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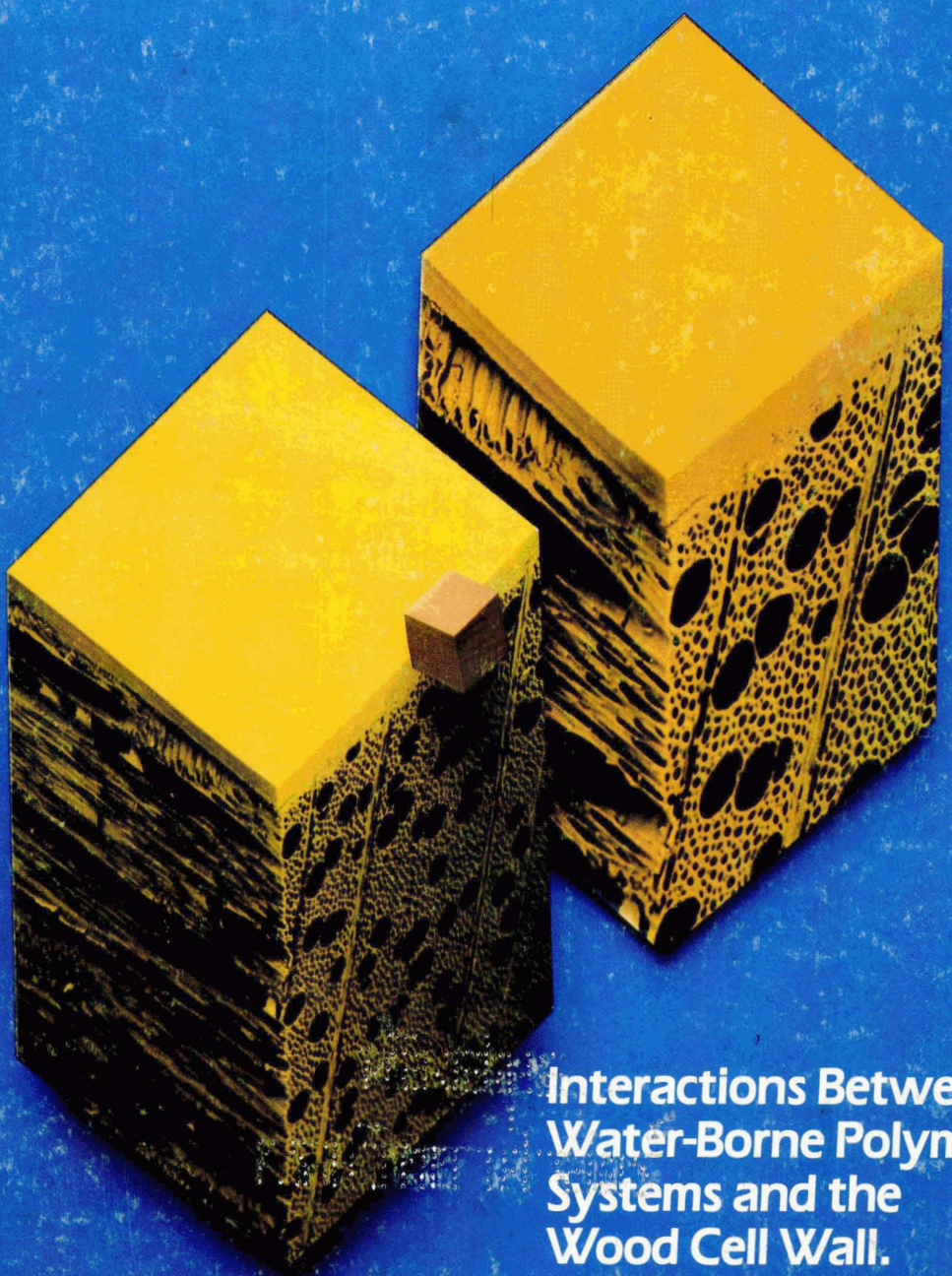
October 1985



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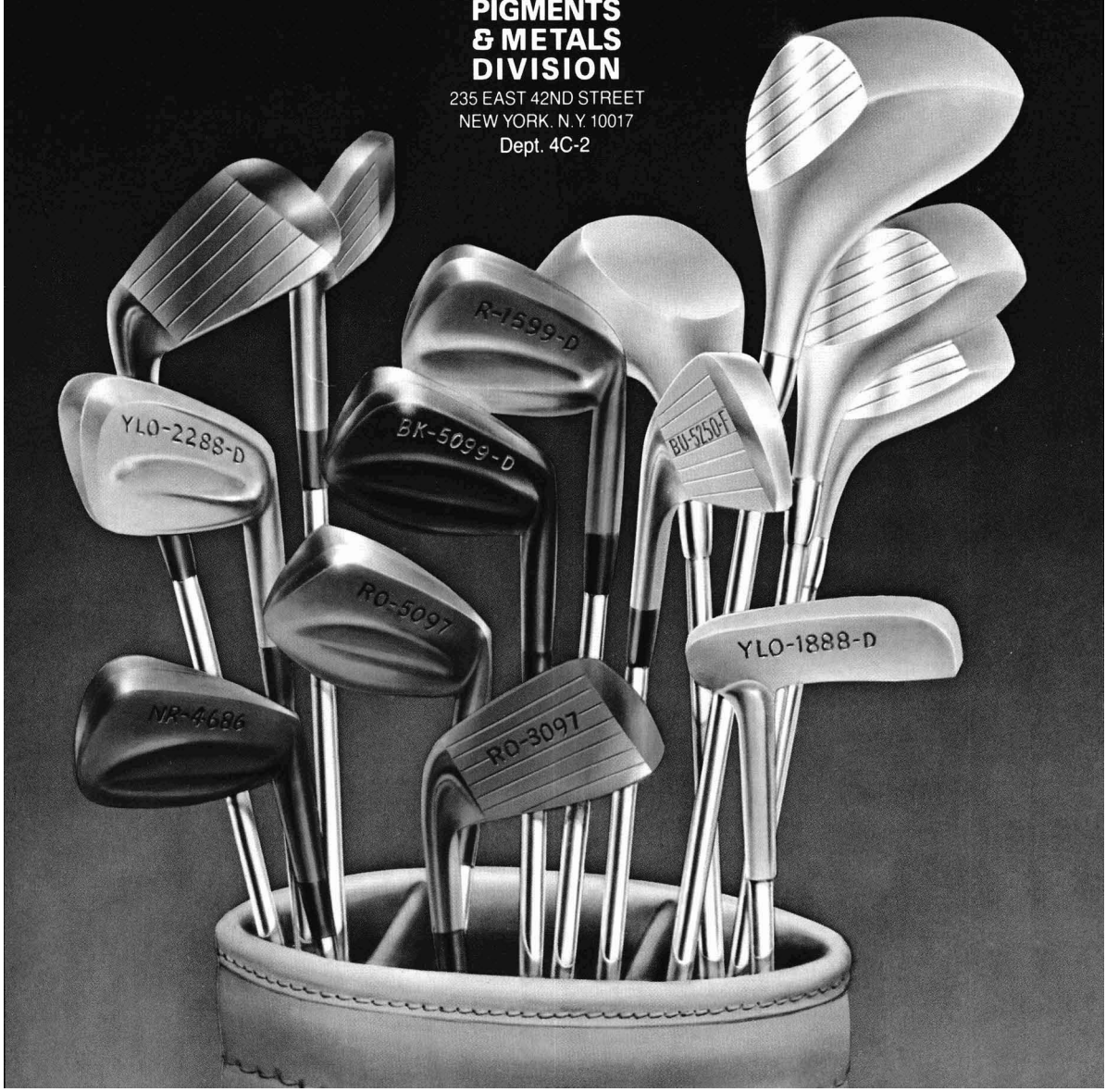

OCTOBER 1985

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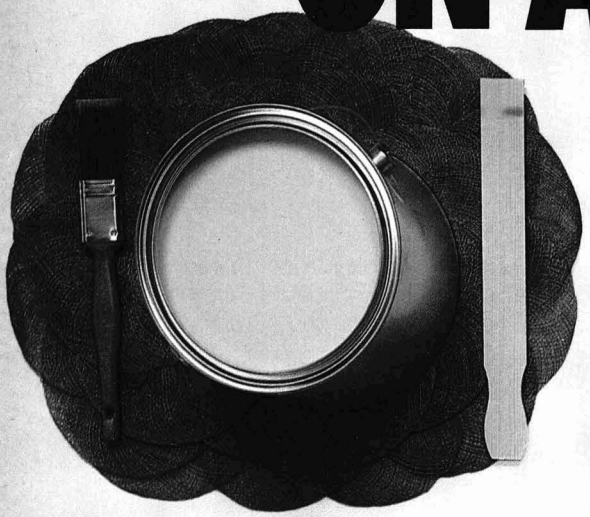

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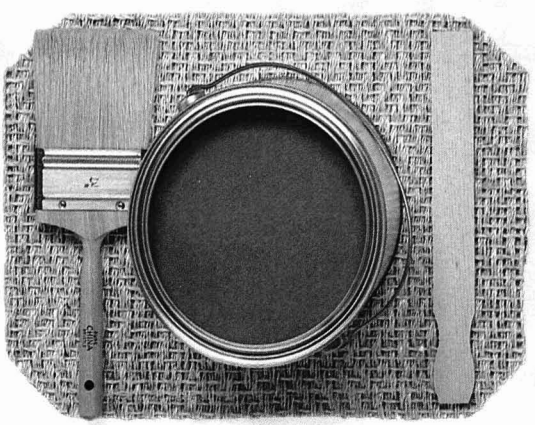

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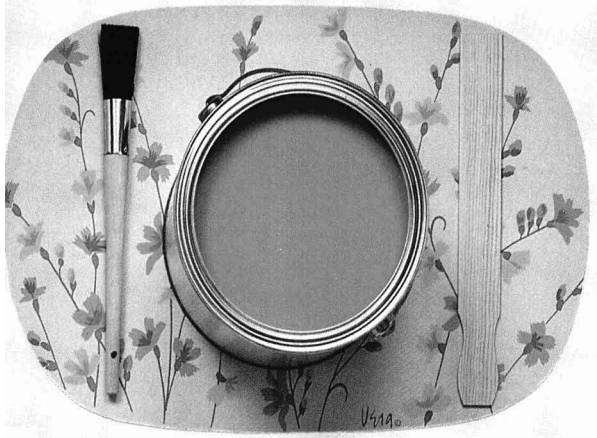

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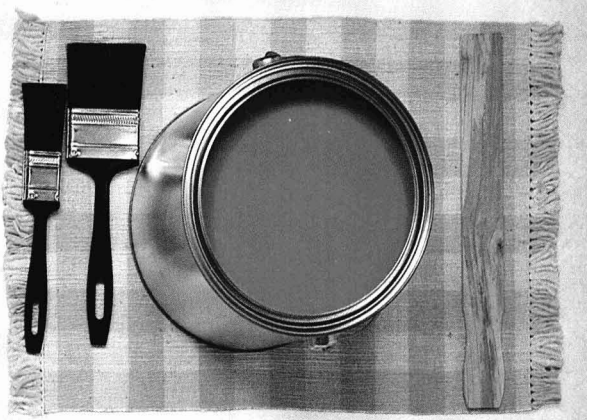

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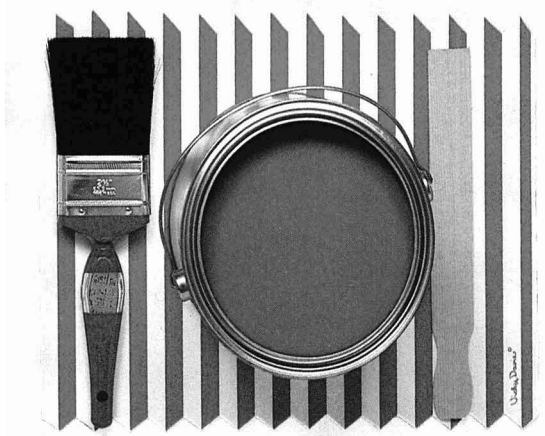

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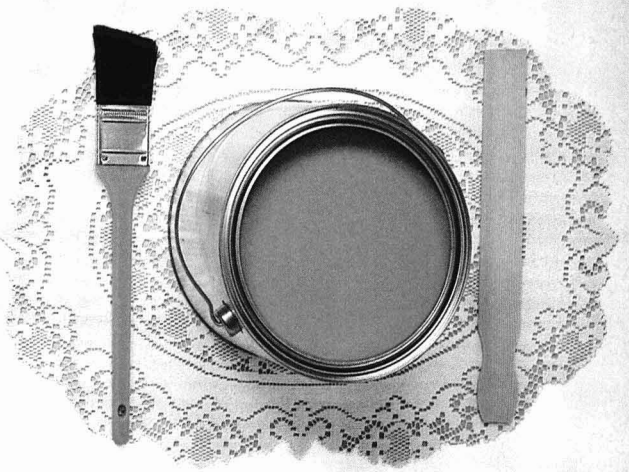

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Modern Times and The Right Stuff

I think we all remember with delight scenes of the great comedian, Charlie Chaplin, starring in the silent film classics. His character, although perplexed, frustrated, and constantly being undone by the machines of his age, perseveres and proves that the human spirit is made of durable stuff.

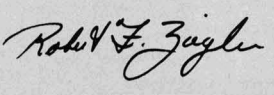
We have all been faced with the prospect of transition, a testing of fundamental beliefs, and compromising with the new. The process, a vital one, is known by such terms as "growth" and "revitalization." In the coatings industry it is known as "survival."

The "Modern Times" faced here is a script containing many characters: government regulation, specialized coating demands, new formulation parameters, antiquated factories, reduced profit margins, and higher costs. No one director can turn this story into a hit; there is no panacea for survival and growth.

However, those who attended the Federation's Annual Meeting and Paint Show this month in St. Louis have a head start in determining the future of their companies. Without exception the technical program presentations reflected the theme of the meeting, "Coatings R&D: Today's Investment in Tomorrow," with heavy emphasis on *investment*. That investment is made not only in dollars and cents, but also in time, patience, and ideas. Complementing the program sessions were the exhibits of 244 companies in the "Big 50" Paint Show. The top technical and sales personnel were available to explain their products and answer questions.

For those who did not attend, the next twelve months will be spent trying to cope with an increasingly heavy burden of problems, whose solutions may very well have been addressed in St. Louis. Not sending a representative to the convention may have cost a company real dollars in the end.

The reality of "Modern Times" is here. How we face the inevitability of change will demonstrate if we have the "Right Stuff."



Robert F. Ziegler,
Editor

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Abstracts of Papers in This Issue

INTERACTIONS BETWEEN WATER-BORNE POLYMER SYSTEMS AND THE WOOD CELL WALL—W.B. Smith, et al.

Journal of Coatings Technology, 57, No. 729, 27 (Oct. 1985)

Interactions between small cross-sectional wafers of basswood and eastern white pine and several water-borne polymer systems and water-soluble organic cosolvents were investigated with the primary interest focused on the penetration of cell-wall capillaries by water-borne polymers. The water-borne polymer systems utilized alkyd, acrylic, and drying oil resins and polyethylene glycol while the solvents were ethylene glycol monobutyl ether and diethylene glycol monobutyl ether. After treatment and redrying, cell-wall capillary penetration was ascertained by comparing the residual volume bulking of water-borne polymer treated wafers and swelling of organic-cosolvent treated wafers to water-treated control wafers.

Results show that wood cell-wall capillary penetration by water-borne polymers is controlled by the molecular size and water solubility of the polymer. The water-soluble solvents, when applied in aqueous solution, both penetrate and swell wood cell-wall capillaries, often becoming entrapped to such an extent that prolonged oven drying does not remove them. With organic cosolvents in the treating solution a definite determination of wood cell-wall capillary penetration by resin polymer molecules was not possible.

STATISTICAL MODEL FOR EMULSION PAINT ABOVE THE CRITICAL PVC—D.A. Holtzen and W.H. Morrison, Jr.

Journal of Coatings Technology, 57, No. 729, 37 (Oct. 1985)

An empirical model that provides cost effective starting point formulations for given hiding power and film properties of a paint was developed. This model reduces significantly the number of experiments required using a conventional trial and error approach. The model was developed for emulsion paints between 50 and 70 pigment volume concentration. The effects of different grades of titanium dioxide pigments and various extenders is also predicted for a given paint formulation.

LOW TEMPERATURE CROSSLINKING STUDIES: INSERTION REACTIONS INVOLVING TITANIUM ALKOXIDES AND ISOCYANATE CONTAINING POLYMERS — J. C. Graham and T.-W. Wang

Journal of Coatings Technology, 57, No. 729, 47 (Oct. 1985)

Linear copolymers containing varying amounts of isocyanate were prepared from isocyanatoethyl methacrylate (IEM) and methyl methacrylate. These polymers were reacted with bis (ethyl-3-oxo-butanolato-0′,0^3) bis (2-propanolato) titanium (Tyzor DC®) under ambient moisture and dry conditions to form crosslinked systems. The course of the reactions were followed using Sward hardness and infrared and nuclear magnetic resonance spectroscopy.

The results of these studies show that although Tyzor DC reacts rapidly with isocyanate containing methacrylate copolymers under both ambient conditions and under nitrogen, the first condition yields appreciably harder coatings as determined by the Sward hardness method indicating a distinct difference in the crosslinking reactions involved. In addition, model reactions between aromatic and aliphatic isocyanates and Tyzor DC in solvent systems revealed a significant steric factor and the reversibility of the insertion reaction at temperatures slightly higher than room temperature.

PHYSICAL AND CHEMICAL CONSIDERATIONS ON COPPER PHTHALOCYANINE—C.C. Wanser

Journal of Coatings Technology, 57, No. 729, 57 (Oct. 1985)

Copper phthalocyanine is a member of a class of highly colored synthetic organic compounds with properties as pigments of excellent strength, chemical, light, and heat resistance. The two most important polymorphs are the beta form (greenish-blue shade) and the alpha form (reddish-blue shade). Halogenated copper phthalocyanines represent the green shades of this pigment. Modifications of copper phthalocyanine by physical and chemical means are made for use in coatings, inks, and plastic systems. Particle size and shape, flocculation resistance, crystal stability, and texture are important considerations in the end-use of these pigments. Chemical and physical variations may change the hue and optical properties in various systems.

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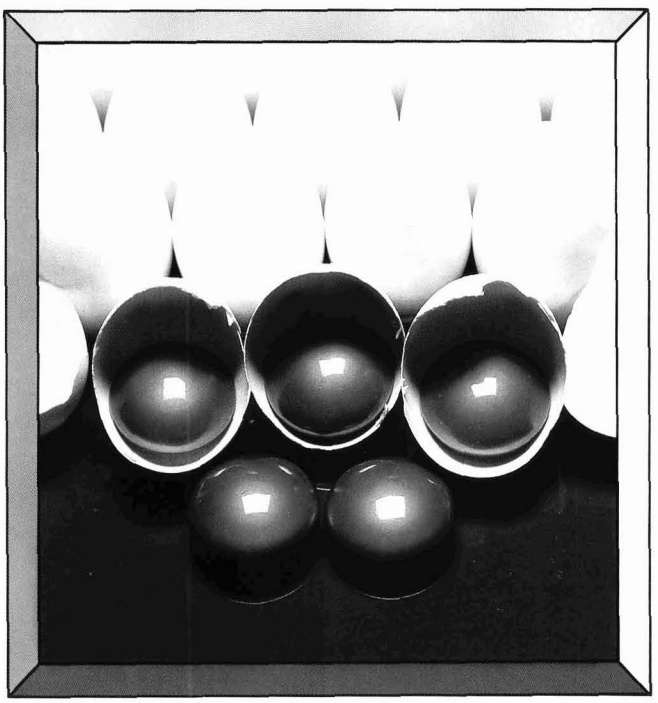

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Demand for Specialty Adhesives and Coatings Expected to Increase Over Next Five Years

Spurred by the rapidly growing fabricated polymer composites business, which is valued at $8 billion in the United States, demand for specialty adhesives and coatings is expected to increase dramatically.

As the use of polymer composites increases due to (1) ease of fabrication, (2) lower weight or superior performance characteristics, or (3) cost savings, there will be a corresponding increase in the use of various specialty adhesives and coatings for these polymer composite parts.

Consumption of all polymer composites are forecast to increase by 8% to 10% a year over the next five years. However, due to the anticipated growth in current applications in addition to new applications, the increase in demand for specialty adhesives and coatings is expected to be even greater over this time period, according to preliminary results from a new market survey by C. H. Kline & Co.

Adhesives provide a means of bonding polymer composites to each other or to such other more traditional structural components as steel, aluminum, or alloys. Coatings typically provide erosion resistance or other protective characteristics as well as being used to pretreat polymer composite surfaces for bonding. To date, adhesives and coatings for polymer composites have found wide use in automotive and aerospace applications. For example, in the automotive industry, adhesives are used extensively for bonding fiberglass reinforced polymers either to themselves or to other metal parts. The Pontiac Fiero and Chevrolet Corvette are two examples of where this technology is employed. Increased use is expected from such emerging technologies as General Motors' Hybrid car (H-body) which is expected to use both metal and polymer exterior panels.

In addition, adhesives and coatings are finding wider use in the aerospace industry as the use of composite parts continue to gain in acceptance. End uses in aerospace include such technologies as seen in the Lear Fan-Jet, reportedly an all-polymer composite aircraft. Further, polymer composites are used in other applications, such as structural wing and frame parts.

Specialty adhesives and coatings for polymer composites are typically formulated with epoxy or urethane resins. These resins exhibit properties ranging from a high degree of flexibility in the bond to a high structural strength. Additionally, such other systems as modified acrylics and elastomerics are employed in selected applications where lower costs or a particular performance characteristic is required.

Spurred by the growing demand for polymer composites, consumption of specialty adhesives and coatings for composite applications will increase by over 10% a year through 1990. Demand will be fueled not only by growth in existing composite applications but also by the continued replacement of metal parts by polymer composites. In addition, the acceptance of bonding technology as opposed to the more traditional metal fastening methods will also increase the demand for these products.

A new study proposed by C. H. Kline will provide a technological and business assessment of this important area of the polymer composites business. The study will look at the major markets in the forefront of using polymer composites, which include aerospace, automotive, construction, electronics, and other markets. Since most of the growth in the use of adhesives and coatings for polymer composites will come from new applications, the report will place particular emphasis on emerging fabrication technology and the impact new applications will have on related adhesives and coatings.

The proposed survey will involve approximately 175 interviews with manufacturers of polymer composites and fabricated parts, suppliers of adhesives and coatings, and end users which assemble finished parts. The report, *Specialty Adhesives and Coatings for Polymer Composites 1985*, is available only by subscription from C. H. Kline and Co., 330 Passaic Avenue, Fairfield, New Jersey 07006.

NPCA Small Paint Manufacturers Breakfast To Focus on Topic of Gainsharing, Nov. 4

"Gainsharing: Is It Right for Your Company?" will be the subject of the Small Paint Manufacturers Breakfast, to be held as part of the NPCA Annual Meeting in New Orleans.

Scheduled for November 4, the breakfast will feature Robert C. Dailey, Associate Professor of organizational behavior at the Tulane University School of Business. He will discuss the emerging concept of gainsharing, which provides financial incentives for employee involvement in operational efficiencies that result in cost savings. Mr. Dailey will also address such topics as how gainsharing differs from profit-sharing, and the role of gainsharing in company success.

NPCA Annual Meeting, Nov. 4-6, To Feature Forum Sessions

The Annual Meeting of the National Paint and Coatings Association will be held in New Orleans, LA, from November 4-6. Among the highlights will be forum sessions on communication, management, and government regulations.

Scheduled for Monday, November 4, Arch Lustberg will discuss improving communication skills. Mr. Lustberg, considered by many to be the country's leading authority on dynamic speech, is the author of "Testifying with Impact" and "Winning at Confrontation," and has produced award-winning Broadway productions, albums, and television series.

On Tuesday morning, November 5, Dr. Thomas R. Horton, President of the American Management Association, will address the topic of "American Management: Fantasies and Realities." Prior to joining AMA in 1982, Dr. Horton had completed a 27-year career at the IBM Corporation, where he pioneered a number of advanced computer applications in such areas as weather forecasting, air traffic control, and space computation.

Tuesday afternoon's forum will be devoted to an update on government regulatory matters, conducted by members of NPCA's Government Affairs staff.

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Macbeth Receives Export Achievement Award

In recognition of its international marketing efforts, Macbeth, a division of Kollmorgen Corp., Newburgh, NY, has received the Governor's Award for Achievement in Export from the New York State Department of Commerce. The award was accepted by Calvin S. McCamy, Macbeth's Vice-President for Research, from Ron Neufeld, Regional Director of the State Department of Commerce.

A manufacturer of color measurement instruments, systems and standards, Macbeth exported 35% of the products it sold in 1984. This figure represents an increase of 2% over the firm's foreign sales in 1983. Macbeth's achievements in export sales were especially notable, Mr. Neufeld said, in view of the strength of the U.S. dollar compared with other currencies which makes it more difficult for American goods to compete with foreign products overseas.

McCloskey Varnish Changes Name

McCloskey Varnish Company, the 131-year-old manufacturer of specialty polymers, resins, and stains for the coatings industry, has changed its name to The McCloskey Corporation.

The Philadelphia-based corporation's additional complexes in Los Angeles and in Portland, OR, are to be designated The McCloskey Corporation-California and The McCloskey Corporation-Oregon, respectively.

Cardolite Purchases 3M Chemical Business

Cardolite Corp., Newark, NJ, is a newly formed company resulting from the management buyout of 3M Company's chemical business derived from cashew nut shell liquid, a naturally occurring alkylphenol. Cardolite now owns and operates the former 3M facility in Newark, NJ, used to produce these chemicals. Most of the products sold by Cardolite Corp. to the friction, coatings, and specialty chemical industries appear under the Cardolite trademark. The firm also manufactures and sells Turpol® rubber additives and Furatone® impregnating resins.

Amoco Enters Fine Acids Markets

Following the recent modification of a process unit at its Decatur, AL, plant, Amoco Chemicals Corp., Chicago, IL, has entered the fine acids markets. The facility, which has been under construction since late 1984, will use Amoco's proprietary oxidation process.

A variety of multifunctional aromatic acids for specialty applications are being produced and test-marketed. "These acids have emerged because new technology demands greater performance," said William A. Miller, Vice-President of Planning and Development. He added, "Amoco is responding to the demands created by new engineering applications, and is helping its customers produce the tailor-made, high-performance materials needed in a competitive marketplace."

Slated for initial introduction are 2,6-naphthalenedicarboxylic acid and its dimethyl ester, and trimesic acid. Also considered for production are meta-toluic acid, phenylindan dicarboxylic acid, 5-tert-butyl isophthalic acid, pyromellitic dianhydride, and a variety of other acids and anhydrides.

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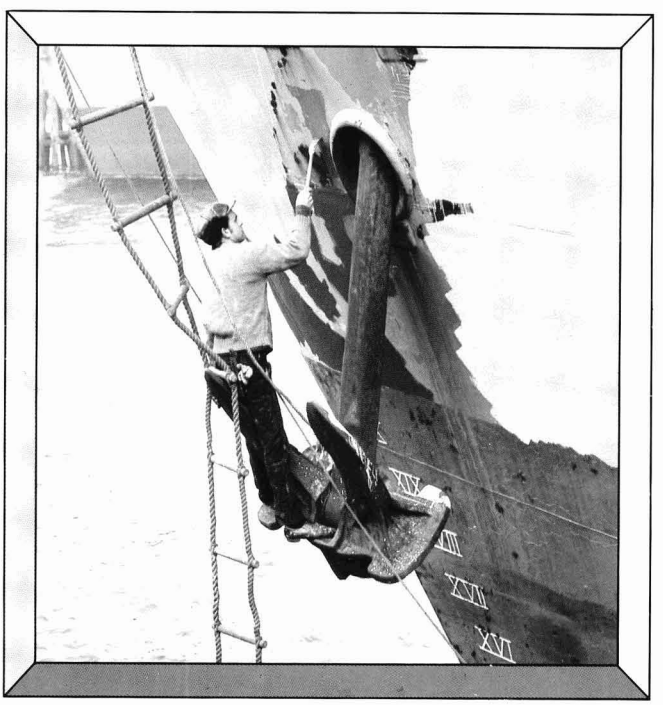

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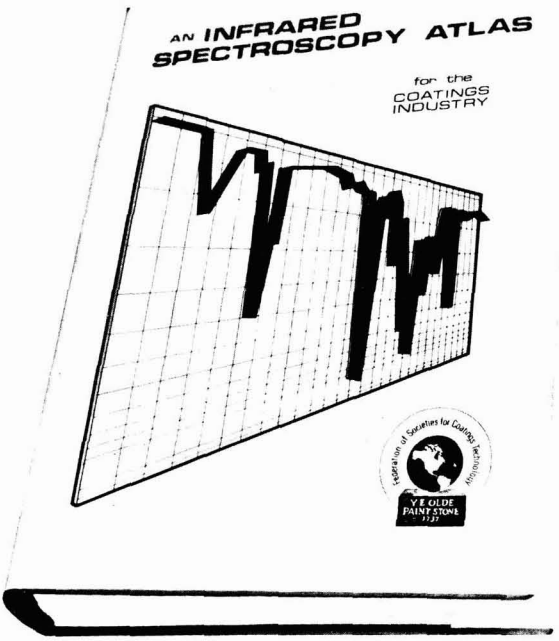

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Environmental Control Update on Hazardous Wastes, Paint Filter Test

New Uniform Manifest For Hazardous Wastes

On September 20, 1984, the regulation mandating the use of a uniform manifest for transporting hazardous wastes went into effect. As part of the Hazardous Solid Waste Act, Congress amended Section 3002 of RCRA to add a new subsection (b) requiring that, effective September 1, 1985, the manifest required by subsection (a)(5) must contain a certification by the generator regarding efforts taken by him to minimize the amount and toxicity of wastes generated. The generator must certify that he has a program in place to reduce the volume or quantity and toxicity of waste generated, to the degree determined by him to be economically practicable, and that the proposed method of treatment, storage, or disposal is that practicable method currently available to the generator which minimizes the present and future threat to human health and the environment.

The new manifest (EPA Form 8700-22) must be used after September 1, 1985. Any generator generating greater than 1000 kilograms of hazardous waste in any calendar month must sign (*by hand*) the certification. Certain small quantity generators of less than 1000 kilograms of hazardous waste per month are exempt from the waste minimization manifest certification requirement, except that if they accumulate on-site more than the above threshold amounts they become subject to the full Subtitle C program (including the waste minimization requirements) for the increment of waste that exceeds these.

There is also a new requirement that generators submit, at least once every two years, a report describing their efforts to minimize waste generation. Also, RCRA permits for the treatment, storage, or disposal of hazardous waste on the premises where the waste was generated must contain a certification by the permittee regarding efforts taken to minimize the amount and toxicity of the generated wastes. The permittee must record the waste minimization certification in the written operating record kept at the facility.

