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JOURNAL OF  
COATINGS  
TECHNOLOGY

JCTAX 61 (778) 1-86 (1989)

November 1989



*A Review of  
Organic Coating  
Technology for  
U.S. Naval Aircraft*







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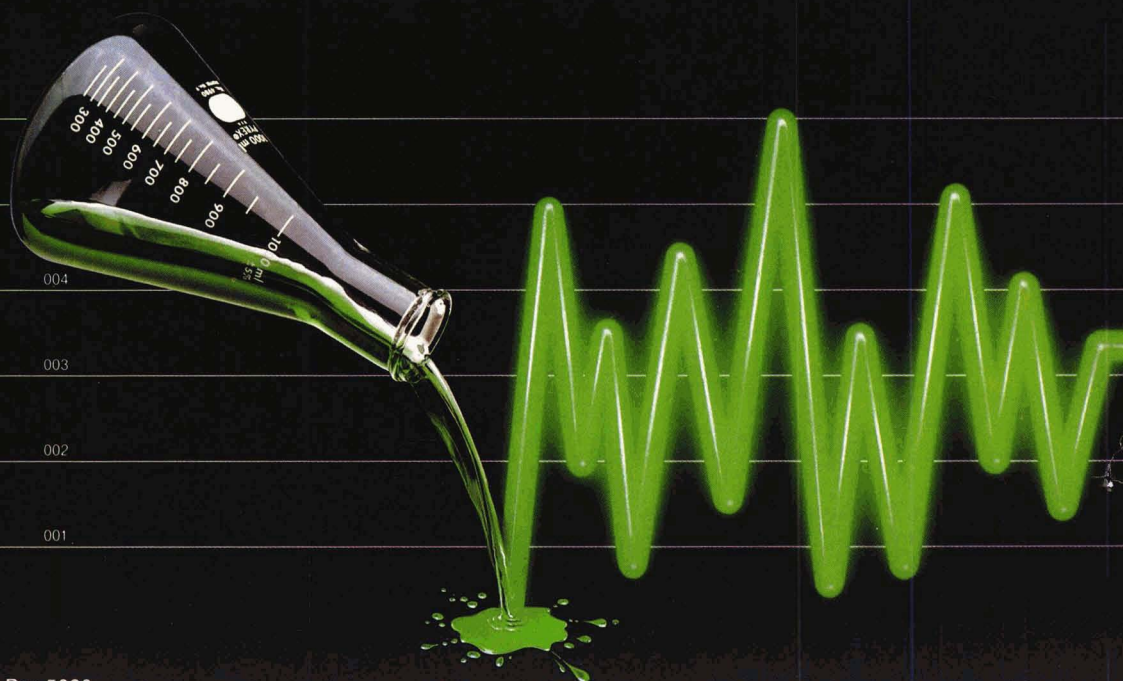
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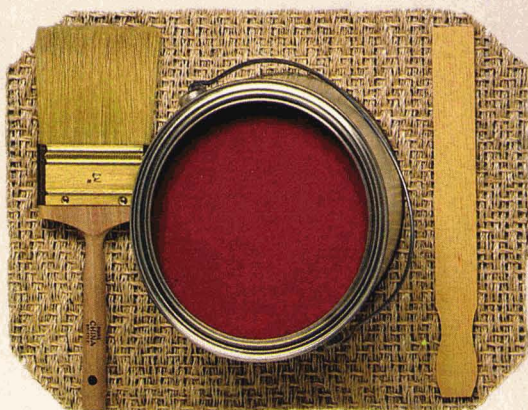
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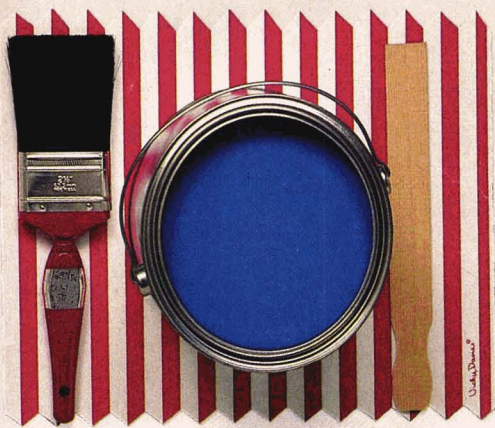
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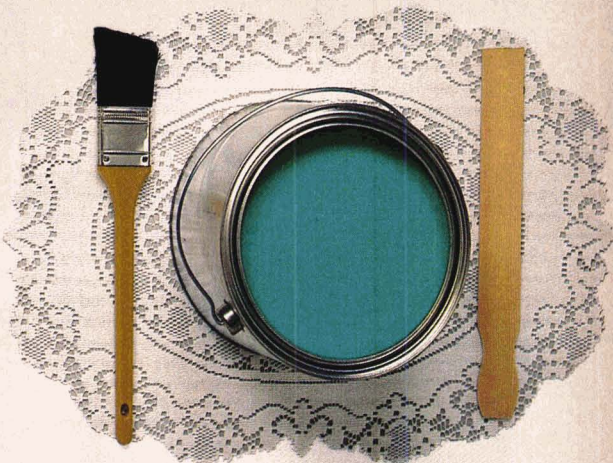
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## Something for Everyone!

If the JOURNAL OF COATINGS TECHNOLOGY has achieved recognition by the coatings industry worldwide, it is primarily as a result of the efforts of "a few good men"—and women—who serve on the Federation's Publications Committee and the JCT Editorial Review Board. Although they battle to maintain the highest standards, these people are far from literary "Dirty Harrys," brandishing lethal red pens, and daring potential authors to "make our day." Instead, these members have carefully developed guidelines which are designed to assist authors in the successful publication of their work in the JCT.

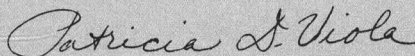
As noted on the newly revised "Guide for Authors," presented in this issue (*see pages 29-30*), several avenues are open for the expression of ideas using the forum of the JCT.

The foundation of the JOURNAL is built on *original research papers* voluntarily submitted by authors for publication. These are reviewed by selected members of the Editorial Review Board and must be prepared in accordance with the prescribed guidelines. Another avenue for authors is in the publication of *critical review articles*. Requiring more than computer literature searches, the committee judges these papers on the author's ability to provide some perspective or new insight into the data presented. (*The paper by Charles Hegedus, et al., which begins on page 31 of this issue reviews organic coatings technology for U.S. Naval aircraft and provides an example of this type of article.*)

Over the years, additions have been made to the regular features of the JCT in an attempt to provide a wider scope and appeal for the readership. The popular *Open Forum* is one such addition. These articles give a nontechnical approach to topics that impact on the coatings industry.

Finally, our most recent addition was actually offered in the JCT several years ago. Proving that "everything old is new again," the JCT re-introduces "*Back to Basics*." Once a popular feature of the JCT, these articles answer the need for practical "how to" information pertinent to coatings technology. (*On page 67, Fred Stieg addresses the topic of the effect of extender on crowding of titanium pigment, providing a tutorial on this much-debated subject.*)

Through publication of a variety of topics and formats, it is hoped that the JCT will continue to provide cohesive, informative issues that meet the needs of its readers. Of course, we appreciate any feedback on the issues and we offer another department for that—*Letters to the Editor*. We welcome your comments and look forward to hearing from you!



Patricia D. Viola  
Editor



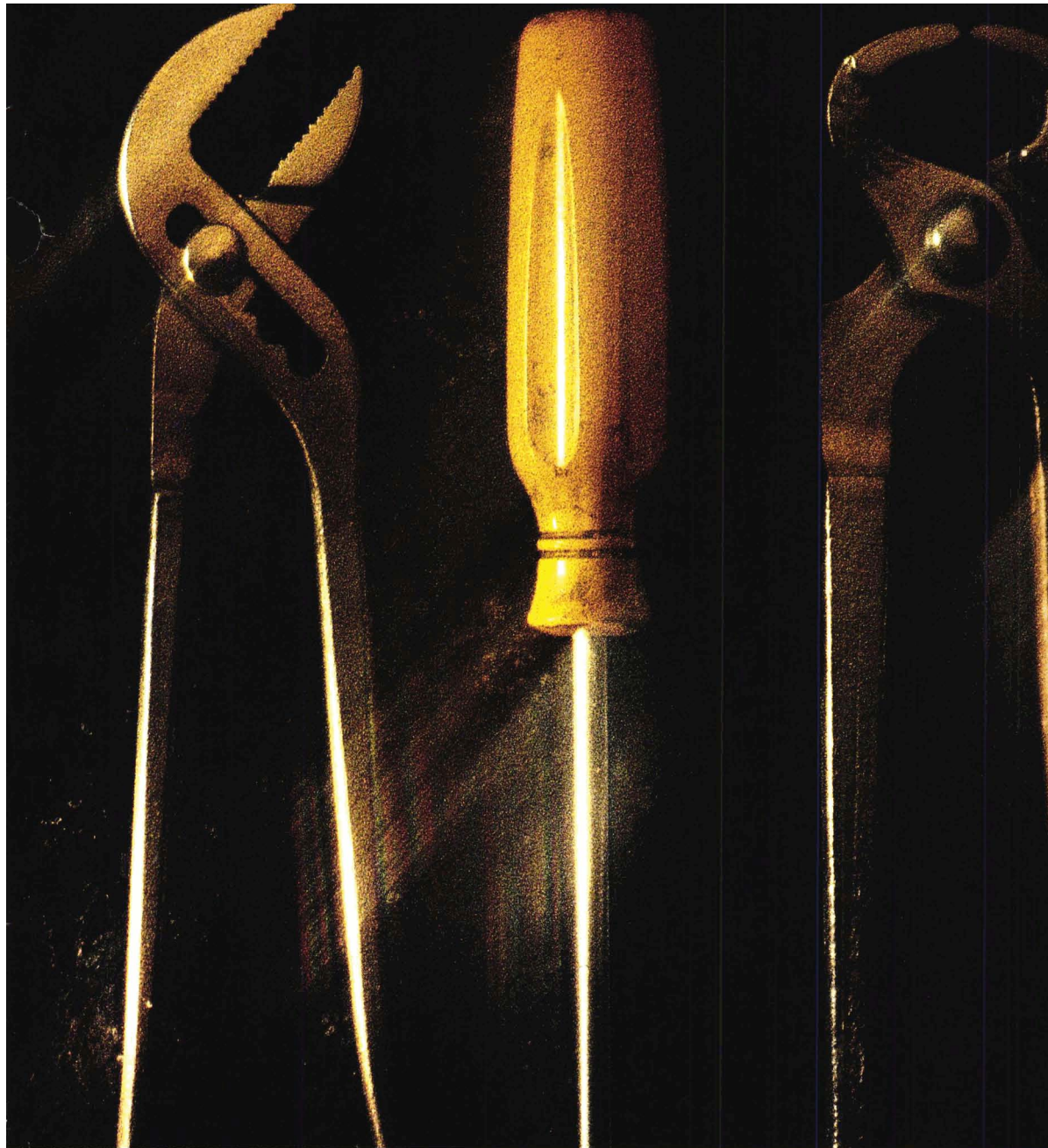
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# Abstracts of Papers in This Issue

## **A REVIEW OF ORGANIC COATING TECHNOLOGY FOR U.S. NAVAL AIRCRAFT—C.R. Hegedus, et al.**

Journal of Coatings Technology, 61, No. 778, 31 (Nov. 1989)

Navy aircraft are subject to one of the most corrosive environments known. The primary defense against this environment is the organic coating system. The chemistry and properties of standard primers, topcoats, rain erosion, and fuel tank coatings are described. Increased emphasis on reducing organic solvent emissions has caused the development of a number of compliant coatings with reduced volatile organic compound (VOC) content. Waterborne, high-solids, and exempt-solvent coatings which are replacing the standard materials are described.

The advancement of polymer and pigment chemistry has subsequently caused the development of coatings with diverse and unique properties. A self-priming topcoat has been developed to replace the current two-coat system with one coating. Flexible primers have been developed to reduce coating system cracking and chipping which is common on highly flexed areas. A high temperature organic coating (370°C) has been designed to provide corrosion and oxidation inhibition for metallic substrates. Finally, a temporary coating which was designed for quick and convenient field application is described. These coatings are currently being field tested and are expected to serve as the next generation of aircraft coating systems. A brief discussion on future trends of aerospace coatings is provided.

## **COMPONENT INTERACTIONS AND THEIR INFLUENCE ON THE UNIFORMITY OF COATING FILMS—M. Laleq, M. Bricault, and H.P. Schreiber**

Journal of Coatings Technology, 61, No. 778, 45 (Nov. 1989)

Acid/base characteristics and surface energies have been determined for polymers, blends, and pigmented systems, and patterned after coating formulations. Acid/base interactions appear to generate compositional heterogeneities due to the preferential association of interacting species. Surface heterogeneities caused by thermodynamic demands to minimize surface free energy in coating films also have been proposed. These physicochemical effects provide useful insights for product design and optimization.

## **CHEMICAL AND THERMAL STABILITY OF PHOSPHATE LAYERS ON COLD-ROLLED AND ELECTROGALVANIZED STEEL—W.J. van Ooij and A. Sabata**

Journal of Coatings Technology, 61, No. 778, 51 (Nov. 1989)

The chemical stability of modern phosphate coatings on two types of automotive steels has been studied, namely cold-rolled steel (CRS) and electrogalvanized steel (EG). Unpainted spray- and dip-phosphated panels were immersed in solutions of different pH for 30 minutes and the changes in the phosphate crystals were studied by SEM, EDX, and XRD. Some of the objectives of this study were to determine the effects of the presence of phosphophyllite or of Ni and Mn in the coatings and of a chromium post-rinse on the acid or alkali resistance of the crystals. The effect of baking on the phosphate coating and on its stability was also studied. Best results were obtained with the dip-phosphated and Mn-containing phosphate coatings. No effect of the chromate post-rinse was observed for any of the systems studied. The behavior of the phosphate at low and high pH is dissimilar and the reaction products formed are different for coatings with and without phosphophyllite. The effects of thermal treatments and subsequent exposure to water have also been studied. The behavior of the coatings on the two substrates was different.

## *Special Issue Notice* **January '90**

This special issue of the JCT will feature highlights of the 1989 Annual Meeting and Paint Show in New Orleans, November 8-10. This Wrap-up Issue will feature information on all exhibitors, with emphasis on products and special booth features; photo displays of award-winning booths; as well as a complete review of important Annual Meeting and Paint Show happenings.



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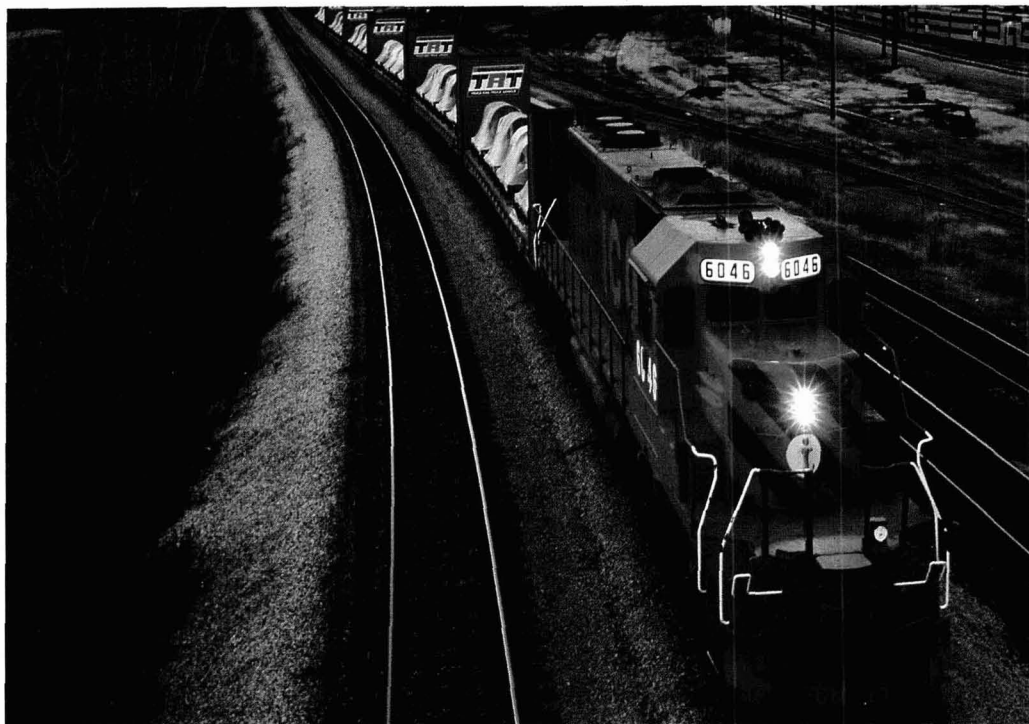
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# 1989-1990

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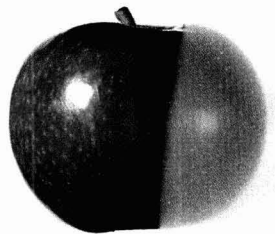
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Left: Clear emulsion-based coating formulated with liquid Tinuvin 1130 UVA

Right: Clear emulsion-based coating formulated with a standard solid UVA

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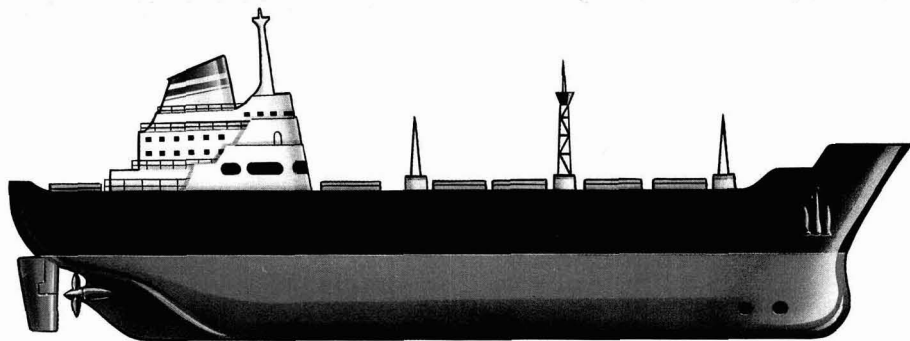
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# Regulatory UPDATE

NOVEMBER 1989

This digest of current regulatory activity pertinent to the coatings industry is published to inform readers of actions which could affect them and their firms, and is designed to provide sufficient data to enable those interested to seek additional information. Material is supplied by Roy F. Weston, Inc., Washington, D.C.

## Clean Air Act Update—

### House:

The House Energy and Commerce Health Subcommittee, chaired by Rep. Waxman (D-CA), continues to markup a modified substitute for the Administration's bill. The substitute was offered by Rep. Lent (R-NY), and full committee Chairman Rep. Dingell (D-MI). Markup to date has focused on motor vehicle requirements and alternative fuel provisions. Rep. Waxman's intent to amend the Administration bill with more stringent provisions has not met with total success, and this is expected to continue as the subcommittee starts to address air toxics and nonattainment provisions. Amendments are expected to be offered for the air toxics provisions, including one that would specify the maximum level of cancer risk to be tolerated from industrial plants. Rep. Waxman is expected to propose stricter amendments to the Administration's bill provision that would require maximum available control technology (MACT) on major industrial sources of air toxics. The Administration bill would require determining whether those standards are sufficient or to issue more stringent health-based standards within seven years after a MACT standard is issued. Rep. Waxman's amendment would propose that health standards limit the lifetime cancer risk of the most exposed individual living near a plant to one in a million.

Other toxics amendments expected to be proposed include setting a floor in determining MACT for controlling emissions from plants that are major sources of toxic pollutants, and requiring all industry categories that emit a toxic pollutant to be regulated under technology-based standards.

The substitute offered by Rep. Dingell and Rep. Lent, lead sponsors of the Administration's bill (HR 3030), is intended to expedite markup through the subcommittee and changes many discretionary provisions in the Administration bill to mandatory provisions. The substitute bill does not make any changes in the acid rain or alternative fuels provisions.

### Senate:

Sen. Baucus, (D-MT) introduced a nonattainment bill (S1630). The bill would impose new requirements on mobile and stationary sources in areas that have not attained the national ambient air quality standards for ozone, carbon monoxide, and particulate matter. Provisions include:

- Classification of areas into categories depending on severity of nonattainment: moderate, serious, severe, and ex-

treme. Serious areas would have to implement VOC emission offsets for new sources, as would areas classified as severe or extreme.

- All requirements in existing state implementation plans not yet instituted would have to be implemented within 18 months of enactment.

An air toxics bill (S816) was introduced in April 1989, an acid rain bill (S57) was introduced in January 1989. The Administration's bill (S1490) was introduced in August 1989. It is expected that these bills will be combined into a comprehensive package for markup by the Senate Environment and Public Works Subcommittee on Environmental Protection, chaired by Sen. Baucus.

**EPA Lists Methyl Bromide Production Wastes as RCRA Hazardous Substances**—EPA has promulgated a final rule listing as hazardous two wastes generated during production of methyl bromide. EPA is also listing these wastes under Superfund and establishing reportable quantities applicable to these wastes. See 54 Federal Register 41402, October 6, 1989.

The specific wastes are the wastewater from the reactor and acid dryer from the production of methyl bromide (EPA Hazardous Waste No. K131) and spent adsorbent and wastewater separator solids from the production of methyl bromide (EPA Hazardous Waste No. K132). These wastes were proposed by EPA as hazardous in April 1985 (see 50 Federal Register 16432). Methyl bromide and dimethyl sulfate are the hazardous constituents of concern in these wastes.

EPA has listed these wastes as hazardous because of the constituent's moderate solubilities in water and high solubilities in organic solvents and are therefore expected to be mobile in the environment. EPA has received several comments in response to the proposed rule and has decided that the comments do not refute EPA's reasons for listing the wastes as hazardous.

The effective date of this final rule is April 6, 1990. For further information, contact the RCRA/Superfund Hotline at (800) 424-9346 or at (202) 382-3000 in Washington, D.C. and Alaska. Also contact Cate Jenkins, Office of Solid Waste, (OS-332), U.S. EPA, 401 M Street, S.W., Washington, D.C. 20460, (202) 382-4786. For information on the Superfund technical rule, contact Ivette Vega, Response Standards Division, (OS-210), U.S. EPA, 401 M Street, S.W., Washington, D.C. 20460, (202) 382-2463.

The Regulatory Update is made available as a service to FSCT members, to assist them in making independent inquiries about matters of particular interest to them. Although all reasonable steps have been taken to ensure the reliability of the Regulatory Update, the FSCT cannot guarantee its completeness or accuracy.

**Packaging Materials for Use Under High Temperature Conditions in Microwave Ovens**—The Food and Drug Administration (FDA) is considering publishing a proposal that would amend the indirect food additives regulations by establishing maximum temperatures of use for certain food-packaging materials. Should FDA choose to amend the indirect food additives regulations, the maximum temperatures of use for the materials in the proposal would be based in part upon use and safety information that is submitted in response to this Advance Notice of Proposed Rulemaking (ANPR). See 54 Federal Register 37340 (September 8, 1989).

The types of data that FDA considers necessary for its review of whether to establish temperature limits are:

- Information on the chemical composition and construction of heat susceptor packaging.
- Information on the temperature levels reached by various components of the package under actual conditions of use in microwave ovens.
- Migration data obtained under maximum time/temperature conditions of use.
- Identity of breakdown products under maximum time/temperature conditions of use.
- Available toxicological safety data for component chemicals and breakdown products.
- Current and anticipated use information (e.g., types of food contacted, cooking/heating times, and temperatures).

These data should be submitted to the Division of Food and Color Additives, (HFF-335), Food and Drug Administration, 200 C Street, S.W., Washington, D.C. 20204, as a letter, as a food additive master file, or as part of a food additive petition.

These data should be submitted to FDA no later than December 7, 1989.

Comments on the proposed ANPR in general were due by November 7, 1989, to Dockets Management Branch, (HFA-305), Food and Drug Administration, Room 4-62, 5600 Fishers Lane, Rockville, MD 20857.

For further information, contact Edward Machuga, Center for Food Safety and Applied Nutrition, (HFF-335), Food and Drug Administration, 200 C Street, S.W., Washington, D.C. 20204, (202) 472-5690.

**EPA Seeks Data on Methylene Chloride Substitute**—EPA's Office of Toxic Substances is seeking published or unpublished data on a potential substitute for methylene chloride, N-methylpyrrolidene (NMP), CAS No. 872-50-4.

NMP is currently under review by the Consumer Product Safety Commission (CPSC) because it is used as a substitute for methylene chloride in paint strippers. EPA is supporting CPSC in its review of health effects data, and will develop a proposed test rule under Section 4 of the Toxic Substances Control Act (TSCA). Section 4 gives EPA authority to require manufacturers or processors of chemicals to test the toxic effects of a designated substance.

EPA is also interested in any current testing of NMP, or ongoing assessment activities of other organizations. All information submitted in response will be placed in a public file and made available for public inspection, unless a submitter claims confidentiality under Section 14 of TSCA.

For more information, contact Robert Jones, OTS Test Rules Development Branch, Offices of Pesticides and Toxic Substances, U.S. EPA, 401 M Street, S.W., Washington, D.C. 20460, (202) 475-8150. All information should be sent to Lynn Marcus, TSCA Public Docket Officer, (TS-793), Office of Pesticides and Toxic Substances, U.S. EPA, Room NE-6004, 401 M Street, S.W., Washington, D.C. 20460.

**Methyl Ethyl Ketoxime Final Test Rule**—EPA is issuing a final test rule requiring manufacturers and processors of methyl ethyl ketoxime (MEKO), CAS No. 96-29-7, to perform testing for health effects. This final rule is part of the overall implementation of Section 4 of the Toxic Substances Control Act (TSCA). See 54 Federal Register 37799 (September 13, 1989).

All required tests must be conducted in accordance with EPA's TSCA Good Laboratory Practice Standards in 40 CFR Part 792, and guidelines set forth in 40 CFR Part 798.

The rule became effective on October 27, 1989.

For further information, contact Michael Stahl, Director, Environmental Assistance Division, (TS-799), Office of Toxic Substances, Room EB-44, U.S. EPA, 401 M Street, S.W., Washington, D.C. 20460, (202) 554-1404.

**Decision Guide for Identifying Substance-Specific Data Needs for Toxicological Profiles**—The Agency for Toxic Substances and Disease Registry (ATSDR) has issued for public comment a Decision Guide for use in identifying the substance-specific information needed to achieve the mission of preventing or mitigating adverse human health effects that result from hazardous substances in the environment. Section 104(i)(2) of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) as amended by the 1986 Superfund Amendments and Reauthorization Act (SARA) requires the ATSDR to:

- (1) prepare a list of hazardous substances found at NPL sites in order of priority
- (2) develop toxicological profiles of those substances
- (3) design and implement a research program to fill data gaps associated with the substances.

The priority list has been prepared (see 52 Federal Register 12866 and 53 Federal Register 41280). The first 10 final toxicological profiles have been issued (see 54 Federal Register 14037 and 54 Federal Register 26417). The Decision Guide is a first step in preparing a research program. See 54 Federal Register 37618 (September 11, 1989).

In developing and implementing a research program, ATSDR is required to coordinate with EPA, the National Toxicology Program (NTP), and the Interagency Testing Committee (ITS). The costs of research programs conducted by ATSDR are to be borne by the manufacturers and processors of the hazardous substances in question.

Comments on the Decision Guide were due on October 26, 1989 to Edward Skowronski, Research Analysis Branch, Agency for Toxic Substances and Disease Registry, Docket ATSDR-11, Mailstop E-29, 1600 Clifton Road, N.E., Atlanta, GA 30333, (404) 639-0730.

**FDA Removes D&C Red Dye No. 36 from Provisional Listing**—The Food and Drug Administration (FDA) has removed D&C Red Dye No. 36 from regulations that provide for provisional listing of color additives. This action is to correct an error that included D&C Red Dye No. 36 in regulations after the confirmed effective date for the permanent listing of D&C Red Dye No. 36 for general use in drugs and cosmetics, except for use in the area of the eye. See 54 Federal Register 37307 (September 8, 1989).

The effective date of this action was September 8, 1989. For more information, contact Gerald L. McCowin, Center for Food Safety and Applied Nutrition, (HFF-33D), Food and Drug Administration, 200 C Street, S.W., Washington, D.C. 20204, (202) 472-5676.



**Final Report Published on Tribromomethane**—The National Toxicology Program (NTP) has published a final toxicology and carcinogenicity report on tribromomethane, a chemical intermediate and solvent. See 54 Federal Register 40748 (October 3, 1989).

Tribromomethane has been identified as a drinking water contaminant from water chlorination. There was some evidence of carcinogenicity for male rats and clear evidence of carcinogenicity for female rats.

For more information, contact Ronald L. Melnick at NTP, P.O. Box 12233, Research Triangle Park, NC 27709, (919) 541-4142. For free copies of the report, "Toxicology and Carcinogenesis Studies of Tribromomethane (Bromoform) in F344/N Rats and B6C3F<sub>1</sub> Mice (Gavage Studies) (TR 350)," contact the NTP Public Information Office, MD B2-04, P.O. Box 12233, Research Triangle Park, NC 27709, (919) 541-3991.

**Response Claims Procedures for the Hazardous Substance Superfund**—On September 13, 1989, EPA proposed regulations to establish procedures for filing, evaluating, and resolving claims for costs incurred for responding to releases of hazardous substances, pollutants, or contaminants asserted against the Hazardous Substance Superfund established under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) and the Reauthorization Act of 1986 (SARA). The regulation also established procedures for notifying concerned parties regarding limitations on the payment of response claims. The claims process has two parts: the preauthorization process and the claims award process.

The proposed regulation is to be used by an individual, private entity, potentially responsible party, or foreign entity eligible to submit a claim pursuant to Section 111(a)(2) or 122(b)(1) of CERCLA.

Comments on the proposed rule are due on November 13, 1989.

Written comments should be sent in triplicate to the Superfund Docket, Room 2427, U.S. EPA, 401 M Street, S.W., Washington, D.C. 20460.

For further information, contact William Ross, Office of Emergency and Remedial Response, (WH-220), U.S. EPA, 401 M Street, S.W., Washington, D.C. 20460, (202) 382-4645, or the RCRA/CERCLA Hotline, (800) 424-9346. See 54 Federal Register 37892 (September 13, 1989).

**National Emission Standards for Hazardous Air Pollutants: Benzene**—In response to a petition filed by the National Resources Defense Council, et al., the D.C. Circuit Court ordered EPA to either propose standards for benzene emissions or publish a notice of intent not to regulate. EPA chose to establish standards limiting benzene emissions from the following source categories: industrial solvent use, benzene waste operations, benzene transfer operations, and the gasoline marketing system. EPA also proposes **not** to regulate the chemical manufacturing process vent source category. See 54 Federal Register 38083 (September 14, 1989).

The chemical manufacturing process vent source category includes three aniline, two benzene sulfonic acids, three chlorobenzene, one cumene, 32 ethylene, one hydroquinone, four linear alkylbenzene, four nitrobenzene, and four phenol units that emit benzene through process vents. EPA has also considered cyclohexane facilities, resorcinol and pyridine manufacturing processes but EPA found these processes were not emitting benzene through process vents. The pro-

posed rule category does not include ethylbenzene and styrene (EB/S) process vents, previously examined under the EB/S source category or equipment leak emissions of benzene from chemical plant process units regulated under 40 CFR Part 61, Subpart J.

Both the proposed standard and the proposal not to regulate chemical manufacturing process vent sources are being developed under Section 112 of the Clean Air Act.

EPA proposed to hold a public hearing on these matters on October 11, 1989, if so requested. No such request was made and no hearing was held. However, comments on the proposal rule are due by November 13, 1989. They should be sent in duplicate to: Air Docket Section, (LE-131), Attention: Docket OAQPS 79-3 Part I, for health effects, listing and regulation of benzene; Docket A-89-03, for benzene emissions from process vents at chemical plants; Docket A-89-04, for benzene transfer operations; Docket A-89-05, for benzene solvent use; Docket A-89-06, for benzene waste operations; U.S. EPA, 401 M Street, S.W., Washington, D.C. 20460.

For further information, contact Doug Bell or Janet Meyer at (919) 541-5568 and (919) 541-5254, respectively, for inquiries on benzene emissions and regulations; Scott Voorhees at (919) 541-5348 for date on the health effects of benzene and the risk assessment.

**EPA Allows Public Hearing on 1986 Vinyl Chloride Provisions**—EPA is proposing to grant a request in a petition by the Society of Plastics Industry to clarify certain provisions of EPA's 1986 final rule revising the national emission standard for vinyl chloride. Petitioners' requests for changes in regard to area monitoring requirements, relief value discharge standard, and request for stay of the 1986 revised provisions were denied. See 54 Federal Register 38938 (September 21, 1989).

EPA held a public hearing on October 18, 1989 on these proposed clarifications and allowed presentation of arguments concerning the proposed revisions. Comments are due November 20, 1989 in duplicate to Central Docket Section, (LE-131), Attention: Docket No. A-81-21, U.S. EPA, 401 M Street, S.W., Washington, D.C. 20460. For more information, contact Shirley Tabler, Standards Development Branch, Emission Standards Division, (MD-13), U.S. EPA, Research Triangle Park, NC 27711, (919) 541-5256.

**Establishment and Open Meeting of the Negotiated Rulemaking Advisory Committee for the VOC Equipment Leak Rule**—EPA has established an Advisory Committee to negotiate a rule to control emissions of toxic volatile organic compounds (VOCs) from chemical equipment leaks. The Committee's first meeting was held on September 27, 1989, and addressed outstanding procedure matters, approaches for resolving substantive issues, and the initiation of issue resolution. The current objectives of the proposed regulatory negotiation are: revise emission factors for fugitive VOC emissions from equipment leaks to better quantify the leaks; develop a new control standard for VOC emissions based on best technology and lowest achievable rates; and develop a format that could be used to implement a risk based standard for VOC emissions from equipment leaks. See 54 Federal Register 37725 (September 12, 1989).

For further information on substantive matters, contact Robert Ajax, Office of Air Quality Planning and Standards, U.S. EPA at (919) 541-5597, or on procedural matters, Deborah Dalton, Regulatory Negotiation Project, U.S. EPA at (202) 382-5495.

**Standards of Performance for New Stationary Sources: Polymeric Coating of Supporting Substances**—Effective September 11, 1989, EPA promulgated standards of performance for new, modified, and reconstructed facilities that perform polymeric coating of supporting substrates. These standards were first proposed in the Federal Register of April 30, 1987 (see 52 Federal Register 15906), and implement Section 111 of the Clean Air Act. The intended effect of these standards is to control volatile organic compounds (VOCs) emissions from each new, modified, or reconstructed coating operation and the associated coating mix preparation equipment used to prepare the coating. Only on-site mix equipment is considered part of an affected facility. "Polymeric coating of supporting substrates" is defined by EPA as a web-coating that applies an elastomer or other polymeric material onto a supporting substrate such as: woven, knit, and nonwoven textiles, fiberglass, yard, and cord. Coatings include natural and synthetic rubber, urethane, polyvinyl chloride, acrylic, epoxy, silicone, phenolic, and nitrocellulose. See 54 Federal Register 37534 (September 11, 1989).

With these standards, EPA seeks to reduce VOC emissions to the level achievable by the best demonstrated technology (BDT) of continuous emission reduction, considering costs, nonair quality health, environmental impacts, and energy requirements. Under the new standards, an operation with projected VOC use of less than 95 Mg/yr (megagrams/year) would be required to monitor VOC use. An operation with projected VOC use of 95 Mg/yr or greater would be required to reduce VOC emissions by 90% using an emission capture system and control device (emission reduction standard), or install approved total enclosure and vent emissions to a 95% efficient control device (alternative standard).

The standards apply to affected facilities for which construction, reconstruction, or modification began after April 30, 1987.

For further information regarding policy issues, contact Doug Bell or Laura Butler at (919) 541-5568 and (919) 541-5267, respectively. For responses to technical questions, call

James Berry at (919) 541-5606, or Robert Blaszcak at (919) 541-5408. Emissions Standards Division, (MD-13), Research Triangle Park, NC 27711.

**Hazardous Waste Management System: Testing and Monitoring Activities**—EPA has issued a final rule (40 CFR Parts 260 and 261) which adopts 47 testing methods for use in meeting Subtitle C of the Resource Conservation and Recovery Act (RCRA). The methods contained in the rule are already known to and used by the regulated community, and are found in the third edition of "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," Office of Solid Waste Publication SW-846, and in Update 1 of that edition.

The effective date of this rule was October 30, 1989.

For general information, contact the RCRA Hotline at (800) 424-9346. For technical information, contact Charles Sellers, Office of Solid Waste, OS-331, U.S. EPA, 401 M Street, S.W., Washington, D.C. 20460, (202) 382-3282. See 54 Federal Register 188 (September 29, 1989, p. 40260).

**House Begins Markup Waste Reduction Bill**—House Energy and Commerce Subcommittee for hazardous materials began markup in October on a waste reduction bill previously introduced by Rep. Wolpe (D-MI).

HR1457 would:

- require additional submission information from companies on efforts to reduce waste chemicals to information on toxic chemical releases already required to be submitted to EPA and state officials under the Community Right-to-Know Act.

- establish an office within EPA to develop and implement a strategy to reduce the production of hazardous wastes, and this office would review and advise other EPA program offices on promoting an integrated approach to reducing production of hazardous wastes.

Both the House and Senate passed similar measures last year, but could not resolve differences before adjourning.

