2) was synthesized as in equation (3).



The imine (3) was then added to a solution of an isocyanate terminated prepolymer resulting in an adduct with latent amine functionality as in equation (4).

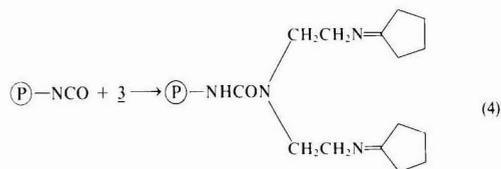


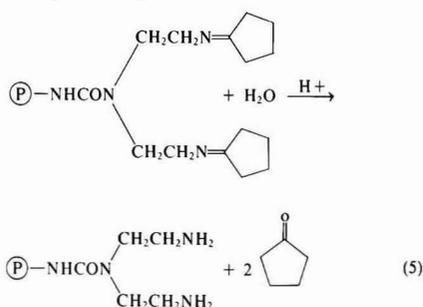
Table 6—Cationic Polyurethane Formulation Properties

	2722-108	2722-144	2722-120	2722-137	2722-183	2722-158
Composition	IPDI/CHDM	MDI/Q650	DesW/PCP-0200	TDI/BisA	TDI/DANT ^a /CHDM	TDI/DANT ^a
Solubility param (δ)	9.7	10.1	10.4	11.3	12.0	12.9
Equivalent wgt.	2118	2011	4038	2688	3227	2025
Epoxy resin (DER) ^b	331	331	331	331	331	331
Solids, percent	23.2	22.2	25.9	26.0	33.5	28.7

(a) Dantocol DHE, bis(2-hydroxyethyl)dimethylhydantoin, Glyco Chem. Co.

(b) Dow Chem. Co.

The adduct is made water dispersible by protonation with acid. Upon dispersion into water the latent amines are liberated as the ammonium cation by hydrolysis of the imine. Amine functionality is generated in film formation by evaporation of the acid. This sequence is indicated in equation (5).



CATIONIC POLYURETHANE DISPERSION PROPERTIES: Using the above sequence of reactions for aqueous dispersion, a series of cationic polyurethanes varying solely in solubility parameter was synthesized. The series covered a range of values from 10H to 13H.

Attempts to maintain consistent equivalent weights within the series as well as with the previous series of anionic polyurethanes were made. Again, consistency in this fashion would rule out the effect of molecular weight on performance. And comparison to the series of anionic dispersions would permit an evaluation of the crosslink bond upon fluid resistance.

After preparing the cationic dispersion, clear-film formulations were made by blending epoxy crosslinking

agents and film formation aids. Table 6 summarizes the dispersion properties of the series of cationic polyurethanes. The isocyanate and polyol components of each polymer can be identified by reference to Table 2 except where indicated in a footnote.

All formulations employed DER 331 as the epoxy crosslinking agent. And variations in solids content are reflective of reduction to spray viscosity (about 25 seconds in a No. 2 Zahn cup).

FLUID RESISTANCE PERFORMANCE: The formulations were spray applied to aluminum and allowed to dry seven days under constant temperature and humidity. The panels were given a pencil hardness rating, immersed in the appropriate test fluid, and again rated for pencil hardness. Table 7 summarizes the fluid resistance performance of the series of cationic polyurethane dispersions.

Once again performance is generally as expected. As the solubility parameter of the polymer approaches that of the fluid, softening is experienced.

The case of Skydrol 500B is interesting because resistance is displayed around solubility parameter of 12.9H. In the case of anionic urethanes, resistance was just developing at this point. The difference in performance may be interpreted in terms of the different crosslink mechanisms.

Water is seen to severely attack all the films and this observation is consistent with the class of anionic polyurethanes.

PREDICTING THE ONSET OF FLUID RESISTANCE: As for the case of anionic polyurethanes, performance in each fluid was displayed graphically to predict at which solu-

Table 7—Fluid Resistance of Cationic Polyurethane Formulations

Formulation (δ)	Film Thickness, mil	Original Hardness	Lub. Oil ($\delta, 8$)	Water ($\delta, 23$)	H5606 ($\delta, 7$)	Skydrol 500B ($\delta, 11$)	TT-S-735 ($\delta, 7.5$)
2722-108 (9.7)	1.3-2.0	HB	HB	<4B	HB	DF ^a	HB
2722-144 (10.1)	0.4-0.8	HB	<4B	<4B	HB	<4B	<4B
2722-120 (10.4)	0.7-1.2	HB, H	2B, 3B	3B, <4B	H	<4B	<4B
2722-137 (11.3)	0.3-0.5	HB, H	HB, F	DF ^a	F	DF ^a	F
2722-183 (12.0)	1.0-1.4	HB	H, 2H	<4B	HB	DF ^a	HB
2722-158 (12.9)	0.6-1.2	HB	2H	<4B	HB	F, H	HB

(a) Dissolved film.

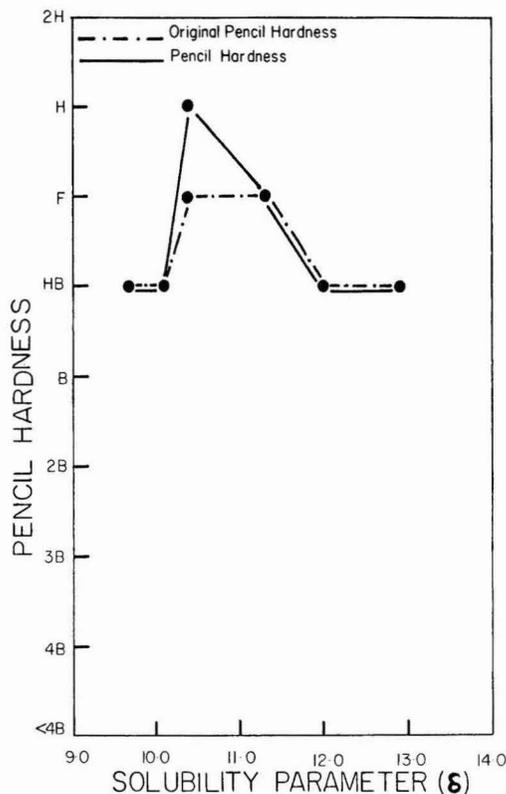


Figure 6—Resistance to Mil H5606 hydraulic fluid ($\delta \sim 7H$) as a function of cationic polyurethane solubility parameter

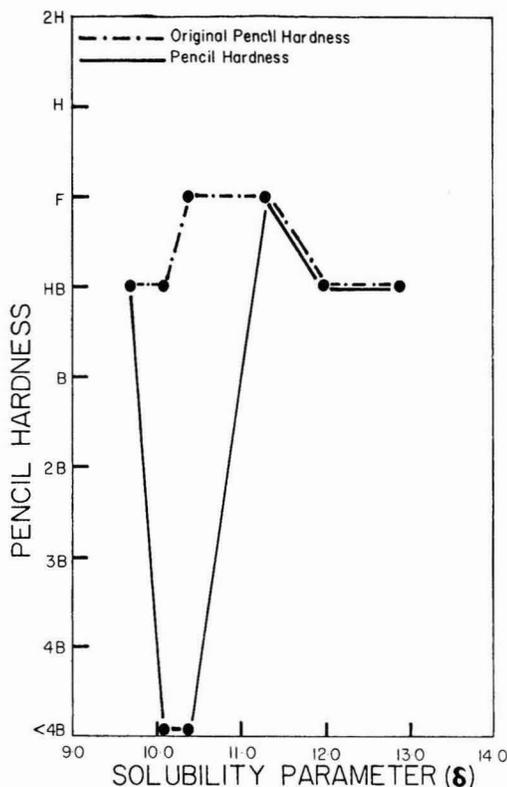


Figure 7—Resistance to TT-S-735 type III hydrocarbon ($\delta \sim 7.5H$) as a function of cationic urethane solubility parameter

bility parameter value resistance can be anticipated. Figures 6—10 relate the data in Table 7 for each fluid.

Figure 6 indicates that cationic polyurethanes of solubility parameter greater than 9.7H should be capable of resisting Mil H5606 hydraulic fluid when blended with epoxy crosslinking agents of the bisphenol A type.

Figure 7 relates resistance to TT-S-735 type III hydrocarbon for cationic polyurethanes above solubility parameter 11.0H.

Figure 8 indicates expected resistance to diester lubricating oil at 11.3H.

Resistance to Skydrol 500B can be expected above values of 12.7H as indicated in Figure 9.

As was true for the class of anionic urethanes, resistance to water is difficult to achieve under ambient conditions and probably involves other considerations in addition to the polymeric solubility parameter.

EXPERIMENTAL

The total evaluation of the polymers discussed in the text is a three-step process after design. The polymers were synthesized in organic solvent. The solutions were then converted to aqueous colloidal dispersions. Finally, the dispersions were made into unpigmented formula-

tions by mixing with crosslinking agents and film-formation aids.

These formulations were spray applied to aluminum substrates which were given an alodine conversion coating. The panels were dried under constant temperature and humidity and evaluated as described in the text.

Since the number of formulations and intermediates is extensive, only general procedures for synthesis, dispersion, and formulation will be described in the interest of brevity. Experimental detail can be found in an annual report released by the U.S. Air Force^{1b} which is available through the National Technical Information Service (NTIS).

SYNTHESIS

After design of the hypothetical polyurethanes having the preferred solubility parameter values, the polymers were synthesized in organic medium. The case of cationic urethanes required introduction of amine functionality as an additional step. As described in equation (4), addition of ketimine precursor 3 accomplished this transformation.

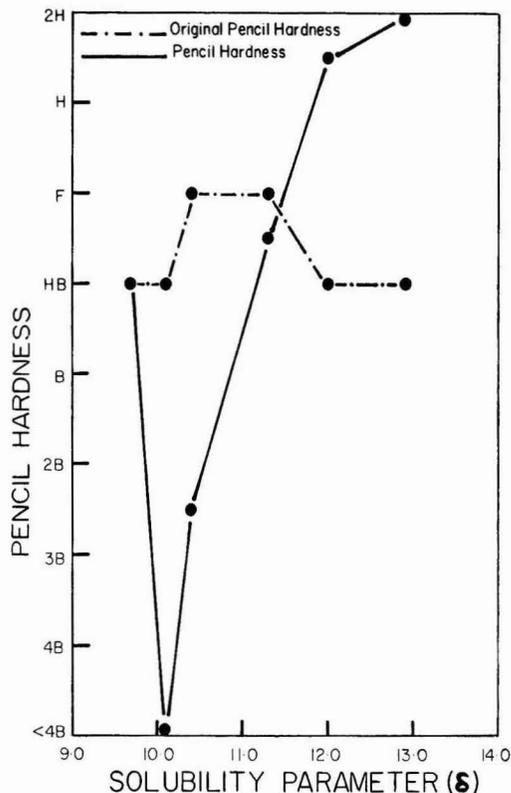


Figure 8—Resistance to diester lubricating oil ($\delta \sim 8H$) related in terms of cationic polyurethane solubility parameter

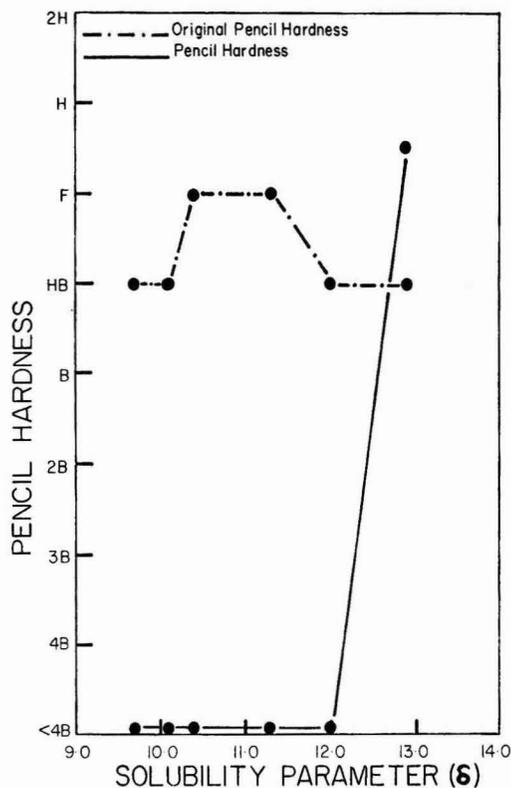


Figure 9—Resistance to Skydrol 500B ($\delta \sim 11H$) related to cationic polyurethane solubility parameter

KETIMINE PRECURSOR (3): Into a two liter single-neck round-bottom flask is poured 95.3 g of diethylenetriamine (1) and enough anhydrous benzene to make a final volume of 1.5 liters. To this solution is added two grams of Dowex® 50W-X8 ion exchange resin (as catalyst) and 174.6 g of cyclopentanone (2).

The flask is then equipped with a Dean-Stark trap and a reflux condenser fitted with a drying tube. The mixture is stirred with a magnetic spin-bar and heated to reflux. Reflux is continued until the stoichiometric amount of water is collected.

The solution is then cooled, filtered, and concentrated by rotary flash evaporation under water aspirator vacuum. The product is transferred to an amber glass bottle, sealed under a nitrogen atmosphere, and refrigerated. IR (cm^{-1}): 3300 (bd. singlet), N-H; 1670 (sharp singlet), C=N.

CARBOXY-FUNCTIONAL POLYURETHANES: A four-neck round-bottom flask is equipped with mechanical stirrer, thermometer, reflux condenser (with drying tube), pressure equalized dropping funnel, and inlet for dry nitrogen. Into the flask is poured the solvent mixture and the mixture of diol reactants. The flask contents are set under a nitrogen atmosphere and heated to reaction temperature (usually 80°C). Stirring is continued until solution

is complete and the temperature is thermostatted to $\pm 1^\circ\text{C}$. At this point a catalytic amount of dibutyltin dilaurate is added.

To the solution is then added the isocyanate reactant. Addition is made to occur dropwise over a three-hour period and the temperature is maintained. After addition is complete, the isocyanate content is monitored by reaction with *n*-butylamine until the theoretical value is attained. The reaction is terminated by cooling and the solution is transferred to a glass container.

AMINE-FUNCTIONAL POLYURETHANES: An isocyanate terminated polyurethane is synthesized by a procedure analogous to that described for carboxy-functional urethanes. The reaction is terminated when the desired isocyanate content and corresponding equivalent weight is attained.

The solution is cooled to 60°C and an equivalent amount of ketimine, 3, is added dropwise. The mixture is then stirred with good mechanical agitation for 30 minutes. The infrared absorption of the isocyanate functional group is then monitored. When the absorption can no longer be detected, the mixture is cooled and the resulting viscous solution is poured into a glass container. The amine equivalent weight of the polymer is then determined by titration with perchloric acid.

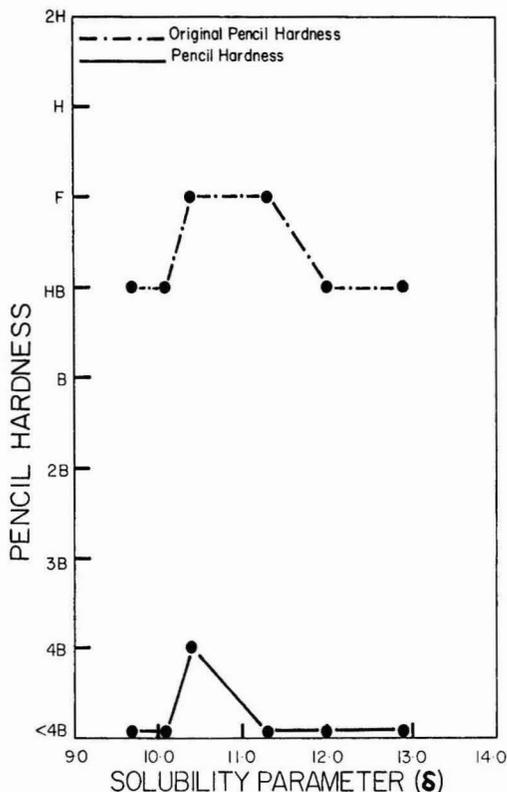


Figure 10—Effect of cationic polyurethane solubility parameter on deionized water ($\delta \sim 23H$) resistance

Aqueous Dispersion

The polyurethane solutions are converted to aqueous colloidal dispersions by the following general procedure:

The polymer solution is weighed into a stainless steel beaker and diluted with a volatile water-miscible cosolvent (in this case tetrahydrofuran). The sample is then reacted with an equivalent amount of neutralizing reagent. For anionic dispersions, the reagent is triethyl amine; for cationic dispersions the reagent is glacial acetic acid.

The mixture is stirred with a high speed dispersator under low shear conditions until the mixture is uniform. The speed is then increased to provide high shear conditions and water is then added slowly in small increments to prevent shock precipitation. Addition of water is continued with increasing viscosity until inversion is indicated by dramatic viscosity reduction. The remainder of the water is added with slow speed mixing.

The resultant dispersion is then transferred to a tared flask. The dispersion is concentrated by rotary flash evaporation under water aspirator vacuum. The concentration is then adjusted by addition of water as needed. Finally, the concentrated dispersion is filtered through a fine grade paint strainer.

Aqueous Clear-film Formulations

The aqueous polymer dispersions are converted to clear-film formulations by mixing with crosslinking agents and in some cases film-formation aids. The carboxy-functional urethane dispersions are blended with polyfunctional aziridine resins; the amine-functional polyurethanes are mixed with polyfunctional epoxides.

No special technique is required for the preparation of these formulations. The materials are merely blended under normal low shear agitation to insure complete mixing. However, since both types of crosslinking agents are slowly hydrolyzed, the final formulation is mixed and filtered immediately prior to spray application.

SUMMARY

Using the method of atomic group contributions, polyurethanes of preferred solubility parameter values were synthesized in organic solution. The solutions were transformed to aqueous dispersion using both cationic and anionic dispersion techniques.

The dispersions were formulated with crosslinking agents designed for use under ambient film formation conditions and these formulations were evaluated as potential aircraft coatings.

Results indicate that the solubility parameter concept is useful in predicting resistance to certain organic fluids. Water appears to be a special case and does not apply.

ACKNOWLEDGMENTS

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Anticorrosive Effect of Some β -Diketones In Polymeric Coatings on Low Carbon Steel

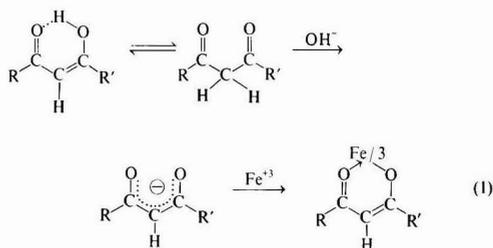
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Corrosion protection of polymeric coatings on carbon steel using a methyl/n-butyl methacrylate copolymer resin, can be enhanced with Fe(III) β -diketonate complexes applied first to the low carbon steel surface. The iron β -diketonate complexes were formed by reaction of the β -diketone complexing agents with iron at the surface. An increase in corrosion resistance was demonstrated by several corrosion tests and the extent of the increase was found to be related to the type of complexes developed. The increased corrosion protection of such polymeric top-coatings is probably due to chemical interactions formed between the polymeric components and the functional groups of the iron complex. This likely strengthens the bonding of the coating.

INTRODUCTION

Complexing agents have long been used as effective corrosion inhibitors for metal protection in an aqueous corrosive media. However, there have been few reports of their use in improving the performance of polymeric coatings on metallic surfaces. It was found in this experiment that the corrosion resistance of the polymeric films on a low carbon steel surface, which had been previously treated with β -diketones, generally increases. This is shown by the condensing humidity test, an intermittent total immersion test, and an atmospheric exposure test.

The β -diketones of interest include acetylacetone (acac), benzoylacetone (bac), and dibenzoylmethane (dbm). All form very stable complexes with many metals and metallic ions, such as iron.^{1,2} It has been reported^{3,4} that iron ions react very rapidly with β -diketones to form Fe(III) β -diketonates in a basic medium, e.g., sodium acetate, aqueous ammonia, or urea, as shown below:



where $R = R' = \text{CH}_3$ for acac; $R = \text{CH}_3$, $R' = \text{C}_6\text{H}_5$ for bac; and $R = R' = \text{C}_6\text{H}_5$ for dbm.

The stability constants of the above three Fe(III) β -diketonates have been determined by many workers⁵⁻⁹ and decrease in the order of acac < bac < dbm. The different stabilities of the β -diketonates have been explained in terms of the effectiveness of the delocalization of electrons in the chelating ring.¹⁰⁻¹⁴

EXPERIMENTAL

Specimens used in this study were cut from a large low carbon steel sheet of 1.4 mm thickness into a rectangular shape of 30 mm \times 40 mm and had the following chemical composition: (in wt%) C, 0.07; Mn, 0.37; P, 0.013; S, 0.009; Al, 0.002; Cr, 0.01; Ni, 0.02; Cu, 0.01; Mo, 0.01; Si, 0.01; N, 0.003.

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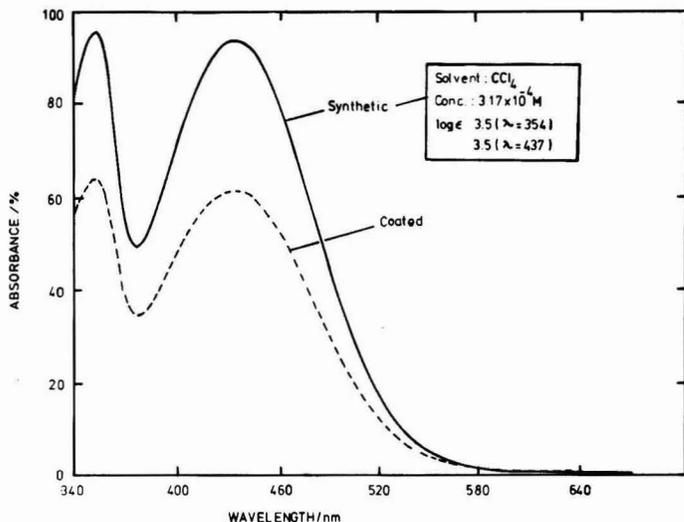


Figure 1—Visible spectra of synthetic and coated Fe(acac)₃

The as-received specimens were first electrochemically degreased in a solution of NaOH, Na₂CO₃, and Na₃PO₄ 12H₂O (10 g, 20g, and 50 g, respectively per liter) at a cathodic current density of 40 mA cm⁻² for one minute to remove oils, and cleaned by dipping into a 15 wt % hydrochloric acid (sp gr 1.16) solution containing 0.1 wt % hexamethylenetetraamine as inhibitor. If necessary, the latter process can be accelerated by gentle heating. The specimens were then neutralized in a 0.4 wt % Na₂CO₃ solution, rinsed with distilled water and acetone, and finally dried and stored in a desiccator before use.

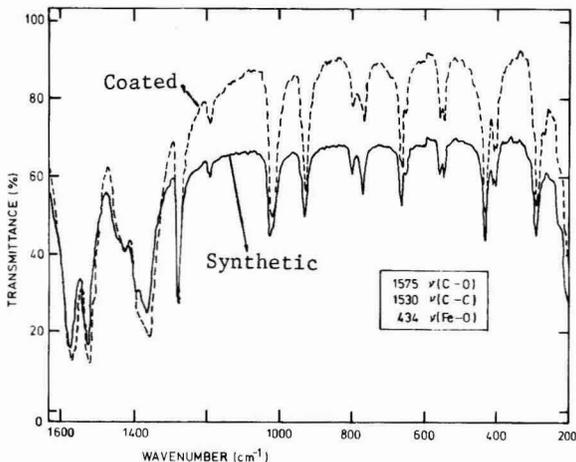
All complexing agents were of reagent grade. The polymeric coating material was a methyl/n-butyl methacrylate bead copolymer, Elvacite®.¹⁵ The polymer

solution was prepared by dissolving the copolymer in methyl ethyl ketone in a ratio of copolymer to solvent of 1:5. The Elvacite resin is fast dissolving and has a low viscosity with quick solvent release. Hence, during the polymeric coating process, the solvent has little effect on the complex layer which developed at the metallic surface of the specimen.

The coating was processed in two steps: (1) complex formation at the metallic surface of the specimen by immersing the latter in a complexing agent-containing solution of 50 mL for 30 seconds, then removing the coupon and drying it in air; and (2) applying polymeric coating by brushing the complex-coated surface (or bare surface of the control specimen) with a piece of glass rod wetted with the polymeric resin parallel to the surface, from bottom to top. In coating a control specimen, step (1) is omitted.

Elvacite is a registered trademark of E.I. du Pont de Nemours & Co., Inc.

Figure 2—Infrared spectra of synthetic and coated Fe(acac)₃ (KBr disk)



The orange to red-colored iron complex layer developed at the specimen surface was identified by visible and infrared spectroscopy. This was done by first stripping off the complex layer in carbon tetrachloride, and then running the visible and infrared spectra of the solution. These spectra were then run and compared with those of the synthetic Fe(III) β -diketonates in carbon tetrachloride. The correct composition of the synthetic compounds has been confirmed by microanalysis.

After polymeric coating, which was applied to either the surface having formed complexes or to the bare surface of the control, all specimens were subjected to corrosion testing¹⁶⁻¹⁸ as described below:

(1) *Condensing humidity test*—This is operated in a humidity chamber at 40°C and 90% relative humidity. Specimens and controls were hung in the chamber for two hours, then cooled to 0°C in a refrigerator for one hour to allow effective condensation of water vapor at the surface, and finally dried thoroughly in air for about 24 hours before the next cycle.

(2) *Intermittent total immersion test*—Specimens and controls were fully immersed in a 3 wt % NaCl solution for two hours at room temperature, then dried in air for about 24 hours before the next cycle.

(3) *Atmospheric exposure test*—Specimens and controls were suspended in an alternate position at an angle of about 30 degrees from the horizontal plane situated southward on the roof of the University Science Building. The university campus is located in a rural district with a harbor on its eastern side. The test was conducted in summer when the climate was typically warm (with an average temperature around 28°C) and humid (with an average relative humidity of 85%).