Delisting Hazardous Wastes

EPA has published a guidance document, "Petitions to Delist Hazardous Wastes—A Guidance Manual." This manual is available through the National Technical Information Service (NTIS) at a cost of $19.00 per hard copy or $4.50 per microfiche copy. The NTIS document identification number (for the purpose of ordering) is PB-85-194488. This manual describes the data which should be included in a delisting petition submitted under 40 CFR 260-22 of the RCRA regulation.

The address for ordering the document is: National Technical Information Service (NTIS), 5285 Port Royal Rd., Springfield, VA 22161.

Paint Filter Test

On June 14, 1985, the paint filter test for determining free liquids in solid hazardous waste became effective.

A 60 mesh conical paint filter is used and can be supported by: (1) a ring stand alone; (2) a fluted glass funnel; or (3) a glass funnel with an open mouth that allows at least one inch of the filter mesh to protrude. 100 mL (100 gms if the material is viscous) of the waste is placed in the filter (or funnel) to capture any liquid that passes through the filter. The sample must be placed in the filter for five minutes. If any liquid passed through the filter during the five minute period, the waste would be considered to hold free liquids.

The paint filter test is referred to as "Update II to SW-846" and is available from the Superintendent of Documents, U.S. Government Printing Office (GPO), Washington, D.C. 20401 (202-783-3228) (GPO Number 055-002-81001-2).

SIDNEY J. RUBIN,
Chairman
Environmental Control Committee

FSCT Technical Advisory Committee Set to Meet in October

Saul Spindel, Chairman of the Federation's Technical Advisory Committee, has announced that the committee and Society Technical Committee chairmen will conduct their first fall joint meeting October 29-30. The meeting will take place at the Inntowner Hotel, Madison, Wisconsin.

The two-day conference will include a tour of U.S. Forest Products Laboratory in Madison. The tour will be conducted by Dr. William C. Feist, of the laboratory staff.

Incoming Society Technical Committee chairmen are particularly urged to attend.

ASTM Seeks Participants for Flash Point Group

ASTM's Coordinating Committee for Flash Point and Related Properties seeks additional participants. The committee provides a forum for industry, government, trade associations, standards writing bodies, and other personnel interested in flash point, and coordinates all activities of ASTM technical committees which involve the determination of flash point. For information, contact Committee Chairman J.E. Rogerson, 395 E. U.S. 22-3, Maineville, OH 45039.

CALL FOR PAPERS

Advances in Coatings Technology 29th Annual Technical Conference NASA Lewis Research Center Cleveland, OH

The Educational Committee of the Cleveland Society for Coatings Technology invite all interested persons to submit papers for presentation at the 29th Technical Conference, "Advances in Coatings Technology," to be held in April of 1986.

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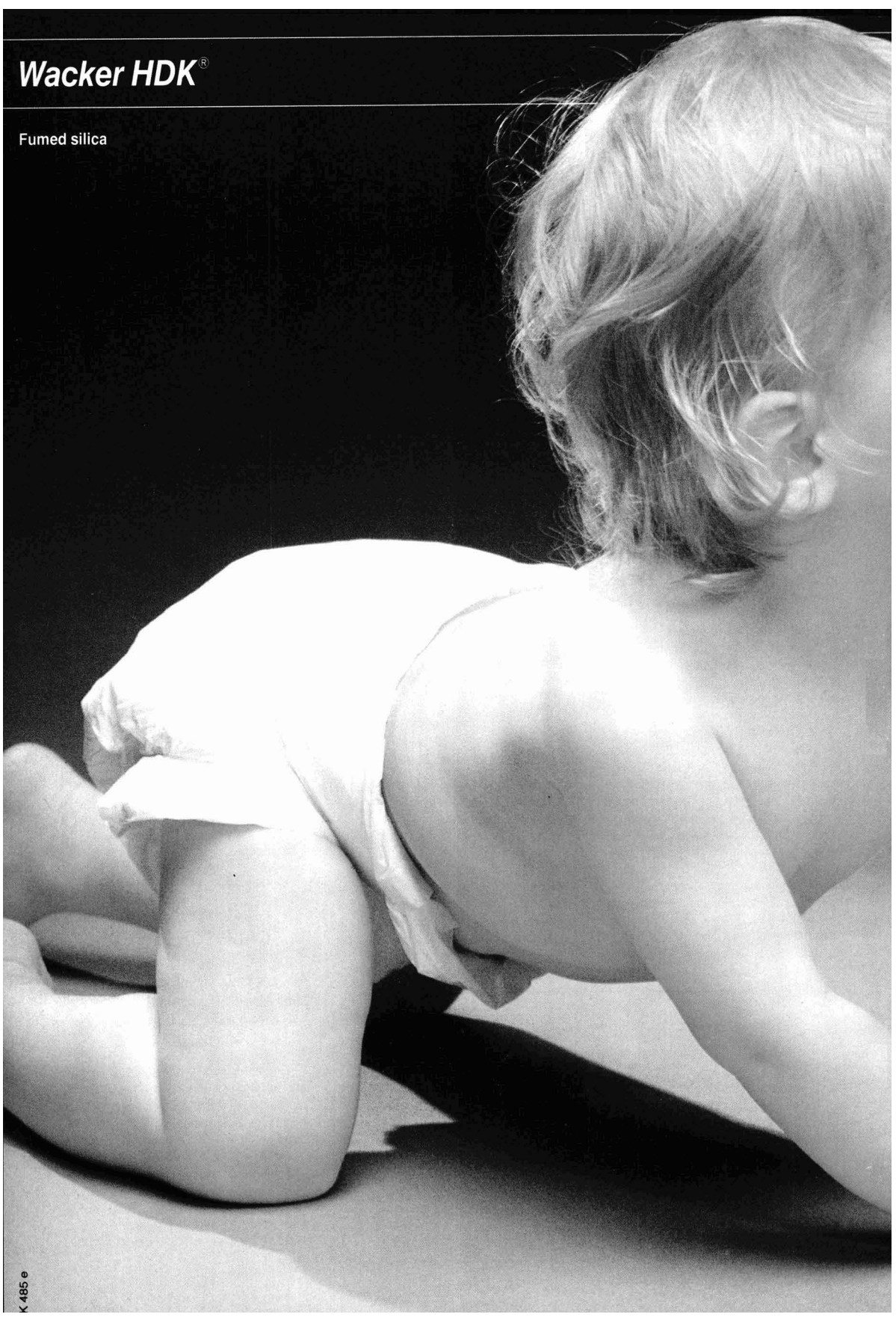

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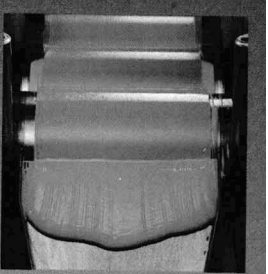

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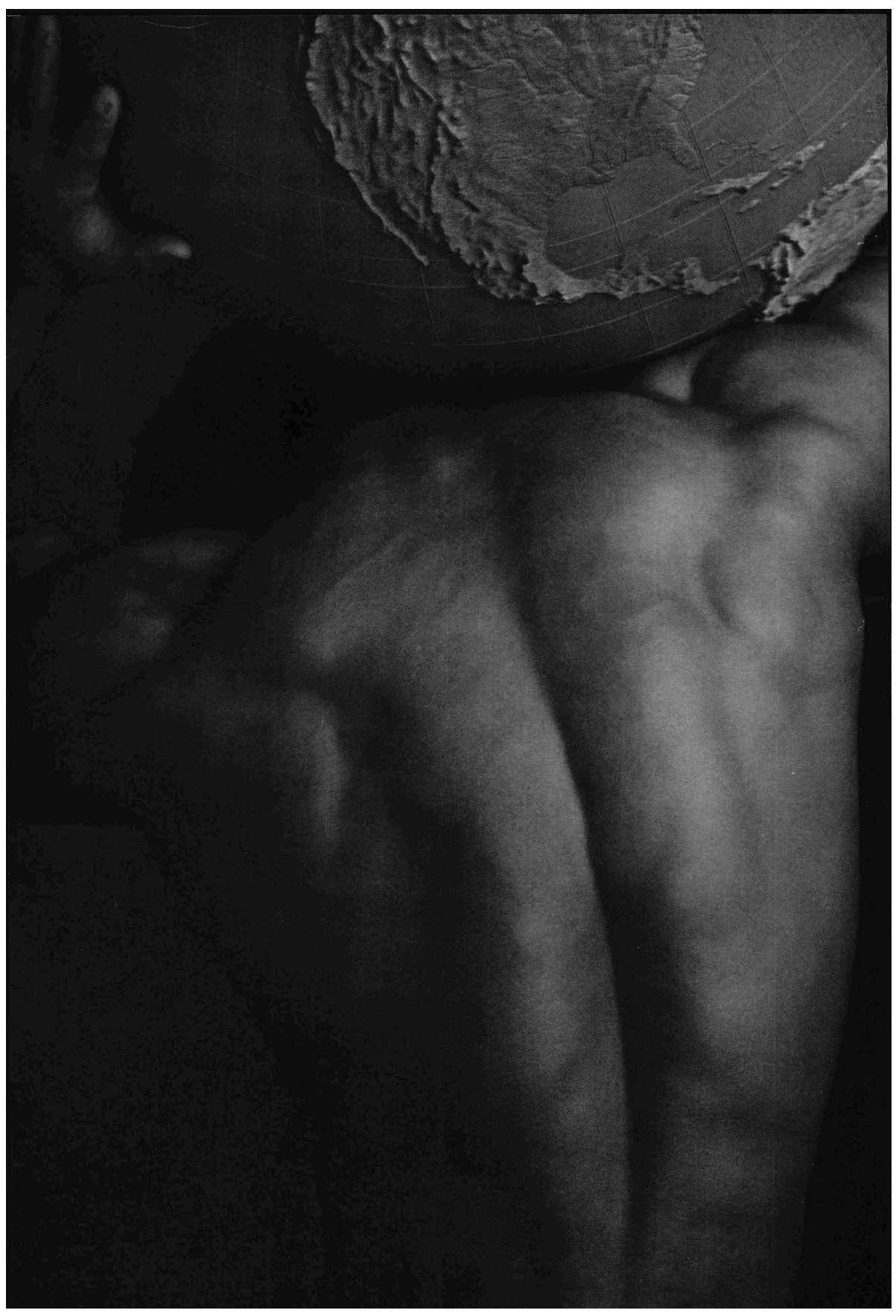

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Interactions Between Water-Borne Polymer Systems And the Wood Cell Wall

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Interactions between small cross-sectional wafers of basswood and eastern white pine and several water-borne polymer systems and water-soluble organic cosolvents were investigated with the primary interest focused on the penetration of cell-wall capillaries by water-borne polymers. The water-borne polymer systems utilized alkyd, acrylic, and drying oil resins and polyethylene glycol while the solvents were ethylene glycol monobutyl ether and diethylene glycol monobutyl ether. After treatment and redrying, cell-wall capillary penetration was ascertained by comparing the residual volume bulking of water-borne polymer treated wafers and swelling of organic-cosolvent treated wafers to water-treated control wafers.

Results show that wood cell-wall capillary penetration by water-borne polymers is controlled by the molecular size and water solubility of the polymer. The water-soluble solvents, when applied in aqueous solution, both penetrate and swell wood cell-wall capillaries, often becoming entrapped to such an extent that prolonged oven drying does not remove them. With organic cosolvents in the treating solution a definite determination of wood cell-wall capillary penetration by resin polymer molecules was not possible.

INTRODUCTION

Wood is a porous, hygroscopic material that exhibits a high degree of swelling and shrinking upon the gain or loss of sorbed moisture. As this characteristic is detrimental in most situations where wood is used, considerable research has been directed toward methods and materials to minimize this moisture-induced dimensional instability. The more effective methods have involved the deposition of polymeric materials within wood cell-wall capillaries.[1] Drawbacks exist, however, in that some of the materials used, such as polyethylene glycol (PEG), remain water-soluble and are subject to leaching when exposed to liquid water, while others, such as phenol formaldehyde, require secondary treatments such as heat to effect polymerization. Traditional wood coating materials such as organic-solvent-borne alkyd resins and drying oils are dissolved in non-polar solvents and, therefore, will not penetrate wood cell-wall capillaries.

Recently, water-borne coatings (water-borne polymer systems) have been developed in which the principal component, the resin, is soluble in water and polar organic cosolvent mixtures. Upon evaporation of solvents and exposure to air, however, these resins polymerize and become water insoluble. An interesting characteristic of these polymer systems is that the resin (an alkyd, acrylic, or drying oil polymer) must be of relatively low molecular weight (under 5,000-10,000) to be soluble in aqueous solutions. Since polymeric materials up to a molecular weight of 3,000 in aqueous solutions penetrate wood cell-wall capillaries,[2] this characteristic introduces a possible wood cell-wall penetration by the resin. This possibility of depositing a water-borne polymer in wood cell walls that can polymerize *in situ* upon simple air exposure creates extraordinary opportunities for wood dimensional stabilization, for excellent wood adhesion in coatings, and for fixing various chemicals such as wood preservatives and fire.retardants within the wood cell wall.[3]

Stamm and Hansen[4] described the following mechanisms by which cell-wall bulking occurs and effects dimensional stability in wood. Upon treatment with

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aqueous solutions of highly water-soluble chemicals, both gross and cell-wall capillaries take up the solution in a concentration only slightly less than that of the original bulk solution. When removed from the solution and allowed to dry, evaporation of water from the gross capillaries occurs, increasing the chemical concentration there, which then promotes further diffusion of the chemical into cell-wall capillaries. Normal wood drying shrinkage is prevented by an amount proportional to the volume of chemical deposited in the cell walls. The degree of dimensional stability attainable in this way is dependent upon the solubility of the bulking chemical and the rate at which the wood is subsequently dried. Cell wall penetration and bulking by resins soluble in polar organic solvents should occur by this same process, so long as those polar solvents have the ability to swell wood.

Bulking treatments that stabilize wood dimensionally were first demonstrated with inorganic salt solutions by Stamm.[5] Salts, however, are not feasible practically because of such drawbacks as water leachability, corrosiveness to machine tools and fasteners, and relatively low solubility in water. Stamm[6] also studied sugars as wood bulking agents. They are better than salts because of their greater solubility in water and noncorrosiveness, but they make wood more susceptible to decay.

One of the most effective cell-wall bulking chemicals, to date, is polyethylene glycol. Its potential as a wood stabilizer was first reported by Stamm.[7] He found that Sitka spruce cross-sectional wafers saturated with 25 percent by weight aqueous solutions of several polyethylene glycols, of molecular weights 1000 (PEG-1000) and less, and dried slowly, exhibited no shrinkage. This indicated virtually complete replacement of cell-wall capillary bound water with polyethylene glycol molecules.

Water-insoluble chemicals can be utilized as wood bulking agents if a replacement process is used to deposit them within the cell-wall structure. An example of this is the replacement of water in swollen wood with ethylene glycol monoethyl ether (EGMEE) followed by the replacement of the solvent by a wax or fatty acid.[4] Such treatments achieve virtually complete bulking and are non-leachable in water. Limitations, however, include the difficulty in carrying out the replacement process and the fact that the treated wood will not readily accept adhesives or finishes.

Recent advances in polymer chemistry have led to the development of water-soluble and water-dispersible alkyd, acrylic, and drying-oil binders that rival the performance of the more traditional organic-solvent-borne coatings.[8,9] Being dispersed in water-miscible polar solvent/water mixtures, as opposed to non-polar hydrocarbon solvents, these resins have the potential to penetrate the wood cell wall, which should improve adhesion of coatings to wood and the performance of wood treatments. Latex dispersions, although they are water-borne polymers, are not of interest in this study because even the smallest latex particles are too large to penetrate cell-wall capillaries.

Resin systems, whether used as coatings or as penetrating treatments for wood, generally require that the resin be diluted to between 5 and 50 percent non-volatile prior to application. With traditional solvent-reducible resins this is accomplished simply by adding the desired amount of a solvent such as mineral spirits. Water-borne systems, however, are more complex. They often require the addition of both neutralizing amines and organic cosolvents in relatively specific proportions to maintain solubility.

One reason for complexity in these water-borne systems is that they may not exist as true solutions. Solomon,[10] among others, believes that many of the so-called water-soluble polymers, rather than forming true solutions in water, exist as colloidal dispersions that appear clear because the particle size is below that visible to the eye. It is for this reason that the resins in these systems are more properly referred to as water-reducible, water-dispersible, or water-borne instead of water-soluble.

The diameter of wood cell-wall capillaries imposes a restriction on the size of molecules that can penetrate the wood cell wall. While direct visual measurement of their size has not been possible, a number of investigators have reported limiting dimensions based upon indirect observations, ranging from 32-1000 Å (3.2-100 nm) in diameter.[11-15]

Table 1—Description of Water-Borne Polymer Systems

Polymer System	% Non-volatile Resin in System	% Cosolvent in System	Acid Value	pH	General Description of Polymer
A	25	11.5 EGMBE	50	8-8.5	Water-borne medium-oil-modified polyurethane for interior use
B	25	9.8 EGMBE 1.7 n-butyl alcohol 3.8 isobutyl alcohol	50	8-8.5	Water-borne medium-oil alkyd for exterior use
C	25	15.0 EGMBE	85-95	7.5-8.3	Water-dispersible linseed oil polymer
D	25	—	120	9.0	Low-molecular-weight acrylic leveling aid for floor polish
E	—	—	—	9.0-9.5	Acrylic-latex vehicle
30% D and 70% E	25	—	—	—	
F	25	16.6 DEGMBE	78	—	Proprietary experimental resin air-curing soluble polymer
G	—	—	5-6	—	High-quality traditional organic-solvent borne polyurethane for interior use

Table 2—Polymer Molecular Size

Polymer	Refractive Index $\bar{M}n$	Refractive Index $\bar{M}w$	Refractive Index $\bar{M}w/\bar{M}n$	U.V. Absorbance $\bar{M}n$	U.V. Absorbance $\bar{M}w$	U.V. Absorbance $\bar{M}w/\bar{M}n$
A	1400	2500	1.8	1400	4300	3.1
B	1200	4400	3.6	1400	11400	8.4
C	1200	2400	2.0	1050	2900	2.8
D	750	850	1.1	—	—	—
F	1800	31900	17.8	1200	27700	23.1
G	6400	46500	7.3	10700	66100	6.2

Tarkow, et al.[2] treated Sitka spruce heartwood cross sections with aqueous monodisperse PEG solutions varying in molecular weight from 200 to 20,000. By using a solute exclusion technique they found the limiting size for cell-wall capillary penetration was approximately equal to the size of a PEG-3000 molecule. Accepting that PEG-3000 assumes a random coil conformation in aqueous solution with a radius of gyration of 18-20 Å (1.8-2.0 nm), the diameter of swollen cell-wall capillaries should be approximately 40 Å.

Robison[16] analyzed interactions between a brominated alkyd resin and basswood. Using scanning electron microscopy (SEM) and energy-dispersive analysis of X-rays (EDAX) he found evidence of cell-wall penetration by the alkyd resin. Based upon the molecular weight and intrinsic viscosity of the resin he calculated a 32 Å diameter limiting cell-wall capillary pore size for basswood.

To determine experimentally whether low-molecular-weight resin polymers penetrate the wood cell wall, volume changes were studied. Small cross-sectional wafers of basswood and eastern white pine were vacuum impregnated with aqueous/organic-cosolvent solutions and water-borne polymer systems. The treated wafers were then subjected to exposure conditions such as air drying, oven drying, vacuum-oven drying, high humidities, and liquid water. If a treated wafer remained in a swollen or "bulked" state, when compared to a control wafer after various exposure conditions, it was considered to have polymeric material deposited in its cell-wall capillaries. The validity of this method for determining cell-wall capillary penetration is well documented.[2,17,18]

EXPERIMENTAL

Green, never-dried basswood (*Tilia americana* L.) and eastern white pine (*Pinus strobus* L.) boards were machined into square billets approximately 20 mm on a side, with the growth rings oriented parallel to one edge. Cross-sectional wafers, approximately 4 mm in thickness, were cut sequentially from these billets and assigned to treatment groups for each bulking experiment. For example, if there were 10 treatment groups with five wafers per group in a particular bulking experiment, 50 wafers were cut from a billet and assigned to each group in the following manner: group 1, wafers 1, 11, 21, 31, 41; group 2, wafers 2, 12, 22, 32, 42; group 3, wafers 3, 13, 23, 33, 43; and so on. Bulking information was obtained by measuring and comparing volume changes exhibited by wafers in particular treatment groups upon subjection to exposure conditions such as air drying, oven drying, vacuum-oven drying, high humidities, and leaching with liquid water. A mercury immersion procedure was used to determine volumetric changes. This procedure involved placing a jar of mercury on an electronic balance, submerging a wafer completely into the mercury, and recording the weight of mercury displaced by the wood wafer. The wafer volume was obtained by dividing the displaced weight by the density of mercury. Wafer weights were determined to 1 mg on an electronic balance. Impregnation of wafers with solvents or water-borne polymer systems was accomplished by vacuum impregnation followed by soaking for three days to allow material to diffuse into cell-wall capillaries.

Since molecular weight is such an important limiting factor for polymer penetration of wood cell-wall capillaries, gel permeation chromatography (GPC) was utilized to characterize accurately the molecular weight distribution of the resin polymers in question. GPC works on the principle that a polymer molecule in solution has an "effective size" that is closely related to the molecular weight of the polymer.

Four Waters Associates, Inc. μ-Styragel packed columns with the following porosity designations, 100Å, 500Å, 1,000Å, and 10,000Å, were utilized with tetrahydrofuran (THF) as the eluting solvent at a flow rate of 2 mL per minute. Because different size molecules in a particular polymer may have more or less UV absorbance than others, both refractometer and UV absorbance detectors were used in this study to provide a more complete characterization of the polymers.

Intrinsic viscosity is one of the most accurately measurable properties of a polymer in solution and can be used to estimate the effective size or hydrodynamic volume a polymer has in different solvents. It was used in this study because while the effective size of a polymer in THF may be small enough to allow cell-wall penetration, that same polymer when dissolved in an aqueous/organic cosolvent solution may swell to such an extent that penetration would be precluded. The intrinsic viscosity of the polymers in their treating solvents is important, therefore, because while the GPC molecular weight determinations had to be made in tetrahydrofuran, wood is treated with polymer systems in aqueous/organic cosolvent solutions and wood cell-wall capillary pore openings are only exposed to polymer molecules in these aqueous/organic cosolvent solutions.

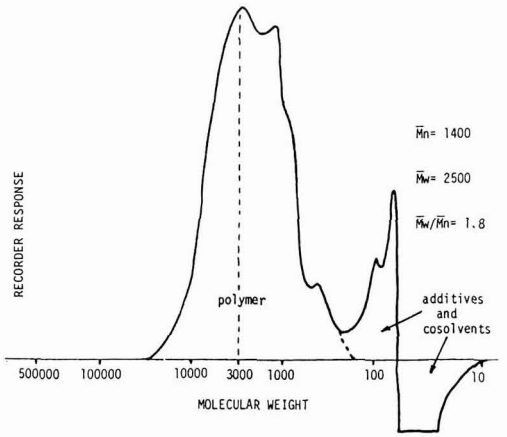

Figure 1—Polymer system A molecular weight, R.I. trace

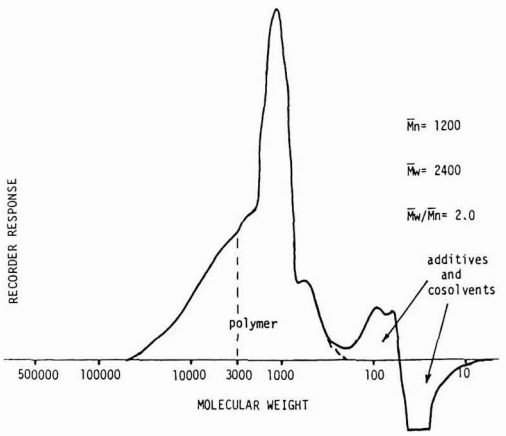

Figure 3—Polymer system C molecular weight, R.I. trace

One of the characteristics of water-borne polymer systems is the presence of relatively low-molecular-weight binder resins.[19] A variety of water-borne polymer systems were tested in this study in an attempt to determine if cell-wall capillary penetration by a water-soluble binder resin does occur, and if it does, whether the resin will cure *in situ* and become non-leachable. *Table* 1 gives a brief description of the polymer systems used while GPC molecular weight distribution information of particular polymers can be found in *Table* 2 and *Figures* 1-4. Characteristics of Polymer System G are included in *Tables* 1 and 2 to allow a molecular size comparison of a traditional organic-solvent-borne polymer to the new water-borne polymers. All of the polymer systems were applied at approximately 25 percent non-volatile or resin solids concentration.

In addition to the various polymer systems being tested, water, 20 percent EGMBE (ethylene glycol monobutyl ether), 20 percent DEGMBE (diethylene glycol monobutyl ether), and 25 percent PEG-1540 were tested as controls. The concentration of 20 percent by weight for the two cosolvents in aqueous solution was chosen because the 20/80 organic cosolvent/water ratio is commonly used with water-borne polymer systems. The PEG-1540 was used as a known wood-cell wall bulking material. Although it does not have the popular reputation of PEG-1000, it was shown by Stamm[4] to be very close to PEG-1000 in wood bulking capability. In addition, having a somewhat larger molecule, PEG-1540 should more closely duplicate the cell-wall bulking that could be found with an alkyd or acrylic resin.

All of the water-borne polymer systems tested are what would be referred to as water-soluble, water-reducible, or water-dispersible, depending upon individual custom. One of the systems, a 30/70 mixture of Polymer System D and Polymer System E (*Table* 1), utilized a combination of a high-molecular-weight emulsion with a low-molecular-weight water-soluble vehicle. Hortensius[20] reported that a combination such as this performed well as a wood coating. In this case it is anticipated that the low-molecular-weight resin, System D, will penetrate and bulk wood cell-wall capillaries and the high-molecular-weight emulsion vehicle, System E, will form a film on cell lumen surfaces to prevent the low molecular weight material from being leached.

Because these polymer systems are water-borne and have materials such as organic cosolvents, previously

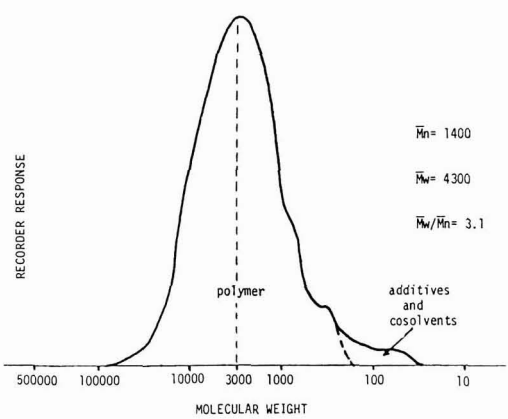

Figure 2—Polymer system A molecular weight, U.V. trace

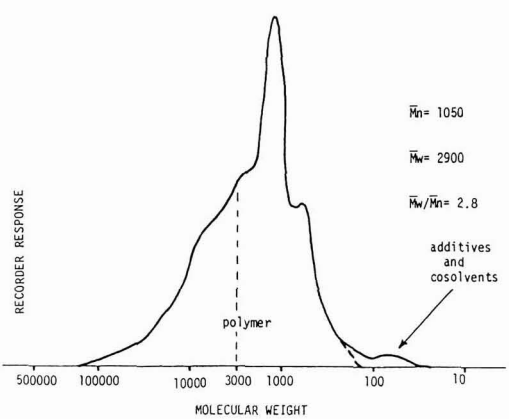

Figure 4—Polymer system C molecular weight, U.V. trace

shown to interact with wood cell-wall capillaries,[21] one would expect that ambient temperature and humidity conditions could have an effect on cell-wall bulking. In light of this possibility, relative humidity and temperature were controlled and observed during this study.

It has often been suggested that the maximum cell-wall-capillary penetration occurs when green wood is treated.[22,23] Oven-dry wood, however, is preferred for cell-wall penetration studies because it provides a convenient base for weight and dimensional measurements. The question arose, therefore, as to whether oven drying irreversibly constricts wood-cell-wall capillaries to such an extent that they do not allow the passage of polymer molecules. To answer this question, basswood and white pine wafers were utilized in the green condition (116 and 76 percent moisture content, respectively) and conditioned to initial moisture contents of oven-dry, 8 percent, and 18 percent before vacuum impregnation with water-borne polymers.

DISCUSSION

As can be seen in *Figure* 5, PEG-1540 is a very effective cell-wall bulking agent for basswood. Only the basswood results are shown but white pine behaved in a similar manner throughout these experiments. This confirms Stamm's[7] work and shows that, like PEG-1000, PEG-1540-treated wood exhibits very little shrinkage, until exposed to liquid water. When comparing the results of the PEG-1540 treatment with the water-borne polymer systems it becomes quite obvious that while those polymers may penetrate cell-wall capillaries they in no way compare to the bulking achievable with polyethylene glycol.

An interesting observation of both basswood and white pine treated with PEG-1540 is that a slight swelling occurs upon oven drying. This is contrary to one of the most fundamental aspects of wood science, that wood shrinks upon drying. A possible explanation is that, being thoroughly bulked with the PEG, the wood wafers are constrained from further shrinkage. The heat of oven drying, however, causes the PEG within the wood cell wall to undergo thermal expansion, thus shrinking cell-wall capillary pore openings. Consequently, the PEG, subjected to thermal expansion but being unable to escape through cell-wall-capillary pore openings causes the wood to swell. This explanation is substantiated by the greater hygroscopicity that the PEG-treated wood exhibits after this thermal swelling, readily observable by comparing PEG-1540-treated basswood with that of water treatment (*Figure* 5). Apparently, the forced swelling opened the cell-wall-capillary structure, resulting in a greater number of sorption sites and therefore a greater hygroscopicity.