## SUMMARY CALENDAR OF REGULATORY ACTIONS

September 11, 1989	New performance standards for polymeric coating of substrates effective. (See this issue.)
September 22, 1989	FDA regulations for safe use of substance for water repellent in paper and paperboard effective. Comments were due October 23. (See this issue.)
September 26, 1989	FDA regulations for safe use of adhesive and polymers antioxidant effective. (See this issue.)
October 26, 1989	Comments were due on ATDSR Decision Guide. (See this issue.)
October 27, 1989	Final test rule effective for methyl ethyl ketoxime. (See this issue.)
October 30, 1989	New EPA test methods for RCRA effective. (See this issue.)
November 13, 1989	EPA rule on delay of closure for hazardous waste management facilities. (See this issue.)
November 13, 1989	Comments due on EPA's response claims procedures for Superfund. (See this issue.)
November 13, 1989	Comments due on EPA's proposed standards for benzene emissions. (See this issue.)
November 20, 1989	Comments due on EPA's 1986 vinyl chloride provisions and clarifications. (See this issue.)
December 7, 1989	Data due to support FDA consideration of packaging materials for use under high temperature conditions in microwave ovens. Comments were due November 7. (See this issue.)
January 1, 1990	New Jersey limits on VOC content on architectural coatings effective. (See May 1989 issue.)
June 13, 1990	OSHA extends administrative stay on formaldehyde occupational exposure. (See October 1989 issue.)



## Union Carbide Corp. Becomes a Holding Company; Joint Venture to Market Toner Resins Formed

Union Carbide Corporation, Danbury, CT, has become a holding company and its domestic chemicals and plastics business became a separate wholly owned subsidiary named Union Carbide Chemicals and Plastics Company Inc.

The holding company has assumed the Union Carbide Corporation name.

In addition, each outstanding share of the corporation's common stock became one share of the holding company's common stock, and the corporation's stockholders automatically became stockholders of the holding company.

The chemicals and plastics subsidiary joins two other core business subsidiaries—Union Carbide Industrial Gases Inc. and UCAR Carbon Company Inc.

Officers of Union Carbide have all been appointed officers of the holding company. The elected officers of the holding company are: R.D. Kennedy, Chairman, Presi-

dent, and Chief Executive Officer; R.P. Krass, Vice President and President—UCAR Carbon Company Inc.; H.W. Lichtenberger, Vice President and President—Union Carbide Chemicals and Plastics Company Inc.; J.R. MacLean, Vice President and President—Union Carbide Industrial Gases Inc.; J.E. Geoghan, Vice President and General Counsel; J.A. Clerico, Vice President, Treasurer, Principal Financial Officer; G.E. Playford, Vice President—Strategic Planning; J.B. Powers, Vice President; O.J. Romary, Vice President and Secretary; C.L. Smith, Vice President—Health, Safety, and Environmental Affairs; R.V. Welty, Vice President—Human Resources; R.S. Wishart, Vice President—Public Affairs; and J.K.

Wulff, Vice President, Controller, and Principal Accounting Officer.

In other news, Union Carbide Chemicals and Plastics Company Inc. and Sekisui Chemical Co., Ltd., Japan, have established a joint venture to manufacture resins for toner markets in North and South America. The joint venture will be called Hexatec Polymers.

Hexatec will supply custom styrene-acrylic resins for toners used in copying machines and laser printers.

To supply the toner market, the joint venture will initially import resins from Japan until manufacturing facilities are developed for production in the U.S. The venture will use Sekisui's technology and both companies' marketing capabilities.

### Witco Buys Union Camp's Tallow Fatty Acids Business

Witco Corporation, New York, NY, has purchased the tallow fatty acids business from Union Camp Corporation, Wayne, NJ.

The purchase, for an undisclosed amount of cash, includes among other things, formulations and manufacturing technology related to the Union Camp tallow fatty acids business. Products will be supplied from Witco's Humko Chemical Division plants in Newark, NJ, and Memphis, TN.

In other news, Witco has licensed carbon black technology to a company in Indonesia for use in building a plant to supply its tire industry.

The new plant is scheduled to begin operations early in 1990. Its design incorporates technology supplied by Witco's Concarb Division and will include a computerized process control system.

The facility will have an initial annual capacity of 30,000 metric tons of tread carcass grades of carbon black.

Witco manufactures specialty chemical and petroleum products and engineered materials.

### Morton International Opens New State-of-the-Art Automotive Tech Center in Rochester Hills, MI

Morton International, Inc., Chicago, IL, has opened its state-of-the-art Automotive Technical Center, in Rochester Hills, MI, a northern suburb of Detroit.

The 64,000-sq ft facility rests on 6.5 acres approximately 25 miles north of Detroit. Both the Specialty Chemicals Group and Automotive Safety Products are represented at the Technical Center.

Five of Morton's seven automotive business units are housed at the location, including: Automotive and Industrial Finishes (Bee Chemical Company and Injecta Color liquid colorants), Powder Coatings,

Adhesives and Coatings (industrial and windshield adhesives), and Inflatable Restraint Systems (airbags).

The Center's high-tech equipment includes: an automotive paint application trial line, capable of painting automotive body panels under simulated production line conditions; a 400-ton injection molding machine; a spectrophotometer to duplicate OEM masters; and a spraymation machine which minimizes operator variance.

Morton International is a manufacturer and marketer of specialty chemicals.

### Specialty Solution Facility Planned for Beaumont, TX

The Goodyear Tire and Rubber Company, Akron, OH, is investing \$64 million for both the construction of a new specialty solution polymer facility in Beaumont, TX, and an expansion of its current synthetic rubber facilities at Beaumont.

The new specialty solution polymer facility will have a capacity of more than 50 million pounds per year and the expansion of the existing facility will increase the current capacity by an additional 30 million pounds.

The specialty polymer facility will create more than 50 permanent jobs in the plant and 350 construction jobs in the Beaumont area.

The new special polymer plant will have multicatalyst and multisolvent system capability.

Expected completion of the expansion is January 1992.

## Construction Contracts Rise 2% in July, Reversing Two-Month Decline; Public Works/Utilities Projects Key to New Building Increase

Contracting for new construction reversed a two-month decline by edging up 2% in July according to the F.W. Dodge Group, McGraw-Hill Information Services Company, New York, NY.

A rebound of public works/utilities projects was largely responsible for lifting July's annualized total of newly started construction to \$256.6 billion. This was the highest monthly annualized value reported since April.

In July, the Dodge Index, which relates current construction contracting to a 100 base in 1982, advanced to 163 from June's 160.

"Nonbuilding" construction contracts, for public works and utilities, advanced 5% in July to an annualized rate of \$45.7 billion. Despite July's pickup, public works construction remains the weakest of the major categories of contracting through the first seven months of 1989.

July contracts for nonresidential buildings totaled \$91.8 billion. The total reflects a modest 2% advance over June's value.

### Akzo Acquires Reliance From Tyler Corporation

Akzo NV, the Dutch-based international chemical company, has acquired Reliance Universal, Inc., from Tyler Corporation, Dallas, TX. The transaction followed approval by Tyler shareholders.

Gary L. Fulk, former President and Chief Executive Officer of Reliance, headquartered in Louisville, KY, has been named President and Chief Executive Officer of the combined businesses in North America. He will report to Cees Zaaij in The Netherlands, who heads the worldwide Akzo Coatings Division.

Reliance is a producer of industrial coatings for woods, metals, and plastics.

### Stauffer Chemical Renamed Rhone-Poulenc Basic Chem.

The Stauffer Chemical Company, Shelton, CT, a division of Rhone-Poulenc Inc., has been renamed Rhone-Poulenc Basic Chemicals Company. As a result of this change, the name of Stauffer Chemical Company of Wyoming has been changed to Rhone-Poulenc of Wyoming Company.

The name changes were effective immediately.

The basic chemicals businesses of the former Stauffer Chemical, consisting of sulfuric acid and sulfur-based products, phosphorous products, soda ash, and waste recycling and recovery services, were acquired by Rhone-Poulenc in December 1987.

Office construction dominated July's nonresidential building activity. Three large new projects—in Los Angeles, CA, Chicago, IL, and Jersey City, NJ—alone will add another 4½-million sq ft of space to the office market.

Apart from July's surprising 24% increase in office construction, the month revealed a sharp gain in contracting for health care facilities, as well as double-digit declines for stores/shopping centers and factories.

July's value of newly-started residential buildings increased 1% to an annualized rate of \$199.1 billion. Single family home building advanced 2% while contracting

for multi-family housing and hotels declined slightly.

Through the first seven months of 1989, the unadjusted total of all newly-started construction was \$149.8 billion, 1% less than in the same period of 1988. The value of both nonresidential building and housing was unchanged from their totals of one-year ago, but nonbuilding construction was 7% below the seven month total for 1988.

By region, the North Central held a 2% lead over last year through seven months. The Northeast and West both showed a 1% deficit, while contracting in the South trailed last year's cumulative total by 4%.

### NDPA Board Approves Automation Institute And Funds for Hazmat Training Program

The Board of Directors of the National Decorating Products Association (NDPA), St. Louis, MO, has approved an institute to promote development of computer software and education of the retailer, plus funds for a hazardous materials training program.

The board approved establishment of a Decorating Products Retail Automation Institute. The institute will serve as a liaison to computer software and hardware firms to promote development of automation-related programs and seminars for the decorating products retailer.

The institute will operate as a subsidiary of NDPA with an affiliate to related associations, Window Coverings Association of America, and National Association of Floor Covering Retailers.

The institute will be funded by contributions from sponsoring manufacturers based upon their sales to decorating products retailers.

Also, the NDPA board appropriated \$30,000 to launch a hazardous materials training program. The program will include a two-tape video training program

covering employee's right-to-know about hazardous materials and how to handle those materials. The training is designed to help retailers meet OSHA requirements.

### Sherwin-Williams to Acquire Whittaker's Coating Group

The Sherwin-Williams Company has reached an agreement in principle to acquire the Chemical Coating Group of The Whittaker Corporation. Terms have not been disclosed.

The company said the transaction is expected to be completed this fall.

The Whittaker Coatings Group, which produces industrial coatings, is comprised of eight operating divisions, 10 manufacturing facilities in nine states, and two research laboratories.

The Chemical Coating Group sale is part of The Whittaker Corporation's divestiture plan.

### Kerr-McGee to Expand Sodium Chlorate Production

Plans to expand its sodium chlorate production in the southeastern U.S. by 60,000 tons have been announced by the Kerr-McGee Chemical Corporation, Oklahoma City, OK. The expansion was announced following completion of preliminary engineering studies.

The increased production is expected to be on-stream by mid-1991, bringing Kerr-McGee's total sodium chlorate capacity to 123,000 tons per year.

Further studies are underway to determine whether the expansion will take place at the company's current facility in Hamilton, MS, or at an unnamed location in the Southeast.

### Coatings Business Unit Formed at Angus Chemical

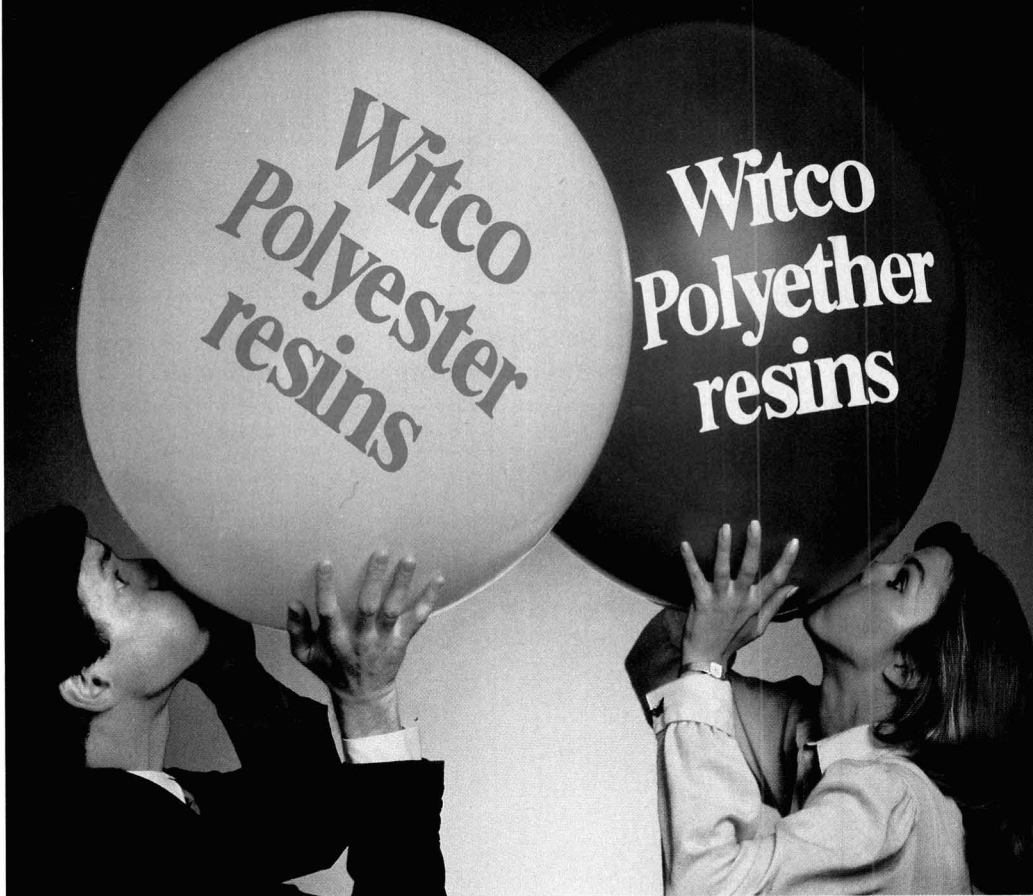
A new coatings business unit has been formed at Angus Chemical Company, Northbrook, IL. The unit will support the coatings industry with a line of performance products, technical capabilities, and formulation assistance.

One important objective of the unit will be to support AMP-95™ in the latex paint and pigment dispersant market.

Lowell D. Pals has been appointed Vice President and General Manager of the new coatings unit.



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## Ultrasonic Paint Measurement Technique Developed by Southwest Research Institute

A nondestructive testing (NDT) measurement technique that can be used to check the thickness of paint layers applied to new cars has been developed at the Southwest Research Institute (SwRI), San Antonio, TX.

Dr. Glenn M. Light, Manager—Nondestructive Evaluation Assessment Section at SwRI, Dr. G.P. Singh, of KARTA Technology (a SwRI employee at the time of development), and Dr. F.D. McDaniel, of North Texas State University, are co-developers of the method.

The ultrasonic measurement technique recently has generated interest as an improved way to spot check paint application uniformity on new autos.

According to Dr. Light, high-frequency ultrasonics can be used to measure layer thicknesses above 0.001 inch. The three layers of paint typically applied to a car,

the primer, colorant, and clear topcoat, each fall within the range of 0.001-0.005 in.

The technique can determine individual layer thicknesses almost instantaneously after the ultrasonic velocity in the sample material has been determined. Equipment consists of a transducer with pulser/receiver and amplifier electronics. By subjecting the sample to a coupling mechanism, such as water and measuring the time change amplitudes as ultrasound waves move through the water, the thickness of the sample can be determined.

A possible future application for ultrasonics is the detection of clusters of tiny defects in ceramic engine blocks during the green state, prior to firing, at which point it might be possible to machine the defects out of the engine to ensure its serviceability, stated Dr. Light.

## Skeist Releases Study on Water-Soluble Polymers; Report Includes Industry Projections For 1993

A new multi-client techno-economic marketing study, "Water-Soluble Polymers, IV," has been released by Skeist Inc., Whippany, NJ, consultants to the polymer industries.

The study is an in-depth examination of the utilization of these polymers in five businesses—superabsorbents, water treatment, petroleum recovery, papermaking aids, and mineral processing. Of these, water treatment shows the brightest future for the next five years. During the past few years, superabsorbents experienced phenomenal growth.

Information is provided on the markets, raw materials, technological trends, marketing shifts, competing systems, prices, suppliers, end users, and volumes for 1988 with projections for 1993.

The Industry Review gives a comprehensive assessment of the structure of this business, including a competitive analysis of some 40 producers. The impact of government regulations is fully discussed.

The subscription fee is \$7,500. A free brochure, with Table of Contents, is available from Skeist Inc., 375 Route 10, Whippany, NJ 07981.

## ARCO Begins Construction Of Polyols Plant in France

ARCO Chemical Company, Newtown Square, PA, has started construction of a new facility to manufacture both conventional and polymer polyols at its Fos-sur-Mer, France site.

The plant will have a capacity of approximately 220 MM pounds per year and will be adjacent to the existing 330 MM pounds per year propylene oxide and 790 MM pounds per year tertiary butyl alcohol unit at Fos-sur-Mer.

Mechanical completion of the polyols facility is scheduled for the third quarter of 1990, at which time ARCO Chemical's production of polyols, under an operating agreement at the existing Lavera, France plant will cease.

In addition, ARCO is expanding the Fos-sur-Mer propylene oxide capacity by approximately 110 MM pounds per year

with an accompanying increase in methyl tertiary butyl ether output of approximately 3,000 barrels per day. The expansions also are targeted for start-up in the third quarter of 1990.

## Ambrose Company Relocates To Site in Arlington, WA

The Ambrose Company, Bellevue, WA, has relocated to Jensen Business Park in Arlington, WA. Ambrose was founded in 1935 in Seattle, WA.

The company will now control all manufacturing within the walls of its 23,000-sq ft operation at the industrial park site.

Marco Dalessi, newly-appointed Vice President—Fabrication, will direct the manufacturing phase of the business, as well as a custom-manufacturing shop.





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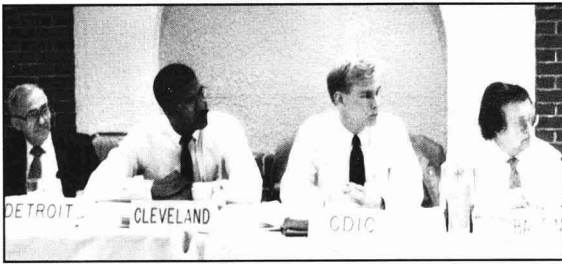
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## Society Educational Committee Chairmen Meet With FSCT Educational Steering Committee in Montreal

Representatives of Society Educational Committees met recently with members of the Federation Educational Steering Committee in Montreal, Canada. Topics of discussion included scholarship programs of the Federation and individual Societies; an update of progress achieved in the promotion of coatings career opportunities through the videotape, "The Choice"; an examination of the rules governing the A.L. Hendry Award competition; and Society-sponsored educational activities.

The meeting was chaired by Sidney Lauren, of the New England Society, Chairman of the FSCT Educational Steering Committee. In addition, the following Steering Committee members attended: Ted Fuhs (Chicago); John Gordon (Los Angeles); Berger Justen (Southern); Dave Kittredge (Los Angeles); Carl Knauss (Cleveland); and John Oates (New York).

Society Educational Committee representatives included: Alex Chasan (Baltimore); Gregory Gerhardt (C-D-I-C); DeVilla Moncrief (Cleveland); Bohdan Melnyk (Detroit); Art McDermott (Houston); Jim O'Brien (Kansas City); Temo Pereda (Mexico); Charles Craib (Montreal); Al Yokubonis (Northwestern); John Mitchell (Pacific Northwest); Tom Brown (Philadelphia); Jim Bohannon (Piedmont); Dick Mullen (Rocky Mountain); Peter Duncker (Toronto); and Ed Walker (Western New York).

Guests attending were: Kurt Weitz, Federation Treasurer; Gilles Belisle, Montreal Society President; and Renald Veilleux, member of Montreal Society Educational Committee.

Also attending were Thomas A. Kdcis, FSCT Director of Field Services, and Patricia D. Viola, Editor of JOURNAL OF COATINGS TECHNOLOGY.

### Review of Federation/Society Scholarship Programs

Federation scholarship funding for 1989-90 academic year, as approved by the Executive Committee, totals \$40,000. This compares with \$36,000 appropriated for 1988-89.

The following schools will receive the same level of funding as last year: University of Detroit—\$2,000; Eastern Michigan University—\$6,000; Kent State University—\$5,000; University of Missouri-Rolla—\$8,000; North Dakota State Uni-

versity—\$9,000; and University of Southern Mississippi—\$6,000.

The University of Waterloo (Canada) has been added to the roster of schools receiving Federation scholarship support, and will receive \$4,000.

In reviewing the appropriations, Chairman Lauren commented that the scholarship program makes a meaningful contribution to the well-being of the coatings industry, by helping to attract and train students for careers in coatings.

He expressed the hope that the level of Federation funding could be increased, in recognition of the success the scholarship-supported schools have had in placing their graduates in the industry.

Increasing financial support, however, underscores the need for comprehensive documentation, to provide an adequate basis for evaluating each school's needs and coatings program performance. He said current procedures will be refined to assure that schools submit the requisite information, and that funding recommendations are accompanied by appropriate supporting data.

It was noted, meanwhile, that substantial funding is provided by Constituent Societies, who have ongoing scholarship programs. These are either grants-in-aid to schools supported by the Federation program, or direct student grants—usually to children of members.

It was agreed that compilation of Society scholarship data would be a useful reference source.

Accordingly, Chairman Lauren will draft a letter for distribution to Society Presidents (copying Educational Committee Chairmen), requesting information on scholarship programs, including amount of funding, criteria, procedures, how administered, etc., to be submitted to Federation headquarters.

### A.L. Hendry Award Competition

Established in 1986 by the Southern Society in memory of an honored Past-President, Alfred L. Hendry, the Award (\$1,000) is for the best paper on some aspect of coatings technology, authored by an undergraduate student enrolled in a college program.

Chairman Lauren noted that faculty support is important to student participation and that professors are more likely to coop-

erate as advisors to students wishing to compete if they are accorded recognition as co-authors.

Accordingly, a recommendation will be forwarded to the Executive Committee that the rules governing the competition be amended to reflect contributions of the faculty advisor to a student paper based on original research, by including his/her name as co-author—but that the cash prize be presented only to the student, who must be the principal author. It is also recommended that competition eligibility be extended to include review papers (no faculty co-author on these). (*Ed. Note: Both recommendations were recently approved by the Executive Committee.*)

### Federation Career Videotape, "The Choice"

Reports on presentations of "The Choice" to student groups reflect generally good reaction to the career message.

Establishing an Ad Hoc Committee for continuing these presentations would ease burden on Educational Committee members, and could utilize special talents of Society members who might be more adept at promotion efforts. Enthusiastic individuals who are motivated and who have ability and energy to "sell the industry" can provide a personal touch to complement the message of the tape.

### Society Reports on Activities

The following are summaries of reports on Society educational activities:

#### Baltimore

Sponsored a 10-lecture course on basic and intermediate coatings technology at Catonsville Community College; a total of 19 students registered, all of whom attended regularly, and were awarded certificates of course completion . . . FSCT videotape, "The Choice," was shown at two local high schools . . . Held Education Night for members, at which representative from National Institute for Standards and Technology spoke on "Predicting Coatings Service Life."

#### C-D-I-C

Major activity is arranging for interesting personal/professional after-dinner pres-

entations at Society monthly meetings (technical presentation precedes dinner). Recent presentations included such topics as: pictorial tour of USSR; limiting personal liability; playing better golf; and travelogue on the Amazon River. This programming continues to be well-received by membership, and has contributed to recent increase in attendance at monthly meetings.

### Chicago

SYMCO '89, held August 29-30, focused on regulatory affairs and compliant coatings . . . Planning presentation of coatings technology course, to begin in early 1990 . . . Continuing \$1,000 grant-in-aid, to help defray costs of student tuition and other scholastic fees. Established a Joint Education Fund with local Paint and Coatings Association to award two additional \$2,000 grants-in-aid . . . Provided financial assistance for Eastern Michigan University's June "Coatings Workshop" for local high school chemistry teachers . . . Sponsored Management Seminar on "Creative Management with a Personal Touch," which won an MMA Award for excellence.

### Cleveland

Continuing participation in Northeast Ohio Science Fair; award cash prizes to winning student projects (in both junior and senior-level categories) which relate to coatings industry . . . Planning to present "The Choice" videotape at several Career Day events . . . Will provide on-site assistance for the back-to-back Inter-Society Color Council Annual Meeting and the Federation co-sponsored Symposium on Color and Appearance Instrumentation, to be held April 22-26, 1990, at the Cleveland Airport Marriott . . . Major activity is development of Annual Advances in Coatings Technology Conference. This year's event was held June 6-7 and featured sessions on instrumentation and methods, environmental considerations, pigment management, and processing.

### Detroit

Continuing sponsorship of evening coatings technology courses at University of Detroit, which now number seven: Electrodeposition; Fundamentals of Automotive Paint Systems (I and II); Principles of Color Technology; Polymer Technology for Coatings; Surface Coatings Technology; and a Coatings Lab course. Planning to videotape the lecture portion of the Color Tech course . . . Cooperating with University of Detroit Dept. of Continuing Professional Education in offering in-house training courses, which are tailored to participating company needs . . . Annual FOCUS conference was held April 19 and had as its theme, "Automotive Corrosion—Are We Winning the Battle?" . . . Contributed \$3,500 toward a graduate research project undertaken by

Polymer Institute at the University of Detroit . . . Contributed funds to assist Eastern Michigan University "Coatings Workshop" for high school chemistry teachers . . . Again held Education Night: speaker was Bob Bailey, Vice President/General Manager of Lilly Industrial Coatings, who presented, "It's Your Future—Manage It."

### Houston

Awarded scholarships to three children of Society members . . . Membership dues were increased \$10, all of which is to be allocated to Scholarship fund . . . Considering establishment of Society Foundation as a separate entity to administer scholarship program . . . Assisting in arrangements for 1990 Southwestern Paint Convention, which Society will host March 14-16.

### Kansas City

Principal ongoing activity is participation in local Science Fair, judging student entries and providing awards. Each of three winners receives savings bonds (their high school science departments each receive check in matching amount); presentations are made at Society monthly meeting (parents and teachers also attend), which is designated Education Night; this year's event also featured presentation by chemistry professor at William Jewell College, who put on a chemistry "Magic Show" . . . Scholarship funding (\$500) again provided for University of Missouri-Rolla, to be used for students pursuing degrees in polymer science and coatings-related areas.

### Los Angeles

Sponsorship of basic coatings technology course continues; students are awarded certificates for successful course completion . . . Copies of "The Choice" videotape were distributed to 49 area high schools. Presentations have been made at Los Angeles high schools, with good reaction to the industry career promotion message; several students have been placed in internship programs at area paint firms . . . Have succeeded in setting up a degree program at California Polytechnic State University (Cal-Poly) at San Luis Obispo. A curriculum outline has been presented to the State University Academic Senate for approval. Program is expected to begin in the Fall of 1990. Funding assistance will be required to outfit a laboratory, provide for instructors, and offer scholarship assistance; a number of paint manufacturers and supplier firms have already promised financial support.

### Louisville

Continuing sponsorship of Surface Coatings Technology program at University of Louisville. Co-sponsored by University's Dept. of Chemical Engineer-

ing, program covers the fundamentals of coatings in four semesters; courses are designed as single unit, however, each unit can be taken independently. Program currently covers: Non-Resinous Coatings Raw Materials; Coatings Formulation and End-Uses; Application of Coatings; and Resinous Raw Materials.

### Mexico

Sponsored presentations of FSCT seminars (Level I and II) on Statistical Process Control, for Society members . . . Assisting in development of program for Society technical meeting, September 6-8 in Monterrey; major topics to be covered include: formulation principles; rheology; polymers; pigments/color; additives; and government regulations . . . Evaluating potential for establishing post-graduate study program at National University of Mexico.

### Montreal

Sponsored 12-week coatings course, with notes in English and French . . . Planning effort to recruit students from junior college via Career Nights and various promotional efforts . . . Meeting with local colleges to discuss development of curriculum for chemical technology which would include coatings-related courses.

### New England

Continuing close liaison with M.S. Coatings and Adhesives program at the University of Lowell, which attracts many local coatings personnel to evening classes . . . Inaugurated scholarship funding for best coatings-related essays authored by full-time college students; awards (\$500 each) were presented to three students in this year's competition . . . Continued program of offering cash prizes (total of \$1,000) for the best student papers on coatings topics . . . Discussing prospects for establishing evening B.S. program in coatings at University of Lowell.

### New York

Joint sponsorship of coatings courses with local PCA continues. Understanding the Basics of Coatings I & II, a two-year, four-semester coatings technology curriculum is presented at Fairleigh Dickinson University; each one-year, two-semester module of the program is a complete course in itself and may be independently taken. Certificate is awarded to those completing course; each of the four semesters qualifies for three CEU's of credit . . . Laboratory Course for Paint Technicians is designed for newcomers to the industry and consists of combination of basic theory, demonstrations, and "hands-on" lab exercises; lectures are presented by industry specialists and given at laboratory of lecturer's em-



ployer. Course is offered when survey of membership indicates sufficient interest . . . Continuing efforts to establish a Coatings Center at Fairleigh Dickinson; currently developing teaching outline for review by university faculty/administration . . . Seeking interested Societies to join in developing Laboratory Exercise Curriculum, that could be used independently or in conjunction with lecture course . . . Assisted in developing schedule of speakers who updated members on various regulatory matters, at seminars held immediately preceding regular monthly meetings . . . Interested in acquiring support material (literature, lab experiments, etc.) for use in Career Day programs.

### **Northwestern**

Continue to maintain close liaison with North Dakota State University. Contributed \$3,000 to scholarship program there; contemplating establishment of a scholarship award to NDSU science students, as well . . . Education Night focused on "Careers in Coatings" and featured several NDSU graduates who related how their educational backgrounds led them to coatings careers; presentations were followed by showing of "The Choice" videotape. Guests included representatives from the Minnesota Science Teachers' Association and area high school science students . . . Annual Spring Symposium had as its theme "Health and Safety" . . . Planning expansion of Educational Committee to provide adequate staffing for visitations to area high schools, junior colleges, and colleges, to show "The Choice" and promote coatings career opportunities.

### **Pacific Northwest**

Portland, Seattle, and Vancouver Sections each contributed \$1,000 to scholarship fund . . . Portland Section organized series of half-day seminars on formulation

topics dealing with new or state-of-the-art technology . . . Vancouver Section to schedule presentations at local high schools to promote interest in coatings careers.

### **Philadelphia**

Sponsoring three coatings technology courses: Introduction to Coatings Technology; Formulation Technology; and Application and Testing of Coatings; these are designed to be full-semester duration, upper level undergraduate/graduate level offerings . . . Annual seminar this year focused on "Understanding and Dealing with the TiO<sub>2</sub> Shortage."

### **Piedmont**

Developing and maintaining close relationship with High Point College and The University of North Carolina at Greensboro . . . Co-sponsoring (with American Chemical Society) October 10 seminar on "Low Temperature Curing" . . . Planning scholarship funding program for students majoring in chemistry at High Point College . . . Planning to continue presentations of "The Choice" videotape to area schools this Fall . . . Continuing sponsorship of summer co-op programs for local college chemistry majors; have placed two students in this year's program.

### **Rocky Mountain**

Sponsored seminar on governmental regulations, featuring discussions on waste storage and disposal, SARA Title III, and dealing with regulatory agencies . . . Facing difficulty in pursuing activities with limited Society membership.

### **Southern**

Continuing close liaison with University of Southern Mississippi, through fund-

ing scholarship program and sponsoring annual Water-Borne and Higher-Solids Coatings Symposium . . . Continuing sponsorship of A.L. Hendry Award for best undergraduate student-authored paper on some aspect of coatings technology . . . Sponsored series of one-day technical seminars on various topics at Society Sections.

### **Toronto**

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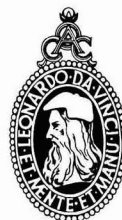
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- (1) Pascal, R.H. and Reig, F.L., "Pigment Colors and Surfactant Selection," *Official Digest*, 36, No. 475 (Part 1), 839 (1964).
- (2) Davidson, H.R., "Use and Misuse of Computers in Color Control," *JOURNAL OF COATINGS TECHNOLOGY*, 54, No. 691, 55 (1982).
- (3) Stephen, H.G., "Hydrogen Bonding—Key to Dispersion?," *J. Oil & Colour Chemists' Assoc.*, 65, No. 5, 191 (1982).
- (4) Patton, T. (Ed.), "Pigment Handbook," Vol. 1, John Wiley & Sons, Inc., New York, 1973.
- (5) Henderson, W.A. Jr. and Singh, B. (to American Cyanamid Co.), U.S. Patent 4,361,518 (Nov. 30, 1982).

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# A Review of Organic Coating Technology For U.S. Naval Aircraft

Charles R. Hegedus, David F. Pulley, Stephen J. Spadafora,  
Anthony T. Eng, and Donald J. Hirst\*

Naval Air Development Center\*

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Navy aircraft are subject to one of the most corrosive environments known. The primary defense against this environment is the organic coating system. The chemistry and properties of standard primers, topcoats, rain erosion, and fuel tank coatings are described. Increased emphasis on reducing organic solvent emissions has caused the development of a number of compliant coatings with reduced volatile organic compound (VOC) content. Water-borne, high-solids, and exempt-solvent coatings which are replacing the standard materials are described.

The advancement of polymer and pigment chemistry has subsequently caused the development of coatings with diverse and unique properties. A self-

priming topcoat has been developed to replace the current two-coat system with one coating. Flexible primers have been developed to reduce coating system cracking and chipping which is common on highly flexed areas. A high temperature organic coating (370°C) has been designed to provide corrosion and oxidation inhibition for metallic substrates. Finally, a temporary coating which was designed for quick and convenient field application is described. These coatings are currently being field tested and are expected to serve as the next generation of aircraft coating systems. A brief discussion on future trends of aerospace coatings is provided.

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## INTRODUCTION

Navy aircraft are normally deployed at coastal land bases or on board aircraft carriers. The continuous proximity to salt water and high humidity combined with atmospheric impurities make U.S. Naval aircraft subject to one of the most corrosive natural environments known (*Figure 1*). Careful consideration of materials deterioration is essential due to the high cost of these vehicles and the severe environment in which they operate. Variables such as system design, materials selection, service conditions, and maintenance policies play a significant role in an aircraft's response to its environment.<sup>1-6</sup> These variables impact equipment readiness, performance, and lifetime.

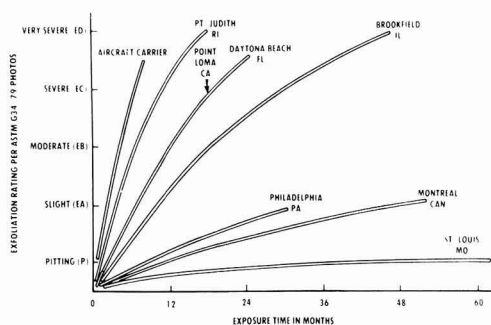
Aircraft considerations begin with design and materials selection. Although aluminum is the most common material used on aircraft structures, there are numerous alloys of aluminum, steel, titanium, and magnesium, all of which can be treated with a myriad of inorganic or metallic coatings prior to painting. The use of polymeric composites, some of which contain carbon fibers which are

highly cathodic,<sup>7</sup> increases the complexity of corrosion control. Other considerations include the type of aircraft, its mission, and maintenance policies. Tactical aircraft fly at altitudes up to 18,000 meters (60,000 ft) at speeds exceeding Mach 2. This can cause aircraft skin temperatures ranging from -54 to 177°C (-65 to 350°F). Flight conditions, especially with helicopters, can cause severe airframe vibration and flexing. During take-off and landing, an aircraft will be impacted by dust, gravel, stones, and de-icing salts. Many operational and maintenance chemicals commonly used or found on aircraft, such as paint strippers, battery acid, de-icing fluids, and cleaners, are corrosive. Exposure to these chemicals, combined with the frequent opening of canopies, radomes, and equipment bays, drastically increase the susceptibility of naval aircraft to corrosion and other forms of materials degradation. Reference (6) provides an excellent documentation of aircraft corrosion causes and case histories. References (8) and (9) describe guidelines and specifications for aircraft corrosion control.

The primary function of organic coatings applied to military aircraft is to protect the airframe from the operational and environmental conditions previously de-

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\* Aerospace Materials Division, Warminster, PA 18974-5000.



scribed. Reference (10) concludes, "The rate controlling parameter for the corrosion of aircraft alloys, excluding the mechanical damage factor, is the degradation time of the protective coating system." This is a clear indication of the critical importance of corrosion and erosion control provided by the coating system. A second function of coatings on military aircraft is to provide camouflage properties to minimize enemy detection and tracking during mission operations. Because of these coating performance benefits and the high cost of aerospace systems, only the most protective and durable paints have been used on aircraft, regardless of application cost. This paper will review technology for current conventional coating systems, compliant primers and topcoats, and state-of-the-art coatings. A discussion on future needs and trends of coatings technology for aircraft is also provided.

## STANDARD FINISHING SYSTEM

For nearly 20 years, the standard organic coating system applied to Navy aircraft has consisted of an epoxy primer and a polyurethane topcoat. Due to its exceptional adhesion, corrosion inhibition, and durability, this system has protected aluminum aircraft structures from the harsh environment in which they operate. The inherent lifetime of this primer and topcoat system on Navy aircraft is four to six years, after which the coating system is chemically or physically removed and the aircraft is repainted. The major cause for paint failure is extended exposure to weather and operational chemicals which embrittle and discolor the coating, causing paint around fastener patterns and seams to crack and chip, thus exposing bare metal. A general description of the finishing system used on Navy aircraft is outlined in military specification MIL-F-7179 and reference (9). In many cases, U.S. Navy, Army, and Air Force use similar coating specifications. However, each service has performance priorities based on mission requirements. For example, the Navy places a large emphasis on corrosion inhibition due to its inherent corrosive environment. (*Table 1 provides a list of the common specifications for Navy aircraft coating systems.*)

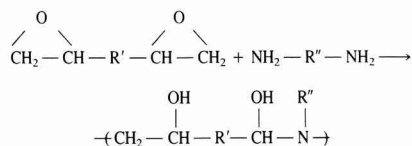
In addition to the standard primer and topcoat which are the primary coatings used in Navy aircraft finishing

systems, other specialized coatings are applied to address specific concerns. Some of these materials are sealants, rain erosion materials, and fuel tank coatings.

## Primer

Epoxy resins are commonly used as binders in high performance primers due to their exceptional adhesion and chemical resistance properties. Two types of epoxy primers are currently applied to Navy aircraft, solvent-borne (MIL-P-23377E) and water-borne (MIL-P-85582A). The water-borne primer is a fairly recent development and is discussed in the compliant coatings section of this paper.