After each cycle or day of testing, the corrosion resistance of both sides of the specimens was evaluated by Champion's method,¹⁹ however, without considering the depth of attack. Each experiment involved at least two test specimens and two controls.

Some selected formulations for preparing the complexing solutions which have resulted in satisfactory complexes on metallic surfaces are given below:

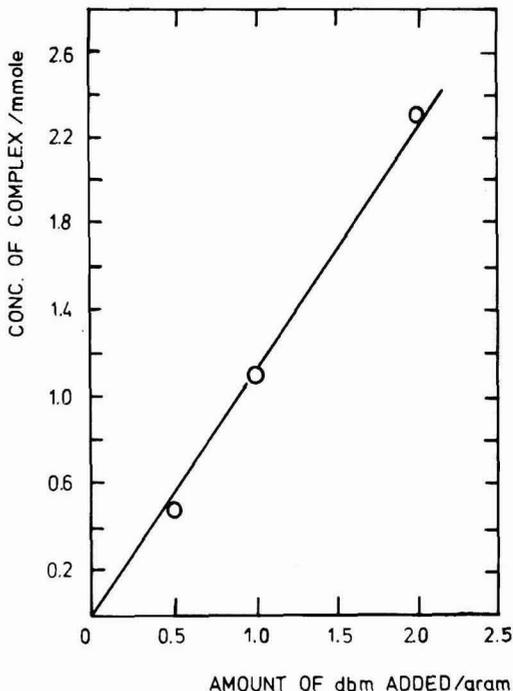


Figure 3—Concentrations of complex Fe(dbm)₃ forming at the substrate surfaces increase with the amount of dbm added in the same mixture solvent as in selected formulation (3)

(1) For acetylacetone complexes: 10 mL of acac in 10 mL of 95% ethanol and 1 mL of 1:1 ammonium hydroxide (one part water to one part concentrated aqueous ammonia). The solution pH was 9.*

(2) For benzoylacetone complexes:

(a) basic medium: 1.5 g of bac in a solvent mixture of 60 mL ethanol and 30 mL water. A few drops of 1:1 ammonium hydroxide were added until the pH was about 9;*

*When the specimen surface was complex-coated in a basic bath, a prior acidic activation with 1 M H₃PO₄ was required to obtain better complex formation.

Table 1—Summary of the Corrosion Testing Results

Corrosion Test	Specimen With Prior Complexing Treatment			Specimen With Polymer Coat Only
	acac	bac	dbm	
		Basic	Acidic	
Condensing humidity test	Rusted in the 5th test cycle.	Rusted in the 4th test cycle.	Rusted in the 5th test cycle.	Rusted in the 6th test cycle.
Intermittent immersion test (Test solution)	Rusted in the 2nd test cycle. (Clear)	Rusted in the 2nd test cycle. (Clear)	Rusted in the 4th test cycle. (Clear)	Rusted in the 6th test cycle. (Clear)
Atmospheric exposure test	Rusted in the 5th test day.	—	—	Did not rust up to 7 days.
				Rusted in the 1st test cycle.
				Rusted in the 1st test cycle. (Yellow)
				Rusted in the 3rd test day.

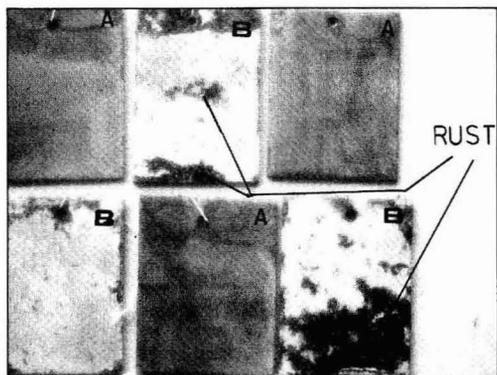


Figure 4—In a condensing humidity test, the Elvacite polymeric films with complexing agent dbm as a primer (A) show better anticorrosive performance than those without (B)

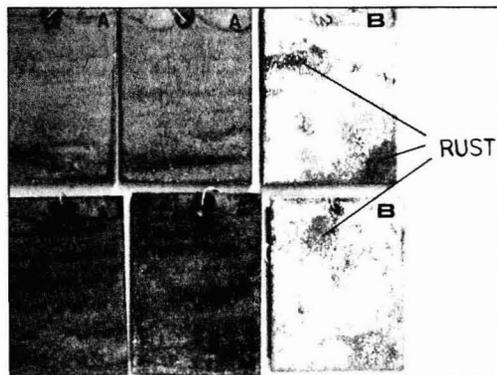


Figure 5—In an intermittent immersion test, the Elvacite polymeric films with complexing agent dbm as a primer (A) show better anticorrosive performance than those without (B)

(b) acidic medium: 1.5 g of bac in a solvent mixture of 60 mL ethanol and 20 mL water. A few drops of 1 M H_3PO_4 solution were added until the pH was about 2.5.

(3) For dibenzoylmethane complexes: 1 g of dbm in a solvent mixture of 75 mL dioxane and 25 mL water, plus 3 mL of 1 M H_3PO_4 solution. The pH was about 3.

RESULTS AND DISCUSSION

The chemical conversion of iron to Fe(III) β -diketonates occurring at the steel surface was verified by either the change of color in the latter or the visible and infrared spectra of the product on the surface. Figures 1 and 2 show the same spectra of the Fe(acac)₃ complex stripped from the complex-coated specimen plus that of

the synthetic compound. The visible spectra also allow the estimation of the concentration of complex formed at the surface, whereas the extent of uniformity of the complexes can be estimated by the distribution of the orange to red color of the complex on the surface.

With the preparative formulations of the complexing solutions described in the experimental section, the concentration of all three complexes formed on the surfaces was of the order of 10^{-3} mmole per sheet, or 10^{-5} mmole per cm^2 .

The complex layer produced by the basic acac-bath with prior acidic activation of the metallic surface was not quite uniform. This is most probably due to the slow vaporization of the aqueous solvent carried by the activation process, when hydroxides can therefore be formed immediately following the complexing process. However, the acidic acac-bath coating offering an in situ activation of the surface was incomplete because a large part of the Fe(acac)₃ complex failed to adhere to the surface. This provided a greater chance of hydroxide formation. The acidic bac-bath produced a more uniform Fe(bac)₃ complex layer than its basic counterpart because of the larger stability constant of Fe(bac)₃. Apparently, the acidic dbm-complex is the highest in uniformity among the three β -diketonates. This can be attributed to the rapid vaporization of the solvent after complex formation and the larger stability constant of the complex.

The concentrations of Fe(dbm)₃ complexes developed at the metallic surface increase with the amount of dbm in the complexing bath as shown in Figure 3. Results of corrosion tests on the testing and control specimens are summarized in Table 1.

Filiform corrosion occurred earlier in all of the control specimens which were coated solely by the Elvacite resin under the specified testing conditions. Figures 4 and 5 show clearly the better anticorrosive effect of the specimens whose surfaces were first chemically converted to iron complexes and then top coated with the polymeric film.

The increase of corrosion resistance varies with the complex species formed at the surface in the order of

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Since, in the same environment, the anticorrosive properties of a polymeric film or coating is primarily determined by the nature of the latter as well as its binding to the metallic surface, the general increase of corrosion resistance of the Elvacite film on a complexed surface strongly suggests a reinforced binding introduced by the complex in between the polymeric film and the metallic surface.

Obviously, the stability and uniform distribution of the complex layer at the surface influence the extent of the anticorrosive effect. It is proposed that some chemical interactions which enhance the compatibility between the phenyl- and/or methyl- groups of the complex and the organic components of the Elvacite film prevail, while the complex itself is bonded chemically to the metallic surface. More detailed information of such chemical interactions will require further investigation.

The consistence of the observed sequence in Equation (2) with the increase of stability constants of the corresponding complex species supports the above proposed model, although the sequence may also reflect the degree of compatibility of the functional groups of the complex with the organic components of the polymeric film. Nevertheless, the order in equation (2) clearly indicates the decisive effect of iron complexes on the enhancement of corrosion resistance of the polymeric film.

SUMMARY

The corrosion resistance of a polymeric Elvacite coating on low carbon steel surfaces has been found to be significantly improved if the substrate surface is first treated with a properly prepared solution of some β -diketones. Essentially, such an effect increases with the

following order of complexes formed at the substrate surface $\text{Fe}(\text{acac})_3 < \text{Fe}(\text{bac})_3 < \text{Fe}(\text{dbm})_3$, and the effectiveness of these complexes is explained in terms of their stability constants as well as the hypothesized compatibility of their functional groups with the components of the polymeric film.

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Acid Catalyzed Curing Polyester/Melamine Resins With Latent Catalysts

W.J. Mijs, W.J. Muizebelt, and J.B. Reesink
Akzo Research*

In this paper, new latent catalysts (oximeesters of sulphonic acids) for the acid catalyzed curing of polyester/melamine resins are introduced. Mechanism and kinetics of the thermolysis reaction of these sulphonic acid esters, during the curing process have been elucidated.

The curing process was followed by monitoring changes in physical and chemical parameters of the resin system. Four methods were used: Fourier infrared spectroscopy recording emission and transmission spectra of the curing film; thermogravimetry; dynamic spring analysis determining Tg and loss tangent $\tan \delta$; and, finally, the "sag balance" viscosimeter.

Tests have been done to connect fundamental physical properties with end use properties of cured paint films to compare various catalysts. The test results indicate that crosslinking performance of the oxime-blocked sulphonic acid catalysts is similar to that of the usual amine/sulphonic acid salt catalysts. Low conductivity of the sulphonic acid oximeester resin systems, however, offers potential for electrostatic spraying. Furthermore, less absorption to some pigments was found with these catalysts compared to amine sulphonic acid salts.

INTRODUCTION

In modern high solids or solvent free organic coatings, resins of relatively low molecular weights are used compared to conventional systems. This is necessary because of application viscosity requirements when

using conventional spraying equipment. As a consequence, effective curing to an insoluble three-dimensional network involves a greater amount of chemical reactions than with conventional solvent-based systems.¹ The curing phase thus, has become much more critical with respect to end use properties. This stresses the importance of characterization of the curing process and careful analysis of chemical reactions during curing.

In this paper, the acid catalyzed curing process of high solids polyester/melamine resins using new blocked acid catalysts is analyzed. Methods monitoring curing by measurement of mechanical and rheological properties and by functional group analysis with Fourier infrared and thermogravimetric techniques are introduced.

Composition of the Resin System

The high solids low molecular polyester resin system containing commercial grades of "Hexamethoxymethyl-melamine" (HMMM)[†] was of the following composition²:

Mixture of equal parts by weight of (a) di(mono-decanoic acid ester of trimethylol propane) isophthalate; (b) di(neopentylglycol) (iso/ortho)phthalate (iso:ortho = 1:2). Polyester resin HMMM = 3:1 by weight.

Latent Catalysts for Curing

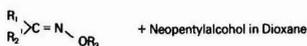
Curing with HMMM is an acid catalyzed process.³ The common catalyst is p-toluene sulphonic acid, frequently used in "blocked" form as its amine salt to ensure a satisfactory potlife of the resin. However, free acids and salts impart a too high conductivity to the resin to permit electrostatic spraying. Moreover, adsorption to some pigments is sometimes a problem.

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[†] The actual functionality of the curing agent used was not 6 but 4 to 5.

Table 1—Kinetics of Decomposition of Oximtosylates

R ₁	R ₂	R ₃	k x 10 ² (sec ⁻¹) ^{xxx}	ΔH [‡] kcal/mole	ΔS [‡] (e.u.)
CH ₃ *)	C ₂ H ₅ *)	Tos	1900	20,7	-15
Ph	PhC(=O)	Tos	130	24,2	-12
Ph	PhC(=O)	Mes	93	25,2	-10
O ₂ N - (C ₆ H ₄) *)	CH ₃ *)	Tos	64	23,9	-14
Ph	COOC ₂ H ₅	Tos	12,7	25,1	-14
COOC ₂ H ₅	Ph	Tos	27	24,6	-14
C ₆ H ₅ **)	COOC ₂ H ₅ **)	Tos	42	24,4	-14



*) Configuration not established
 **) Syn- and anti- have equal reactivity
 ***) at 130°C

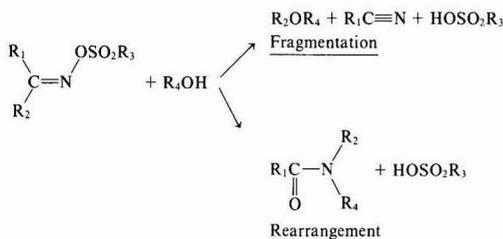
Table 2—Some Properties of a High Solid Polyester/Melamine System Cured With Blocked Acid Catalysts

Catalyst	Benziloximtosylate	p.tol. sulphonic acid diethanolamine
Solid Content (%) (1h, 130°C)	72.3	71.2
Layer thickness (um)	50–55	40–45
Gloss (20°)	90	96
Persoz hardness	257	298
Erikson indentation (mm)	8,2	7,4
Conical bending (mm)	11	12
Impact face/reverse	32/32	50/30
Xylene test (15') ^{*)}	8–9	10
Kestemich test ^{*)} (3 cycles):		
– Gloss (60°)	95	97
– Blistering	9	12
– Cracking	12	12
– Discoloration	12	12

*) rating: 0 = worst, 12 = best

Consequently, new latent catalysts (oximeesters of sulphonic acids), in which the acid and the blocking group are covalently bound, were designed. The acid is liberated by a thermal dissociation reaction. These catalysts were found to offer a good balance between potlife requirements and curing speed,⁴ as is substantiated below.

Thermolysis of oxime sulphonic esters can yield Beckmann fragmentation and/or Beckmann rearrangement products according to the nature of R₁ and R₂. The reaction proceeds as follows in the presence of an H donating species (R₄OH) like resin-OH, alcohols, water:



Mixtures of fragmentation and rearrangement are possible and the position of R₁ and R₂ may vary in the products according to their chemical nature and the stereochemical configuration in the oxime.⁵ At times, when water was present, hydrolysis products from intermediates in the rearrangement reaction were also found.

Products found in this curing process model reaction were in accordance with this scheme; e.g., the oxime-ester with R₁=PhC=O, R₂=Ph, R₃=pC₆H₅CH₃ gave mainly Beckmann fragmentation products, PhC≡N, PhCOOR₄ and p-toluenesulphonic acid. On the other hand, benzo-phenoneoximtosylate (R₁=R₂=Ph, R₃=pC₆H₅CH₃) mainly yielded Beckmann rearrangement products: PhCNH.Ph + R₄OC(=O)Ph + H₂NPh.

The ester and aniline presumably arise from hydrolysis (some water being present in the model reaction) of the rearrangement intermediate Ph-N=C(=O)-Ph. The

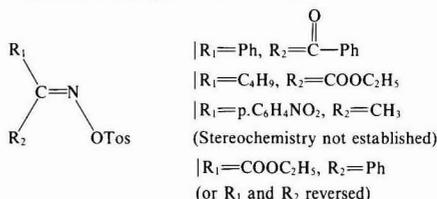


important point in any case is the formation of the sulphonic acid, which catalyzes the HMMM curing. The dissociation (thermolysis) rate of the latent catalyst is an important factor in the overall curing rate. Dissociation rates of several oximesulphonic acid esters at 80°–150°C in the presence of neopentylalcohol as a resin-OH model were measured and the corresponding activation parameters were calculated.

From the data presented in Table 1 it is clear that dissociation rates can vary considerably according to the nature of groups R₁ and R₂. Electron accepting groups generally have a retarding effect on the reaction rate, probably through destabilization of the intermediate carbonium ion in rearrangement of fragmentation.

From this range of compounds the optimal catalyst can now be selected. This is illustrated in Table 2. Persoz hardness and rub test values are used as indicators for cure response at 130°C and the viscosity increase is used to represent potlife at 40°C. The dissociation rates at different temperatures of the latent catalysts correlate well with cure response and potlife of the resin system.

The best catalysts in both respects seem to be:



The dissociation of the oximesulphonic acid esters is a first order reaction. Consequently, half-lives could be

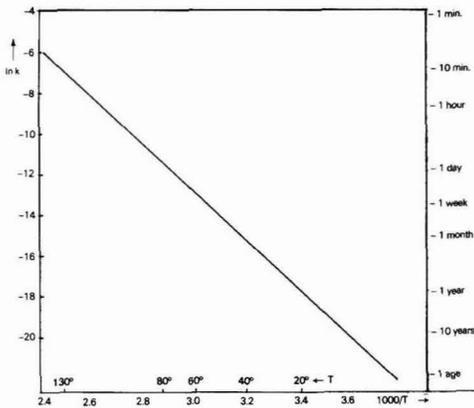
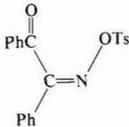


Figure 1—Half-life of benziloximtosylate as a function of temperature

established for each compound as a function of temperature. These half-lives are important factors which determine the curing rate at a given temperature. In Figure 1 the logarithm of the dissociation rate constant k is plotted against $1000/T$ for benziloximtosylate



with an indication of the corresponding half-life of this latent catalyst. As could be expected, the oximesulphonic ester resin systems have a relatively low conductivity which offers potential for electrostatic spraying. Measured under identical conditions at room temperature, the resistance of benziloximesulphonic ester/polyester resin was about 30 times higher than the similar amine/*p*-toluenesulphonic ester system.

Absorption to pigments was also less with the oximesulphonic ester catalysts compared to the amine salts, e.g., the *p*-toluenesulphonic acid concentration in the polyester-HMMM-tioxide RCR2 pigment paint decreased by 30–35% at a 0.6% sulphonic acid/diethylaniline level within six hours at room temperature. Kronos RN 59 even absorbed 55% of the acid. Using benziloximtosylate, only 1–5% absorption of sulphonic acid to the pigment was found.

CHARACTERIZATION OF THE CURING PROCESS

The acid catalyzed curing process of a "hexamethoxymethylmelamine" (HMMM)/polyester high solid resin has been characterized by several independent methods, which are also more generally applicable:

- (1) Fourier infrared and thermogravimetric analysis monitoring changes in *chemical* parameters during the curing process;

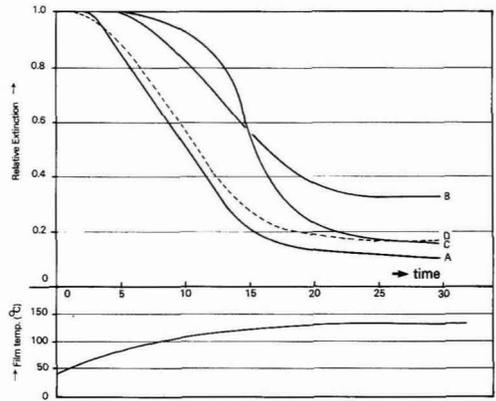


Figure 2—Infrared extinction ($E_{915 \text{ cm}^{-1}}/E_{815 \text{ cm}^{-1}}$) during acid catalyzed curing as a function of temperature

- (2) Dynamic spring analysis and "sagging balance" viscosimetry monitoring changes in *physical* parameters during the curing process.

Fourier Transform Infrared (FTIR)

With FTIR, emission and transmission spectra can be obtained very rapidly (within a few seconds). A good signal-to-noise ratio renders this technique eminently suitable to study dynamic processes. Thus, curing of a coating on a sheet substrate can be followed by recording FTIR emission spectra. Transmission spectra can be obtained on a KBr substrate.

In the case of our high solid polyester/HMMM system, the HMMM OCH_3 group (915 cm^{-1}) absorption intensity was measured against the triazine ring absorption (815 cm^{-1}) as a standard. The former decreases and the latter remains constant as the curing proceeds.

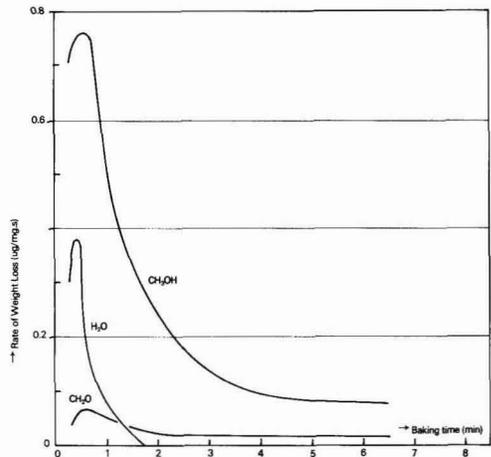


Figure 3—Type and degree of crosslinking by GC of high solids film emissions during curing at 130°C

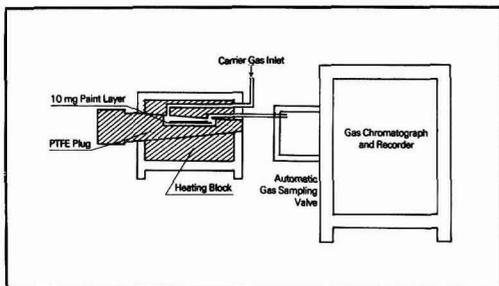


Figure 4—Mini baking oven with on-line gas chromatograph

Four catalysts were investigated: free *p*-sulphonic acid, (A); amine salt of *p*-toluene sulphonic acid, (B); benziloximotosylate, (C); and benzophenoneoximotosylate (D).

During the measurements the temperature was increased from 40°C to 130°C over 15 minutes, then held constant.

The results of the experiments are shown in Figure 2. Catalysts A and D are already reactive from about 60°C, whereas, B and C start at about 90°C. Noteworthy is the fast curing reaction in the case of C. The difference in curing reactivity between catalysts C and D is in agreement with the kinetic data on the corresponding dissociation reactions (compare with Table I). It is understandable that the polyester systems with A and D have a poor potlife at ambient temperatures.

Thermogravimetric Analysis (TGA)

Curing with HMMM gives rise to the emission of volatile products (methanol, water, and formaldehyde). The authors measured the rate of emission of these volatiles (see Figure 3) by coupling a curing oven to a gas chromatograph (see Figure 4). Curing with two catalysts (benziloximotosylate and benzophenoneoximotosylate) was investigated by thermogravimetric analysis. The maximum temperature was kept at 90°C. The difference in reactivity between benzophenoneoxime and benziloximotosylates is again clearly apparent (Figure 5). Some weight loss is recorded in the blank experiment without a catalyst.

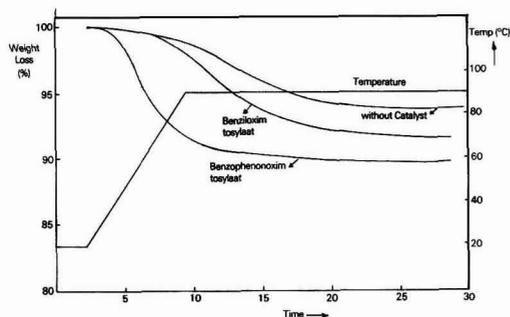


Figure 5—TGA curves of acid catalyzed curing

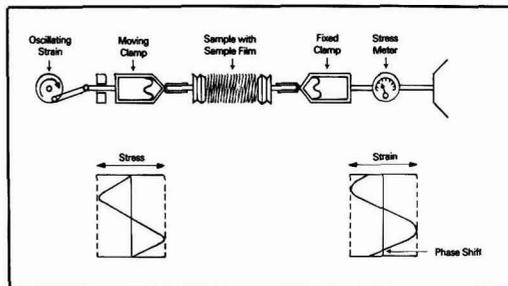


Figure 6—Dynamic spring analysis in 'rheovibron' system

Dynamic Spring Analysis

With dynamic spring analysis,⁶ the curing reaction can be followed in terms of changes in mechanical properties (E-modulus and loss factor). A schematic representation is given in Figure 6.

A piece of steel spring is clamped at both ends with the aid of screw thread pieces, which are screwed into the spring and provided with air channels to allow the escape of gaseous reaction products. After mounting the spring in a Rheovibron dynamic viscoelastometer, the wet coating is applied and the system is heated in less than one minute to the reaction temperature.

The enforced sinusoidal strain induces a sinusoidal stress. The stress level is a measure for the E-modulus (stiffness) of the combination coating plus spring. The stress-strain phase difference gives the loss factor $\tan \delta$.

Three polyester/HMMM/catalyst A, B, and C systems were studied with this method.

Relative rigidity (since the film's thickness is not known) against time in a 90°C curing experiment is given in Figure 7. The differences in reactivity are clearly seen: the increase in relative rigidity going along with the curing process starts first with the system containing free *p*-toluene sulphonic acid (A), then with the amine salt (B), and finally with the benziloximotosylate (C) systems. Noteworthy again is the relatively long induction period followed by a steep increase in relative rigidity with System C. In terms of practical application this would represent a good potlife at room temperature and relatively rapid curing at elevated temperatures. The order of reactivity $A > B > C$ is in agreement with the FTIR and TGA experiments.

With dynamic spring analysis one can also measure the loss factor $\tan \delta$, the maximum of which corresponds to the glass transition temperature (T_g). The T_g shifts to higher temperatures during the crosslinking process, due to restricted polymer chain movements.

The loss factor was determined as a function of temperature of a sample (polyester/HMMM/benziloximotosylate) which has been cumulatively exposed to increasing periods of curing at 90°C (Figure 8). Heating (at moderate temperatures 20–90°C) and cooling for the $\tan \delta$ analysis was done as quickly as possible to prevent additional curing during the measurement. From Figure 8 one can clearly see the T_g shift accompanying the curing process of the sample heated at 90°C for periods of 26, 40, 60, 120, and 165 minutes.

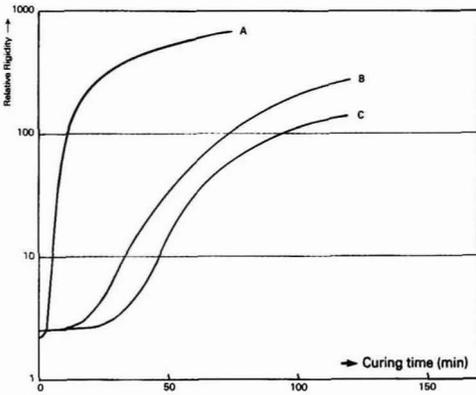


Figure 7—Rigidity by DSA of high solids films during baking at 90°C

The maximum of the loss curve also decreases, presumably due to the more complicated and varied types of chain movements in the network.