Figure 5 clearly shows the abilities of 20 percent EGMBE and DEGMBE aqueous solutions to penetrate and swell wood. While some of this swelling is retained over extensive periods of drying, it is eliminated upon leaching with liquid water. Leaching with liquid water also removes some of the PEG-1540 from basswood and white pine, observable from the above 50 percent reduction in air-dried volume and the decrease in weight. In the air-dry condition before water leaching, PEG-treated basswood had a weight percent gain of 64 percent, which

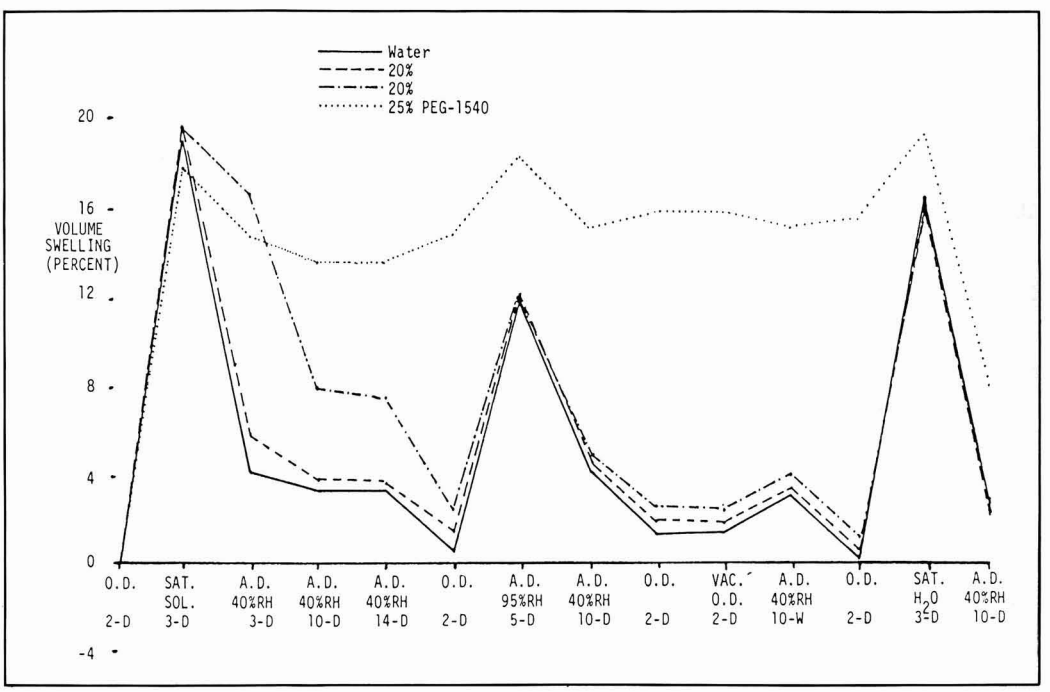

Figure 5—Volume changes of basswood treated with water-based solutions and subsequently exposed to varying conditions

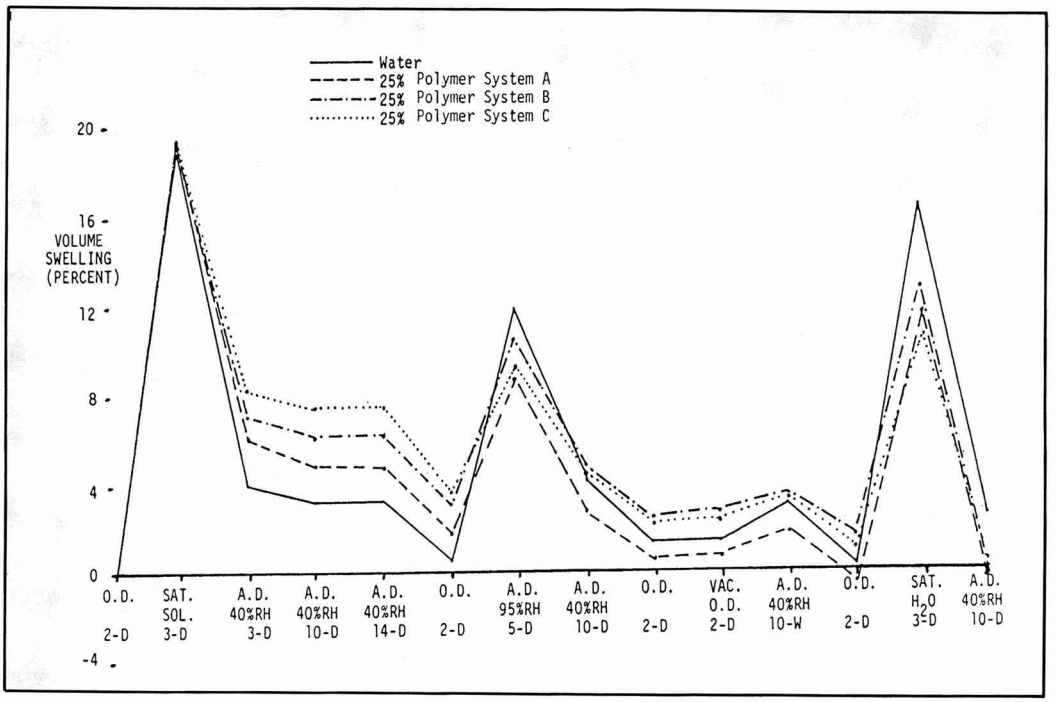

Figure 6—Volume changes of basswood treated with water-borne polymer systems and subsequently exposed to varying conditions

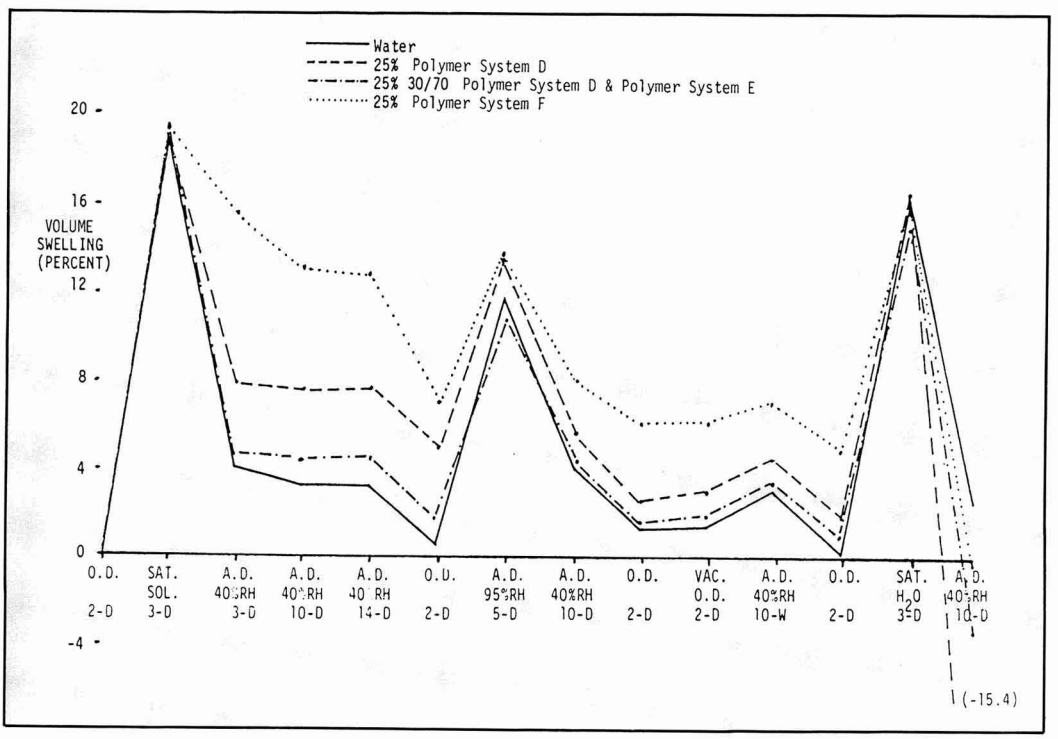

Figure 7—Volume changes of basswood treated with water-borne polymer systems and subsequently exposed to varying conditions

after leaching is reduced to 23 percent. Under the same conditions white pine drops from 59 to 29 percent weight gain.

Since Polymer Systems A, B, and C all contain EGMBE, it is of interest to compare their swelling at various exposure conditions to that found with the aqueous EGMBE solution. Basswood and white pine will be considered together because their results are similar.

Each of these three polymer systems exhibited greater bulking upon air drying than the 20 percent EGMBE solution (*Figures* 5 and 6). A significant difference, however, is that the polymer treatments added approximately 75 and 85 percent, respectively, to the original dry weight of basswood and white pine, while the EGMBE solution added only five and six percent, respectively. Therefore, although the increased swelling of the polymer systems over that of EGMBE could be due to polymer deposited in cell-wall capillaries, it could also be due to trapped solvents that are restrained from evaporating or escaping by the resin loading obstructing cell-wall capillary pore openings. The second argument is substantiated by observing swelling behavior at 95 percent relative humidity and leaching in water. The polymer-treated wood samples did not swell as much as the water-treated samples. This is an indication of the high polymer loadings in cell lumina hindering moisture access to sorption sites. The complete loss of bulking after leaching with water is further evidence that the original swelling was due to entrapped cosolvents.

Polymer System F (*Figure* 7) exhibited a greater degree of bulking over that of its cosolvent alone (20 percent DEGMBE solution, *Figure* 5) than did Polymer Systems A, B, or C. Therefore, this polymer is more likely than the others to actually have achieved cell-wall capillary penetration. However, upon leaching with liquid water, the cell-wall bulking was not retained. For both basswood and white pine there was only a small decrease in weight for Polymer System F due to leaching with water between the last two air-dry conditions. This small weight decrease with the accompanying loss of cell-wall bulking, was caused either by the leaching of entrapped DEGMBE or polymer molecules that had not been able to cure into a non-leachable state within the cell-wall capillaries. Which of these is the correct explanation is not known at this time.

Polymer System D showed definite penetration and bulking of both basswood and white pine cell walls as there is no cosolvent in this system to create bulking artifacts (*Figure* 7). The major problem with this system, however, is that it has a relatively high acid value of 120 (*Table* 1). This means that a correspondingly large amount of an amine, usually ammonia, is necessary for resin solubility and that the final cured polymer will have lower resistance to leaching with liquid water.[19] Smith[24] observed a similar effect, but to a greater degree, when maple was treated with a cell-wall penetrating, water-borne polymer system having an acid value of 130 and utilizing dimethyl amino ethanol for neutralization. Amines are known to degrade wood constituent material,[17,25] and could be the cause of this phenomena. That the excessive shrinkage did not occur in white pine treated with System D is probably due to its higher lignin content. The combination of the high-molecular-weight film-forming emulsion, System E, with System D did not contribute to cell wall bulking performance (*Figure* 7).

Table 3—Percent Volume Shrinkage of Basswood from Swollen Condition to 14 Days Air-Drying at 40% RH

Treatment	Preimpregnation Moisture Content[a] (Oven Dry) 0	8	18	(Green) 116
Water	13.3	13.8	13.3	13.2
20% EGMBE	13.6	12.6	12.4	12.2
20% DEGMBE	10.3	9.2	8.9	8.9
25% PEG-1540	3.6	3.0	2.7	2.8
25% Polymer System A	12.4	12.3	12.2	11.9
25% Polymer System B	11.3	10.8	10.4	10.6
25% Polymer System C	10.0	9.5	9.5	9.5
25% Polymer System D	9.6	9.6	9.2	10.7
25% 30/70 Polymer System D & Polymer System E	12.7	12.7	12.5	13.3
25% Polymer System F	5.6	5.1	5.1	7.8

(a) Percent of oven-dry weight.

Table 3 shows the shrinkage of basswood from the treated, swollen condition after 14 days of air drying. As can be seen from these results, there are no significant shrinkage differences between the different initial moisture content treatment groups. Even PEG-1540, which because it bulks wood cell walls (*Figure* 5) and causes much less shrinkage than other treatments, is not noticeably affected by wood moisture content at the time of treatment. This is not really surprising, however, when it is recalled that these small wood wafers were impregnated under vacuum, virtually eliminating gross penetration problems, and the readily available water in the water-borne polymer systems ensures that the cell-wall capillaries become sufficiently swollen. These results show that the use of oven-dry wafers to provide a more convenient starting point in cell-wall bulking studies is justifiable and dependable when water is present in the treating liquid.

In order to determine if the effective size in solution of the water-borne polymer resins varies according to the solution solvent a number of intrinsic viscosity measurements were made. PEG-1540 has a 21 percent higher intrinsic viscosity in water than in THF (*Figure* 8). This means that its effective size in wood treating aqueous

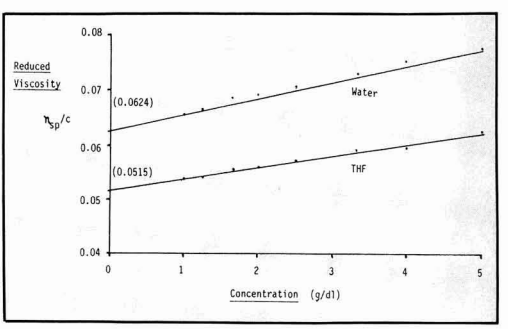

Figure 8—Intrinsic viscosity of PEG-1540

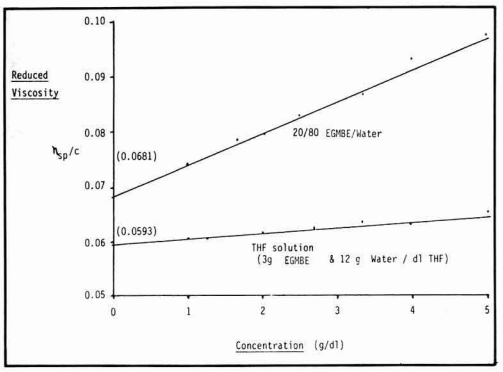

Figure 9—Intrinsic viscosity of polymer system A

solutions is approximately 21 percent larger than its effective size in the THF used in the GPC columns.

Polymer Systems A, C, and F (*Figures* 9, 10, 11) were only available in a diluted form, with cosolvents and water already added. These solvents and water, therefore, had to be added to the THF-diluting solution in order to maintain a consistent ratio of the multi-solvent mixtures upon dilution. The presence of these other solvents in the THF-diluting solution means that the THF solution intrinsic viscosity can only be compared qualitatively, and not quantitatively, to the wood treating water solution.

When Polymer System A (*Figure* 9) is diluted with 20 percent EGMBE, its intrinsic viscosity is 15 percent higher than when it is diluted with a high concentration of THF. This indicates that when it is in a wood treating aqueous solution, System A is approximately 15 percent larger than would have been anticipated from GPC results alone. The intrinsic viscosities of System F (*Figure* 11) indicate an approximately 8 percent larger size in the water solution.

Interestingly, the intrinsic viscosities of Polymer System C (*Figure* 10) point to that polymer being 37 percent smaller in the water solution than in the THF solution. This smaller size in a wood treating aqueous solution increases the likelihood that this polymer penetrates cell-wall capillaries. A comparison of the data on Polymers A and C in *Table* 2 and *Figure* 6 substantiates this premise. While molecular size of the two polymers are quite similar, Polymer C exhibits a greater residual volume bulking

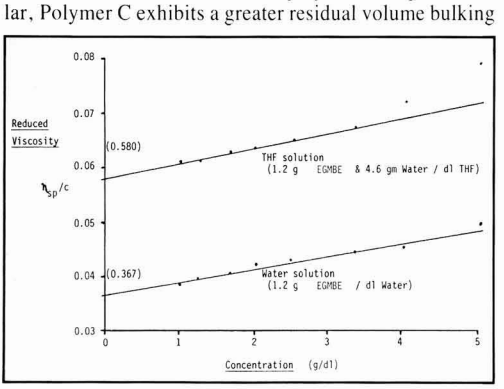

Figure 10—Intrinsic viscosity of polymer system C

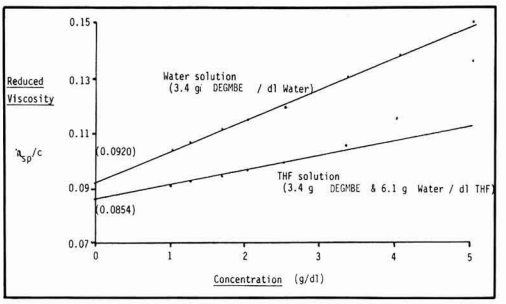

Figure 11—Intrinsic viscosity of polymer system F

than Polymer A, evidence of a greater cell-wall capillary penetration.

An interesting observation was made with Polymer Systems C and F. These two polymers could not be diluted below 25 percent polymer concentration with the 20 percent EGMBE and DEGMBE aqueous solutions that they were dissolved in, without resin precipitation. They could, however, be diluted with distilled water. This non-ideal solution behavior indicates significant polymer/solvent interactions that could affect the effective size of the polymer in solution.

CONCLUSIONS

This research has shown that, aside from the polymer itself, several other factors affect interactions between the wood cell wall and water-borne polymer systems. These include the organic cosolvents and neutralizing amines necessary for the aqueous solubilization of the polymer resins, and the ambient relative humidity.

Although the GPC molecular weight distribution experiments indicated substantial low-molecular-weight fractions in the water-borne polymer systems of this study, definite polymer penetration of cell-wall capillaries, as evidenced by cell-wall bulking, only occurred with Polymer System D and PEG-1540. With Polymer System F, even though significant cell-wall bulking occurred, polymer penetration of cell-wall capillaries could not be confirmed because an organic cosolvent, DEGMBE, was present in the treating solution and leaching with liquid water eliminated the cell-wall bulking. This raises the question, unresolved at this time, of whether the original bulking was due to adsorbed solvent or low-molecular-weight fractions of the System F polymer which had not been able to cure into a nonleachable state within the cell-wall capillaries.

The intrinsic viscosity work has shown that a number of the water-borne polymers in this study could not be diluted with their polar-organic-cosolvent/water mixtures below a certain level without resin flocculation. This deviation from an ideal, or true, solution is indicative of significant polymer/solvent interactions that may cause the effective size of the polymer in solution to be considerably larger than would be expected from the GPC studies. McEwan[26] and Wicks, et al.,[27] finding similar non-ideal behavior in many water-borne polymer sys-

tems, have suggested, based upon experimental evidence such as viscometry and light scattering, that even though the polymer solutions appear clear to the eye, the polymer is actually in a sort of micellar solution with organic cosolvent. Such a situation would explain non-ideal behavior in solution and indicate increases in the effective size of the polymer in solution to the point that molecules would no longer be capable of penetrating wood cell-wall capillaries.

Water-borne polymers with a high acid value require less organic cosolvent for water solubility, a good characteristic when dealing with wood. This research has provided evidence, however, that the high level of amines necessary for the water solubilization of these polymers can cause cellulose degradation. The excessive cell-wall shrinkage and collapse caused by this degradation would be detrimental to any wood treated with this type of water-borne polymer system.

Another complicating factor is whether or not these resin polymers, which definitely cure into a non-leachable form as a surface coating and within wood cell lumens, will cure when they are within wood cell-wall capillaries. The possible lack of free oxygen and inability of separate resin molecules to react together within a constricted capillary structure may prevent curing of any resin deposited within cell-wall capillaries, thus leaving that resin liable to leaching by exposure to liquid water.

Water-borne polymer systems have proven to be quite complicated as far as their interactions with wood are concerned. The potential, however, that non-leachable, one-step, cell-wall penetrating materials have for improving the performance of wood coatings, adhesives, and preservatives justifies continued research in this area.

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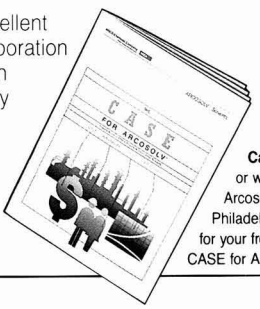

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Statistical Model for Emulsion Paint Above the Critical PVC

Dwight A. Holtzen and William H. Morrison, Jr.
E. I. du Pont de Nemours & Co., Inc.*

An empirical model that provides cost effective starting point formulations for given hiding power and film properties of a paint was developed. This model reduces significantly the number of experiments required using a conventional trial and error approach. The model was developed for emulsion paints between 50 and 70 pigment volume concentration. The effects of different grades of titanium dioxide pigments and various extenders is also predicted for a given paint formulation.

INTRODUCTION

Formulation of paint products is a tedious task, requiring the preparation of many paints before a final formulation is reached. Even then, the paint is rarely completely optimized because of the enormous number of trial and error experiments that must be conducted. There have been many attempts to reduce the number of experiments needed to formulate a paint by calculating approximate starting point formulas. The most successful of the mathematical methods involves calculating the critical PVC,[1] using either the reduced pigment volume concentration,[2,3] or the reduced binder concept.[4] The field has been published extensively, and the reader is referred to the above references.[5] Recent work[6] published during the course of the present study used a statistical design approach to optimize a formulation.

An important property for pigment manufacturers and those who use pigments is hiding efficiency of primary pigments used in paints. Much fundamental work has been done in the area,[7] but direct application by paint formulators is not straightforward.

The development of an empirical model which correlates hiding power and film properties (such as stain resistance and film porosity) to composition is important for the following reasons:

(1) The need exists for a method that gives starting point formulations. The effects of TiO_2 and extender type on paint cost and film properties (such as substituting enamel TiO_2 grades for flat grades) should be predicted.

(2) The paint formulator needs to know what changes can be made to lower cost without lowering quality (spreading rate and film integrity) and if pigments are being used efficiently.

(3) A complete evaluation of the performance of new or competitive pigments could be done with the preparation of relatively few paints. Comparisons now rely mainly on pound-for-pound substitution (or volume substitution) in a few paint formulas that are not completely optimized for each pigment being evaluated.

Others have tried to do this in a nonmathematical, more limited fashion.[8] This paper describes an empirical mathematical model for paints formulated above the CPVC which can be manipulated by a computer, accomplishing these goals.

Scope of the Model

Realizing that all types of paint (i.e, trade sales/industrial, interior, exterior) probably could *not* be included in one empirical model, the study was limited to interior flat paints above the CPVC. Interior flat paint was chosen because of: (1) an interest in the effectiveness of surface treatments on the hiding efficiency of interior high hiding grades of TiO_2, (2) the high percentage of total gallons produced, (3) the large amount of effort to reduce its cost, and (4) the wide variation in formulations, especially amounts and types of extenders.

Since there are large, sudden changes in properties at the CPVC, paints above and below critical cannot easily be described by one mathematical model. About 70% of the volume of interior flat paint is above the CPVC, with

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Table 1—Paint System

- TiO_2: Enamel and Flat Grade
- Extenders (Two):
  Large Partial Size $CaCO_3$
  One Fine Extender (Clay or Silica)
- Resin: Vinyl Acrylic
- Coalescent (Min. For Film Formation)
  Carbitol Acetate (1% of Resin Solids)
- Dispersants (Balanced to Pigment Content)
- Defoamer: As Needed
- Thickener: As Needed

50 TO 70 PVC RANGE
27% VOLUME SOLIDS

about 85% of this in the 50-70 PVC range.[9] Most of the remaining 15% is above 70 PVC and contains very small amounts of TiO_2 (depending primarily on dry flat hiding).

In summary, the model was developed for compositions typical of emulsion paints in the 50-70 PVC range, and included both normal and fine extenders. The fine extenders were either silica or clay. One enamel grade and one high hiding flat grade of TiO_2 were included. Hiding power (oil and total) and stain resistance were the paint properties of interest.

EXPERIMENTAL

Paints used in this study were made from predispersed pigments. Prime pigments and extenders were dispersed at maximum pigment loadings and optimum surfactant content.[10] Grinds were made on a high-speed disc mill.

Drawdowns for determination of hiding power were made with an automatic drawdown blade under controlled speed, temperature, and humidity conditions. Reflectance using a CIE filter was measured on dried drawdowns over the black and white Morest charts. Relative hiding power was calculated from classical Kubelka-Munk equations[11] relating scattering to reflectance.

Stain resistance was evaluated relative to a standard formula containing 1.7 lb TiO_2/gal, 55 PVC at 33.5% solids by volume. The K&N ink was removed after a given period of time with a laboratory towel. Reflectance measurements were then made using a CIE Y filter. Reflectance values of standard and sample were transformed to K/S values using the Kubelka-Munk formula relating R_∞ to K/S.[11]

Relative staining value was then calculated from:

$$\text{Stain} = \frac{(K/S)\ \text{standard}}{(K/S)\ \text{sample}} \times 100$$

Relative staining values greater than 100 implies less ink remaining on the samples vs the standard formula (better stain resistance for the sample).

The paint system used is shown in *Table* 1. The resin (vinyl acrylic) is a widely used interior emulsion resin. Particle size and glass transition temperature (Tg) of the resin have major effects[5] on the properties (hiding, film integrity) studied. The resin used has typical values of Tg (22°C) and mean particle size (0.40μm) for interior emulsion paints.

Two extenders were used. The first was a standard calcium carbonate (1 μm mean particle size) which was chosen because of its normal distribution of particle size about the mean and its uniformity. The second extender was either a fine silica or a delaminated clay. The particle size of these materials is shown in *Table* 2. Extenders, which have a large particle size relative to the prime pigment, influence hiding by acting as a diluent (see Prediction Section). They usually have other purposes in the paint (e.g., control sheen, affect color linearity with tint, and reduce cost). Very fine extenders were included in addition to a carbonate diluent, to study their effect on

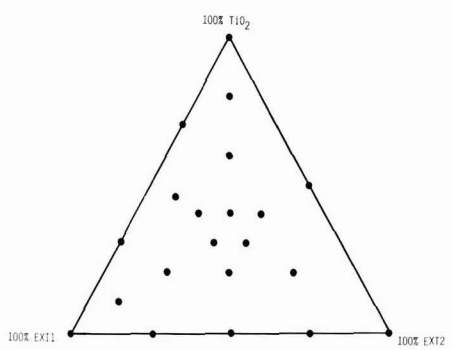

Figure 1 — Compositions in volume %'s of the pigments in the paint compositions originally selected for study. Ext 1 = Fine Extender, Ext 2 = Calcium Carbonate (large particle size). This surface is at constant PVC. Three PVC's were used; 50, 60, and 70

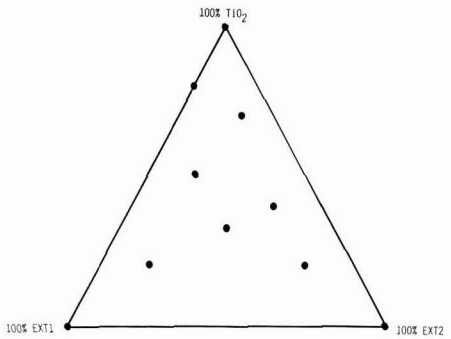

Figure 2 — Minimum number of compositions in volume %'s of the pigments in the paint formulation for study. Ext 1 = Fine Extender, Ext 2 = Calcium Carbonate (large particle size). This surface is at constant PVC. Three PVC's were used; 50, 60, and 70

Table 2—Particle Sizes of Ingredients

Extender	Mean Particle Size (μm)
Calcium carbonate (large particle)	1.0
Silica (fine particle)	0.12
Clay (delaminated)	0.30 (thickness)
	2-10 (diameter)
Silica (fumed)	0.014
Resin (vinyl acrylic)	0.40

Table 3 — Regression Coefficients for Hiding— EXT1 = Fine Silica

	Sx Total			Sx Oil	
	Enamel Grade	Flat Grade		Enamel Grade	Flat Grade
A0	−3.415	−3.981	B0	−0.1849	−0.2537
A1	0.1602	0.1794	B1	0.08459	0.07855
A2	−0.0001345	0.0003956	B2	0.0005358	0.0001849
A3	0.09306	0.1179	B3	0.003837	0.006984
A4	−0.0001054	−0.0006314	B4	−0.00001885	−0.0001115
A5	0.08312	0.08695	B5	0.008676	0.007978
A6	−0.0002972	−0.0002197	B6	−0.00003324	−0.00003145
A7	0.001397	0.001267	B7	0.0004557	0.0002087
R^2	0.98	0.99	R^2	0.98	0.99
Sigma	0.303	0.285	Sigma	0.109	0.094

the hiding efficiency of the TiO_2 and paint film properties. They have been shown to increase the hiding ability of enamel grades of TiO_2.[12] We were interested in confirming these results in a more encompassing model, and to compare the efficiency of these fine extenders with the surface treatment level typically found on a high hiding flat and an enamel grade TiO_2.

Only enough coalescent was present to ensure good film formation. The amount of surfactant(s) was adjusted according to pigment level in each paint. Other ingredients such as glycols, fungicides, biocides, etc. which could affect film properties were omitted.

Data Collection

The compositions in *Figure* 1 were made at three PVC's; 50, 60, and 70. After several data sets were collected with 63 points, as shown in *Figure* 1 (21 points at each of the PVC's), it was found that the same "goodness" of fit (shown by correlation coefficient square "R^2" and total residuals) could be obtained with 30 data points as shown in *Figure* 2 (10 points at each of the three PVC's). Most of the data was collected with the 30-point sets. Each point was measured for oil and total (dry) hiding, and stain resistance as described in the experimental section.

The points in *Figures* 1 and 2 were chosen to give good statistical balance over the range of compositions included in the analysis. Certain points which normally would have been included (e.g., high Ext. 2, low Ext. 1 and TiO_2 PVC's) were not because of difficulty in making acceptable paints. Certain areas (e.g., the center area of *Figures* 1 and 2) were given more weight because many commercial systems are formulated there.

Regression Fitting of Data

The mathematical fitting of the data was done using a standard multiple regression program.[13] Several types of mathematical models were tried including logarithmic, exponential, second and third order polynomials, and inverse functions. Combinations of the above were also examined. The simplest equations (i.e., contained the fewest terms) giving good fits to the data are shown in equations (1), (2), and (3).

$$\text{Sxtotal} = A_o + A_1PVC_{TiO_2} + A_2PVC^2_{TiO_2} + A_3PVC_{Ext\,1} + A_4PVC^2_{Ext\,1} + A_5PVC_{Ext\,2} + A_6\,PVC^2_{Ext\,2} + A_7PVC_{TiO_2}\,PVC_{Ext\,1} \quad (1)$$

$$\text{Sxoil} = B_o + B_1PVC_{TiO_2} + B_2PVC^2_{TiO_2} + B_3PVC_{Ext\,1} + B_4PVC^2_{Ext\,1} + B_5PVC_{Ext\,2} + B_6PVC^2_{Ext\,2} + B_7PVC_{TiO\,2}\,PVC_{Ext\,1} \quad (2)$$

The regression coefficients for various paint systems are in *Tables* 3 to 6, along with the values of R^2 (the portion of the sum of the squares of the dependent variable which is explained by the multiple regression equation), and an estimate of the standard deviation of the dependent variable based on the regression fit. Negative coefficients for the square terms in the hiding equations indicate a negative curvature of the hiding power surface as the concentration of pigment (either TiO_2 or extender)

Table 4 — Regression Coefficients for Hiding— EXT1 = Delaminated Clay

	Sx Total			Sx Oil	
	Enamel Grade	Flat Grade		Enamel Grade	Flat Grade
A0	−2.956	−3.882	B0	−0.02139	−0.1331
A1	0.1422	0.1655	B1	0.08197	0.07439
A2	0.00007655	0.0005727	B2	−0.0005271	−0.0001355
A3	0.09390	0.08776	B3	0.01084	0.006661
A4	−0.0004797	−0.0001468	B4	−0.0001649	−0.00007072
A5	0.08384	0.09603	B5	0.007933	0.006856
A6	−0.0004049	−0.0003905	B6	−0.0001117	−0.00007503
A7	0.0009606	0.001072	B7	0.0001815	0.0001201
R^2	1.00	0.99	R^2	0.99	1.00
Sigma	0.191	0.236	Sigma	0.095	0.056

Table 5 — Regression Coefficients for Stain— EXT1 = Fine Silica

	Enamel Grade Stain TiO_2	Flat Grade Stain TiO_2
C0	1414	555.2
C1	−24.25	−11.21
C2	0.1664	0.07737
C3	−3455	−1197
C4	3374	1280
C5	12.57	6.301
R^2	0.89	0.82
Sigma	21.9	11.5

Table 6 — Regression Coefficients for Stain— EXT1 = Delaminated Clay

	Enamel Grade Stain TiO_2	Flat Grade Stain TiO_2
C0	9162	8226
C1	−201.2	−219.2
C2	1.257	1.516
C3	−11770	−4960
C4	7223	2150
C5	84.33	49.33
R^2	0.97	0.84
Sigma	29.8	40.9

is raised. This is not surprising because the crowding of pigment lowers scattering efficiency.

$$\text{Stain} = C_o + C_1PVC_{Total} + C_2PVC^2_{Total} + C_3(Sx_{Oil}/Sx)_{Total} + C_4\,(Sx_{Oil}/Sx_{Dry})^2 + C_5PVC_{Total}\,(Sx_{Oil}/Sx_{Total}) \quad (3)$$

For the hiding data, the fits are very good. The equation should predict the hiding values (Sx oil and Sx total at 1 mil wet film) accurately to within $\pm 2\sigma$. Extrapolation of the regression fitted polynomial to areas of composition outside the data is not valid. The equations apply only to the 50-70 PVC range as defined by the data. The experimental standard deviations for the hiding data are about 0.30 as compared with the values shown in *Tables* 3 and 4 for the fitting error.