The solvent-borne primer is manufactured and packaged as a two-component epoxy/polyamide system. Table 2 lists a control formulation which meets the requirements of MIL-P-23377E. One component contains an epoxy resin which is the product of a condensation reaction between epichlorohydrin and bisphenol A. The second component is a solution of a multi-functional polyamide resin in a solvent blend. Upon mixing the two components, which is done just prior to application of the primer, reaction of epoxide and amide groups within the resins ensues:



where:

R' is the bisphenol A derivative

$R''$  is a polyamide chain.

The product of this reaction is a highly crosslinked polymer which forms the matrix of the primer film. The chemical and mechanical properties of the epoxy matrix cause the primer to be adherent, chemically resistant, and durable. References (11) and (12) provide detailed discussions of epoxy resin chemistry for coatings.

The epoxide component of the primer contains various pigments, including titanium dioxide, strontium chromate, and extenders. Strontium chromate is the most critical of these pigments since it is well known as an exceptional corrosion inhibitor, especially for aluminum.<sup>13</sup> A number of specific mechanisms of corrosion inhibition by chromates have been proposed.<sup>14-19</sup> In general, protection is attributed to passivation of the metal surface by the chromate ions which inhibit the anodic reaction.<sup>18</sup> Therefore, when the primer is applied, not only does it form a barrier between the metallic substrate and the corrosive environment, but active corrosion inhibition is initiated by leaching of the chromate pigment(s). The latter mechanism is especially critical when breaks occur in the paint film. In this case, the chromate ions leach out of the coating, protecting the exposed metal. The leaching rate of the chromate is considered a critical factor in the inhibition mechanism.<sup>19</sup> Since the diffusion rate of pigments varies for different polymer binders, it is essential that the coating is formulated and designed to provide the optimum leaching rate for corrosion protection. In many solvent-borne epoxy primers for aircraft,



strontium chromate yields optimum results. However, other binders used in recently developed primers allow the use of barium chromate, zinc chromate, and other inhibitors.

Titanium dioxide in the primer enhances durability, chemical resistance, and opacity of the applied coating.<sup>20,21</sup> The extender pigments can be silicas, silicates, carbonates, or sulfates.<sup>22</sup> The extenders are normally inexpensive and provide a cost-effective component which "fills" the coating and reduces gloss of the applied film. The surface irregularities which cause gloss reduction also act as anchors for a topcoat, thus enhancing intercoat adhesion by enhanced mechanical attachment. Reference (19) provides a comprehensive review of epoxy primer technology for aircraft applications.

Upon mixing the two components of the epoxy/polyamide primer, the curing reaction begins. At this stage, the coating is suitable for spray application. After application, 15 to 23  $\mu\text{m}$  (0.6 to 0.9 mils) thick, the coating is tack-free within one hour and dry hard in less than six hours. If a topcoat is to be applied, it is usually done within this time period to ensure proper adhesion. The primer is almost entirely cured within seven days, at which time the desired dry film properties are obtained.

The critical performance properties of MIL-P-23377E are listed in Table 3. As previously indicated, adhesion and corrosion inhibition are essential. Since water is a primary factor in reducing adhesion of paint films,<sup>23,24</sup> adhesion of the primer is characterized after 24-hr immersion in water by a tape test. The required performance is the highest rating described in ASTM D 3359 method A, which is a 5A—no coating removal. Adhesion is also characterized by a method which quantifies the force required to scrape the primer from the substrate (ASTM D 2197, method A).

**Table 1—Common Specifications for Navy Aircraft Coating Systems**

Number	Title
MIL-H-5606E .....	Hydraulic Fluid, Petroleum Base, Aircraft, Missile, and Ordnance
MIL-F-7179E .....	Finishes and Coatings, Protection of Aerospace Weapons Systems, Structures and Parts, General Specification for
MIL-C-22750D .....	Coating, Epoxy Polyamide
MIL-P-23377E .....	Primer Coating, Epoxy Polyamide, Chemical and Solvent Resistant
MIL-L-23699 .....	Lubricating Oil, Aircraft Turbine Engines, Synthetic Base
MIL-C-27725B .....	Coating, Corrosion Preventive, for Aircraft Integral Fuel Tanks
MIL-R-81294B .....	Remover, Paint, Epoxy and Polyurethane Systems
MIL-C-81706 .....	Chemical Conversion Materials for Coating Aluminum and Aluminum Alloys
MIL-H-83282B .....	Hydraulic Fluid, Fire Resistant, Synthetic Hydrocarbon Base, Aircraft
MIL-C-83286B .....	Coating Urethane, Aliphatic Isocyanate for Aerospace Application
MIL-C-85285 .....	Coating, Polyurethane, Aliphatic, Weather Resistant, Low Infra Red Reflective
MIL-C-85322 .....	Coating, Elastomeric, Polyurethane, Rain Erosion Resistant, for Exterior Aircraft Use
MIL-P-85582 .....	Primer Coating: Epoxy, VOC Compliant, Chemical and Solvent Resistant

**Table 2—Epoxy Primer (MIL-P-23377E) Control Formulation**

Component A	Wt %
Epoxy resin <sup>a</sup> .....	18.2
Strontium chromate .....	12.0
Titanium dioxide .....	2.3
Magnesium silicate .....	5.5
Silica .....	2.9
Cellosolve acetate .....	16.4
Methyl ethyl ketone .....	4.1
<b>Component B</b>	
Polyamide resin <sup>b</sup> .....	7.5
Isopropanol .....	24.5
Toluene .....	6.6
<b>Total</b> .....	100.0

(a) Shell Epon 1001-T75.

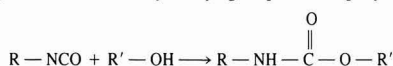
(b) Henkel Versamid 115.

Corrosion inhibition is evaluated by applying the primer to a chromate conversion coated (MIL-C-5541), 2024 T-3 (QQ-A-250/4) panel. After curing for seven days, an "X" is scribed through the coating so that the substrate is exposed. It is subsequently placed in a 5% NaCl spray chamber (ASTM B 117) for 1000 hr. Following exposure, suitable primers show no uplifting of the coating or corrosion of the substrate. An aluminum/graphite epoxy specimen, which forms a galvanic couple, is also exposed for 500 hr. In addition, resistance to filiform corrosion is required.

## Topcoat

A topcoat is applied to military aircraft to enhance environmental protection and provide desired optical properties, usually camouflage. Aliphatic polyurethane coatings are ideal for this application due to their superior weather and chemical resistance, durability, and flexibility. It is interesting to note that although polyurethane topcoats have become increasingly popular over the last 20 years, initial versions of these materials were applied to German aircraft during World War II. The coatings used by the U.S. Navy are specified in MIL-C-83286B and MIL-C-85285B. The latter specification has been revised recently to address high-solids, low volatile content (VOC) coatings which will be discussed in the next section of this review.

Polyurethanes are a huge class of polymers with a wide variety of chemical and physical properties. Most urethanes used in aircraft coatings are classified as ASTM Type V which are two-component, reactive materials. One component of the coating is a solution of hexamethylene diisocyanate (HDI). The second component contains a hydroxylated polyester. Upon mixing, the isocyanate groups react with the hydroxyl groups of the polyester:



where:

R is aliphatic hydrocarbon chain

R' is aliphatic polyester chain.

The resulting polymer is flexible, yet extremely durable and chemical resistant. Aliphatic isocyanates and polyesters are used in topcoats because they provide outstanding weather resistance compared to aromatics, which degrade when exposed to ultraviolet light. References (25-28) provide more detailed discussions about polyurethane chemistry.

The main pigment used in white and gray topcoats is rutile titanium dioxide, which is dispersed in the polyester component. Due to its light scattering and chemical stability, it is an ideal pigment to provide whiteness and hiding power to the topcoat.<sup>20,21</sup> Other fillers can be used as tinting pigments for additional colors, and extenders may be added for gloss reduction.

When the two components are combined and the polyurethane reaction begins, the coating is ready for application. This coating is normally spray applied to a dry film thickness of  $53.3 \pm 2.5 \mu\text{m}$  ( $2.1 \pm 0.1$  mils). The coating is set-to-touch and dry hard (when cured at room temperature) within two and six hours, respectively. Although the painted surface can be handled after six hours without damage to the coating, full cure properties are normally not obtained for approximately seven days.

The performance properties required in MIL-C-83286B are listed in Table 3. The most critical requirements are weather resistance, chemical resistance, and flexibility. Weather resistance is evaluated by laboratory exposure in an accelerated weathering chamber (ASTM G 26, Type BF) for 500 hr. This chamber is a continuous cycle of high intensity ultraviolet light and water spray. Although studies have shown that there is no precise correlation with outdoor exposure,<sup>29-31</sup> the accelerated exposure does indicate if the coating is susceptible to ultraviolet and/or water degradation. These results are used in conjunction with actual outdoor exposure for one year in Florida. Both accelerated and real-time weathering conditions cause only minimal changes in the color, gloss, and flexibility of Navy aircraft topcoats.

Chemical stability is evaluated by exposure of the applied topcoat to various operational fluids such as lubricating oil, hydraulic fluid, and hydrocarbons at elevated temperatures and/or extended durations. In addition, the coating is subjected to 149°C (300°F) for 4 hr. Suitable topcoats show no defects other than slight discoloration after exposure to these conditions.

Flexibility requirements for MIL-C-83286B topcoats include impact and mandrel bend tests. A 60% elongation of the coating after impact at room temperature and a 0.95 cm (0.375 in.) 180° bend at -51°C (-60°F) are required of gloss colors without cracking of the film. Flexibility requirements for low gloss colors are less stringent since it is difficult to formulate flexible low gloss coatings due to high pigment concentrations which normally embrittle the film.

The epoxy/polyurethane coating system previously described has performed well in its function of protecting aircraft structures. However, environmental and safety issues, as well as state-of-the-art advances in polymer and pigment technology, have caused a rapid evolution of organic coating science. It has become evident that major improvements in coating system performance with less hazardous and toxic effects on our environment are possi-

ble. Current and future advances in aerospace coating technology are discussed later in this overview.

## Sealants

Although the standard epoxy primer provides excellent adhesion and corrosion inhibition, it is brittle. This lack of ductility results in cracking of the paint system on highly flexed areas of the aircraft. In order to improve the overall flexibility of the standard Navy coating system, sealants are frequently incorporated into aircraft finishing systems. These sprayable materials are applied between the primer and the topcoat at thicknesses up to 203  $\mu\text{m}$  (8 mils) and are primarily formulated from polysulfide and polyurethane binders. Their elastic nature minimizes cracking of the paint system. Common specifications covering these materials are MIL-S-8802, MIL-S-81733, MIL-S-83430, and MIL-C-85322 (Type II). Critical requirements in these specifications are low-temperature flexibility (mandrel bend tests), chemical resistance (fluid immersion at elevated temperatures), and corrosion resistance (5% NaCl salt spray tests). Although these sealants mainly provide corrosion protection by the formation of a relatively impermeable barrier, MIL-S-81733 and MIL-C-85322 Type II sealants also contain strontium chromate for chemical corrosion inhibition. An in depth discussion of the specific chemistry and application properties of sealants is beyond the scope of this paper and will not be addressed here. However, a detailed discussion of this technology is provided in references (32) and (33).

## Rain Erosion Materials

In addition to their harsh environment, aircraft must also endure seemingly harmless natural conditions that can deteriorate the performance of coatings and their underlying structures. One example of this phenomenon occurs when airborne debris, like sand or rain droplets, impact aircraft leading edges and radomes during flight. The force of impact from these particles can erode the coating system and adversely affect the underlying substrate. The standard paint system does not provide adequate protection against this condition. Even when applied at two to three times its normal thickness, the coating system erodes prematurely.

One method of determining the rain erosion resistance of materials is the use of a whirling-arm facility which simulates flight through a rain storm. The test fixture at this laboratory consists of an engine which rotates an extended arm at speeds up to 833 kilometers per hour (500 mph). Stationed above the arm are tubes through which water is allowed to flow at a rate corresponding to an average rain fall of 1.27 cm (0.5 in.) per hour. As the arm turns, specimens pass directly below the nozzle, allowing for the maximum consistent impact of the water droplets on the specimen. A material with a failure time of less than five minutes in this test would not provide adequate protection against erosion during aircraft deployment. Endurance times of 5 to 20 min indicate a coating that would provide protection, but would also require continual maintenance and repair. Finally, an

**Table 3—Critical Performance Characteristics of Aircraft Epoxy Primer (MIL-P-23377E) and Polyurethane Topcoat (MIL-C-83286B)**

Property	Test Method	MIL-P-23377E	MIL-C-83286B
Drying time	ASTM <sup>a</sup> D 1640	—	2 hr
Set-to-touch		—	—
Tack-free		1 hr	—
Dry-hard		6 hr	6 hr
Pot life	Gel Time	8 hr	6 hr
Gloss (60°)	ASTM D 523	20 max	—
Gloss colors		—	90 min
Camouflage		—	7-12
Adhesion			
Wet tape	ASTM D 3359 A	No uplifting-5	No uplifting-5
Scrape	ASTM D 2197 A	3 kg	—
Flexibility			
GE impact	FTMS-141 <sup>b</sup> 6226	10	—
Gloss colors		—	60
Camouflage		—	20
Mandrel bend	ASTM D 1737 (–51°C)	—	0.95 cm
Gloss colors		—	1.27 cm
Camouflage		—	—
Strippability (% surface stripped)	MIL-R-81294	>90%	—
Humidity resistance	95% RH & 49°C	—	30 Days
Heat resistance	150°C	—	4 hr
Fluid resistance		Resistance to total immersion	
Lubricating oil	MIL-L-23699 @ 121°C	24 hr	24 hr
Hydraulic fluid	MIL-H-83282 @ 65°C	24 hr	—
Hydraulic fluid	MIL-H-5606 @ 25°C	—	7 Days
Distilled water	Total immersion	96 hr (49°C)	96 hr (38°C)
Hydrocarbon	25°C	MEK wipe	7 Days
Skydrol 500B	MIL-H-83306 @ 25°C	—	7 Days
Corrosion resistance			
5% NaCl salt spray			
Aluminum	ASTM B 117	1000 hr	500 hr
Al-Gr/Ep	ASTM B 117	500 hr	—
Filiform	ASTM D 2803	1000 hr	—
Weather resistance			
Accelerated	ASTM G 26, Type BF	—	500 hr
Outdoor	Florida exposure	—	1 Year

(a) ASTM: American Society for Testing and Materials.

(b) FTMS-141: Federal Test Method Standard 141B.

elastomeric system lasting 20 min or greater would exhibit an extended field lifetime while not requiring continual maintenance between rework operations.<sup>34</sup>

When tested in this facility, the standard polyurethane topcoat is completely removed within a few minutes. In an attempt to improve performance, teflon particles were incorporated into the standard topcoat. However, this material only lasted five minutes in the whirling arm test before complete failure. Since this response only meets the minimum standards for protection, materials specifically designed for rain erosion resistance are employed. Two types of materials are used for this application, elastomeric coatings and tapes. Both types of material exhibit properties that offer advantages for different specific uses.

The rain erosion resistant coating used on Navy aircraft is a two-component polyurethane material. One component consists of a pigmented high molecular weight polyether type polyurethane. The other component contains a clear ketimine (blocked diamine) resin that acts as both a crosslinking agent and a chain extender.<sup>25</sup> When combined, the two components form an elastomeric coating which can absorb and dissipate the energy of impacting

rain droplets, thus preventing failure. This coating can typically withstand 30 min in whirling arm tests. Flexibility is characterized by a 0.635 cm (¼ in.) mandrel bend at –51°C (–60°F), whereas the standard topcoat only requires a 0.95 cm (⅜ in.) or 1.27 cm (½ in.) mandrel. However, in order to exhibit high elasticity, the polymer crosslink density is decreased, causing poorer chemical resistance and weathering properties. Therefore, to improve the finishing system durability, these materials are normally overcoated with the standard topcoat.

Although elastomeric coatings offer increased resistance to rain erosion, the optimum protection for Navy aircraft is provided by elastomeric tapes. These materials can be clear or pigmented, polyurethane-based films and are supplied with or without an adhesive backing. Unlike coatings, these tapes are bonded to the surface and do not require a drying time. Early versions of these materials were clear aromatic type polyurethanes. Although durable, these aromatic materials had poor weatherability. The latest versions of these materials, however, are aliphatic. These new materials are extremely durable (lasting up to 70 min in whirling arm tests) and have excellent weatherability.<sup>35</sup>



## Internal Fuel Tank Coatings

Certain internal areas of aircraft are exposed to selective environments which pose unique problems. One example is fuel tanks. Aviation fuels contain additives which may be corrosive. If left unprotected, fuel tanks would corrode and leak. In order to protect these areas, the Navy uses a fuel tank coating (MIL-C-27725B, Type II, Class A) which is a two-component urethane material designed for application to nonferrous surfaces. When the two components are combined, they form a highly cross-linked urethane binder with a high degree of chemical resistance. The fluid resistance requirements for this material are significantly more severe than those of the standard primer and topcoat. The conventional topcoat must withstand 24-hr immersion on unscribed panels without degradation, whereas the fuel tank coating specifications requires 14 days immersion of specimens with scribes through the coating. This high degree of chemical resistance is necessary because the coating is not only subjected to the various chemicals contained in aviation fuels, but it is also exposed to aircraft operational chemicals, salt water and dilute acidic solutions.<sup>36</sup>

Another use for MIL-C-27725B was uncovered in an investigation of corrosion resistant coatings for environmental control system (ECS) ducts.<sup>37</sup> ECS ducts on F-14 aircraft are regularly subjected to temperatures of 204°C (400°F). Because this fuel tank coating is highly cross-linked, it also has good resistance to thermal degradation. When tested against other candidate materials for this application, the polyurethane coating provided the best overall adhesion, and chemical and corrosion resistance while remaining stable up to 227°C (440°F).

## COMPLIANT PRIMERS AND TOPCOATS

### Environmental Regulations

The Environmental Protection Agency, by enforcing the Clean Air Act, has tried to standardize organic emission regulations for paint application in the aerospace industry. The most stringent regulations within the United States have been implemented by California because of severe air-quality problems. By definition, aircraft and the weapons attached to them fall under the aerospace rules:

District	Aerospace Rule
Bay Area .....	Regulation 8, Rule 29
San Diego .....	Rule 67.3
South Coast .....	Rule 1124

These rules limit the amount of volatile organic compounds (VOC) in the paints at the point of application. The current maximum limits for primers and topcoats for aerospace equipment are 350 and 420 grams per liter of paint, respectively. Measurements of VOC content must include not only solvents but any volatile chemicals in the resin, pigment, or additive components of each paint. Exempt solvents, such as water and 1,1,1-trichloroethane, are not included in the volatile content or the total paint volume. The most common test method for VOC measurement is ASTM D 3960.

The Navy has numerous rework facilities and contractor plants throughout California and, due to the large volume of paint used for aerospace systems, has agreed to abide by the aforementioned regulations and to implement compliant coatings. In addition, organic coatings under development for use on Navy systems are being formulated to meet these requirements. Specific technologies which are being utilized to meet this goal are water-borne and high-solids (low volatiles), and exempt solvents coatings.

### Water-borne Technology

Water can be used in organic coatings to form either solutions or emulsions. However, the solubility of most polymers in water, especially those suited for high performance coatings, is very limited. Monomers and polymers which can be diluted with water have chemical structures that are hydrophilic. Unfortunately, this affinity for water usually results in paint films with poor resistance to moisture, making them impractical for use in a marine environment.

Generally, the most durable water-borne finishes are emulsions which employ a combination of water and organic cosolvents. This technology has been used to develop a water-borne, epoxy primer suitable for aerospace applications.<sup>38</sup> It is produced as a two-component, high-solids, solvent-borne material. One component contains the pigmented epoxide resin solution and the other contains a polyamide curing agent solution. Prior to application, the two components are mixed and water is added, forming an emulsion. Formulation of the coating without water is an important advantage since it eliminates the concern for freeze-thaw stability.

Upon application and cure, the coating has similar chemical and physical characteristics to the standard aerospace epoxy primers. In the laboratory, this water-borne primer has met all of the performance requirements of specification MIL-P-23377E. The primary difference is in the use of barium chromate as the corrosion inhibiting pigment in the water-borne primer, rather than the commonly used strontium chromate. This substitution was made during development because of the incompatibility of the curing agent with the strontium ion, resulting in excessively quick reaction times and a short pot life. Initially, there was concern that the barium chromate, which is less soluble in water than strontium chromate, would not inhibit filiform corrosion as effectively. Extensive laboratory testing<sup>39</sup> and application to hundreds of fleet aircraft have demonstrated the quality of this new primer. In 1984, specification MIL-P-85582A was established for water-borne, epoxy primers with a maximum VOC content of 340 g/L. The compatibility problem with strontium chromate has been resolved and a separate class within the specification has been established for primers with this inhibitor.

The major adjustment on the part of the applicator is necessitated by the slower drying time, particularly when the humidity is high. Drying can be assisted by increasing the air flow across the wet primer. The coating is dry-hard in 6 hr with an air velocity of 27 m/min (88 ft/min) which is common in paint application hangers with forced air

movement. The low volatility of water also results in a smoother surface finish which is evident even after top-coating. The higher density of water (compared to organic solvents) yields significantly less overspray and improves the transfer efficiency. Organic contaminants which may be on the substrate, such as hydraulic fluid, are exposed by the primer due to its high surface tension and incompatibility with these organic fluids. Another interesting result from the use of this coating is a reduced chromate concentration in the surrounding atmosphere during application. This results from density and surface chemistry effects which: (1) reduce overspray and (2) cause any overspray which does occur to be carried quickly to the ground. Clean-up of application equipment can be accomplished in many cases with soap and water.

### High-solids Technology

The development of high-solids coatings for aerospace equipment has been a slow, deliberate process. Reduced solvent concentrations generally yield a higher viscosity, shorter pot life, and rougher surface finish. The use of low molecular weight resins to reduce the viscosity also may shorten the pot life and decrease the flexibility of the applied film. The pot life can be increased by chemically inhibiting the polymers from the crosslinking reaction until they are "unblocked" by moisture in the air. By inhibiting the reaction, drying time is also extended. The use of high-boiling solvents which improve the surface finish further increases the drying time. These solvents can sometimes cause a new phenomenon, where sharp edges are uncoated and exposed due to paint flow-out.

Recently, a number of breakthroughs have occurred. An evaluation of high-solids, epoxy primers at this laboratory has indicated that the technology is feasible.<sup>39</sup> As expected, they exhibit a higher viscosity, shorter pot life, and longer drying time. However, one primer has met all of the performance requirements in specification MIL-P-23377E. Despite the high viscosity, it can be sprayed to a dry-film thickness of approximately 19  $\mu\text{m}$  (0.75 mils) as required for aircraft application. The test results have been used to prepare a revision to the specification (with a separate high-solids class). The maximum VOC content will be 340 g/L.

The chemistry required for high solids epoxy primers is also appropriate for VOC reduction of epoxy topcoats. Although epoxy topcoats have poor weather resistance and flexibility, they are currently used to touch up damaged aircraft coating systems. (Use of the standard polyurethane topcoat is restricted on ships.) Two epoxy topcoats having a VOC of 340 g/L have been evaluated<sup>39</sup> and specification MIL-C-22750 has been revised to include provisions for their use. This revision will be issued in the near future.

Most of the research and development in the area of VOC compliant topcoats has been devoted toward polyurethanes since they represent the optimum performance attainable with regard to flexibility, durability, and weatherability. Specification MIL-C-85285B has been revised to accommodate high-solids materials at a maximum VOC content of 420 g/L (for aircraft application). A number of coatings have qualified to the specification

requirement and are being applied to aircraft in areas with VOC regulations.

### Exempt Solvent Technology

1,1,1-trichloroethane (TCA) is listed as an exempt solvent in VOC regulations issued by 45 of the 50 states. Since it is not photochemically reactive, it does not contribute to the generation of "smog" in industrial areas. However, if it reaches the upper layer of the earth's atmosphere, TCA will react with and degrade the ozone layer.<sup>40</sup> For this reason, TCA may only be a stop-gap solution to existing environmental regulations.

TCA is compatible with most organic resins when used in combination with other suitable solvents. It has a number of advantages, including no appreciable odor or flammability and minimal overspray (due to its high density). Applicators often prefer this option, since it requires no modifications to existing procedures or equipment. Coating performance is virtually unchanged from the original noncompliant paints. In addition, more paint suppliers can furnish materials at lower cost in comparison to the new water-borne and high-solids coatings.

However, there are many reasons to restrict the use of TCA in paints. In addition to the potential ozone problem, chlorinated solvents are treated as a hazardous waste and require expensive disposal methods. They react with aluminum, particularly at high temperatures or pressures. Therefore, airless spray equipment must have all wetted parts composed of stainless steel or other resistant materials.

A special concern for the Navy is the tendency of aggressive ions such as chlorine to cause stress-corrosion cracking within high-strength, structural alloys. Extensive testing by Grumman Aircraft Systems, the McDon-

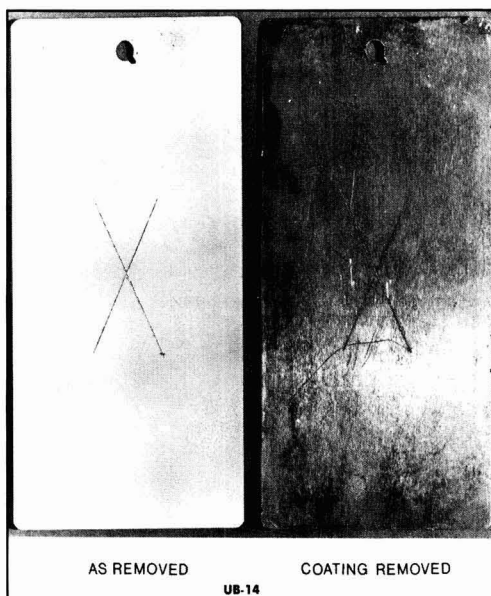


Figure 2—Aluminum specimens painted with UNICOAT after exposure to 5% salt spray for 2000 hr

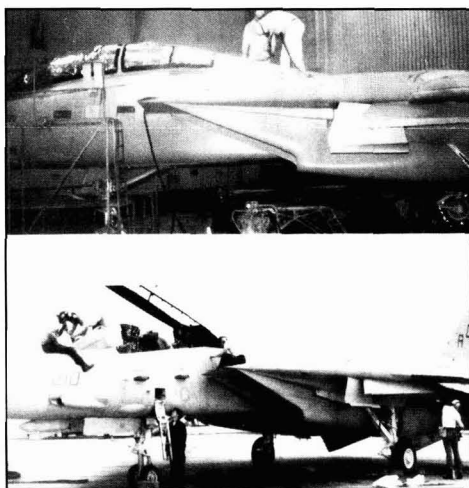


Figure 3—An F-14 aircraft during and after application of UNICOAT

nell Aircraft Company, and our laboratory<sup>41</sup> revealed that TCA could be used with certain restrictions. First, the solvent must be an inhibited grade such as Dow Chemical's Chlorothene SM. Then, each paint specification utilizing TCA must limit the maximum solvent retention to 1% by weight after seven days air-dry in accordance with test method ASTM F 151. Finally, paints containing TCA must not be used in high-temperature applications, such as engine components, or where the solvent can become entrapped, such as fasteners (wet installation) or faying surfaces. In 1987, specification MIL-P-23377E was the first to include a separate exempt-solvent class. Since then, epoxy primers from numerous coatings manufacturers have been developed and qualified to this specification.

The main approach for reducing VOCs of primers and topcoats has been by utilizing water-borne and high-solids technology, respectively. This approach has been successful. However, many specialty coatings are not compatible with either of these methods and must utilize exempt solvent technology. Although exempt solvents still pose environmental and health concerns, limited usage is appropriate for application of critical coatings. Since the area of low VOC coatings is receiving much research and development attention, major strides are expected in the near future.

## STATE-OF-THE-ART COATINGS

Although the current paint system performs well, the individual coatings exhibit several deficiencies and concerns. The epoxy primer is brittle, especially at low temperatures ( $-51^{\circ}\text{C}$ ), resulting in extensive cracking of the paint system in highly flexed areas of the aircraft. The sealants are soft, easily deformed, and difficult to apply and remove. In addition, increased awareness and concern for the environment and worker safety have caused local, state, and federal governments to limit VOC emissions from painting operations. The carcinogenic effects

of chromates which are used in aircraft primers present another concern about current coating systems. The coatings discussed in the following have recently been developed to address some or all of these issues. Individually, they exhibit improvements in handling and performance properties. Together, they provide attractive alternatives to the aerospace finishing industry.

### UNICOAT, A Self-Priming Topcoat

A coating has been developed in this laboratory<sup>42</sup> which can be applied directly to metallic and polymeric substrates. It exhibits the adhesion and corrosion inhibition of a primer with the chemical and weather resistance, durability, and flexibility of a topcoat. As such, the coating was designed to replace the standard epoxy primer/polyurethane topcoat system. This coating is a two-component polyurethane which is similar in chemistry to the standard aircraft topcoat. However, the binder in this coating was specifically designed to adhere directly to the substrate while still maintaining flexibility, and chemical and weather resistance. The polyester component contains titanium dioxide, vesiculated beads, zinc molybdate, zinc phosphate, and organo-zinc salt pigments. The titanium dioxide provides hiding power and whiteness, while the vesiculated beads enhance opacity and reduce gloss.<sup>20,21,43,44</sup> The remaining three pigments provide outstanding corrosion inhibition without the use of chromate or lead. This coating has similar application properties to the standard topcoat and it is applied to a dry film thickness of approximately  $50\text{ }\mu\text{m}$  (2 mils). The VOC of the application-ready coating is less than  $415\text{ g/L}$  which meets the current regulations for aerospace equipment topcoats. The admixed viscosity is 44 sec through a Ford 4 (ASTM D 1200) viscometer. UNICOAT can be applied by airless, air-assisted airless, electrostatic, or conventional spray equipment.

References (42), (45), and (46) describe the laboratory performance of UNICOAT. In general, the properties meet or exceed the performance requirements of the standard coating system (Table 2). Scribed aluminum specimens were exposed to 5% salt spray for over 2000 hr without substantial substrate corrosion (Figure 2). Tape and scrape adhesion of the coating after seven days immersion in water at  $65^{\circ}\text{C}$  ( $150^{\circ}\text{C}$ ) far surpass the requirements listed in Table 2. The scrape adhesion following this exposure is greater than 5 kg compared to the 3 kg requirement of the epoxy primer (with no exposure).

Following laboratory characterization of UNICOAT, additional specimens were exposed on active Navy ships

Table 4—Flexibility of Aircraft Elastomeric Primer and Epoxy Primer Performance

Test	Polyurethane Primer	Epoxy Primer
Impact elongation ( $21^{\circ}\text{C}$ )	60%	20%
Mandel bend ( $-51^{\circ}\text{C}$ )	2.54 cm	0.32 cm
Tensile elongation ( $21^{\circ}\text{C}$ )	90%	7%
Tensile strength ( $21^{\circ}\text{C}$ )	24.15 MPa (3500 PSI)	15.14 MPa (2200 PSI)



for nine months. The results confirmed the coating's durability and corrosion protection in actual environmental conditions. Subsequently, four aircraft (an F-14 and three H-3's) were entirely painted with the self-priming topcoat (see Figure 3). In all cases, production engineering personnel were pleased with its application and appearance. One year after application to the F-14, including nine months deployment at sea, the coating remained in excellent condition and evaluations from field personnel were exceptionally high. Improved durability and cleanability of UNICOAT has been observed in combination with a substantial decrease in required corrosion control maintenance on this aircraft. Field tests for the H-3 aircraft are producing similar results.<sup>47</sup> One of these aircraft was deoxidized but not chromate conversion coated, eliminating all of the chromates from its finishing system.

Preliminary results strongly indicate that the advantages provided by UNICOAT include significant reductions in:

- (1) Paint application time, manpower, and materials;
- (2) Emissions of hazardous materials; and
- (3) Aircraft maintenance.

The estimated annual coat savings for the Navy due to the application of one coating in lieu of the current two-coat system for aircraft is substantial. These savings result from a 30% reduction in the required manpower and materials for painting an aircraft. Emission of hazardous materials are reduced in that: (1) the VOC content of this coating system is far less than that of the standard primer and topcoat system and (2) the coating eliminates the use of chromates.

### Flexible Primer

Since the standard epoxy primer is relatively brittle, sealants are sometimes spray applied between the primer and topcoat in aircraft finishing systems. An attractive alternative is to utilize a coating that possesses the adhesion of a primer and the flexibility of a sealant, thus eliminating the logistical and application problems inherent in stocking and applying two materials instead of one. An elastomeric primer which provides these benefits has been characterized and implemented on Navy aircraft.<sup>48-50</sup> This material is a one-component polyurethane which reacts and cures when exposed to moisture. The pigment system contains strontium chromate for corrosion inhibition and extender pigments for gloss control. The coating conforms with MIL-P-85853. The major performance properties required by this specification are corrosion resistance, adhesion, chemical resistance, and flexibility. Most of the requirements in the flexible primer specification are similar to those in the standard primer specification (MIL-P-23377E). However, three requirements for film flexibility are significantly more stringent than those exhibited by the standard primer. A comparison of their flexibility properties is provided in Table 4. Figure 4 is a graph of stress-strain behavior, comparing an epoxy primer, polyurethane topcoat, polysulfide sealant, and polyurethane elastomeric primer.

Application of this coating to numerous Navy aircraft has resulted in fewer coating system failures due to crack-

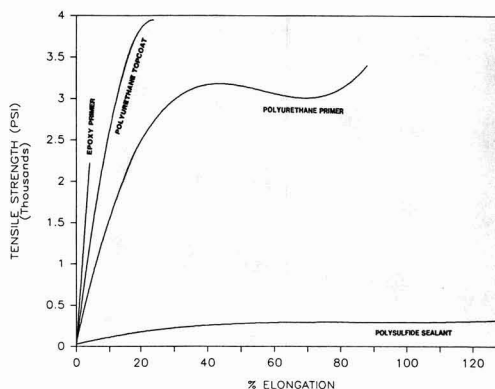


Figure 4—Stress-strain behavior for coatings applied to Navy aircraft

ing and chipping. As stated previously, one of the major coating failure mechanisms on aircraft is cracking around fasteners, thus exposing bare metal. Case histories and field reports on aircraft with the elastomeric urethane primer have indicated a 40% reduction in maintenance man-hours required for corrosion on the E-2C aircraft.

### High Temperature Resistant Coating

Various areas of naval aircraft are routinely subjected to elevated temperatures during operation. The standard paint system was only designed to resist thermal exposures up to 176°C (350°F) for short durations. Therefore, two types of materials are employed for application in these areas: ceramic coatings and high temperature resistant organic coatings. Since ceramic coatings are beyond the scope of this review, they will not be discussed. The current high temperature coating used on Navy aircraft is covered under TT-P-28 and is designed to withstand temperatures up to 650°C (1200°F). This material is a silicone paint with aluminum pigment. It can be applied by conventional air spray and is cured by heating to 204°C (400°F) for 1 hr or upon elevated temperature exposure under component operation. During the curing period, the binder system for this coating will oxidize, leaving a durable barrier layer of silicone oxide/aluminum to protect the underlying substrate from adverse conditions. Although this material provides adequate barrier protection in the high temperature range, the performance diminishes dramatically in the mid-temperature range (260-370°C, 500-700°F) or when damaged.

Recently, a material was developed at this laboratory<sup>51</sup> that withstands exposure to temperatures in the 260-370°C range, while providing superior protection to the underlying substrate. This heat resistant coating consists of a blend of silicone and silicone alkyl copolymer resins with a binary pigment system consisting of leafing aluminum pigment and zinc dust. This pigment system not only provides barrier protection via the leafing aluminum flakes, but also chemical corrosion protection through the sacrificial zinc dust. Figure 5 illustrates corrosion protection provided by this coating on mild steel. The coating

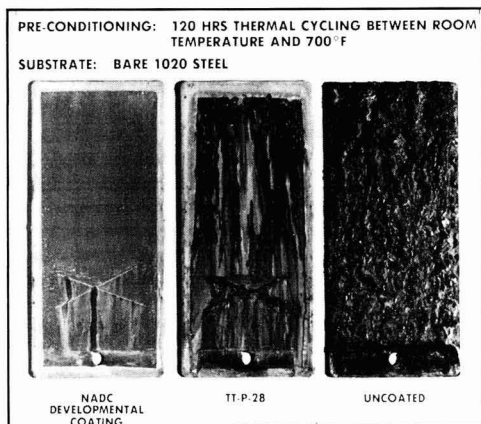


Figure 5—Steel specimens following 500 hr exposure in 5% salt spray

can be applied by conventional air spray and requires very little surface preparation. The coating dries hard at room temperature within 1 hour. Although the dried film has sufficient integrity to be handled without causing damage to the coating, it does not obtain its complete durability and chemical resistance until it attains full cure during service exposure. The cured film has good adhesion and provides corrosion protection to the substrate as indicated in Figure 5. In addition, since this material is used around hot engine components, it is resistant to engine fluids. Finally, the impact flexibility is superior to the TT-P-28 material. The coating is currently being investigated at field activities.

### Temporary Camouflage Paint

The standard camouflage scheme on Navy aircraft was designed as an all-terrain scheme which is generally applicable around the world. Military aircraft often participate in rapidly deployed missions or "war game" exercises that require a theater specific camouflage scheme to match the terrain (i.e., desert, forest, mountain). In these scenarios, a temporary camouflage paint that could be applied over the existing permanent paint system in the field on short notice would be an asset. Of course, the coating would be required to be easily removed after several weeks without damage to the permanent paint system.

Such coatings have been formulated in this laboratory with water-borne acrylic resins (similar to those used in floor waxes) and weather resistant pigments. The resins can be dissolved and removed by alkaline solutions. However, they are resistant to sunlight, rain, and most aircraft fluids. The paints were evaluated for their ease of application and removal, and for residual effects on the underlying coating system. Film properties such as adhesion, flexibility, and durability were determined before and after exposure to heat, humidity, and accelerated weathering. Available proprietary paints using this same technology were also evaluated for comparative purposes.