Viscosimetric Measurements With the "Sagging Balance"

The sagging balance (Figure 9) was designed by Overdiep to carry out viscosity measurements on high solid resins during curing.⁷ The liquid sample is spread on a Bonder plate in a thin (<200 μm) layer). The downward end of the plate is connected to an automatic recording balance and the whole system is placed in an oven. The angle of the plate can be controlled.

Severe sagging occurs with lower molecular weight high solid resins during the curing process. To control the rheology, Buter^{2,8-9} has introduced low molecular urea compounds as "sag control agents" (SCA's). These

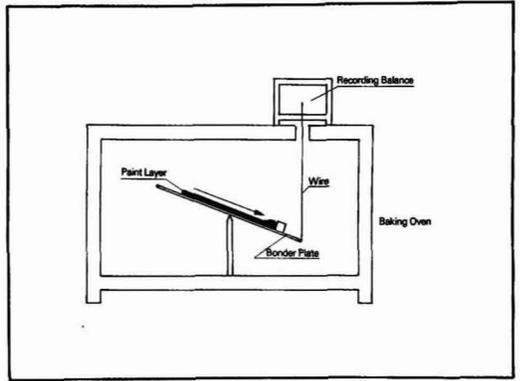


Figure 9—Sag balance mechanism

compounds disappear at higher temperatures (>100°C) by an acid catalyzed reaction, thus allowing for sufficient flow before curing is completed. The authors have investigated samples of the high solid polyester/melamine system mentioned before, containing a SCA and one of the four catalysts free p-toluenesulphonic acid (A), the diethanolamine salt of p-toluene sulphonic acid (B), benziloximesulphonic ester (C), and benzophenone oxime sulphonic ester (D), with the sagging balance.

The measurements are represented in Figure 10. The integrated fluidity ($\phi = \int \frac{dt}{\mu}$, t = time, μ = viscosity) calculated from the weight increase at the downward end of the plate using the formula given in reference (7) is plotted against time. The temperature increased to 130°C in about four minutes.

When using the free acid (A) as a catalyst, rapid build up of the chemical network prevents almost any sagging, whereas with the reactive catalyst (D) sagging is only slight.

There is almost identical behavior with catalysts B and C; C, however, has the longest induction period before sagging occurs. The D and A systems have insuffi-

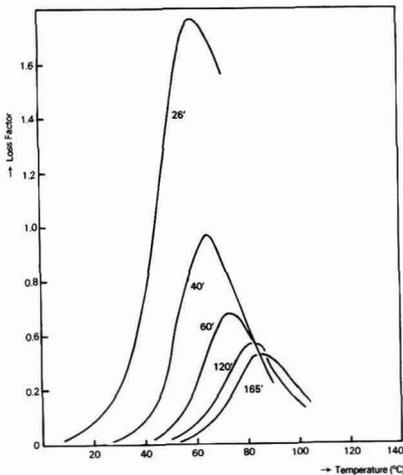


Figure 8—Loss factor and glass transition temperature by DSA of high solids films during curing at 90°C

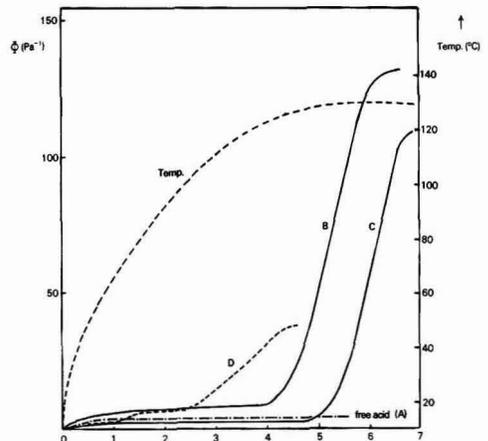


Figure 10—Sagging balance analysis of curing polyesters

cient flow, the chemical network formation occurs too quickly. But with B and C, flow is satisfactory before the increase of the integrated fluidity is levelled off by the crosslinking process.

The order of reactivity of the catalysts again is $A > D > B > C$ as was found previously above.

TEST DATA

To gain an impression of the performance of the high solid polyester/HMMM/benziloximetosylate resin in practical paint tests, the authors made a comparison between two samples: one, with the oximetosylate and the other with the usual amine/*p*-toluene sulphonic acid catalyst (Table 2).

Amounts of catalyst corresponding to 0.6–0.7 weight % of *p*-toluene sulphonic acid were used. The test results clearly indicate that the oximesulphonic esters are as effective crosslinking catalysts as the amine salts.

EXPERIMENTAL

Preparation of β -benzil Monoxime Tosylate

An amount of benzil (210 g) was suspended in 200 mL of ethanol and cooled to -5°C . To this suspension, 70 g of hydroxylamine hydrochloride were gradually added in portions at such a rate that the temperature of the reaction mixture did not exceed 0°C . Subsequently, 120 g of NaOH in 600 mL of water were added with vigorous stirring at a temperature of -5° to -3°C . After addition stirring was continued for 1.5 hours. The reaction mixture was diluted with one litre of cold water and the nonconverted benzil was filtered off. The filtrate was acidified with acetic acid and a white product precipitated. This product was washed with water and dried in air. The yield was 209 g of α -benzil monoxime. This was converted into the β -isomer by refluxing in benzene containing 15 g of active carbon. The yield of β -benzil monoxime was 152 g.

Of this substance, 67.5 g were dissolved in 150 mL of tetrahydrofuran and cooled at -5°C . Triethylamine (45.5 g) was added and subsequently, over a period of 45 minutes at -5° to 0°C , with vigorous stirring, 57 g of *p*-toluene sulphonyl chloride in 150 mL of tetrahydrofuran were added. After 30 minutes stirring, the mixture was poured into one litre of cold water and 40 mL of concentrated hydrochloric acid. The crude product was filtered and recrystallized from a mixture of three parts of ethanol and two parts of acetone to give 58.7 g of pure benzil monoxime tosylate (melting point 113.3° – 115.5°C).

Benzophenoneoximetosylate

To a cold solution of 12 g (0.3 mol) sodium hydroxide in 150 mL water, 59.1 g benzophenoxime (0.3 mol) in 900 mL of acetone were added. To this solution, 58.14 g (0.306 mol) *p*-toluenesulphonylchloride in 300 mL of acetone were added in about one hour with ice cooling. After the mixture had been stirred for an additional hour, it was poured into 900 mL of benzene. The benzene solution was washed repeatedly with ice water, dried, and then diluted with 600 mL of *n*-hexane. After cooling,

benzophenoneoximetosylate was (74.1 g, m.p. 72.9 – 82.3°C dec.) separated.

Preparation of *p*-Nitroacetophenone Oxime Tosylate

An amount of hydroxylamine hydrochloride (42 g = 0.6 moles) was dissolved in 90 mL of water and cooled in ice. Subsequently, 18 g of NaOH (0.3 moles) in 50 mL of water were added with proper stirring and cooling. To the resulting solution were added 49.5 g of *p*-nitroacetophenone in 200 mL of ethanol followed by heating under reflux. Next, to dissolve all solids, another 200 mL of ethanol were added. The solution was refluxed further for one hour. *p*-Nitroacetophenone oxime (51 g) was isolated by cooling. In the following step, 18 g of *p*-nitroacetophenone oxime in 300 mL of acetone were added at 0°C to a solution of 4 g of NaOH (0.1 mole) in 50 mL of water. Next, 19.4 g (0.102 moles) of *p*-toluene sulphonyl chloride in 100 mL of acetone were added, with stirring at 5° to 10°C . The solution was stirred for one hour at 0° to 5°C and poured into 300 mL of benzene, washed four times with 125 mL of cold water, and dried. By distilling off the solvent a yellow residue was obtained, which was recrystallized from a mixture of benzene and hexane (1:1), yield 22.4 g m.p. 123.4° to 125.4°C .

Preparation of α -Oximinocaproic Acid Ethyl Ester Tosylate

The mixture of 21.6 g of (0.1 mole) of *n*-butyl-diethyl malonate and 15.6 g (0.133 moles) of iso amyl nitrite were added. A solution of 2.3 g of Na (0.1 mole) in 45 mL of ethanol (anhydrous) with stirring, over a period of 1.5 hours at -10° to -8°C was added. Stirring was continued for 16 hours at -10°C . After evaporation and further treatment with cold water, extraction with ether, acidification, and a second extraction with ether, 13.8 g of α -oximino-caproic acid ethyl ester were obtained.

Of this, 5.3 g (0.03 moles) were dissolved in 25 mL of tetrahydrofuran, cooled to -10°C . To this solution were added 4.55 g (0.045 moles) of triethylamine, and in one hour at -10° to 0°C , a solution of 5.7 g (0.03 moles) of *p*-toluene sulphonyl chloride in 25 mL of tetrahydrofuran were added. After one hour stirring at 0°C , the reaction mixture was stored for 16 hours at 4°C and subsequently poured into cold dilute hydrochloric acid extracted with diethyl ether and dried. After evaporating the solvent the product (3.6 g) was recrystallized from ethanol m.p. 43.2° – 46.8°C .

Kinetic Measurements

Rates of decomposition were measured in neopentyl alcohol/dioxide mixtures (generally 4:3). Sealed tubes containing 0.5 mL portions of solutions of oximetosylate were placed in an oil bath thermostat and withdrawn at appropriate times.

The solutions were then diluted with a constant volume of methanol/water (80:20) which was also used for subsequent HPLC-analysis on a Zichrosorb 10RP18 column. A plot of log (peak area) of oximetosylate vs time gave straight lines indicating first order kinetics. Experiments

over the full range of neopentyl alcohol concentrations (0–100%) gave nearly identical rate constants. The decomposition of the oximatosylates investigated therefore proceeds via a monomolecular dissociation as the rate-determining step.

ACKNOWLEDGMENT

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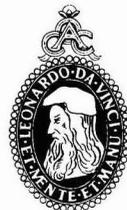
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Interrelationships Between Pigment Surface Energies And Pigment Dispersions In Polymer Solutions

G. Dale Cheever and John C. Ulicny
General Motors Research Laboratories*

In the manufacture of paints, plastics, elastomers, and adhesives, good dispersion of pigments and other fillers is essential in producing a quality product. Although many theoretical examples exist in the literature describing the factors required for dispersion, a practical method is needed to measure these pigment and solution properties and relate them to dispersion.

The critical surface tension of wetting (γ_C) of 18 commercial pigments representing hiding, extender, colored, white, organic, and inorganic types were measured with a capillary flow technique. A relationship was found between pigment dispersion and the γ_C values of eight representative pigments and the surface tension of an aqueous polyvinyl alcohol solution.

INTRODUCTION

Pigments are finely divided powders which are essentially insoluble in the media in which they are suspended or dispersed. Dispersion is defined as the separation of the individual pigment particles by the suspending liquid and maintaining this dispersed or deflocculated state. The requirements for a pigment are: ease of dispersion, color and opacity, brightness, gloss and gloss retention, good flow, lightfastness, and weathering resistance. Thus, good pigment dispersion is a necessary prerequisite for a successful paint. Three separate stages have been recognized in the process of dispersing pigments in polymer liquids.¹⁻⁴

Wetting—The pigment-air interface is replaced by a pigment-liquid interface. For complete wetting to occur, adsorbed air and other contaminants must be essentially removed;

Mechanical Disruption—The powder is thoroughly mixed with the liquid, and the agglomerates and large particles present are broken down by mechanical energy into the desired size; and

Stabilization—The desired deflocculated and dispersed conditions shown above must be maintained for periods of time dictated by the end use of the pigment-liquid system.

These three stages which are needed to form a stable dispersion may occur very rapidly, but are distinct in character. Parfitt² states that it is questionable whether the three stages given above ever reach completion in practical paint systems. Also, for some end uses, partially flocculated pigment-resin systems may actually be needed (Table 1).

The goal of the research reported in this paper is to express pigment-polymer interactions in terms of basic parameters, such as surface energies and relate dispersion in terms of these surface energies. Surface energies and interactions were measured in pigment capillary beds. This experimental approach will now be examined.

Pigment Capillary Beds

The flow of standard liquids was measured under capillary pressures generated by a pigment bed contained within a vertical glass tube. This method was adopted from work reported by Crowl.⁵ Analyses of the data gave the advancing contact angle, θ , of the flowing liquids on the pigment particles. Utilizing the work of Zisman,⁶ surface energies for each pigment were obtained from the measured $\cos\theta$ values.

Presented by Dr. Cheever at the 60th Annual Meeting of the Federation of Societies for Coatings Technology in Washington, D.C., November 3, 1982.

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Table 1—Paint Properties Resulting From the State of Pigment Dispersion

Performance Properties	Pigment Dispersion State	
	Deflocculated	Flocculated
Rate of sedimentation	Slow	Rapid
Sedimentation volume	Low	High
Texture of sediment	Poor (dense; hard to redisperse)	Good (soft; easily redispersed)
Flow	Excellent	Poor
Leveling	Excellent	Poor
Sag resistance	Poor	Good
Hiding (opacity)	Good	Poor
Color development	Good	Fair
Brushing	Poor (drags)	Good (easy)
Gloss	Good	Poor
Film durability	Good	Fair

The authors have developed a second method of analyzing flow data in pigment capillary beds from mass transport phenomena described by Bird, Stewart, and Lightfoot (BSL).⁷ These two methods will be compared. They share a common goal, namely to compute $\cos\theta$ for each liquid-pigment combination.

Pigment Surface Energies—Crowl Methods

It was shown⁵ that

$$\frac{\ell^2}{t} = \frac{(r/k^2) \gamma_{LV} \cos\theta}{2\eta} \tag{1}$$

where

- ℓ = length of liquid flow (cm) in capillary-Crowl⁵ method,
- t = time of flow (sec),
- $\frac{\ell^2}{t}$ = slope of plots of ℓ^2 vs t (cm^2/sec),
- γ_{LV} = liquid-vapor surface tension (mN/m),
- r = radius of capillary (cm),
- θ = advancing contact angle (degree),
- k = a factor to estimate the tortuous path of the capillaries, and
- η = absolute liquid viscosity (mPa·s).

When a flowing liquid exhibits complete wetting, i.e., $\cos\theta = 1$, a value of (r/k^2) can be calculated from equation (1). The liquid surface tension and viscosity are assumed to be bulk values. Once (r/k^2) has been calculated for a given pigment, $\cos\theta$ can be obtained for other liquids of interest for which $\cos\theta < 1$. The assumptions are:

- (1) Liquid flow is laminar.
- (2) Pigment packing for the same pigment bed is constant, i.e., (r/k^2) is constant.
- (3) Gravity is negligible.

The separate quantities, r and k , would have to be approximated from independent measurements which are difficult to obtain and are subject to questions of interpretation, e.g., what is the capillary radius and the path of flow in commercial pigments.

Pigment Surface Energies—This Work

The main departure of the present work from that reported by Crowl⁵ is that the derived $\cos\theta$ values can be

obtained from readily measurable pigment characteristics. It is shown in Appendices A and B that

$$\frac{\ell^2}{t} = \frac{2\gamma_{LV} \bar{D}_p \cos\theta}{25\eta} \frac{\epsilon^2}{(1-\epsilon)} \tag{2}$$

where

- ℓ = flow distance in packed bed (cm) - present work,
 - \bar{D}_p = mean particle diameter (cm), and
 - ϵ = pigment void fraction
- $$= \frac{\text{volume of the voids}}{\text{volume of the pigment bed}}$$

Thus, ℓ^2/t is the same numerical value as ℓ^2/t , γ_{LV} and η are used in both methods. The pigment void fraction, ϵ , is measured from the pigment weight, height, and bulk density.

The pigments are all examined in a scanning electron microscope, and the measured particle size distribution is compared with the mean particle diameter, \bar{D}_p , determined at $\cos\theta = 1$, equation (2). The assumptions are:

- (1) Liquid flow is laminar.
- (2) The void fraction is constant for a given pigment.
- (3) The particle size distribution with the resulting \bar{D}_p is constant.
- (4) Gravity is negligible.

Pigment Surface Energies— γ_C

Considering the Zisman⁶ relationships needed for equations (1) and (2),

$$\cos\theta = 1 + b(\gamma_C - \gamma_{LV}), \text{ valid for } \gamma_{LV} \geq \gamma_C. \tag{3}$$

where

- b = slope of the Zisman plot, i.e., a plot of $\cos\theta$ vs. γ_{LV} , and
- γ_C = critical surface tension of wetting.⁶

Measurement Criteria of Pigment Dispersions

The degree of pigment dispersion and flocculation were measured by the pigment sedimentation volume technique.^{1-4,8,9} This technique is illustrated in Figures 1 and 2. The consequences of flocculated and deflocculated pigment suspensions on paint properties are summarized in Table 1.^{2,3,8} Thus, deflocculated pigment systems give small, compacted, hard-to-redisperse sediments, and cloudy solutions. Flocculated pigment systems result in large, loose, easy-to-redisperse sediments, and clear liquids. For deflocculated pigment systems, there is slow, persistent settling to a hard-packed cake. The pigment particles and aggregates have settled essentially individually. The cloudy supernatant resulted from the smaller particles remaining in solution. However, in flocculated pigment systems, there is rapid settling to a soft, disorder cake. The clear supernatant resulted from the sweeping of essentially all pigment particles from the solution by the settling flocculates. It is seen that, the relative volume of the sediment, its compactness, and the clarity of the supernatant as a function of time reveal the degree of the pigment dispersion and deflocculation.

Patton³ defines the compactness of sediment in terms of a terminal sediment volume (TSV). The quantity TSV is defined as the ratio of the volume of the final

compacted solids produced by gravitational settling to the theoretical dry solids volume determined from the bulk density. The smaller the values of TSV the more compact is the sediment and the less has been the degree of flocculation in the solution.

The examination of *Table 1* shows that the state of the pigment dispersion can markedly affect paint properties. Every paint system is a balance between a number of properties, with the result that the coating application, use, and durability criteria dictate which of these properties are paramount in importance. To achieve desired appearance and durability, compromises may occur in handling and application characteristics. The flocculation and sedimentation behavior, thus, can give both desirable and undesirable end-use properties.

EXPERIMENTAL

Pigments

The pigments used in this study, their classification, sources, and bulk density are given in *Table 2*. The pigment classifications follow the criteria established by Madson¹⁰ and Fuller and Love.¹¹ In addition to the single pigments, the gray and red primer surfacer and 50:50 (volume/volume) barytes/ calcium carbonate pigment blends are given in *Table 2*.

The pigments were used in the as-received condition. The primer surfacers are blends of pigments which are used in solvent-based primer surfacers. The gray primer surfacer pigment blend consisted primarily of barytes and clay, with small amounts of lampblack and titanium dioxide. The red oxide primer surfacer pigment blend also consisted primarily of barytes and clay but with small amounts of red iron oxide, basic lead silico chromate, and titanium dioxide.

The four titanium dioxide pigments represent both anatase and rutile crystallographic forms. Titanium dioxide and carbon black are used in paints, plastics, and elastomers.

The three FMC pigments are microcrystalline cellulose products which are used in paints, lotion, gels, as food additives, and as foam controllers.

Zinc phosphate is a corrosion inhibitor pigment for primers.

Solvents

The solvents utilized in this study to characterize pigment surfaces are listed in *Table 3*. The sources of the liquids, their purity, surface tension, and viscosity are given. The surface tension and viscosity values presented were taken from the literature.^{12,13} Our surface tension values, obtained by the duNouy ring method, agreed satisfactorily with these literature values.

Pigment Capillary Beds

The apparatus used for measuring the capillary flow of liquids in pigment beds is shown in *Figure 3*. A weight of pigment was placed in a 10 × 30 mm Pyrex tube fitted with a surgical cotton plug. The pigment was packed to a

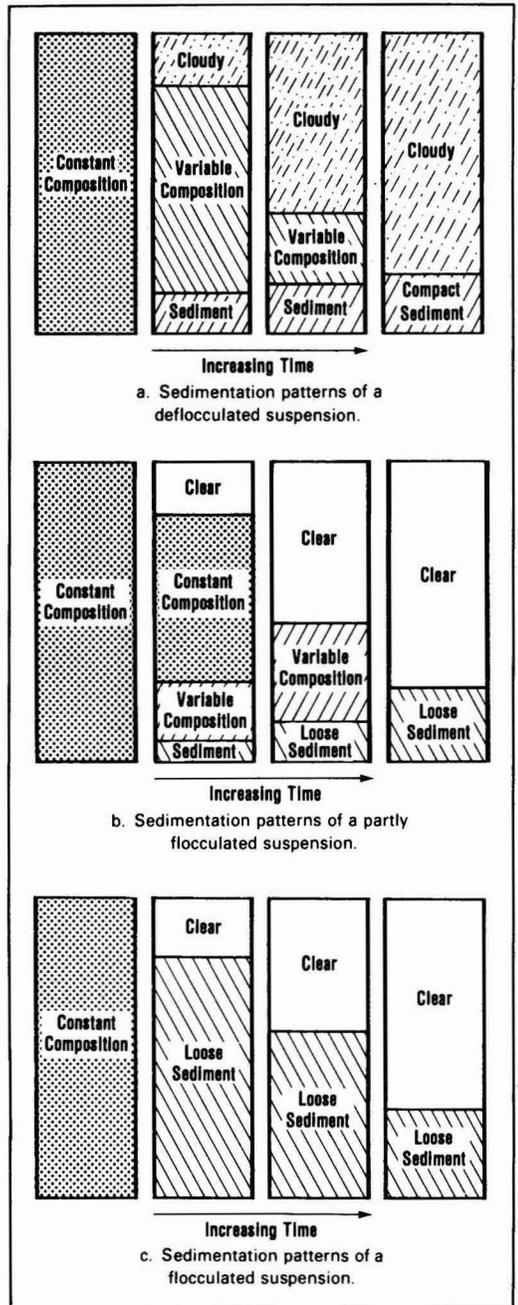


Figure 1—Schematic representations of sedimentation patterns as a function of degree of dispersion²

standard height with a hand vibrator engraver. The inverted tube and machinist rule were placed in contact with the liquid and the liquid front rising through the bed was measured as a function of time. The initial liquid was

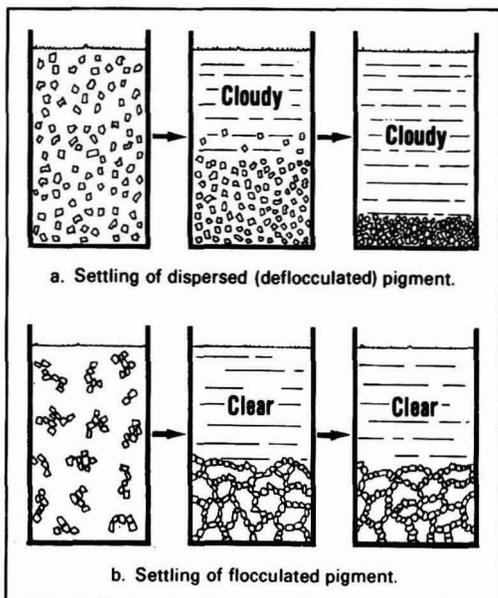


Figure 2—Schematic illustration of the two basic types of pigment settling³

toluene or hexane which gave complete wetting ($\cos\theta = 1$). These liquids gave the packing constant (r/k^2) for equation (1) and \bar{D}_p for equation (2). A new sample of the same pigment was packed to the same height and the flow rate of the next liquid determined. This procedure was repeated to obtain the flow rate for each liquid-pigment combination of interest.

Preparation of Dispersions

RESINS: Solutions of Gelvatol® 20/30 grade polyvinyl alcohol (PVA) were prepared in deionized-distilled water at a concentration of 11% by weight (11 g resin per 100 g solution). Water was heated to boiling, the required PVA resin added, and stirred with an air-driven motor for 30 minutes. The heat was turned off, and the solution stirred at room temperature for 24 hours.

The physical properties of the polyvinyl alcohol resin and the resulting aqueous solutions are given in *Table 4*. Liquid surface tensions were measured with a Fisher Scientific recording automatic tensiometer, using the duNouy platinum ring method with appropriate corrections. Density measurements were obtained with the Westphal hydrostatic technique.¹⁴ Viscosities were obtained in a kinematic viscometer.¹⁵ The resulting kinematic viscosities were converted to absolute viscosities by multiplying by the solution density.

PREPARATION OF RESIN-PIGMENT DISPERSIONS: Dispersions of the pigments described in *Table 2* were prepared

Table 2—Identification of Pigments

Classification	Source	Name	Bulk Density, ^a (g/mL)
White hiding pigments (titanium dioxide)	Glidden Pigments Chemical/Metallurgical Div., SCM Corp.	RGMC ^b	3.9
		R-CL6 ^c	4.0
		R-88S ^c	4.1
		R-77 ^c	4.1
White extender pigments	Inmont Corp.	Talc	2.7
		Clay	2.6
		Barytes	4.3
		Calcium Carbonate	2.7
		English Micro Mica 3000	2.7
		4X Mineralite Mica	2.7
		50:50 V/V Barytes/Calcium Carbonate	3.5
Colored inorganic pigment	Inmont Corp.	Red Iron Oxide	4.9
Black pigment	Inmont Corp.	Carbon Black	1.5
Primer surfacers	Commercial Grades	Gray P/S	3.7
		Red P/S	3.7
		Inorganic pigment	Fisher Scientific Co.
Microcrystalline	Food Machinery Corp. (FMC)	AVICEL RC-591	1.5
		AVICEL PH-101	1.5
Cellulose		AVILOID CT-581	1.5

(a) Data obtained from pigment suppliers.

(b) Titanium dioxide, anatase crystal form.

(c) Titanium dioxide, rutile crystal form.