There is more error in the calculated values of stain using the regression equation (R^2 values low). The stain test (as practiced in this laboratory) has a high experimental error, with standard deviations of ~20. The accuracy of the predicted values are in the same error range (see *Tables* 5 and 6). If the error in the stain test could be reduced, the regression fittings would be much better.

With mathematical expressions for relative staining value and hiding, a computer program can be written to predict compositions which will satisfy certain values of these parameters.

TI02 GRADE FLAT EXTENDER = SI02 TITLE —

PIGMENT VOLUME CONCENTRATION IS AT .340 VOLUME SOLIDS FRACTION. ALL PVC BLENDS ARE EST. TO GIVE TOTAL SX = 2.75 AND OILED HIDING SX = 1.65 AT 1 MIL WET FILM WITH TOTAL PVC FROM 50-70. TOTAL MATERIAL COSTS (PIGMENT + RESIN) ARE BASED ON 1/17/84 PRICES. FOR STAIN TFW-178 IS THE CONTROL AND = 100. VALUES GREATER THAN 100 INDICATE IMPROVED STAIN RESISTANCE.

PVC TOTAL	PVC TI02	PVC SI02	PVC CAC03	COST $/GAL	STAIN	SXOIL/SXDRY = 0.60
50.	18.6	1.0	30.4	2.73	120.	
50.	18.3	2.0	29.7	2.74	120.	
50.	18.0	3.0	29.0	2.76	120.	
50.	17.7	4.0	28.3	2.77	120.	
50.	17.5	5.0	27.5	2.79	120.	
50.	17.2	6.0	26.8	2.80	120.	
50.	17.0	7.0	26.0	2.82	120.	
50.	16.7	8.0	25.3	2.83	120.	
50.	16.5	9.0	24.5	2.85	120.	
50.	16.3	10.0	23.7	2.87	120.	
55.	17.2	0.0	37.8	2.52	123.	
55.	16.9	1.0	37.1	2.53	123.	
55.	16.6	2.0	36.4	2.55	123.	
55.	16.3	3.0	35.7	2.56	123.	

FINISHED

Figure 3 — Computer printout for the flat grade TiO_2, fine silica, and carbonate (large particle size)

COMPUTER PROGRAM

A computer program in FORTRAN was written to manipulate the regression equations for hiding and stain. A detailed listing of the program is given in the Appendix. A brief description follows.

The two simultaneous equations for oil and dry hiding are stated in terms of three variables [see equations (1) and (2)]; PVC TiO_2, PVC EXT 1, and PVC EXT 2. Two of the variables can be related to the other in terms of total PVC.

$$\text{PVC EXT 2} = (\text{PVC TOTAL} - \text{PVC EXT 1}) - \text{PVC } TiO_2 \quad (4)$$

This value for PVC EXT 2 can be substituted into the two equations for oil and total hide and the equations can be simplified to two quadratic equations in terms of PVC TiO_2 as the independent variable.

$$O = PVC^2_{TiO_2}\,(A_2 + A_6) + PVC_{TiO_2}\,(A_1 + A_7 PVC_{EXT\,1} - 2A_6H - A_5) + K + (A_0 - S_x) \quad (5)$$

$$H = PVC_{TOTAL} - PVC_{EXT\,1} \quad (6)$$

$$K = A_5H + A_6H^2 + A_3PVC_{EXT\,1} + A_4\,PVC^2_{EXT\,1} \quad (7)$$

The two quadratic equations (one for Sx total and one for Sx oil with the B coefficients substituting for the A's) are then solved for all cases where their roots are equal and real for all values of PVC TOTAL and PVC EXT 1. There are no unique solutions but by iterating on PVC TOTAL and PVC EXT 1, a map of solutions can be generated for any given values of Sx oil and Sx total. Relative staining values can then be calculated for each solution.

The input data to the program comprises three parameters: volume solids fraction of the paint (used to adjust Sx total and Sx oil); Sx oil at 1 mil wet film; and Sx total at 1 mil wet film. Since all the data are at constant spreading rate, the hiding values are at equal wet film thickness. One mil was chosen as a convenient thickness to measure hiding. The program then calculates all possi-

ble compositions of PVC TOTAL, PVC TiO_2, and PVC EXTENDERS which will satisfy those values and prints out this information, along with calculated stain values as shown in *Figure* 3.

The sum of the experimental and fitting error in the output is estimated to be about ±5% of the predicted PVC's. The next section will discuss how well the model describes commercial paint systems.

COMMERCIAL PAINT SYSTEMS—PREDICTION

Several interior flat paints were made based on commercial formulations. All toning (colored) pigments were omitted. Measured values of Sx total and Sx oil (1 mil wet) along with volume solids were used to predict PVC's with the computer program. Within the map of predicted solutions were values which corresponded well with the actual PVC's in the paint. The results are shown in *Table* 7. These paints covered resins from acrylic to PVAC, widely varying levels of TiO_2 and widely varying types of extenders, coalescents and other ingredients.

In the two cases where the difference between observed and predicted levels of TiO_2 PVC are large, (see *Table* 7, fourth and sixth paints), the predicted values are lower. This discrepancy could be the result of an inefficient use of the TiO_2 in the paint. Either poor dispersion or poor dispersion stability could be the cause. The discrepancy could be due also to effects mentioned in the next section. Because the paint system used in this study is well balanced (i.e., the TiO_2 and extenders are well dispersed and stable), the few discrepancies found in *Table* 7 are to be expected.

Figures 3 and 4 illustrate the use of fine extenders in combination with enamel grade TiO_2 to produce pigmentation equal in cost and performance to highly treated flat grades. The ability to match the performance of highly treated flat grades with the combination of a fine extender and an enamel grade TiO_2 was a general phenomena observed at widely different values of oil and total hiding.

Table 7 — Actual vs Predicted Compositions For Commercial Paints

	TOTAL PVC	TiO_2 PVC	EXTENDER PVC
Actual	55	16	39
Predicted[a]	55	16	39
Actual	59	22	37
Predicted[a]	60	22	38
Actual	58	14	44
Predicted[a]	55	14	41
	60	13	47
Actual	69	15	54
Predicted[a]	70	9	61
Actual	49	22	27
Predicted[a]	50	20	30
Actual	62	19	43
Predicted[a]	60	11	49
Actual	70	13	57
Predicted[a]	70	10	60

[a]Total PVC was set at every 5 PVC from 50-70 PVC, intermediate total PVC's were not calculated.

TI02 GRADE ENAMEL EXTENDER = SI02 TITLE —

PIGMENT VOLUME CONCENTRATION IS AT .340 VOLUME SOLIDS FRACTION. ALL PVC BLENDS ARE EST. TO GIVE TOTAL SX = 2.75 AND OILED HIDING SX = 1.65 AT 1 MIL WET FILM WITH TOTAL PVC FROM 50-70. TOTAL MATERIAL COSTS (PIGMENT + RESIN) ARE BASED ON 1/17/84 PRICES. FOR STAIN TFW-178 IS THE CONTROL AND = 100. VALUES GREATER THAN 100 INDICATE IMPROVED STAIN RESISTANCE.

PVC TOTAL	PVC TI02	PVC SI02	PVC CAC03	COST $/GAL	STAIN	SXOIL/SXDRY = 0.60
50.	16.6	9.0	24.4	3.00	136.	
50.	16.4	10.0	23.6	3.01	136.	
50.	16.1	11.0	22.9	3.03	136.	
50.	15.9	12.0	22.1	3.04	136.	
50.	15.6	13.0	21.4	3.06	136.	
50.	15.4	14.0	20.6	3.07	136.	
50.	15.2	15.0	19.8	3.09	136.	
50.	15.0	16.0	19.0	3.11	136.	
50.	14.8	17.0	18.2	3.13	136.	
50.	14.6	18.0	17.4	3.14	136.	
50.	14.4	19.0	16.6	3.16	136.	
50.	14.3	20.0	15.7	3.18	136.	
55.	16.4	3.0	35.6	2.70	140.	
55.	16.1	4.0	34.9	2.71	140.	
55.	15.8	5.0	34.2	2.72	140.	
55.	15.5	6.0	33.5	2.73	140.	
55.	15.2	7.0	32.8	2.75	140.	
55.	15.0	8.0	32.0	2.76	140.	
55.	14.7	9.0	31.3	2.77	140.	
55.	14.5	10.0	30.5	2.79	140.	
55.	14.2	11.0	29.8	2.80	140.	
60.	15.6	0.0	44.4	2.46	152.	
60.	15.3	1.0	43.7	2.47	152.	
60.	15.0	2.0	43.0	2.48	152.	
60.	14.7	3.0	42.3	2.49	152.	
60.	14.4	4.0	41.6	2.50	152.	
60.	14.1	5.0	40.9	2.51	152.	
65.	14.1	0.0	50.9	2.26	173.	

FINISHED

Figure 4 — Computer printout for the enamel grade TiO_2, fine silica, and carbonate (large particle size)

We found no exceptions. Generally, the total PVC of the paint containing the enamel grade/fine extender combination has to be raised to get equal cost at equal film integrity. This is due to the lower binder demand of this combination vs the flat grade.

Fine extender silica and clay are approximately equally efficient optical spacers of enamel grade TiO_2 (see *Figures* 6 and 7). The higher price of silica made formulations using it more costly than clay (see *Figures* 4 and 5). Some limited data with fumed silica of 0.014 μm size showed further improvements in hiding efficiency for enamel grades (see *Figure* 8). None of the extenders investigated improved the efficiency of the TiO_2 to the level of the flat grades. This is illustrated in *Figures* 6 and 7 which show the hiding power for TiO_2/fine extender combinations. The flat grade's hiding is maximum at zero extender (acting only as a diluent) while the enamel grade

hiding efficiency goes through a maximum at a unique volume fraction for that TiO_2 extender combination. This maximum is always lower for the enamel grade/extender combination. The lower hiding is compensated by the lower cost of the enamel grade/fine extender combination.

The model also shows the advantage of delaminated clay on relative staining values. The clay probably forms sheet-like layers (''leafing'') and acts as a sealer at or near the surface of the film (see *Figures* 4 and 5).

A coarse carbonate/TiO_2 ladder (i.e., staying on the line connecting 100% TiO_2 with 100% Ext 2 in *Figure* 1) showed the interaction term (PVC_{TiO_2} PVC_{EXT_2}) to be unimportant (zero). Thus, coarse carbonate acts only as a diluent for TiO_2 in terms of hiding efficiency.

TI02 GRADE ENAMEL EXTENDER = CLAY TITLE —

PIGMENT VOLUME CONCENTRATION IS AT .340 VOLUME SOLIDS FRACTION. ALL PVC BLENDS ARE EST. TO GIVE TOTAL SX = 2.75 AND OILED HIDING SX = 1.65 AT 1 MIL WET FILM WITH TOTAL PVC FROM 50-70. TOTAL MATERIAL COSTS (PIGMENT + RESIN) ARE BASED ON 1/17/84 PRICES. FOR STAIN TFW-178 IS THE CONTROL AND = 100. VALUES GREATER THAN 100 INDICATE IMPROVED STAIN RESISTANCE.

PVC TOTAL	PVC TI02	PVC CLAY	PVC CAC03	COST $/GAL	STAIN	SXOIL/SXDRY = 0.60
50.	15.7	9.0	25.3	2.64	313.	
50.	15.4	10.0	24.6	2.62	313.	
50.	15.2	11.0	23.8	2.60	313.	
50.	14.9	12.0	23.1	2.58	313.	
50.	14.7	13.0	22.3	2.57	313.	
50.	14.5	14.0	21.5	2.55	313.	
50.	14.3	15.0	20.7	2.54	313.	
50.	14.1	16.0	19.9	2.52	313.	
50.	14.0	17.0	19.0	2.51	313.	
50.	13.8	18.0	18.2	2.50	313.	
50.	13.7	19.0	17.3	2.49	313.	
50.	13.6	20.0	16.4	2.49	313.	
50.	13.5	21.0	15.5	2.48	313.	
50.	13.4	22.0	14.6	2.47	313.	
50.	13.3	23.0	13.7	2.47	313.	
50.	13.3	24.0	12.7	2.46	313.	
50.	13.2	25.0	11.8	2.46	313.	
50.	13.2	26.0	10.8	2.46	313.	
50.	13.2	27.0	9.8	2.46	313.	
50.	13.1	28.0	8.9	2.46	313.	
50.	13.2	29.0	7.8	2.46	313.	
50.	13.2	30.0	6.8	2.47	313.	
50.	13.2	31.0	5.8	2.47	313.	
50.	13.3	32.0	4.7	2.48	313.	
50.	13.3	33.0	3.7	2.48	313.	
50.	13.4	34.0	2.6	2.49	313.	
50.	13.5	35.0	1.5	2.50	313.	
50.	13.6	36.0	0.4	2.51	313.	
55.	17.2	0.0	37.8	2.66	220.	
55.	16.7	1.0	37.3	2.63	220.	
55.	16.3	2.0	36.7	2.60	220.	
55.	16.0	3.0	36.0	2.57	220.	
55.	15.6	4.0	35.4	2.54	220.	
55.	15.2	5.0	34.8	2.52	220.	
55.	14.9	6.0	34.1	2.49	220.	
55.	14.6	7.0	33.4	2.47	220.	
55.	14.3	8.0	32.7	2.44	220.	
55.	14.0	9.0	32.0	2.42	220.	
55.	13.7	10.0	31.3	2.40	220.	
55.	13.4	11.0	30.6	2.38	220.	
60.	15.9	0.0	44.1	2.48	189.	
60.	15.4	1.0	43.6	2.45	189.	
60.	15.0	2.0	43.0	2.42	189.	

FINISHED

Figure 5 — Computer printout for the enamel grade TiO_2, delaminated clay, and carbonate (large particle size)

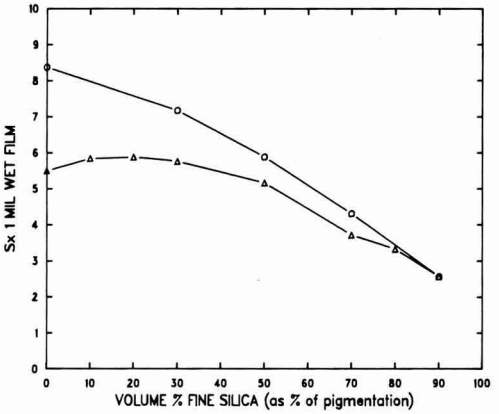

Figure 6 — Hiding power vs extender volume % for blends of TiO_2 flat grade (O) or enamel (Δ) grade/fine silica. The paint is at 60 PVC. For the enamel grade Sx (MAX) = 5.8 at ~20% extender

LIMITATION OF MODEL

Resin Tg, resin particle size, and coalescent efficiency and level all significantly affect CPVC.[5] The effects can be described by relatively simple functions; however, when incorporated into a single equation, it becomes too cumbersome. Even with these limitations, the model does

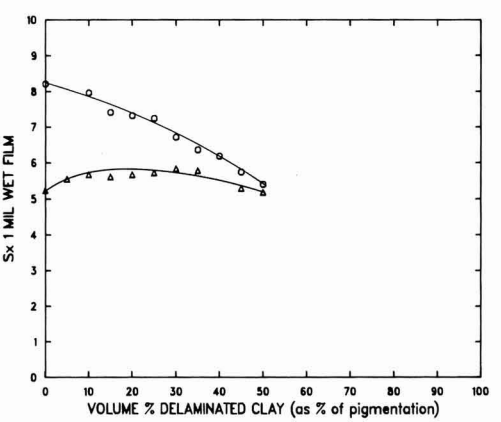

Figure 7 — Hiding power vs extender volume % for blends of TiO_2 flat grade (O) or enamel (Δ) grade/delaminated clay. The paint is at 60 PVC. For the enamel grade Sx (MAX) = 5.8 at ~20% extender

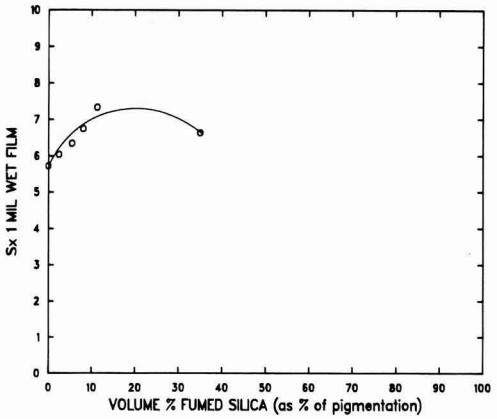

Figure 8 — Hiding power vs extender volume % for blends of TiO_2 enamel grade/fine fumed silica. The paint is at 60 PVC. For the enamel grade Sx (MAX) = 7.6 at ~20% extender

give good "starting point" formulations (see *Table* 7). The known types of changes that occur with changes in resin and coalescent can be used to modify the predicted results of the model in a qualitative way. Use of the model to predict paint compositions outside the 50-70 PVC range is unsound.

SUMMARY

With relatively few paints an empirical statistical model of a flat paint above the critical pigment volume concentration was constructed. Good starting point formulas can be calculated using the model for a paint of unknown composition from its measured volume solids, Sx oil, and Sx total, or using specifications for these parameters for a new paint. The effects of extender type on two titanium dioxide pigments, an enamel and a flat grade, showing how similar paints can be formulated at equal cost from either, was demonstrated with the model.

A computer program designed to manipulate the regression equations for hiding power and relative staining values is also provided.

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APPENDIX

```
$     TY HIDCALC.FOR
            DIMENSION X(4,2),ITLE(10)
            IQ='N'
100         CONTINUE
C     ********************************************
C     A VALUES ARE DRY HIDING COEFFICIENTS
C
C     B VALUES ARE OILED HIDING COEFFICIENTS
C
C     C VALUES ARE STAIN COEFFICIENTS
C
C
C     COST VALUES:  D00 = RESIN
      ($ Per Gal)   D1  = T102
                    D2  = FINE EXTENDER
                    D3  = COARSE EXTENDER
C     ********************************************
      D00=6.39
      D3=1.75
      TYPE 110
110   FORMAT(' T102 PIGMENT TYPE
      (R902,R931       )=',$)
      ACCEPT 111,ITI02
111   FORMAT(A4)
      IF (ITI02  .EQ. ' ') GO TO 200
      TYPE 112
112   FORMAT(' FINE EXTENDER TYPE(SI02,CLAY)=',$)
      ACCEPT 113, IEXTN
113   FORMAT(A4)

      IF (ITI02 .EQ. 'R931') GO TO 114
      IF (1EXTN .EQ. 'SI02') GO TO 115
C     ********************************************
C     COEFFICIENTS FOR ENAMEL/CLAY
C     ********************************************
      A0=2.956
      A1=0.1422
      A11=0.00007655
      A2=0.09390
      A22=-0.0004797
```

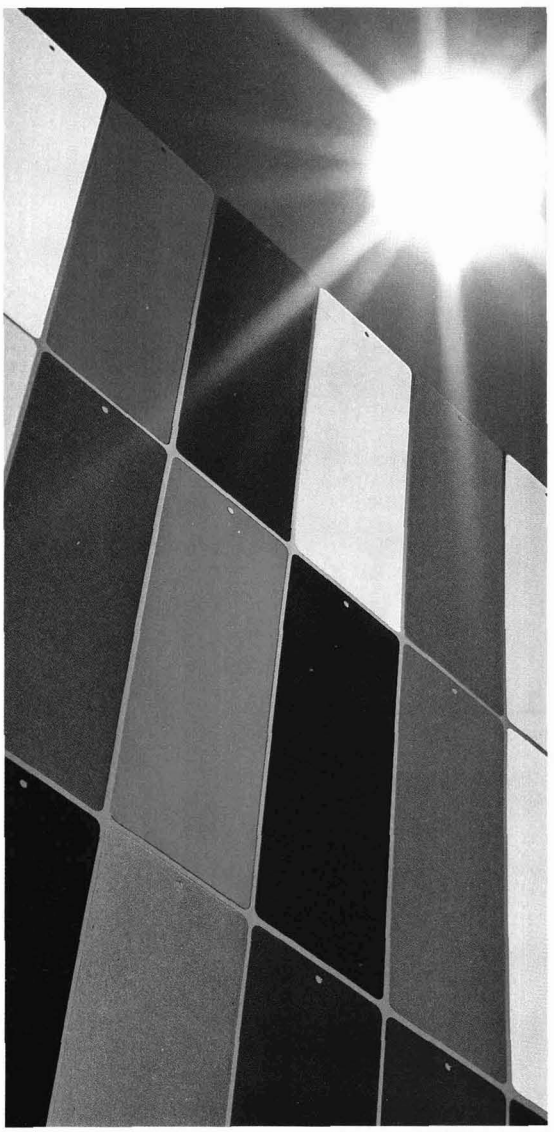
```
      A3=0.08384
      A33=-0.0004049
      A12=0.0009606
      B0=-0.02139
      B1=0.08197
      B11=-0.0005271
      B2=0.01084
      B22=-0.0001649
      B3=0.007933
      B33=-0.0001117
      B12=0.0001815
      C1=9162.
      C2=-201.2
      C3=1.257
      C4=-11770.
      C5=7223.
      C6=84.33
      D1=25.01
      D2=2.15
      GO TO 116
C     ********************************************
C     COEFFICIENTS FOR ENAMEL/SILICA
C     ********************************************
115   A0=-3.415
      A1=0.1602
      A11=-0.0001345
      A2=0.09306
      A22=-0.0001054
      A3=0.08312
      A33=-0.0002972
      A12=0.001397
      B0=-0.1849
      B1=0.08459
      B11=-0.0005358
      B2=0.003837
      B22=-0.00001885
      B3=0.008676
      B33=-0.00003324
      B12=0.0004557
      C1=1414.
      C2=-24.25
      C3=0.1664
      C4=-3455.
      C5=3374.
      C6=12.57
      D1=25.01
      D2=11.63
      GO TO 116
114   IF (IEXTN. .EQ. 'SI02') GO TO 127
C     ********************************************
C     COEFFICIENTS FOR FLAT/CLAY
C     ********************************************
      A0=-3.882
      A1=0.1655
      A11=0.0005727
      A2=0.08776
      A22=-0.0001468
      A3=0.09603
      A33=-0.0003905
      A12=0.001072
      B0=-0.1331
      B1=0.07439
      B11=-0.0001355
      B2=0.006661
      B22=-0.00007072
      B3=0.006856
      B33=-0.00007503
      B12=0.0001201
      C1=8226.
      C2=-219.2
      C3=1.516
      C4=-4960.
      C5=2150.
      C6=49.33
      D1=22.51
      D2=2.15
      GO TO 116
C     ********************************************
C     COEFFICIENTS FOR FLAT/SILICA
C     ********************************************
127   A0=-3.981
      A1=0.1794
      A11=0.0003956
      A2=0.1179
      A22=-0.0006314
      A3=0.08695
      A33=-0.0002197
      A12=0.001267
      B0=-0.2537
      B1=0.07855
      B11=-0.0001849
      B2=0.006984
      B22=-0.0001115
      B3=0.007978
      B33=-0.00003145
      B12=0.0002087
      C1=555.2
      C2=-11.21
      C3=0.07737
      C4=-1197.
      C5=1280.
      C6=6.301
      D1=22.51
      D2=11.63

116   TYPE 117
117   FORMAT(' INCREMENT OF TOTAL PVC=',$)
      ACCEPT 118,DPVC
118   FORMAT(F)
      TYPE 119
119   FORMAT(' INCREMENT OF FINE EXTENDER PVC=',$)
      ACCEPT 120,DX2
120   FORMAT(F)
      IF (IQ .EQ. 'Y') GO TO 160
      TYPE 140
140   FORMAT(' ENTER TITLE ',$)
      ACCEPT 150,(ITLE(I),I=1,5)
150   FORMAT(5A4)
      TYPE 121
121   FORMAT(' TOTAL SOLIDS (VOLUME FRACTION)=',$)
      ACCEPT 122,SOLIDS
122   FORMAT(F)
      TYPE 123
123   FORMAT(' TOTAL SX=',$)
      ACCEPT 124,XTSX
124   FORMAT(F)
      TYPE 125
125   FORMAT(' OILED SX=',$)
      ACCEPT 126,XOSX
126   FORMAT(F)
      TYPE 170
170   FORMAT(' WILL THESE SAME #,S BE USED FOR ALL
      CASES, Y/N?',$)
      ACCEPT 128,IQ
```

```
128     FORMAT(A)
160     TSX=XTSX
        OSX=XOSX
        AT=A11+A33
        A0=B11+B33
        PVC=50.-DPVC
        SXR=OSX/TSX
        TYPE 180,ITI02,IEXTN,(ITLE(I),I=1,5),SOLIDS,
        TSX,OSX
180     FORMAT(1H1,
        1'TIO2 GRADE ='A4,2X,'EXTENDER ='A4,5X,'TITLE
        -',1X,5A4,//
        1' PIGMENT VOLUME CONCENTRATION IS AT 'F4.3'
        VOLUME SOLIDS FRACTION.'/
        1' ALL PVC BLENDS ARE EST. TO GIVE TOTAL SX =
        'F4.2' AND OILED'/
        1' HIDING SX = 'F4.2' AT 1 MIL WET FILM WITH TOTAL
        PVC FROM 50-70.'/
        1' TOTAL MATERIAL COSTS (PIGMENT+RESIN) ARE
        BASED ON 1/17/84 PRICES.'/
        1' FOR STAIN TFW-178 IS THE CONTROL AND = 100.
        VALUES GREATER THAN'/
        1' 100 INDICATE IMPROVED STAIN RESISTANCE.')
        TYPE 190,IEXTN,SXR
190     FORMAT(/
        1' PVC   PVC   PVC   PVC   COST'/
        1' TOTAL   TIO2   'A4'   CACO3   $/GAL
        STAIN      SXOIL/SXDRY='F4.2,/)
        TSX=(0.27/SOLIDS)*TSX
        OSX=(0.27/SOLIDS)*OSX
1       X2=0.
        PVC=PVC+DPVC
        IF (PVC .GT. 70.) TYPE 55
55      FORMAT(/25X,'FINISHED')
        IF (PVC .GT. 70.) GO TO 100
2       IF (X2 .GT. 70.) GO TO 1
        X(3,1)=-1.
        X(3,2)=-1.
        H=PVC-X2
        BT=A1+A12*X2-2.*A33*H-A3
        BO=B1+B12*X2-2.*B33*H-B3
        CT=A3*H+A33*H**2+A2*X2+A22*X2**
        2+A0-TSX
        CO=B3*H+B33*H**2+B2*X2+B22*X2**
        2+B0-OSX
        DISCT=BT**2-4.*AT*CT
        DISCO=BO**2-4.*AO*CO
        IF (DISCT .LT. 0.) GO TO 6
        IF (DISCO .LT. 0.) GO TO 6
        SQDT=SQRT(DISCT)
        SQDO=SQRT(DISCO)
        X(1,1) = (-BT+SQDT)/(2.*AT)
        X(1,2) = (-BT-SQDT)/(2.*AT)
        X(2,1) = (-BO+SQDO)/(2.*AO)
        X(2,2) = (-BO-SQDO)/(2.*AO)
        DO 5 K=1,2
        DO 5 J=1,2
        DIFF=X(1,J)-X(2,K)
        IF (ABS(DIFF) .GE. 2.) GO TO 5
        X(4,J)=(X(1,J)+X(2,K))/2.
        SUM=X(4,J)+X2
        IF (X(4,J) .GE. 0.   .AND.   SUM   .LE. PVC)
        X(3,J)=PVC-SUM
5       CONTINUE
        DO 6 J=1,2
        IF (X(3,J) .LT. 0.) GO TO 6
        COST=SOLIDS*(D00*(1.-PVC/100.)+D1*X(4,J)/
        100.+D2*X2/100.+D3*X(3,J) 1/100.)
        STAIN=C1+C2*PVC+C3*PVC**2+C4*SXR+C5
        *SXR**2+C6*PVC*SXR
        TYPE 210,PVC,X(4,J),X2,X(3,J),COST,STAIN
210     FORMAT(F6.0, 3F7.1, F7.2, 2X, F6.0)
6       CONTINUE
        X2=X2+DX2
        GO TO 2
200     CONTINUE
        END
```

In sunlight, coatings can die in months

With CIBA-GEIGY light stabilizers, they can live for years



*U.S. Patent Nos. 4,314,933; 4,426,471; 4,426,472; and 4,344,876 assigned to CIBA-GEIGY Corporation.

Cracking, delamination and catastrophic failure often occur in coatings exposed to the sun. Consequently, the service life of both coating and substrate is shortened, and the cost of maintenance goes up.

Commercial use has shown that most types of coatings can be protected against photodegradation by two classes of CIBA-GEIGY light stabilizers.

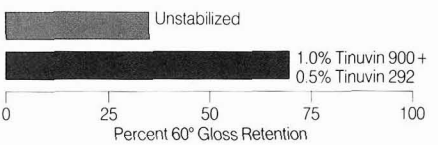
The first class is the hindered amine light stabilizers (HALS), such as Tinuvin® 292, Tinuvin 144 and Tinuvin 079L. They protect gloss and help prevent loss of physical properties that may result in chalking, erosion, water permeability and checking.

Ultraviolet light absorbers (UVA) such as Tinuvin 328, Tinuvin 900 and CGL-1130 are the other class. UVAs prevent UV light from degrading light-sensitive materials, especially primers, mildewcides and pigments. Thus, they protect performance characteristics, such as adhesion between primer and top coats. Depending on the pigment used, a UVA and HALS combination will improve color retention.

While a CIBA-GEIGY UVA or HALS by itself provides good protection for coatings, usage has shown that combinations of the two may give better results. This development is so important that CIBA-GEIGY has four patents* covering the use of HALS and combinations of HALS and UVAs in acrylic, polyester, alkyd and polyurethane coatings.

Percent 60° Gloss Retention After Two Years of Exposure

Sample: Two-Component Solid White Acrylic Aliphatic Polyurethane Coating
Exposure: 5° South Florida Black Box



Manufacturers of maintenance, coil and powder coatings can now formulate their products to yield substantially longer service life.

For more information on light stabilization of coatings, call 800 431-1900 (in New York, 914 347-4700). Or write to: Additives Department, CIBA-GEIGY Corporation, Three Skyline Drive, Hawthorne, NY 10532.