The test results for the temporary paints have indicated favorable properties for the intended purpose, although resistance to high humidity is limited. Optimum candidates will be service tested on several fleet aircraft in the near future.

### FUTURE TRENDS OF AEROSPACE COATINGS

Research and development of organic coatings for aerospace applications is a continuous, dynamic process which is caused by:

- (1) changes in aircraft design, materials, and performance.
- (2) implementation of stringent legislation on material usage, emissions, and disposal.
- (3) desires to improve individual coating and finishing system performance.

New structural alloys and composites, which have different characteristics than previously used materials, often require the use of coatings to address their specific needs. In many cases, these coatings must exhibit properties different than standard materials. The rapid implementation of strict legislation on the emission and disposal of toxic and hazardous materials may be the most pressing issue on organic coating technology. These rules limit the use of organic solvents, chromates, lead, and other common paint ingredients, thus posing a need for new technology which addresses this void. Finally, as aerospace system performance becomes more demanding, so does the requirement for environmental and detection/weapon protection by coatings.

The approach to improve aircraft finishing system performance is investigation and utilization of high performance polymers and pigments. In addition, there is a trend to replace subjective and qualitative tests with quantitative procedures for analysis of coating performance. Polymer research has mainly been concerned with: (1) moderate enhancement of all mechanical, physical, and chemical properties without detrimental tradeoff effects and (2) significant improvement of specific properties. The first issue is being addressed to obtain potential coating matrix polymers which can be used to improve overall properties of generic high performance coatings (for example, modification of polymer chemistry and morphology for improvement of both flexibility and weather resistance). Normally, polymer tailoring to enhance flexibility counteracts improved chemical and weather resistance. The second issue concerns substantial improvement of a single property for a specific end purpose (for example, modifications causing durability and resistance when exposed to extreme environments such as temperature, acid, alkaline, etc.).

This laboratory is currently involved with investigations of polythioethers, polyurethanes, and toughened epoxies for low VOC flexible primers. The objective of these efforts is to improve coating system flexibility while obtaining adequate adhesion and chemical resistance. Elimination of volatile organics and toxic corrosion inhibitors is also a major requirement. Polymer research and development which has not been initiated but is deemed necessary includes low VOC substitutes for polyvinyl butyral wash primers, epoxy and alkyl silicate

zinc-rich primers, alkyd and acrylic lacquer topcoats, and high temperature binders.

Emphasis on pigment research has centered on non-toxic corrosion inhibitors and pigment-polymer interactions. Since chromates, the primary corrosion inhibitors for aluminum, are suspected carcinogens, a large effort to obtain suitable chromate substitutes has begun.<sup>42,52-55</sup> Molybdates, phosphates, borates, nitrites, and thioglycollates are the primary candidates and, in some instances, they have performed exceptionally well. This laboratory is also concerned with corrosion inhibitor delivery systems such as encapsulated spheres and beads which have a polymer matrix or shell, in which the inhibitors are contained. Using this approach, it may be possible to design corrosion inhibitors and their release to the necessary substrate.

Further research involves molecular interactions between pigment particles and polymer chains. These interactions are believed to be responsible for many of the macroscopic properties of coatings and other particulate filled composites.<sup>56</sup> The approach of this research is to characterize pigment surfaces and polymer chemistry, analyze the type and extent of interactions, and correlate this data with macroscopic properties. By understanding these phenomena, pigment surfaces may be modified to obtain specified pigment and coating properties.

Finally, in order to better understand the performance of organic coatings, new techniques are being devised and utilized. Up until the last 10-20 years, coatings technology has been an art rather than a science. The demand for improved and innovative paints has forced technologists to take a rigorous approach to paint development and analysis. Long-standing methods for characterizing properties such as adhesion and corrosion inhibition were simple, qualitative, and often subjective. Recent advances have been toward exact scientific techniques for mechanical, chemical, and electrochemical behavior. With the advancement of technology, the future of organic coatings will meet the demands of military aircraft.

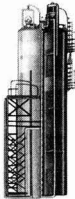
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# Component Interactions and Their Influence On the Uniformity of Coating Films

M. Laleq

Pulp and Paper Research Institute\*  
and

M. Bricault and H.P. Schreiber

Ecole Polytechnique†

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Acid/base characteristics and surface energies have been determined for polymers, blends, and pigmented systems, and patterned after coating formulations. Acid/base interactions appear to generate compositional heterogeneities due to the preferential association of interacting species. Surface heterogeneities caused by thermodynamic demands to minimize surface free energy in coating films also have been proposed. These physicochemical effects provide useful insights for product design and optimization.

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## INTRODUCTION

Polymer coatings are multicomponent, complex systems frequently based on macromolecules which contain polar groupings. In such systems, the existence of specific (i.e., nondispersive) interactions among the components may be taken for granted. It is obvious that these interactions may affect numerous properties of the system, including its rheology, mechanical, and adhesion characteristics. Understanding the nature of specific interactions is therefore an important challenge to coatings scientists and technologists. The purpose of this paper is to contribute to that understanding.

Early routes to the evaluation of interactions among the components of film-forming systems relied heavily on the solubility parameter.<sup>1</sup> The concept as originally introduced has been modified extensively<sup>2,3</sup> to account for

contributions from dispersive and nondispersive sources. More recently, the technique of inverse gas chromatography (IGC) has been used as a very convenient approach to the measurement of interaction thermodynamics in polymer systems.<sup>4-7</sup> The method was first used in situations where only dispersive forces were involved. Subsequently, IGC also has become a vehicle for characterizing nondispersive interactions in polymers. According to Fowkes,<sup>8</sup> nondispersive forces frequently may be considered as acid/base (ab) forces, and he has used the theoretical basis of Drago and coworkers<sup>9,10</sup> to generate ab interaction parameters for various components of polymer systems. Also, in this context, the IGC method has been evolved as a convenient route to the evaluation of such interaction numbers. As a result of these developments, there is promise that the formulator and user of coatings may become more discriminating in optimizing the performance of coatings systems.

In seeking the common goal of numerical characterization for ab interactions, alternative treatments of IGC data may be chosen.<sup>11,12</sup> The one applied here uses vapors known to be acceptors and donors, as probes in the volatile phase, and evaluates the ratio of specific retention volumes ( $V^{\circ}g$ ) as an acid/base interaction parameter ( $\Omega$ ).<sup>12,13</sup> Thus, for acidic solids,

$$\Omega_a = 1 - (V^{\circ}g)_b / (V^{\circ}g)_a < 0 \quad (1)$$

and for basic solids,

$$\Omega_b = (V^{\circ}g)_b / (V^{\circ}g)_a - 1 > 0 \quad (2)$$

The parameter is a comparative index of acidity or basicity. Different absolute values will be obtained for various choices of acid or base vapor probes, and it is important, therefore, to make comparisons with this restriction in mind.

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\*570 St. John's Rd., Pte. Claire, Quebec, Canada H3C 3A7.

†Département de Génie Chimique, Campus de L'Université de Montréal, 2900, boul. Édouard-Montpetit, Montréal, Quebec, Canada.

**Table 1—Specific Retentions and Interaction Parameters for Starting Polymers**

T(°C)	nC <sub>8</sub>	V <sup>g</sup> for		C <sub>4</sub> (OH)	C <sub>4</sub> (NH <sub>2</sub> )	Ω
		nC <sub>6</sub>				
Solid I						
30.....	345	310	581	830	—0.42	
40.....	293	227	363	602	—0.65	
50.....	202	104	188	374	—0.99	
60.....	167	115	126	236	—0.87	
70.....	107	73.9	76.5	152	—0.99	
80.....	85.9	61.4	50.4	99.8	—0.98	
90.....	60.3	44.8	25.7	51.6	—1.00	
100.....	47.2	30.6	18.6	38.1	—1.05	
110.....	38.1	23.5	13.0	23.6	—0.70	
Solid II						
30.....	330	255	438	1271	—1.36	
40.....	281	205	356	814	—1.29	
50.....	206	158	216	560	—1.59	
60.....	163	116	147	436	—1.97	
70.....	108	73.0	91.7	322	—2.51	
80.....	77.1	51.5	70.2	230	—2.28	
90.....	51.4	40.7	45.2	151	—2.34	
100.....	40.3	31.4	39.3	126	—2.21	
110.....	32.7	27.8	30.8	91.1	—1.96	
Solid III						
30.....	455	405	1122	680	0.65	
40.....	392	316	750	456	0.64	
50.....	294	241	415	235	0.77	
60.....	119	146	319	176	0.81	
70.....	160	81.6	174	98.6	0.76	
80.....	111	53.5	126	68.9	0.83	
90.....	75.5	40.9	84.1	46.1	0.82	
100.....	50.2	33.3	61.3	33.5	0.83	
110.....	30.7	24.4	41.6	26.7	0.56	

Various uses may be made of IGC data, in addition to calculations of  $\Omega$ . Since the basis of IGC is the thermodynamic drive for partitioning the vapor phase between the carrier gas and the solid stationary phase,<sup>4,14</sup> it can be shown that the heats of interaction between stationary and vapor phases ( $\Delta H_i$ ) are given by

$$\Delta H_i = \partial \ln V^g / \partial 1/T \quad (3)$$

This relationship has been used in the present work to help characterize the surface interaction tendencies of components in film-forming systems. Additional surface characterization has been obtained from contact angle measurements, and in combination, these data have been used to draw conclusions on the state of homogeneity in the test systems.

## EXPERIMENTAL

### Materials

Three polymeric vehicles have been selected to illustrate the concepts advanced in this work. All are commercial products prepared by proprietary processes and used as supplied. They are:

THIXOTROPIC ALKYD, hereafter coded (I): This is a linseed oil-based binder with mono- and diglyceride esters in a ratio of 3:2, and an Mw ~ 2,000. The resin is modified by being covalently bonded with a diurea additive, present at 12 wt %.

ALKYD 2 (II): Composition same as in I, but lacking the diurea additive.

MELAMINE (III): Setamine™ 141 is an isobutanol type resin.

The polymers were borne in 1:1 volume mixtures of xylene and butyl glycol acetate. They were studied singly and in the following combinations: I/II (1:1), I/III (1:1), II/III (1:1), and I/II/III (1:0.25:0.4). The three-component mix was patterned after industrial practices. The latter three compositions examine the surface states of alkyds combined with their melamine crosslinker moiety. The combination I/II is intended to test the effect of a sag-control agent on otherwise identical resin formulations.

The polymer vehicles were studied as clearcoats and the system I/II/III also was examined in filled versions using the following: rutile TiO<sub>2</sub>, 8.2 m<sup>2</sup>/g surface area; iron oxide (Fe<sub>2</sub>O<sub>3</sub>), 3.5 m<sup>2</sup>/g surface area; and glass spheres, 0.08 mm in diameter. In all cases, filled polymer films contained 10 wt % particulates.

### Inverse Gas Chromatography

All polymers, polymer mixtures, and filled systems were deposited onto screened (60/100 mesh) Chromosorb W support from solutions containing about 2% polymer solids. Following solvent evaporation under vacuum in a Rotovap, the coated support was baked at 130°C/30 min prior to packing into stainless steel columns, which were previously degreased, washed, and dried. Standard washing procedures<sup>5</sup> were used to ensure that the supported polymer phase accounted from 8-12% of the total weight of column packing.

Different approaches were required for the pure particulates. Glass beads were used as received and packed into columns 25 cm long, containing 4-6 g of material. Rutile and iron oxides were tumble-blended with the glass until visual and microscopic inspection showed that thin, contiguous films of these pigments had formed on the glass substrates. The coated beads were then packed into similar steel columns. As in the previous example, solids were conditioned at 130°C/30 min prior to column packing.

Columns were placed in a Perkin-Elmer Sigma-2 chromatograph, equipped with hot wire detectors. Helium (He) gas was used as a carrier at flow rates of 12-15 (± 0.1) ml/min. Retention volumes were obtained by injecting minute quantities of vapor via 1-μl Hamilton microsyringes. The vapors were selected to represent donor and acceptor properties, as well as materials able to interact through dispersive forces alone. Used were: acids (Drago designation)—n-butanol (C<sub>4</sub>OH) and chloroform (CHL); bases—n-butyl amine (C<sub>4</sub>NH<sub>2</sub>) and dibutyl ether (DBE); and dispersive-force vapors—n-hexane and n-octane, (nC<sub>6</sub> and nC<sub>8</sub>).

At least triplicate injections were made in all cases, with column temperatures ranging from 30 to 110°C. At each experimental temperature, each column was swept with He for at least 30 min prior to vapor injection. Data reproducibility throughout was better than ± 4%. In cases

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where nonsymmetrical elution peaks were generated, the protocol of identifying retention times was that described in references (12) and (13).

### Surface Energetics

A Rame-Hart A-100 goniometer was used to measure contact angles on samples of single polymers and unfilled polymer mixtures supported on glass microscope slides. Film thicknesses were uniformly near 0.002 cm. Prior to data accumulation, all film samples were given the same thermal conditioning as in the IGC sequence. In addition to values of  $\gamma_s$ , the procedure of Schultz and coworkers<sup>15,16</sup> was applied for computations of the polar contribution ( $\gamma_p^d$ ), to the overall surface energy. Fluids involved in the work included hexane, octane and cyclohexane, water, glycerol, and formamide. An uncertainty of approximately  $\pm 8\%$  is associated with surface energy values quoted in succeeding sections of this paper.

## RESULTS AND DISCUSSION

### Single Polymers

Retention volume data for the three vehicle polymers are illustrated in Table 1. For simplicity, the  $V_g^0$  data are restricted to the nonpolar probes and to the strong acid/base pair of vapors,  $C_4OH$ , and  $C_4NH_2$ . Table 1 also contains values of  $\Omega$  calculated from the alcohol and amine retention data following equations (1) and (2). Characteristic differences are immediately evident among the three polymers. I and II are strongly acidic, alkyd II more so than I. In contrast, the melamine III registers as a base. In all cases,  $\Omega$  is temperature dependent and the variations follow similar trends. Inherent acidity or basicity at first increases with rising temperature, but subsequently begins to decrease. Very similar behavior has been found in simpler macromolecules.<sup>17</sup>

The trends previously discussed seem to be reasonable; polar probe molecules will be retained by the polymer surface through a combination of dispersive and nondispersive forces. Of these, the weaker dispersive forces will tend to dominate when polymer and probe are both donors or both acceptors, while stronger but shorter-range acid/base forces will tend to predominate in donor/acceptor contacts. These tendencies should become more pronounced with rising temperature, as long as the thermal energy needed to desorb molecules held by acid/base

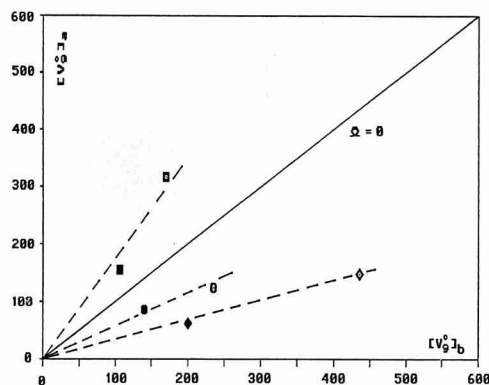


Figure 1—Relationship between retention volumes for selected acid/base probes on polymer surfaces.  $T = 60^\circ\text{C}$ . Solid I:  $\square$  —  $C_4(OH)/C_4(NH_2)$ ;  $\blacksquare$  — CHL/DBE. Solid II:  $\diamond$  —  $C_4(OH)/C_4(NH_2)$ ;  $\blacklozenge$  — CHL/DBE. Solid III:  $\square$  —  $C_4(OH)/C_4(NH_2)$ ;  $\blacksquare$  — CHL/DBE.

forces is not exceeded. However, given that the short-range forces are thermolabile, it follows that at sufficiently high temperatures all substrate-vapor interactions will reduce to dispersive forces. As a result, all  $\Omega$  values should tend to zero. The temperatures where maxima occur and where  $\Omega = 0$  should be specific to substrate/vapor pairs. In the present cases, I attains maximum acidity near  $90^\circ\text{C}$ ; II in the same temperature interval; while the melamine attains maximum basicity in the broad temperature range of  $60$ – $100^\circ\text{C}$ . It follows from Table 1 that I/III and II/III combinations should be miscible, driven by acid/base interactions. The diurea moiety in I reduces the degree of acidity relative to compound II. Clearly, these materials are expected to be miscible and the purpose of studying their mixture is to specify the effect of the additive on surface and interaction characteristics. A point of practical concern is the obvious difference in apparent miscibilities of system components at processing temperatures (e.g.,  $> 100^\circ\text{C}$ ) and at much lower use-temperatures. In general, an influence on system properties may be inferred from this and has indeed been documented for various multicomponent systems.<sup>17,18</sup>

As already noted, the quoted  $\Omega$  values are valid only for the chosen pair of acid/base vapors. In principle, of course, the same value should be obtained for any combination of vapors with similar donor and acceptor potentials (as given by Drago's Ea, Ca, Eb, and Cb indexes),<sup>9-10</sup> provided the molecular dimensions in these pairs are not too dissimilar. The concept is illustrated by Figure 1. Under isothermal conditions (e.g.,  $60^\circ\text{C}$  as in Figure 1), a solid able to interact through dispersion forces only (e.g., a polyolefin) should generate  $V_g^0$  values for any suitable combination of donor/acceptor probe molecules such that the points fall on the solid line designating  $\Omega = 0$ . Similarly, a solid with donor or acceptor capabilities should generate a line of constant, nonzero  $\Omega$ , if the choice of probes has been consistent with at least the previously stated restrictions. In the present work, the pair CHL/DBE was chosen as a weaker acid/base counterpart to the

Table 2—Acid/Base Force Contributions to  $\Delta H_i$  in Polymer/Vapor Pairs

Solid	I	II	III
Vapor	( $-\Delta H^{\text{ab}}$ in KJ/mol)		
$nC_6$ .....	0 (20.5) <sup>a</sup>	0 (24.0) <sup>a</sup>	0 (16.3) <sup>a</sup>
$C_4(OH)$ .....	1.9	0.5	13.7
$CHCl_3$ .....	2.5	3.0	8.8
$C_4NH_2$ .....	17.2	20.6	-1.5
DBE .....	11.0	17.5	2.8

(a)- $\Delta H_f^\circ$  (in KJ mol<sup>-1</sup>).

**Table 3—Retention Volumes and Interaction Parameters for Polymer Combinations**

Composition	Temp(°C) = V°g for				$\Omega$ Exp.	$\Omega$ Calc.
	nC <sub>6</sub>	C <sub>4</sub> (OH)	C <sub>4</sub> (NH <sub>2</sub> )			
I/II(1:1)	30.....	245	284	446	-0.57	-0.89
	50.....	144	163	355	-1.18	-1.29
	70.....	81.3	107	257	-1.40	-1.75
	90.....	59.0	73.0	173	-1.37	-1.67
	110.....	30.3	51.5	115	-1.24	-1.33
I + III(1:1)	30.....	149	111	103	0.08	+0.11
	50.....	127	73.5	76.4	-0.04	-0.11
	70.....	84.1	55.9	67.1	-0.20	-0.12
	90.....	53.7	40.6	47.1	-0.16	-0.09
	110.....	22.5	27.7	31.3	-0.13	-0.07
II/III(1:1)	30.....	149	672	907	-0.71	-0.35
	50.....	188	411	580	-0.83	-0.41
	70.....	129	117	325	-1.76	-1.20
	90.....	91.6	85.4	186	-1.52	-1.18
	110.....	40.2	37.5	83.3	-1.40	-1.22
I/II/III (1:0.25:0.4)	30.....	74.2	148	275	-0.86	-0.31
	50.....	55.3	73.5	173	-1.35	-0.67
	70.....	22.9	37.2	93.5	-1.51	-0.82
	90.....	18.6	29.8	78.0	-1.61	-1.26
	110.....	9.0	10.7	18.4	-0.72	-0.72

alcohol/amine combination, and the relevant V°g values have been entered in Figure 1. Evidently, the two pairs selected do sense very similar interaction balances with each of the three polymers; it is obvious by inspection, however, that other probe combinations (e.g., CHL/amine or alcohol/DBE) would lead to very different  $\Omega$  values.

The available data may be used to estimate the contributions made by acid/base forces to overall heats of solid/probe interactions. Following Fowkes,<sup>8,19</sup> we note that

$$\Delta H_i = \Delta H_i^d + \Delta H_i^{ab} \quad (4)$$

Assuming that the hexane probe is dimensionally consistent with the butyl alcohol and amine, then  $\Delta H_i^d$  may be obtained from appropriate slopes of  $\ln V^\circ g$  vs  $1/T$ , as called for in equation (3).  $\Delta H_i^{ab}$  can then be estimated by subtraction, using overall interaction heats obtained from the slopes of plots involving the polar probe molecules.

The relationship between  $\ln V^\circ g$  and  $1/T$  is shown for solid I in Figure 2. Reasonably good straight lines are noted, and these are typical of results also obtained for solids II and III, and for CHL and DBE probes. The resulting enthalpies are entered in Table 2. Numerically, the values are very similar to those reported by Fowkes<sup>19</sup> for acid/base enthalpies involving acrylic and vinyl poly-

mers. The inherent acidity of I and II is reflected by strong enthalpic contributions when basic probes are used. The more energetic interaction between these solids and the amine, as compared with the DBE, is quite pronounced. Interestingly, the  $\Delta H_i^{ab}$  contribution arising from CHL is greater than from the alcohol probe, suggesting that acid/acid repulsion limits the distance of approach of the alcohol molecule to the extent that dispersion forces become dominant. The basic solid III produces analogous results, with the acid probes now giving appreciable enthalpic effects, and very weak contributions stemming from the use of basic probes. In this case, the amine probe appears to be more strongly repelled from the substrate than the weaker base DBE.

### Polymer Mixtures

Table 3 presents an overview of retention data for the polymer combinations. Two sets of  $\Omega$  are given; in addition to the experimental datum a calculation has been made based on the assumption that in a miscible, randomly dispersed blend, the surface composition would be determined by the respective weight fractions,  $w$ , of components according to

$$\Omega_j = w_1\Omega_1 + w_2\Omega_2 + \dots + w_j\Omega_j \quad (5)$$

There is no clearcut theoretical justification for the previously stated convention. It has been chosen on grounds of convenience, but more complex combinatorial formulas may be more realistic and volume, rather than weight fractions, may turn out to be more advisable.

The aforementioned uncertainties notwithstanding, the results are consistent with the inferences drawn from the inherent donor, acceptor characteristics of the polymers. A point of particular interest is the lack of agreement between calculated and observed  $\Omega$  values for I/II mixtures. Here the experimental values are consistently higher than those calculated on the basis of simple combinatorial rules. The reduced acidity of I, due to its diurea moiety, seems to result in a form of "immiscibility," driven by the need to minimize the surface energy of the compound. That is, the surface composition of the mixture may reflect differences between the surface energetics of I and II, the respective  $\Omega$  values suggesting that I should be preferentially surface localized. The suggestion of an excess surface concentration of I in these mixtures is to be raised again later in the paper. In contrast, the agreement between sets of  $\Omega$  for mixtures of I/III is good. Speculatively, the miscibility arising from favorable ab interactions has produced polymer surface compositions

**Table 4—Surface Energy Parameters for Starting Polymers and Polymer Combinations**

T(°C)	30		50		70		90	
$\gamma$ (in J m <sup>-2</sup> )	$\gamma_s$	$\gamma_s^p$	$\gamma_s$	$\gamma_s^p$	$\gamma_s$	$\gamma_s^p$	$\gamma_s$	$\gamma_s^p$
I.....	42.6	14.3	41.7	13.9	41.4	13.0	40.5	10.6
II.....	47.7	16.8	47.0	16.3	46.7	16.0	45.5	12.8
III.....	38.8	9.5	38.3	9.3	38.0	9.0	37.4	8.3
I/II(1:1).....	43.3	14.4	43.9	14.1	42.7	13.6	41.7	11.7
I/III(1:1).....	40.5	9.1	40.0	8.7	39.3	8.3	38.3	7.9
II/III(1:1).....	40.5	9.4	40.1	10.2	39.7	9.9	39.2	8.8
I/II/III(1:0.25:0.04).....	43.8	14.6	43.4	14.0	42.8	12.9	41.6	11.5



**Table 5—Acid/Base Parameters for Filled Polymer Systems**

T(°C)	30	50	90
<b><math>\Omega</math> for:</b>			
I/II/III Vehicle.....	-0.86	-1.35	-1.61
TiO <sub>2</sub> .....	1.26	1.40	1.87
Fe <sub>2</sub> O <sub>3</sub> .....	-0.43	-0.46	-0.54
Glass.....	0.07	0.04	0.06
<b>Vehicle + Glass</b>			
Calc.....	-0.79	-1.31	-1.48
Exp.....	-0.82	-1.36	-1.50
<b>Vehicle + TiO<sub>2</sub></b>			
Calc.....	-0.65	-1.08	-1.26
Exp.....	-0.41	-0.72	-0.99
<b>Vehicle + Fe<sub>2</sub>O<sub>3</sub></b>			
Calc.....	-0.80	-1.27	-1.50
Exp.....	-1.16	-1.44	-1.64

that are consistent with the stoichiometry of the system. In II/III mixtures, ab interactions again will favor compositional uniformity, yet the calculated and experimental  $\Omega$  values differ somewhat, particularly at lower temperatures. (As implied already, we would expect discrepancies among experimental and calculated  $\Omega$  values to diminish as the temperature rises into regions which reduce the importance of specific interactions). Compositional heterogeneity again may be inferred. Evidently, ab interaction may be a desirable but insufficient driving force for the formation of isotropic polymer films. Again, surface energetics will be examined to help clarify the issue. Finally, compositional anisotropy is strongly inferred for the system I/II/III, the surface of which is excessively acidic. The experimental  $\Omega$  values here suggest a surface excess of polymer II.

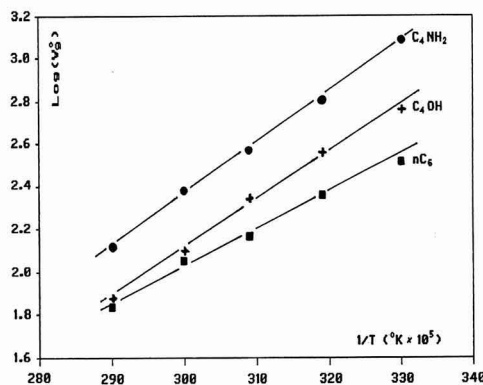
The inferences of anisotropy arising from the IGC results may be examined more closely through surface energy evaluations. These are reported in Table 4. It is at once evident that higher surface energies are produced in II compared with I, with  $\gamma^p$ s also consistently higher in II, indicating more pronounced surface polarity. The lowest surface energies are produced in solid III. Since the combination of I/II generates  $\gamma$  values that are much closer to those of I than of II, we conclude that the requirement to minimize surface free energy leads to surface enrichment in I. The entire film will, therefore, be compositionally heterogeneous. In I/III, the  $\gamma$ s values are essentially as expected from the composition, but the  $\gamma^p$ s are lower than either I or III. This might be accounted for if I and III were coupled through acid/base interactions, with the responsible moieties oriented into the bulk of the film, leaving the surface enriched in nonpolar structures inherent in these polymers. Surface heterogeneity caused by orientational degrees of freedom in macromolecules has been reported recently.<sup>13</sup> In II/III combinations, the situation is similar, but the inherently high  $\gamma^p$ s values for II are reflected here, the  $\gamma^p$ s for the mixture being somewhat higher than in the I/III case. It seems, therefore, that surface energetics combine with acid/base interactions to produce distinct types of heterogeneity in the present coating films. Immiscibility appears to result in local compositional heterogeneity, while, in miscible pairs, the heterogeneity is surface localized due to chain dynamics

effects. The data for the complex three-component system do not point decisively either to compositional or to conformational anisotropies, and, therefore, do not add greatly to the inferences drawn from  $\Omega$  information. This may be due to the very different scales of surface probing represented by the two methods. The surface tension method probes surface areas given by the dimension of droplets used in the determinations; the IGC method probes surfaces on a molecular scale. Further work will be needed to resolve questions posed by these differences where multicomponent systems are involved.

### Filled Polymers

Interaction parameters for vehicle I/II/III filled with glass, rutile titanium dioxide, or iron oxide solids, are presented in Table 5, along with  $\Omega$  data for the three fillers. For brevity, the tabulation is limited to three temperatures. In the group of fillers, the nonagglomerated rutile has been surface-modified by the manufacturer, the result being a pigment of appreciable surface basicity. In contrast, the iron pigment reports surface acidity. The glass is a neutral surface; moreover,  $V^g$  values were very low, typically ranging from 0.8 to 3.0 mL/g. Pristine glass is known to be a very active surface and we assumed that the passive surfaces in these specimens were due to the sorption of atmospheric gases which had not been removed during the imposed thermal conditioning. The three fillers, therefore, represented wide variations in potential interactions with components of the polymer blend. As before, two sets of  $\Omega$  were entered in Table 5; the experimental and the calculated data based on the assumptions relating to equation (5).

The two sets of values are in excellent agreement for glass-filled polymers, but in considerable disagreement for rutile- and iron oxide-filled materials. The neutral, surface-passivated glass does not interfere with the contribution made by the other components of the system to its surface interaction capacity. There is then no evidence for any preferential association between the glass and other components of system I/II/III. The surface interaction potential in rutile-filled films is more basic than



**Figure 2—Temperature variation of retention volumes for polymer I**

would be expected from the composition, while films filled with iron oxide are more acidic than expected. It is suggested that in these materials, preferential associations within the pigmented mass do take place. Such associations, driven by acid/base interactions, have been proposed on other occasions.<sup>13,19,20</sup> Here, the more highly acidic polymer II may be preferentially sorbed by the rutile, while the iron pigment would preferentially sorb the basic melamine III. Each of these events would affect the local composition near pigment particles, and, assuming the solids to be uniformly dispersed, throughout the bulk of the polymer. The results in Table 5 are consistent with these hypotheses, if the final proviso is made that the pigment concentration at and near the polymer surface is somewhat lower than in the bulk of the sample. Given the need to minimize surface free energies and the higher density of pigment particles, this proviso seems to be consistent with thermodynamic, as well as with sedimentation principles.

## CONCLUSIONS

The IGC method, combined with surface energy data, has been shown to be useful for measuring surface interaction characteristics of some film-forming alkyd polymers, their blends, and of a filled polymer system, modeled after coating formulations.

Acid/base interactions have been measured for combinations of alkyd/melamine vehicles and for a mixture of two identical alkyds, one containing a urea modifier. Evidence has been given that acid/base concepts are implicated in miscibility/immiscibility considerations in these multicomponent polymers.

Acid/base interactions contribute to compositional heterogeneity in the polymer blends studied. Surface heterogeneity, driven by the conformational flexibility of macromolecules and by the need to minimize surface free energy, is another form of anisotropy present in the polymer films examined in this work.

Acid/base forces have been cited as sources of apparent compositional heterogeneity in two of the three filled, multicomponent polymer systems of this research. It appears that acid/base and surface energy sources may generate compositional heterogeneity in many complex,

film-forming polymer systems used in the protective coatings field.

## ACKNOWLEDGMENTS

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# Chemical and Thermal Stability of Phosphate Layers on Cold-Rolled And Electrogalvanized Steels

W.J. van Ooij<sup>†</sup> and A. Sabata  
Colorado School of Mines\*

The chemical stability of modern phosphate coatings on two types of automotive steels has been studied, namely cold-rolled steel (CRS) and electrogalvanized steel (EG). Unpainted spray- and dip-phosphated panels were immersed in solutions of different pH for 30 minutes and the changes in the phosphate crystals were studied by SEM, EDX, and XRD. Some of the objectives of this study were to determine the effects of the presence of phosphophyllite or of Ni and Mn in the coatings and of a chromium postrinse on the acid or alkali resistance of the crystals. The effect of baking on the phosphate coating and on its

stability was also studied. Best results were obtained with the dip-phosphated and Mn-containing phosphate coatings. No effect of the chromate postrinse was observed for any of the systems studied. The behavior of the phosphate at low and high pH is dissimilar and the reaction products formed are different for coatings with and without phosphophyllite. The effects of thermal treatments and subsequent exposure to water have also been studied. The behavior of the coatings on the two substrates was different.

## INTRODUCTION

The increased use of precoated steels in automotive bodies has prompted several studies of the cosmetic corrosion mechanism of such steels in recent years.<sup>1-5</sup> The phosphating process prior to the application of the paint film is generally assumed to be an important factor in assuring optimum performance of the painted steel in tests for the evaluation of cosmetic corrosion resistance of the entire system.<sup>6</sup> Depending on the conditions of the test, corrosion under a paint film is associated with either a low or high pH. Phosphate coatings on cold-rolled steel (CRS) have been extensively studied in terms of their alkali resistance because corrosion propagation here involves a very high pH in many test conditions.<sup>7</sup> The alkali resistance of a phosphate coating is also important in the cathodic electrocoating process which is also associated with a high pH. Phosphate coatings on modern zinc-coated steels have been shown to be less alkali resistant.

This is attributed to the absence of phosphophyllite which can only be formed on CRS and which has been demonstrated to be more alkali resistant than hopeite.<sup>8,9</sup> Therefore, recent trends in the improvement of phosphate coatings on CRS have been aimed at an increase of its phosphophyllite content and for zinc-coated steels at the development of coatings containing other alkali-resistant phosphates such as those of nickel or manganese.

Detailed studies of the rate and mechanism of the attack of phosphate crystals on CRS in aqueous solutions of various pH values have been published,<sup>8,10-12</sup> but very little has been reported so far for modern phosphate coatings on zinc-coated steels. Wyvill and Miyoshi, et al., both reported a higher rate of dissolution for hopeite than for phosphophyllite in a solution of pH 13,<sup>9,13</sup> although it was shown by Maeda that very stable hopeite coatings can be obtained if they have the appropriate crystallographic orientation.<sup>14</sup>

A comparison of the mechanisms of attack of phosphophyllite and hopeite at high and low pH was recently published by Servais, et al.<sup>11</sup> Some of their findings were

\*Dept. of Chemistry and Geochemistry, Golden, CO 80401.

<sup>†</sup>Dr. van Ooij's new address is: Armco R&T, 703 Curtis St., Middletown, OH 45043.

that during paint baking partial dehydration of both types of phosphates occurred. The phosphophyllite became completely amorphous, whereas the hopeite remained crystalline but now contained two water molecules rather than four. The phosphophyllite crystals did not rehydrate in subsequent contact with water, whereas hopeite did. Another result was that the mechanism of attack was different at high and low pH. The authors used this finding to estimate the pH in certain conditions of corrosion testing.

In this paper, we report on the pH stability of modern phosphate coatings on CRS and EG. In particular, we wanted to compare the alkali stability of hopeite and phosphophyllite and the effects of the presence of nickel and manganese in hopeite coatings. The type of process, i.e., dip or spray, the effect of baking at 180°C, and the effect of a chromium postrinse on the pH stability have also been studied. The information in the literature on the mechanism by which such a postrinse improves the corrosion performance of the painted system is somewhat conflicting. Two mechanisms have been proposed, i.e., a pore blocking mechanism<sup>15</sup> and a stabilization against high pH attack as a result of the deposition of a thin zinc chromate film on the phosphate surface.<sup>10,16</sup>

To simulate the effects of paint baking on the phosphate crystals, we studied the effects of a thermal treatment at 180°C and also the effect of subsequent rehydration conditions on the crystal structure of the phosphates on CRS and EG.

## EXPERIMENTAL

### Phosphated Panels

Test panels were obtained from Parker/Amchem. The CRS panels were dip and spray phosphated in Granodine 958 and Granodine 952, respectively. Both are modern nickel- and manganese-containing low-zinc phosphate processes. The coating weights were 2.50 and 2.00 g/m<sup>2</sup>, respectively. These were compared with the older system Granodine 902, a spray phosphate system of about 1.39 g/m<sup>2</sup> which contained nickel but no manganese. All panels were obtained with and without the standard chromium rinse (Deoxyllyte 41) which is part of the system. The phosphophyllite contents of the coatings were specified by the supplier as 80% for the dip-phosphate system and 60% for the two spray systems. The EG panels were phosphated in the same processes. Their coating weights were 2.22 g/m<sup>2</sup> for Granodine 902, 2.78-3.05 g/m<sup>2</sup> for

Granodine 958, and 2.00 g/m<sup>2</sup> for Granodine 952. Here, too, panels were obtained with and without the standard chromium rinse. All panels were studied in the as-received condition.

### Test Conditions

Heating of the panels was done in air in a laboratory oven. Rehydration of the baked panels was carried out by immersion in demineralized water of pH 7. The effect of the pH on the phosphate was studied by immersion of 1.5 × 1.5 cm<sup>2</sup> samples in solutions of HCl or NaOH. The cut edges of the samples were not coated or sealed in these experiments. All samples were of the same thickness, i.e., 0.76 mm. Changes in pH during phosphate attack were adjusted. The temperature of the solutions was 25°C. In some experiments, the test solutions were stirred moderately in the same way for all samples. Following immersion, the samples were rinsed with water and blown dry in air.

### Phosphate Characterization

The morphology and composition were determined by SEM/EDX using a JEOL JX 840 scanning electron microscope with a Tracor Northern EDX system TN-5500. All analyses were done with a 15 kV primary beam voltage so as to minimize the contribution of the metal below the phosphate in the EDX spectra. Quantification was done using the standard software of the X-ray analyzer. All results are presented as weight-percent. The crystallographic structure of the phosphate coatings was determined in a Rigaku-200 system using Cu-K $\alpha$  radiation. The experimental conditions were 40 kV, 70 mA, and 1°/min scan rate with a time constant of 1.