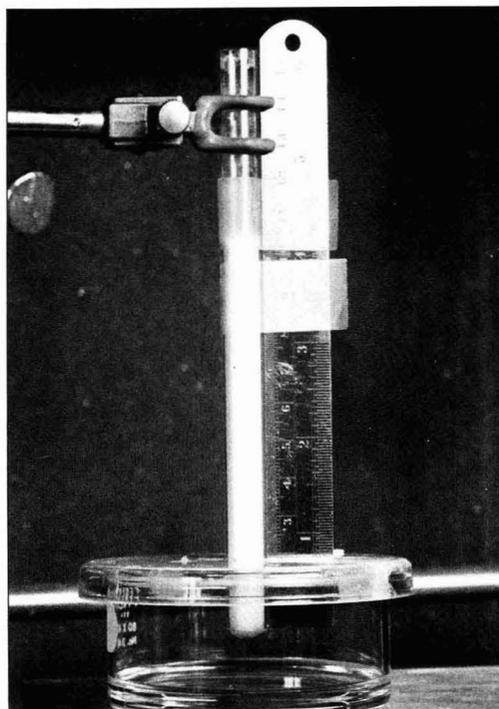
in 11% PVA solutions contained in 250-mL polypropylene bottles. The required amounts of pigment and resin solutions were added to give pigment to binder ratios (g pigment/100 g binder resin or P/B) of 100, 200, and 300. The pigments and resins remained undisturbed for 30 mins (30 min soak time). Next, 3-mm diameter steel shot was added to correspond to 40% by volume of the total pigment-resin-solvent blend. The dispersions were shaken 30 min on a paint shaker, drained, and set aside.

MEASUREMENT OF PIGMENT SEDIMENTATION VOLUME^{2,3}: The pigment sediment heights were measured and converted into sedimentation volumes by calibrating the 250-mL polypropylene bottles with known volumes of distilled-deionized water. The sedimentation volumes were measured as a function of time, and TSV values

Table 3—Solvents Used in Characterizing Pigment Surfaces

Liquid	Source	Purity	γ_{LV} 25°C (mN/m)	Viscosity at 25°C (mPa·s)
Benzyl alcohol	Merck	Reagent	39.0	5.8
Tritolyl phosphate	Eastman	Tech.	40.4	
Methylene iodide	Eastman	98% Min.	51.1	2.6
Nitromethane	Eastman	Spectro.	36.0	0.62
Glycerin	Baker	Reagent	63.3	954
Water	^a	^a	72.0	1.0
Ethylene glycol	Baker	Reagent	47.7	19.9
Chlorobenzene	Matheson, Coleman and Bell	98% Min.	33.6	0.84
Formamide	Baker	Reagent	58.4	3.3
Furfural	Baker	Baker An- alyzed	43.5	1.49
Aniline	Mallinck- rodt	AR	43.1	3.8 (15°)
1-Bromonaphthalene	Baker	Baker	43.9	6.0
Heptane	Aldrich	Spectro.	21.2	0.39
Decane	Aldrich	Spectro.	24.6	0.92
Isopropyl alcohol	Baker	Baker An- alyzed	21.2	1.77
Toluene	Baker	Spectro.	28.0	0.56
Hexane	Baker	Spectro.	17.4	0.29
Benzyl ether	Aldrich	99%	40.6	4.4
Nitrobenzene	Baker	Baker An- alyzed	43.6	1.9
Methanol	Matheson, Coleman and Bell	Reagent A.C.S.	22.1	0.50

(a) The sample was distilled from deionized water in a Barnstead water still.


Figure 3—Apparatus used to measure flow of liquids in pigment beds

Pigment Capillary Beds—Crowl Method⁵

Values of the pigment bed packing constants, r/k^2 , [equation (1)] for each pigment were determined with toluene or hexane. These solvents completely wetted the pigment surfaces; therefore, $\cos\theta = 1$. They are presented in Table 5. The rate of penetration of a given liquid was measured, plotted as l^2 vs t , and the slope l^2/t , determined. From a knowledge of r/k^2 , γ_{LV} , and η , the value of $\cos\theta$ was determined for the liquid and pigment of interest [equation (1)]. With some liquids and pigments, the slope was measured from the l^2 vs t curves after one minute. This time was required for the liquid to saturate the cotton plug and then proceed up the pigment bed. The slope remained constant for 10–12 minutes. This constant slope was used in equation (1).

were calculated by obtaining the theoretical dry solids volume from the total pigment present and the bulk pigment density (Table 2). This calculation assumes negligible volume contribution from the polymers themselves and negligible pigment left in solution. These assumptions were verified with pigment solutions which had settled for long time periods. The solids concentrations in the solutions were 11%, which are the concentrations of the resin-solvent solutions alone. In general, sedimentation took considerable time to reach completion and was asymptotic in nature. Depending on the pigment, complete sedimentation required 10 to 400 days. Viscosity differences are assumed to be negligible for the times requiring complete sedimentation.

RESULTS AND DISCUSSION

Pigments

As-received pigment samples were examined in a JEOL JSM-U3 scanning electron microscope (SEM) or an ISI Model DS130 unit. Photomicrographs are shown in Figures 4-7 for RGMC and R-CL6 TiO₂, talc and clay, barytes and red iron oxide, and gray and red primer surfaces, respectively.

Table 4—Physical Properties of Polyvinyl Alcohol Polymers and Aqueous Solutions

Resin	\bar{M}_w	ρ (g/mL)	11% w/w solutions ^a		
			γ_{LV} (mN/m)	η (mPa·s)	ρ (g/mL)
PVA	10,000 ^b	1.20 ^b	42.4	42.9	1.0279

(a) Measurements made at 23–24°C.
(b) Data obtained from resin supplier.

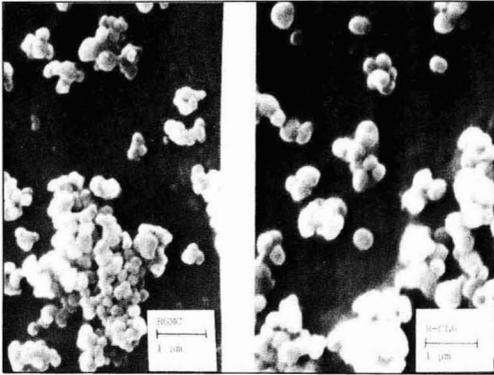


Figure 4—Scanning electron micrographs of RGMC and R-CL6 TiO₂ pigments as received

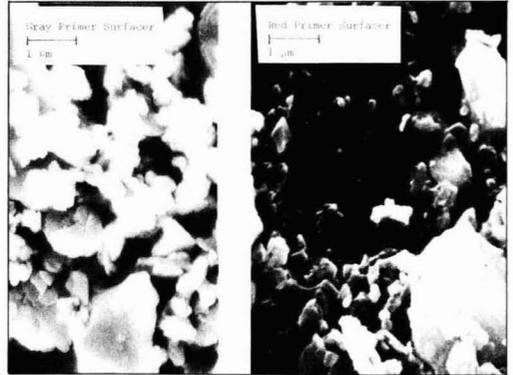


Figure 7—Scanning electron micrographs of gray and red primer surfacer pigments as received

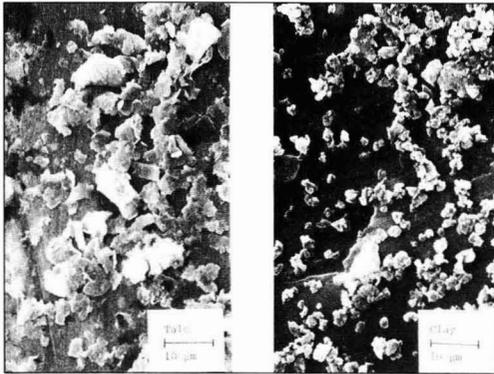


Figure 5—Scanning electron micrographs of talc and clay pigments as received

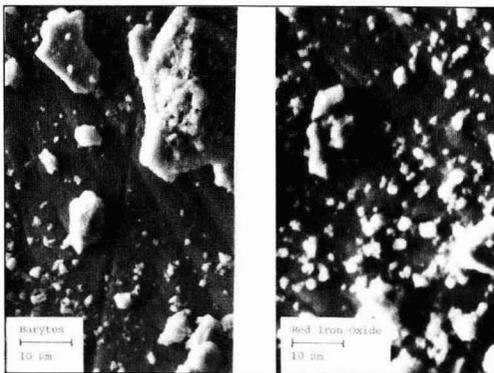


Figure 6—Scanning electron micrographs of barytes and red iron oxide pigments as received

The Zisman plots of $\cos\theta$ vs γ_{LV} for the pigments were made, and the resulting γ_C values ($\cos\theta = 1$) are given in *Tables 6 through 10*. In *Tables 6–10*, the wettability properties (γ_C) of the pigment mixtures appeared to be dominated by a single pigment. This effect occurred in the mixtures of barytes and calcium carbonate and in the gray and red primer surfacers. The liquid-solid interactions measured were essentially those of barytes.

Pigment Capillary Beds—New Method

The Crowl flow data were examined with the new approach, and the values of \bar{D}_p were calculated from equation (2). A summary of the flow rates, L^2/t , and the values of ϵ , \bar{D}_p , (r/k^2) , and γ_C are given in *Table 11* for eight representative pigments. The values of \bar{D}_p and (r/k^2) were all calculated from toluene L^2/t flow rates except for talc which were obtained from aniline L^2/t flow rates. Toluene caused separation in the talc pigment bed.

Comparison of \bar{D}_p Values Obtained From Flow and Microscopy Measurements

Comparing *Table 11* with *Figures 4–7*, shows that there is favorable agreement between \bar{D}_p values obtained from flow tests and those measured in the SEM. An exception is talc which gives a flow \bar{D}_p of $0.12 \mu\text{m}$, but the average particle size measured is $2\text{--}4 \mu\text{m}$. The reason for the discrepancy is not known.

Terminal Sediment Volumes

The TSV values for the pigment-PVA systems at P/B ratios of 100, 200, and 300 are given in *Table 12*. The TSV results will be discussed later in this paper.

Surface Energy-Pigment Dispersion Relationships

An empirical approach was taken in relating surface energies and pigment dispersion. It was recognized that

Table 5—Summary of Measured Pigment Packing Constants

Pigment	Sample Number	r/k^2 cm
Carbon black	8-92	4.72×10^{-6}
Barytes	8-113	1.28×10^{-5}
Calcium carbonate	8-131	2.74×10^{-5}
Talc	8-148	1.00×10^{-5}
Red iron oxide	9-9	1.50×10^{-5}
R-88S	9-25	2.02×10^{-5}
R-CL6	9-39	2.17×10^{-5}
RGMC	9-55	3.33×10^{-5}
R-77	9-67	4.22×10^{-5}
BaSO ₄ /CaCO ₃ 50-50 blend	9-140	1.59×10^{-5}
Gray primer surfacer blend	10-51	4.10×10^{-5}
Red iron oxide primer surfacer blend	9-108	2.48×10^{-5}
Clay	9-122	2.28×10^{-5}
English Mirco mica 3000	10-35	2.36×10^{-5}
4X Mineralite mica	10-61	4.21×10^{-5}
Aviloid CT-581	10-24	6.59×10^{-5}
Avicel PH-101	10-9	1.60×10^{-4}
Avicel RC-591	9-150	7.62×10^{-5}

Table 7—Surface Characterization of Three Microcrystalline Pigments and Clay

Pigment	Liquid	γ_{LV} (mN/m)	$\cos\theta$	γ_C (mN/m)
Aviloid CT-581	Formamide	58.4	0.00	47.5
	Methylene iodide	51.1	0.763	
	Ethylene glycol	47.7	0.908	
Avicel PH-101	Water	72.0	0.340	40.0
	Formamide	58.4	0.250	
	Methylene iodide	51.1	0.870	
	Ethylene glycol	47.7	0.577	
	Furfural	43.5	0.836	
	Benzyl ether	40.6	1.00	
Avicel RC-591	Formamide	58.4	0.569	43.9
	Methylene iodide	51.1	0.747	
	Ethylene glycol	47.7	0.886	
Clay	Water	72.0	0.172	29.2
	Formamide	58.4	0.209	
	Methylene iodide	51.1	0.468	
	Furfural	43.5	0.643	
	Aniline	43.1	0.430	

at least three factors were needed to be considered: pigment dispersion, pigment surface energy, and solution surface energy. Pigment dispersion was chosen because of its importance in affecting paint properties (Table 1). This property was measured by the TSV values which are sediment properties, where the larger the TSV values the larger the degree of pigment flocculation in the solution. For pigment and solution surface energies, the critical surface tension of wetting, γ_C , was chosen because

Zisman⁶ has shown that γ_C is a useful index of surface wettability. This property was discussed earlier in this paper as one of the key elements in displacing adsorbed gases for obtaining stable pigment dispersions. Also, values of γ_C have been measured for many surfaces and are available in the chemical literature. Liquid surface tension was used as the factor for the liquid surface energy because: it is a universal factor in describing liquid-solid interaction phenomena and has been

Table 6—Surface Characterization of Four Titanium Dioxide Pigments

Pigment	Liquid	γ_{LV} (mN/m)	$\cos\theta$	γ_C (mN/m)
RGMC anatase titanium dioxide	Water	72.0	0.007	42.9
	Formamide	58.4	0.079	
	Methylene iodide	51.1	0.773	
	Aniline	43.1	1.00	
R-77 Rutile titanium dioxide	Water	72.0	0.180	35.6
	Formamide	58.4	0.080	
	Methylene iodide	51.1	0.270	
	Ethylene glycol	47.7	0.100	
	Benzyl ether	40.6	0.765	
R-88S Rutile titanium dioxide	Water	72.0	0.180	35.9
	Formamide	58.4	0.220	
	Methylene iodide	51.1	0.262	
	Aniline	43.1	0.736	
R-CL6 Rutile titanium dioxide	Water	72.0	0.388	41.1
	Formamide	58.4	0.594	
	Methylene iodide	51.1	0.614	
	Ethylene glycol	47.7	0.868	
	Aniline	43.1	1.00	

Table 8—Surface Characterization of Barytes, Calcium Carbonate, a Blend of Barytes and Calcium Carbonate, and Red Iron Oxide

Pigment	Liquid	γ_{LV} (mN/m)	$\cos\theta$	γ_C (mN/m)
Barytes	Water	72.0	0.100	43.4
	Formamide	58.4	0.062	
	Methylene iodide	51.1	0.323	
	Ethylene glycol	47.7	0.743	
	Nitrobenzene	43.6	1.00	
Calcium carbonate	Water	72.0	0.24	25.1
	Methylene iodide	51.1	0.47	
	Aniline	43.1	0.35	
	Benzyl alcohol	39.0	0.41	
Barytes/Calcium carbonate	Formamide	58.4	0.068	43.3
	Methylene iodide	51.1	0.530	
	Ethylene glycol	47.7	0.677	
Red iron oxide	Water	72.0	0.28	28.0
	Formamide	58.4	0.26	
	Methylene iodide	51.1	0.40	
	Aniline	43.1	0.45	
	Benzyl alcohol	39.0	0.35	

Table 9—Surface Characterization of Talc, Carbon Black, and Two Mica Pigments

Pigment	Liquid	γ_{LV} (mN/m)	$\cos\theta$	γ_C (mN/m)
Talc	Water	72.0	0.00	48.0
	Formamide	58.4	0.569	
	Methylene iodide	51.1	0.838	
	Ethylene glycol	47.7	1.00	
	Aniline	43.1	1.00	
Carbon black	Water	72.0	0.073	40.0
	Methylene iodide	51.1	0.696	
	Benzyl alcohol	39.0	1.00	
4X Mineralite mica	Water	72.0	0.370	45.7
	Formamide	58.4	0.635	
	Methylene iodide	51.1	0.776	
	Ethylene glycol	47.7	1.00	
English Micro mica 3000	Water	72.0	0.521	36.1
	Formamide	58.4	0.741	
	Formamide	58.4	0.790	
	Methylene iodide	51.1	0.681	
	Ethylene glycol	47.7	0.767	
	Benzyl ether	40.6	0.970	

Table 10—Surface Characterization of Combination Pigment Blends Used in Gray and Red Iron Oxide Primer Surfacer

Pigment	Liquid	γ_{LV} (mN/m)	$\cos\theta$	γ_C (mN/m)
Combination for gray primer surfacer	Formamide	58.4	0.047	39.6
	Methylene iodine	51.1	0.334	
	Ethylene glycol	47.7	0.559	
Combination for red iron oxide surfacer	Water	72.0	0.083	35.8
	Formamide	58.4	0.083	
	Methylene iodine	51.1	0.326	
	Benzyl ether	40.6	0.767	

Table 11—Summary of Pigment Bed Flow Data

No.	Pigment	ϵ	L_2^2/t^a cm ² /sec	$\bar{D}_p, \mu\text{m}$	$(r/k^2), \mu\text{m}$	γ_C (mN/m)
1	RGMC	0.79	8.31×10^{-2}	0.70	0.33	42.9
2	R-CL6	0.71	5.40×10^{-2}	0.76	0.22	41.1
3	Talc ^b	0.86	0.55×10^{-2}	0.12	0.10	48.0
4	Clay	0.77	5.68×10^{-2}	0.57	0.23	29.2
5	Barytes	0.48	3.18×10^{-2}	1.82	0.13	43.4
6	Red iron oxide	0.62	3.74×10^{-2}	0.91	0.15	28.0
7	Gray primer surfacer	0.70	10.24×10^{-2}	1.56	0.41	39.6
8	Red primer surfacer	0.67	6.18×10^{-2}	1.12	0.25	35.8

(a) Measured flow rates with toluene, assume $\cos\theta = 1$.(b) Measured flow rates with aniline, assume $\cos\theta = 1$.**Table 12—Terminal Sediment Volumes Of Suspensions in Polyvinyl Alcohol Solutions**

No.	Pigment	Terminal Sediment Volumes			
		P/B	100	200	300
1	RGMC	4.79	8.91	7.54	
2	R-CL6	6.14	5.70	6.09	
3	Talc	3.64	3.00	2.78	
4	Clay	5.42	4.65	7.06	
5	Barytes	4.22	3.32	3.01	
6	Red iron oxide	8.41	6.96	6.05	
7	Gray P/S	4.09	2.85	2.59	
8	Red P/S	4.09	3.88	3.50	

measured for many systems and reported. The absolute value of this difference, $|\gamma_C - \gamma_{LV}|$, was used because, in practice, the difference can be both negative and positive. Table 13 lists a summary of the γ_C, γ_{LV} , and $|\gamma_C - \gamma_{LV}|$ values for pigment dispersions in 11% PVA solutions. The γ_{LV} values were measured in P/B slurries of 100, and these values of γ_{LV} were used for all the pigment-resin concentrations. To relate pigment dispersion to pigment and solution surface energies, the results are plotted in Figures 8 and 9 for the polyvinyl alcohol-pigment dispersions. Referring to Figures 8 and 9, there are minima in the PVA-pigment TSV values (maxima in pigment dispersion) as a function of $|\gamma_C - \gamma_{LV}|$.

The relevance of these results is: relationships have been found to exist between pigment surface energies measured by basic flow equations of simple liquids in pigment capillary beds and dispersion properties in a polymer solution. It follows, then, that desired flocculation and coating properties (Table 1) can be generated by the proper choice of pigment surface energies, γ_C , and the polymer-solvent surface energies, γ_{LV} (Figures 8 and 9). The practical application of this work is that in an important aqueous polymer solution (polyvinyl alcohol in water), pigments giving the best dispersion can be selected.

Table 13—Surface Energies of Pigments And Pigment-Resin Dispersions

No.	Name	γ_C , (mN/m)	Pigment-PVA Resin Dispersions ^a	
			γ_{LV} , (mN/m)	$ \gamma_C - \gamma_{LV} $, (mN/m)
1	RGMC	42.9	42.4	0.5
2	R-CL6	41.1	41.7	0.6
3	Talc	48.0	43.3	4.7
4	Clay	29.2	41.1	11.9
5	Barytes	43.4	37.8	5.6
6	Red iron oxide	28.0	41.2	13.2
7	Gray P/S	39.6	42.0	2.4
8	Red P/S	35.8	42.0	6.2

(a) The liquid surface tensions, γ_{LV} , were measured in pigment-resin dispersions with pigment concentrations of P/B 100.

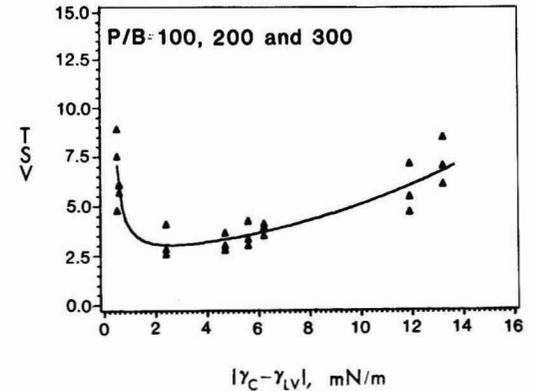
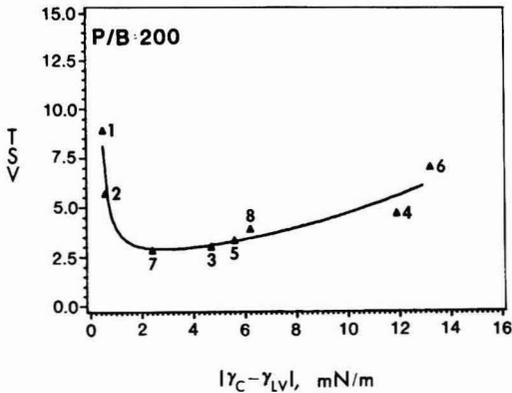
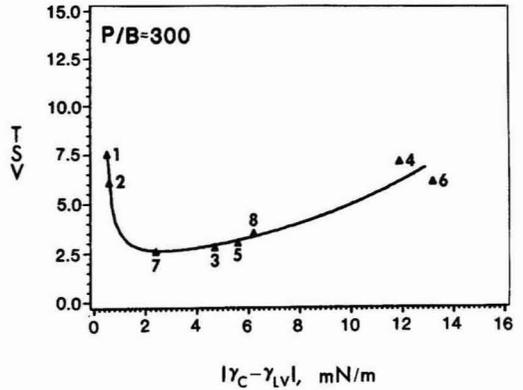
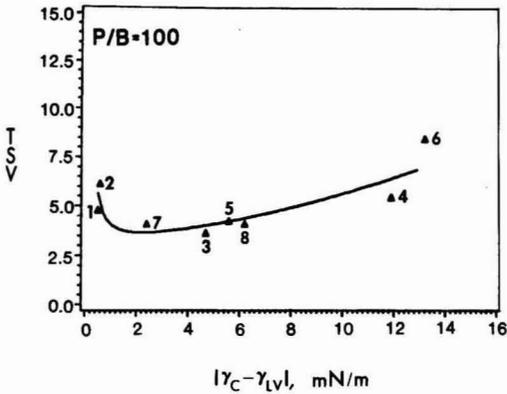


Figure 8—Relationships between sediment volume and pigment/solution energies at P/B values of 100 and 200 for polyvinyl alcohol dispersions

Figure 9—Relationships between sediment volume and pigment/solution energies at P/B values of 300 and a combination of 100, 200, and 300 for polyvinyl alcohol dispersions

SUMMARY

The capillary bed liquid rise technique was successfully applied to 18 pigments or pigment blends to obtain surface-liquid interactions and energies. Flow equations of simple liquids in pigment capillary beds were derived from basic mass transport relationships. The analyses of these flow data gave the liquid-pigment contact angles which are measures of the energy of interaction between the liquids and the pigments. For a wide variety of pigments, dispersion was expressed as a function of the terminal sediment volume (TSV) in a model aqueous polymer solution, polyvinyl alcohol/water. Maxima in dispersion are clearly indicated for the polyvinyl alcohol/water system with particular combinations of pigment and resin solutions. Thus, success has been achieved in relating fundamental pigment and solution surface energies to an important paint property, namely, dispersion in a polyvinyl alcohol solution. Also, pigment mixtures can be formulated such that liquid-pigment interactions are dominated by a specific pigment in the

mixture. These approaches show promise as aids in the preparation of paints.

ACKNOWLEDGMENTS

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APPENDIX A

DERIVATION OF WASHBURN EQUATION

The Washburn equation¹⁶ is recognized as a general relationship which is useful in many applications where liquid flow in capillaries is involved. For a single circular tube of length ℓ , the average velocity ν_{ave} , for laminar, pressure-driven steady flow is (BSL equation 2.3-18)⁷:

$$\nu_{ave} = \frac{\Delta P R^2}{8\eta\ell} = \frac{d\ell}{dt} \tag{A-1}$$

where

- ΔP = pressure drop (mPa),
- R = radius of capillaries (cm), and
- t = time (sec).

The type of flow which obeys the criteria given above is known as Poiseuille flow.

The pressure drop across a capillary due to surface tension is

$$\Delta P = \frac{2\gamma_{LV} \cos\theta}{R\ell} \tag{A-2}$$

Substituting A-2 into A-1 gives

$$\frac{d\ell}{dt} = \frac{\gamma_{LV} \cos\theta R}{4\eta\ell} \tag{A-3}$$

Separating and integrating gives

$$\int_0^\ell \ell d\ell = \int_0^t \frac{\gamma_{LV} R \cos\theta}{4\eta} dt \tag{A-4}$$

or

$$\frac{\ell^2}{2} = \frac{\gamma_{LV} R \cos\theta}{4\eta} t \tag{A-5}$$

or

$$\frac{\ell^2}{t} = \frac{\gamma_{LV} R \cos\theta}{2\eta} \text{ Washburn's Equation} \tag{A-6}$$

The derivation of Washburn's equation assumes a horizontal capillary or negligible effects of gravity.