CIBA–GEIGY

Low Temperature Crosslinking Studies: Insertion Reactions Involving Titanium Alkoxides And Isocyanate Containing Polymers

John C. Graham and Ta-Wei Wang†
Eastern Michigan University*

Linear copolymers containing varying amounts of isocyanate were prepared from isocyanatoethyl methacrylate (IEM) and methyl methacrylate. These polymers were reacted with bis (ethyl-3-oxo-butanolato-0′,0[3]) bis (2-propanolato) titanium (Tyzor DC®) under ambient moisture and dry conditions to form crosslinked systems. The course of the reactions were followed using Sward Hardness and infrared and nuclear magnetic resonance spectroscopy.

The results of these studies show that although Tyzor DC reacts rapidly with isocyanate containing methacrylate copolymers under both ambient conditions and under nitrogen, the first condition yields appreciably harder coatings as determined by the Sward Hardness method indicating a distinct difference in the crosslinking reactions involved. In addition, model reactions between aromatic and aliphatic isocyanates and Tyzor DC in solvent systems revealed a significant steric factor and the reversibility of the insertion reaction at temperatures slightly higher than room temperature.

INTRODUCTION

Insertion reactions involve the addition of a metal compound (*I*) to a neutral unsaturated acceptor molecule (*II*) resulting in an adduct (*III*) where the unsaturated molecule has inserted itself between the metal and the group which was originally bonded to the metal [equation (1)].

$$\underset{(I)}{M-X} + \underset{(II)}{Y} \rightarrow \underset{(III)}{M-Y-X} \qquad (1)$$

As the mechanisms of organometallic reactions have been gradually clarified, the existence of general insertion reactions has become more apparent.[1,2]

Isocyanates are probably the most powerful acceptors studied, forming adducts which are the products of addition across the N=C group to form carbamates rather than across the C=O group to form carbimidates. The reactions and reactivity of isocyanates can best be understood by considering the electronic structure of the isocyanate group where internal conjugation leads to charge separation between the nitrogen, carbon, and oxygen atoms, as indicated in equation (2), where charged structures *IV* and *VI* are major contributors.[3]

$$\underset{(IV)}{R-\overset{(-)}{N}-\overset{(+)}{C}=O} \leftrightarrow \underset{(V)}{R-N=C=O} \leftrightarrow \underset{(VI)}{R-\overset{(+)}{N}=\overset{(-)}{C}-O} \qquad (2)$$

$$\updownarrow$$

$$\underset{(VII)}{R-\overset{(+)}{N}\equiv C-\overset{(-)}{O}}$$

Although considerable work has been carried out on insertion reactions,[4] and Burger[5] has reported that titanium alkoxides and isocyanates form 1:1 adducts, the first report of polyinsertions involving metal alkoxides and isocyanates appeared by Meth-Cohn in 1970.[6] He reported that titanium alkoxides (*VIII*) react rapidly with either alkyl or aryl isocyanates (*IX*) at 0°C by insertion into the Ti-O bonds to form mono-, di-, tri and tetra -*N*- titanourethanes (*X*) according to equation (3),

$$\underset{(VIII)}{Ti(OR^1)_4} + \underset{(IX)}{nR^2NCO} \xrightarrow{0°C} \underset{(X)}{(R^1O)_{4-n}\,Ti\,(N<^{R^2}_{CO_2R^1})_n} \qquad (3)$$

Presented, in part, at the 62nd Annual Meeting of the Federation of Societies for Coatings Technology in Chicago, IL, October 24, 1984.

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A thorough literature search has not revealed any additional publications in this area since then, although publications exist regarding similar reactions, such as between titanium alkoxides and acetyl isocyanates.[7-9]

The major drawback in utilizing the insertion reaction between isocyanates and titanium alkoxides to develop low temperature crosslinking systems lies in the general reactivity of titanium alkoxides towards nucleophiles such as alcohols[10-14] and especially water.

Titanium alkoxides are in many respects analogous to, although more reactive than, tetralkoxysilanes. Their alkoxide groups hydrolyze rapidly on exposure to water yielding high molecular-weight condensation polymers *(XI)* with backbones consisting of alternating atoms of titanium and oxygen. The products of the partial hydrolysis of titanium alkoxides were first reported by Boyd[15] in 1951 who outlined the following scheme [equation (4)].

$$(RO)_4Ti + (n\text{-}1)H_2O \rightarrow (RO)_3TiO\left[\begin{matrix}OR\\|\\Ti\text{-}O\\|\\OR\end{matrix}\right]_{n-2} Ti(OR)_3 + 2(n\text{-}1)ROH \quad (4)$$

(XI)

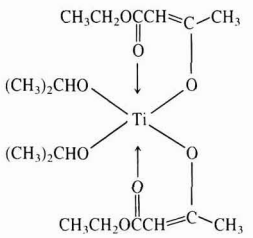
According to Boyd, further hydrolysis of the alkoxy containing products proceeds stepwise, even at room temperature, until titanium dioxide or hydrated titanium dioxide ($TiO_2 \cdot nH_2O$) is produced.

Although the ease of hydrolysis of simple titanium alkoxides, such as titanium tetra-isopropoxide *(XII)*, obviates the use of these materials in practical

$$Ti\left[OCH(CH_3)_2\right]_4$$

(XII)

crosslinking reactions, titanium alkoxides containing bulky groups are reported to be more resistant to hydrolysis. An evaluation of available titanates showed that the commercially available titanium chelate compound bis-(ethyl-3-oxo-butanolato-O^1,O^3)bis(2-propanolato) titanium (Tyzor DC®) *(XIII)* resists hydrolysis[16] and this material was examined as a potential crosslinking agent for isocyanate containing copolymers.



(XIII)

EXPERIMENTAL

Raw Materials

Phenyl isocyanate, cyclohexyl isocyanate, bis(ethyl-3-oxobutanolato-O^1,O^3) bis(2-propanolato)titanium (Tyzor DC) and isocyanatoethyl methacrylate (IEM) were commercial materials of the highest available purity and were used without further purification. Tyzor DC was obtained from the DuPont Co. IEM was obtained from the Dow Chemical Co. All the solvents used were purified in the usual manner.

Insertion Reactions Of Isocyanate Model Compounds

Tyzor DC in dry toluene was treated with the required molar amount of isocyanate in the same solvent, generally at ambient temperature. The course and extent of the reaction was monitored by the disappearance of the isocyanate absorption band in the infrared region of the electromagnetic spectrum using infrared solvent cells. Strict anhydrous conditions were maintained throughout.

Spectral Analysis

A FT-80A 80MHz Varian NMR spectrophotometer and a Perkin-Elmer infrared spectrophotometer were used for the spectral identification. A Waters Associates Gel Permeation Chromatograph (E-Linear column) was used to determine the molecular weight of the polymers.

Tyzor is a registered tradename of DuPont Co.

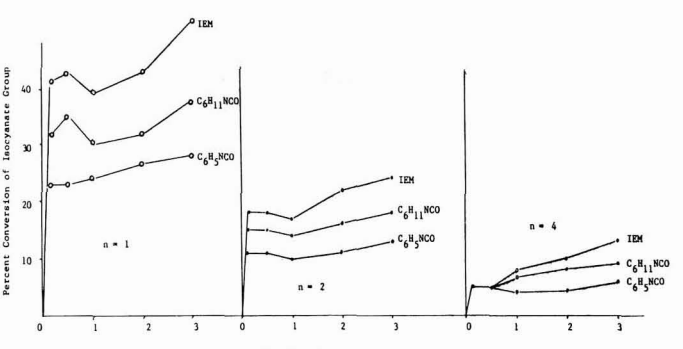


Figure 1—Percent conversion of isocyanate as a function of the amount and type of isocyanate. Legend: n RNCO + Tyzor DC → Product. Reactions run at 30% concentration by weight in toluene. Isocyanate content determined by IR analysis

Triphenyl Isocyanurate

After four weeks, crystals began to separate from a solution of phenyl isocyanate and Tyzor DC (2:1 and 4:1). These crystals were filtered and washed with dry hexane to yield triphenyl isocyanurate, white needles, m.p. 258° - 260°C; λ_{max} 1710 cm^{-1} (C=O) (in dry toluene); singlet centered at 7.42 ppm (monosubstituted benzene C_6H_5); mass m/e 357. The m.p. quoted in the literature[20] varies from 162-285°C and 285-286°C.

Copolymerization Reactions

A typical solution polymerization for preparing a copolymer containing 30% by weight of IEM and 70% methyl methacrylate (MMA) follows: One hundred grams of urethane grade 2-ethoxyethyl acetate, predried over 4-H molecular sieves, is heated to 115°C in a dry, nitrogen purged, 3-necked 500mL round bottom flask. A mixture of sieve dried IEM(30g) and MMA(70g) and azobisisobutyronitrile (AIBN) (1.5g) are added drop-wise to the stirred heated solvent over a two-hour period and post-reacted by adding additional AIBN (0.5g) in 25mL of 2-ethoxyethyl acetate over three hours while maintaining the temperature at 115°C. Purification of the polymer was accomplished by adding the copolymer in 2-ethoxyethyl acetate to dry hexane with the precipitation of the polymer. The precipitated polymer was filtered and vacuum dried at room temperature overnight to remove any residual solvent. The products contain 7.3% (by weight) of isocyanate (91.25% of the theoretical isocyanate) as determined by titration.[21]

To determine molecular weights, the dissolved polymer was post reacted with excess dry methanol at 65°C for two hours, precipitated with hexane and dried in a vacuum oven at 60°C for three hours, then analyzed by GPC. The molecular weight of the second polymeric system containing 4.6% of isocyanate (92% of the theoretical isocyanate) was determined by the same procedure.

Crosslinking Reaction

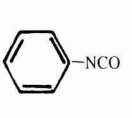
The required amount of Tyzor DC in dry toluene was mixed with the required molar amount of isocyanate containing polymer (IEM/MMA) in a Erlenmeyer flask at ambient temperature and shaken vigorously. These liquid samples were applied to steel panels by using a 0.002 inch Bird applicator either in (a) ambient air or (b) nitrogen controlled dry box and the degree of crosslinking determined periodically using the Sward Hardness method.

Infrared analysis of the polymer crosslinking reaction was carried out using a single NaCl prism for ambient conditions and sealed between two NaCl prisms for dry conditions.

RESULTS AND DISCUSSIONS

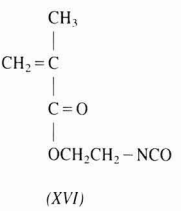
Model Reaction Studies

To establish the scope of this reaction, Tyzor DC was reacted with varying amounts of phenyl isocyanate *(XIV)*, cyclohexyl isocyanate *(XV)*, and 2-isocyanatoethyl methacrylate *(XVI)* in solution. Under these conditions,



(XIV)

(XV)



(XVI)

all of the model isocyanates reacted with Tyzor DC, with insertion occurring exclusively across the Ti-O Pr^i bond according to the following scheme [equation (5)].

$$nRNCO + Ti(EEAA)_2(OPr^i)_2 \rightarrow (EEAA)_2(OPr^i)_{2-n}Ti(NCOPr^i)_n \quad (5)$$

(O above the second N-C bond; R below the second N-C bond)

Where: EEAA = Ethyl Acetoacetate
OPr^i = Isopropoxide

Each reaction was monitored by infrared spectroscopy using ^1H and ^{13}C NMR spectroscopy to examine the structure of the products.

REACTION OF PHENYL ISOCYANATE WITH TYZOR DC: Although Meth-Cohn reported that one molar equivalent of titanium tetraisopropoxide *(XII)* reacts readily and reversibly with four molar equivalents of phenyl isocyanate in benzene solution at room temperature,[6] we observed with Tyzor DC a much lower initial conversion of the

Table I—Moles of Phenyl Isocyanate Converted In the Reaction with Tyzor DC

	Time				
n[a]	5 min.	1/2 Hr.	1 Hr.	2 Hrs.	3 Hrs.
1.....	0.23	0.23	0.24	0.27	0.28
2.....	0.22	0.22	0.20	0.22	0.26
4.....	0.20	0.20	0.16	0.16	0.22

(a) Moles of isocyanate to 1.0 mole of Tyzor DC.

Table 2—^1H & ^{13}C NMR Spectra Chemical Shifts Of Solutions of Insertion Products in Dry Toluene

	—CH=Proton	—CH Proton	—CH= Carbon	—C—H Carbon	Isopropyl Me Carbon
n[a]	Δδ (ppm)[b]	Δδ (ppm)[b]	Δδ (ppm)[c]	Δδ (ppm)[c]	Δδ (ppm)[c]
1.....0		+0.02	+0.09	+1.10	−0.03
2.....0		+0.03	+0.07	+0.59	−0.03

A negative value indicates that the resonance is the up-field from the reference.

(a) Moles of isocyanate to 1.0 mole of Tyzor DC.
(b) Refers to the chemical shift difference comparing to the same group in Tyzor DC in ^1H NMR: i.e. $\delta_{-CH} = 4.95$ ppm, $\delta_{-CH} = 4.85$ ppm.
(c) Refers to the chemical shift difference comparing to the same group in Tyzor DC in ^{13}C NMR; i.e. $\delta_{-CH} = 88.91$ ppm. $\delta_{-CH} = 67.91$ ppm, $\delta_{-CH_3} = 25.09$ ppm.

Table 3—Moles of Cyclohexyl Isocyanate Converted In the Reaction with Tyzor DC

	Time				
n[a]	5 min.	1/2 Hr.	1 Hr.	2 Hrs.	3 Hrs.
1.....	0.32	0.35	0.30	0.32	0.38
2.....	0.28	0.28	0.27	0.30	0.35
4.....	0.20	0.20	0.28	0.32	0.32

(a) Moles of isocyanate for 1.0 mole of Tyzor DC.

Table 4—Moles of Isocyanatoethyl Methacrylate Converted in the Reaction with Tyzor DC

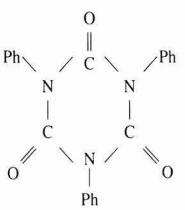
	Time				
n[a]	5 min.	1/2 Hr.	1 Hr.	2 Hrs.	3 Hrs.
1.....	0.41	0.43	0.39	0.43	0.52
2.....	0.36	0.36	0.34	0.44	0.48
4.....	0.20	0.20	0.34	0.40	0.48

(a) Moles of isocyanate for 1 mole of Tyzor DC.

isocyanate with the infrared spectrum showing a maximum of 0.28 moles of isocyanate converted for each mole of Tyzor DC during the first three hours of the reaction (*Table* 1, *Figure* 1). Even after prolonged periods of time, infrared analysis showed that less than two moles of isocyanate are reacting for each mole of tetrafunctional Tyzor DC.

After four days, NMR analysis of the liquid products showed the isopropoxide methine protons as a broadened multiplet overlapping with the methylene protons of the ethyl acetoacetate ligands, indicating the existence of considerable non-equivalence due to the presence of crowded and different substituents around the central titanium atom. Increasing the probe temperature to 55°C shifted the ^{1}H NMR spectra and caused an increase in the NCO absorption in the infrared, as the products reverted back to the starting compounds. The downfield shift of the isopropoxide methine protons coupled with no shift in the olefinic protons ($-CH=C$) indicates that the insertion reaction occurs primarily, if not exclusively, across the Ti-OPr^i bond rather than the Ti-ethyl acetoacetate bond (*Table* 2). This evidence is supported by ^{13}C NMR where the methine carbons of the isopropoxide group (C_7 and C_7') appear as two sharp peaks near 69 ppm indicating the presence of different substituents on the isopropoxide groups (*Figure* 2).

In addition, the appearance of the amide carbonyl absorption band (1700 cm^{-1}) in the IR spectrum confirmed the structure of the products. No NH absorption, indicative of urethanes, was observed indicating that water vapor had not entered the system. When the liquids from the 1:2 and 1:4 adducts were set aside for approximately four weeks, white crystals formed in small amounts (< 7%). By mass spectroscopy and ^{1}H NMR these crystals were identified as triphenylisocyanurate *(XVII)*.



(XVII)

REACTION OF CYCLOHEXYL ISOCYANATE WITH TYZOR DC: The reaction of cyclohexyl isocyanate with Tyzor DC was faster than the reaction of phenyl isocyanate, with approximately 0.35 moles of the available isocyanate converted during the first three hours of the reaction (*Table* 3 and *Figure* 1).

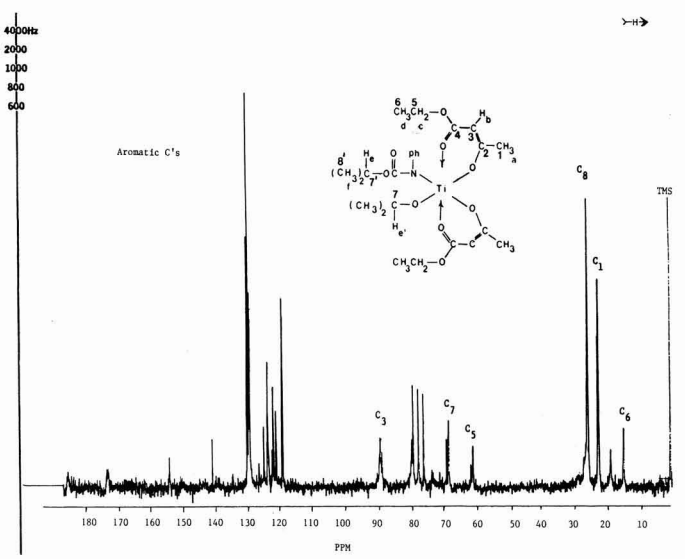


Figure 2—^{13}C NMR spectrum of the product from the reaction of phenyl isocyanate with Tyzor DC (1:1) at room temperature

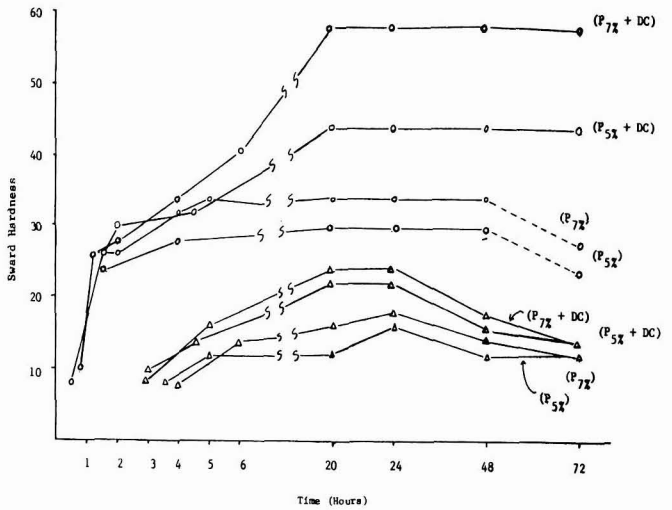


Figure 3—Sward Hardness data generated from the reaction of isocyanate containing polymer with Tyzor DC. Legend: o = ambient conditions; Δ = under nitrogen; 1:1 ratio of isocyanate to Tyzor DC used in all runs. All samples run at room temperature; subscripts indicate the approximate amount of NCO in percent by weight of the polymer

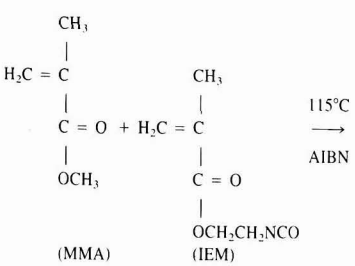
REACTION OF 2-ISOCYANATOETHYL METHACRYLATE (IEM) WITH TYZOR DC: IEM proved to be the fastest isocyanate studied with approximately 0.50 moles of the available isocyanate groups converted during the first three hours of the reaction (*Table* 4 and *Figure* 1). Within three days, the NCO peak had completely disappeared for the 1:1 adduct.

The order of reactivity, as reproduced in equation (6), is best explained on the basis of the steric factor, rather than the electron inductive effect, since the bulkiness of the groups attached to the isocyanate functionality parallels the reactivity of the model compounds.

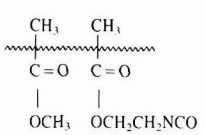
$$CH_2 = C(CH_3)COCH_2CH_2NCO > C_6H_{11}NCO > C_6H_5NCO \quad (6)$$

Synthesis of Isocyanate Containing Copolymers

Acrylate copolymers containing varying amounts of pendent isocyanate groups were synthesized from methyl methacrylate and 2-isocyanatoethyl methacrylate according to the following reaction [equation (7)].



(7)



(MMA/IEM Copolymer)

The polymer, as purified by known procedure, was determined to have a $\bar{M}n$ = 11,000 g/mole and a $\bar{M}w$ =

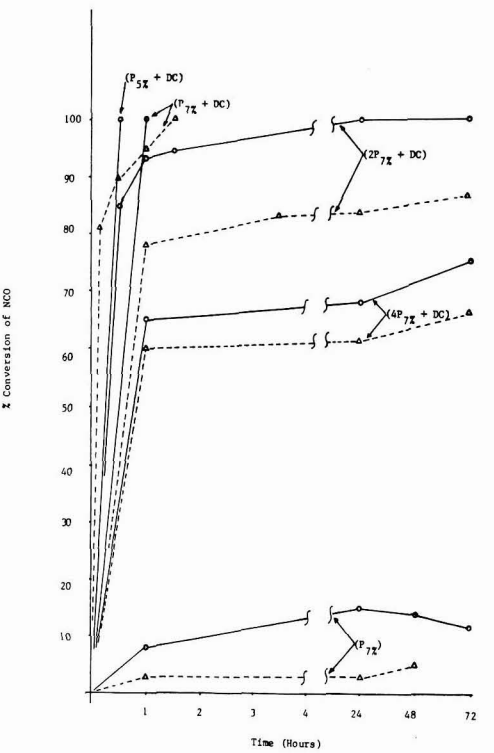


Figure 4—Conversion of the isocyanate containing polymer by reaction with Tyzor DC under atmospheric (ambient) conditions and under nitrogen. Legend: o = ambient conditions; Δ = under nitrogen. Ratios are based on moles of NCO per mole of Tyzor DC. All samples run at room temperature as 30% by weight solutions in toluene; subscripts indicate the approximate amount of NCO in percent by weight of the polymer

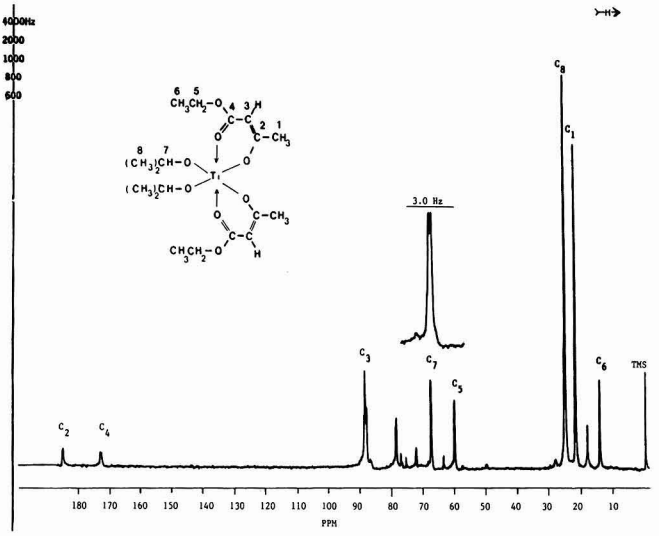


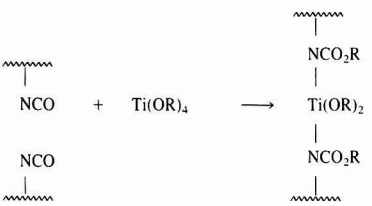
Figure 5—^{13}C NMR spectrum of Tyzor DC

26,500 g/mole by GPC analysis when compared to a polystyrene standard calibration curve.

Polymer Crosslinking Studies

Two polymeric methacrylate systems containing 4.6% NCO and 7.3% NCO were mixed with 1 mole equivalent of Tyzor DC, deposited as films and allowed to crosslink in air containing ambient moisture (ca. 50% RH) and in a controlled dry nitrogen atmosphere using the Sward Hardness method as a measure of the degree of crosslinking. Polymer systems free of Tyzor DC were used as controls in order to evaluate the difference between crosslinking via (a) the titanium compound and (b) normal curing of the isocyanate by atmospheric moisture [equation (8)].

CROSSLINKING VIA TITANATE COMPOUND:



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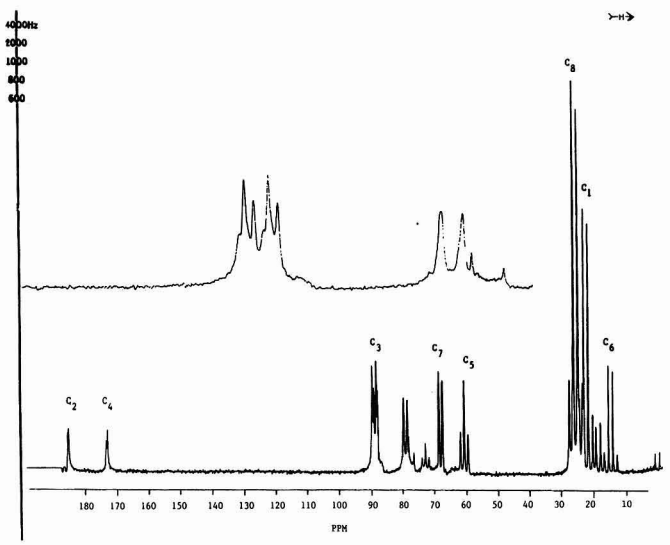


Figure 6—Off-resonance decoupled ^{13}C NMR spectrum of Tyzor DC

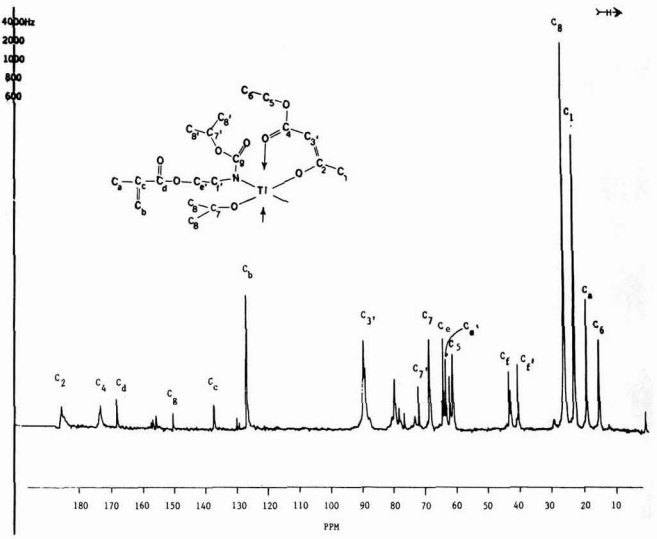
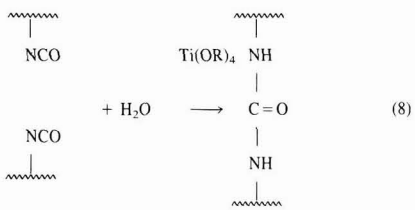


Figure 7—^{13}C NMR spectrum or reaction product of IEM and Tyzor DC (1:1)

CROSSLINKING VIA TITANATE CATALYZED MOISTURE CURE:



(8)

The results, as indicated by the Sward Hardness data (*Figure* 3), show a rapid increase of the hardness using Tyzor DC as the crosslinker in ambient moisture with maximum hardness achieved within 20 hours at room temperature with no further increase observed up to 72 hours. By comparison, polymers without Tyzor DC, cured under conditions of ambient moisture, did not achieve as great a degree of cure as indicated by the Sward Hardness value. Under nitrogen, the hardness of the polymer alone and the polymer mixed with Tyzor DC increased only slightly and never achieved the hardness level of the systems cured in ambient air.

Although the Sward Hardness data indicates that Tyzor DC, under conditions of ambient moisture, is an effective crosslinker for isocyanate containing polymer systems whereas little, if any, crosslinking occurs under nitrogen,

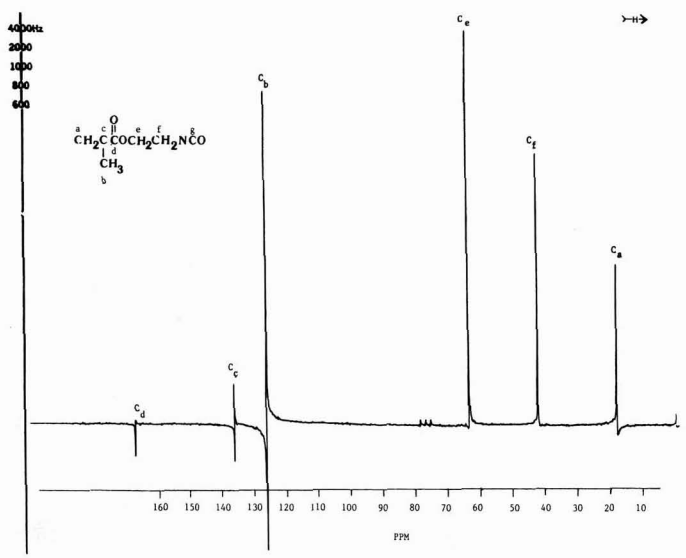
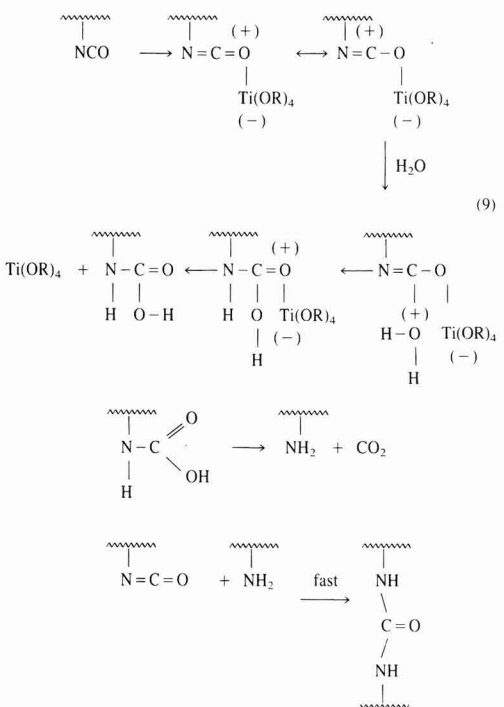


Figure 8—^{13}C NMR spectrum of 2-isocyanatoethyl methacrylate (IEM)

interpretation of the infrared data leads us to an entirely different conclusion. Examination of the IR data *(Figure* 4) shows a rapid decrease of the NCO peak over a one hour period when the polymer is mixed with Tyzor DC and allowed to react as a thin film either in contact with air or under dry nitrogen. This data, in conjunction with the previous efforts reported on the model compounds as well as the Sward Hardness data indicates that although Tyzor DC reacts rapidly with NCO groups under nitrogen the resulting product does not contribute in a meaningful fashion to the hardness as indicated by the Sward Hardness. In air, the reaction is as rapid as evidenced by the disappearance of the NCO group, but in this case, the hardness does increase rapidly. From this information, we conclude that the NCO groups are being consumed differently when reacted with Tyzor DC in contact with atmospheric moisture or under nitrogen.