## RESULTS

### Phosphate Characterization

Figures 1-6 show the morphologies of the six different phosphate coatings. The chromium rinse had no effect on the crystal morphology for any of these systems. The Mn-containing phosphate crystals are considerably smaller than those without Mn for both CRS and EG. Further, the spray phosphated Mn-containing systems have somewhat smaller crystals than the corresponding dip phosphates and all three-cation coatings are somewhat more granular than the well-known needle-like structure obtained without Mn, in agreement with literature.<sup>17</sup> The analysis results, as per EDX of the phosphated panels, are given in Table 1. Ni is detected in all materials. It is highest in the dip-phosphated EG system. The others have approximately the same content. Mn is detected in Granodine 958 and 952 and is higher in the former (dip) than the latter (spray) system. The amounts of Mn detected in CRS and EG are about equal for each process. Chromium could not be detected in any of the coatings. It appears from the data in Table 1 that part of the Fe signal in the CRS systems and part of the Zn signal in EG systems stems from the metallic substrate. However, the Zn/P ratios obtained from the CRS systems allow us to estimate

Table 1—EDX Analysis of the As-Received Panels (wt%)\*

Material	Fe	Zn	P	Ni	Mn
CRS 902 .....	59.11	29.91	9.97	0.99	0
CRS 952 .....	73.56	17.78	5.63	0.10	2.00
CRS 958 .....	42.12	36.28	15.16	1.35	4.85
EG 902 .....	0	84.10	14.41	1.00	0
EG 952 .....	0	82.28	13.39	0.91	2.90
EG 958 .....	0	78.44	15.41	1.88	3.95

(a) Average of three values taken at 15 kV and 100X magnification.



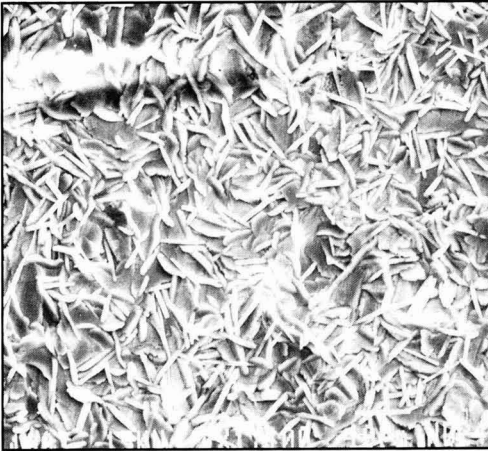


Figure 1—Secondary electron (s.e.) micrograph of as-received CRS 902 phosphate system

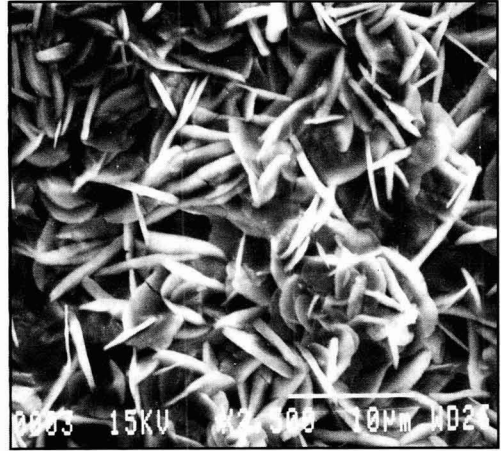


Figure 4—S.e. image of EG 902

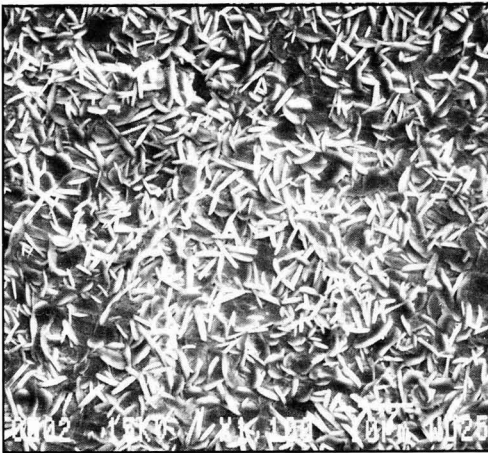


Figure 2—S.e. image of CRS 952

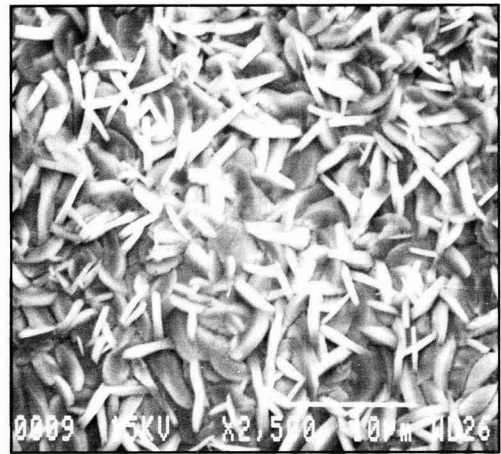


Figure 5—S.e. image of EG 952

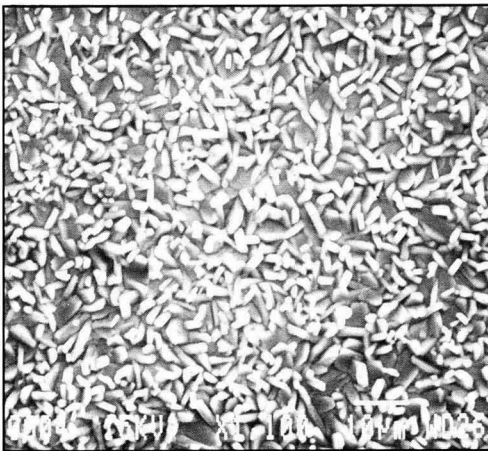


Figure 3—S.e. image of CRS 958

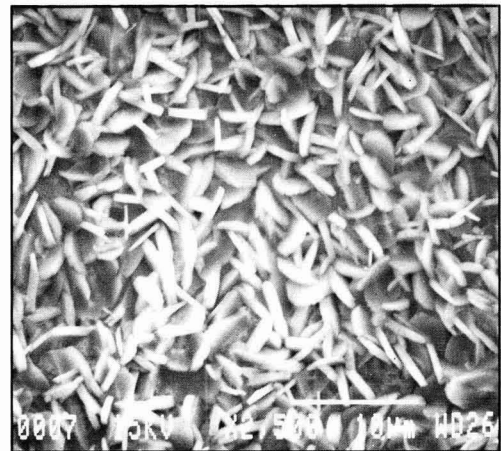


Figure 6—S.e. image of EG 958

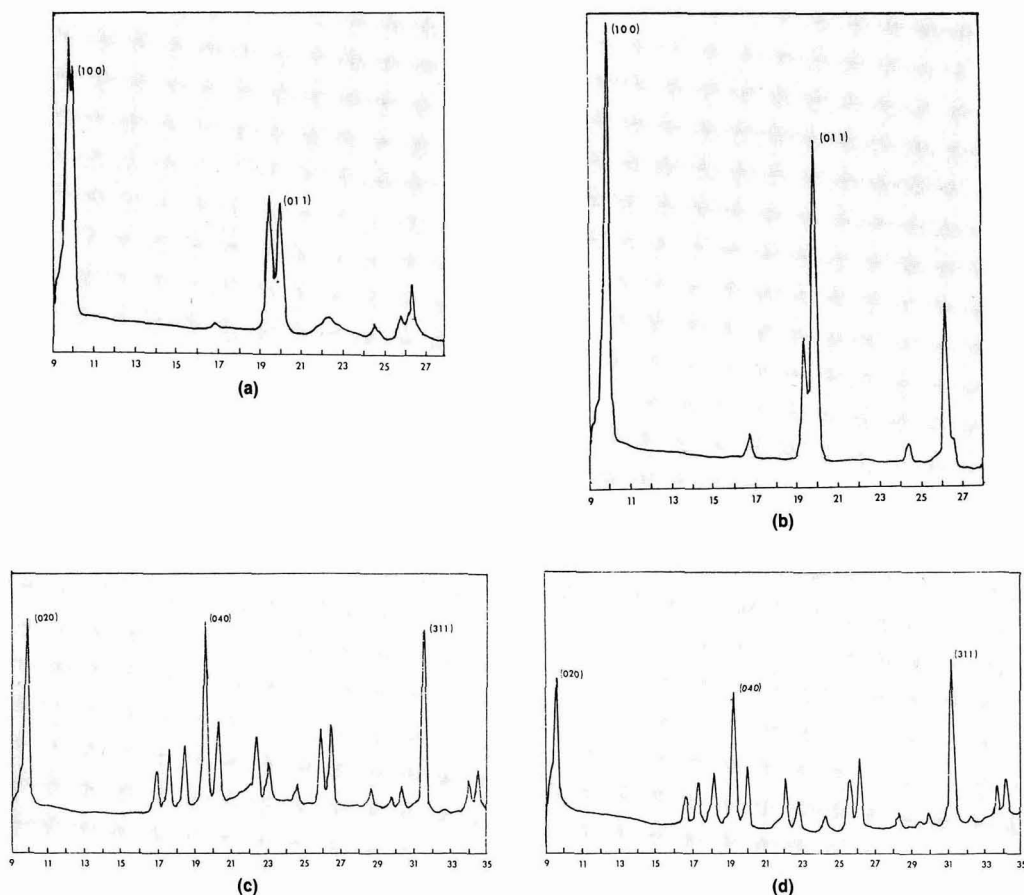


Figure 7—X-ray diffractograms of phosphate crystals on CRS 902 (a), CRS 958 (b), EG 902 (c), and EG 958 (d)

the phosphophyllite content on the CRS panels. This is based on a 1/1 Zn/P ratio in phosphophyllite and a 3/2 ratio in hopeite, so any mixture of the two phosphates should give a Zn/P ratio between 1.0 and 1.5. If we apply a correction for the amounts of Ni and Mn, which are believed to be in the same mixed phosphate forms as phosphophyllite,<sup>17</sup> we find 70-75% phosphophyllite in Granodine 958, 60-70% in Granodine 952, and 15-20% in Granodine 902. These values are lower than those reported by the supplier which were obtained by using the standard P-ratio method. This method is unreliable because it does not consider the effects of texture and structure factor calculations. EDX, on the other hand, appears to be a rapid and simple method for the estimation of the phosphophyllite contents of phosphates on CRS.

The X-ray diffractograms of the phosphates are given in Figure 7 which shows hopeite on EG spray and dip systems and two different mixtures of hopeite and phosphophyllite on CRS. All peaks in this figure stem from

these phosphates since diffraction peaks from Fe and Zn do not appear below an angle  $2\theta$  of  $35^\circ$ . All peaks are easily identified and annotated using the standard powder diffraction files. The diffractograms shown here are more intense and of higher resolution than those published in literature.<sup>11</sup> The difference with respect to other, high quality diffractograms of hopeite<sup>6</sup> must be attributed to different crystal orientations (texture) in our systems. Such orientations have been related to a texture in the zinc coating.<sup>18,19</sup> The intensity of the (020) peak in hopeite was shown to increase with the (002) plane intensity of the zinc layer, which is a basal plane orientation. In Figure 8a, the sections of the diffractograms containing the peaks of the metal substrates are shown. It is seen that the zinc substrate in the older system Granodine 902 has a strong (101) orientation, whereas the zinc substrate in the newer systems Granodine 958 and 952 has a strong (002) preferred orientation. The hopeite crystals on zinc substrates with higher basal plane orientation in various samples of 958 and 952 were found to have a stronger (020) plane orientation.

In Figure 8b, the diffraction peaks of the steel of the old and the newer phosphate systems are shown. Here, the metal substrate of the Granodine 902 shows a preferred (110) orientation. Although it is not clear what the significance of this observation is, it is seen that the hopeite mixed with the phosphophyllite in both types of CRS samples (Figure 7) shows fewer hopeite diffraction

peaks than the hopeite on EG, which suggests a different orientation of the hopeite on EG than on CRS.

A remarkable observation is that the intensity of the diffraction peaks from the Ni- and Mn-containing systems 952 and 958 is lower than from the older 902 system, despite the higher coating weights. This suggests that part of the phosphate coating in the former systems is amor-

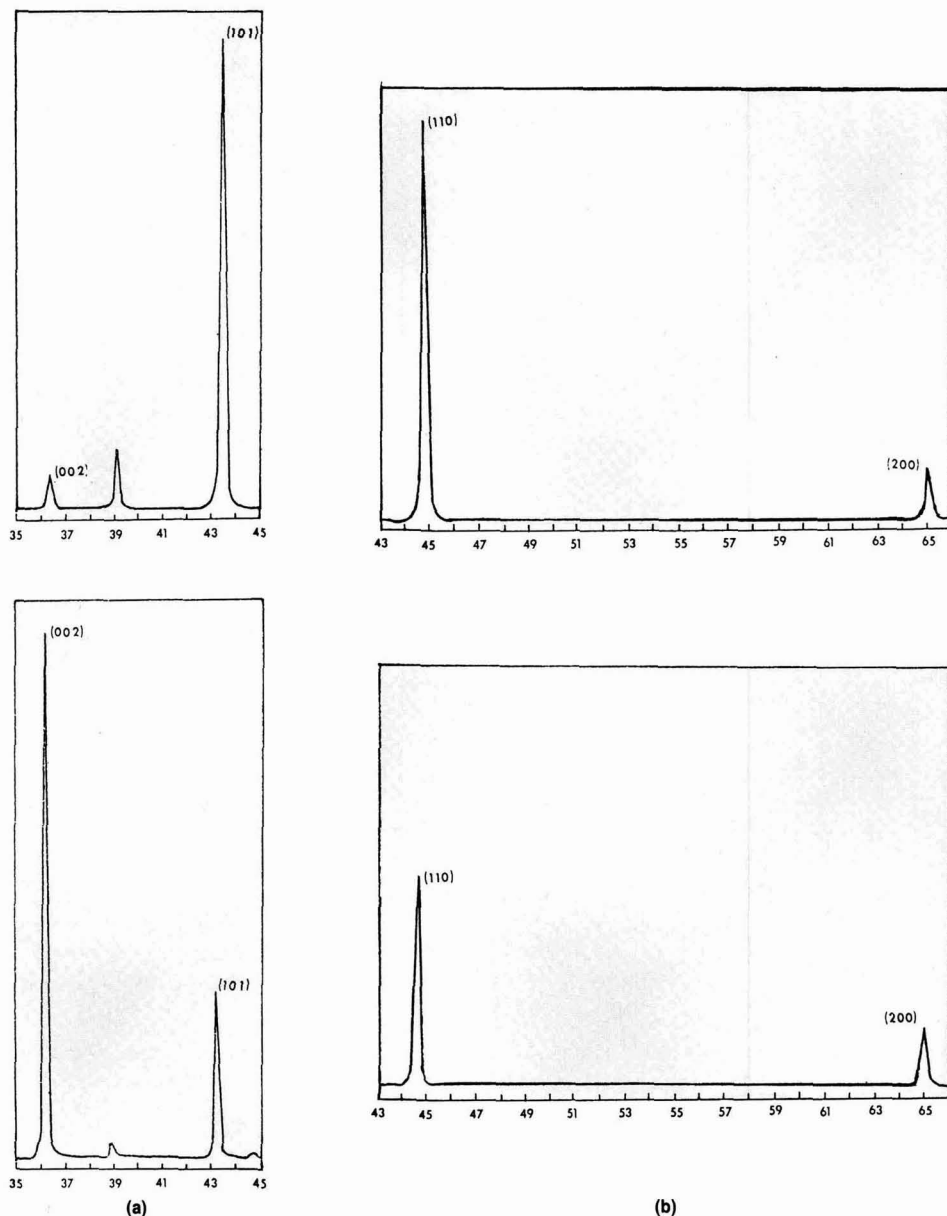


Figure 8—X-ray diffractograms of the substrates of the samples of Figure 7 showing the orientations of the zinc coating in 902 and 958 (a) and of the steel in 902 and 958 (b)

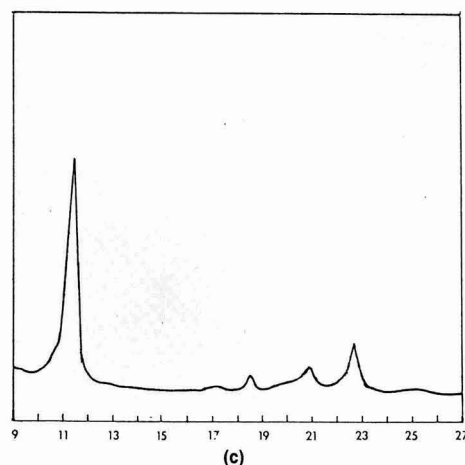
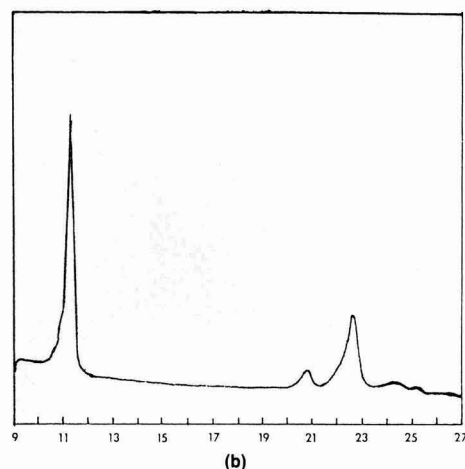
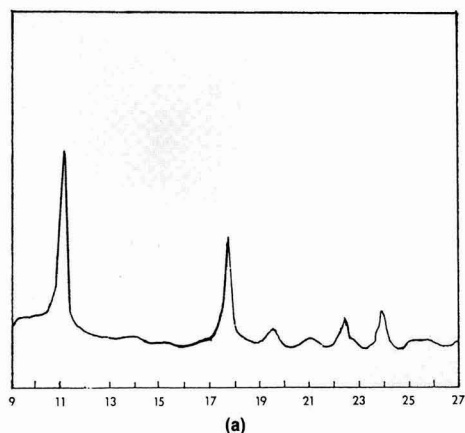


Figure 9—X-ray diffractograms of the phosphate crystals on EG 958 (a), on CRS 902 (b), and on CRS 958 (c) after baking at 180°C for 30 min

phous. It has been postulated previously that in the presence of Fe, or of Ni with Mn, part of the phosphate becomes amorphous.<sup>20, 21</sup>

### Baking of the Phosphate Coatings

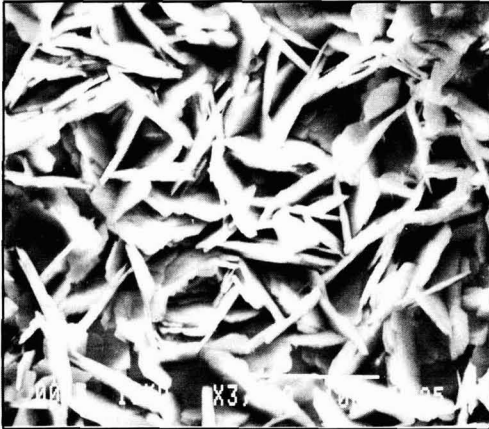
To study the effect that the baking of the commonly used cathodic electrocoat system might have on the crystallinity and orientation of hopeite and phosphophyllite coatings, samples of all panels were heated at 185°C in air for 30 min. It has already been demonstrated that under such conditions partial dehydration of hopeite to the dihydrate occurs.<sup>19</sup> Servais, et al., claimed that phosphophyllite had become completely amorphous after baking for 30 min at 160°C and higher.<sup>11</sup> Hopeite in this study dehydrated partly at 160°C and higher and this process was complete at 200°C.

Baking of our phosphate systems had no noticeable effect on the morphology and the composition of the crystals. X-ray diffraction indicated, however, that complete dehydration to the dihydrate had occurred for EG with Granodine 958 and 902, i.e., with or without Mn. The diffractogram is shown in Figure 9. All peaks can be identified as stemming from  $\text{Zn}_3(\text{PO}_4)_2 \cdot 2\text{H}_2\text{O}$ . Also in Figure 9, the diffractograms are shown of the baked CRS samples with Granodine 902 and 958, which are very similar. These spectra can also be identified as the dihydrate of zinc phosphate, but probably with a different orientation as compared with that on EG (Figure 9a). There are no peaks observed which can be attributed to a crystalline dehydrated form of phosphophyllite, so this component of the phosphate layer has probably become amorphous, in agreement with literature.<sup>11</sup> From this result, it cannot be determined whether this amorphous form of phosphophyllite has dehydrated or not. The intensity difference observed in the spectra of the baked 958 and 902 phosphates is in agreement with the different hopeite contents of these phosphates before baking, namely 70-75 and 15-20%.

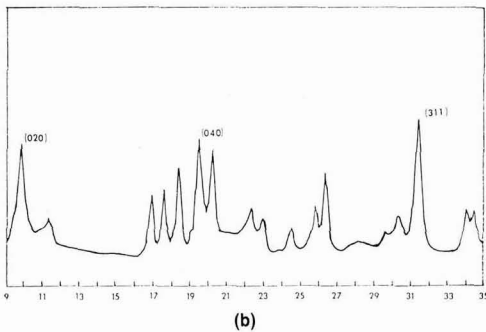
### Rehydration of Baked Phosphates

The baked phosphates were immersed in water for one week. The composition of the 902 system on EG did not change during this treatment, but the crystal morphology changed noticeably, as is shown in Figure 10a. Many crystals seem to have cracked or burst, possibly because of high stresses which develop during the rehydration process, in agreement with literature.<sup>11</sup> Figure 10b indicates that the dihydrate has largely been rehydrated to the original hopeite (compare with Figure 7). However, although all peaks in the diffractogram of the rehydrated hopeite are identical to those of the original phosphate, the crystal orientation has changed. After rehydration, a preferred (311) orientation is observed. Similar results were obtained when the sample was rehydrated in a nitrogen-containing atmosphere, i.e., when  $\text{N}_2$  was bubbled through the water. However, the rate of rehydration was slowed down when there was no oxygen present, and also with oxygen present but with the cut edges of the sample covered so as to exclude a contribution from a corrosion cell between the phosphate surface and the steel surface at





(a)



(b)

Figure 10—S.e. image (a) and X-ray diffractogram (b) of the phosphate crystals on EG 902 after baking at 180°C for 30 min followed by rehydration in water for one week at ambient temperature

the edges. These results indicate that the rehydration reactions are probably dependent on the pH at the phosphate surface which, in turn, is affected by corrosion reactions.

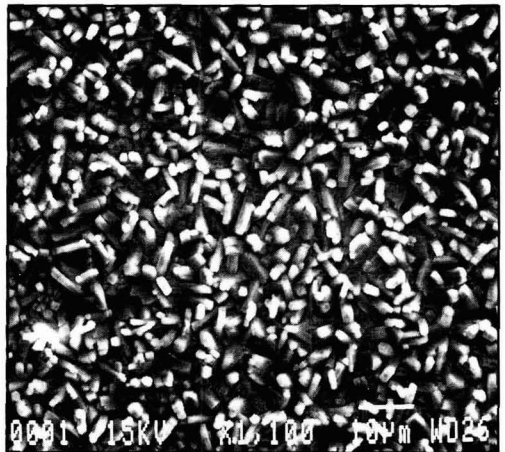
The results obtained with Granodine 958 on EG were quite different. In the absence of a corrosion cell, i.e., in  $N_2$  or with the cut edges taped, a small degree of rehydration was observed after four weeks. Clearly the rate of this reaction was less than in Granodine 902. However, in the presence of a corrosion cell, i.e., with oxygen present, large amounts of a crystalline form of  $Zn(OH)_2$  were formed and no rehydration was observed at all. Upon heating of the rehydrated sample at 180°C, this  $Zn(OH)_2$  was converted completely to  $ZnO$  and very little of the original phosphate was left. These results must be interpreted as effects of the presence of Mn in the coatings.

The behavior of the phosphates on CRS was different from that on EG, as can be concluded from Figure 11 (Granodine 958). After rehydration, the crystal morphology has not changed noticeably (Figure 11a). Neither the amorphous phosphophyllite nor the zinc phosphate dihydrate seems to rehydrate rapidly to the original crystalline

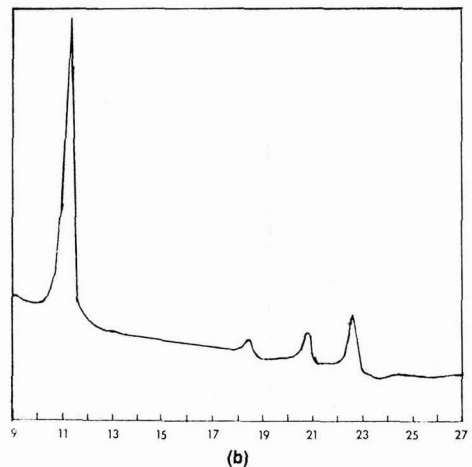
structure. The  $Zn_3(PO_4)_2 \cdot 2H_2O$  peaks in the diffractogram observed for system 902 decreased very slowly with simultaneous formation of some hopeite, but system 958 did not show any sign of rehydration after two weeks.

In summary, it was found that pure hopeite on EG dehydrates completely to the dihydrate form in 30 min at the electrocoat baking temperature. It rehydrates back to the tetrahydrate within one week when immersed in water. However, the rehydrated form has a different preferred crystal orientation.

Phosphophyllite transforms to an amorphous form during baking. This process is irreversible. The hopeite mixed with phosphophyllite on CRS dehydrates to the dihydrate but is not transformed back to the original tetrahydrate, so its behavior is different from that on EG. The presence of Mn seems to retard the rehydration processes in both substrates.



(a)



(b)

Figure 11—As Figure 10 but for phosphate crystals on CRS 958

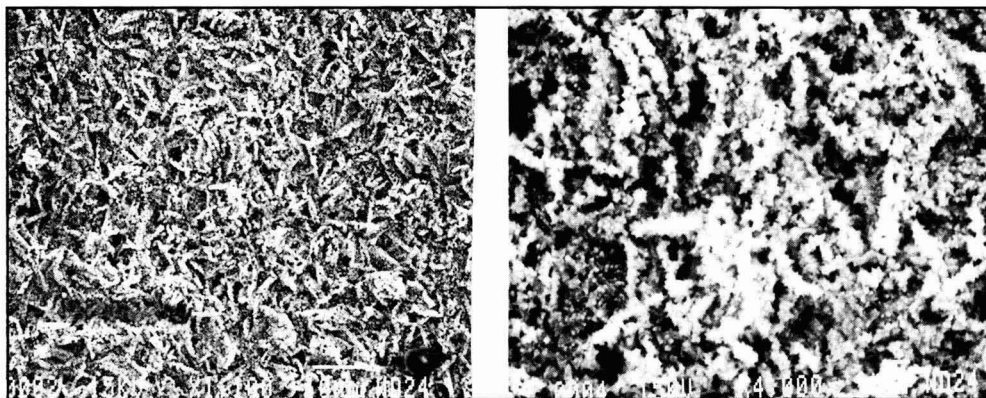
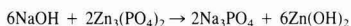


Figure 12—S.e. images of EG 902 following immersion in a nonstirred NaOH solution of pH 13 for 3 min at 60°C

### Stability of the Phosphates at High pH

The Granodine 902 phosphates on EG and CRS and the 958 on CRS were treated for 3 min at 60°C and pH 13 in NaOH solution. The solutions were not stirred. *Figure 12* shows the appearance of the treated crystals on EG. The phosphate before treatment is shown in *Figure 4*. The crystals on EG have disappeared and they have been replaced with a material which consists of very fine particles which grow on the surface of the original phosphate crystals. EDX and XRD analysis indicated that there was no hopeite left. XRD detected only small amounts of ZnO. The Ni content had not changed. So, it seems likely that, under the conditions of this experiment, the zinc phosphate is transformed to zinc hydroxide, which is largely amorphous and which transforms only slowly to ZnO.<sup>22</sup> The layer of reaction products is devoid of phosphorus, so it seems that the net reaction is:



The resultant zinc hydroxide does not dissolve completely which indicates that the pH at the surface must have been between 8.5 and 10.5.<sup>22</sup> As the solution was not stirred, such a drop of pH can be expected because the OH<sup>-</sup> ions are consumed in the exchange reaction with the PO<sub>4</sub><sup>3-</sup> ions.

*Figure 13* shows the CRS surfaces after the NaOH treatment, which can be compared with the as-received systems shown in *Figures 1* and *3*. EDX indicated that all of the phosphorus had dissolved in both systems, but most of the zinc was still present. The nickel and manganese contents had remained constant. By XRD, only a small amount of ZnO could be detected. The morphology of the phosphate coatings had changed somewhat, but not in the same way as for the hopeite on EG. Although some of the phosphate layer may have dissolved, it does not seem that particles have precipitated onto the surface here. In fact, some sort of mixed zinc-iron oxide has formed, which has almost the same morphology as the original phosphate crystals. This is in agreement with earlier reports on alkali attack of phosphophyllite-containing phosphate coatings,<sup>10,11</sup> and the results therefore confirm that the mechanism of PO<sub>4</sub><sup>3-</sup> ion dissolution

from hopeite on EG and from hopeite/phosphophyllite on CRS is quite different.

### Dependence of Phosphate Attack on pH and Stirring

The stability of the phosphate coatings on EG and CRS was also studied in a wide range of pH values, including acid solutions. In the same experiments, the effect of stirring the solutions was studied as well. Such an effect would indicate changes in the pH at the surfaces during phosphate attack. A demonstration of diffusion-controlled reactions could be helpful in the understanding of the mechanism of phosphate attack. In these experiments, the panels were immersed in solutions of different pH for 30 min at 25°C. The same panels as previously described were treated in runs with and without moderate stirring.

In *Figures 14a* and *b*, the dissolution curves are given for CRS and EG samples phosphated in Granodine 902 with and without stirring. It is observed that stirring accelerates the dissolution at high and low pH for both systems, but the effect of stirring is more pronounced at the low pH end of the curves for both systems. Further, the sensitivity to alkali and the rate of dissolution is identical for the two types of phosphates. At the low pH end, however, the EG system is considerably more resistant, both in stirred and in nonstirred solutions. The morphologies and compositions of the phosphates in these experiments were monitored. At low pH, they dissolve entirely, with or without stirring. At the high pH end of the curves, the reaction product left on the metal surface depends strongly on the stirring conditions. The hopeite on EG dissolves completely in the stirred solution, but forms an amorphous Zn(OH)<sub>2</sub> layer in the absence of stirring. Its morphology is shown in *Figure 15a*, which is different from the zinc hydroxide layer formed at higher temperatures (*Figure 12*). The phosphate on CRS forms the same reaction product with and without stirring and this product is practically identical in composition to the one formed at 60°C (*Figure 13a*). This product was tentatively identified as an amorphous mixed zinc-iron oxide. The product formed at 25°C, shown in *Figure 15b*, reveals less attack and cracking, i.e., it resembles the origi-

nal phosphate coating even more than after treatment at 60°C.

Figure 14c shows the zinc content of the phosphate on CRS as a function of pH. It can be seen that part of the zinc dissolves, but above pH 12 the composition remains constant, regardless of the degree of stirring of the solution. This composition is the mixed zinc-iron oxide, which does not dissolve further at high pH. These phenomena cannot simply be explained by assuming that the hopeite dissolves in alkali and the remaining phosphophyllite is converted to the zinc-iron oxide. If this were correct, there should be a difference between the stirred and nonstirred solutions, because we have observed that hopeite dissolves completely in a stirred but not in a stagnant solution (Figure 15a). Clearly the mechanism is more complicated.

### Comparison of Stabilities of Various Phosphates

The experiments previously described for one type of phosphate on EG and CRS were repeated for all available phosphate systems in stirred solutions only. The purpose of these experiments was to investigate the effects of the presence of Mn, of a chromium rinse, of the phosphophyllite content, and of the type of process (spray vs dip) on the chemical stability of modern phosphate coatings.

All systems behaved qualitatively in the same way as described. They all dissolved completely in acidic liquids without leaving any residue. At high pH, all phosphates on EG dissolved completely, all coatings on CRS left a residue of the amorphous zinc-iron oxide with approximately the same morphology as the original phosphate crystals. In Figure 16, the Granodine 958 systems on EG and CRS are shown following treatment at pH 12.9, which shows a partial retention of the original morphology in the former and a complete retention with very little damage and cracks in the latter, despite the complete leaching of the phosphate. On the low pH end, Ni and Mn also dissolved completely from all substrates; at the high

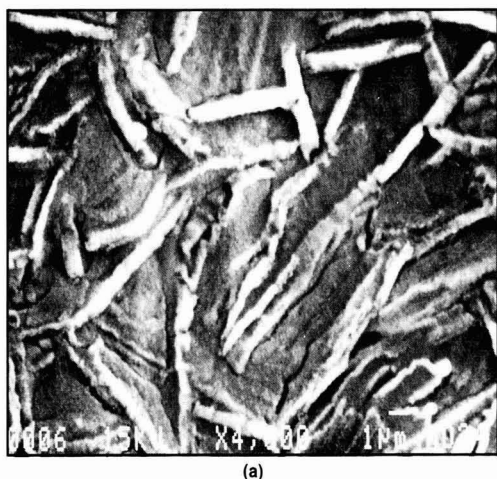
pH end they did not dissolve. This is exhibited in Figure 17 for Granodine 958 and 952. The curves seem to suggest that in acidic environments the Ni is dissolved more slowly than Mn.

In Figures 18a and b, the effect of the chromium postrinse is shown for the dissolution curves of 958 for EG and CRS and the 952 system on EG. No significant effect for any of these modern systems is observed at low pH. At high pH, the systems 952 on EG and 958 on CRS seem to be slightly more stable with the chromium rinse. However, the as-received systems with and without rinse did not have identical coating weights in this experiment and it is conceivable that the observed effect is, at least in part, due to these coating weight differences. The morphologies and compositions of the corresponding systems with and without postrinse, before and after attack, were also identical.

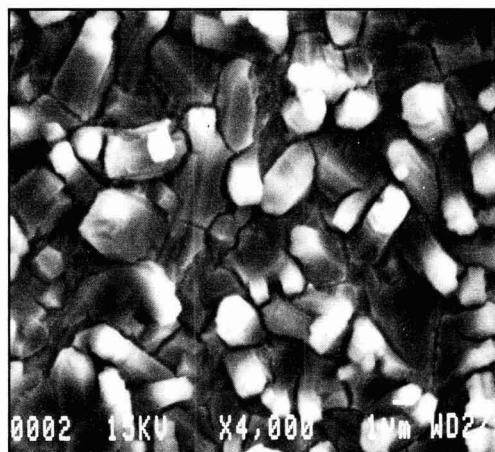
In Figure 19, the curves are given for the 902 and 952 spray systems on EG and CRS. These experiments were carried out with the postrinsed systems only. Comparison of these curves should thus indicate an improvement of the alkali or acid stability due to the incorporation of Mn in the coating, which has already been demonstrated to give rise to considerably smaller crystals. The curves show, however, that in these experiments no significant effect of the modification of the phosphate on its rate of attack at high or low pH can be detected. This conclusion holds for both EG and CRS.

In Figure 20, the performance of the modern Mn-containing systems in the spray and the dip versions for both substrates is compared. They were all postrinsed. It can be seen that, for both EG and CRS, the dip versions are more stable than the spray versions and this can be concluded for both the low and the high pH end of these experiments, although the effect is more significant in high pH conditions.

Finally, when comparing the acid and alkali stability of all systems studied (Figures 18-20), it can be concluded that the spray system 902 on CRS displays the highest



(a)



(b)

Figure 13—S.e. images of CRS 902 (a) and CRS 958 (b) after the same treatment as in Figure 12

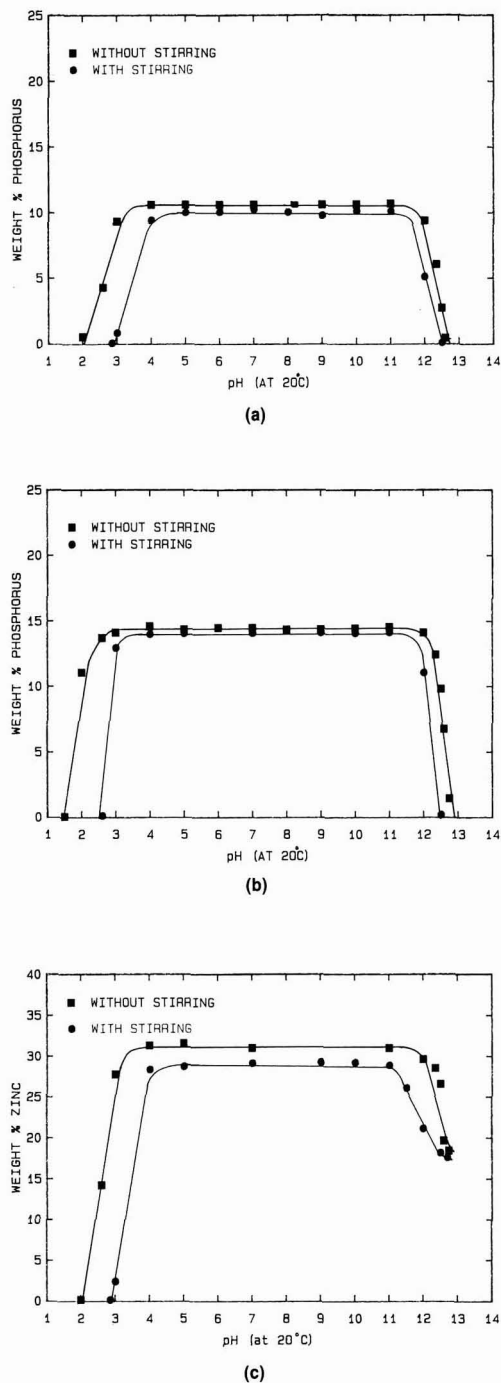


Figure 14—Effect of stirring on the rate of phosphate dissolution during immersion in solutions of different pH for 30 min at 25°C; (a) wt% P, CRS 902; (b) wt% P, EG 902; (c) wt% Zn, CRS 902

sensitivity to attack at both low and high pH, the dip system 958 on CRS is the most stable at high pH, whereas the same system on EG has the best stability in low pH conditions.

When comparing the pH stabilities of the various phosphate systems, as was done, it should be realized that these comparisons were made with unbaked systems. In actual painted automotive panels, however, the phosphates have always been subjected to baking following the E-coat deposition. The crystallographic changes that occur upon baking have been described in a previous section. The experiments in which the pH stabilities were studied are only meaningful, however, if the behavior of the various phosphates in such experiments is the same before and after baking. No studies have been reported in literature of the effect of baking on the chemical stability of phosphates.