APPENDIX B

DERIVATION OF EQUATION FOR LIQUID FLOW IN A PACKED BED

In Washburn's equation, the model for liquid flow involves a single capillary of radius R . In the derivation which follows, the packed bed is pictured as being composed as capillaries of radius R (BSL 6.4-4 to 6.4-6).⁷ For laminar, pressure-driven flow through a packed bed of length L .

$$\nu_{ave} = \frac{\Delta P R_h^2}{2\eta L} \tag{B-1}$$

where

- R_h = hydraulic radius
- = $\frac{\text{cross section available for flow}}{\text{wetted perimeter}}$

The superficial velocity, ν_o , is equal to

$$\nu_o = \nu_{ave} (\epsilon) \tag{B-2}$$

where

$$\begin{aligned} \epsilon &= \text{void fraction of bed} \\ &= \frac{\text{volume of voids}}{\text{volume of bed}} \end{aligned}$$

or

$$\nu_o = \frac{dL}{dt} = \frac{\Delta P R_h^2}{2\eta L} \epsilon \tag{B-3}$$

If we assume for capillary rise in a packed bed,

$$R = 2R_h, \tag{B-4}$$

which is exactly true for a cylindrical capillary, then

$$\Delta P = \frac{\gamma_{LV} \cos\theta}{R_h}, \tag{B-5}$$

Combining (B-3) and (B-5),

$$\frac{dL}{dt} = \frac{\gamma_{LV} R_h \cos\theta}{2\eta L} \epsilon. \tag{B-6}$$

Separating and integrating as before gives

$$\frac{L_2}{t} = \frac{\gamma_{LV} R_h \cos\theta}{\eta} \epsilon. \tag{B-7}$$

From BSL 6.4-4 to 6.4-6,

$$R_h = \frac{\epsilon}{a} = \frac{\epsilon}{a_v(1-\epsilon)} = \frac{\epsilon \bar{D}_p}{6(1-\epsilon)}, \tag{B-8}$$

where

$$R_h = \frac{\text{volume available for flow}}{\text{total wetted surface}} \cdot \frac{\text{volume of voids}}{\text{volume of bed}} = \frac{\text{wetted surface}}{\text{volume of bed}}$$

$$= \frac{\epsilon}{a},$$

$$a = \frac{\text{wetted surface}}{\text{volume of bed}}$$

$$= a_v(1-\epsilon),$$

$$a_v = \frac{\text{total particle surface}}{\text{volume of particles}}, \text{ and}$$

$$\bar{D}_p = \frac{6}{a_v}.$$

Substituting for R_h in (B-7) gives

$$\frac{L^2}{t} = \frac{\gamma_{LV} \cos\theta \bar{D}_p}{6\eta} \frac{\epsilon^2}{(1-\epsilon)}. \tag{B-9}$$

According to BSL, experimental data show that the flow rates predicted by (B-3) are too large and that the value 2 should be replaced by the value 25/6. If this recommended value is used and carried through (B-3) to (B-8), the final result is

$$\frac{L^2}{t} = \frac{2 \gamma_{LV} \cos\theta \bar{D}_p}{25\eta} \frac{\epsilon^2}{(1-\epsilon)}. \tag{B-10}$$

This is equation (2) of the body of this paper. Again, this derivation assumes laminar flow with negligible gravity effects.

APPENDIX C

NOMENCLATURE

- v_{avc} — average velocity through circular tube (cm/sec)
- v_o —superficial velocity
= $\frac{\text{volumetric rate}}{\text{cross sectional area}}$ (cm/sec)
- ΔP — pressure driving force (mPa)
- R — tube radius (cm)
- l — flow length in capillary (cm) - Crowl⁵ method
- t — time (sec)
- R_h — hydraulic radius (cm)
= $\frac{\text{cross section}}{\text{wetted perimeter}}$
- η — liquid absolute viscosity (mPa·s)
- γ_{LV} — liquid-vapor surface tension (mN/m)
- L — flow distance in packed bed (cm) - present work

- ϵ — void fraction
= $\frac{\text{volume of voids}}{\text{volume of bed}}$
= $\frac{\text{volume of voids}}{\text{volume of voids} + \text{volume of pigment}}$
- a — $\frac{\text{wetted surface}}{\text{volume of bed}}$ (cm⁻¹)
- a_v — $\frac{\text{total particle surface}}{\text{volume of particles}}$ (cm⁻¹)
- \bar{D}_p — mean particle diameter (cm)
- ρ — liquid density (g/cm³)
- ρ_p — particle density (g/cm³)
- k — parameter in Crowl equation

Society Meetings

C-D-I-C

NOV.

"New Trade Sales Slurry Plant"

Technical Chairman Mike McGroarty discussed the three meetings which the technical committee has held at Battelle, Columbus, OH. A project was proposed to investigate the accuracy of ASTM D2369-81, Volatile Content of Coatings. According to Mr. McGroarty, three types of coatings will be tested with and without the addition of a high boiling solvent.

After a tour of Paint American Manufacturing, a new trade sales slurry plant in Dayton, OH, Robert Rightmeyer, of Warehouse Paint Centers, Paint America Co., discussed "TRADE SALES SLURRY PLANT."

Mr. Rightmeyer spoke about the motivating factors behind the venture of the new slurry plant. Having control of the quality and consistency of the trade sales products merchandised through Warehouse Paint Centers was very important, according to Mr. Rightmeyer.

The design of the slurry manufacturing facilities was created by the late Wayne Malden, of Process Design Engineering, Chicago, IL, said Mr. Rightmeyer. The plant began operation in February 1982 with 5,000 square feet of production area on the ground level with mezzanine levels in certain areas.

Mr. Rightmeyer explained the process flow. This incorporates the use of six, 15,000-gallon storage tanks which contain three pigment or extender slurries and two emulsion latices. Mixing is facilitated in 2,000- and 1,000-gallon processing tanks at low speed, low energy, activated from control panels, which automate the process.

According to Mr. Rightmeyer, versatility of raw materials is the key in the streamlining of formulations producing the diverse array of trade sales products. Many of the processing innovations were developed by Paint America for exclusive use at the latex slurry plant, including the "Micrex Technology," said Mr. Rightmeyer.

DAVID KINDER, Secretary

LOS ANGELES

NOV.

"Digital Control Systems"

Lloyd Haanstra, of the Environmental Committee, reported on the progress of CARB rule 1113, and explained that a forthcoming workshop would define a

quick dry enamel. Mr. Haanstra also discussed the fact that the Air Quality Board was picking up samples of products from various sources, testing them, and then charging the manufacturers for the testing. He felt the committee should write in protest of this practice.

John O'Meara, E&C Controls Co., discussed "DIGITAL CONTROL SYSTEMS IN THE MANUFACTURE OF VARIOUS PRODUCTS."

Mr. O'Meara showed that computers had become cheaper, and discussed the various places in everyday life that were controlled by computers.

Basically, he explained if one has a process to control, one has to define what to do, write down the details of the process, and find someone who can work out the hardware and software that can handle the process. He suggested that one would do best to wind up with a full service vendor.

The cost of a system was pointed out, with the hardware costing, about 25% of the total, and software, 75%.

Q. Do you feel that there has been a lack of acceptance of automation/computerization in the coating industry, and if so, what are the main reasons?

A. Our industry, like some others, do not know a great deal about automation by computers, and therefore, there has been a fear or discomfort with the idea.

Q. Which computer company uses Word Star Software and how do you find a competent person to interface between operation and computer?

A. Word Star is a copyrighted software program that can be used on about 50% of computers in use. To get a person on an industrial application, he must become totally dependent on the automatic system - and learn it manually also.

Q. Most paint companies want similar things from a computer system, like making batch tickets, inventory, pricing, color controls, etc. Why can't the paint society as a whole ask a computer company to develop a general program and let every member company share it, and let every member company share the cost?

A. The system needed is already available by a number of companies that will satisfy approximately 95% of the need. They range in price from \$5,000 to \$500,000 depending on size of the operation.

MICHAEL GILDON, Secretary



New England Society President, John Fitzwater, (left), of Polyvinyl Chemical Industries, Inc. presented the MMA Award to Immediate Past-President, Robert Modrack, of Benjamin Moore Co. The Society was recognized for its contribution to the industry derived from its 1982 Coatings Tech Expo

NEW ENGLAND

NOV.

"Rheology Control of High Solids Coatings"

Dennis G. Miller, of Cab-O-Sil Div., Cabot Corp., discussed "RHEOLOGY CONTROL OF HIGH SOLIDS COATINGS."

Mr. Miller stated that due to Federal regulations limiting the emission of organic solvents from surface coatings operations, the paint industry is in the process of reformulating many of its products. Four approaches are possible: high solids solvent-borne coatings, water-borne coatings, powder coatings, and radiation-cured coatings. Marketing data for the last three years show that high solids solvent-borne coatings are gaining acceptance in many broad areas of usage, such as metal furniture, automotive topcoat, can coatings, flat stock, etc., stated Mr. Miller.

Based on performance properties of the coatings containing various levels of the thixotropes studied, only fumed silica provided adequate anti-settling and anti-sag control, explained Mr. Miller. Fumed silica also exacted the smallest price in gloss reduction. The additional raw material costs per gallon in using fumed silica at levels of 0.17 and 0.35 wt. % are low, 5.8 and 12.0 cents, respectively.

The rheological investigations established two major points, said Mr. Miller:

(1) No thixotropy was observed in any of the coatings at 75% wt. solids using a

Ferranti-Shirley cone and plate viscometer. The control and the coating containing 0.35 wt. % fumed silica showed no thixotropy when adjusted to 80, 85, and 90, wt. solids to stimulate the coating after application.

(2) Yield value was determined to be the major rheological property controlling the sag resistance of the high solids coatings. A simple yield point tensiometer was used to determine the yield points of the selected coatings. The control and the coating containing 0.35 wt. % fumed silica were tested at 75, 80, 85, and 90 % wt. solids. Good correlations between the calculated maximum dry film thickness without sag and the observed sag resistance of the coatings were obtained.

Q. When you sprayed your panels did you use automatic spray equipment?

A. No, they used a Binks model 18 hand gun and they were sprayed by hand. A total of eight panels were hand sprayed with a mil and one half thickness. Then we would increase our findings.

Q. Did you do any temperature effect studies?

A. No, all these materials were sprayed at 77°. Hot sprays were not attempted.

Q. Have you done any effect on gloss and other color systems like a black?

A. No, we only tested a white and all the materials were tinted with a small amount of red iron oxide, yellow iron oxide, and carbon black dispersion so they could tell the panels apart.

Q. Do you have any reports on the effect of gloss in blacks?

A. No, we have not. General problem with coatings in blacks is getting the proper dispersion in the black.

Q. Did you use any ethylene glycol as an additive?

A. No, this approach had not been tried, just the fumed silica.

CHARLES J. HOAR, *Secretary*

NEW YORK NOV. "Diatomaceous Silica in Coatings"

Tom Remmers, of Maville Products, Inc., spoke on "THE USE OF DIATOMACEOUS SILICA IN COATINGS."

Aided with slides, Mr. Remmers explained that diatoms are plants that extract silica from water and that when they die they settle to the ocean floor. With changes in land patterns over time, some of the diatomite was raised from ocean bottom to land (as was the case in California). Current California deposits were formed some 15 million years ago. In the U.S., major sites are located



1982-83 Officers of the Dallas Society. (Left to right): Vice-President—T. Leon Everett; President—William Wentworth; and Secretary—Terence W. La Baw. Not present are Treasurer—Van G. Falcone and Society Representative—Carlos Dorris

in California and in the State of Washington.

Mr. Remmers showed how diatomite was mined. There are a few major types of diatomite used by the paint industry: natural grade, calcined grade, and the flux calcined grade. Natural is off white color, flux calcined is white, and calcined is pink in color, explained Mr. Remmers.

The use of diatomite in paint is flattening. Diatomite does not just take up space in a can.

Diatomite Grades for us, in paint, can disperse on high speed mixers up to A Hegman 5-1/2. Medium flux calcined grade is the "workhorse" giving a 1-2 Hegman. The major use is in emulsion type paints, said Mr. Remmers. The finer diatomite is ground the less the efficiency for flattening. Diatomite particles of less than four microns do not contribute to flattening. It does not work like talc. Grind methods other than disc dispenser can destroy particle shape and thus destroy the ability of diatomite to flatten, stated Mr. Remmers.

Slides indicated levels of diatomite, Hegman fineness, and respective gloss and sheen.

Other properties of diatomite, according to Mr. Remmers, are burnish resistance, stain removal, fast solvent release, and good sand properties.

Factors affecting gloss and sheen are particle size, particle shape, oil absorption, and particle distribution. Comparison at various loading levels to talc were made showing gloss and sheen. At 1/4 lb./gal. diatomite shows lower gloss and sheen readings than talc. Various pigment formulas were examined by Mr. Remmers for different type paints, each containing diatomite as a flattener.

Diatomite as a possible replacement

for other extender was examined. With hydrogels it is generally not possible to use diatomite in place of hydrogels especially in long pigmented or clear finishes, stressed Mr. Remmers.

Q. How does diatomite affect the brightness of bright blues, greens? I think they whiten the color.

A. Customers have said the same thing. Our lab has not done any work along these lines. I would agree with you. A good idea would be to publish colors in different gloss range.

Q. For burnish resistance how well does diatomite work?

A. It is better than talc, calcined clay. There are items that are better than diatomite.

MIKE ISKOWITZ, *Secretary*

NORTHWESTERN NOV. "Water-Borne Epoxy Materials"

President Don Emch presented Treasurer Rich Johnson with the MMA Award plaque, which was won for the Society's contribution through its educational programs.

John C. Avery, of Celanese Plastics & Specialists Co., presented a lecture on "TWO COMPONENT WATER-BORNE EPOXY MATERIALS FOR INDUSTRIAL MAINTENANCE COATING SYSTEMS."

Mr. Avery discussed the history, epoxy component selection, converter selection, system selection, sales incentives, and applications of epoxy materials.

"LEVELING AND SURFACE FLOW IN PAINTS AND COATINGS, AND THEIR RE-



1982-83 Officers of the Louisville Society. (Left to right): Secretary—W.J. Morris; Society Representative—James A. Hoek; Treasurer—Edward D. Thomasson; Immediate Past-President—Phillip W. Harbaugh; President-Elect—John A. Lanning; and President—Fred E. Newhouse

LATIONSHIP TO SURFACE PHENOMENA, SUCH AS SURFACE TENSION, FOAMING, ETC.” was discussed by Wolfgang Zinnert, of Byk-Mallinckrodt U.S.A., Inc.

Mr. Zinnert explained the types of products used to prevent surface related problems. He then drew comparisons of these types of products.

Measuring surface tension and its relationship to surface active agents was discussed. Mr. Zinnert also discussed the types of silicones and their various applications.

ROBERT MADY, *Secretary*

PIEDMONT

NOV.

“Investigation of Abrasion Resistance”

Education Committee Chairman, Bob Matejka, reported on the society-sponsored polymer course and the questionnaire which is being prepared to obtain input on the value of the course. Mr. Matejka also solicited support from member companies to participate in the newly proposed one-semester internship program at UNC-G. Dr. Wid Painter, of GTI, then discussed a two-year chemical technology program and explained a co-op program he wishes to begin with the industry for his students.

W. M. Neuberg, of Shamrock Chemicals Corp., discussed “AN INVESTIGATION OF ABRASION RESISTANCE.”

With the aid of slides, Mr. Neuberg showed how wax particles, such as polyethylene wax, play a role in a coating film. Through polarized transmitted light microscopy, he demonstrated how wax crystals melt under pressure of abrasion and become liquid lubricant to protect the film they are in. Mr. Neuberg

explained that polytetrafluoroethylene does not melt under pressure, rather, these discrete particles sheared along their crystalline plane to supply platelets to lubricate. He suggested that PTFE maximizes abrasion resistance and mar proofing of a coating film when used with polyethylene wax. According to Mr. Neuberg, a 3% PE and 0.5% PTFE on total paint weight is a good starting point for investigation.

PHILLIP WONG, *Secretary*

PITTSBURGH

NOV.

“Basics of Spray Finishing”

Jack Adams, of Binks Manufacturing Co., gave a talk entitled, “BASICS OF SPRAY FINISHING.”

Mr. Adams began by describing the first spray painter (60,000 years ago) who decided to embellish his scratchings on

cave walls by blowing natural pigment powders into the crevices with a hollow bone (the first powder coatings). This “spray gun” was soon put out of business when cave artists turned to paints made by mixing the powders with tree sap.

Mr. Adams pointed out that spray painting began again about 60 years ago when people were looking for a better way to apply paints. Spraying is only one of many ways to get paint onto ware, but it often is the best method for a given product. He stressed that spray painting works very well when it is under control. Unfortunately, too many people do not have their spray operation under adequate control. Mr. Adams indicated that one of the purposes of his talk was to alert people to the means of control and the problems that occur with lack of control. He went on to say that successful application depended on the MOE principle (M = MATERIAL, O = OPERATOR, E = EQUIPMENT): *MATERIAL*—the material has to be the right one, prepared properly, reduced to proper viscosity, etc. If there is a problem with the material, get back to the supplier; *OPERATOR*—a good, well-trained operator is a necessity. A good spray operator is every bit the craftsman that a machinist or cabinet maker is. Every operator must be trained and his supervisor must be properly trained; and *EQUIPMENT*—must be the right equipment for the job, must be maintained, have the right nozzles, etc.

Mr. Adams then surveyed the three basic spraying methods: conventional (air), airless (hydraulic), and electrostatic. In conventional (air) spraying the emerging stream of paint is struck by converging jets of air which break the paint into small particles. In airless (hydraulic) spraying the paint is under high pressure and is forced through a small hole at high velocity. The expansion of the fluid as it comes out of the hole is so rapid that it



Northwestern Society Officers for 1982-83. (Left to right): Secretary—Robert Mady; President—Donaldson Emch; Treasurer—Richard Johnson; and Vice-President—Herbert Davidson

breaks up into droplets. In electrostatic spraying electrical potential is used to break the paint into droplets. An electrical charge is applied to the paint and the ware is grounded. The paint is broken into strings which tend to break up further as they travel toward the grounded surface.

The advantages of conventional spray are control and versatility, stated Mr. Adams. Conventional spraying is the most controllable of all spray processes. To prove this Mr. Adams showed a transparency of a very fine painting of a clipper ship in full sail - done free hand with auto lacquers and a full sized conventional sprayer! Because of its versatility conventional spray techniques can be used to spray anything from very heavy mastics to solvents with viscosities lower than that of water.

Mr. Adams then discussed the different kinds of conventional spray methods. Siphon spraying uses the lowest cost equipment and is best for low volume painting operations. It allows easy clean-up. The disadvantages are that the maximum volume is one quart, only low viscosity materials may be sprayed, and the number of spraying positions is restricted, said Mr. Adams. Pressure feed equipment is more flexible and gives better control. It can be used to apply more material, faster. Most automobiles are sprayed with pressure feed spray guns giving application rates of 36-40 fluid oz. per minute per gun. The limitations of this technique are that it involves a greater volume of air and it creates more fog than Siphon spraying.

Mr. Adams' coverage of spray methods led him to a discussion of spray guns. He pointed out that all kinds are available and that their forms follow their functions. A spray gun essentially is a handle designed to hold valves and a nozzle in position. He proceeded to show transparencies of a wide variety of guns ranging from heavy duty drop forged guns for mastics and other heavy materials to small light-weight air brushes. He indicated that there was a vast number of designs available, but that the keys to operation of all of them were the air and fluid nozzles. These parts determine atomization and spray volume. Fluid nozzles come in different sizes and materials depending on the fluid being sprayed. Case-hardened steel is used for solvent systems, stainless steel for corrosives, and tungsten carbide for abrasive materials. There are several different types of air nozzles, including external mix, internal mix, and special effects, all of which can be used on the same gun. Specific combinations of air and fluid nozzles can be used to control particle size, spray pattern, and rate of delivery so as to minimize overspray, rebound (off spray), and fallout and maximize both the



1982-83 Officers and Board Members of the Philadelphia Society. Seated (left to right): Honorary Director—J. Richard Kiefer; Society Representative—Carl Fuller; President—Ralph Myers; and Assistant Treasurer—Phillip Reitano. Standing (left to right): Member-at-Large—Donald Denny; President-Elect—Frank Bartusevic; Treasurer—William Georgov; Membership Chairman—Stuart Lipskin; and Member-at-Large—Thomas L. Peta

amount of paint that reaches the ware and the quality of the resultant film.

Mr. Adams quoted the adage "When the pattern is right, the pressure is right" and stressed that an operator should spray at the lowest pressure that gave the proper spray pattern. He then proceeded to show the audience what a proper spray pattern looks like. The preferred pattern or fan is elliptical with the long axes being vertical. The paint appearance should be dense and smooth, dark in the body of the pattern and fine edges. Since the fluid nozzle naturally gives a circular pattern, the air must be used to "squeeze" the paint stream until the natural pattern is distorted to the desired shape. He then showed a number of incorrect patterns and paint densities ranging from wet centers where the paint is not "squeezed" enough, through formation of two streaks with no paint at the center (too much "squeezing"), to a series of patterns when the guns were damaged, out of adjustment, or partially clogged.

Mr. Adams next surveyed operator variables, including the distance of the gun from the work (there is a correct distance for each type of gun), stroking speed, pattern overlap (50% of each spray pass), spray gun attitude (keeping gun square with work). Distance is particularly important, stressed Mr. Adams. If the gun is too far away, the result will be a dry and sandy finish, with too much solvent lost on the way. If the gun is too close, the coating will be very wet, too heavy, and will sag.

The use of other components of the spray system to cut cost and increase effectiveness was discussed. Mr. Adams

explained the advantages of using a paint heater to lower viscosity and keep temperature constant throughout the day. This allows lower pressure and, therefore a lower flow rate which can save a considerable amount of money on paint waste. Reducing flow by one fluid three per minute would save roughly \$8000 per year per gun.

Compressed air supply was then discussed. Mr. Adams stressed that this must be controlled and maintained. A system must have a good quality, clean, well maintained regulator. The regulator screens must be kept clean. In addition, the air supply itself must be clean. A system should include an oil/water extractor prior to the regulator. The filter in the extractor must be changed periodically. The compressor must not be forgotten either. It must be the proper size, both in terms of volume and pressure.

Mr. Adams explained airless spray. He began by indicating why airless spray is so popular. It reduces fog, is cleaner, is fast (fastest technique yet - can paint inside of three bedroom house in 45 minutes), saves material, and cuts cost. Because the forward velocity of the paint is due to the release of hydraulic pressure, the particles slow down rapidly and tend to stay on the part. The initial high velocity makes the process hazardous, however. A careless operator can blow paint into his fingers or hand. Unless the correct treatment is carried out immediately, the person can lose fingers or even a hand. Where this method is used the plant doctor or nurse must know what to do about hydraulic injection of paint into the body. All equipment comes with warning labels concerning this hazard.



1982-83 Officers of the Western New York Society. (Left to right): Education Chairman—Art Smith; Secretary—Don Kressin; Membership Chairman—Dick Foss; Vice-President—Mike Kaufman; Society Representative—Robert Hoerner; Technical Committee Chairman—Bob Gillespie; and President—Bill Martin. Missing from the photo is Treasurer, Charles C. Tabbi (Regrettably, Mr. Hoerner died December 21—see *Obituary*, p. 77. Eugene LeVea, of Pratt & Lambert, Inc., will serve as temporary Society Representative)

Mr. Adams pointed out that airless spray patterns were similar to those of conventional equipment, but not as fine. He recommended using the lowest pressure to give a satisfactory pattern. The source of the pressure is a high ratio (up to 50:1) pump which can be used in conjunction with a paint heater. He recommended using paint heaters on all automatic equipment. The constant temperature helps to insure constant spray quality.

Electrostatic spraying was explained. Mr. Adams began by considering the paint transfer efficiencies of various processes. Conventional air spray is only 30-40% efficient and airless is 35-40%, whereas electrostatic air spray is 50-60% efficient and high speed electrostatic bells are an incredible 85-94% efficient. The original electrostatic process involved an air spray gun with an ionized zone just beyond the nozzle. The charged particles were attracted to the grounded part being painted. The next development was the rotating disc which was more efficient, but gave less control. Changing the shape of the disc to a bell or cup allows directional control and the greatest efficiency. There also is increased interest in electrostatic air guns, mainly because of the high degree of control. Electrostatic equipment does have drawbacks, stressed Mr. Adams. The charged particles always go to the nearest grounded surface. Therefore, they do not go into corners, cavities, crevices, etc.

Mr. Adams discussed robots. Robotic machines have been developed that control all operator variables in spraying and give very constant results. These machines can be programmed to handle any given product. The means of producing the desired finish has to be well understood in order to do this, however. An improperly programmed robot will produce as bad a

job as an improperly trained human operator.

Mr. Adams concluded by pointing out that most paint problems were due to simple, basic mistakes - often due to the operator never having been shown how to spray properly. Other factors were incorrectly adjusted equipment, poor maintenance, etc. He stressed that it is the basics that count. If you know the basics, you can solve the problems.

CLIFFORD SCHOFF, *Secretary*

PITTSBURGH

DEC.

"Quality vs Performance Testing"

John R. Holleran and Edward J. Battis, Metallurgical Department Manager and Chief Chemist, respectively, of the Pittsburgh Testing Laboratory, gave talks on their specialties as practiced at the laboratory.

Mr. Holleran discussed the instruments and techniques used by testing laboratories to test and evaluate materials and described some typical problems and projects. He mentioned the use of the Scanning Electron Microscope (SEM) to determine failure mechanisms and analyze surface morphologies of materials. The dispersive x-ray unit (EDAX) attached to the SEM is used to identify elements present in a specimen. A separate technique, x-ray diffraction, is used for identification of crystalline compounds. These and other instruments had been applied to many different projects by Mr. Holleran. He described their use in analysis of factors involved in the failure of metal structures in mines - bad welds, poor designs, stresses, etc.

Ed Battis, the Chief Chemist at Pittsburgh Testing Laboratory, spoke on

"QUALITY VS PERFORMANCE TESTING". He dealt with the differences between these types of tests and gave examples in terms of paints and his own particular specialty, asphalts.

Quality tests tell the investigator how much pigment and vehicle the paint contains, its viscosity, the fineness of grind, and other properties related to the design and manufacture of the paint, he said.