At this point, we are tempted to conclude that Tyzor DC reacts with isocyanate containing copolymers under nitrogen to form a crosslinked polymer where the crosslinking group involves the insertion of the NCO group into a limited number of Ti-OPr^i bonds in the Tyzor DC molecule leading to a soft polymer. On the other hand, in the presence of atmospheric moisture, the crosslinking reaction appears to be more efficient leading to a harder polymer as evidenced by the Sward Hardness data. Since urea groups tend to produce hard polymers it is possible, in this instance, that Tyzor DC functions as a catalyst in promoting the reaction between atmospheric moisture and the isocyanate group. The following scheme outlines a potential mechanism for this reaction [equation (9)].[22,23]



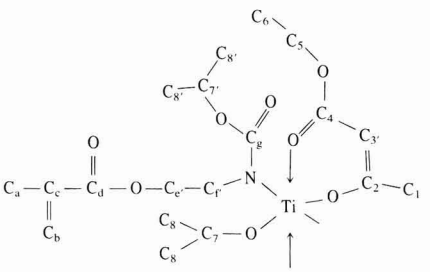
SPECTROSCOPIC STUDIES

Tyzor DC

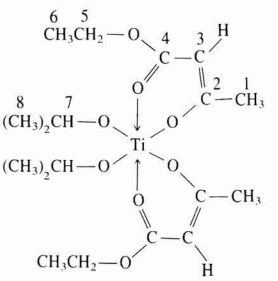
The ^{13}C and ^{13}C off-resonance decoupled spectrum of Tyzor DC are reproduced in *Figures* 5 and 6. The ^{1}H NMR spectrum is consistent with known data.[17-19] In the case of ^{13}C NMR spectra, carbon atoms 2,3,4, and 7 are split into doublets due to the existance of this compound in the *cis* configuration which belongs to C_2 symmetry point group. The assignment of the peaks is based on the ^{13}C off-resonance decoupled spectrum (*Figure* 6).

Reaction of Tyzor DC With 2-Isocyanatoethyl Methacrylate

The ^{13}C NMR spectrum for the 1:1 adduct (XVIII) (*Figure* 7) when compared to the ^{13}C NMR spectrum of Tyzor DC *(XIX)* (*Figure* 5) shows a shift of the methine carbon of the isopropyl group from 0.68 ppm to 0.73 ppm (see 7 and 7′ of *Figure* 7) indicating insertion of IEM into the isopropoxide group. No evidence for insertion in the ethylacetoacetate group was observed from the NMR spectra. Shifts in the field positions of carbons e and f are also observed as expected for the insertion reaction (*Figures* 7 and 8).



(XVIII)



(XIX)

CONCLUSIONS

In conclusion, it appears that Tyzor DC functions as an ineffective crosslinker in reactions with isocyanate containing polymers, at or near room temperature, except in the presence of ambient moisture where effective cross-

linking, as evidenced by Sward Hardness, is observed to take place. Under these conditions, Tyzor DC might well be functioning as a catalyst in promoting the reaction between ambient moisture and the isocyanate functionality of the polymer. Although the reaction between Tyzor DC and isocyanates appears reversible in solution, at temperatures below 55°C no evidence of reversibility has been observed in unpigmented thin films. Efforts are continuing to elucidate the crosslinking reactions that occur between Tyzor DC and similar titanium compounds and isocyanates in the presence of ambient moisture at or near room temperature.

ACKNOWLEDGMENT

The authors wish to thank the Detroit Society for Coatings Technology and the Graduate School Research Fund at Eastern Michigan University for their support of this research. This article was submitted by Mr. Wang in partial fulfillment of an M.S. Degree in Chemistry at Eastern Michigan University.

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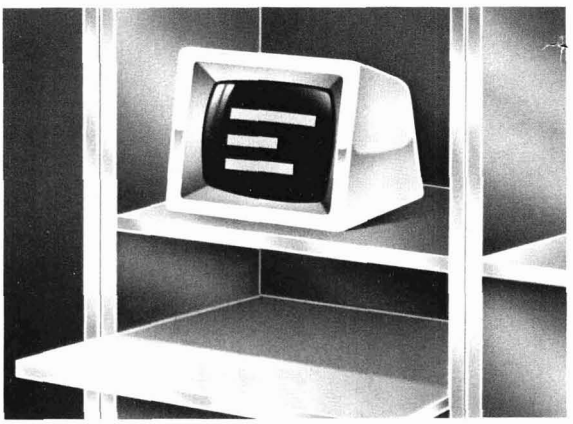
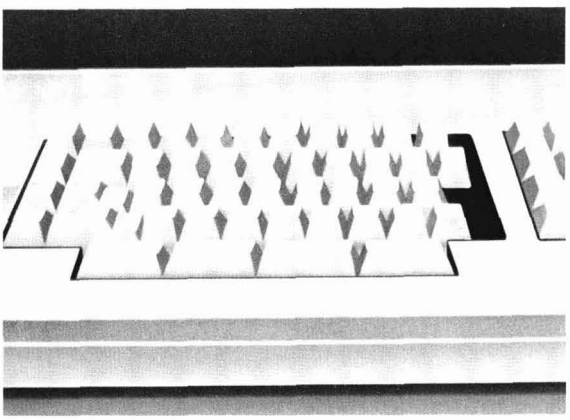
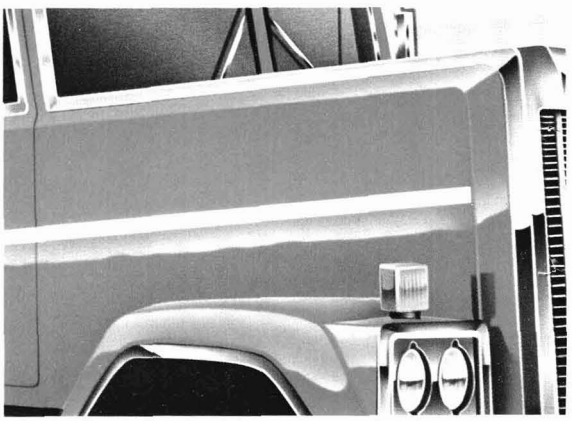
Errata

The ordinate in *Figure* 5 of the article, "Water-Borne Corrosion Inhibitive Primers Evolve to be Competitive," by the Golden Gate Society (JCT, July 1985, p 71), should read *Time to Rust Rating = 9, hours.*

Are Your Urethane Finishes TOUGH ENOUGH?







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Physical and Chemical Considerations On Copper Phthalocyanine

Calvin C. Wanser
CIBA-GEIGY Corporation*

Copper phthalocyanine is a member of a class of highly colored synthetic organic compounds with properties as pigments of excellent strength, chemical, light, and heat resistance. The two most important polymorphs are the beta form (greenish-blue shade) and the alpha form (reddish-blue shade). Halogenated copper phthalocyanines represent the green shades of this pigment. Modifications of copper phthalocyanine by physical and chemical means are made for use in coatings, inks, and plastic systems. Particle size and shape, flocculation resistance, crystal stability, and texture are important considerations in the end-use of these pigments. Chemical and physical variations may change the hue and optical properties in various systems.

INTRODUCTION

Copper phthalocyanine pigments have experienced a phenomenal rise in popularity over the past few decades. This is due to their properties of high strength, chemical and light resistance, cleanliness, and durability. The growth rate of these pigments has been rapid, increasing by 100% during the period 1952-1959.[1] Domestic copper phthalocyanine blue and green production in 1960 was 5.8 million pounds.[2a] The 1982 production was over 11.0 million pounds.[2b]

HISTORICAL AND BASIC PHTHALOCYANINE CHEMISTRY

Phthalocyanine pigments are a class of highly colored synthetic compounds whose basic structure is the tetrabenzoporphyrazine chromophore. Metal-free phthalocyanine was unknowingly prepared by Braun and Tcherniac[3] in 1907. They noted that when *o*-cyanobenzamide was heated above its melting point, a low yield of blue compound was formed. Copper phthalocyanine was first prepared by Dediesbach and Von der Weid[4] in 1927. They observed the formation of a deep blue crystalline solid by heating *o*-dibromobenzene, cuprous cyanide, and pyridine in a sealed tube. Dediesbach and Von der Weid formulated this product as a complex compound of the three starting materials.

About the time of publication of this work, Scottish Dyes Corporation observed the formation of a blue-green, highly colored impurity when ammonia was passed through molten phthalic anhydride in iron equipment. This compound was iron phthalocyanine. The company recognized the potentialities of such pigments and referred the matter to Professor R.P. Linstead.

The work of Linstead[5] and his co-workers formed the basis of our present knowledge of phthalocyanine compounds. Linstead coined the name phthalocyanine. Phthalo is derived from the name of the generic compounds, and cyanine refers to the deep blue coloration. Metal-free phthalocyanine contains two isoindole hydrogens which are readily replaced by metals, forming metal phthalocyanines. These metal derivatives may be electrovalent or covalent in nature. The electrovalent metal phthalocyanines are, in general, complexes of phthalocyanine and alkali or alkali earth metals (such as Na, K, Ca). Stable covalent metal phthalocyanines are formed from such metals as Cu, Ni, Co, and Zn. Less stable covalent derivatives are formed from Sn, Mg, Mn, and Be, for example. Many trivalent metals form phthalocyanine derivatives. In such cases, two of the valence bonds of the metal are linked to the phthalocyanine complex; the third remains ionic and reactive. Ferric chloride, for example, reacts with phthalonitrile to form chloroferric phthalocyanine. Hydrolysis of this compound

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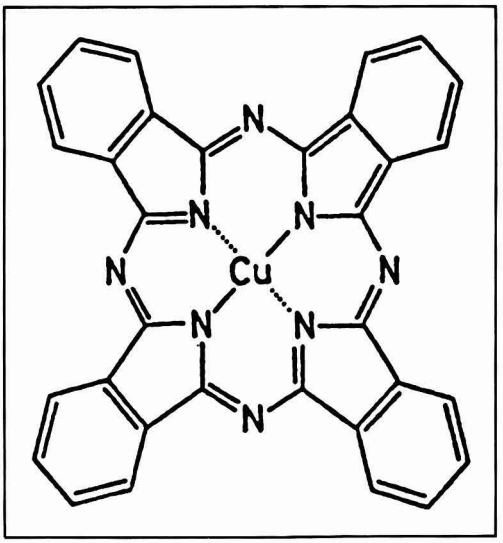

Figure 1—Copper phthalocyanine

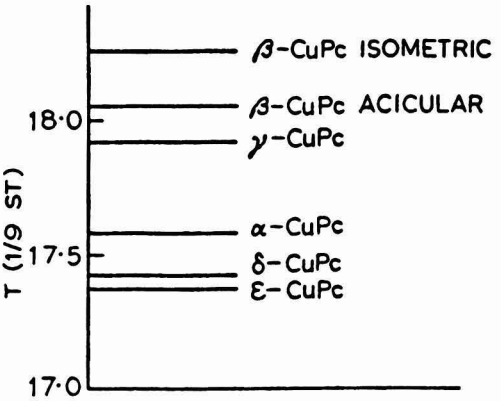

Figure 3—Differences in shade of five different crystal modifications of copper phthalocyanine as well as beta copper phthalocyanines of different particle shapes are noted. Beta copper phthalocyanine (isometric particles) are greener than beta copper phthalocyanine (acicular particles). Redness of shade is maximum at epsilon copper phthalocyanine. (Sappok, 1978)

yields hydroxy-ferric phthalocyanine and hydrogen chloride.

In addition to substitution of metals at the two active hydrogens in the central ring systems, 16 replaceable hydrogen atoms are present on the outer benzenoid rings. These hydrogen atoms are more reactive to halogenation than the isoindole hydrogens. Substitution may occur directly or indirectly on the intermediates for the phthalocyanine synthesis. Such substitution will result in products of varying hue and working properties.

COPPER PHTHALOCYANINE AND CRYSTAL MODIFICATIONS

The most important commercial phthalocyanine blue and green pigments are based on copper phthalocyanine (*Figure* 1). Their outstanding properties are high strength, brightness, and resistance to light, heat, and all common chemical agents except strong oxidizing agents (such as nitric acid). An example of the excellent stability

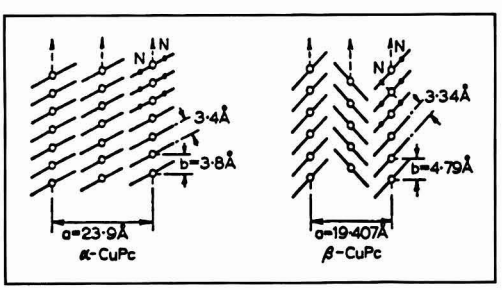

Figure 2—Alpha and beta copper phthalocyanines are represented by two different crystallographic planes with differences noted in the interplanar spacings. o:Copper, •:Nitrogen. (Sappok, 1978)

of copper phthalocyanine is the fact that it can be sublimed unchanged at temperatures as high as 600°C.

Copper phthalocyanine blue is approximately twice as strong as iron blue and four to seven times as strong as peacock blue lakes. Although copper phthalocyanine blues are much stronger than iron blues, the latter represent better money value based on the relative cost. The choice, therefore, of copper phthalocyanines over relatively lower cost pigments would depend upon the superior performance characteristics of the phthalocyanines.

The major use of copper phthalocyanine pigments is in inks. The general use of these pigments may be broken down as follows:

Coatings	30%
Inks (including Textiles)	55%
Plastics	15%
Total	100%

Different crystal forms of copper phthalocyanines are not merely differences in crystal shape but arise from different arrangements of the molecules in the crystal lattice.[6] This translates into differences in fundamental physical properties as well as pigmentary performance. For example, their X-ray diffraction patterns and infrared spectra are quite different.

Alpha and beta forms of copper phthalocyanine are of considerable practical importance (*Figure* 2). The alpha form is redder in shade and is almost always of a very small particle size. It tends to revert to the beta form under the action of heat and organic solvents. Thermodynamically, the alpha form is less favored. The beta form is greener in shade and the particle size may vary from very small up to needles two centimeters long. This form is favored thermodynamically. Other crystal forms exist and the shade variations are illustrated in *Figure* 3.

The shade shifts redder to greener from epsilon to delta to alpha to gamma to beta (acicular) to beta (isometric). Beta copper phthalocyanine is one of the modifications

which exhibits a proven dependency of hue on particle shape.[7]

Crystal Growth of Copper Phthalocyanine

Crystallization derives from the existence of alpha and beta forms of copper phthalocyanine. It can occur in any media but is most often recognized as "can fading" of paints containing aromatic solvents. When a paint is formulated with unstabilized alpha form and stored, color strength is lost and microscopical examination shows that the pigment is transformed to large needle-like crystals (*Figure* 4). These are found to be the beta form and regrinding will not break up these crystals and restore the original shade of the paint.

The mechanism of crystallization depends on the limited, but significant, solubility of copper phthalocyanine in solvents, particularly aromatic solvents. There is a dynamic equilibrium between the undissolved copper phthalocyanine molecules which are in the majority and the comparatively few dissolved ones. Molecules are continuously passing from the particle surfaces into solution and emerging from solution onto particle surfaces. Molecules coming out of solution would be expected to arrange themselves in the stable beta pattern. In addition, there is the process of Ostwald ripening in which larger particles in suspension grow at the expense of the smaller particles. The smaller particles are most likely to be the alpha form.

Beta copper phthalocyanine or the stabilized alpha form may be used to overcome crystallization defects. Stabilized alpha form is usually obtained by introducing a substituent so that the arrangement of the molecules to give the beta crystal is sterically difficult or impossible. By far the most important substituent is a single chlorine atom in the 4-position. Only the alpha form of 4-monochloro copper phthalocyanine exists, so that crystallization defects are not possible. It is interesting to note that 3-monochloro copper phthalocyanine has both alpha and beta forms presumably because the molecular shape is closer to the parent compound. It is not necessary to use pure monochloro copper phthalocyanine to obtain stability in practice. A mixture with unchlorinated material will afford crystallization resistance possibly because the chlorinated material causes a discontinuity on the growing crystal faces. Such mixtures can be made from chlorinated intermediates or by chlorination of copper phthalocyanine itself. These mixtures tend to be greener than pure alpha form.

Other substituents prevent formation of the beta form. For example, nitro, amino, and phenacetyl groups. Other metal phthalocyanines, such as tin, magnesium, and aluminum, suppress crystallization when present in quite small amounts. Such metals may upset the regular arrangement of the beta form crystal lattice.

A standard test for evaluating crystal stability is to grind the color and corresponding control in a lacquer system. Grinds of the white extender (TiO_2) are made at the same time. The blue is tinted off in the ratio of 12 parts pigment and 88 parts white. One set of panels is sprayed immediately. The remainder of the lacquer tint is aged one week at 140°F, and a second set of panels

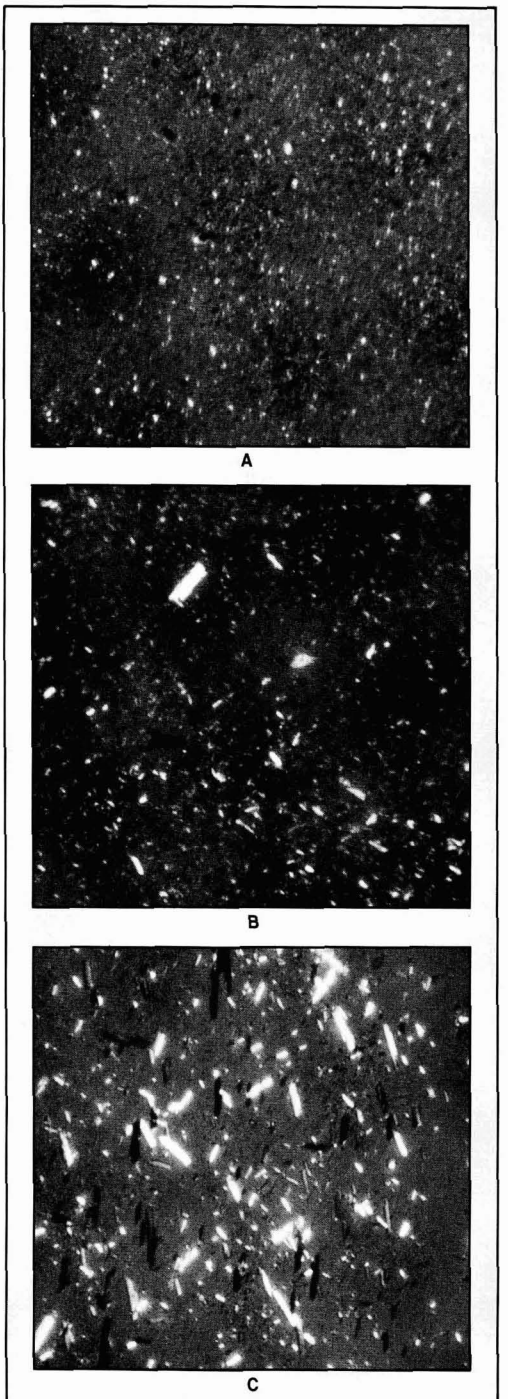

Figure 4— Crystals of unstabilized alpha copper phthalocyanine in a crystallizing medium: (A) alpha form dispersed in a crystallizing medium prior to crystal growth (250×); (B) partial crystal growth of the alpha form in the medium (250×); (C) extensive crystal growth of the alpha form in the medium (250×)

sprayed. The test may be extended to a month if desired. Loss of strength on aging is a measure of the degree of crystallization.

COPPER PHTHALOCYANINE SYNTHESIS AND PIGMENTATION

Several approaches exist for the preparation of copper phthalocyanine. The two more general synthetic routes involve:

(1) The reaction of a phthalic acid derivative, such as phthalic anhydride, with urea and a metal salt, such as cupric chloride, at temperatures approximating 200°C.

(2) The reaction of phthalonitrile and a copper salt, usually in an inert solvent, at about 200°C.

The products obtained from the above reactions are impure and the ultimate particle size too large for pigment application. It is necessary, therefore, to remove impurities and reduce the crystal size.

Conditioning Techniques

Pigment manufacturers strive to obtain particle sizes which maximize full tone intensity and transparency, tinting strength, and clarity of color. As the particle size decreases, texture deficiencies begin to occur as well as higher rheology. The attainment of size ranges which permit ease of application in various pigmented systems is referred to as conditioning. Acid pasting consists essentially of dissolving the copper phthalocyanine crude in concentrated sulfuric acid, followed by hydrolysis in excess water to reprecipitate pigment as finely divided particles almost completely in the alpha form. This is a physical action where pigment purification also occurs. The concentration of pigment in the acid may be varied and the precipitation temperature is a very important factor. It may vary from below 0°C-100°C or even higher if a closed system is employed for the dilution. Drip or spray methods of hydrolysis frequently produce some large particles. Introduction of a thin film of pasting into the water under pressure can produce a more uniform particle size. Control of size can also be obtained by causing precipitation from sulfuric acid by a change in temperature. For example, acid solution is made at 70-100°C and precipitation is effected by rapid cooling to -10°C-$+30$°C. Surfactants may be used during the hydrolysis to improve dispersibility of the product. Precipitation in the presence of water immiscible solvents can afford texture and strength enhancements.[7]

Another conditioning technique is known as the permutoid swelling. Here the pigment is treated with sulfuric acid at a lower concentration insufficient to dissolve it, but sufficient to form a slurry of the copper phthalocyanine sulfate as coarse green crystals. These crystals then hydrolyze to yield the alpha form on addition of water. Acid concentration may vary between 60-80% sulfuric acid and one can start with either dry copper phthalocyanine or presscake. Another consideration for particle size reduction is the type of agitation during the permutoid swelling. The alpha form is usually produced from this process. At sulfuric acid concentrations below about 60%, some beta form may remain unchanged. Acid treat-

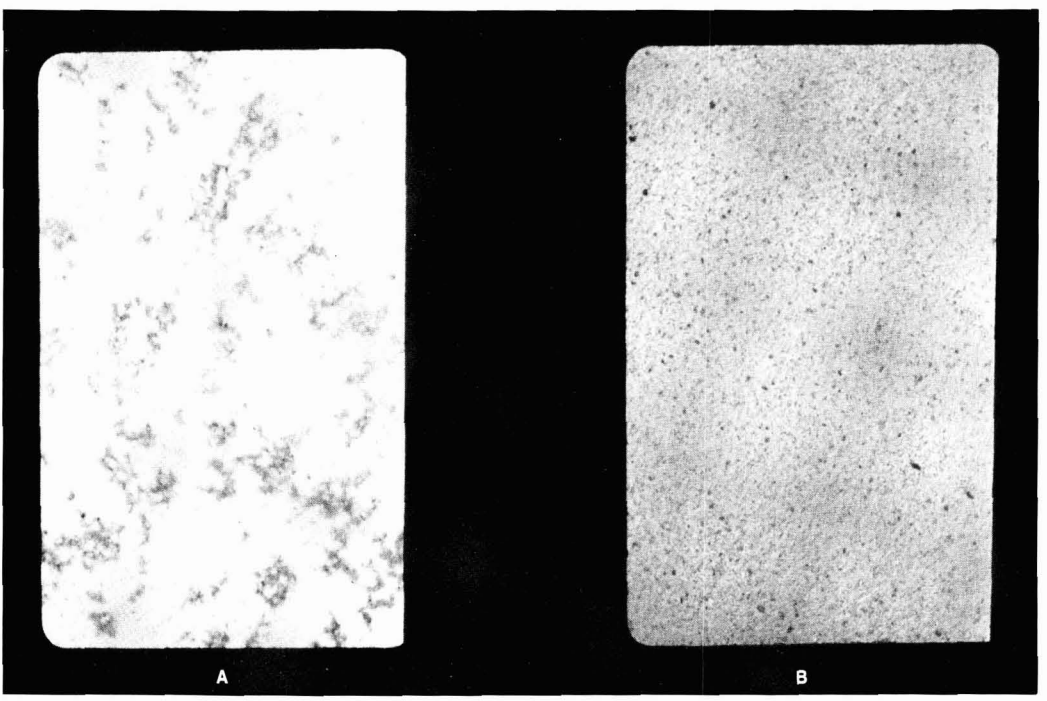

Figure 5—Flocculation of alpha form copper phthalocyanine in a vehicle system: (A) alpha form dispersed in the system prior to flocculation (250×); (B) alpha form after flocculation occurs (250×)

ment processes may also be applied to substituted copper phthalocyanines or mixed metal phthalocyanines.

Grinding techniques may be employed for conditioning copper phthalocyanine. In salt grinding, crude copper phthalocyanine is mixed with a grinding aid, such as inorganic salt, and the mixture subjected to attrition grinding in a ball mill, for example. This causes particle size reduction and also a phase change, any beta form being converted to the alpha form.

A variation would be to wet grind with a non-crystallizing solvent. The grinding process is much more time dependent than acid pasting. Production of beta copper phthalocyanine can be obtained by the salt grind process. One may start with the alpha or beta form and treat with a crystallizing solvent during or after grinding. If no solvent is present, the alpha form is produced regardless of the starting material.

The production of a satisfactory pigment depends on the balance of two mutually opposing forces: grinding, which increases pigment strength and tends to produce the alpha form; and crystallization, which decreases pigment strength and produces the beta form. Efficiency of the milling and increased grinding time gives increased strength. If these are overdone, however, an unacceptable amount of alpha is formed. Excess solvent and higher temperatures help to preserve the beta form, but if they are not carefully controlled a weak product is obtained. This type of grinding may also be accomplished with small media (such as steel shot) under aqueous conditions.

FLOCCULATION OF COPPER PHTHALOCYANINE

Copper phthalocyanine is planar and possesses molecular symmetry. It has a very small dipole and there is the absence of repulsive forces between the pigment particles. Flocculation is affected by the initial particle size and shape and surface characteristics of the pigment. It is equally affected by the vehicle. Ease of wetting and adsorption of the medium or solvent play a role and selective adsorption may be a complicating factor.

To prepare a copper phthalocyanine which will not flocculate significantly in any medium is a difficult task (*Figure* 5). The most generally successful means of conferring resistance to flocculation consists of the introduction of polar groups which impart a charge on the particle surface. Groups such as sulfonic and carboxylic acid are examples of this technique. However, less polar substituents such as hydroxymethyl or salts of *o*-carboxybenzamidomethyl groups are reported to be effective. This type of substitution must be carried out with care. If overdone, solubility in alkali may reduce the fastness of the pigment below an acceptable level. As found with stabilization against crystallization where a proportion of monochloro copper phthalocyanine confers resistance to a blend with unsubstituted material, only some of the copper phthalocyanine molecules need to be treated to yield flocculation resistance.

It should be noted that beyond a certain limit no further improvement is gained by increasing the content of polar molecules. It is customary to apply this method to a blue already stabilized against crystallization and, in addition, to coat the particles with a suitable resin to overcome poor texture or dispersibility thought to result from the presence of polar groups. The improvement here may also be due to an increased tendency of polar materials to associate with TiO_2.

Other methods of overcoming flocculation defects are:

(1) Addition of aluminum benzoate.

(2) Formation of copper phthalocyanine in the presence of titanium or zirconium oxides to yield an intimate mixture.

(3) Additions of vanadyl or tin phthalocyanines reduce flocculation, possibly because the central metal atom has valencies not taken up by the phthalocyanine ring which carry oxygen or chlorine atoms perpendicular to the plane of the molecule and upset its electronic symmetry.

It should be noted that beta form copper phthalocyanine is much less liable to flocculation defects than the alpha form or non-crystallizing alpha form. Furthermore, a cleaner and greener shade exists with the beta form.

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From 1956 to the present time, he has been involved in research and development work on organic pigment colors for Imperial Paper Color and Chemical Corporation, Hercules Incorporated, and CIBA-GEIGY Corporation. Mr. Wanser is currently employed by CIBA-GEIGY Corporation at Glens Falls, NY, as a Staff Scientist.

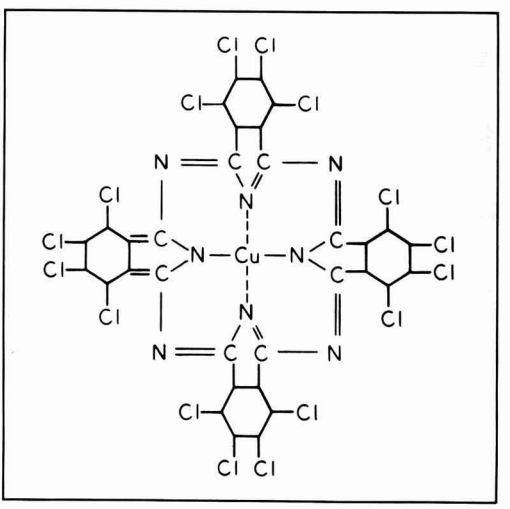

Figure 6—Phthalocyanine green. Hexadecachloro copper phthalocyanine

Since flocculation is reversible under conditions of high turbulence, as in the spraying of a paint, a test procedure is devised for evaluating flocculation. This is accomplished by comparing the sprayed and poured portions of the same paint grind. An alkyd enamel system is employed. The tinted paint contains one part of blue to 10 parts TiO_2 on a dry basis. After spraying a panel with the tint, a portion is then poured over the freshly sprayed panel. The apparent strength difference between the sprayed and poured portions is a measure of the degree of flocculation.

COPPER PHTHALOCYANINE GREEN

Copper phthalocyanine has 16 hydrogens on the benzenoid rings that may be replaced by chlorine, bromine, or fluorine. The pigment changes in shade from blue to green as the number of chlorine atoms in the molecule increases. The effect of the first atoms on shade is minor, and the effect increases rapidly to about 10-14 atoms and is less significant again for the final two atoms (*Figure* 6). Many commercial phthalocyanine greens contain 14-15 chlorine atoms. A combination of bromine and chlorine yields a yellower shade product than chlorine alone.

Pigmentation processes for phthalocyanine green may be similar to those used for the blue. Some processes require modification. It is, however, less complicated than with blue since dimorphism, and hence crystallization, does not occur. Flocculation tendencies are minimal. Although there are not two crystal forms of phthalocyanine green, there is a distinction between more or less crystalline forms. Crystallinity may be increased by appropriate solvent treatments. There is no substantial growth as compared to copper phthalocyanine blue. Green microcrystals can be characterized by X-ray diffraction and by electron microscopy—the more crystalline green is cleaner, stronger, and disperses easier than the starting material and has a sharper X-ray diffraction pattern.

SUMMARY

Copper phthalocyanines are outstanding pigments, offering excellent strength, brightness, and fastness properties. Their economy relative to lower cost pigments is dependent upon superior performance characteristics.

The major disadvantages are the tendencies to flocculate in paints and lacquers, crystallize in certain solvent systems, and exhibit texture deficiencies. These problems, as well as the search for new pigments in this class, form the basis for continuing research on synthesis modifications and pigment after-treatments.

Particle size, shape, distribution, and crystal structure as affecting pigment working properties are important considerations in innovative work.

References

(1) "Phthalocyanine Pigments; Explosion of Color Paints a Bright Future," *Oil, Paint & Drug Reporter*, Nov. 2, 1959.
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(7) Sappok, R., "Recent Progress in the Physical Chemistry of Organic Pigments with Special Reference to Phthalocyanines," *J. of the Oil and Colour Chemists' Assoc., 61*, No. 8, 299 (1978).
(8) Moser, F.H., and Thomas, A.L., "Phthalocyanine Compounds," Reinhold Publishing Corporation, 1963.