In Figure 21, the pH stability curves are shown for system 902 on EG and on CRS before and after baking at 180°C for 30 min. For CRS, no difference either at high or low pH is detected. EG, on the other hand, shows a slight decrease in stability at low pH, whereas at high pH the stability is slightly improved. In both cases, however, the difference is marginal.

## DISCUSSION AND CONCLUSIONS

Phosphate coatings on EG consist of hopeite,  $\text{Zn}_3(\text{PO}_4)_2 \cdot 4\text{H}_2\text{O}$ , entirely. This is largely crystalline, although there is some evidence, based on diffraction peak intensities, that the incorporation of Mn into the crystals leads to a partial amorphotization of the zinc phosphate layer. In addition, the phosphate crystals containing Mn are of smaller dimensions and the Mn also seems to promote a nodular rather than a needle-like structure. This might be related to a more effective nucleation of the phosphate crystals if the baths contain both Ni and Mn.<sup>23</sup>

Upon baking, all phosphates on EG dehydrate readily and completely to the crystalline dihydrate. This takes place within 30 min at 185°C, i.e., it can be expected to be complete during the stoving of the commonly used electrocoat system. Upon exposure to water, the dihydrate tends to rehydrate to the tetrahydrate again; but for system 902, this process is associated with a severe cracking of the crystals (Figure 10a) and with a change in crystal orientation. The cracking may be related to the generation of severe stresses in the crystals as a result of the different lattice parameters of the two types of crystals. Such cracking is not observed in the dehydration step. This difference between the behavior in the dehydration and rehydration step may be related to the temperatures at which these processes proceed. Possibly the crystals can absorb the stresses generated in the dehydration process better because of the elevated temperature which facilitates stress relief by ionic mobility. The observed change in the orientation of the crystals after the dehydration and rehydration cycle provides some experimental evidence for such ionic mobility.

The rate of rehydration of the dihydrate is markedly retarded for systems containing Mn (952 and 958), in agreement with literature.<sup>20</sup> The mechanism of this phe-



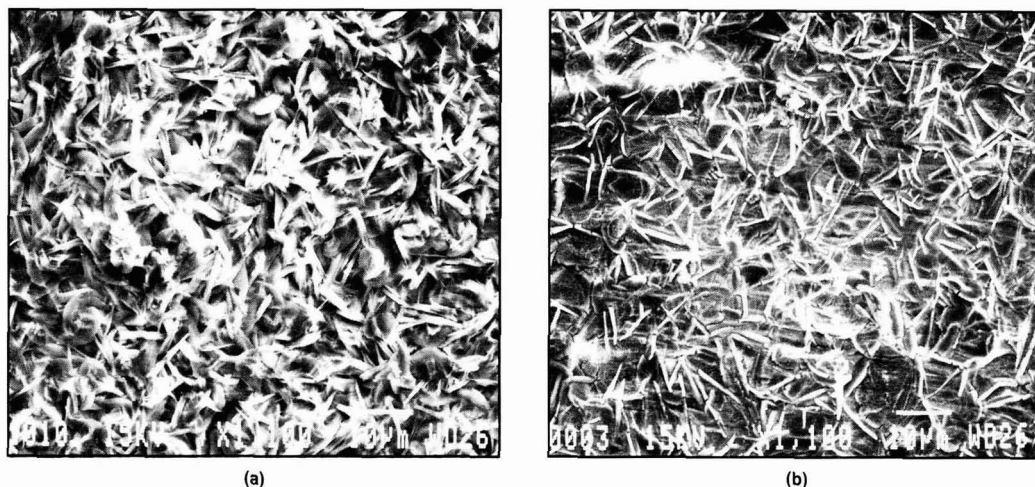


Figure 15—S.e. images of residues of EG 902 (a) and CRS 902 (b) after immersion in a nonstirred NaOH solution of pH 12.75 for 30 min at 25°C

nomenon is not known, because it is not exactly known what lattice positions the Mn ions occupy and whether it is present in an amorphous or crystalline form. It should be noted, however, that no difference was observed between the X-ray diffractograms of the dihydrates in systems with and without Mn and the same conclusion holds for the tetrahydrates, which would be consistent with Mn present as an amorphous phosphate. This is further in agreement with our observation that the intensities of the diffraction peaks for 952 and 958 are lower than for 902.

The retardation of the rehydration rate of the zinc phosphate by Mn could well be the underlying mecha-

nism why the wet adhesion of the EC-coat on Mn-containing phosphate systems is improved, as has been reported.<sup>20</sup> It should be pointed out that in the wet adhesion test, the phosphate is initially in the form of the dihydrate. Water then diffuses to the interface and rehydrates the phosphate surface, which is associated with cracking and reorientation effects, as previously discussed. These effects can be assumed to be detrimental to the adhesion and the improvement of the wet adhesion by the incorporation of Mn is caused by the inhibition of this rehydration reaction at the interface. Both the dry and wet adhesion of the EC-coat to the tetrahydrate (hopeite) are

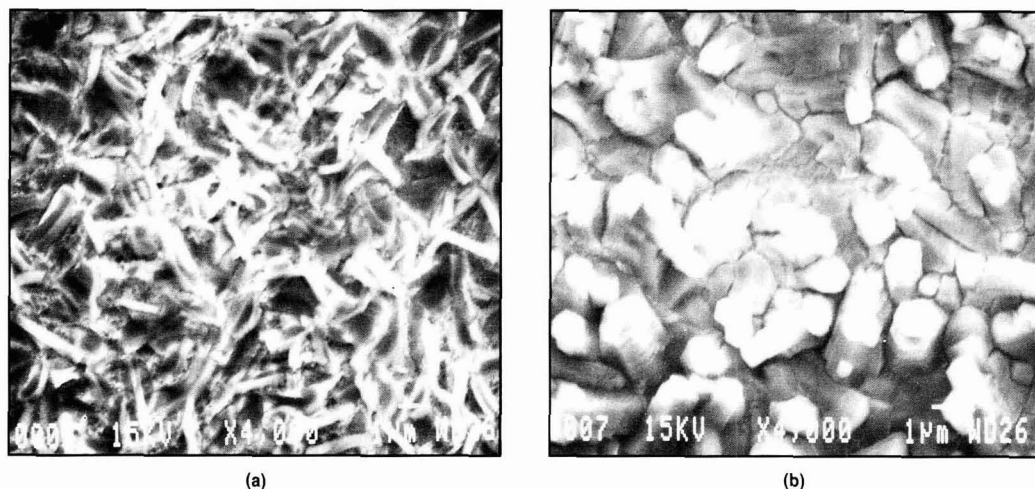


Figure 16—S.e. images of residues of EG 958 (a) and CRS 958 (b) after immersion in a stirred NaOH solution of pH 12.9 for 30 min at 25°C

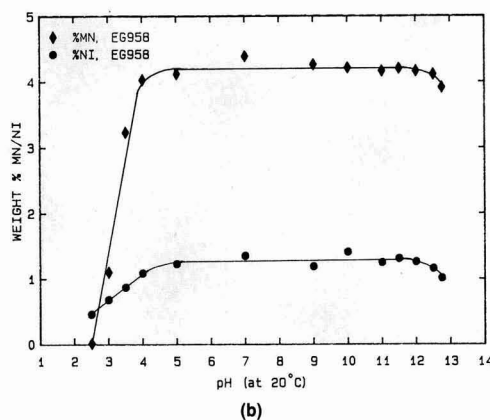
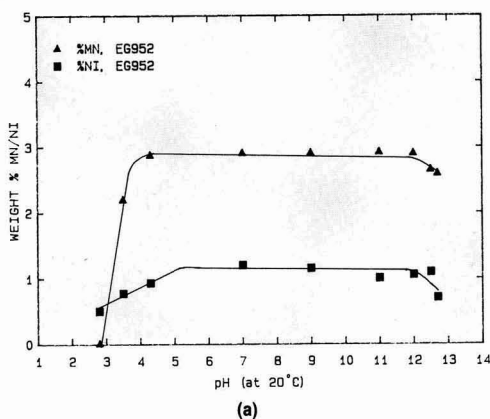


Figure 17—Dissolution of Ni and Mn during immersion in stirred solutions of different pH for 30 min at 25°C; (a) EG 952; (b) EG 958

probably good, but such adhesion cannot actually be evaluated because the curing of the paint can only be carried out at high temperatures where the tetrahydrate becomes converted to the dihydrate.

Rehydration effects of the dihydrate leading to a deterioration of the paint-phosphate interface can also be expected to take place during atmospheric exposure or in accelerated corrosion tests. This could be a contributing factor to the rate of cosmetic corrosion propagation and could be expected to be dependent on the percentage wet time, as is actually observed.<sup>24</sup> At high degrees of wet time, phosphated EG systems do not perform very well. We have actually observed the rehydration of the dihydrate to hopeite in undervehicle corrosion tests, which will be published elsewhere.

The phosphate coatings on CRS were all found to consist of a mixture of hopeite and phosphophyllite. The amount of the latter was higher for the dip phosphated systems. These observations are in agreement with literature. Similar to EG, the coatings with Mn consist of smaller phosphate crystals. The hopeite component of the phosphate coatings on CRS show a strong preferred (020)

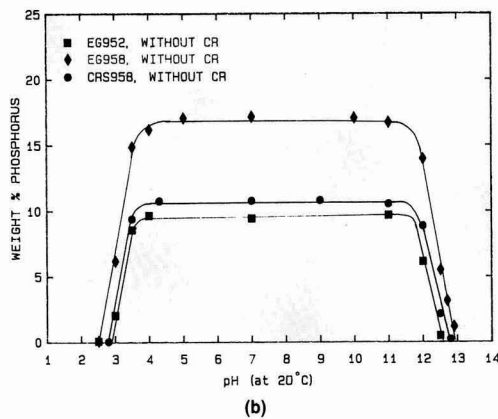
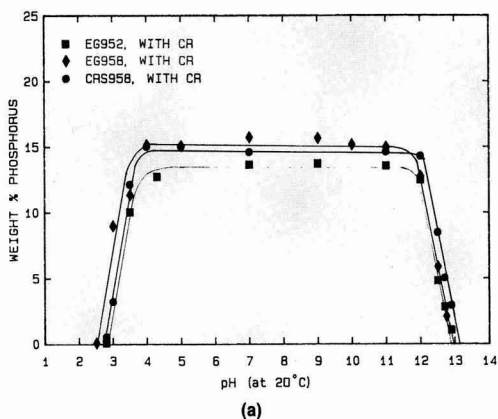


Figure 18—Effect of chrome postrinse treatment on the rate of phosphate dissolution after immersion in stirred solutions of different pH for 30 min at 25°C

orientation, i.e., a totally different texture than the hopeite on EG. The XRD results also suggest that in all systems some amorphous material is present, as reported in literature for mixtures of hopeite and phosphophyllite.<sup>21</sup>

Upon baking the phosphates on CRS, the hopeite component dehydrates to the dihydrate, as in EG, and the phosphophyllite becomes completely amorphous. This suggests strongly that some dehydration takes place as well, but the reaction product, i.e., dihydrate or anhydrous, cannot be identified from the XRD experiments. Experiments using Mössbauer spectroscopy are currently being carried out in an attempt to identify the amorphous reaction products. The results of these studies will be published elsewhere.

The amorphotization process leading to a completely disordered structure is indicative of a high ionic mobility at 180°C, as was discussed for EG. During immersion in water, the zinc phosphate dihydrate rehydrates as in EG, but at a much slower rate. The system with the high phosphophyllite content hardly rehydrates at all, so the effect of Fe on the rehydration of zinc phosphate dihy-

phosphate appears to be similar to that of Mn. The phosphophyllite remains completely amorphous during the immersion, so no conclusions can be drawn on the possible rehydration of the amorphous phosphophyllite component. However, as opposed to the pure hopeite coatings on EG, no cracking or morphological changes occurred in the dehydration and immersion steps of this experiment. This provides strong evidence that the relatively poor wet adhesion of paint coatings to pure hopeite is primarily related to the cracking of the crystals as a result of the rehydration. This model is partly in agreement with conclusions drawn in recent studies of wet paint adhesion to EG and Zn-Ni alloy plated steel.<sup>6</sup> The authors attributed the poor wet adhesion to EG in part to the phosphate rehydration which was supposed to generate interfacial shear stresses. This process did not actually lead to paint delamination because the phosphate-paint bond was assumed to be very strong. However, a second process led to a weakening of this bond, which, in conjunction with the interfacial shear stresses, resulted in a poor wet adhesion. This process is a hydrolysis of the phosphate to  $\text{Zn}(\text{OH})_2$  during immersion which partly decomposed to ZnO which was assumed to adhere poorly to the paint, although no evidence for this was presented. In Zn-Ni coated systems,  $\text{Zn}(\text{OH})_2$  has been shown to be stabilized by traces of  $\text{Ni}^{2+}$  ions,<sup>9</sup> so no ZnO is formed here. The adhesion between the paint and the  $\text{Zn}(\text{OH})_2$ -covered surface was found to be better than to ZnO. In our experiments, no ZnO or  $\text{Zn}(\text{OH})_2$  formation was detected and we feel that the formation of the latter at the sample edges or around pores in certain cases is the result of a corrosion reaction rather than hydrolysis of the phosphate. Therefore, the loss of wet adhesion to the materials investigated is probably caused mainly by the interfacial stresses induced by the cracking of the crystals.

### Reactions of the Phosphate Coatings

At low pH conditions, all phosphates dissolve completely. No residues are left on the zinc or steel surfaces. Obviously, the mechanism of this attack, e.g., in HCl, is a double decomposition of the phosphates to zinc or iron

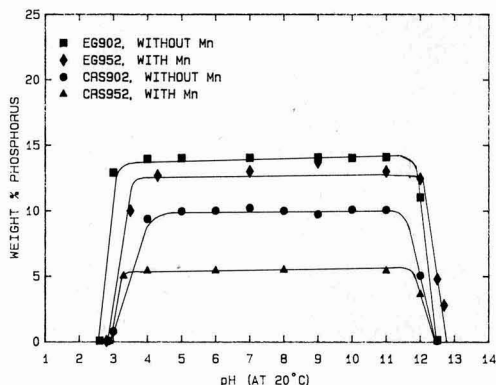


Figure 19—Comparison of the rates of phosphate dissolution for the spray systems on EG and CRS with and without Mn; conditions as in Figure 18

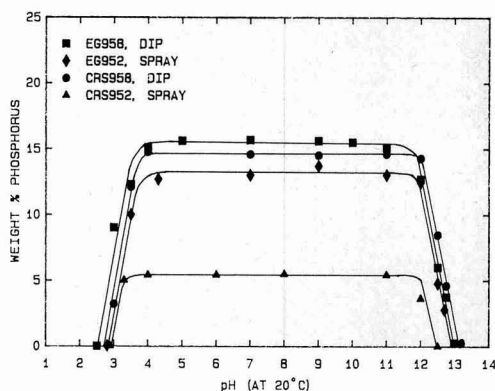
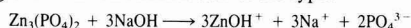


Figure 20—Comparison of the rates of phosphate dissolution for the spray and dip systems on EG and CRS; conditions as in Figure 18

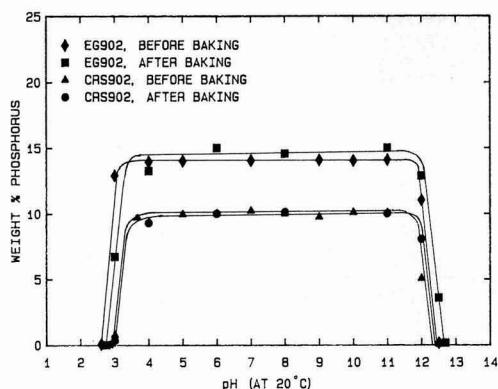
chlorides and phosphoric acid, the latter being a much weaker acid than HCl. The differences observed between the rates of dissolution in the pH range 2.5-3.0 are attributed to differences between the solubility products of the various phosphates and possibly also to differences between crystal shapes, dimensions, and surface areas. Mn and Ni do not have a marked effect on the rate of this dissolution because their phosphates also dissolve, although at a different rate (Figure 17). The effect of the stirring of the solution on the rate of phosphate dissolution is in agreement with this simple mechanism. Because of the consumption of hydrogen ions in the dissolution process, a concentration gradient develops in the boundary layer at the crystal surface. The rate of phosphate dissolution becomes diffusion-controlled. Stirring of the solution lowers the concentration gradient and the rate of attack of the crystals is increased.

At the high pH end of the dissolution curve, an accelerating effect of stirring the solution is also observed, indicating a diffusion-controlled reaction here, too. Obviously, this must be related to the diffusion of  $\text{OH}^-$  ions here. Since hydroxyl ions have a higher rate of diffusion than the strongly hydrated hydronium ions, the effect of stirring should be less pronounced at high pH than at low pH, which is indeed observed.

At high pH, hopeite generally dissolves completely, leaving behind a residue containing Mn and Ni only (Figure 17). Since the  $\text{PO}_4^{3-}$  ions have been leached out completely, the Mn and Ni residues are probably oxides or hydroxides, which are not amphoteric. A zinc-containing residue is only observed if the solution is not stirred. Because of the pH gradient at the phosphate surface, this suggests a dissolution reaction of the type:



If the solution is not stirred, the pH of the boundary layer may be around 9-10 and  $\text{ZnOH}^+$  reacts with  $\text{OH}^-$  to form  $\text{Zn}(\text{OH})_2$  particles which nucleate and grow at the phosphate surface. The result is a layer of  $\text{Zn}(\text{OH})_2$  with a morphology that vaguely resembles the original phosphate layer (Figures 12 and 15a), the more so if the reaction proceeds at room temperature. In that case, the amorphous  $\text{Zn}(\text{OH})_2$  particles are almost identical in



**Figure 21—Comparison of the rates of phosphate dissolution for EG 902 and CRS 902 before and after baking at 180°C for 30 min**

morphology to the original phosphate crystals. This observation suggests that the exchange between  $\text{PO}_4^{3-}$  and  $\text{OH}^-$  ions in such conditions takes place in the crystal itself, i.e., it can be interpreted as a solid state reaction. A high ionic mobility in phosphate crystals has been demonstrated, e.g., for Fe in phosphophyllite.<sup>26</sup>

If the pH is above 10, i.e., if the solution is stirred, the  $\text{ZnOH}^+$  ions react with excess  $\text{OH}^-$  ions to form zincate ions which are soluble:



The result is a complete dissolution of the hopeite. Experiments to confirm the mechanism of phosphate dissolution are in progress.

Phosphate coatings containing phosphophyllite behave quite differently from pure hopeite coatings in alkaline solutions. Irrespective of whether the solution is stirred or not, the result of alkaline attack is always a complete leaching of all phosphate ions, leaving behind most of the zinc and all of the iron. If the reaction is carried out at room temperature, the original phosphate crystal morphology is retained without any detectable cracking or dissolution. This behavior was found to be independent of the original phosphophyllite content, although the high phosphophyllite coating showed a slightly higher tendency for cracking during the attack.

This behavior of the phosphate coatings further seems to be independent of the presence of Ni and Mn, since it has been reported previously for phosphophyllite-containing coatings without Ni and/or Mn.<sup>10,11,27</sup> The exact nature and composition of the phosphate-free residue with the original crystal morphology cannot be established yet without further analysis. However, for both phosphophyllite contents, the reaction product consists of Fe, Zn, and O only and both products are completely amorphous. Baking of the residue at 180°C does not cause any morphological or other detectable changes in this material and it remains amorphous, as opposed to the amorphous  $\text{Zn}(\text{OH})_2$  formed from pure hopeite on EG which dehydrates to crystalline ZnO upon baking. As expected, it dissolves rapidly in low pH solutions before and after baking. It has been suggested that this material is an iron zincate.<sup>10,28</sup> Experiments to elucidate its struc-

ture further by means of Mössbauer spectroscopy are in progress.

The mechanism of formation of such an iron zincate must be attributed to a solid state exchange reaction with or without stirring, as was also postulated for pure hopeite in the nonstirred solutions. The argument for this mechanism is the retention of the crystal shape, dimension, and morphology, regardless of the phosphophyllite content. It is impossible that this would be the result of a dissolution followed by a precipitation reaction. Such a solid state reaction requires a rapid diffusion of phosphate ions out of the crystals and a diffusion of  $\text{OH}^-$  ions into the crystals. Such a diffusion process requires a high ionic mobility at ambient temperatures. Evidence for such a mobility is the observation that the reaction product is completely amorphous. Another pertinent observation is the electron-stimulated rapid diffusion of Fe to the phosphate surface which has been reported in AES analysis of phosphophyllite-containing coatings.<sup>10</sup> The diffusion of  $\text{Fe}^{2+}$  ions through the crystal lattice may be the underlying mechanism why the reaction product is independent of the stirring of the solution.

Our observations are therefore in agreement with the following model. At high pH, phosphate ions are completely replaced with  $\text{OH}^-$  ions in a solid state process. The phosphophyllite thus forms  $\text{Zn}_2\text{Fe}(\text{OH})_6$  which is insoluble in alkali. The hopeite is completely converted to  $\text{Zn}(\text{OH})_2$  but now it does not dissolve as it does from EG substrates, but it forms an insoluble double salt with  $\text{Zn}_2\text{Fe}(\text{OH})_6$  of the composition  $x\text{Zn}(\text{OH})_2 \cdot y\text{Zn}_2\text{Fe}(\text{OH})_6$  instead. The ratio  $x/y$  is variable and depends on the original composition of the phosphate coating. Zinc is known to form many of such stable and highly insoluble double salts involving the hydroxide and in such salts the hydroxide does not easily dehydrate. A well-known example is the corrosion product  $4\text{Zn}(\text{OH})_2 \cdot \text{ZnCl}_2$  formed in corrosion tests involving chloride ions.<sup>2,3</sup> The formation of the postulated mixed compound is conceivable because of the similarity of the crystallographic orientation of the hopeite and the phosphophyllite as was shown in Figure 7. This suggests that the hopeite has a preferred orientation with a d-spacing similar to that in phosphophyllite. This intimate mixing of crystals with the same d-spacing will facilitate the formation of the double salt after replacement of the phosphate with hydroxyl ions.

In conclusion, the quality of modern phosphate coatings on EG and CRS has been much improved in recent years. This is the result of the incorporation of a few percent of Ni and Mn in the coatings. These metals are present as amorphous phosphates. Several effects of these metals on the behavior of the phosphate conversion coating were observed. First, the crystals of coatings with Ni and Mn are considerably finer. Quantitative data on this effect as a function of Ni and Mn concentrations were recently reported by others.<sup>29</sup> Further, the alkali stability of the phosphate coating is enhanced by the incorporation of the metal ions. This is of importance for the protection of the coating in conditions of high pH, e.g., during the cathodic electrocoating process and in certain corrosion conditions. However, the stability of the coatings in low pH conditions is not affected by the presence of Ni and Mn. Another effect of these metal ions that we observed



is that the rehydration of baked, partly dehydrated phosphate coatings on EG is markedly retarded. This is probably the underlying mechanism why the wet adhesion to phosphated EG is improved. Such wet adhesion has been reported to be poor for EG. The best overall systems that we studied were those obtained by dip phosphating which had more Mn incorporated in the system than the spray phosphated panels. Finally, the presence of Mn seems to increase the amount of phosphophyllite formed in spray systems.

The presence of Ni and Mn did not appear to have an effect on the orientation of the crystalline components of the phosphates, nor on the composition of the reaction products formed during attack by alkali or acid. In an acidic environment, the entire coating dissolves, whereas at high pH, pure hopeite on EG either dissolves or forms amorphous zinc hydroxide, depending on the conditions. The phosphates on CRS always form a complex double salt which seems to consist of an iron zincate and zinc hydroxide. This product retains the original phosphate crystal morphology.

Clearly, the behavior of modern phosphate coatings is very complex and more work needs to be done before their contribution to the corrosion processes of pre-coated steels in different environments can be completely understood. In actual service conditions, the corrosion conditions under paint coatings may be quite different from those used in this study, and probably involves a combination of concurrent rehydration and hydrolysis reactions resulting in phosphate degradation.

There is evidence that the rate of attack and the products are strongly dependent on these conditions. For instance, it has been reported that in microblisters formed in salt spray tests of painted CRS, the phosphate coatings dissolve completely.<sup>10</sup> This was attributed to a complexing effect of a high concentration of phosphate ions in the blisters which cannot diffuse away as in our experiments reported here. We have recently observed that certain inorganic ions, e.g.,  $\text{Cl}^-$ , can have a strong effect on the behavior of phosphate coatings at high pH. These results will be published elsewhere.<sup>30</sup> It has also been reported that the composition of the phosphate residues on CRS is dependent on the type of alkali cation as well.<sup>28</sup>

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# Effect of Extender on Crowding Of Titanium Pigment

Fred B. Stieg  
Pigmentech Consulting\*

## Introduction

Ever since my original observation that the decrease in the hiding power of any titanium pigment, occurring as formula PVC increased in a single-pigment system, could be related to the decreased separation of individual pigment particles in the dry paint film,<sup>1</sup> and similar early descriptions of hiding power variations when the titanium was combined with extenders of different particle sizes,<sup>2</sup> there has been a general tendency among other spokesmen for the titanium pigment industry to refer to the cause of both phenomena as "crowding."<sup>3,4</sup>

While the reduced spacing of titanium particles as their concentration is increased in a fixed volume of dry film might well be described as crowding, no legitimate reason exists for applying the same name to the effect produced by the replacement of titanium dioxide (TiO<sub>2</sub>), in a similarly fixed volume, with an equal volume of coarse extender.

The name applied would normally cause little concern, but the concept of crowding has been employed to disparage commercial spacing extenders as being "too coarse"<sup>5</sup> to perform a service for which they already have been employed by a generation of paint formulators. The confusion generated should be dispelled by reviewing the facts.

## Effect of Extender Particle Size on Packing

In essence, the "crowding theory" is no more than a casual assumption that any substitution of large-for-small pigmentary

particles in a fixed volume of total film will result in the small particles being packed more closely together than they would be if all particles were of the same smaller size. There has been no supporting evidence, but this assumption has been widely accepted among paint formulators.

This acceptance may be attributed to the exposure that most of us had had to that "packing" presumed to exist at the CPVC,<sup>6</sup> with all of the available space between the coarsest particles completely filled with particles of the next smaller size, and so on down to the finest.

CPVC curves for two-component systems, based upon oil absorption,<sup>7</sup> had also provided evidence that the replacement of TiO<sub>2</sub> with coarse extender resulted in just such packing, some mixtures requiring less oil to coat the pigment surfaces and to fill the residual voids than was required for either titanium or extender alone. The larger the particle size of the extender, the steeper the slope of the plotted CPVC curve, and the more pronounced the "peak" at maximum packing.

It has been too easy, unfortunately, to overlook the fact that the packing that had taken place actually had occurred, *not because of the presence of coarse extender, but because the system no longer possessed the same total volume*. Less oil was present, because less oil was required to reach an end-point in the oil-absorption test procedure, and the coarser the extender, the less oil was required. Had the total volume of the system been kept constant by always adding the same volume of oil, a different conclusion might have been reached as to the crowding effect of such coarse extenders on titanium pigment, although the results would have been of little value in identifying the CPVC. However, this is

what we commonly do when formulating paint.

At approximately 52 PVC, a pure enamel-grade rutile, rubbed out in oil, will be so crowded that the paste will not flow, or even wet a substrate. This is the pigment's CPVC in oil. At the same PVC, a blend, with 35% of the titanium pigment volume replaced by a calcium carbonate, and rubbed out using the same oil, will display both flow and wetting, because the CPVC of the blend is now almost 60%, and its dispersion at 52 PVC will contain 38½% of "free oil" in excess of that required to simply wet the pigment (see Figure 1).<sup>8</sup> The TiO<sub>2</sub> particles will obviously have become less tightly packed as a result of their dilution with extender, rather than crowded.

The use of the word "obviously" may seem difficult to justify, in view of the widespread acceptance of the crowding concept and the credentials of those who

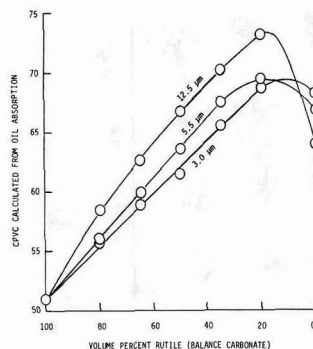


Figure 1—Evidence of packing in CPVC curves for different particle sizes

\*903 Beachview Dr., Jekyll Island, GA 31520.

have espoused it, but the calculations are simple, and the conclusion unavoidable:

#### At 52 PVC

52.0 volumes  $\text{TiO}_2$   
48.0 volumes required oil  
100.0 volumes total

#### and

33.8 volumes  $\text{TiO}_2$   
18.2 volumes calcium carbonate  
34.7 volumes required oil ( $52.0 \times 0.40/0.60$ )  
13.3 volumes free oil  
100.0 volumes total

### Crowding and Spacing Of Titanium Dioxide

Another possible reason for the erroneous assumption of crowding, when  $\text{TiO}_2$  is replaced by a coarser extender, is the fact that those who have drawn two-dimensional diagrams to illustrate the effect have infrequently observed the formulator's logical practice of making such substitutions on an equal-volume basis.<sup>5</sup>

If we assume, for the sake of simplicity, that both the  $\text{TiO}_2$  and the extender are of completely uniform particle size, any single particle of an 0.8- $\mu\text{m}$  spacing extender will be replacing 64 particles of a 0.2- $\mu\text{m}$   $\text{TiO}_2$ , on an equal-volume basis. This single particle, however, will possess only one-fourth the surface area of the 64 titanium particles that it has replaced. This means that less binder will be required to maintain the spacing layer around it than was consumed in spacing the original titanium particles, and the binder that once filled the remainder of the space between them is no longer needed for that purpose.

That freed binder adds to the total volume within which the remaining titanium particles may be expected to redistribute themselves at a slightly greater particle-separation than before. This may be illustrated as in Figures 2 and 3, using a cubic distribution of particles for simplicity of

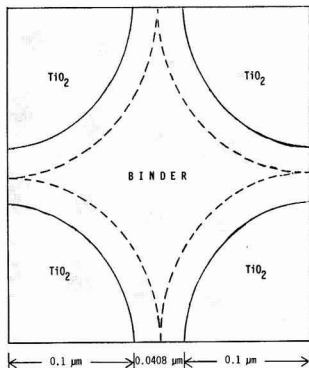


Figure 2— $\text{TiO}_2$  dispersion at 30 PVC

drawing, in a 30 PVC "film." It has been assumed that each particle will be centered at one corner of a cubic section of that film. Since a cube has eight corners, and one-eighth of each particle's volume falls within the cube, the space between particles may be easily visualized as a function of PVC and particle size.

For  $\text{TiO}_2$  alone (diameter 0.2  $\mu\text{m}$ ), the volume of a single particle will be:

$$\begin{aligned}\text{vol TiO}_2 &= 0.5236 \times 0.2^3 \text{ cubic } \mu\text{m} \\ &= 0.00419\end{aligned}$$

and since the cubic section contains only one such particle, its total volume at 30 PVC will be:

$$\begin{aligned}\text{vol cube} &= 0.00419/0.30 \text{ cubic } \mu\text{m} \\ &= 0.01396\end{aligned}$$

Each side of the cube will be the cube root of this latter number, or 0.2408  $\mu\text{m}$ , making the linear space between each of the individual particles 0.0408  $\mu\text{m}$  ( $0.2408 - 0.1 - 0.1$ ). This "stand-off" spacing may be thought of, for the purposes of the model, as having been contributed by a 0.0204  $\mu\text{m}$ -thick layer of binder around each individual particle. However, each particle also may be thought of as associated with a total of 0.00977 cubic microns of binder ( $0.01396 \times 0.70$ ), a volume that will be referred to at a later point.

The four-times larger extender particles may be treated in a similar manner (see Figure 3). The spacing of these particles, however, will be influenced by a much lower concentration, since they are replacing only 20% of the  $\text{TiO}_2$ . For the extender alone:

$$\begin{aligned}\text{vol particle} &= 0.5236 \times 0.8^3 \text{ cubic } \mu\text{m} \\ &= 0.2681 \\ \text{vol cube} &= 0.2681/(0.3 \times 0.2) \\ &= 4.4680 \text{ cubic } \mu\text{m}\end{aligned}$$

A cube of film of this volume, if pigmented only with the  $\text{TiO}_2$ , as in Figure 2, would contain  $4.4680/0.01396$  particles, or a total of 320, of which the extender has replaced 64, leaving 256.

The 64 titanium particles would have been associated with a total volume of binder equivalent to  $0.00977 \times 64 = 0.6255$  cubic  $\mu\text{m}$ . However, providing the larger extender particle with an equally thick 0.0204- $\mu\text{m}$  "stand-off" layer will require significantly less binder because its surface area will be only one-fourth that of the 64 titanium dioxide particles which it has replaced:

$$\begin{aligned}\text{vol ext + stand-off layer} &= 0.3112 \text{ cubic } \mu\text{m} \\ \text{vol ext particle} &= 0.2681 \text{ cubic } \mu\text{m} \\ \text{vol stand-off layer} &= 0.0431 \text{ cubic } \mu\text{m}\end{aligned}$$

This means that the extended system will possess additional binder with which to increase the stand-off layer between the particles of pigment and extender. Rather than being crowded by the presence of the

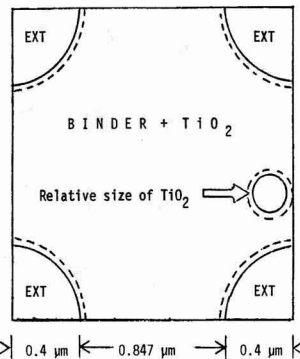


Figure 3—Dispersion of 0.8  $\mu\text{m}$  extender

coarser extender, the remaining 256 titanium particles will be provided with an opportunity to increase particle separation, and the coarser the extender, the greater the increase in free binder per particle of titanium!

It follows, moreover, that if the extender particles were smaller than  $\text{TiO}_2$ , as claimed to be necessary for a spacing effect in a recent publication,<sup>5</sup> an increase in surface area would be produced, a greater volume of binder would be required to coat them, and all of the titanium particles would have to move closer together to maintain a uniform spacing in the reduced volume of free binder. Thus, while crowding may indeed occur, it does not occur for the reasons so often postulated. This is consistent with the findings of Dr. R.W. Craft<sup>9</sup> of the Du Pont Company, in respect to the inferior spacing effects of ultra-fine blanc fixe when compared with less-fine extenders.

### Hiding Power and Extender Particle Size

If the loss of  $\text{TiO}_2$  hiding efficiency with an increased extender particle size can not be attributed to crowding, why does this loss occur?

It seems probable that the sheer size of the discontinuity in the light-scattering pattern represented by the extender particle may well be the basic reason.

When  $\text{TiO}_2$  is replaced by equal volumes of any extender, the number of scattering-centers replaced by a single extender particle increases as the cube of the ratio of the extender diameter to that of  $\text{TiO}_2$ . In the previous model involving on 0.8- $\mu\text{m}$  extender, one extender particle replaced 64 titanium particles:

$$0.8/0.2 = 4.0 \text{ and } 4^3 = 64 \text{ particles}$$

For a 3.0- $\mu\text{m}$  extender, the replacement becomes:

$$3.0/0.2 = 15.0 \text{ and } 15^3 = 3,375 \text{ particles}$$



Surface area will be proportional to the square of the particle's diameter, so that coarser extender will possess a surface area 14 times as great as that of the 0.8- $\mu$ m extender:

$$3^2/.8^2 = 14.0625$$

Since both extenders would be completely surrounded at the standoff spacing by a layer of titanium particles (equally spaced from each other), this means that the number of titanium particles in that layer will be approximately 14 times greater for the coarse extender than for the finer.

Since any extender will have a refractive index essentially the same as binder, it represents a discontinuity in the light-scattering pattern created by the  $\text{TiO}_2$  particles, each of which is an independent light-scattering center. If this discontinuity has a detrimental effect on dependent scattering, then the larger the extender particle, the greater the number of adjacent light-scattering sources likely to be affected by its presence.

On the other hand, such a discontinuity also represents an increase in the particle-to-particle separation for titanium dioxide particles located on opposite sides of it, and increased particle-separation is generally recognized as beneficial to light-scattering efficiency. However, while light-scattering efficiency in single-pigment systems is known to increase with dilution for  $\text{TiO}_2$ , it is also known that the increase does not continue at dilutions greater than 10 PVC<sup>2</sup> since scattering-efficiency becomes constant at higher dilutions.

Experiments have commonly shown<sup>4,9</sup> that an extender with a particle size such as that employed in the hypothetical titanium-replacement of Figure 3 may be introduced as a volume replacement for vehicle solids, when raising the PVC of a test system, without reducing hiding power.

Since this replacement would surely result in forcing all of the titanium particles closer together—an action that we accept as detrimental to hiding efficiency—it is possible to speculate that the effect of this crowding is roughly compensated for by the spacing provided to the titanium particles immediately surrounding it by the larger bulk of the extender particle.

As previously noted, however, experiments show the light-scattering efficiency of  $\text{TiO}_2$  becomes constant after a separation of approximately one diameter has been attained. One might also, therefore, further speculate that extender particle bulk, above some limiting particle-size, would be creating a greater separation than required for maximum light-scattering. Further, the excess bulk would, in a sense, be lost as a source of increased light-scattering efficiency, and become merely diluting volume.

Finally, as the extender particle becomes larger, it represents a correspondingly greater proportion of the total dry-film thickness, constituting what has been described as a "hole"<sup>11</sup> or "window"<sup>10</sup> through the film. The window term was actually applied to a nonfilm forming latex particle, to explain why hiding power decreased as its particle size increased, the nonfilm forming latex being considered to be a "plastic extender," producing effects similar to mineral extenders of similar particle size.