The results tell the paint purchaser whether he is getting a quality product, i.e. whether everything is in it that is supposed to be there and whether it is put together properly, added Mr. Battis.

Performance (acceptance) tests tell the purchaser whether the paint will do the job that he expects. They are paint film tests related to the end use and the specifications set down for the paint by the purchaser. Examples include drying time, gloss, scrub, and corrosion resistance, and exposure. The results tell the purchaser whether the paint film meets his specifications, he said.

Mr. Battis finished with a discussion of asphalt for airport runways. The paving material is composed of asphalt cement (softening point greater than 100°C), stone, sand, and slag mixed in certain specified proportions. Specifications for airport runways are very strict - much tougher than for roads. Before a contractor can carry out a paving job he must put down a test strip which is tested as it is applied and after it is completed. The strip must pass all the tests or a new strip has to be put down. Once an acceptable strip has been applied, the contractor can go on to building the runway itself. The Federal Aviation Agency sets the quality specs - how much asphalt, the exact aggregate mix, gravel size, etc. If the material meets the quality tests, the final product *should* meet performance tests. However, this is not always true. Performance tests include air void measurement, break resistance, and flexibility. Asphalt pavement is a flexible material. It must give, but not too much. Some tests are done on freshly prepared pavement mix, whereas others are carried out in the field after paving.

CLIFFORD SCHOFF, *Secretary*

ROCKY MOUNTAIN

NOV.

"Computer Controls in the Coatings Industry"

John O'Meara, of FMC Controls, presented "COMPUTER CONTROLS IN THE COATINGS INDUSTRY."

LUIS O. GARCIA, *Secretary*

"Silicone Resins in Maintenance Applications"

William Finzel, of Dow Corning, spoke on "PERFORMANCE OF SILICONE MODIFIED ORGANIC RESINS IN MAINTENANCE APPLICATIONS."

Mr. Finzel related that protective coatings containing silicone modified resins are being used as maintenance topcoats in mild to moderate corrosive atmospheres. Silicone content improves overall weatherability as measured by gloss retention, chalk resistance and color retention, he said.

Although silicone-containing paints in maintenance applications can vary in silicone content as high as 100% and offer high temperature resistance, the discussion for this paper will be limited to 30% silicone modification and ambient temperature cure, he said.

Mr. Finzel discussed three subjects: (1) low VOC silicone alkyd copolymers; (2) durable topcoat comparison; and (3) silicone acrylic cold blends.

In the first study, Mr. Finzel compared the VOC (Volatile Organic Compound) content of silicone alkyd paints with naphtha mineral spirits, an active solvent blend, chlorinated solvent and water reducible silicone alkyd resin. A variety of lower VOC paints are possible, he said, depending on the VOC requirement and silicone alkyd resin most acceptable for the specific maintenance application.

Six topcoat resin systems were compared, he said, in similar pigmented coatings in the second study for physical properties, chemical resistance and accelerated weatherability. The resins tested are commercially available silicone alkyd, alkyd, silicone acrylic, acrylic, two part polyester aliphatic urethane, and two part weatherable epoxy. Silicone modification improves weatherability of the alkyd but does not affect chemical resistance of the alkyd or acrylic, said Mr. Finzel. The urethane and epoxy systems exhibit unique coating properties, he added.

A more detailed study of cold blended acrylic resins were reviewed in the third and last section. Florida and Michigan outdoor weathering data were shown along with heat resistance of coatings containing silicone cold blended resins. Acrylic and silicone modified acrylic systems are superior to corresponding alkyd and silicone modified alkyd resins in weatherability and heat resistance, he said. Acrylic and silicone resin selection is important for optimum compatibility, film flexibility, adhesion and durability, he concluded.



St. Louis Society Officers for 1982-83. (Left to right): Secretary—William Truskowski; Educational Chairman—Susan Bailey; Treasurer—Charles L. Grubbs; Technical Chairman—Harold Cook; and President—Joseph J. Wrobel

Q. Does solvent resistance improve by silicone modifying acrylics?

A. No, being thermoplastic they have less solvent resistance than silicone alkyds.

Q. Are cold blended silicone acrylics as effective as U.V. modified acrylics?

A. No work has been done in this area.

Q. Are cold blended silicones as

efficient as silicone copolymers? What about costs?

A. Usually a cold blend is more brittle, also the properties of the organic product blended will determine properties. The cost of the cold blend will usually be higher than the copolymer. The copolymer can be tailored to specific applications.

WILLIAM A. TRUSKOWSKI, Secretary

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SAMPLES**

Future Society Meetings

Birmingham

(Mar. 3)—"THE ROLE OF THE ANALYTICAL LABORATORY IN A MODERN PAINT COMPANY"—Dr. S. Bryan, Donald MacPherson & Co. Ltd.

(Apr. 14)—"TRANSPORT AS IT APPLIES TO THE PAINT INDUSTRY"—O. Thomas, Freight Transport Assoc.

(May 5)—"USE OF ANTI-STATIC DEVICES IN THE PAINT INDUSTRY"—G. Wheatcroft, 3M Co.

Chicago

(Mar. 7)—"CUSTOM COMPUTER SYSTEMS FOR COATINGS CHEMISTS"—Bruce Banther, East Chester Corp. "EXPERIMENTAL DESIGN AND STATISTICAL ANALYSIS OF DATA"—Dr. R.D. Snee, E.I. duPont de Nemours Co.

(Apr. 4)—"ACID RAIN TRENDS IN THE U.S."—Dr. Gary Stensland, University of Illinois. "ASPECTS OF RHEOLOGY IN COATINGS"—Dr. J.E. Glass, North Dakota State University.

Cleveland

(Feb. 15)—"DISPERSANTS: BASIC CONCEPTS AND EFFECT ON PAINT PROPERTIES"—Dr. W.K. Asbeck.

(Apr. 19)—"ATTRITOR GRINDING AND DISPERSING EQUIPMENT"—Arno Szegvari.

(May 17)—"A FUNNY THING HAPPENED ON THE WAY TO THE COATINGS TECHNICAL MEETING"—Frank Borrelle, FSCT Executive Vice-President.

Golden Gate

(Mar. 14)—"RECENT DEVELOPMENT IN EPOXY RESIN"—Dr. Ron Bauer, Shell Development Co.

(Apr. 18)—"CHLORINATED SOLVENTS IN COMPLIANCE COATINGS"—Hank George, Consultant.

(May 16)—"BENEFITS OF COMPOSITE DISPERSANTS IN COATINGS AND COLORANTS"—Elio Cohen, Daniel Products Co.

(June 13)—"MANUFACTURING COMMITTEE'S SEMINAR."

Houston

(Mar. 9)—"Education Committee Symposium. "SECURITY: EDP (COMPUTER), PLANT, AND PERSONNEL."—Mark Dante, Chairman, Shell Development Co.

(Apr.)—"PAST PRESIDENT'S NIGHT." (May 11)—"IMPACT OF THE SUPER-

FUND LAW ON THE PAINT INDUSTRY"—Tom Graves, NPCA Legal Staff.

Kansas City

(Mar.)—LADIES NIGHT.

(Apr. 14)—"HIGH SOLIDS COATINGS—PAST, PRESENT, AND FUTURE"—Speaker from Spencer Kellogg, Div. of Textron, Inc.

(May 12)—"FEDERATION OFFICERS' VISIT. Update on Federation Activities; Education Committee will present Science Pioneer Awards; and Election of Officers.

(June 10-11)—"JOINT MEETING of the Kansas City/St. Louis Societies.

Los Angeles

(Mar. 9)—"RECENT DEVELOPMENT IN EPOXY RESIN"—Dr. Ron Bauer, Shell Development Co.

(Apr. 13)—"CHLORINATED SOLVENTS IN COMPLIANCE COATINGS"—Hank George, Consultant.

New England

(Feb. 17)—"RECENT ADVANCES IN TANK CLEANING SYSTEMS WITH CAUSTIC RECYCLING."

(Mar. 17)—"FORMULATING WITH MICROSOPHERES."

(Apr. 14)—"ANNUAL WESTERN MEETING. "REVIEW OF THE ALYCOL ETHER SITUATION."

(May 20)—"BIENNIAL SYMPOSIUM ON COATINGS TECHNOLOGY."

Northwestern

(Mar. 8)—"SOCIETY SYMPOSIUM"—H.C. Ting, Chairman.

(Apr. 5)—"EDUCATION NIGHT"—James Lawlor, Chairman.

(May 3)—"MANUFACTURING PROGRAM"—Richard Munding, Chairman.

Philadelphia

(Mar. 10)—"FEDERATION VISIT.

(Apr. 15)—"AWARDS NIGHT.

(May 12)—"NEWER TECHNOLOGICAL AREAS OF WATER-BORNE RESINS"—Don Hogan, Polyvinyl Chemical Corp.

FSCT Membership Anniversaries

25-YEAR MEMBERS

New York

Joseph Cantor, of Josad Consultants, Inc.
Bernard Fleisher, of Continental Technical Finishes Corp.

Fred Gartenlaub, of New York Bronze Powder Co., Inc.

Lawrence H. Gates, of North American Paint.

Albert J. Kirsch, of American Cyanamid Co.

Marvin Kress, of Red Devil Paints & Chemicals.

Max Kronstein, of Manhattan College.
Edwin S. Leister, of Duralac Chemical Corp.

Bernard Lepselter, of R & A Specialty Chemical Co.

Sanford M. Liedeker, of The Supply Co., Inc.

Joseph Lo Re, of Verflex Co., Inc.
Sidney Mandel, of United Paint & Varnish Co.

Saul Mark, of Continental Technical Finishes Corp.

John G. Pappas, of Benjamin Moore & Co.
Saul Samit, of Koppers Co., Inc.

Lawrence Shatkin, of Paragon Paint & Varnish Corp.

Michael A. Spero, of Pan Chemical Corp.
Fred R. Spohrer, of F.O. Pierce Co.

Peter Tortorici, of Dux Paints & Chemicals, Inc.

Lou Weiner, of Red Devil Paints & Chemicals.

David Werther, of Mobil Chemical Co.
M.A. Zecchin, of Ultra Adhesives, Inc.

Constituent Society Meetings and Secretaries

Piedmont

(Feb. 16)—JOINT MEETING WITH PIEDMONT PCA.

(Mar. 16)—FEDERATION OFFICER VISIT.

(Apr. 20)—"PLANT SAFETY-HANDLING SOLVENT AND NITROCELLULOSE"—Speaker from Hercules, Inc.

(May 18)—"SOLVENTS FOR HIGH SOLIDS COATINGS"—R. Readshaw, Union Carbide Corp.

(June 15)—"FINISHING POLYOLEFIN PLASTICS"—T.E. Parsons, Eastman Chemical Products, Inc.

Pittsburgh

(Mar. 7)—"RELATIONSHIPS BETWEEN PARTICLE SIZE AND OPTICAL PROPERTIES OF IRON OXIDE PIGMENTS"—Dr. Rolf Odenthal, Mobay Chemical Corp.

(Apr. 4)—"CHLORINATED SOLVENTS—AN ALTERNATIVE TECHNOLOGY IN COATINGS"—Jed Fulkerson, Dow Chemical Co.

(May 2)—"WHAT IS THE STRUCTURAL STEEL PAINTING COUNCIL?"—John Keane, Mellon Institute.

St. Louis

(Feb. 15)—"LAS VEGAS NIGHT."

(Mar. 15)—"HIGH SOLIDS COATINGS"—Speaker from Freeman Chemical Co.

(Apr. 19)—"EDUCATION NIGHT."

(May 17)—"MANUFACTURING NIGHT."

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BALTIMORE (Third Thursday—Eudowood Gardens, Towson, MD).

BIRMINGHAM (First Thursday—Westbourne Site, Edgbaston). D. H. CLEMENT. Holden Surface Ctg. Ltd., Bordesley Green Rd., Birmingham B9 4TQ, England.

CHICAGO (First Monday—meeting sites in various suburban locations). FRED FOOTE, U.S. Gypsum Co., 700 N. Rte. 45, Libertyville, IL 60048.

C-D-I-C (Second Monday—Sept., Jan., Apr., June in Columbus; Oct., Dec., Mar., May in Cincinnati; Nov., Feb., in Dayton). ROBERT A. BURTZLAFF, Potter Paint Co. of Ind., P.O. Box 265, Cambridge City, IN 47327.

CLEVELAND (Third Tuesday—meeting sites vary). RAY PODLEWSKI, Mansfield Paint Co., P.O. Box 998, Mansfield, OH 44901.

DALLAS (Thursday following second Wednesday—Steak & Ale Restaurant). T. LEON EVERETT, Dan-Tex Paint & Ctg. Mfg., Inc., P.O. Box 18045, Dallas, TX 75218.

DETROIT (Fourth Tuesday—meeting sites vary). PETER BURNETT, Wyandotte Paint Products, Inc., 650 Stephenson Hwy., Troy, MI 48084.

GOLDEN GATE (Monday before third Wednesday—Alternate between Sabella's Restaurant on Fisherman's Wharf and the Sea Wolf at Jack London Square, San Francisco). ROBERT MILLER, Frank W. Dunne Co., 1007 41st St., Oakland, CA 94608.

HOUSTON (Second Wednesday—Sonny Look's, Houston, TX) RICHARD D. BATCHELOR, Valspar Corp., 2503 W. 11th St., Houston, TX 77008.

KANSAS CITY (Second Thursday—Cascone's Restaurant, Kansas City, MO). GENE WAYENBERG, Tnemece Co., Inc., P.O. Box 1749, Kansas City, MO 64141.

LOS ANGELES (Second Wednesday—Steven's Steak House, Commerce, CA). MICHAEL GILDON, Guardsman Chemicals, 9845 Miller Way, Southgate, CA 90280.

LOUISVILLE (Third Wednesday—Howard Johnson's, Louisville, KY). W. JERRY MORRIS, Celanese Plastics & Specialties Co., P.O. Box 99038, Jefferson-town, KY 40299.

MEXICO (Fourth Thursday—meeting sites vary). TERESA SUAREZ, Sherwin-Williams Co., Mexico, D.F., Mexico.

MONTREAL (First Wednesday—Bill Wong's Restaurant). M. MEGELAS, International Paints Ltd., P.O. Box 190, Outremont, Que., Can., H2V 4M9.

NEW ENGLAND (Third Thursday—Fantasia Restaurant, Cambridge). CHARLES J. HOAR, Union Chemicals Div., 67 Walnut Ave., Clark, NJ 07066.

NEW YORK (Second Tuesday—Landmark II, East Rutherford, NJ). MICHAEL ISKOWITZ, Koppers Co., Inc., 480 Frelinghuysen Ave., Newark, NJ 07114.

NORTHWESTERN (Tuesday after first Monday—Boulevard Cafe, Golden Valley, MN). ROBERT MADY, George C. Brandt, Inc., 2975 Long Lake Rd., St. Paul, MN 55113.

PACIFIC NORTHWEST (Portland Section—Tuesday following second Wednesday; Seattle Section—the day after Portland; British Columbia Section—the day after Seattle). WILLIAM SHACKELFORD, Gaco-Western, Inc., P.O. Box 88698, Seattle, WA 98188.

PHILADELPHIA (Second Thursday—Valle's Steak House. WILLIAM GEORGOV, J.M. Huber Corp., P.O. Box 310, Havre de Grace, MD 21078.

PIEDMONT (Third Wednesday—Howard Johnson's, Brentwood exit of I-85, High Point, NC.) JAMES E. HUSTED, Mobil Chemical Co., P.O. Box 2438, High Point, NC 27261.

PITTSBURGH (First Monday—Skibo Hall, Carnegie Mellon Univ.). CLIFFORD SCHOFF, PPG Industries, Inc., R&D Center, P.O. Box 9, Allison Park, PA 15101.

ROCKY MOUNTAIN (Monday following first Wednesday—Gusthaus Ridgeview, Lakewood, CO). LUIS O. GARCIA, Kelly-Moore Paint Co., 3600 E. 45th Ave., Denver, CO 80216.

ST. LOUIS (Third Tuesday—Salad Bowl Restaurant). WILLIAM TRUSZKOWSKI, Mozel Chemical Products Co., 4003 Park Ave., St. Louis, MO 63110.

SOUTHERN (Gulf Coast Section—Various Dates; Central Florida Section—Third Thursday after first Monday; Atlanta Section—Third Thursday; Memphis Section—Second Tuesday; Miami Section—Tuesday prior to Central Florida Section). S.G. SANFILIPPO, Reichhold Chemicals, Inc., Technical Services Lab., P.O. Box 1610, Tuscaloosa, LA 35401.

TORONTO (Second Monday—Cambridge Motor Hotel). R. H. STEVENSON, Tenneco Chems., Canada Ltd., 235 Orenda Rd., Bramalea, Ont., Can., L6T 1E6.

WESTERN NEW YORK (Third Tuesday—The Red Mill, Clarence, NY). DONALD M. KRESSIN, Spencer Kellogg Div., Textron, Inc., P.O. Box 210, Buffalo, NY 14225.

BALTIMORE

Active

COPELAND, DEREK R.—Lenmar, Inc. Baltimore, MD.
 DUNN, LARRY D.—Rust-Oleum Corp., Hagerstown, MD.
 FRAZIER, ROBERT F.—duPont de Nemours & Co., Inc., Front Royal, VA.
 GREENWELL, DANIEL A.—Duron, Inc., Beltsville.
 KARWACKI, VICTOR Z.—O'Brien Corp., Baltimore.
 KJONIKSEN, OLE CHR.—Jotun Baltimore Copper Paint Co. Baltimore.
 MARCINKUS, FABIAN—Lenmar, Inc. Baltimore.
 RICHARDSON, W. A.—Valspar Corp., Baltimore.
 SHEIKH, MUHAMMAD SAIED—Rust-Oleum Corp., Hagerstown.
 VAN HORN, ROBERT A.—Contact Paint & Chemical Co., Baltimore.
 WINTERLING, WILLIAM C.—O'Brien Corp., Baltimore.

Associate

ARBUCKLE, WENDY—Union Chemicals Div., Baltimore, MD.
 BROWN, ROBERT R.—Colloids, Inc., West Point, VA.
 GROSSMAN, JUDITH E.—NL Chemicals, Hightstown, NJ.
 HYDE, TIMOTHY S.—Reichhold Chemicals, Inc., Blue Bell, PA.
 KUHLE, JOANNE—W. R. Grace Co., Baltimore.
 LOVE, EVERETTE H. JR.—Diamond Shamrock Co., Matthews, NC.
 ROWE, RICHARD C.—McWhorter Inc., Baltimore.

BIRMINGHAM

Active

BEST, ANTHONY J.—Intercoat Paints Ltd., Midland, England.
 CHIPPINGTON, K.A.—Olympic Varnish Co. Ltd., Glos. England.
 DOWNES, KENNETH JAMES—Blythe Burrell Colours, Oxon, England.
 HARMAN, DENIS J.—Postans Ltd., Birmingham, England.
 JAMES, DAVID GEORGE—Holdens Surface Coatings Ltd., Birmingham.
 MORRIS, JOHN—Lawrence Industries, Surrey, England.
 TAYLOR, MARK STEPHEN—Postans Ltd., Birmingham.
 WELLS, JANET M.—Croda Paints Ltd., Birmingham.

C-D-I-C

Active

BURNS, DAVID J.—Hanna Chemical Coatings Co., Columbus, OH.
 CUNNINGHAM, PATRICIA—Lilly Industrial Coatings, Indianapolis, IN.
 GABLE, BRUCE T.—Perfection Paint & Color Co., Indianapolis.
 HAWKINS, BILL D.—Lilly Industrial Coatings, Indianapolis.
 JASWA, JEFFREY A.—Hanna Chemical Coatings Co., Columbus.
 MAYES, DEBBIE J.—Hanna Chemical Coatings Co., Columbus.
 MCGROARTY, MICHAEL L.—DeSoto Inc., Columbus.
 MESSERLY, MARK T.—Hanna Chemical Coatings Co., Columbus.
 RAYMAN, THOMAS—Hanna Chemical Coatings Co., Columbus.
 RUBIN, ALIPO R. JR.—Hilton-Davis Chemical Co., Cincinnati, OH.

Associate

HELD, WILLIAM L.—Paul Uhlich & Co., Cincinnati, OH.

CLEVELAND

Active

ANDERSON, PATRICK J.—Body Brothers, Inc. Bedford, OH.
 BLACK, DAVID K.—Body Brothers, Inc., Bedford.
 BONFICH, WILLIAM—PPG Industries, Inc., Cleveland, OH.
 DAMKO, STEPHEN F.—Sherwin-Williams Co., Cleveland.
 DAVIS, SHARON L.—Glidden Coatings & Resins Div. SCM Corp., Strongsville, OH.
 DONOVAN, PATRICK K.—Parr, Inc., Cleveland.
 FERCH, DONALD—Sherwin-Williams Co., Cleveland.
 GRIBBLE, PETER R.—Glidden Coatings & Resins Div., SCM Corp., Strongsville.
 HARSCH, EUGENE—Wooster Brush Co., Wooster, OH.
 KOSTANSEK, PAUL—Werner G. Smith, Inc., Cleveland.
 MCDANIEL, ROBERT L.—Lord Corp., Erie, PA
 PONTIUS, J.D.—Sherwin-Williams Co., Cleveland.
 ROTH, THOMAS—Morgan Adhesives Co., Akron, OH.
 SROCKY, M.P.—Foseco, Inc., Cleveland.
 SOLOMON, MARK S.—Body Brothers, Inc., Bedford.
 SONDHE, R.S.—Poly-Crab, Inc., Solon, OH.
 WISNIEWSKI, KARL M.—Parr, Inc. Cleveland.

Associate

BARBER, TOM—Maroon Chemical Group, Cleveland, OH.
 BUFFINGTON, ROY—A.T.&T. Long Lines, Cleveland.
 ROCCO, JAMES—A.T.&T. Long Lines, Cleveland.
 SEEGOTT, PAUL—Seegott Inc., Chagrin Falls, OH.
 WOOLLARD, ROBERT F.—Dow Chemical Co., Cleveland.

Educator and Student

GRAF, ROBERT T.—Case Western Reserve Univ., Cleveland, OH.

DETROIT

Active

BASUTTI, TONY—Armor Paint, Oldcastle Windsor, Ont., Can.
 FLEISCHER, STEFAN—Saran Protective Coatings, Detroit, MI.
 FREER, DAVID E.—Ford Motor Co., Mt Clemens, MI.
 GABRIEL, GABRIEL J.—Mercury Paint Co., Detroit.
 GLINSKI, JAMES M.—Chrysler Chemical Div., Trenton, MI.
 HAYS, DONALD R.—G. M. Fisher Body, Birmingham, MI.
 HENDRICK, PAT—Wyandotte Paint Prods., Inc., Troy, MI.
 HENRY, ROBERT L.—Wyandotte Paints Products, Inc., Troy.
 KYDD, A. THOMAS—Sherwin-Williams Co., Southfield, MI.
 LABANA, S. S.—Ford Motor Co., Dearborn, MI.
 MELGARY, LAWRENCE—Northern Coatings, Menominee, MI.
 MUNBY, JAMES E.—Ciba-Geigy Corp., Troy.
 PICKARD, KARIN—Inmont Corp., Detroit.
 POTOCZEK, STEPHEN Seibert Oxidermo, Inc., Romulus, MI.
 SAGE, MARVIN—Park Chemical Co., Detroit.
 THOMPSON, ANTHONY B.—NL Chemicals Co., Middleburg Hts, OH.
 TRAIKOFF, ROBERT M. Volkswagen of America, Warren, MI.
 WOLPERT, STEPHEN—KMS Fusion, Ann Arbor, MI.

Associate

COLLINSON, CHARLES L.—Baker & Collinson Inc., Detroit, MI.
 DRAKE, SHEILA—Caschem Inc., Palantine, IL.



1982-83 Officers of the Pittsburgh Society. (Left to right): President—William Cibulas; Secretary—Cliff Schoff; Treasurer—Joseph Mascla; Society Representative—Ed Vandervort; Immediate Past-President—Richard Trudel; and President-Elect—Mike Gillen

GALLETINE, DON—George E. Moser & Son Inc., Detroit.
 HOWARD, JOSEPH B.—Mobay Chemical Corp., Troy, MI.
 JAKUBOWSKI, JAMES H.—Spencer Kellogg Div., Textron, Inc., Troy.

HOUSTON

Active

FIELDS, MIKE—Reichhold Chemicals, Inc., Houston, TX.
 GREEN, BILL—Dyco Chemicals & Coatings, Houston.
 JUDLIN, JAMES W.—Devoe & Reynolds Co., Inc., Houston.
 UNIETIS, FRANK—AMF Tuboscope, Inc. Houston.
 VARUGHESE, KURUVILA—O'Brien Corp., Houston.

Associate

FARBER, CHERYL A.—Dow Corning Corp., Houston, TX.
 KEEFER, TONY—Henkel Corp., Houston.
 MCBRIDE, VORNELIUS J.—Reichhold Chemicals, Inc. Houston.

NORTHWESTERN

Active

CLINNIN, DAVID—Cargill, Inc., Minneapolis, MN.
 HAVEN, MICHAEL D.—Ceramic Industrial Coatings, Osseo, MN.
 GLEM, MARK W.—Creative Paint Manufacturing, Minneapolis.

PHILADELPHIA

Active

CASTOR JR., WILLIAM S.—Gulf & Western Industries, Bethlehem, PA.
 LIPSON, FRED J.—Girard Paint Products, Inc., Montgomeryville, PA.
 RUDDICK, DOUGLAS H.—New Jersey Zinc Co., Inc., Palmerton, PA.
 VORNDRAN, JEFFREY—Delkote, Inc., Penns Grove, NJ.