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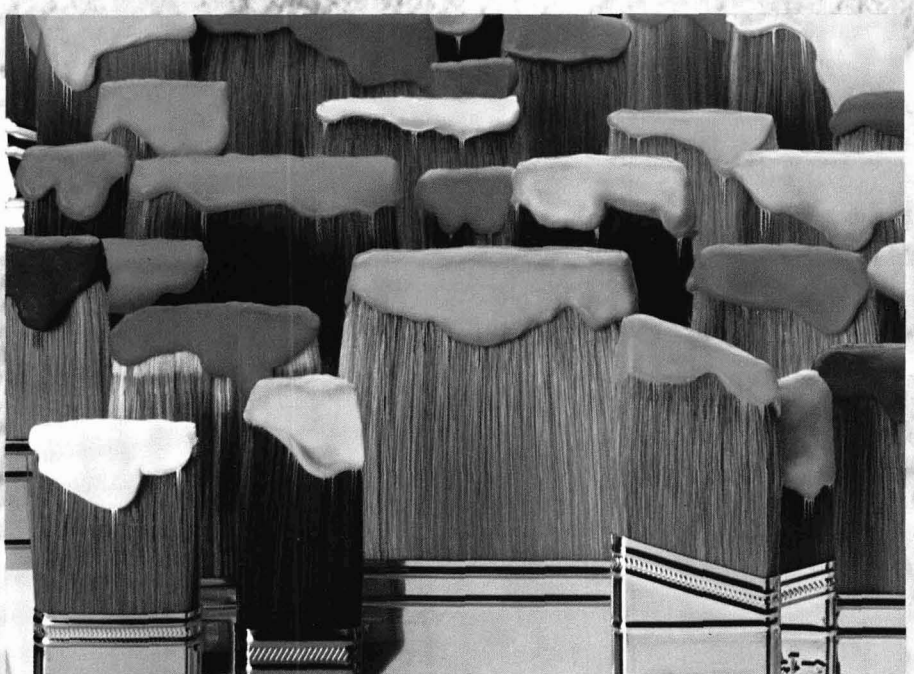

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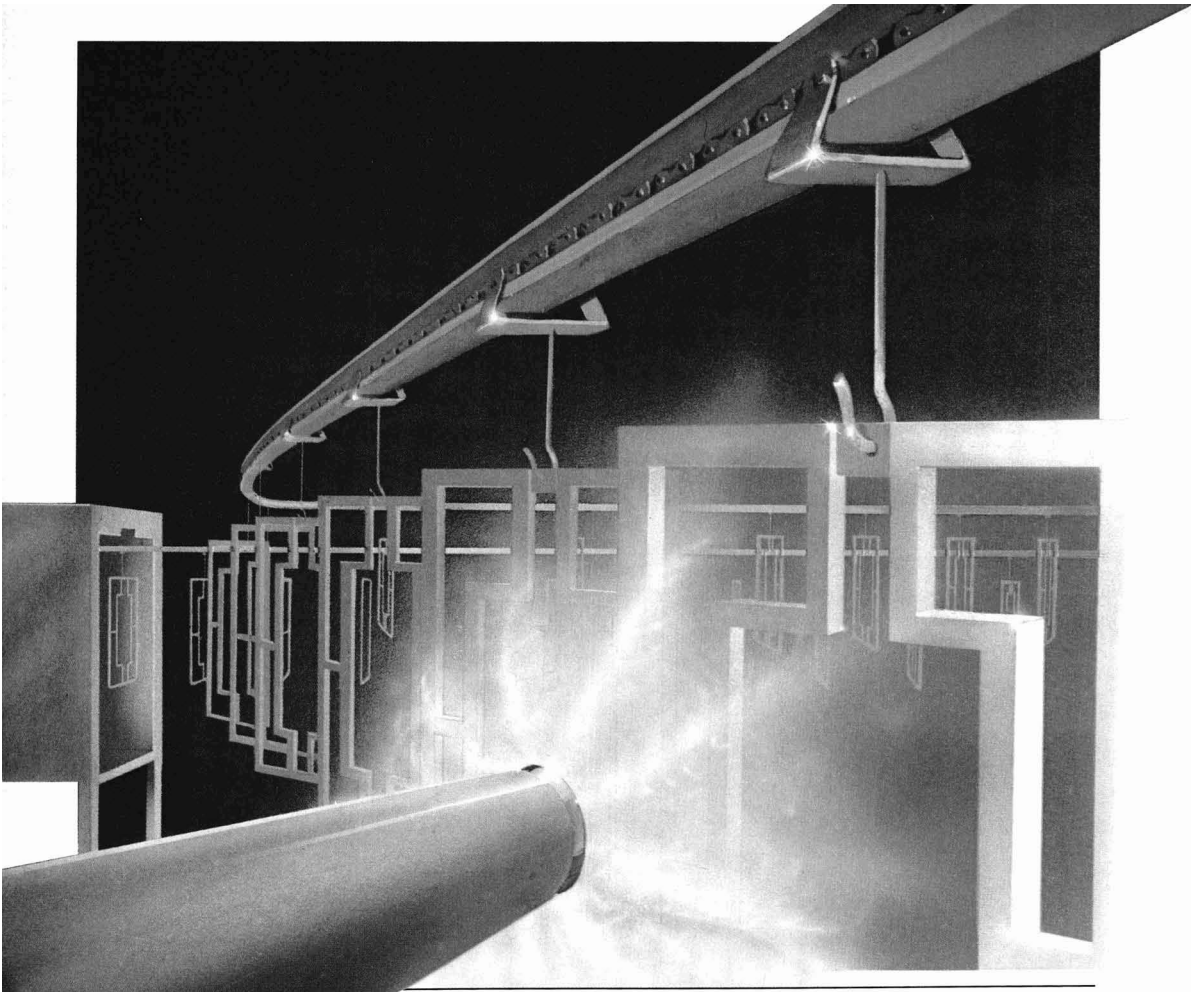

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"Future Scientific Developments" Will Be Theme Of September 1986 FATIPEC Congress in Venice

"The Scientific Developments in the Coatings and Printing Ink Industries on the Doorway of the 21st Century" will be the theme of the September 21-26, 1986 Congress of FATIPEC (the XVIIIth) in Venice, Italy.

FATIPEC is the Federation of Associations of Technicians in the Paint, Varnish, Lacquer, and Printing Ink Industries of Continental Europe. It is composed of seven member associations: from France, Belgium, Holland, Germany, Italy, Switzerland, and Hungary. The official languages of FATIPEC are French, German, and English.

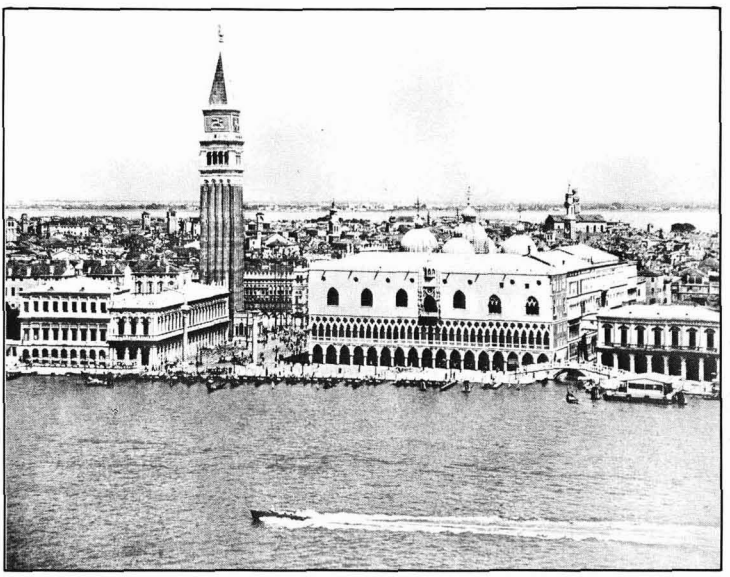
The hosts for the meeting in Venice will be the Italian member of FATIPEC: AITIVA (Associazione Italiana Tecnici Industrie Vernici e Affini) and the Italian Chemical Industry Association: FEDERCHIMICA (Federazione Nazionale dell'Industria Chimica Gruppo Industrie Vernici ed Inchiostri da Stampa).

The President of FATIPEC is Dr. Gianni Varasi, Managing Director of Maxfin S.p.A. Holding. The Vice-President, who will be in charge of arrangements for the event in Venice, is Dr. Amleto Poluzzi, of Ashland Chemical Co.

Anyone wishing to present a paper at the Congress must contact: Segreteria Organizzativa, del XVIII Congresso FATIPEC, c/o MGR s.r.l., Piazza S. Ambrogio 16, 20123 Milano, Italy.

Registration for the Congress is now open and forms are available from the Federation office in Philadelphia, or from the above address in Milano.

Running concurrently with the program sessions will be FATIPEXPO, an exhibition of raw materials, finished products, machinery, and other equipment.



Highlighting the Venetian waterfront are the San Marco belltower and the Doges' Palace

Gondolas provide a relaxing mode of transportation along the many canals of Venice

Toronto Society Initiates Certificate Coatings Program

Following recent government approval, a certificate coatings program has been instituted this fall by the Toronto Society for Coatings Technology. In addition, tacit approval has been given to the Society to initiate a part-time diploma program in 1986, the first in North America.

Directed by Peter Rodak, of George Brown College of Applied Arts and Technology, in Ontario, courses have been set for the first two years. The Society will designate courses for the third year when it is approved. Guest speakers from the coatings industry will complement the programs.

Evolved from the original Toronto Society courses started in 1949, these programs are designed to broaden the coatings knowledge of industry lab technicians, high school graduates, and sales representatives. Participants in these past courses, held at George Brown, can use their credits towards the two-year program, and towards the three-year program following approval. In addition, the Ontario government has sanctioned that a maximum of 50% of the needed credits can be obtained through equivalent work experience.

For additional information, contact Peter Rodak, George Brown College of Applied Arts and Technology, P.O. Box 1015, Stn. B, Toronto, Ontario, Canada M5T 2T9.

CALL FOR PAPERS

Water-Borne and Higher-Solids Coatings Symposium
New Orleans, Louisiana
February 5-7, 1986

The Southern Society for Coatings Technology and the Department of Polymer Science at the University of Southern Mississippi invite all interested persons to submit papers for presentation at the 13th Annual Water-Borne and Higher-Solids Coatings Symposium.

Papers relating to the chemistry, formulation, and marketing of water-borne, higher-solids and other advanced coating systems are solicited. Papers relating to engineering aspects of coating systems or solvent abatement are also solicited.

Title and abstract should be submitted as soon as possible to:

Dr. Gordon L. Nelson, Chairman
Department of Polymer Science
University of Southern Mississippi
Southern Station Box 10076,
Hattiesburg, MS 39406-0076

The completed paper should be submitted by *December 15, 1985*. Papers to be presented at the Symposium will be chosen based on abstracts.

It is preferred that all papers be original and of scientific value.

For additional information, contact Dr. Nelson at the above address.

DuPont Safety Management Package Offered

DuPont Safety Management Services is offering a special safety program which could reduce injuries up to 50% for companies or plants with 100 or fewer employees. Based on proven, cost effective techniques, the safety management package is a one-year program tailored to each client's specific needs.

For further information about DuPont's Small Business Package, contact DuPont Company, Safety Management Services, Barley Mill Plaza 19, Wilmington, DE 19898.

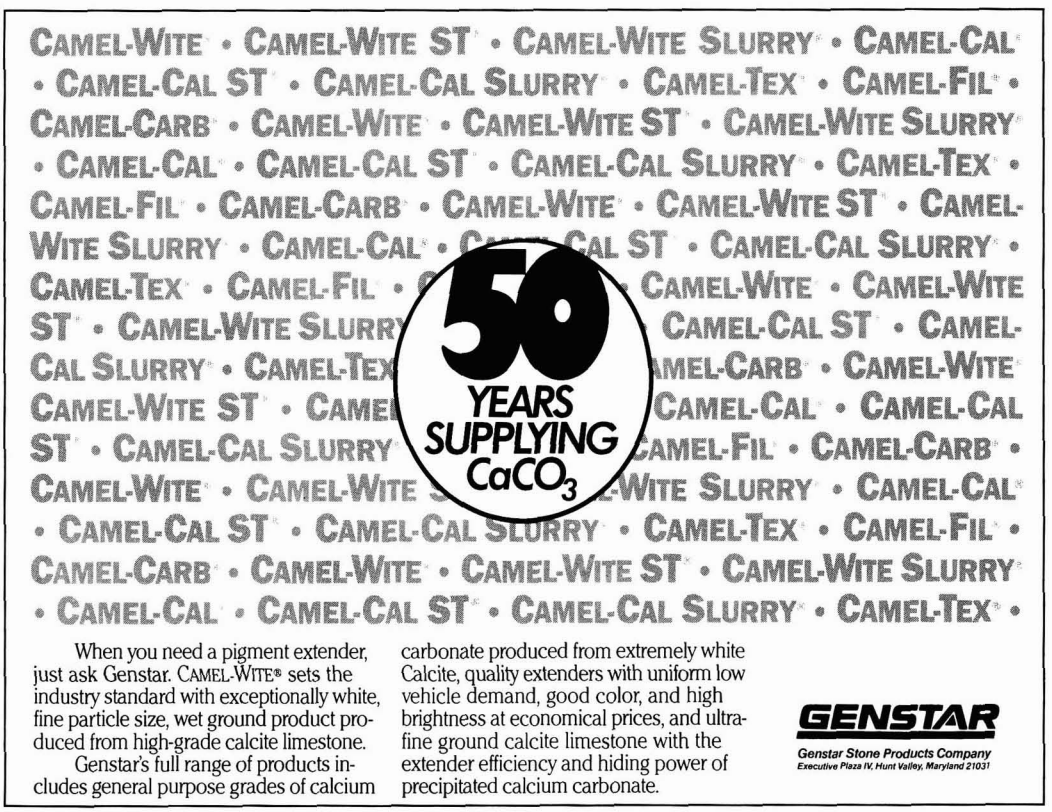
International Plastics Show Scheduled in China

CHINAPLAS 86, the third of a series of international exhibitions for plastics and rubber processing equipment and raw materials, will be held on June 10-16, 1986 at the China International Exhibition Centre, Beijing, China. Organized by Adsale Exhibition Services of Hong Kong and represented in the U.S. by Kallman Associates of Ridgewood, NJ, the trade fair and seminar program will emphasize plastics as a substitute for wood and steel products.

According to Adsale, China intends to update its equipment for producing polyethylene film for agriculture, as well as new materials for packaging. Among the areas to be discussed are multilayer films, rigid and soft PVC films, woven and nonwoven plastic sacks, boxes, bottles and containers, as well as injection molding machines and plastic foam products.

The 1984 show was held in Shanghai and attracted over 95,000 trade visitors and 198 international displays.

For complete details, contact Kallman Associates, 5 Maple Court, Ridgewood, NJ.



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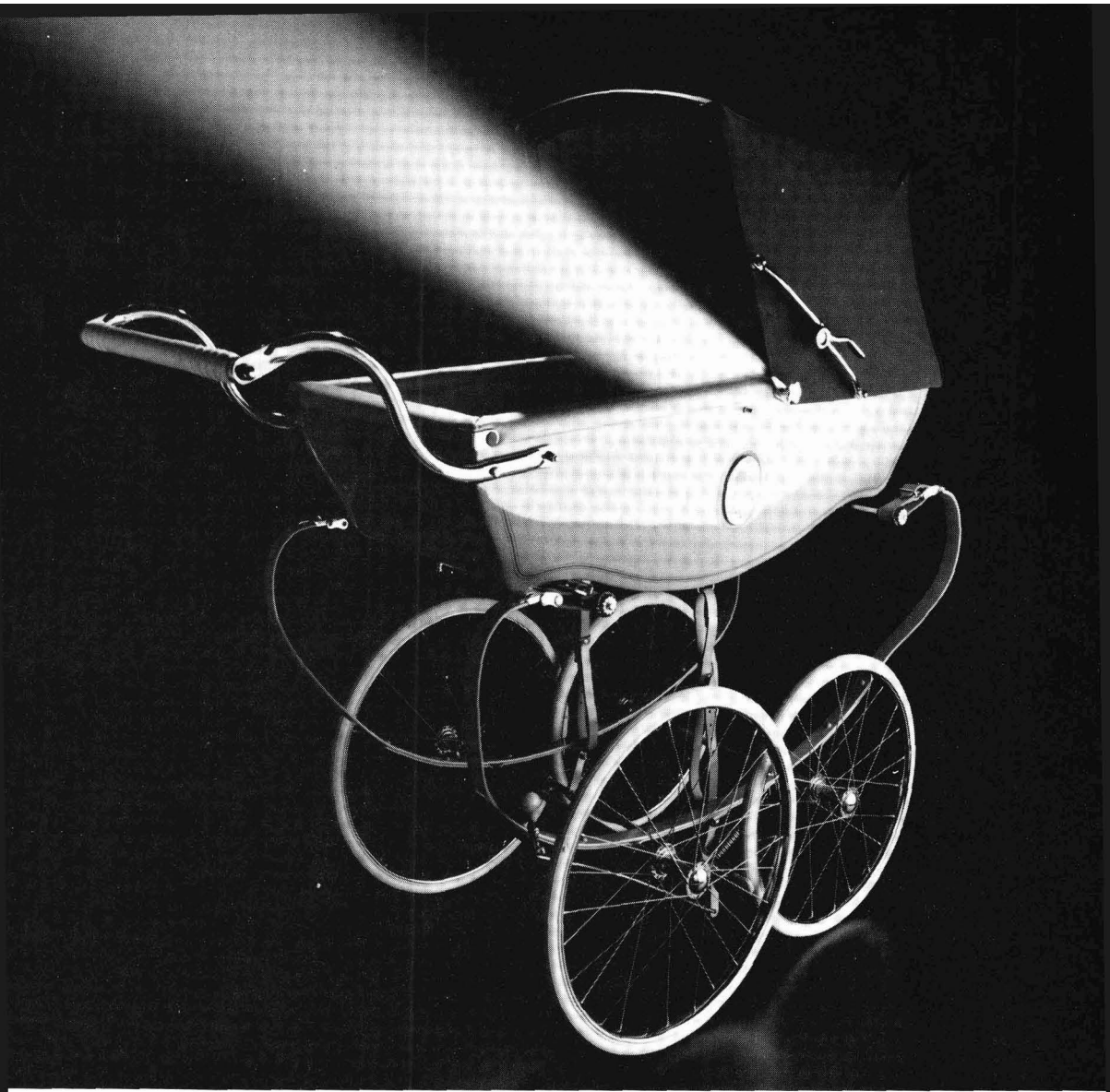

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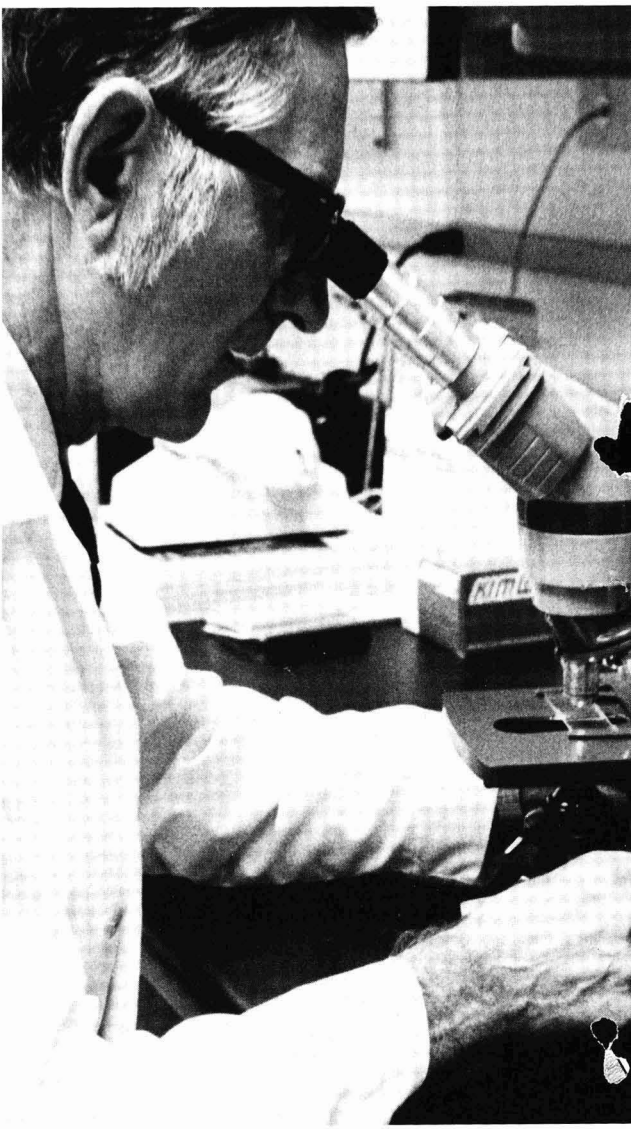
Ongoing research at DuPont's Haskell Laboratory for Toxicology and Industrial Medicine supports the use of DuPont DBE solvent as a substitute for E-series glycol ethers and acetates:

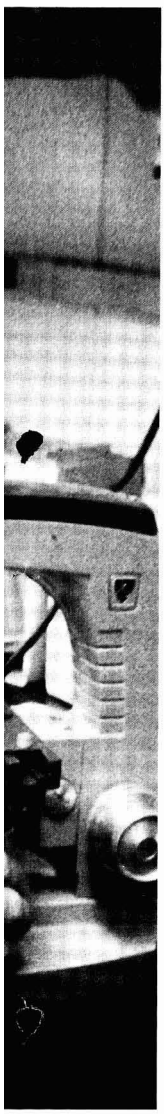
- DuPont DBE solvent has no effect on potency and fertility.
- DuPont DBE is not uniquely toxic to the fetus in teratogenic (fetal development) studies.
- Mutagenicity (genetic change) tests are negative.

Toxicity testing is continuing. Since the introduction of DuPont DBE as a blending solvent more than 10 years ago, no human health problems have been reported.

DuPont DBE cuts costs, too—as you'll see on the facing page.



to reformulate DBE solvent



Cost data underscore its proven economy

Reformulating with blends of DuPont DBE solvent can be your most cost-effective route to replacing E-series glycol ethers and acetates. The cost table (right) shows that DuPont DBE is a low-cost high-boiling ester solvent. As a result, you can hold down costs by making DuPont DBE your retarder solvent when formulating with P-series glycol ethers and acetates. The reformulating chart (below) shows how blends with DuPont DBE can ease the problem of solvent replacement.

Wide Application. DuPont DBE's solvency for many polymers accounts for its use in a wide range of coatings, both conventional and high-solids. In addition to automative topcoats and primers, these uses include steel and aluminum coil coatings, interior and exterior can coatings and finishes for appliances and metal furniture.

COMPARISON OF REPRESENTATIVE PRICES OF SLOW ESTER-TYPE SOLVENTS*

	¢/LB.	$/GAL.
Du PONT DBE	41.5-44	3.77-3.99
EE ACETATE	50.0-60.5	4.05-4.90
PM ACETATE	53.0	4.29
DPM ACETATE	53.0-69.5	4.29-5.63
DB ACETATE	64.5	5.06

Delivered Tank Truck. Sources include CMR, industry sources and distributor price lists. Survey made in November 1984 in Chicago, Cincinnati, Detroit and Cleveland.

Free sample. Dial (800) 527-2601 for a free sample or help in reformulating with DuPont DBE solvent.

Use DBE as the slow solvent with PM Acetate and reduce replacement costs

	EPOXY ENAMEL		HIGH SOLIDS POLYESTER		ACRYLIC LACQUER	
SOLVENT	OLD FORMULA (WT.%)	REPLACEMENT* FORMULA (WT/%)	OLD FORMULA (WT.%)	REPLACEMENT* FORMULA (WT.%)	OLD FORMULA (WT.%)	REPLACEMENT* FORMULA (WT.%)
Du PONT DBE	–	15	–	12	–	5
PM ACETATE	–	25	–	38	–	8
MEK	–	–	47	50	26	26
N-BUTANOL	–	10	–	–	–	–
TOLUENE	–	–	–	–	61	61
XYLENE	50	50	–	–	–	–
EE ACETATE	50	–	53	–	13	–
$/GALLON	2.65	2.51	3.28	3.22	1.95	1.95
LB./GALLON	7.58	7.54	7.34	7.37	7.13	7.17

Cost of solvent, cents/pound: Du Pont DBE 41.5; PM Acetate 53.0; MEK 36.0; N-Butanol 36.0; Toluene 17.9; Xylene 17.3; EE Acetate 50.0

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CALL FOR PAPERS

60th Colloid and Surface Symposium
Georgia Institute of Technology, Atlanta, GA
June 15-18, 1986

Focusing on "Gas-Solid Interactions" and "Liquid-Liquid, Liquid-Solid Interactions," the Georgia Institute of Technology will offer its 60th Colloid and Surface Science Symposium on June 15-18, 1986.

Symposia: Gas-Solid Interactions

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K.S.W. Sing (Plenary Lecturer)
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England

CATALYSIS: METAL SUPPORT INTERACTIONS
M.A. Vannice
Dept. of Chemical Engineering
The Pennsylvania State University
133 Fenske Laboratory
University Park, PA 16802

CATALYSIS: NOVEL CATALYSTS
Glenn Schrader
Dept. of Chemical Engineering
Iowa State University
Ames, IA 50011

FINE PARTICLES: FLAME SYNTHESIS
George Mulholland
Center for Fire Research
U.S. Dept. of Commerce
National Bureau of Standards
Washington, D.C. 20234

CHEMICAL VAPOR DEPOSITION
K.F. Jensen
Chemical Engineering and Materials Science
University of Minnesota
421 Washington Ave. S.E.
Minneapolis, MN 55455

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Exxon Research and Engineering
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SOLID SURFACE MODIFICATION
R.F. Hochman
School of Materials Engineering
Georgia Institute of Technology
Atlanta, GA 30332-0100

ZEOLITES/MOLECULAR SIEVES
T.L. Thomas
Zeolite Research Program
Georgia Tech Research Institute
Georgia Institute of Technology
Atlanta, GA 30332

Symposia: Liquid-Liquid, Liquid-Solid Interactions

EMULSION POLYMERS AND POLYMERIZATION: FUNDAMENTAL STUDIES AND APPLICATIONS

J.W. Vanderhoff
Emulsion Polymer Institute
Sinclair Laboratory #7
Lehigh University
Bethlehem, PA 18015

Interested persons may submit papers in the following topics to the appropriate chairmen by December 1, 1985. The deadline for abstracts on standard ACS abstract forms is February 1, 1986.

SURFACE PHENOMENA IN ENHANCED OIL RECOVERY
D.T. Wasan
Dept. of Chemical Engineering
Illinois Institute of Technology
Chicago, IL 60616

MICROEMULSIONS
D.O. Shah
Dept. of Chemical Engineering
University of Florida
Gainesville, FL 32611

STRUCTURE AND PROPERTIES OF COLLOIDAL DISPERSIONS
Raj Rajagopalan
Dept. of Chemical Engineering
Rensselaer Polytechnic Institute
Troy, NJ 12180-3590

SOLID-LIQUID SEPARATION PROCESSES FOR ULTRAFINES SUSPENSIONS
Eric Clayfield
Georgia Mining and Mineral
Resources Institute
Georgia Institute of Technology
Atlanta, GA 30332

MICELLAR SOLUTIONS
A.D. King
Dept. of Chemistry
University of Georgia
Athens, GA 30602

NONTOXIC CONTROL OF INTERFACIAL FOULING
R.E. Baier
Health Care Instruments
and Devices Institute
Parker Hall
State University of New York
Buffalo, NY 14214

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Empire State Paper Research Institute
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and Forestry
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BIOLOGICAL SURFACES
Steve Hilbert
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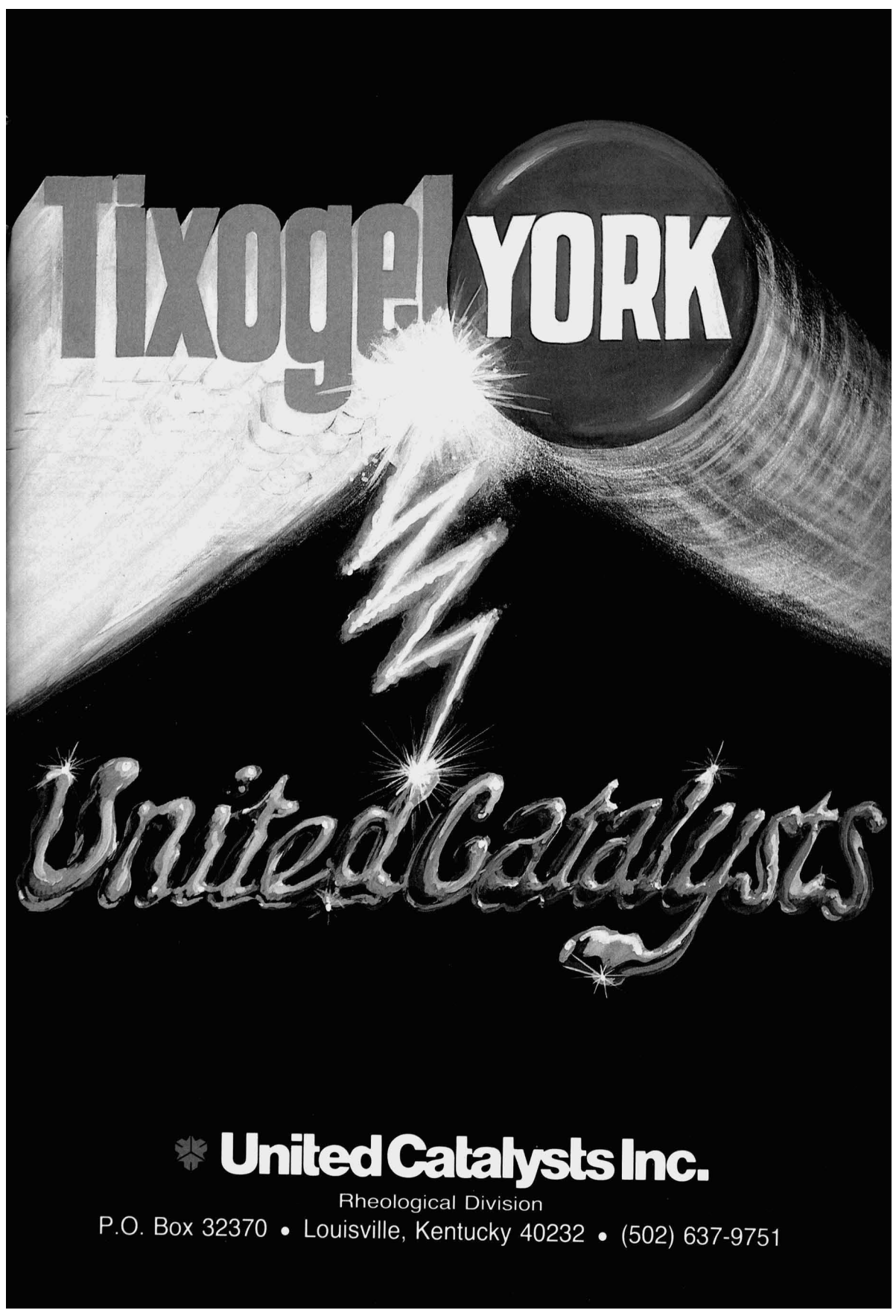
GENERAL PAPERS
T.R. Rybolt
Dept. of Chemistry
University of Tennessee/Chattanooga
Chattanooga, TN 37403

For additional information, contact the Symposium Chairman, Michael J. Matteson, School of Chemical Engineering, Georgia Institute of Technology, Atlanta, GA 30332.