It should be noted, however, that the apparent CPVC was found to decrease with increases in the particle size of normal latex binders, and with increases in their glass transition temperature, in earlier work.<sup>11</sup> Taken together, these characteristics of a latex binder, plus the amount of associated plasticizers or coalescents, were considered to establish the "binding power index" (BPI) of the latex. This could be calculated by a comparison of the CPVC for an extender, as calculated from oil absorption, with its apparent CPVC in the latex, as established by the onset of high dry-hiding.<sup>12</sup>

$$\text{BPI} = (1 - \text{CPVC}) / (1 - \text{CPVC}')$$

where CPVC' = apparent CPVC in latex system  
CPVC = CPVC of pigmentation from O.A.

BPI = binding power index

This will be recognized as consistent with Berardi's definition of his binding power index as a ratio of the amount of oil required to completely bind a given quantity of pigment, to the amount of latex required to produce the same result.<sup>13</sup>

### Effect of BPI Above the CPVC

By this interpretation, the packing of pigment particles above the CPVC is not changed by any differences in latex BPI. They exist in a condition of maximum packing, as in a solvent system, but the ratio of air-to-binder in the inter-particulate voids is not determined only by the availability of binder, but also by the relative ability of the binder to penetrate into the packing, as determined by its particle size and ability to deform at ambient temperatures.

The density of the packing itself is, of course, still determined by the relative particle sizes of pigment and extender, as is the size of the pores and voids to be penetrated by the binder. Crowding is a normal condition, since, as in the CPVC curves previously described, no free binder exists, and BPI is the only variable. Consequently, at the same formula PVC and the same pigmentation, high dry-hiding will decrease as BPI increases, and increase as BPI decreases.<sup>14</sup>

It is possible to speculate that a nonfilm-forming latex might be considered to be a latex vehicle with a BPI of zero, rather than a pigment. In which case, its admixtures with a normal, film-forming latex might be expected to possess a BPI somewhere between that of the film-forming latex and zero, while a reduction in formula PVC would be created by its replacement of any quantity of  $\text{TiO}_2$ .

This concept has been successfully used to predict the effect upon hiding power of a replacement of  $\text{TiO}_2$  with "microvoids" below the CPVC (with an accuracy of about  $\pm 2\%$ ) in unpublished work.

If a "window-through-the-film" does increase with extender particle size, then this effect for any large particle should become more pronounced as film thickness decreases. This does not appear to occur, however, according to the published work of one respected worker. The scattering coefficient of  $\text{TiO}_2$  in latex flat wall paints, as calculated using the Kubelka-Munk equations, has been found to vary with both PVC and the film thickness at which "S" is measured, generally tending to increase as film thickness decreases, for PVCs above the critical, and in the presence of large-particle-size extender.

There is a temptation to interpret these data as the result of increased surface texture due to the extender, or to increased porosity due to some change in film formation, but the same investigator has found a similar effect in very thin films of gloss paints at PVCs of 15-30%, in the absence of extender. It therefore seems possible that his observation in respect to the extender-containing flat latex paints is simply evidence of the nonapplicability of Kubelka-Munk theory at low film thicknesses, rather than a negation of the window hypothesis. There may be still other interpretations, however.

### The Dilution Efficiency Of Extenders

The previous comments as to the relationship of hiding power to extender particle size are necessarily speculative, since theorists have yet to provide mathematical models to predict the effects of multiple scattering in simple  $\text{TiO}_2$  dispersions above 10 PVC after quite a few years of effort, although work still continues.

Furthermore, the relationship becomes even more complicated above the CPVC, where the pigment and extender combination already exists in a condition of maximum packing, and hiding power is augmented by "high dry-hiding" due to dry-film porosity. The addition of very fine extender, under these circumstances, may be found to increase high dry-hiding without increasing apparent porosity, as evalu-

**Table 1—Values Obtained for the Calcium Carbonate Extenders of Figure 1**

Mean d	Ratio (0.8/d)	E/d Value
0.8	1.00	1.00
3.0	0.27	0.30
5.5	0.15	0.15
12.5	0.06	0.00

ated by enamel holdout or the penetration of some stains, due to a reduction in pore diameter by an even closer packing. Since a precise scientific explanation for the complications introduced by extenders must await a solution of the more basic problem, dependent scattering, it has become necessary to take a more pragmatic approach.<sup>16</sup>

The Dilution Efficiency concept was developed as a means for quantifying the effect of extender particle size on hiding power, as calculated using a semi-empirical equation for predicting the hiding power of  $\text{TiO}_2$ :<sup>2</sup>

$$\text{hiding power/lb TiO}_2 = 409 \times [0.9045 - \text{PVC}^{(1/3)}]$$

where  $\text{PVCe}$  = effective PVC for  $\text{TiO}_2$

$$\text{PVCe} = \text{vol TiO}_2 / \text{vols (TiO}_2 + \text{ext} \times \text{E/d} + \text{binder})$$

where E/d = dilution efficiency of extender

The volume of an extender multiplied by its dilution efficiency was considered to represent its "effective volume" as a diluent. A series of extenders of different types and particle sizes were compared at a fixed volume (9.50 gal) in a 40 PVC latex test formulation containing 2.20 lb of enamel-grade rutile  $\text{TiO}_2$  per gallon.

Test grinds were compared for relative hiding power by the tinting-strength method of ASTM D 2745, using a high-brightness 20 micrometer classified natural calcium carbonate as a "zero" (O) standard. The Kubelka-Munk relationship used is independent of film thickness, permitting relative white hiding power at equal brightness to be calculated from reflectance readings for test aliquots shaded with exactly the same amount of dispersed black colorant.

Hiding power relative to the standard for each test extender, was determined. Then calculations were made to identify the effective volume of each that would produce the effective PVC ( $\text{PVCe}$ ) required to provide the same relative values using the hiding power equation that was described

earlier, and the constants from the test formulation. This effective volume for each extender, divided by 9.50, was its dilution efficiency (E/d).<sup>17</sup>

As expected, the E/d values ranked in reverse order of particle size; all extenders with a mean particle-diameter greater than about 10  $\mu\text{m}$  were found to yield zero values, while all those below 0.8  $\mu\text{m}$  earned a 1.0 rating. Table 1 shows the values obtained for the calcium carbonate extenders of Figure 1.

No mathematical relationship between size and E/d was postulated, although the values calculated were surprisingly close to being inversely proportional to particle diameter (d), as shown in Table 1.

It will be noted that there may be nothing inconsistent between the earlier interpretation and the present speculation that extender particle bulk exceeding some limiting particle-diameter may be "waste space." It is even a bit easier to conceive how an excessively large spacing might be nonproductive than it is to picture a mixture of particle sizes—some effective spacers, and some not. However, in the end it would make no difference whether 70% of the volume of a common 3.0- $\mu\text{m}$  extender is thought of as waste space, or if 30% of it is identified as "effective volume."

## Conclusions

The common conception that replacement of any  $\text{TiO}_2$  with extenders of larger particle size will result in crowding, has no apparent basis in fact. Both the experimental evidence and geometric analysis suggest that the reverse must be true below the CPVC.

Ultra-fine extenders (finer than  $\text{TiO}_2$ ) are less effective as spacers than those of the same size, or larger, for use below the CPVC.

Above the CPVC, ultra-fine extender enhances high dry-hiding.

There is a limiting extender particle-size above which extender bulk is no longer fully effective as a spacing medium. From experimental evidence, the limiting particle-diameter is approximately four times that of  $\text{TiO}_2$ , or about 0.8  $\mu\text{m}$ .

The previously stated "limit" will undoubtedly vary with extender particles of varying shape, and quite possibly with the width of the particle-size distribution for the extender employed. However, the effect of such uncontrollable variables upon hiding power has been found to be small

enough to permit hiding power to be predicted by computer software<sup>18</sup> with an accuracy well within that of most experimental determinations, for both porous and nonporous systems.

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# Society Meetings

## LOS ANGELES .....SEPT.

### "Hiding Effects of $TiO_2$ "

A moment of silence was observed in memory of Pacific Northwest Society member Emil Iraola, of Olympic Home Care Products Co., who died recently.

The Society officers installed for 1989-90 included: President—Parker Pace, of Behr Process Corp.; Vice President—James F. Calkin, of E.T. Horn Co.; Secretary—Sandra Dickinson, of McWhorter Co.; and Treasurer—James D. Hall, of Sinclair Paint Co.

Jan P. Van Zelm, of Byk Chemie U.S.A., will continue to serve as the Society Representative to the Federation Board of Directors.

Robert Backlin, of Hüls America, Inc., presented the traditional President's gavel to Mr. Pace.

President Pace presented Immediate Past-President Melinda R. Rutledge, of Allo Colouring Co., with a plaque and thanked her for the devotion she displayed during her term of office.

Society Committee Chairmen for 1989-90 were announced as follows: Awards—Ray DiMaio, of Koppers Co., Inc.; Budget and Audit—Maurice Samson, Retired; Computer—Donald I. Jordon, of Cargill, Inc.; Constitution and By-Laws—Henry J. Kirsch, of Trans Western Chem., Inc.; Employment—John C. Plant, of John K. Bice Co., Inc.; Environmental Affairs—Dave Muggee, of E.T. Horn Co.; Entertainment—Robert Oskey, of McCloskey Corp.; Good Fellowship—Clarence Meyers, Society Honorary Member; Library—Al L. Curado, of Dunn-Edwards Corp.; Manufacturing—Anne M. Probizanski, of Ampro Technologies; Meetings—Earl B. Smith, Retired; Membership—Mr. Hall; Nominating—Ms. Rutledge; Photographer and Publicity—V.C. Bud Jenkins, of Ellis Paint Co.; Program and Scholarship—Mr. Calkin; Technical—Santos Delos Santos, of Davis Colors; Textbook—Trevelyan Whittington, Society Honorary Member; and Yearbook—Denese R. Crick, of AC Products, Inc.

Geneva H. Wells, of H.M. Royal, Inc., accepted the Chairmanship of the 1993 Western Coatings Societies' Symposium.

Environmental Committee Chairman Muggee briefly commented on topics of

interest. He stated that an increase of the city's sewer fees is anticipated; the Environmental Protection Agency is cracking down on cities; a Superfund Cleanup Bill has been written by Art Torres; generator fees starting at a base of \$2,400 will be initiated; new Department of Transportation regulations state that corrosive, flammable, or poisonous materials will no longer be listed as NOS; and as of March 1990, the specific product must be listed.

Environmental Subcommittee Chairmen were announced: Architectural—L. Lloyd Haanstra, of Decratrend Corp.; Industrial—Dodwell DeSilva, of Guardsman Products, Inc.; Maintenance—Subash V. Shah, of Koppers Co., Inc.; Manufacturing—Wallace E. Brede, of McCloskey Corp.; Marine Coatings—Joseph A. Pagan, Jr., of Devoe Marine Coatings; and Waste Management—Kenneth J. O'Morrow, of Oil and Solvent Process Co.

The meeting's speaker was Rebecca Craft, of E.I. du Pont de Nemours and Co., Inc. Her presentation focused on "HIDING EFFECTS OF  $TiO_2$ ."

The speaker discussed optics and the use of  $TiO_2$  to generate the kind of optics preferred in a paint system in a way that is efficient. She approached the topic by examining the scientific process to get a better understanding of what is going on in the system. Dr. Craft explained hiding and formulating with efficiency, using theoretical systems and practical ideas (i.e., shaving cream and snow are good for hiding but they are not something we would want on our houses). In conclusion, the speaker focused on particle size for maximum scattering, random packing, and spacer pigments.

*Q. Concerning random packing, would a wider particle size distribution be more efficient at hiding than a narrow distribution range due to more random arrangement in the dry film?*

A. If you think about random packing and you have very large particles, it is like pouring sand into a bucket of marbles. The marbles establish the matrix. If you mix sand and sugar, you will get good random mixing. There is nothing in structure to determine what particle might be next to another. If your particle size distribution is too wide or too different, then you do not get to random spacing.

There are some extenders on the market one-tenth the size of  $TiO_2$  particles.

When you use those to space, you do not get good spacing because  $TiO_2$  establishes its own matrix to fill in and fit wherever they might. You do not know if one  $TiO_2$  is next to another.

On the other hand, if the extender particle gets too large, and too large is around five microns, you can defeat the whole idea of hiding. The particle is so large, light sees it as a big window.

Particle size of  $TiO_2$  will determine how good it is scattering. Particle size and particle size distribution is important to look at in the way it is going to pack. The more narrow the distribution, the better off you are going to be.

*Q. Blanc fixe and aluminum trihydrate are recommended as spacer pigments. Which do you think may work best and why?*

A. I do not know because it depends on what you want to achieve. Some systems blanc fixe because maybe the particle size is smaller and the oil absorption is low. In another system it might be aluminum trihydrate.

SANDRA DICKINSON, Secretary

## MONTREAL .....SEPT.

### "Vinyl Coatings"

The following members were nominated for Society officer positions for 1989-90: President—Gilles Belisle, of National Research Council of Canada; Vice President—Robert Benoit, of NL Chem Can-



SEPTEMBER GUEST SPEAKER—Dr. Rebecca Craft, of E.I. du Pont de Nemours & Co., Inc., discusses "Hiding Effects of  $TiO_2$ ," at the September meeting of the Los Angeles Society

ada Inc.; Secretary—Richard Bordeleau, of Tioxide Canada Inc.; Treasurer—Luc Pepin, of SICO, Inc.; Corresponding Secretary—Bruce Bridges, of Reichhold Canada Inc.

Society Honorary Member Horace S. Philipp, of Supply and Services Canada, will continue to serve as the Society Representative to the Federation Board of Directors.

New York Society member Thomas Ginsberg, of Union Carbide Corp., spoke on "NEW DEVELOPMENTS IN VINYL COATINGS."

The speaker said the use of vinyl coatings in high performance coatings is due to their excellent balance of properties.

He stated that conventional vinyl coatings, however, have low solids.

Mr. Ginsberg said the current drive toward high solids coatings has spurred the development of new vinyl resins that allow the formulation of vinyl coatings, which conform to the requirements of U.S. regulations. In the process of formulation development, considerable progress has been made in identifying new approaches that yield coatings of excellent properties for numerous applications. According to the speaker, progress also has been made in the synthesis and production of vinyls, providing opportunities for cost reduction.

The properties and compatibility of new low molecular weight vinyl oligomers were discussed. Mr. Ginsberg said the devel-

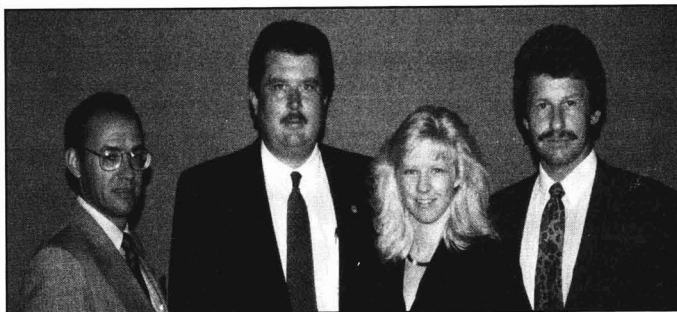
opment of a liquid vinyl resin supplied at 75% solids in methyl ethyl ketone has taken place.

*Q. In the epoxy polyamide-vinyl formulation shown, what trade off of properties could be expected as a result of the vinyl modification?*

A. None. Epoxies, as you know, have excellent alkali resistance but less resistance to acids and vinyls. Therefore, combined, they have been shown to complement each other in this respect and performance equal to urethane-acrylics can be achieved.

G.L. SIMPSON

**ST. LOUIS OFFICERS**—Officers of the St. Louis Society for 1989-90 include (l-r): Immediate Past-President—Robert Wagon; President—Howard Jerome; Secretary—Stanley Sobolewski, Jr.; Assistant Treasurer—Michael Schnurman; Treasurer—Dennis Cahill; Vice President—Terry Gelhot; and Society Representative—John Folkerts



**DALLAS OFFICERS**—Elected to serve as officers for the Dallas Society in 1989-90 are: Treasurer—Leo Michalewicz; Secretary—Harry C. Simmons, Jr.; Vice President—Ronda Miles; and President—Steve Stephens

**BALTIMORE OFFICERS**—1989-90 officers of the Baltimore Society: Vice President—Gary Morgereth; Secretary—Victoria L. Kram; President—Donald Hillard; and Treasurer—Mary Lou Spurrier





# Future Society Meetings

## Baltimore

(Nov. 16)—JOINT AWARDS MEETING WITH BALTIMORE PCA.

## Golden Gate

(Nov. 13)—"SECOND GENERATION COALESCING AGENTS FOR INDUSTRIAL ACRYLIC LATICES"—Dan King, Exxon Chemical.

(Jan. 15)—"WAX EMULSIONS IN WATER-BORNE COATINGS"—Marshall Wiseman, Michelman Inc.

(Mar. 19)—"COUPLING SOLVENT EFFECTS ON WATER-REDUCIBLE ALKYD RESINS"—Rhonda Vance, Dow Chemical.

(Apr. 16)—"THE TRUTH ABOUT ACCELERATED WEATHERING—IT'S SIMPLER THAN YOU THINK"—Douglas M. Grossman, The-Q Panel Co.

(May 14)—"NEW DEVELOPMENTS IN HIGH SOLIDS RESINS AND RHEOLOGY ADDITIVES"—Robert Van Doren, NL Chemicals.

## Kansas City

(Jan. 11)—"NEW SAFETY AND ENVIRONMENTAL REGULATIONS AND HOW THEY AFFECT THE COATINGS INDUSTRY"—Maude Taylor, Buckman Laboratories.

(Feb. 8)—"DEFOAMING AND DE-AIRING"—Jay Adams, Tego Chemie Service USA.

(Mar. 8)—"TANK CLEANING"—Fred Poulos, WashTec. Also Tour of Sinclair & Valentine Ink.

(Apr. 13)—LADIES' NIGHT.

(May 10)—EDUCATION NIGHT.

(June 8-10)—JOINT MEETING OF ST. LOUIS AND KANSAS CITY SOCIETIES. Holiday Inn, Lake of the Ozarks, Missouri.

## Los Angeles

(Jan. 10)—"WAX EMULSIONS IN WATER-BORNE COATINGS"—Marshall Wiseman, Michelman Inc.

(Mar. 14)—"COUPLING SOLVENT EFFECTS ON WATER-REDUCIBLE ALKYD RESINS"—Rhonda Vance, Dow Chemical.

(Apr. 11)—"THE TRUTH ABOUT ACCELERATED WEATHERING—IT'S SIMPLER THAN YOU THINK"—Douglas M. Grossman, The Q-Panel Co.

(May 9)—"NEW DEVELOPMENTS IN HIGH SOLIDS RESINS AND RHEOLOGY ADDITIVES"—Robert Van Doren, NL Chemicals.

## Pacific Northwest

### Portland, Seattle & Vancouver Sections

(Nov. 14-16)—"SECOND GENERATION COALESCING AGENTS FOR INDUSTRIAL ACRYLIC LATICES"—Dan King, Exxon Chemical.

(Jan. 16-18)—"WAX EMULSIONS IN WATER-BORNE COATINGS"—Marshall Wiseman, Michelman Inc.

(Mar. 20-22)—"COUPLING SOLVENT EFFECTS ON WATER-REDUCIBLE ALKYD RESINS"—Rhonda Vance, Dow Chemical.

(Apr. 17-19)—"THE TRUTH ABOUT ACCELERATED WEATHERING—IT'S SIMPLER THAN YOU THINK"—Douglas M. Grossman, The Q-Panel Co.

(May 15-17)—"NEW DEVELOPMENTS IN HIGH SOLIDS RESINS AND RHEOLOGY ADDITIVES"—Robert Van Doren, NL Chemicals.

## Philadelphia

(Nov. 16)—"LOW VOC ALKYDS"—Richard Johnson, Cargill.

(Dec. 14)—70th Anniversary of the Society. "DRIERS FOR LOW VOC ALKYDS"—Marvin Landau, Hüls.

(Jan. 11)—JOINT MEETING WITH PHILADELPHIA PCA. "ECONOMIC FORECAST FOR 1990"—Frederick Dickson, ICI.

(Feb. 8)—"TRENDS IN THE COATINGS INDUSTRY"—Chuck Reitter, *American Paint & Coatings Journal*.

(Mar. 8)—"EPOXY RESINS FOR LOW VOC COATINGS"—William Collins, CIBA-GEIGY Corp.

(Apr. 28)—AWARDS BANQUET.

(MAY)—MANUFACTURING SEMINAR.

### Technical Committee Meetings

(Dec. 4)—"DYNAMIC SURFACE PROPERTIES OF SURFACTANTS"—Paul Berger, Witco.

(Jan. 4)—"SILICONES FOR TODAY'S PAINT INDUSTRY"—Edward Orr, Byk-Chemie, U.S.A.

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## Constituent Society Meetings and Secretaries

**BALTIMORE** (Third Thursday—Snyder's Restaurant, Linthicum, MD). VICTORIA KRAM, Lenmar, Inc. 150 S. Calverton Rd., Baltimore, MD 21223.

**BIRMINGHAM** (First Thursday—Strathallan Hotel, Birmingham, England). BERNARD MYATT, Worrall's Powders Ltd., St. Clements Rd., Aston, Birmingham, England B7 5AH.

**CDIC** (Second Monday—Sept., Dec., Mar. in Columbus; Oct., Jan., Apr. in Cincinnati; Nov., Feb., May in Dayton). JAMES FLANAGAN, Flanagan Associates, Inc., 10999 Reed Hartman Hwy., Cincinnati, OH 45242.

**CHICAGO** (First Monday—alternate between Sharko's Restaurant, Villa Park, IL and Como Inn, Chicago, IL). THEODORE FUHS, General Paint & Chemical Co., 201 Jandus Rd., Cary, IL 60013.

**CLEVELAND** (Third Thursday—meeting sites vary). BEN CARLOZZO, Tremco, Inc., 10701 Shaker Blvd., Cleveland, OH 44104.

**DALLAS** (Thursday following second Wednesday—The Harvey Hotel, Dallas, TX). HARRY C. SIMMONS, Jr., Sherwin-Williams Co., 2802 W. Miller Rd., Garland, TX 75041.

**DETROIT** (Second Tuesday—meeting sites vary). LIANA C. ROBERTS, A.T. Caldas Co., 1985 W. Big Beaver, Troy, MI 48084.

**GOLDEN GATE** (Monday before third Wednesday—alternate between Francisco's in Oakland, CA and Holiday Inn in S. San Francisco). MARGARET R. HARTMANN, Midland Div./Dexter Corp., 31500 Hayman St., Hayward, CA 94544.

**HOUSTON** (Second Wednesday—Sonny Look's Sir-Loin Inn, Houston, TX). JOSEPH CARAVELLO, Guardsman Products, 11502 Charles St., Houston, TX 77041.

**KANSAS CITY** (Second Thursday—Cascone's Restaurant, Kansas City, MO). H. JEFF LAURENT, F.R. Hall, Inc., 1920 Swift Ave., N. Kansas City, MO 64116.

**LOS ANGELES** (Second Wednesday—Steven's Steakhouse, Commerce, CA). SANDRA L. DICKINSON, McWhorter Co., 5501 E. Slauson Ave., Los Angeles, CA 90040.

**LOUISVILLE** (Third Wednesday—Executive West Motor Hotel, Louisville, KY). LLOYD BROWNING, Kelley Technical Coatings, Inc., 1445 S. 15th St., Louisville, KY 40210.

**MEXICO** (Fourth Thursday—meeting sites vary). ANTONIO JUAREZ, Amercoat Mexicana, via Gustavo Baz 3999, 54030 Tlalnepantla, edo de Mexico.

**MONTREAL** (First Wednesday—Bill Wong's Restaurant, Montreal). BRUCE BRIDGES, Reichhold Canada Inc., P.O. Box 120, St. Therese, Que., Canada J7E 4J1.

**NEW ENGLAND** (Third Thursday—Sheraton Lexington Hotel, Lexington, MA). JOSEPH H. WEINBURG, Permutthane Coatings, P.O. Box 3039, Peabody, MA 01961.

**NEW YORK** (Second Tuesday—Landmark II, East Rutherford, NJ). JEFFREY C. KAYE, MacArthur Petro & Solvents Co., 126 Passaic St., Newark, NJ 07104.

**NORTHWESTERN** (First Tuesday after first Monday—Jax Cafe, Minneapolis, MN). DANIEL W. DECHANE, Valspar Corp., P.O. Box 1461, Minneapolis, MN 55440.

**PACIFIC NORTHWEST** (PORTLAND SECTION—Tuesday before third Wednesday; SEATTLE SECTION—the day after Portland; BRITISH COLUMBIA SECTION—the day after Seattle). STEVE REARDEN, Imperial Paint Co., 2526 N.E. Yeon Ave., Portland, OR 97210.

**PHILADELPHIA** (Second Thursday—Williamson's Restaurant, GSB Bldg., Bala Cynwyd, PA). PETER KUMZA, VIP Products Corp., 3805 Frankford Ave., Philadelphia, PA 19124.

**PIEDMONT** (Third Wednesday—Ramada Inn Airport, Greensboro, NC). RUBY JOHANNSEN, Southchem, Inc., P.O. Box 9026, Greensboro, NC 27429.

**PITTSBURGH** (Second Monday—Montemurro's Restaurant, Sharpsburg, PA). JOSEPH POWELL, Union Carbide Corp., P.O. Box 979, Latrobe, PA 15650.

**ROCKY MOUNTAIN** (Monday following first Wednesday—Holiday Inn North, Denver, CO). GARY SCHINGECK, Diamond Vogel/Komac, 1201 Osage St., Denver, CO 80204.

**ST. LOUIS** (Third Tuesday—Salad Bowl Restaurant, St. Louis, MO). STANLEY SOBOLESKI, U.S. Paint Div., 831 S. 21st St., St. Louis, MO 63103.

**SOUTHERN** (GULF COAST SECTION—third Thursday; CENTRAL FLORIDA SECTION—third Thursday after first Monday; ATLANTA SECTION—third Thursday; MEMPHIS SECTION—bi-monthly on second Tuesday; and MIAMI SECTION—Tuesday prior to Central Florida Section). VERNON SAULS, McCullough & Benton, P.O. Box 272360, Tampa, FL 33688.

**TORONTO** (Second Monday—Cambridge Motor Hotel, Toronto). VIK RANA, Ashland Chemicals, 2620 Royal Windsor Dr., Mississauga, Ont., Canada L5J 4E7.

**WESTERN NEW YORK** (Third Tuesday—meeting sites vary). MARKO MARKOFF, 182 Farmingdale Rd., Cheektowaga, NY 14225.

(Feb. 1)—"REGULATORY UPDATE OF FORMALDEHYDE"—Jeffrey D. Felder, Monsanto Chemical Co.

(Mar. 1)—"NOVEL ISOCYANATE FREE CHEMISTRY"—Kenneth Van Dyke, Hüls.

(Apr. 5)—"PIGMENT SYNERGISM"—John Baiker, NYCO.

## Rocky Mountain

(Jan. 8)—"WAX EMULSIONS IN WATER-BORNE COATINGS"—Marshall Wise-man, Michelman Inc.

(Mar. 12)—"COUPLING SOLVENT EFFECTS ON WATER-REDUCIBLE ALKYL RESINS"—Rhonda Vance, Dow Chemical.

(Apr. 10)—"THE TRUTH ABOUT ACCELERATED WEATHERING—IT'S SIMPLER THAN YOU THINK"—Douglas M. Grossman, The Q-Panel Co.

(May 7)—"NEW DEVELOPMENTS IN HIGH SOLIDS RESINS AND RHEOLOGY ADDITIVES"—Robert Van Doren, NL Chemicals.

## St. Louis

(Nov. 21)—"INERTING IN COATINGS PLANTS, SAFETY AND ECONOMY"—Gary Halpern, Neutronics, Inc.

(Jan. 16)—Special Education Night. "TRENDS IN THE COATINGS INDUSTRY"—Chuck Reitter, *American Paint & Coatings Journal*.

(Mar. 20)—"THE TRUTH ABOUT ACCELERATED WEATHERING—IT'S SIMPLER THAN YOU THINK"—Douglas M. Grossman, The Q-Panel Co.

(Apr. 17)—MANUFACTURING MEETING.

(May 15)—Past-Presidents' Night. "TiO<sub>2</sub> UPDATE—DOMESTIC AND WORLD"—Louis Griffis, Kerr McGee.

(June 8-10)—JOINT MEETING OF ST. LOUIS AND KANSAS CITY SOCIETIES. Holiday Inn, Lake of the Ozarks, Missouri.

## Western New York

(Nov. 21)—"PERFORMANCE SOLVENTS FOR HIGHER SOLIDS AND WATER-BORNE COATINGS"—Ron Readshaw, Union Carbide Corp. Scotch'n Sirlolin, Buffalo, NY.

(Dec. 19)—"POLYFUNCTIONAL ISOCYANATE OXAZOLIDONE RESINS"—Bernie Taub, Spencer Kellogg/Reichhold Chemicals, Inc. Fanny's, Amherst, NY.

(Jan. 16)—JOINT MEETING WITH BUFFALO PCA. Buffalo Raceway, Hamburg, NY.

(Feb. 20)—Subject to be announced. Protocol, Williamsville, NY.

(Apr. 17)—"FACTORS OF INFLUENCE IN MATERIAL WEATHERABILITY"—Mike Crewdson, SubTropical Testing Service. Cambria's, Nepew, NY.

(May 15)—"WATER-BORNE COATINGS FOR WOOD"—Theodore DeDonno, Rohm and Haas Co. Shooters Restaurant, Buffalo, NY.

**Robert Jurczynszyn** has been appointed Corporate Manager—Regulatory Affairs by Akzo Coatings Inc., Troy, MI. He will be responsible for managing Akzo's external communications and corporate involvement with regard to health, safety, environmental, and regulatory issues. Mr. Jurczynszyn also will serve as the company's technical spokesman at regulatory agency meetings, hearings, and chemical industry association meetings. He has been with Akzo since 1963 and is a member of the Detroit Society.

Akzo has announced that **Terrance R. Francek** has been named Director—Strategic Planning. He will direct the development of long-range strategic plans throughout Akzo's U.S. coatings operations. Mr. Francek most recently served as Market Manager—General Industrial Business Unit.

Hercules Incorporated, Wilmington, DE, has named **R. Michael Alberts** Manufacturing Manager for six plants producing paper chemicals, resins, coatings, and additives. He will be responsible for production at the Savannah, GA; Franklin, VA; Portland, OR; Milwaukee, WI; Kalamazoo, MI; and Chicopee, MA plants. Mr. Alberts previously was the Product Director of Hercules Paper Chemicals.

Betz Laboratories, Inc., Trevose, PA, has named **Beth Arnold** Marketing Development Specialist for Betz Analytical Services. She will be responsible for developing and planning the marketing/sales program for environmental and industrial testing. Ms. Arnold will be based out of the company's Southern Laboratories, in The Woodlands, TX.

Cookson Pigments, Newark, NJ, announces a series of appointments and promotions within the company. The following personnel changes have been effected: **John P. Rafter**, Vice President—Finance and Administration; **David E. Waldron**, Vice President—Marketing; **Robert F. Mitchell**, Vice President—Operations; **Donald Gray**, Vice President—Research and Development; **John M. Kourouglis**, Production Manager—Manufacturing; **Lawrence Brady**, Production Manager—Inorganics Division; and **Christopher G. Patrignani**, Production Manager—Organics Division.



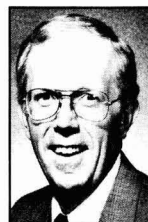
R. Jurczynszyn



R.M. Alberts



M.V. Guzzo



L.D. Pals

**Michael V. Guzzo** has joined Micro Powders, Inc., Tarrytown, NY, as Operations Manager. His primary responsibility will be to coordinate the activities of manufacturing facilities in the United States and the management of domestic sales and executive offices. Mr. Guzzo also will be involved with the management of international sales.

The United States Department of Agriculture (USDA) Forest Service's Forest Products Laboratory, Madison, WI, presented the USDA Superior Service Award to **William C. Feist** at their 43rd Annual Honors Award Ceremony. U.S. Secretary of Agriculture **Clayton Yeutter** presented Mr. Feist the award for his outstanding technology transfer of research information on the proper methods of finishing wood.

Earlier this year, Mr. Feist received the Federal Laboratory Consortium's 1989 Special Award for Excellence in Technology Transfer for his work with the wood finishing industry. Also, the Forest Service presented him with its Excellence in Technology Transfer Award.

Mr. Feist, a Research Chemist with USDA, has been a project leader for wood surface chemistry and protection for the past five years.

Union Carbide Corporation, Danbury, CT, has appointed **Charles R. Kline** Vice President—Operations for the Solvents and Coatings Materials Division. He replaces **Tyler T. Elgin**, who retired. Mr. Kline joined Union Carbide in 1962 and has held various management positions in the chemicals and plastics business areas. He most recently was Director of Operations—Solvents and Coatings Materials Division.

ANGUS Chemical Company, Northbrook, IL, has named **Lowell D. Pals** Vice President and General Manager of its newly established Coatings Business Unit. Mr. Pals is a member of the Chicago Society.

The Specialty Coatings Division of The Dexter Corporation, Waukegan, IL, has appointed **Gerald McComas** Senior Research Chemist—Automotive Coatings. He will be accountable for the development of coatings for plastic and metal automotive/truck applications. Mr. McComas is a member of the Chicago Society.

**Ted E. Walsh** has joined Pico Chemical Corporation, Tinley Park, IL, as National Account Representative—Coatings Maintenance Division. He will be responsible for sales and service activities related to latex emulsion, paint, adhesive, and coatings manufacturers. Mr. Walsh was employed by Union Carbide Corporation for more than 18 years, most recently serving as Services Supervisor—UCAR Emulsion Systems Division.

The American Society for Testing and Materials, Philadelphia, PA, has honored **William H. Safranek** as the 1989 recipient of the Frederick A. Lowenheim Award. Mr. Safranek, Technical Editor for the American Electroplaters and Surface Finishers Society, Orlando, FL, was recognized by Committee B-8 on Metallic and Inorganic Coatings for high competence and professionalism in technology of metallic and inorganic finishes, as exemplified by Dr. Frederick A. Lowenheim. The award was presented by Committee B-8 at ceremonies on May 23, in Kansas City, MO.

**Francis D. O'Neill**, of Teledyne Rodney Metals, was elected to a second one-year term as President of the National Coil Coaters Association (NCCA), Philadelphia, PA. The election took place at the 1989 NCCA Annual Meeting in Marco Island, FL.

Also re-elected to serve one-year terms as Vice Presidents were: **Robert W. Moorman**, of The Glidden Company and **Frank Graziano**, of Pre Finish Metals. **Charles Donnelly**, of Reliance Universal, Inc., was elected to serve another one-year term as Secretary-Treasurer for NCCA.

Dr. Graziano is a member of the Chicago Society.

Morehouse Industries, Anaheim, CA, has appointed **Jim Swartwout** President and Chief Operating Officer. Mr. Swartwout has held management positions at Farr Company and a division of Allis-Chalmers, as well as serving as Executive Vice-President and Chief Operating Officer of Delphian Corporation.

**Herb Waltenspiel** has been appointed Industry Manager of raw materials to the coatings industry for Kraft Chemical Company, Melrose Park, IL. Mr. Waltenspiel has served as a Coatings Development Chemist for Dexter Specialty Chemicals and most recently worked for Rhone Poulenc, marketing specialty chemicals to coatings manufacturers, resin manufacturers, and distributors. He will work out of Kraft's Melrose Park headquarters.

**Herman Jay Lanson**, Honorary Member of the St. Louis Society, was recently featured in "Polymer Science Pioneers"—a series which appeared in *Polymer News*. The series, authored by Prof. Raymond B. Seymour, of the University of Southern Mississippi, cites the contributions of "polymer science pioneers," such as Mr. Lanson.

As noted in the article, Mr. Lanson began his professional career with Nuodex Products Co. and later was employed with Crown Oil Products Co., Grand Rapids Varnish Corp., General Electric, P.D. George Co., and U.S. Vehicle and Chemical Co.

In 1961, Mr. Lanson started his own firm, Lanson Chemical Co. (now LanChem). LanChem was acquired in 1984 by Akzo, but continued to be operated under the director of Mr. Lanson and his son, Elliott.

In addition to serving as an Adjunct Professor of Polymer Chemistry at Washington University, he helped to organize a course on industrial chemistry at St. Louis

The Canadian Paint and Coatings Association, at a meeting of the Board of Directors that followed the 77th Annual Meeting, elected **W.R. "Bill" MacInnis**, of Interlab Paints, Chairman of the Board, and **D.T. "Denis" Wright**, of ICI Paints (Canada) Inc., Vice Chairman of the Board. Both will serve for a one-year term.

PPG Industries, Inc., Pittsburgh, PA, has announced that **Richard Q. La Fond** will assume the position of Vice President—Trade Products, Coatings and Resins Group. Mr. La Fond will have global responsibility for PPG's trade paint business. He previously worked at General Electric Company, Fairfield, CT.

**William E. Mahoney**, Executive Vice-President—Chemical Group, of Witco Corporation, New York, NY, has been elected to Witco's Board of Directors. He has more than 26 years of experience with the company's Humko Chemical Division and has held a series of sales and marketing management positions, culminating in Senior Vice-President—Sales and Marketing for Humko.

**Richard Carney** has been named Technical Sales Representative for Croda Inks Corporation, Niles, IL. He will be headquartered at the company's Cincinnati, OH, location. Mr. Carney's previous experience includes 17 years in the converting/ink industry in various capacities, including management, manufacturing, and sales.

University and has been a long-time lecturer in the coatings short courses of the University of Missouri-Rolla.

Mr. Lanson was a Secretary and member of the Board of Trustees of the Paint Research Institute (now Coatings Industry Education Fund).

A Past-President of the St. Louis Society for Coatings Technology, he was the 1979 recipient of the Society's Gateway Award for meritorious service. Mr. Lanson is a member of the American Chemical Society, American Oil Chemists Society, and Society of Plastics Engineers, and is a fellow of the American Institute of Chemists.

His many achievements include publishing reports on polymer science and technology in technical journals, comprehensive articles on alkyds and polyester resins in *Mark's Encyclopedia of Polymer Science and Technology* and Tess and Poehlein's book on *Applied Polymer Science*, and an article on drying oils for Kirk-Othmer's *Encyclopedia of Chemical Technology*.