Associate

BETTS, WILLARD JR.—Reichhold Chemicals, Inc., Dover, DE.
 DUNNE, VINCENT F.—McKesson Chemical Co., Philadelphia, PA.
 FIRESTONE, SAM—S.E. Firestone Assoc., Philadelphia.
 HYDE, TIMOTHY S.—Reichhold Chemicals, Inc., Blue Bell, PA.
 KENNARD, JOE—PPG Industries, Dover.
 MATTERN, WILLIAM J.—Reichard-Coulston, Inc., Bethlehem, PA.
 ROYAL, LEW—H. M. Royal, Inc., Trenton, NJ.
 VERMEYCHUK, J. G.—Hercules Inc., Wilmington, DE.

PIEDMONT

Active

EVERTS, JACK A.—Paint Products Co., Walkertown, NC
 WATERS, BECKY—Ashland Chemical Co., Charlotte, NC

Associate

HOUSTON, JERRY A.—Rohm & Haas Co., Charlotte, NC
 SURIANI, DAVID M.—BASF Wyandotte, Matthews, NC

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Rochester Institute to Hold Summer Color Courses

Three summer courses will be presented by the Rochester Institute of Technology, featuring Dr. Fred W. Billmeyer, Jr., of Rensselaer Polytechnic Institute, and Max Saltzman, of the University of California at Los Angeles.

Color Technology for Management will be offered June 1-2. It covers the principles of color technology as they influence management decisions. The program explores the physical and perceptual aspects of color, color measurement, color differences and tolerances, and color matching. Typical problems in the production and sale of colored products will be confronted, and managerial solutions based on the principles of color technology will be presented. The course will show how to improve productivity by applying the principles of color technology to reduce waste and make better use of manpower and equipment. The course is designed solely for executive and management personnel responsible for the production and sale of colored products. Anyone directly involved in color matching and color control is encouraged to enroll in either or both of the other two courses. Tuition

for *Color Technology for Management* is \$550.

Principles of Color Technology will be offered twice, June 6-10 and June 13-17. The course provides information on color description, color-order systems, measurement principles, color-difference calculations and tolerances, computer color matching, and colorant properties. Laboratory periods provide hands-on experience in measurement, computation and problem-solving using the latest commercial equipment. The course is of value to individuals from a variety of disciplines and organization levels who are interested in color science, and especially to industrial personnel involved in color matching and color control. Attendance is limited to ensure maximum participation in laboratory sessions. Tuition is \$600.

Advances in Color Technology will be offered June 20-24. It provides current, advanced-level information on developments in and techniques of color science and technology. Topics include instruments, calibration, and measurement errors; terminology and standards; color spaces and color differences; color ap-

pearance; and turbid-medium theory and color matching. Selected advanced laboratory workshops are included. The course is designed for those with two or more years of direct experience in instrumental color measurement. Industrial personnel involved in color matching and color control at an advanced level are cordially invited. Elementary material is *not* included, and applicants without previous experience should enroll in *Principles of Color Technology*. Tuition for *Advances in Color Technology* is \$600.

For more information on these programs, contact Val Johnson, T&E Center Seminar Coordinator, Rochester Institute of Technology, One Lomb Memorial Drive, P. O. Box 9887, Rochester, NY 14623.

Chromatographic Course To Be Held at Kent State

"Fundamentals of Chromatographic Analysis," will be presented at Kent State University, Kent, OH, April 18-21. The course is cosponsored by the KSU Chemistry Department, IBM Instruments and the University Conference Bureau.

The course will provide a coherent overview of chemical separations via chromatographic methods. It will be directed towards the beginning to intermediate chromatographer and will be unique from most programs in that material will be included on gas, liquid and thin-layer methods. The course will stress the three techniques as complementary rather than competing processes and will be a blend of fundamental information on theory and instrumentation with applications.

The lecturers will be Dr. Roger K. Gilpin, IBM Instruments, and adjunct Associate Professor at Kent State University; Dr. Theodore Provder, Senior Scientist, Glidden Coatings and Resins Division of SCM Corporation; Dr. Fred E. Regnier, Professor, Purdue University; and Dr. Neil D. Danielson, Assistant Professor, Miami University. Instruments will be shown by Ronald L. Lewis of IBM Instruments.

Additional information can be obtained from Carl J. Knauss, Chromatographic Course Coordinator, Chemistry Department, Kent State University, Kent, OH, 44242.

Du Pont to Hold Seminar on Explosion Prevention

Industrial explosion prevention and protection techniques developed by a Du Pont Company expert will be presented in a seminar and workshop in New Orleans, April 19-21. The two and one-half day session, "Industrial Explosion Prevention and Protection," will be taught by Dr. Frank T. Bodurtha, a widely recognized authority in the field. Dr. Bodurtha, of Du Pont's Engineering Department, has worked for more than 20 years in explosion prevention and protection.

The program is designed for industrial, academic and governmental scientists; engineers with safety, process and management responsibilities; and research and development personnel. It is also appropriate for those in manufacturing, operations, engineering, maintenance and design engineering.

The seminar will teach participants how to recognize explosion hazards, save time in analysis and correction of hazards and determine low-cost solutions to ex-

plosion problems. Also included are techniques for determining when assumed explosion hazards do not exist, explosion investigation and decreasing injuries and property losses.

A few of the subjects covered in lecture and workshop sessions are flammability limits of gases and vapors; ignition sources and auto-oxidation; explosion pressure and blast effects; and explosion containment, suppression and venting. Also included are atmospheric releases, flame arresters, behavior of dense stack gases and a number of other topics.

Early registration is recommended because class size is limited to 40 persons. The \$795 registration fee includes luncheons, coffee breaks and meeting materials. In-house sessions can be arranged for companies or institutions on a reduced-fee basis. For a brochure and registration form write: Explosion Protection, Du Pont Company, Room X-39700, Wilmington, DE 19898.

Carnegie-Mellon to Sponsor 'Colloids and Interfaces' May 23-27

"Colloids and Interfaces", a five-day seminar now in its eighth consecutive year, will be offered May 23-27 at Carnegie-Mellon University, Pittsburgh, PA.

This intensive seminar continually attracts and responds to the needs of those

scientists and engineers involved with the unique effects of colloid and surface phenomena in industrial processes.

Designed specifically for anyone who works with adhesives, sealants, wetting agents, pigments, paints, printing inks, etc., this course will provide a foundation in the fundamentals as well as an introduction to the latest research techniques. The session covers not only equilibrium phenomena but also key aspects of flow and transport near interfaces and in colloidal systems. The course format provides ample problem-solving sessions for individual interaction with the faculty, coupled with basic theory lectures and daily laboratory demonstrations.

Chairman for the course is Dr. Geoffrey D. Parfitt, Professor of Chemical Engineering, Carnegie-Mellon University, and President of the International Association of Colloid and Interface Scientists.

Tuition for the session is \$825.

For information, contact Frank E. Nowak, Director of Post College Professional Education, 405 MMCH, Carnegie-Mellon University, Pittsburgh, PA.

Chicago Society To Sponsor 'Symco '83'

The Chicago Society for Coatings Technology will hold its 13th Annual Chicago Area Coatings Symposium, "Symco '83" on April 19-20 at Knickers Restaurant, Des Plaines, IL. Entitled, "E.T.—Emerging Technologies: A Closer Encounter," the symposium will be devoted to industrial coatings and trade sales.

For additional information, contact Chairman Ross Johnson, The Enterprise Companies, 1191 S. Wheeling Rd., Wheeling, IL 60090.

New York Society To Award Scholarships

The New York Society of Coatings Technology is offering several scholarships to assist students of chemistry and chemical engineering attending recognized institutions of higher learning. Students working in the coatings industry, and students of parents within the industry, will be given preference.

The Society's Scholarship Committee requests that each applicant submit a letter with information about his or her school, course of study, extra curricular activities, means of payment for studies, and why he/she should be awarded the scholarship.

Letters should be sent to the Scholarship Committee Chairman, Marvin Wexler, New York Society, 520 Westfield Ave., Elizabeth, NJ 07208 before March 31, 1983.

CALL FOR PAPERS "RUST NEVER SLEEPS"

8TH ANNUAL **FOCUS** CONFERENCE
FUTURE OF COATINGS UNDER STUDY

Detroit Society for Coatings Technology

May 4, 1983

Management Education Center
Troy, Michigan

Conference will FOCUS on the latest technologies which will be required by automobile manufacturers in order to achieve increased cosmetic and perforation resistance standards. Solicited papers should cover the following areas:

- Corrosion resistant sheet steel
- Cleaning and phosphating update
- New resins and coating systems
- Electrodeposition update
- New and improved corrosive pigments
- New apparatus and test methods for corrosion research

Those wishing to participate are urged to submit a letter of intent including a tentative title of paper as soon as possible and an abstract of about 200 words by February 28, 1983.

Abstracts on any subjects related to automobile corrosion prevention would be welcomed. Please forward all communications to: The Detroit Society for Coatings Technology, 765 Dellwood Drive, Ann Arbor, Michigan 48103. Telephone (313) 589-3660.

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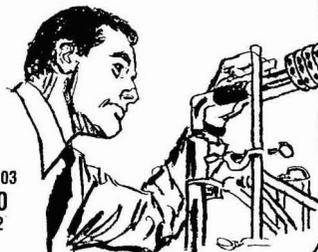
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People

Dr. William G. Boberski has been named Technical Manager, Heavy Duty Maintenance Products in the PPG Industries, Coatings and Resins Div., Research and Development Center, Springdale, PA. He is a member of the Pittsburgh Society.

Frank W. Buck has been promoted to Vice-President and General Merchandising Manager, Filtration and Minerals Div., Manville Products Corp. Based at the firm's Denver headquarters, Mr. Buck has overall marketing responsibility for all filtration and minerals products. He is a member of the Rocky Mountain Society.

Ashland Chemical Co., in a realignment of senior management, has named **John A. Brothers** Senior Vice-President, Operations. Reporting to him are the following group Vice-Presidents: **Anton Dorfmueller**, **Donald L. Coticchia**, and **David J. D'Antoni**. **Scotty B. Patrick** has been named Administrative Vice-President, Technical.

CDI Dispersions, Newark, NJ, has announced the appointment of **Thomas E. Smith** to the position of Product Development Manager.

Glenn E. Taylor has joined the Minerals & Chemicals Div. of Engelhard Corporation as Director of Manufacturing for the Pigments & Extenders Group. Also, **Andrew R. Negele** was named Technical Service Chemist for the Group.

John Blunt has joined Ferro Corporation's Composites Div. as Manager of Research and Development, responsible for the Western Div. Mr. Blunt was formerly with Con/Chem, Inc. as Technical Director. He is a member of the Los Angeles Society.

Also announced by Ferro Corp. were the promotions of **William H. Meek** to Business Manager, Specialty Organics, and **Dennis L. Hammond**, to Technical Director at the firm's chemical division in Bedford, OH.

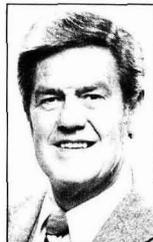
Georgia-Pacific has announced the promotion of **Steven J. Klestinec** to Plant Manager of its Thermosetting Resins plant in Peachtree City, GA.



W.G. Boberski



F.W. Buck



R.O. Yates



G.D. Lutz

Robert O. Yates has been named President of Norris Paint Co., Inc., Salem, OR.

Thomas F. Wagner was named Vice-President—Chemical Coatings of SCM's Glidden Coatings & Resins Div. He replaces **Arne C. Lindholm**, who has retired.

H.D. Wenstrup was appointed Vice-President and Director of Marketing of Chemcentral Corp., Chicago, IL.

Murray Gladstone, of Gladstone & Sons, Woodmere, NY, has been elected President of the National Decorating Products Association, St. Louis, for 1982. **Carol Orr**, of Colour Shoppe Interiors, Saratoga, CA is Vice-President, and **Lynn DeFries**, of Elkhart, IN, is Treasurer.

Nalco Chemical Co., Oak Brook, IL, has named **John B. Cruel**, formerly Vice-President, Human Resources, to the position of Vice-President, Corporate Sales. **Edward J. Mooney** will assume Mr. Cruel's former position. Promoted to General Manager, Specialty Chemicals, was **Graham H. Jackson**.

Randall L. Ehmer has joined Walsh & Associates, Inc., as a Sales Representative based at the firm's North Kansas City office.

Bruce S. Schimmel was promoted to the position of Sales Representative of Alcan paste and pigments for Alcan Ingot and Powders, Union, NJ.

Theodore D'Amico was named Sales Manager for Ultra Adhesives, Inc., Paterson, NJ.

George D. Lutz has been promoted to Senior Vice-President of Bee Chemical Co., Lansing, IL. He will assume responsibility for coated film, coatings, injecta color, and universal color dispersions.

Lan Maycock has been named Product Manager for acetyls and acrylics in the Solvents & Coatings Materials Div., Union Carbide Corp., Danbury, CT.

Edward A. McCarthy has been appointed Technical Sales Specialist for the Packaging Vice-Group of the Midland Div., Dexter Corp. He is a member of the Chicago Society. Also, **John P. Knudtson** has been named Manager, Packaging Product Development.

Henkel Corp., Minneapolis, MN, has announced several organizational changes in its Chemical Products Div. **Eugene M. Fischer** has been appointed Director of Manufacturing Technology, based at the Minneapolis headquarters. **Alan J. Pranica** was promoted to Works Manager and **Brendan J. Murphy** was named Assistant Works Manager at the firm's Kankakee plant.

The appointments of **Carl A. Mason**, **Robert P. Serreti**, and **Larry M. McGrenera** to new manufacturing positions in the Organics Division, have been announced by Witco Chemical Corp. Mr. Mason was assigned to the position of Manager of Manufacturing Operations—Midwest, based at the firm's Clearing plant in Chicago. Mr. Serreti was named Plant Manager of the company's Perth Amboy, NJ plant. Mr. McGrenera was promoted to Production Manager at the Clearing plant where he recently served as Assistant Plant Manager.

Frank E. Bolway, Jr., Chairman and CEO of the D.H. Litter Co., Inc., New York, NY, has announced that **Sidney B. Levinson** has been elected Chairman and **Saul Spindel** was named President of the D/L Laboratories.

Sid Levinson, who previously served as President, will continue as Chief Executive Officer with Saul Spindel as Chief Operating Officer.

Mr. Levinson, who is in his 50th year in the paint and coatings industry, is a graduate chemist and chemical engineer.

He joined the D.H. Litter Co. as its Technical Director in 1952. He became President of the David Litter Laboratories Div. which eventually changed its name to the D/L Laboratories.



Nationally recognized as a consultant in all phases of the technology of paints and coatings, Mr. Levinson has presented over 60 talks and written over 30 articles, chapters in five books, and a manual for the armed services. He is a member of the Federation of Societies for Coatings Technology ASTM, ACS, NACE, and SSPC. Mr. Levinson is a past-president of the New York Society and received their highest award, the PaVaC Award. At present, he is Chairman of Committee D-1, Paints and Coatings of ASTM and has been made an Honorary Member of both Committee D-1 and ASTM. He also is the FSCT delegate to SSPC.

Saul Spindel began his employment with the D/L Laboratories in 1959 as a chemist. He progressed to Group Leader,



Chief Chemist, and then Vice-President and Technical Director, the position he held until his present promotion.

Mr. Spindel is a graduate of Brooklyn College with over 30 years of experience in all phases of paint and coatings technology. He is both a Corrosion Specialist and Professional Engineer in Corrosion. He is a member of the Federation of Societies for Coatings Technology ASTM, ACS, and NACE. Mr. Spindel also is a past president of the New York Society. At present he is Chairman of the Corrosion Committee of the Federation, Society Representative for the New York Society, and a member of the Executive Committee of ASTM Committee C-24 Caulks and Sealants.

Obituary

J. Robert "Bob" English, retired partner of R.T. Hopkins Co., Atlanta, GA, died November 26. He was 69 years old.

Mr. English, elected by the Southern Society in 1982 to Honorary Membership, was a founding member of the Society and served as its President in 1952. He also served as the Publicity Chairman of the Atlanta Section and Society Historian.



Mr. English attended Georgia Tech from 1931 to 1936, and entered the paint industry while still in school, as a co-op student in chemical engineering. He continued to work in formulation and production of paints until World War II, including involvement in development of the original reflectorized traffic paints. During the war he switched to explosives manufacturing for a few years, but returned to coatings in 1945 as Executive Vice President of Leland Moore Paint & Oil Co., of Charleston, S.C. There he also served on the War Production Board, Civilian Production Administration, and Office of Price Administration. During his "second career" as a supplier, he was associated with the universally recognized "Dean" of manufacturers' representatives in the industry, R.T. Hopkins, ably representing several prominent raw material manufacturers.

Robert L. Hoerner, 55, Manager of the Chemical Coatings Laboratory of Pratt & Lambert, Inc., Buffalo, NY, died on December 21.

A 19-year member of the Western New York Society, Mr. Hoerner had served as the society's President in 1968 and was elected in 1982 as its Society Representative.

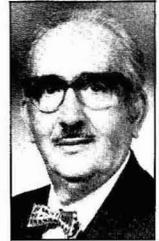
Mr. Hoerner joined Pratt & Lambert, Inc., in 1946 as a laboratory assistant and was promoted through a series of responsibilities until his most recent position which he assumed in 1977.

He was a native of Buffalo and was graduated, summa cum laude, from Canisius College.

He is survived by his wife, Frances.

Dr. David M. Gans, 77, Director Emeritus of Coatings Research Group, Inc., died on December 23.

Dr. Gans joined CRGI as its Director in 1962, when the laboratory was moved from its original location in Bethesda, MD to its present quarters in Cleveland, OH. When he retired in 1972, the CRGI board named him Director Emeritus for his years of distinguished service.



He was an Honorary Member of both the Cleveland Society and of the Federation, which, in 1972, bestowed on him its most prestigious recognition, the Heckel Award. In 1976, he received the Distinguished Service Award of the Cleveland Technical Societies Council.

Dr. Gans was a graduate of the University of Chicago, where he received his Ph.D. in physical chemistry. After teaching there for several years, he served in a series of positions in industry, including nine years with Interchemical (now Inmont) Corp. as Assistant Director of Research; four years with Quaker Chemical Products Co. as Technical Director, and 14 years with the Arco Co. as Vice-President of Research.

He is survived by his wife, Marion (Molly), two sons, Paul and Daniel, and four grandchildren.

Dr. Berthold Winston, of Absolute Coatings, Inc., died October 9. Dr. Winston had been associated with the coatings industry for more than 30 years. He was, for many years, a member of the New York Society, the American Chemical Society, the National Association of Corrosion Engineers, and the American Institute of Chemical Engineers. He is survived by his wife and a daughter.

George Binder, 43, Technical Director for Stabler Paint Manufacturing Co., died December 4.

Robert Lee Gray, Jr., 60, Vice-President, Sales, of the Bisonite Co., Inc., Tonawanda, NY, died on December 14.

Powder-Coated Tubing

A brochure is available which describes HI-COAT™ powder-coated tubing. Its advantages are featured which include: toughness, beauty, durability, easier handling, packing, and shipping. For a copy, contact Battelle Columbus Laboratories, 505 King Ave., Columbus, OH 43201.

In-Mold Plating

A low-cost, energy-efficient method to coat plastic materials with metals is the subject of new literature. The procedure, known as in-mold plating, is explained and its advantages are noted. For additional information, contact Battelle Columbus Laboratories, 505 King Ave., Columbus, OH 43201.

Temperature Instruments

A broad line of non-contacting, infra-red temperature monitoring and indicating instruments is described in an illustrated catalog. The new four-page publication details specifications, operating characteristics, and prices of seven instruments, including two miniaturized, pocket-size units, two pistol-grip instruments, two bench-style models, and a digital emissivity meter. All of the instruments can sense a wide range of temperatures at distances up to 15 feet or greater. For copies of catalog No. 1200-SB1, write LFE Corp., Advertising Dept., 55 Green St., Clinton, MA 01510.

Instruments

The 1983 edition of Transcatalog®, contains complete mail-order descriptions of the test, measurement, and calibration instruments and accessories from 150 selected manufacturers, has been published. More than 120 new items have been added to this expanded edition. Transcatalog is designed for the plant operations, maintenance, electrical, and instrumentation professionals who work with physical parameters in lab, shop, plant, or remote site. Product scope covers temperature, pressure, flow, sound, vibration, pH, conductivity, humidity, velocity, viscosity, frequency, R-C-L, and more. The comprehensive coverage includes applications, specifications, prices, photos, connection diagrams, selection guides, and ordering information. To get a free copy, contact TRANSCAT, Box D-1, Rochester, NY 14606.

ILAC Directory

The ILAC (International Laboratory Accreditation Conference) Directory 1981, a new, basic guide in the field of product testing and world trade, has been recently released by ASTM. The directory is a unique, international guide to which organizations accredit what testing techniques in electrical and mechanical engineering; industrial and domestic equipment; civil and chemical engineering; instrumentation; and biological sciences. Divided into five parts, the directory provides information on general testing arrangements; laboratory accreditation systems; other laboratory accreditation and approval systems; international systems; and tentative systems. Further information can be obtained from ASTM, Sales Services Dept., 1916 Race St., Philadelphia, PA 19103.

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Preservative

A six-page brochure explaining a new packaging concept of marketing Dowicil 75 preservative in water-soluble polyvinyl alcohol (PVA) bags is now available. Several advantages are listed as are concentration charts. For a copy of literature 192-900-982, contact Dow Chemical U.S.A., Designed Products Dept., 2040 Dow Center, Midland, MI 48640.

Photoinitiator

A new, highly efficient nonyellowing photoinitiator, Irgacure® 184, has been introduced in recent literature. Its effectiveness in ultraviolet curable coatings for paper, plastics, flooring, and metals is highlighted. Absorption properties, curing efficiencies, and color resistance characteristics are detailed. For a copy, contact Additive Dept., CIBA-Geigy Corp., Saw Mill River Rd., Ardsley, NY 10502.

Resin

A new coating resin which offers all of the performance characteristics of a pure acrylic resin, at considerably less cost, is the subject of a recently published technical bulletin. The uses of this new modified acrylic copolymer are discussed along with its exceptional advantages. Contact Reichhold Chemicals, Inc., 525 N. Broadway, White Plains, NY 10603 for more information.

Activated Carbon

A line of pelletized activated carbon products especially suited to systems for the recovery of volatile organic solvents in industrial and commercial operations has been introduced in new literature. Witcarb® Columbia® pelletized products lend themselves to solvent recovery systems in a wide range of industries including the recovery of acetates, alcohols and hydrocarbons in the gravure printing field, ketones in the acetate fiber industry, hydrocarbons and chlorinated solvents in rubber production, chlorinated and fluorinated solvents in metal degreasing operations, and ketones, acetates and hydrocarbons in the coating of paper, plastic films, and fabrics. Data are provided on their usefulness in controlling undesirable air emissions and odors. Further information is obtainable from the Inorganic Specialties Div., Witco Chemical Corp., 277 Park Ave., New York, NY 10017.

Urethane Oligomer

A data sheet is available featuring GAFGARD 238 Urethane Oligomer, a general purpose, nonyellowing aliphatic urethane-based oligomer which can be cured with ultraviolet light or electron beam radiation to form a film which exhibits good scuff, stain and solvent resistance, gloss retention, and flexibility. For a copy of #2300-101, write GAF Corp., Commercial Development, 140 W. 51 St., New York, NY 10020.

Surfactant

A new anionic surfactant which exhibits detergency, emulsification, dispersion, foaming and stabilizing properties is the subject of recently introduced literature. Atsurf® 1910, an aqueous alcohol solution of the ammonium salt of a sulfated alkylphenoxypoly-(ethyleneoxy) ethanol, has potential for a variety of applications which are discussed in the brochure. Write ICI Americas, Inc., Wilmington, DE 19897 for additional information.

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Methylene Chloride

A new 21-page brochure outlining the many applications and characteristics of methylene chloride is now available. The advantages of methylene chloride, including efficiency, performance, positive ecological impact and safety are detailed. Some of the major applications for this solvent include vapor degreasing, paint stripping, urethane foam blowing, chemical processing, and in the food and pharmaceutical areas. For copies of the brochure No. 100-5804-81, write Susan King, Inquiry Correspondent, Inorganic Chemicals, Dow Chemical U.S.A., 1703 South Saginaw Rd., Midland, MI 48640.

Technical Index

A comprehensive index to the Goodyear's Chemical Division's technical literature is now available. Annually, the Chemical Division publishes new and revised technical material, brochures, and booklets containing information on the use, compounding, and handling of its complete line of chemical products for the manufacturing industries. The new list of technical material contains more than 150 references to published information. For information, contact Goodyear Chemicals Data Center, P.O. Box 9115, Akron, OH 44305.

Polymer

A polymer designed to improve leveling and increase viscosity of latex enamels is introduced in new literature. Its effectiveness and advantages are listed in a technical bulletin available from Troy Chemical Corp., One Avenue L, Newark, NJ 07105.

Pump

A new four-page brochure discusses how a Netzsch NESP Progressive Cavity Pump and Piping System can be used to transport dewatered sludges, manufacturing process wastes, and a wide range of other semi-liquid materials with high solids concentrations, and high viscosities. Many materials previously moved on conveyors and thought to be unpumpable in closed piping systems over long distances and high lifts are covered. Also included is the principle of operation of the Netzsch NESP Progressive Cavity Pump with bridge breaker and auger feed as well as other technical information. For a free copy of the brochure, write Netzsch Inc., Nemo® Pump Div., 119 Pickering Way, Exton, PA 19341.

Water-Reducible Products

Water-reducible products for trade sales and industrial coatings are featured in a new bulletin. The literature contains a physical properties table for such emulsion products as Wallpol[®] polyvinyl acetate copolymers, vinyl acetate homopolymers, and Synthemul[®] acrylic and styrene/acrylic emulsion resins. Water-reducible acrylic solution resins and specialities are also included. The bulletin is available from Reichhold Chemicals, Inc., 525 North Broadway, White Plains, NY 10603.

Universal Dispersions

Literature is available featuring tinting black, phthalo green, and phthalo blue (R/S, NCF) universal dispersions. Discussed are these compounds' improved properties compared with the existing commercially available products of this type. Also, their advantages such as their ability to exhibit high coloring strength and less flooding and floating tendencies, are listed. For additional information, write CDI Dispersions, 27 Haynes Ave., Newark, NJ 07114.