**William Bowers** has been named President of Bruning Paint Company, Baltimore, MD. He has held positions in sales management and manufacturing in the trade sales paint industry. Mr. Bowers' most recent assignment was Vice President and General Manager of Bruning's Pompano Beach, FL, paint plant.

In other news at Bruning, **Linda K. Stanofski** was appointed Vice President, Controller, and Treasurer. She has been with Bruning for 15 years, 10 of which have been as Corporate Controller.

Also, **Patricia S. Siano** has assumed the position of Director of Service Operations responsible for all areas of customer service related functions, including transportation.

The U.S. National Committee of the International Commission on Illumination has honored **Calvin S. McCamy**, Vice President—Research for Macbeth Division of Kollmorgen Instruments Corporation, Newburgh, NY. Mr. McCamy was recognized by the Commission for his contributions to international standardization over the past 30 years.

## Obituary

**Emil Iraola**, President of the Pacific Northwest Society, died on August 28. He was 55 years old.

Mr. Iraola began his professional career in the coatings industry as an employee of the Preservative Paint Co. in Seattle. At the time of his death, he served as Senior Technical Service Chemist for Olympic Home-Care Products in Seattle, a position that he held for the past 10 years.

A member of the Pacific Northwest Society for 30 years, Mr. Iraola served on its Educational and Technical Committees. In addition, he was Chairman of the Federation's Joint Coatings/Forest Products Subcommittee.

Mr. Iraola is survived by his wife, Mary, five children, and two grandchildren.

**Eugene Z. Smith**, of Smith Chemical & Color Company, died recently. Mr. Smith was a member of the New York Society.





## VOC Abatement System

Technical information is available on a volatile organic compounds (VOC) abatement system designed to enable compliance with VOC legislation specifying control technology for coating, degreasing, and cleaning manufacturing operations. The system combines carbon adsorption and thermal oxidation to destroy VOCs. For further information, contact Custom Engineered Materials Inc., 4039 Avenida de la Plata, Oceanside, CA 92056.

## Glycol Ethers

A glycol ethers handbook which provides formulators with a practical reference guide to the physical properties of a line of glycol ethers has been published. Also included are specification limits, solubility data, plus toxicological, handling, and storage information. For a copy of the Dowanol™ Glycol Ethers Handbook, contact the Performance Products Dept., The Dow Chemical Co., 2020 Willard H. Dow Center, Midland, MI 48674.

## Polyolefin Products

Slip additives, antiblock additives, mold release agents, antistatic agents, anti-fog agents, and lubricants are among the categories highlighted in a new technical brochure. This eight-page, two-color booklet details products designed specifically for the plastic industry. Contact Guillermo Schnitzler, Technical Service Performance Products, Unichema Chemicals, Inc., 4650 S. Racine Ave., Chicago, IL 60609, for a copy of the brochure, "Products for the Polyolefin Industry."

## Adhesion Technology

Data is obtainable on an enhanced adhesion technology for applying coatings and adhesives to plastics and elastomers. The technology, which uses a fluorination process to treat plastic surfaces, is recommended for adhesive bonding, coating decorating, and printing applications on polyolefins. For more information, write Air Products and Chemicals, Inc., Surface Treated Products, 7201 Hamilton Blvd., Allentown, PA 18195-1501.

## Thickness Measuring Gauges

Nine versions of microprocessor-based, hand-held coating/plating thickness measuring gauges are featured in a preliminary data sheet. Described with color illustrations and specifications, the literature is intended for those in the metal finishing industry. Contact Chris Horvath, CMI International, 2301 Arthur Ave., Elk Grove Village, IL 60007, for more information.

## Packaging Machine

A high-speed volumetric (positive displacement) liquid and semi-liquid packaging machine has been introduced in literature. This series of fillers is available with up to 16 fill nozzles in its dual lane configuration. A variety of nozzles and other features are available as well as automatic lid and cap placement. More information on the Model 984 packaging machine can be obtained by writing Ambrose Co., 20325-71st Ave., N.E., Arlington, VA 98223.

## Refrigerated Circulators

A line of refrigerated circulators, designed to save space and reduce noise emission while still offering the same capacity, versatility, precision and safety found in larger models, has been introduced in literature. These models are suited for the petrochemical, aerospace, electronics, semiconductor, pharmaceutical, and bio-tech industries. For more information on models D1-GH, D8-GH, and F3-CH refrigerated circulators, write to Fisons Instruments, 24911 Avenue Stanford, Valencia, CA 91355.

## Chemical Processing Equipment

A 10-page, full-color brochure featuring a wide variety of filter press plates, with tips on how to choose the proper plate for a particular application has been released. For a free copy of this equipment catalog, contact Bob Sterkel, D.R. Sperry & Co., 112 N. Grant St., North Aurora, IL 60542.

## Viscosity Cup Equivalent Chart

A 24 in. x 36 in. laboratory wall-size viscosity cup equivalent chart that will convert tenths of efflux time seconds to centistokes viscosity has been issued. Several columns across the top of the chart include: K constant, C constant, maximum centistoke range, minimum centistoke range, calibration oil, and percent tolerance for all 29 cups. For information on how to obtain a Viscosity Cup Equivalent Chart, contact Paul N. Gardner, Sr., Paul N. Gardner Co., Inc., 316 N.E. First St., Pompano Beach, FL 33060.

## Analyzer

An automatic nitrogen, carbon, and sulphur analyzer is the subject of recently released literature. The instrument reportedly can simultaneously determine nitrogen, carbon, and sulphur concentrations from as low as 100 ppm to 100%, with a reproducibility factor of better than  $\pm 0.3\%$ . For more information on the NA1500 analyzer, contact Fisons Instruments, 24911 Avenue Stanford, Valencia, CA 91355.

## Filtration Unit

A compact filtration unit with all wetted surfaces constructed of heavy duty noncorrosive phenolic materials is described in literature. The model filters up to 5 ppm and has a 20 gallon reservoir tank. The unit measures 19.5 in. x 43.5 in. x 23.5 in. high. For complete technical details, and a copy of the 24-page "Filter Book," write Industrial Filters Co., Box 823, Fairfield, NJ 07006.

## Industrial Scales

A six-page, four-color booklet outlining over 30 industrial products by application, including parts counting, batching and filling, checkweighing, animal weighing, explosion-proof, and SPC/SQC weight analysis, has been issued. For a free copy, contact Ilene Pfifferling at Sartorius Corp., Weighing Systems Div., 140 Wilbur Place, Bohemia, Long Island, NY 11716.

## Silicate Clays

A line of water-soluble synthetic silicate clays is the topic of a data sheet. The product, available in several grades, is cold-swelling and produces clear, highly thixotropic "gels" which thin upon shearing but quickly reform to their original gel structure. For more information on the Laponite® line of patented, water-soluble synthetic silicate clays, contact Waverly Mineral Products Co., 555 City Line Ave., Bala Cynwyd, PA 19004.

## Cylinder

A cylinder for packaging particulate-free and high-purity gas products is the subject of a recently released data sheet. The literature describes how the cylinder combines the properties of 316L electropolished stainless steel, the surface adsorption of aluminum, and the structural integrity of carbon steel. For more information, request Form #6884 from Liquid Carbonic, Advertising Dept., 135 S. LaSalle St., Chicago, IL 60603.

## Electrically Conductive Topcoat

An electrically conductive fluoropolymer topcoat formulated to dissipate static electrical charge is the topic of a recently released data sheet. Applications include business machine and computer components, printer rollers, photocopier fuser rollers, integrated circuit chip magazines, tote-bins, and wafer boats in electronics manufacturing. For complete technical data on Ultralon® T-99 Conductive Topcoat, including its application and handling, contact ICI Fluoropolymers, 475 Creamery Way, Exton, PA 19341.

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Decatur, Indiana and Leven, Fife, Scotland.

## Training Film

Safe handling of oxidizers by firefighters and hazardous materials response teams during an emergency is the subject of a new videotape, "Oxidizers: Identification, Properties and Safe Handling." As the title suggests, the program deals with the different forms of oxidizers, handling instructions in case of spills and fires, and the hazards associated with these materials. For further information, contact the Chemical Manufacturers Association, 2501 M St., N.W., Washington, D.C. 20037.

## Oleic Acid

A high purity oleic acid derived from natural raw materials is highlighted in recently released literature. The product is available in drum or bulk. Samples and product data are available to qualified interested parties upon request. Contact George Walsh, Customer Service Manager, Unichema Chemicals, Inc., 4650 S. Racine Ave., Chicago, IL 60609, for more information on Priolene 6096 high purity oleic acid.

## Radiation Thermometers Standard

American Society for Testing and Materials' (ASTM) Standard Test Method E 1256 on Radiation Thermometers is now available. This standard was developed by E20.02 on Radiation Thermometry, a subcommittee of ASTM standards-writing Committee E-20 on Temperature Measurement. The test method provides for the first time a basis for testing and calibrating noncontact temperature sensors. For a copy of E 1256, contact ASTM Customer Services, 1916 Race St., Philadelphia, PA 19103-1187.

## Polymer

A data sheet describing an aliphatic water-borne urethane polymer has been released. The product reportedly can be crosslinked and applied using all conventional techniques, and is compatible with many of the commercial emulsion polymers in the marketplace. For more information on Sancure 898, contact Sannco Industries, Inc., 300 Whitney St., Leominster, MA 01453-3209.

## Wire Cloth Chart

A two-color, laminated wire cloth specifications chart is being offered. The handy guide provides market grade and "tensile bolt" wire cloth specifications, mesh count, width of opening, wire diameter, and open area percentages. Measurements are in inches and metric system conversion. For a free copy, write Newark Wire Cloth, 351 Verona Ave., Newark, NJ 07104.

## Fluid Mixers

A six-page full-color brochure describing a company's line of fluid mixers for the laboratory has been issued. The bulletin details five mixers and four laboratory stirrers/mixers, as well as mixer accessories. For a copy of brochure designated B-536, contact Maude Hall, Mixing Equipment Co., 221 Rochester St., P.O. Box 190, Avon, NY 14414.

## Color Measurement Systems

A six-page, four-color brochure detailing on-line color measurement systems which measure color continuously on the manufacturing line has been released. Included is information on applications and options for each system. To receive the Color-Probe™ brochure, write HunterLab, 11491 Sunset Hills Rd., Reston, VA 22090.

## Water-Borne Coatings

The development of two water-borne coatings for aircraft interiors has been announced through literature. These air dry coatings are used on supported fabric and plastic surfaces. The products, code numbers 46 Series (flat) and 47 Series (semi-gloss), are washable and meet Douglas Aircraft specifications DPM 5948 and DPM 5949, respectively. Contact Stan Haske, Crown Metro Aerospace Coatings, Inc., 4343 Temple City Blvd., Temple City, CA 91780, for more technical information.

## Shipping and Storage Container

The availability of a mini-bulk shipping and storage container for hazardous materials has been announced through literature. The container is designed for safe handling of corrosive liquids, flammable liquids, and oxidizers. Contact Clawson Tank Co., 4701 White Lake Rd., Clarkston, MI 48016-0350, for additional information on the Poly Jumbo™ Bin mini-bulk shipping and storage container.

## Surface Preparation Products

A 10-page, four-color guide lists and describes a line of products for surface conditioning operations, those important steps that follow dimensioning, such as refining surface imperfections, preparation prior to bonding, refining before final finishing, and final finishing. For a copy of "3M Surface Conditioning Products for Surface Preparation and Finishing," write 3M, Building Service and Cleaning Products Div., Dept. BC89-10, P.O. Box 33600, St. Paul, MN 55133.

## Electrode Rotator

Technical information is available on an electrode rotator which offers a rotational speed that is adjustable to within 1% of the input setting from 50 to 10,000 RPM. The model is available with quick-change electrode assemblies to enable corrosion scientists to readily study the behavior of many different active electrodes. Contact EG&G Princeton Applied Research, Electrochemical Instruments Div., CN 5206, Princeton, NJ 08543-5206, for additional information on the Model 636 Electrode Rotator.

## Water-Repellent Additive

Technical information is available on a water-repellent additive designed specifically for water-reducible exterior stains and coatings. The stir-in wax emulsion contributes water beading and weather-resistant properties to water based paint, lacquers, stains, and coatings. More detailed information on Aquabead™ 418E additive, can be obtained by contacting Micro Powders, Inc., 580 White Plains Rd., Tarrytown, NY 10591.



**Solution to October's "CrossLinks"**

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Available in the U.K. from: Birmingham Paint, Varnish and Lacquer Club, c/o Ray Tennant, Carrs Paints Ltd., Westminster Works, Alvechurch Rd., Birmingham B31 3PG, England

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## Coatings for Wood and Antifouling Compositions Focus of OCCA Symposiums Conducted in October

The Oil & Colour Chemists' Association (OCCA) sponsored two symposiums in October, "Decorative and Protective Coatings for Exterior Wood," and "Organotin Antifouling Compositions."

The conference on "Decorative and Protective Coatings" was sponsored by the Thames Valley Section of OCCA, on October 12, at the Building Research Establishment, in Garston, Watford, England.

The symposium covered aspects of timber as a substrate for coatings and was directed at manufacturers, specifiers, and people connected with the application of wood finishes. Subjects addressed included the following: complex characteristics of wood as a substrate and the effect of micro-organisms on subsequent coatings; standardization and classification of high and low build stains, colored preservatives and varnishes, and gloss, semi-gloss, and matt opaque coatings; natural finishes; use of factory applied joinery finishes; and views and developments in North America, the United Kingdom, and the rest of Europe.

The Marine Paint Forum of British Maritime Technology and OCCA cosponsored the "Antifouling Compositions" seminar, on October 25, at the General Council of British Shipping, in London, England.

The symposium was organized to assist industry personnel in becoming acquainted with the latest legislation being brought about on environmental pollution problems, monitoring, and methods of prevention. Subjects covered included: the

evidence for environmental pollution; current and proposed legislation in the United Kingdom and EEC; techniques available for the measurement of pollution; long-term monitoring; and guide lines to shipyards on the control of pollution.

### CALL FOR PAPERS

#### 16th International Conference

on

#### "Organic Coatings Science & Technology"

July 9-13, 1990 • Athens, Greece

All prospective authors are invited to submit papers for presentation at the 16th International Conference on "Organic Coatings and Science Technology," scheduled for July 9-13, 1990, in Athens, Greece.

The purpose of the conference is to bring together scientists, engineers, and educators in an international forum to discuss recent research and development work covering all aspects of organic coatings. Leading researchers from various countries are invited to present lectures on topics of interest selected by the Scientific Committee.

Abstracts, no more than one page, on subjects related to the science and technology of organic coatings, must be submitted no later than January 1. Manuscripts are not required. However, speakers may submit manuscripts for publication in the proceedings of the conference no later than March 1.

For more information, contact Angelos V. Patsis, Director, Materials Research Laboratory, CSB 209, State University of New York, New Paltz, NY 12561.

## Papers Sought for ASTM Symposium On Water in Walls, October 25-26, 1990

The American Society for Testing and Materials (ASTM), Philadelphia, PA, invites authors to forward papers for a symposium on "Water in Exterior Building Walls: Problems and Solutions," slated for October 25-26, 1990, in Dearborn, MI.

The symposium, sponsored by ASTM standards-writing Committee E-6 on Performance of Building Constructions, will be held following their October 21-25, 1990, standards development meetings.

The conference will cover the causes of water infiltration into building walls, the consequences of uncontrolled water entry, and methods for evaluating and controlling water penetration. Papers are needed on the following topics: nature of water problems; analytical techniques; consequences of water in walls; design

and construction methods to avoid problems; solutions to water problems; and other related issues, such as the influence of wall materials, including exterior insulation and finish systems.

Prospective authors are requested to submit a title, a 300-500 word abstract, and an ASTM Paper Submittal Form, no later than November 15, to Dorothy Savini, Symposia Coordination, ASTM, 1916 Race St., Philadelphia, PA 19103-1187. Paper Submittal Forms are available from Ms. Savini. A Special Technical Publication based on the symposium proceedings is anticipated by ASTM.

More information is available from Symposium Chairman Thomas A. Schwartz, Simpson Gumpertz & Heger Inc., 297 Broadway, Arlington, MA 02174.

## PDCA to Host 106th Meeting On March 6-9, in San Diego

The Painting and Decorating Contractors of America will host their 106th annual convention and trade exhibit, March 6-9, 1990, in San Diego, CA.

Jack Anderson, syndicated columnist, will deliver the Keynote Address during the convention's opening session. His topic will be, "The News Behind the Headlines."

Over 20 educational sessions are scheduled, including leadership, management, and technical sessions.

The convention's trade exhibit will showcase 200 booths featuring paint and coatings manufacturers, equipment manufacturers, suppliers, and contractor service companies.

For more information, contact PDCA, 3913 Old Lee Highway, Suite 33B, Fairfax, VA 22030.

# Book Review

## ORGANIC POLYMER CHEMISTRY Second Edition

By  
K.J. Saunders

Published by  
Chapman and Hall  
29 W. 35th St.  
New York, NY 10001 (1988)  
ix + 502 Pages, \$47.50

Reviewed by  
Robert F. Brady, Jr.  
Naval Research Laboratory  
Washington, DC

This book is a rigorous and readable study of industrially important polymers, the processes by which they are made, and the uses of which they are put. This book covers the organic chemistry of commercially-significant polymers used in adhesives, coatings, fibers, elastomers, and plastics, and is a wel-

come counterpart to books on polymer chemistry which treat only the physical properties of polymers. The aim of this book is to relate the theoretical concepts of organic chemistry to industrial practice, and this aim is comprehensively and admirably achieved.

The first chapter introduces polymer structure, preparative reactions and techniques, and the historical background of polymers. Following this there are 19 chapters each devoted to class of polymers. Each of these chapters treats the raw materials, preparation, properties, and uses of the subject polymer. The book contains chapters on all of the polymers used in modern coatings, including polyurethanes, epoxies, alkyds, vinyls, acrylics, cellulosic, and phenolic polymers. No coatings chemist will fail to find in this volume a useful discussion of the polymers which interest her or him.

The book is attractively printed and bound. Chemical structures and tables of properties are clearly presented, and an extensive index is provided. This book is strongly recommended to those who wish to obtain or update their knowledge of the organic chemistry of polymers.

## CALL FOR PAPERS

**Western Coatings Societies'  
Symposium and Show  
San Francisco • Feb. 18-20, 1991**

Papers are invited for presentation at the 20th Biennial Symposium and Show of the Western Coatings Societies, to be held at the Hilton Hotel and Tower, San Francisco, CA, February 18-20, 1991.

Topics planned include current and emerging technologies of coatings, as well as regulatory issues. Presentations on the science of practical problem-solving for the small coatings manufacturer are particularly desired.

Address inquiries to Dr. Robert D. Athey, Jr., Chairman, Technical Committee, c/o Athey Technologies, P.O., Drawer 1459, El Cerrito, CA 94530.



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# Coming Events

## FEDERATION MEETINGS

For information on FSCT meetings, contact FSCT, 1315 Walnut St., Philadelphia, PA 19107 (215-545-1506).

1989

(Nov. 8-10)—67th Annual Meeting and 54th Paint Industries' Show. New Orleans Hilton and The Rivergate, New Orleans, LA.

1990

(Apr. 25-26)—Symposium on Color and Appearance Instrumentation (SCAI). Sponsored by the FSCT and the Inter-Society Color Council. Cleveland Airport Marriott, Cleveland, OH.

(May 16-19)—Federation "Spring Week." Seminar on the 16th and 17th; Society Officers Meeting on the 18th; Board of Directors Meeting on the 19th. Galt House, Louisville, KY.

(Oct. 29-31)—68th Annual Meeting and 55th Paint Industries' Show. Convention Center, Washington, D.C.

1991

(Nov. 4-6)—69th Annual Meeting and 56th Paint Industries' Show. Convention Center, Toronto, Ontario, Canada.

## SPECIAL SOCIETY MEETINGS

1989

(Dec. 5)—Cleveland Society 75th Anniversary Dinner Party. Bond Court Hotel, Cleveland, OH. (Charles K. Beck, 75th Anniversary Chairman, 8369 Paddock Court, Mentor, OH 44060).

1990

(Feb. 21-23)—Southern Society. 17th Annual Water-Borne and Higher-Solids Coatings Symposium. New Orleans, LA. (Dr. Robson F. Storey, Department of Polymer Science, University of Southern Mississippi, Southern Station Box 10076, Hattiesburg, MS 39406-0076).

(Mar. 14-16)—Southwestern Paint Convention. Houston and Dallas Societies. Doubletree at Post Oak, Houston, TX. (Neil McBride, P.O. Box 841156, Houston, TX 77284-1156).

(Apr. 4-7)—Southern Society. Annual Meeting. Sandestin Beach Hilton, Destin, FL. (James R. Salisbury, Union Carbide Corp., 2043 Steel Dr., Tucker, GA 30084).

(May 3-5)—Pacific Northwest Society. Annual Symposium. Red Lion Hotel, Bellevue, WA.

(June 8-9)—Joint Meeting of the St. Louis and Kansas City Societies. Holiday Inn, Lake of Ozarks, MO.

1991

(Feb. 18-20)—Western Coatings Societies' 20th Biennial Symposium and Show. San Francisco Hilton, San Francisco, CA.

(Apr. 17-21)—Pacific Northwest Society. Annual Symposium. Empress Hotel and Conference Center, Victoria, British Columbia,

Canada. (John P. Berghuis, NL Chemicals Canada, Inc., 3450 Wellington Ave., Vancouver, B.C., Canada, V5R 4Y4).

## OTHER ORGANIZATIONS

1989

(Nov. 12-18)—"Basic, Intermediate, and Advanced Coating Inspection." Courses sponsored by the National Association of Corrosion Engineers (NACE), New Orleans, LA. (NACE Education and Training, NACE, P.O. Box 218340, Houston, TX 77218).

(Nov. 18-20)—National Decorating Products Show sponsored by the National Decorating Products Association. McCormick Place, Chicago, IL. (Lillian Smysor, NDPA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132-2994).

(Nov. 26-Dec. 1)—"Protective Coatings and Linings." Course sponsored by NACE, ICorrST, and CEA, Berkshire, England. (CEA Education Courses, 174 High St., Guildford, Surrey, GU1 3HW).

(Nov. 27-30)—"Introduction to Coatings Technology." Short Course sponsored by Kent State University (KSU), Kent, OH. (Carl J. Knauss, Director, Cooperative and Continuing Education, Chemistry, KSU, Kent, OH 44242).

(Nov. 28-30)—"Level II—Industrial Maintenance Course" sponsored by KTA-Tator, Inc., Pittsburgh, PA. (KTA-Tator, 115 Technology Dr., Pittsburgh, PA 15275).

(Nov. 28-Dec. 1)—The Inter-Society Color Council Williamsburg Conference. Williamsburg, VA. (Roy Berns, Rochester Institute of Technology, P.O. Box 9887, Rochester, NY 14623-0887).

(Dec. 3-8)—SSPC 89. National Conference and Exhibition sponsored by the Steel Structures Painting Council (SSPC). George R. Brown Convention Center, Houston, TX. (Rose Mary Sargent, Meetings Manager, SSPC, 4400 Fifth Ave., Pittsburgh, PA 15213).

(Dec. 3-9)—"Basic and Advanced Coating Inspection." Courses sponsored by the National Association of Corrosion Engineers (NACE), Houston, TX. (NACE Education and Training, NACE, P.O. Box 218340, Houston, TX 77218).

(Dec. 4-6)—"Electrochemical Techniques for Corrosion Measurement" Symposium sponsored by EG&G Princeton Applied Research. Baltimore, MD. (Ruth Rearick, EG&G Princeton Applied Research, CN 5206, Princeton, NJ 08543).

(Dec. 4-8)—"Fundamentals of Chromatographic Analysis." Short Course sponsored by Kent State University (KSU), Kent, OH. (Carl J. Knauss, Director, Cooperative and Continuing Education, Chemistry, KSU, Kent, OH 44242).

(Dec. 9-10)—"Advances in Surface Coatings and Their Applications." National Seminar sponsored by the Oil Technologists' Association of India (Central Zone), Harcourt Butler Technological Institute, Kanpur. (S. Chandra, Convener, Seminar, Oil Technologists' Association of India (C.Z.), H.B. Technological Institute, Kanpur-208 002).

(Dec. 12-15)—"Getting Into Compliance with Environmental Regulations for Paint and Coatings Facilities." Short course sponsored by UC Berkeley Extension, University of New Orleans, New Orleans, LA. (Continuing Education in Engineering, UC Berkeley Extension, 2223 Fulton St., Berkeley, CA 94720).

1990

(Jan. 22-24)—ASTM Committee E-12 Meeting on Appearance of Materials. Embassy Suites Hotel, Ft. Lauderdale, FL. (Sharon Kauffman, ASTM, 1916 Race St., Philadelphia, PA 19103).

(Feb. 18-21)—Adhesion Society Annual Meeting. Sheraton Savannah Resort and Hotel, Savannah, GA. (Adhesion Society President Jim Wightman, Dept. of Chemistry, VPI & SU, Blacksburg, VA 20461).

(Mar. 13-15)—Electrocoat/90. Drawbridge Inn and Convention Center, Ft. Mitchell, KY. (Cindy Puthoff or Anne Goyer, Gardner Management Services, 6600 Clough Pike, Cincinnati, OH 45244).

(Mar. 25-29)—RadTech '90—North America. Radiation Curing Conference and Exposition. Hyatt Regency Chicago, Chicago, IL. (RadTech International North America, 60 Revere Dr., Suite 500, Northbrook, IL 60062).

(Apr. 2-6)—11th International Corrosion Congress. Florence, Italy. (AIM—Associazione Italiana di Metallurgia, Piazzale Rodolfo Morandi, 2, I-20121 Milano, Italy).

(Apr. 22-24)—Inter-Society Color Council Annual Meeting. Cleveland Airport Marriott, Cleveland, OH. (Program Chairman, James E. Grady, Pigments Dept., CIBA-GEIGY Corp., 7187 White Pine Dr., Birmingham, MI 48010).

(Apr. 23-27)—Corrosion/90 sponsored by the National Association of Corrosion Engineers. Bally's Hotel, Las Vegas, NV. (NACE, Conference Manager, P.O. Box 218340, Houston, TX 77218).

(Apr. 30-May 2)—Surface Coating '90. Sponsored by the National Chemical Coaters Association, The Sheraton Sturbridge Resort and Conference Center, Sturbridge, MA. (Shirley Spears, NCCA, P.O. Box 44275, Cincinnati, OH 45244).

(May 2-9)—Surface Treatment '90. Hannover Fairgrounds, Hannover, West Germany. (Hannover Fairs USA Inc., 103 Carnegie Center, Princeton, NJ 08540).

(May 8-10)—Haztech International Conference and Exhibition. Sponsored by the Institute for International Research. George R. Brown Convention Center, Houston, TX. (Rachelle Scheinbach, Executive Director, Institute for International Research—Bellevue, 13555 Bel-Red Rd., Bellevue, WA 98009).

(May 14-15)—"Analysis of Paints and Related Materials." Symposium sponsored by ASTM Committee D-1. Pittsburgh, PA. (Marsha Firman, ASTM, 1916 Race St., Philadelphia, PA 19103).

(May 21-23)—12th International Conference on "Advances in the Stabilization and Controlled Degradation of Polymers." Luzern, Switzerland. (A.V. Patsis, Institute of Materials Science, State University of New York, New Paltz, NY 12561).

(May 30-June 1)—4th International Conference on "Crosslinked Polymers." Luzern, Switzerland. (A.V. Patsis, Institute of Materials Science, State University of New York, New Paltz, NY 12561).

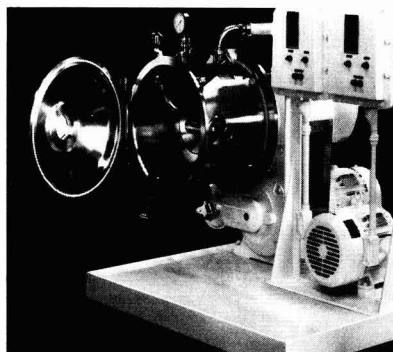
(June 13-15)—Solid Waste and Recycling Technology Conference and Exhibition. Sponsored by the Institute for International Research. Cobo Conference/Exhibition Center, Detroit, MI. (Rachelle Scheinbach, Executive Director, Institute for International Research—Bellevue, 13555 Bel-Red Rd., Bellevue, WA 98009).

(June 18-20)—64th American Chemical Society (ACS) "Colloid and Surface Science" Symposium. Sponsored by ACS Division of Colloid and Surface Chemistry, Lehigh University, Bethlehem, PA. (M.S. El-Aasser, Emulsion Polymers Institute, Lehigh Univ., 111 Research Dr., Bethlehem, PA 18015).

(July 9-13)—16th International Conference on "Organic Coatings Science & Technology." Athens, Greece. (A.V. Patsis, Institute of Materials Science, State University of New York, New Paltz, NY 12561).

(July 23-25)—"Polymer Analysis and Characterization." International symposium sponsored by Du Pont Company and the Czechoslovak Academy of Sciences, Brno, Czechoslovakia. (Howard G. Barth, ISPC Chairman, Du Pont Co., Experimental Station E228/238, P.O. Box 80228, Wilmington, DE 19880-0228 or Josef Janca, Inst. Analytical Chem., Czechoslovak Acad. Sci., Leninova 82, 611 42 Brno, Czechoslovakia).

(Sept. 10-14)—NACE Fall Committee Week/90 sponsored by the National Association of Corrosion Engineers. (NACE, P.O. Box 218340, Houston, TX 77218).



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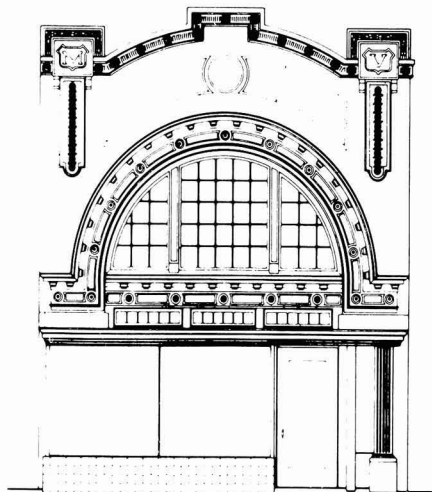
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(Sept. 16-22)—20th FATIPEC Congress. Acropolis, Nice, France. (Jacques Roire, A.F.T.P.V., 5 rue Etex, 75018 Paris, France).

(Sept. 26-28)—Haztech International Conference and Exhibition. Sponsored by the Institute for International Research. Brooks Hall, San Francisco, CA. (Rachelle Scheinbach, Executive Director, Institute for International Research—Bellevue, 13555 Bel-Red Rd., Bellevue, WA 98009).

(Oct. 2-4)—Haztech International Conference and Exhibition. Sponsored by the Institute for International Research. David L. Lawrence Convention Center, Pittsburgh, PA. (Rachelle Scheinbach, Executive Director, Institute for International Research—Bellevue, 13555 Bel-Red Rd., Bellevue, WA 98009).

(Oct. 25-26)—"Water in Exterior Building Walls: Problems and Solutions." Symposium sponsored by ASTM, Dearborn, MI. (Symposium Chairman, Thomas A. Schwartz, Simpson Gumpertz & Heger Inc., 297 Broadway, Arlington, MA 02174).

(Nov. 9-11)—National Decorating Products Show. Sponsored by the National Decorating Products Association (NDPA). Indiana

Convention Center and Hoosier Dome, Indianapolis, IN. (NDPA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132-2994).

1991

(Feb.)—Inter-Society Color Council Williamsburg Conference. Williamsburg, VA. (Louis A. Graham, Lou Graham & Associates, Inc., 1207 Colonial Ave., Greensboro, NC 27408).

(Mar. 4-8)—Corrosion/91 sponsored by the National Association of Corrosion Engineers. Cincinnati, OH. (NACE, Conference Manager, P.O. Box 218340, Houston, TX 77218).

(July 7-12)—Seventh International Conference on Surface and Colloid Science (ICSCS). Sponsored by the International Association of Colloid and Interface Scientists, Université de Technologie de Compiègne, France. (M. Clause, Secretariat of the 7th ICSCS, c/o Wagons-Lits Tourisme, B.P. 244, 92307 Levallois-Perret Cedex, France).

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Federation Officers:

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Patricia D. Viola, Editor

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## 'Humbug' from Hillman

This issue will be devoted to contributions from "old" old friends who have come through many paint smeared years with me and kept in touch.

Seen at almost every New York Society meeting since—since no one can remember when—has been old reliable Moe Bauman—and certainly no "has been." Moe composed the following epic in 1940 when he was a comparatively young bench chemist.

### Formulating Paint

Dazzled and puzzled by my problem today  
Not that it's different, or hard, you may say  
But it never ends; it just isn't funny  
It must be licked or they'll get another dummy.

"Take it easy, it will come your way."  
Maybe your right! I'll plug away  
Then from morning 'till night, the mill goes around  
Wondering, hoping something will be found.

As time goes on, new ideas strike my mind  
This time I'm sure I'm going to find  
A formula that's really great  
And guarantee to be 100% safe.

Hooray! I've found one that looks just right  
Boy, am I happy with all my might!  
For now I can relax; it's all complete  
I think I'll take it easy and take a seat.

Hey there! Get up from that seat  
Yes you! Stand on your feet  
Do you see this product? They say it's the best  
It's a nickel cheaper, and stands a better test.

No use arguing, the other fellow's right.  
Start in working and keep in sight  
That some day you'll find one and lick them all  
And then you'll be on top, and see them fall!

And then there's Ed Raswyk who visited us on our Vermont mountain along with Sid Lauren and Sal Sanfilippo. Add up those years of coatings experience and stories! Ed brought along this newspaper account. Let your imagination supply the bitter end to the story!

### Robber accidentally shoots self, flees

Pittsburgh (AP)—A gas station robbery went awry Wednesday when the bandit accidentally discharged his pistol into his pants, police said.

An unknown male gunman entered a North Negley Ave. gasoline station at about 12:40 am and demanded money, police said.

When the bandit put his pistol back into his belt the weapon fired and the man screamed and ran away. No money was taken.

Milt Glaser, forever young—and I do mean forever—carefully reads his *C&EN News* and found this in August:

Stephen Hirsch of Wayne, NJ, says the current popularity of Broadway's version of *Les Miserables* encouraged him to read the novel. He did so and encountered (on page 1098) an amazing sentence: "Comrades, we will overthrow the government, as sure as there are fifteen acids intermediate between margaric acid and formic acid." This information Hirsch contends is noteworthy for at least three reasons:

- It was known to a nonscientist (Hugo).
- The novel appeared in 1862 and modern organic chemistry was in its formative stages.
- The information is correct!

And, Milt asks: "Have you heard about the oil sheik who cashed a check and the bank bounced?"

We end this column, appropriately, with a prayer from Len Schaeffer who still works, religiously.

Lord, keep me from the habit of thinking I must say something on every subject and on every occasion.

Release me from craving to straighten out everybody's affairs.

Keep my mind free from the recital of endless details—give me wings to get to the point.

I ask for grace enough to listen to tales of others' pains. Help me to endure them with patience. But seal my lips on my own aches and pains—they are increasing and my love of rehearsing them is growing sweeter with the years.

Teach me the glorious lesson that occasionally it is possible that I may be mistaken.

Keep me reasonably sweet; I do not want to be a saint—some of them are hard to live with—but a sour old person is one of the crowning works of the devil.

Give me the ability to see good things in unexpected places, and talents in unexpected people. And give me, O Lord, the grace to tell them so.

Make me thoughtful, but not bossy. With my vast store of wisdom, it seems a pity not to use it all—but Thou knowest, Lord, that I want a few friends at the end.

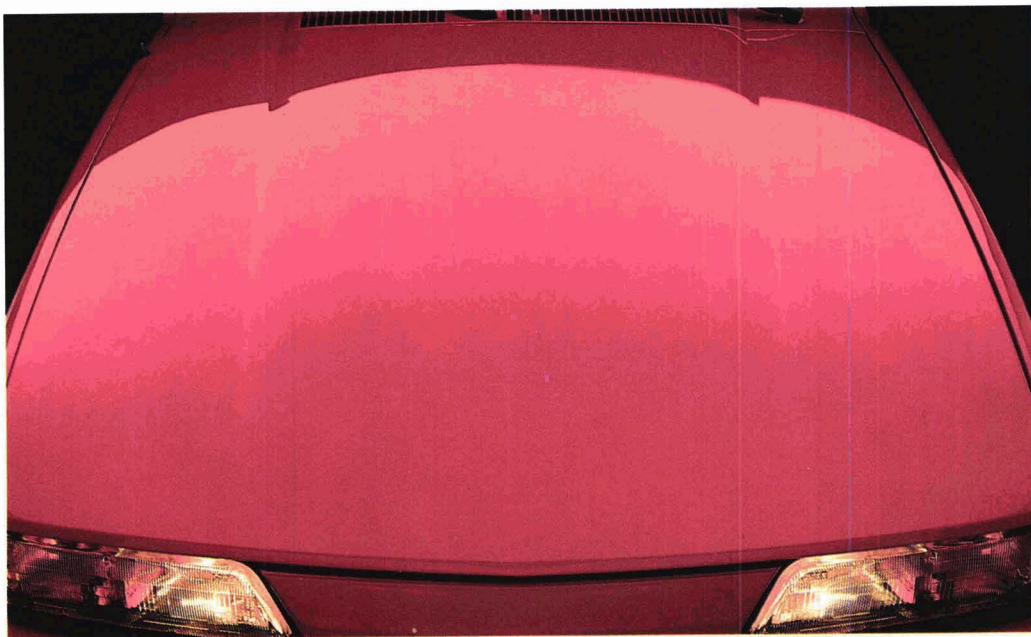
—Herb Hillman

Humbug's Nest

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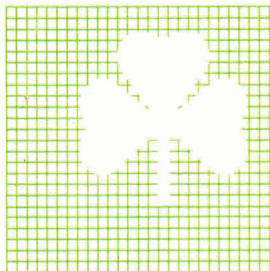


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