Polypropylene

A specialty-grade Pro-fax[®] polypropylene designed for high-speed extrusion coating and laminating applications is featured in recent literature. Its application uses, advantages, and properties are listed. Write Hercules Incorporated, Product Inquiry, 910 Market St., Wilmington, DE 19899 for more information.

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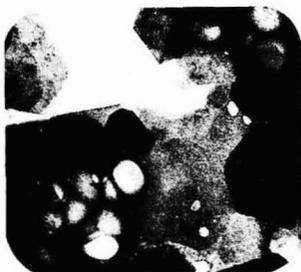
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Merits of Zinc Oxide Further Discussed by AM Participant

TO THE EDITOR:

A panel discussion on performance of non-lead, non-chrome pigments in aqueous and solvent based coatings was held on November 5 at the Annual Meeting in Washington. Each of five panelists presented a talk on a proprietary pigment extolling its merits after which an open forum was held with an invitation for the audience to participate.

In my turn, I began to discuss the merits of zinc oxide, a generic pigment, and was about midway when the moderator stopped me by stating that zinc oxide was not one of the pigments on the program. I defended the relevance of zinc oxide, and indeed my comments had dealt with zinc oxide as a pigment in primers for metal. Nevertheless, he ruled against continuing.

Because attendees may have felt entitled to hear my comments, I include them below:

"The Steel Structures Painting Council (SSPC) conducted a large panel study on primers applied over hand-cleaned as well as blasted steel and found oil/alkyd zinc oxide primer to be in a performance category exceeded only by lead and chromate primers. In addition, a fabrication shop has used this primer for four years under pressure to remove lead pigment. Use of this primer has recently been extended to the entire plant. As a consequence, SSPC has adopted a specification utilizing zinc oxide pigment appearing in the third edition of their painting manuals.

"If red lead pigment has a fine particle size, the required amount per gallon is one pound, not the 19 or 22 pounds per gallon that our industry has used for years.

"With zinc oxide the required level is also one pound per gallon for a one to one linseed oil/long oil alkyd binder. Increasing the alkyd reduces the needed or permissible amount.

"Red lead has three functions: (1) to neutralize film acids; (2) to maintain the steel at a pH which is in the passivation range according to Pourbaix diagram, and; (3) to insolubilize sulfates and chlorides that are present at the steel/paint interface or that have been introduced with other pigments.

"Zinc oxide functions in the first two ways above, that is, neutralizing film acids and maintaining the pH.

"The advantage red lead has over zinc oxide is the insolubilization of chloride and sulfate ions."

WILLIAM C. JOHNSON,
Consultant
Kinterville, PA

In response to Bill Johnson's letter:

At the conclusion of the formal presentations by the panelists, I opened the session up to questions from the floor. Shortly thereafter, Mr. Johnson was recognized and, instead of asking a question, began to discourse about zinc oxide. I listened for what I felt was a reasonable period of time and then interrupted his statement to ask whether or not he was leading up to a question. He said, "No," but rather wanted to discuss the use of zinc-oxide in anti-corrosive

coatings. I requested that, inasmuch as we were on a tight schedule, (the session was the last one on Friday afternoon) he stop reading his prepared statement and wait until the end of the program when, if there was still time, he could have the floor back to continue.

There was time at the end but when I asked if anyone else had anything more to ask or say, I received no response from the audience. Apparently, Mr. Johnson either chose not to speak or had left before the conclusion of the program.

This panel, and the ensuing discussion, was most worthwhile and a credit to the Federation Corrosion Committee which arranged it.

SAUL SPINDEL,
Chairman,
FSCC Corrosion Committee

Author Comments on Zero Contact Angle Fallacy

TO THE EDITOR:

Nearly forty years ago I drew attention [D.M. Gans, *J. Phys. Chem.*, 49, 165 (1945)] to the widely used but incorrect assumption that a liquid which readily wets a solid surface thereby forms a contact angle of zero on that surface. I have expounded at length on this subject in more recent publications, in a series in this journal [D.M. Gans, *JOURNAL OF PAINT TECHNOLOGY*, 39, 501 (1967); 41, 515 (1969); 42, 653 (1970)] and in a chapter on interfacial energetics (D.M. Gans, *Treatise on Coatings*, Vol. 2, Part 2, R.R. Myers and J.S. Long, Eds., Marcel Dekker, New York, 1976, p 249). My dismay should therefore be understandable whenever I see a statement in the literature that affirms this zero contact angle fallacy. For example, Dr. Swaraj Paul, in column 2 on page 61 of his recent review (JCT, September 1982), states:

"Liquid is said to be nonspreading if $\theta > 0$, whereas it is said to wet the surface completely when $\theta = 0$. In the latter case, rate of spread is dependent on the viscosity of the liquid and the roughness of the solid surface".

Nonspreading and wetting in relation to zero contact angles is a complicated subject whose intricacies are exposed in detail in my chapter in the *Treatise on*

Coatings cited above. When spontaneous spreading occurs, the original solid-vapor (SV) surface is replaced by the solid-liquid (SL) interface and the liquid-vapor (LV) surface. After spontaneous spreading, only the SL interface and the LV surface remain. They are parallel to each other. There is no location at which the three phases come together. There is no contact point or line and hence no contact angle. To call this an instance of a zero contact angle is wrong.

What I am talking about is not a mere exercise in semantics. In speculations, interpretations, or calculations dealing with interfacial energetics, where spontaneous spreading is often explicitly or implicitly involved, we must avoid the pitfall of assuming the existence of a zero contact angle since this allows faulty conclusions. I discuss this more fully on pages 272 through 280 in my chapter in *Treatise on Coatings*. In short, zero contact angle is not the same as no contact angle.

If a liquid does not appear to spread on or wet a surface, either (1) this is a classical nonspreading situation with a real finite contact angle or (2) the liquid would but cannot spread spontaneously because of rheological restraints, as in the case of a sufficiently pigmented paint.

DAVID M. GANS,
Beachwood, OH

Coming Events

FEDERATION MEETINGS

(Apr. 26-27)—Federation Seminar on "The Efficient Operation of an Up-to-Date Paint and Coatings Laboratory." Hilton Plaza Inn, Kansas City, MO. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, PA 19107).

(May 19-20)—Spring Meetings. Society Officers on 19th; Board of Directors on 20th. Terrace Hilton Hotel, Cincinnati, OH. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, PA 19107).

(Oct. 12-14)—61st Annual Meeting and 48th Paint Industries' Show. Place Bonaventure, Montreal, Quebec, Canada. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, PA 19107).

1984

(May 17-18)—Spring Meetings. Society Officers on 17th; Board of Directors on 18th. Galt House, Louisville, KY. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, PA 19107).

(Oct. 24-26)—62nd Annual Meeting and 49th Paint Industries' Show. Conrad Hilton Hotel, Chicago, IL. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, PA 19107).

SPECIAL SOCIETY MEETINGS

(Feb. 23-25)—16th Biennial Western Coatings Societies' Symposium and Show. Hyatt Regency, San Francisco, CA. (Ted Favata, Chairman, Triangle Coatings Co., 2222 Third St., Berkeley, CA 94710).

(Mar. 8)—Northwestern Society. Annual Coatings Symposium. Marriott Hotel, Bloomington, MN. (Paul Sara, Valspar Corp., P.O. Box 1461, Minneapolis, MN 55440).

(Mar. 16)—Louisville Society Spring Symposium on Coatings Plant Safety. Marriott Inn, Clarksville, IN. (Don Collier, Porter Paint Co., 400 S. 13th St., Louisville, KY 40203).

(Mar. 23-26)—Southern Society Annual Meeting. Peabody Hotel, Memphis, TN. (William E. Early, Piedmont Paint Mfg. Co., P.O. Box 6223, Stn. B, Greenville, SC 29606).

(Apr. 13-15)—Southwestern Paint Convention. Lowe's Antole Hotel, Dallas, TX.

(Apr. 19-20)—Chicago Society for Coatings Technology Symc '83—"E.T.—Emerging Technologies: A Closer Encounter." Knickers Restaurant, Des Plaines, IL. (Chairman Ross Johnson, The Enterprise Companies, 1191 S. Wheeling Rd., Wheeling, IL 60090).

(Apr. 26-27)—"Advances in Coatings Technology." 26th Annual Technical Conference of the Cleveland Society for Coatings Technology. NASA-Lewis Research Center, Cleveland, OH. (Dr. Richard R. Eley, Glidden Coatings & Resins, P.O. Box 8827, Strongsville, OH 44136).

(May 4)—Detroit Society for Coatings Technology FOCUS—"Corrosion Resistance." Michigan State University Education Center. (Peter Burnett, Wyandotte Paint Products Co., 650 Stephenson Hwy., Troy, MI 48084).

(May 5-7)—Pacific Northwest Society Symposium. Thunderbird Inn, Portland, OR. (Chairman Gerry McKnight, Lilly Industrial Ctg. Inc., 619 S.W. Wood St., Hillsboro, OR 97123).

(June 10-11)—Joint meeting of Kansas City and St. Louis Societies. Holiday Inn, Lake of the Ozarks, MO.

OTHER ORGANIZATIONS

(Feb. 19-20)—Fourth Annual Short Course on "Adhesion Theory and Practice" sponsored by The Adhesion Society. Sheraton Savannah Inn, Savannah, GA. (G.F. Hardy, Treasurer,

The Adhesion Society, c/o Celanese Research Co., P.O. Box 1000, Summit, NJ 07901).

(Feb. 20-23)—The Adhesion Society's Sixth Annual Meeting. Sheraton Savannah Inn, Savannah, GA. (Prof. James Koutsky, University of Wisconsin, Chemical Engineering Dept., 1415 Johnson Dr., Madison, WI 53706).

(Feb. 22-24)—"Estimating for Painting Contractors and Maintenance Engineers" Short Course. Marriott Hotel, Bloomington, MN. (Arts & Sciences Continuing Education, G-7 Humanities-Social Sciences Bldg., University of Missouri-Rolla, Rolla, MO 65401).

(Feb. 24-25)—"Fundamentals of Color" Seminar. Houston, TX. (Jeanne M. Dolan, Macbeth, Div. of Kollmorgen Corp., Little Britain Rd., P.O. Box 950, Newburgh, NY 12550).

(Mar. 1)—"Conversion Coatings as a Substrate for Organic Finishes" Workshop sponsored by the Association for Finishing Processes of the Society of Manufacturing Engineers. Baltimore Convention Center, Baltimore, MD. (Donna Theisen, Technical Activities Dept., SME, One SME Dr., P.O. Box 930, Dearborn, MI 48128).

(Mar. 1-4)—Fourth International Cadmium Conference. Bayerischer Hof Hotel, Munich, West Germany. (Cadmium Council Inc., 292 Madison Ave., New York, NY 10017).

(Mar. 5-6)—Canadian Decorating Products Show. Skyline Hotel, Toronto, Ont. (NDPA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132).

(Mar. 10-11)—"Fundamentals of Color" Seminar. Birmingham, AL. (Jeanne M. Dolan, Macbeth, Div. of Kollmorgen Corp., Little Britain Rd., P.O. Box 950, Newburgh, NY 12550).

(Mar. 14-18)—"Composition of Coatings" Short Course. University of Missouri-Rolla, Rolla, MO. (Norma Fleming, Arts & Sciences, Continuing Education, G-6 H/SS, UMR, Rolla, MO 65401).

(Mar. 24-25)—"Fundamentals of Color" Seminar. Charlotte, NC. (Jeanne M. Dolan, Macbeth, Div. of Kollmorgen Corp., Little Britain Rd., P.O. Box 950, Newburgh, NY 12550).

(Mar. 26-27)—Western Decorating Products Show. Disneyland Hotel, Anaheim, CA. (NDPA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132).

(Mar. 28-31)—"Cutting Costs with Computers and Automated Systems"—National Plant Engineering & Maintenance Conference and Exhibition. McCormick Place, Chicago, IL. (Clapp & Poliak, A Cahners Exposition Group Co., 708 Third Ave., New York, NY 10017.)

(Apr. 10-12)—Inter-Society Color Council Annual Meeting. Louisville, KY. (Fred W. Billmeyer, Dept. of Chemistry, Rensselaer Polytechnic Institute, Troy, NY 12181).

(Apr. 11-12)—23rd Annual Symposium of the Washington Paint Technical Group. Marriott Twin Bridges Hotel, Washington, DC. (Mildred A. Post, Publicity Chairperson, WPTG, P.O. Box 12025, Washington, DC 20005).

(Apr. 12-14)—ASTM Committee C-22 on Porcelain Enamel and Related Ceramic-Metal Systems Meeting. ASTM/Philadelphia Centre, Philadelphia, PA. (Drew Azzara, ASTM, 1916 Race St., Philadelphia, PA 19103).

(Apr. 14-15)—"Fundamentals of Color" Seminar. Seattle, WA. (Jeanne M. Dolan, Macbeth, Div. of Kollmorgen Corp., Little Britain Rd., P.O. Box 950, Newburgh, NY 12550).

(Apr. 17-20)—National Coil Coaters Association Annual Meeting. Marriott's Marco Beach Hotel, Marco Island, FL. (NCCA, 1900 Arch St., Philadelphia, PA 19103).

(Apr. 18-21)—"Fundamentals of Chromatographic Analysis." Kent State University, Kent, OH. (Dr. Carl J. Knauss, Chemistry Dept., Kent State University, Kent, OH 44242).

(Apr. 18-22)—Corrosion/83 sponsored by the National Association of Corrosion Engineers. Anaheim, CA. (NACE, P.O. Box 218340, Houston, TX 77218).

(Apr. 19-21)—Chemical Coaters Association. "Surface Coating 83." Milwaukee, WI. (Chemical Coaters Association, Box 241, Wheaton, IL 60187).

(Apr. 20-22)—ASTM Committee C-3 on Chemical Resistant Nonmetallic Materials Meeting. Galt House, Louisville, KY. (Jim Dwyer, ASTM, 1916 Race St., Philadelphia, PA 19103).

(Apr. 24-28)—"Nuclear Quality-Assured Coatings Work" Course. Pittsburgh, PA. (Carmen A. Rivera, IAT, P.O. Box 32331, Washington, DC 20007).

(Apr. 28-29)—"Fundamentals of Color" Seminar. Don Mills, Ont., Can. (Jeanne M. Dolan, Macbeth, Div. of Kollmorgen Corp., Little Britain Rd., P.O. Box 950, Newburgh, NY 12550).

(May 8-12)—74th Annual Meeting of the American Oil Chemists' Society. Chicago Marriott Hotel, Chicago, IL. (American Oil Chemists' Society, 508 S. Sixth St., Champaign, IL 61820).

(May 9-11)—RADCURE '83 Conference sponsored by AFP/SME. Palais de Beaulieu, Switzerland. (Susan Buhr, Society of Manufacturing Engineers, One SME Drive, P.O. Box 930, Dearborn, MI 48128).

(May 10-12)—Paint Research Association's Fifth International Conference. "Technological Advances in the Coatings Industry." London Penta Hotel, London, England. (PRA, Waldegrave Rd., Teddington, Middlesex TW11 8LD, England).

(May 11-13)—23rd Annual Marine and Offshore Coatings Conference. Hyatt Regency Hotel, Baltimore, MD. (National Paint & Coatings Association, Inc., 1500 Rhode Island Ave., NW, Washington, DC 20005).

(May 12-13)—"Fundamentals of Color" Seminar. Newton Lower Falls, MA. (Jeanne M. Dolan, Macbeth, Div. of Kollmorgen Corp., Little Britain Rd., P.O. Box 950, Newburgh, NY 12550).

(May 19-20)—"Fundamentals of Color" Seminar. Cherry

Hill, NJ. (Jeanne M. Dolan, Macbeth, Div. of Kollmorgen Corp., Little Britain Rd., P.O. Box 950, Newburgh, NY 12550).

(May 23-25)—ASTM D-33 Coatings for Power Generation Facilities Committee Meeting. Galt House, Louisville, KY. (Phil Lively, ASTM, 1916 Race St., Philadelphia, PA 19103).

(May 24-26)—8th Annual Powder and Bulk Solids Conference/Exhibition. Georgia World Congress Center, Atlanta, GA. (Cahners Exposition Group, Cahners Plaza, 1350 E. Touhy Ave., Des Plaines, IL 60018).

(June 1-2)—"Color Technology Management" Course. Rochester Institute of Technology, Rochester, NY. (Val Johnson, T&E Center Seminar Coordinator, Rochester Institute of Technology, P.O. Box 9887, Rochester, NY 14623).

(June 6-10 & 13-17)—"Principles of Color Technology" Course. Rochester Institute of Technology, Rochester, NY. (Val Johnson, T&E Center Seminar Coordinator, Rochester Institute of Technology, P.O. Box 9887, Rochester, NY 14623).

(June 2-3)—"Fundamentals of Color" Seminar. Rosemont, IL. (Jeanne M. Dolan, Macbeth, Div. of Kollmorgen Corp., Little Britain Rd., P.O. Box 950, Newburgh, NY 12550).

(June 15-18)—Oil & Colour Chemists' Association Biennial Conference on "The Efficient Use of Surface Coatings." Viking Hotel, York, England. (R.H. Hamblin, Director and Secretary, OCCA, Priory House, 967 Harrow Rd., Wembley, Middlesex, HA0 2SF, England).

(June 19-22)—Dry Color Manufacturers' Association Annual Meeting. The Greenbrier, White Sulphur Springs, WV. (P.J. Lehr, DCMA, Suite 100, 117 N. 19th St., Arlington, VA 22209).

(June 20-24)—"Advances in Color Technology" Course. Rochester Institute of Technology, Rochester, NY. (Val Johnson, T&E Center Seminar Coordinator, Rochester Institute of Technology, P.O. Box 9887, Rochester, NY 14623).

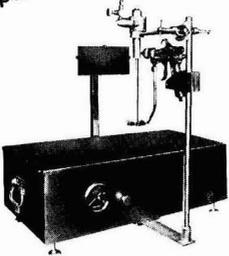
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'Kumbug' from Hillman

I am pleased to note that some occasional encouraging words of friendship from our readers have found their way to my hill top nest in Vermont. I have had helpful comments from Earl Hill, Dick Austin, Russ Horne, Peter Robinson, and Frank Borrelle. And so—I thank my small band of faithful correspondents.

Dick Austin's "Law of Junk" which says:

"The quantity accumulated is directly proportional to the space available" struck a familiar note as I packed to move to our new house.

It also appears that most of my contributors are devoted to developing their own scientific laws. I have lists of Horne's Laws for paint manufacturing, paint formulation, and falling paint cans; Baker's Laws; Murphy's Law; and several others. I will share some of these with you on occasion.

As an appetizer, here are Russ Horne's specifics of Murphy's Law regarding falling paint cans:

"A paint can always falls in such a way as to inflict the greatest damage to personnel and/or property. The subject can will make sure to fall at the precise angles to break toes or splash paint over a brand new suit which is usually being worn by an important visiting dignitary, or both."

• • •

Frank Borrelle, who among other things too horrible to mention, is our Publisher and Executive Vice-President of the Federation, engages in the intellectual pursuit of reading once in a while. Not long ago, he was impressed by the following in the *Polymers Paint Colour Journal* and sent it to me. Those who would be kind enough to explain the big words, please contact Frank directly.

What Is an Engineer?

An engineer is a person who passes as an exacting expert on the basis of being able to turn out with prolific fortitude infinite strings of incomprehensible formulae calculated with micrometric precision from vague assumptions which are based on debatable figures taken from inconclusive experiments carried out with instruments of problematical accuracy by persons of doubtful reliability and questionable mentality for the avowed purpose of annoying and confounding a hopeless chimerical group of fanatics referred to all too frequently as engineers.

• • •

I suppose that this will turn out to be our column devoted to erudition for I have at hand a contribution from Peter Robinson, President of our most erudite group, The Paint Research Institute. President Peter saved this from a yellowing April 1975 copy of *Industrial Research*.

Evaluation of creativity

"I know a systems analyst (in those days he was called a management engineer). He almost had a nervous breakdown trying to apply scientific management principles to the Manhattan Project during World War II. He tried to functionalize and standardize and specialize the work of mathematical physicists. He tried to set up standards. He tried to develop an appropriate appraisal form.

"His first difficulty, the most traumatic, was that he found it impossible to describe the work. He couldn't even produce a job description. He went into the cubicle of a theoretical mathematician and said, 'All right, what are your tasks, duties, and elements?' The fellow said 'What's that?' He went through it again. The fellow finally answered, 'Well, what I am trying to prove is that locally compact sets are not dense in themselves in Hilbert space.' The engineer asked, 'That's a duty? Show me these things.' The fellow said, 'I can't because they don't really exist. They are just abstract ideas which we invent.' The systems analyst exploded. 'You are working with something that doesn't exist? Come on now, tell me your tasks, duties, and elements.'

"Eventually the engineer went to the second page in a book, published by the U.S. Employment Service, which tells how to analyze a job. It says, 'If you don't make any sense of what the incumbent says, watch what he does.' So he began to observe.

"Well, the mathematical physicist did only three things. He drank coffee in the office, he looked at books, and he wrote on the blackboard. That's all he did. Obviously, my analyst was getting nowhere.

"He went to the man's boss, who really wasn't his boss because it turned out that the man really didn't have a boss. 'What's this fellow doing?' he asked. The boss said, "We don't know what he is doing. If we knew what he was doing, we wouldn't have him doing it.' Astonished, the analyst asked, 'Do you mean to tell me that you don't know the tasks, duties, and elements of this subordinate?' The boss responded, 'Hell, no. That's why we've got him doing it. He's the only man in the country who understands this sort of thing.'

"So the analyst went on to the next part of the interview form. 'Now tell me,' he asked the boss, 'how do you know when he is doing the job well? What are the criteria?' The boss answered, 'I haven't the slightest idea.' When the analyst persisted, he finally said, 'Well, I guess he is doing it well when he tells me so.' 'Do you mean to tell me,' shouted the analyst, 'that you depend on a subordinate to tell you when he is doing a job right?' The boss stood his ground. 'That's absolutely true. He's the only person in the country who can understand the proof of these theorems. If he says he's doing it right, he's doing it right. That's why we have him doing it.'"

—Herb Hillman

Color-matching Aptitude Test



Color-matching Aptitude Test Set was created by the Inter-Society Color Council and sponsored by the Federation. It is in world-wide use as a means for estimating color-matching skill. The 1978 edition contains minor refinements over the previous editions (1944, 1953, 1964), and these bring it closer to the original ISCC plan, making it a still more successful tool for evaluating color-matching skill.

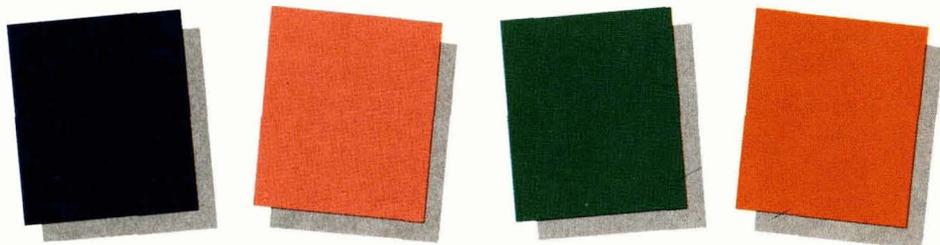
The basic aim of the Test is to provide an objective measure that will aid in determining an individual's ability for performing color-matching tasks accurately. Changes in a person's color-matching skill may occur over a period of time, improving due to training, experience, and motivation, or lessening when removed from practice, or as a result of health disorders. Retesting at regular intervals will provide an indication of any such changes in an individual's ability.

The Test is not designed to indicate or measure "color-blindness" (anomalous color vision). Special tests designed for this purpose should be used.

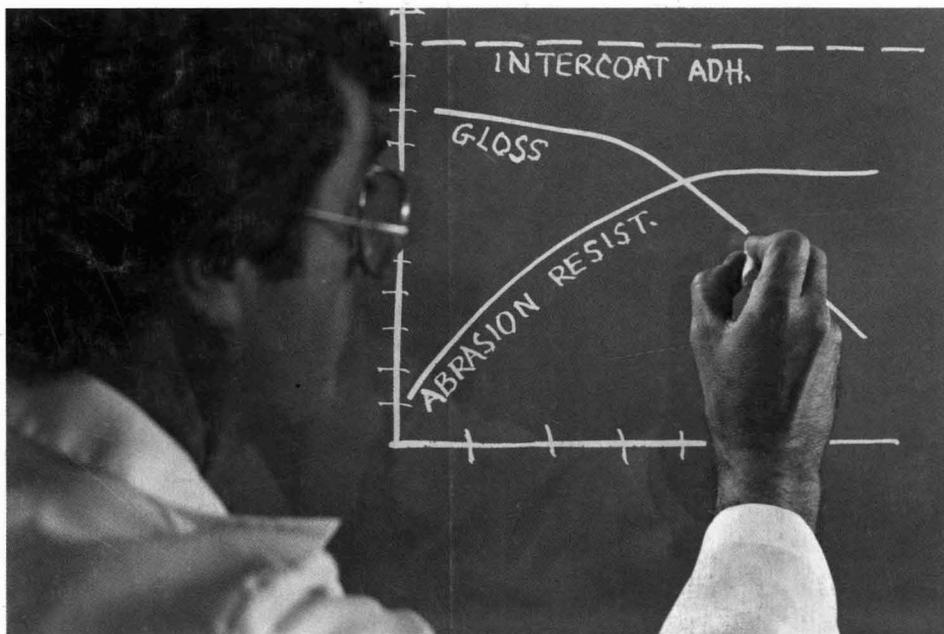
The Color-matching Aptitude Test Set consists of a carrying case, an easel on which are mounted 48 color chips, a dispenser which holds 48 matching chips, score sheets, and a scoring key.

Brochure available upon request. Price: \$400.

Orders must be prepaid. U.S. and Canada—Add \$10.00 shipping. All others—Add \$50.00 shipping.



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