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Constituent Society Meetings and Secretaries

BALTIMORE (Third Thursday—Martin's Eudowood, Towson, MD). ED COUNTRYMAN, Bruning Paint Co., 601 S. Haven St., Baltimore, MD 21224. Virginia Section—Fourth Wednesday, Ramada Inn-East, Williamsburg, VA.

BIRMINGHAM (First Thursday—Strathallan Hotel, Birmingham, England). D.M. HEATH, Holden Surface Ctgs. Ltd., Bordesley Green Rd., Birmingham B9 4TQ England

CHICAGO (First Monday—meeting sites vary). RAYMOND CZICZO, Reliance Universal, Inc., 1915 Industrial Ave., Zion, IL 60099.

CDIC (Second Monday—Sept., Jan., Apr., June in Columbus; Oct., Dec., Mar., May in Cincinnati; and Nov., Feb. in Dayton). SAMUEL KRATZER, D&L Paint Co., 215 Brownsville Ave., Liberty, IN 47343.

CLEVELAND (Third Tuesday—meeting sites vary). RICHARD ELEY, Glidden Coatings & Resins, Div. of SCM Corp., D.P. Joyce Research Center, P.O. Box 8827, Strongsville, OH 44136.

DALLAS (Thursday following second Wednesday—Executive Inn, near Lovefield Airport). FREDERICK T. BEARD, Glidden Coating & Resins, Div. of SCM Corp., 1900 North Josey Ln., Carrolton, TX 75006.

DETROIT (Fourth Tuesday—meeting sites vary). PETER KNIGHT, Dow Chemical Corp., 2020 Dow Center, Midland, MI 48640.

GOLDEN GATE (Monday before third Wednesday—Alternate between Sabella's Restaurant on Fisherman's Wharf and Francesco's in Oakland, CA).

HOUSTON (Second Wednesday—Sonny Look's, Houston, TX). JAMES W. JUDLIN, Devoe & Raynolds, Inc., 676 Kerbyville Rd., Houston, TX 77003.

KANSAS CITY (Second Thursday—Cascone's Restaurant, Kansas City, MO). STEVEN JOHNSON, Cook Paint & Varnish Co., P.O. Box 389, Kansas City, MO 64141.

LOS ANGELES (Second Wednesday—Steven's Steak House, Commerce, CA). MELINDA RUTLEDGE, Allo Chemical Co., P.O. Box 443, Ontario, CA 91761.

LOUISVILLE (Third Wednesday—Breckinridge Inn, Louisville, KY). LARRY F. PITCHFORD, Reynolds Metals Co., P.O. Box 3530, Plant III, Louisville, KY 40232.

MEXICO (Fourth Thursday—meeting sites vary).

MONTREAL (First Wednesday—Bill Wong's Restaurant).

NEW ENGLAND (Third Thursday—LeChateau Restaurant, Waltham, MA). GAIL POLLANO, Polyvinyl Chemical Industries, Inc., 730 Main St., Wilmington, MA 01887.

NEW YORK (Second Tuesday—Landmark II, East Rutherford, NJ). JOHN W. BURLAGE, Pacific Anchor Chemical, 14 Ridgedale Ave., Cedar Knolls, NJ 07927.

NORTHWESTERN (Tuesday after first Monday—Jax Cafe, Minneapolis, MN). JOAN B. LAMBERG, Horton-Earl Co., 750 S. Plaza Dr., St. Paul, MN 55120.

PACIFIC NORTHWEST (Portland Section—Tuesday following second Wednesday; Seattle Section—the day after Portland; British Columbia Section—the day after Seattle). YVON POITRAS, General Paint Co., 950 Raymur Ave., Vancouver, B.C., V6A 3L5, Canada.

PHILADELPHIA (Second Thursday—Dugan's Restaurant, Philadelphia, PA). THOMAS L. PETA, J.C. Osborne Chemicals, Inc., P.O. Box 1310, Merchantville, NJ 08109.

PIEDMONT (Third Wednesday—Howard Johnson's, Brentwood Exit of I-85, High Point, NC). CHARLES HOWARD, Desoto, Inc., P.O. Box 22105, Greensboro, NC 27420.

PITTSBURGH (First Monday—meeting sites vary). MARK TROUTMAN, Bradley Paint Co., 608 W. Crawford St., Connellsville, PA 15425.

ROCKY MOUNTAIN (Monday following first Wednesday—Bernard's, Arvada, CO). MARCY S. BAUGH, Hutson Industries, 60 Tejon St., Denver, CO 80223.

ST. LOUIS (Third Tuesday—Engineers Club). JAMES N. MCDERBY, F.R. Hall & Co., 6300 Bartmer Ind. Dr., St. Louis, MO 63130.

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; Miami Section—Tuesday prior to Central Florida Section). C. LEWIS DAVIS, Ambrosia International, 802 Black Duck Dr., Port Orange, FL 32019.

TORONTO (Second Monday—Cambridge Motor Hotel). HANS WITTMAN, BASF Canada Ltd., 10 Constellation Ct., Rexdale, Ont., Canada M9W 1K1.

WESTERN NEW YORK (Third Tuesday—meeting sites vary). JEAN L. LUCK, Pratt & Lambert Inc., Powder Coatings Div., P.O. Box 22, Buffalo, NY 14240.

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JANI, SAURABH—Frazee Industries, San Diego, CA.

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SERDAS	anti-foaming agents, anti-floating agents
SERVOCIDE	fungicides and bactericides
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People

Richard W. Scott, Staff Scientist at Sherwin-Williams Co., Chicago, IL, is a recipient of the 1985 ASTM Award of Merit. The award, and accompanying honorary title of Fellow of the Society, recognize Mr. Scott's outstanding leadership as Chairman of the Analytical Subcommittee of Committee D-1, directing the work of 24 active task groups and for promoting the use of ASTM standards by the U.S. Environmental Protection Agency. Mr. Scott is a member of the Chicago Society.

The 1985 ASTM Award of Merit was also presented to **Hal D. Swafford**, Technical Manager of Analytical Services, Glidden Coatings & Resins, Div. of SCM Corp., Strongsville, OH. Mr. Swafford was honored for his exceptional contributions to Committee D-1 in the development of new analytical procedures for the determination of trace heavy metals in paints and for the development of numerous other standards for coatings analysis. Mr. Swafford is a member of the Cleveland Society.

Abner Brenner, retired from the U.S. National Bureau of Standards, is the 1985 recipient of the Midgley Medal given annually by the Detroit Section of the American Chemical Society. Dr. Brenner is recognized for outstanding contributions to chemistry related to the automotive industries, particularly exemplified by his pioneering work on electroless plating.

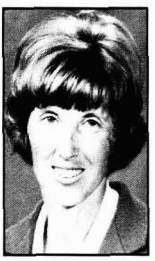
Michael R. Steele has been promoted to Western Regional Sales manager for Vedoc® Powder Coatings, Coatings Div., Ferro Corp., Cleveland, OH. Based at Ferro's Los Angeles facility, Mr. Steele directs sales development, technical support, and distributor sales for a 12-state area.

International Paint/Powder Coatings, a division of International Paint, London, England, has appointed **Al Alfonso** to the position of Technical Representative. Mr. Alfonso will be responsible for product sales and operations in California, Nevada, and Arizona.

A newly created position of Business Manager of Coatings has been established at Rohm and Haas Co., Philadelphia, PA, in order to enhance the firm's responsiveness to the needs of the markets served. **Thomas H. Haag**, formerly Group Market Manager—Trade Sales Coatings, has been appointed to the new post. His duties include coordinating and directing the manufacturing, marketing, research, communication, and financial functions of the firm.

R.W. Scott

H.D. Swafford



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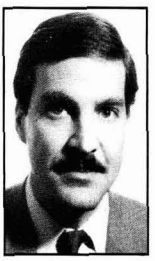

R.C. Ruda

Carol Niemi, a Research Leader with Dow Chemical Co., Midland, MI, has been elected to serve on the national board of the Chemical Coaters Association. As a national director, she will serve on the speakers selection committee for the association's annual seminar. Ms. Niemi is also a member of the Detroit Society for Coatings Technology.

Paul R. Yingst has been named Manager of Manufacturing and Technology for Ball Chemical Co., Glenshaw, PA. In this new position, he will have responsibility for all manufacturing activities, as well as research and development.

Austin A. Wolfrey, Senior Materials Engineer at Raytheon Co., Sudbury, MA, has been named Group Leader of Finishes and Plastics. A member of the New England Society, Mr. Wolfrey brings 11 years of experience in the coatings, plastics, and adhesives industries to the position.

Stanley L. Alekman has been promoted from Director to Vice-President, Research and Development, of Nuodex Inc., Piscataway, NJ. He is responsible for the company's technology, product and process development, technical services, and commercial development.

Michael A. Wood has joined The Valspar Corporation, Minneapolis, MN, as a Sales Service Coordinator. Mr. Wood will be located in Rockford, IL.

Buckman Laboratories, Inc., Memphis, TN, has appointed **M.L. Pulido** to Vice-President of International Marketing. Dr. Pulido joined Buckman as an Agricultural Specialist in 1967. He will oversee all marketing efforts in Asia, Latin America, and the Pacific.

Borden Chemical, a Div. of Borden, Inc., Columbus, OH, has promoted **Robert C. Ruda** to Midwest Regional Sales Manager for the polymer emulsion group of the Thermoplastics Div. A member of the Chicago and Louisville Societies, he will be based at the division's regional offices in Northbrook, IL.

Borg-Warner Chemicals, Parkersburg, WV, has promoted two employees to General Manager. **Thomas W. Beal** becomes General Manager for Surfactant Products and **Edward W. Lovely** moves to General Manager of Polymer Additives. Formerly, Mr. Beal served as General Manager for the firm's Sparmar plant. Mr. Lovely previously served as Marketing Manager and Commercial Development Manager for the company's chemical specialties.

Ian Pike, of South Australia, was installed in July as the 1985-87 President of the Oil and Colour Chemists' Association Australia. Mr. Pike has been associated with the coatings industry for more than 35 years and recently retired as the State Manager of Berger Paints (Australia) Pty. Ltd., South Australia. The 27th Convention of OCCAA will be held in Hunter Valley, South Australia, July 4-7.

Ronald S. Bauer, of Shell Development Co., Houston, TX, is the new Chairman of the Division of Polymeric Materials Science and Engineering, American Chemical Society. Other officers are: Chairman-Elect—**John H. Lupinski**, of General Electric Co., Schenectady, NY; Vice-Chairman—**Ray Dickie**, of Ford Motor Co., Dearborn, MI; Secretary—**Theodore Davidson**, of Ethicon, Inc., Somerville, NJ; and Treasurer—**Theodore Provder**, of Glidden Coatings and Resins, Strongsville, OH. The Division will meet next in New York, April 13-18. The Fall 1986 meeting (September 7-12) will be in Anaheim, CA.

Sherwin-Williams Co., Chicago, IL, has appointed **Jack Andrews** to the position of Sales Representative in the coil business unit. Mr. Andrews will be responsible for account maintenance and business development in the coil coating markets.

Art Gilliam has advanced to Market/Product manager, forest products business unit, for the Chemical Coatings Div. of Sherwin-Williams. Mr. Gilliam joined the division in 1957 and most recently served as Technical Manager for the forest products business unit.

Newton B. Martin has been promoted to Manager of Process Services at the Humko Chemical Div. of Witco Chemical Corp., New York, NY. Mr. Martin has been with Humko since 1968 and most recently served as Group Leader in Applications Development.

Humko Chemical has appointed **Bing-Lin Chen** to Manager of Basic Science in its Research and Development Dept. in Memphis, TN. Mr. Chen assumes responsibility for research and development in fatty-based specialty chemicals.

SCM Chemicals, a division of SCM Corp., has named **Peter C. Firing** President of its Baltimore-based pigments operation. Mr. Firing is Chairman of the Board of SCM Pigments Ltd. in the U.K. and SCM Chemicals Ltd. in Australia.

Carl D. Archer has been promoted to Central Regional Sales Manager, of SCM Pigments, SCM Corp. Mr. Archer will coordinate the sales and service functions for the central U.S.

In addition, **Linda F. Elliott** has accepted the position of Sales Representative for SCM Pigments. She will be responsible for sales and service support for the Colorado, New Mexico, Oklahoma, Arkansas, and Texas areas.

William Zinsser & Co., Inc., Somerset, NJ, has named **James Ochs** to the post of Technical Director. A member of the New York Society, Mr. Ochs most recently served as a Paint Chemist for Benjamin Moore & Co.

Succeeding **Joachim Zirner** in the position of Director of Polyurethane and Coatings Manufacturing at Mobay Chemical Corp., Pittsburgh, PA, is **Helmut Porkert**. Mr. Porkert will direct major components of the production operations at Mobay's Baytown, TX, and New Martinsville, WV, plants.

Avecor, Inc., San Fernando, CA, has named **Richard Woolsey** its first Regional Sales Manager. In this new position, Mr. Woolsey will head the sales coverage in an 11-state territory as well as supervise the efforts of his sales staff in the territory.

Larry M. Wlodar has advanced to Supervisor of Process Engineering at Akzo Coating America, Inc., Troy, MI. Mr. Wlodar will supervise and coordinate the activities of process engineers at the Pontiac, Detroit, and Atlanta facilities.

Akzo Coatings America has promoted **Gregory C. Dildilian** to Marketing Services Director. In this position, he will be responsible for marketing, advertising, and public relations for the corporation.

George O. Trabue, Jr. has been elected a Senior Vice-President and **Thomas G. Fudge** has been elected a Vice-President, of Eastman Chemical Products, Inc., New York, NY. Prior to these promotions, Mr. Trabue served as Vice-President overseeing chemical marketing and Mr. Fudge was Director of Marketing of the Chemicals Div. of Eastman.

In addition, Eastman Chemical has named two Senior Marketing Representatives with the Chemicals Field Marketing Div. **Peter Andriacchi** and **Lumas C. Thomas, Jr.**, both of whom served as Marketing Representatives in the division, will assume new posts.

Literature

Surface Pretreatment Polymer

Recently released information details a new advance in the treatment of rusted steel with a water-dilutable chelating polymer. This non-toxic product reacts chemically with rust and, after drying, forms an hydrophobic protective conversion coat which passivates the rust and offers a solid basis for adhesion to all types of subsequent paint systems. For copies, contact Aceto Chemical Co., Inc., 126-02 Northern Blvd., Flushing, NY 11368.

Air Flow Amplifiers

A complete line of air flow amplifiers is featured in a product bulletin. Applications include industrial ventilating, conveying of materials, and energy efficient parts ejection, drying, or cooling. Contact Michael L. York, Vortech Corp., 10125 Carver Rd., Cincinnati, OH 45242.

Color Computer

A detailed description of the components of a new color computer has been published. The system allows for the display and printout of any of four 3-coordinate standard color systems plus white and white/yellow standards. For full details on the Tricolor LFM-3, contact Yogi Shah, Sitco, 318 Cleveland Ave., Highland Park, NJ 08904.

Coil Coatings

Five coil coatings product groups are featured in a new full-color booklet. Anticorrosion properties, manufacturing benefits, and applications are among the topics discussed. Copies and spec sheets are available from Akzo Coatings America, Inc., Marketing Services Dept., 650 Stephenson Hwy., Troy, MI 48083.

Chemicals Sourcebook

The 1985 edition of the "Suspect Chemicals Sourcebook" has been issued. The guide contains chemical substances regulated or under investigation by federal and state governments and key international organizations. Special coverage of the OSHA Chemical Hazard Communication Standard's provisions is also included. Order inquiries should be addressed to H.G. Miller, International Regulation Organization, 5724 N. Pulaski Rd., Chicago, IL 60646.

Maintenance Coating

The performance benefits of an industrial maintenance coating are outlined in a recently published report. This coating is a self-priming topcoat which provides extended-term protection and is offered in a broad spectrum of rapid response colors. For a copy of the report, "High Solids Systems for Industrial Maintenance and Repair: Technical and Economic Analysis," write to Ameron Protective Coatings Div., 201 N. Berry St., Brea, CA 92621.

KELATE MR6

A new advance in the chemical conversion of rust!

KELATE MR6 A water reducible chelating polymer designed for field application to rusted steel which has been hand or power cleaned or sandblasted.

KELATE MR6 Neutralizes the corrosion process reacts quickly with the rust and transforms iron oxides into a stable and insoluble black metallo organic complex which is ready for painting after reaction. Reaction time is three hours and it is not necessary to wash the surface with water after reaction.

KELATE MR6 Available in concentrated form to the surface coating industry.

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ACETO CHEMICAL CO., INC.

ACETO

Mr V.G. MIATA
126-02 Northern Boulevard,
Flushing, New York 11368

Phone: 718.898.2300

Polyester Resin

A new hydroxyl terminated polyester resin has been introduced in literature. Reported to exhibit outstanding physical properties, possess low color, and produce polyester/urethane powder coatings with smoother finishes, the resin was developed for curing with blocked isocyanates. For copies of the technical data sheet on Arakote 3109, write: Resins Dept., Ciba-Geigy Corp., Three Skyline Dr., Hawthorne, NY 10532.

Self-Cleaning Filters

A line of small automatic cleaning filter models has been improved with the addition of a motor and is described in current literature. The motor automates the self-cleaning feature giving continuous cleaning and eliminates the need for manual operations. Performance specifications on the upgraded filters can be obtained from Michael Bannan, AMF Cuno General Filter Products, 400 Research Pkwy., Meriden, CT 06450.

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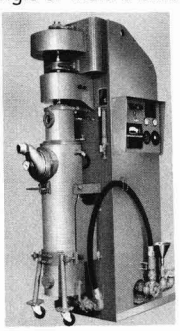

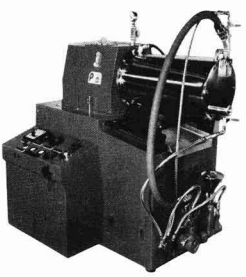

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Filling Machine

Product literature detailing a new filling machine is now available. The new model offers a three foot conveyor to one or both sides, permitting the operator to store many empty and filled containers while another container is being filled. The conveyors fold up when not in use. Inquiries should be forwarded to Beltron Corp., P.O. Box 893, Red Bank, NJ 07701.

Bead Mill

An improved model explosion-proof bead mill is introduced in literature. The new model contains a variable high speed grinding shaft plus a variable speed peristaltic feed pump and can process water-based or solvent-based paint and ink mill bases. For performance specifications, contact Premier Mill Corp., 220 East 23 St., New York, NY 10010.

Specialty Solvent

An eight-page guide provides detailed product information on diethylene glycol monohexyl ether, a clear, colorless, high-boiling solvent of high purity. The solvent is reported to show potential as a coalescing solvent in water-borne coatings. For copies of F-60041A, write to Union Carbide Corp., Specialty Chemicals Div., Dept. K3442, 39 Old Ridgebury Rd., Danbury, CT 06817.

Infrared Library

An infrared library of acrylic polymers and specific modifiers likely to be present in finished acrylic-based products is available on floppy disks or in hard copy. For a brochure outlining the benefits of the materials information, contact Hebe B. Greizerstein, EB Associates Laboratories, Inc., 2211 Main St., Buffalo, NY 14214.

Gloss Meter

The applications of a new reflectance and gloss meter are outlined in a pamphlet. The compact and portable equipment can be used throughout the plant or lab or directly on the production line. Complete details are offered by Testing Machines Inc., 400 Bayview Ave., Amityville, NY 11701.

Anionic Dispersant

Product data on a new anionic pigment dispersant for emulsion paints has been published. The efficient and cost saving pigment is synthesized from readily available raw materials. Contact Tambour Ltd., Industrial Zone, Akko, P.O. Box 2238, Akko 24 101, Israel, to obtain copies on "Dispers-One" dispersant.

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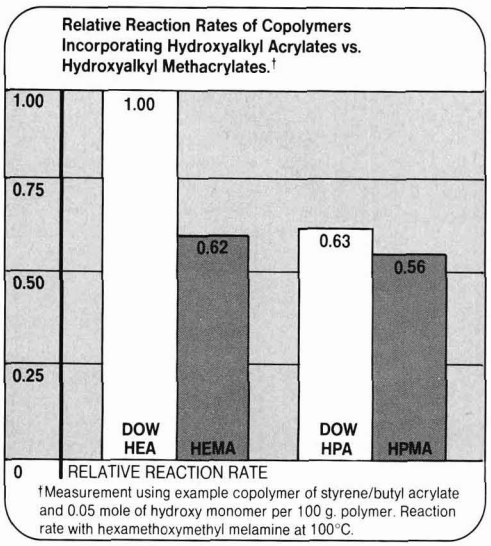
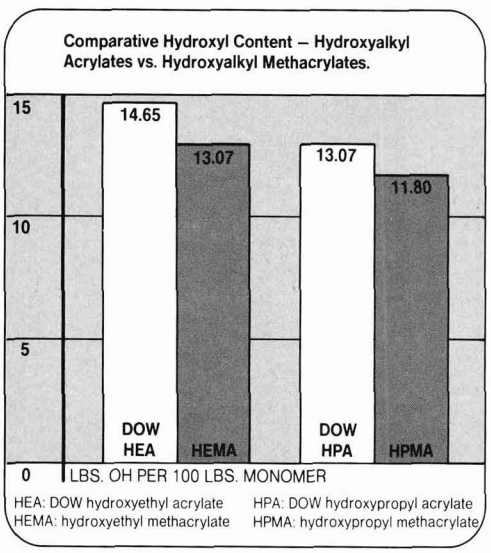
Pound for pound, more crosslinking sites than methacrylates.





DOW hydroxyethyl acrylate (HEA) and hydroxypropyl acrylate (HPA) functional vinyl monomers offer substantially higher OH reactivity than hydroxyalkyl methacrylates. Plus, their lower molecular weights translate into a 12% reduction in functional monomer requirements and lower material costs.

Exceptional versatility for resin polymerization.

DOW HEA and HPA copolymerize with a variety of other vinyl monomers in aqueous and non-aqueous systems. The resulting pendant hydroxyl functionality can be used in crosslinking reactions with polyisocyanates, polyanhydrides and melamine or epoxy resins.

Faster crosslinking and lower temperature requirements save time and money.

When HEA and HPA hydroxyl functionality is added to polymers and copolymers, higher reactivity in crosslinking reactions results. This permits faster curing cycles and accelerated line speeds for increased productivity.

With DOW HEA and HPA, polymers can be converted into radiation curable resins when vinyl unsaturation is introduced through reactions with backbone functional groups. And high reactivity at lower temperatures makes HEA and HPA useful in coatings for plastics and other temperature-sensitive substrates.

Favorable end-use properties.

• Outstanding mechanical and endurance properties in coatings enamels. • Solubility in water and polar solvents. • Stabilization of aqueous polymer dispersions. • Improved adhesion to metals, wood, textiles, paper and other substrates.

Put DOW HEA and HPA to work for you.

HEA and HPA are chemical intermediates useful in the synthesis of coatings resins, latexes, adhesives, radiation-cure oligomers, and other resins. These monomers are not recommended for use as reactive diluents in radiation coatings or as simple additives to other formulations, unless appropriate measures are taken to control vapors and minimize personnel exposure within recommended guidelines.
For complete information, write: The Dow Chemical Company, Coatings and Resins Department, Inquiry Services, 1703 S. Saginaw Road, Midland, Michigan 48674.

Specialty MONOMERS

DOW*

*Trademark of The Dow Chemical Company

Glossmeter

A new glossmeter is the subject of recently released literature. The new model can be equipped with three individual sensing heads, or one multi-angle sensing head. Details on the new model as well as its accessory parts can be obtained from Paul N. Gardner Co., Inc., P.O. Box 10688, Pompano Beach, FL 33061.

Torsion Pendulum

An automatic torsion pendulum for measuring the shear modulus and mechanical damping of plastics, elastomers, and composites has been introduced in literature. To obtain copies, contact Myrenne Instruments, P.O. Box 1383, Fremont, CA 94538.

Air Sampler

The uses and benefits of a new 20 liter/minute high flowrate air sampler are listed in a product guide. The sampler is designed to measure asbestos and very low concentration pollutants, tracer gases, aerosols, particulates and detectable gases. For additional information write to: Bart Catalane, Sales Manager, Gilian Instrument Corp., 8 Dawes Hwy., Wayne, NJ 07470.

Urethane Oligomer

Marked improvements in the gloss retention of coatings for no-wax floor covering is achieved through a recently developed urethane oligomer. The new Sartomer® 9505 aliphatic urethane oligomer reportedly imparts excellent abrasion, scratch, and stain resistance and offers improved heat aging properties as observed in comparative product testing. For copies of test data details, contact Arco Specialty Chemicals, West Chester Plaza, West Chester, PA 19382.

Alcohols

A 15-page booklet describes a wide variety of alcohols used in coatings formulations. Included in the publication are comprehensive tables of physical properties, graphs depicting flash points and freezing points of alcohol/water mixtures, and data on a broad range of constant boiling mixtures composed of alcohols and other solvents. Copies of "UCAR Alcohols for Coatings Applications," designated F-48588, are available from Union Carbide Corp., Solvents and Coatings Materials Div., Dept. K3442, 39 Old Ridgebury Rd., Danbury, CT 06817.

Color Matching System

A four-color pamphlet highlights a recently developed color matching system. Three major components make up the system: a dual-beam spectrophotometer; a desktop personal computer; and a software package tailored to the needs of the specific industries. For copies of the brochure, contact Milton Roy Co., Analytical Products Div., Dept. 6682, 820 Linden Ave., Rochester, NY 14625.

Recovery System

Literature has been published outlining the capabilities of three new and improved blasting and recovery systems. The new models are lighter in weight, allowing for greater ease of use and flexibility. Write to the Inventive Machine Corp., P.O. Box 369, Bolivar, OH 44612, for product specifications on the new Blast N' Vac models.

Packaging Products

Metal and plastic packaging products are covered in a recently published four-color, 12-page booklet. Write to Brockway Standard, Inc., 8613 Roswell Rd., Bldg. 2, Suite 100, Atlanta, GA 30338, for copies.

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GRINDING BEADS

- Faster Production of Product
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SEPR ER 120 A Grinding Beads are produced by electric fusion of oxides to form a closely-bonded internal structure of a crystalline zirconia phase within an amorphous silica phase. The combination imparts high impact resistance, elasticity and a satin smooth surface to a dense material. Compared to a same-size standard grinding bead, ER 120 A offers greater mass and achieves higher kinetic energy. Compared to a same-mass bead, ER 120 A is smaller and offers a greater number of impacts. The end result: Faster processing; enhanced size reduction.

Additional benefits resulting from smooth fusion-produced zirconia-silica beads include a reduced wear rate on equipment, a lower fracture rate for these stronger beads and a highly improved cleaning ability. Furthermore, the number of passes (re-cycles) or residence time needed to achieve a given product size will be reduced.

For complete information including samples, call or write:

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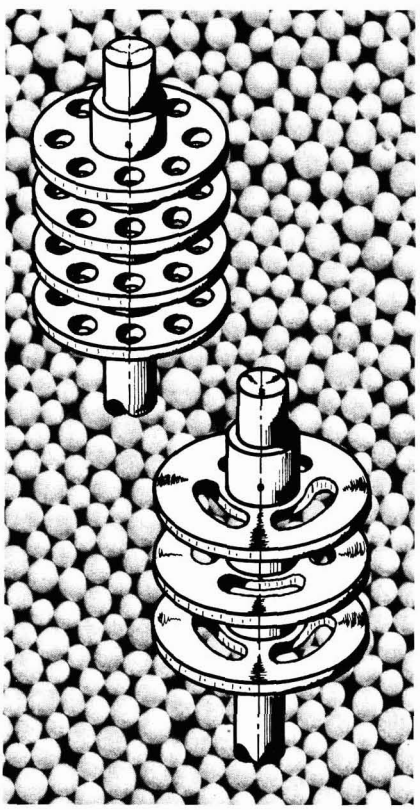


Color System

A recent technical bulletin details an advanced color control system designed to meet the needs of printers and printing related industries concerned with four-color process in publications, product packaging, catalogs, and commercial printing of all types. The system combines an optical sensor, mounted on a vertical movement stand for measurement ease, with an IBM PC/XT. For copies of the bulletin, write to HunterLab, Hunter Associates Laboratory, Inc., 11495 Sunset Hills Rd., Reston, VA 22090.

Testing Chamber

A large-capacity temperature/humidity chamber specially designed to meet proposed ASTM standards for SCAB testing is the subject of a new product bulletin. Capable of reproducing the required freezing, humidity, and heating cycles all in one unit, as well as eliminating the need to repeatedly transfer the test samples throughout the multi-week SCAB test procedure are some of the benefits of the unit. Contact Mr. E. Carroll, Hotpack Corp., 10940 Dutton Rd., Philadelphia, PA 19154, for complete details.

Spectrophotometer

A low cost, high performance color measurement system is discussed in a technical brochure. The system provides spectrophotometric or colorimetric data in the measurement of opaque, translucent, or transparent materials used in a variety of industries. For copies of the brochure, contact HunterLab, Hunter Associates Laboratory, Inc., 11495 Sunset Hills Rd., Reston, VA 22090.

Urethane Additives

An aliphatic amine tetrol which serves as a catalyst and crosslinker in urethane polymers has been added to a line of urethane additives. Applications include semi-flexible and flexible foams, urethane coatings, and urethane adhesives, sealants, and rigid foams. For further details, contact Performance Chemicals, Air Products and Chemicals, Inc., P.O. Box 538, Allentown, PA 18105.

Dispersant Guide

A recently published six-page guide reviews the use of dispersing agents to prevent settling of solids, agglomeration of gases, and formation of pastes or creams in liquid suspensions. Copies of the booklet "How to Handle Your Mixing Problems," are offered by W.R. Grace & Co., Organic Chemicals Div., Marketing Communications Dept., 55 Hayden Ave., Lexington, MA 02173.

Silicone Antifoams

Information on a variety of silicone antifoam compounds, emulsions, and fluids is detailed in a brochure. Data on the selection and use as well as helpful practices for the various products are included in the literature. For copies, contact Hodag Corp., 7247 N. Central Park Ave., Skokie, IL 60076.

Tackifying Resins

Literature is available on a recently introduced line of tackifying resins for pressure sensitive adhesives, hot-melt adhesives, protective coating vehicles, and printing inks. For detailed product specifications on the new resins, write to Product Inquiry, Hercules Incorporated, Hercules Plaza, Wilmington, DE 19894.

Thermometers

A 16-page booklet highlights a complete line of industrial and laboratory thermometers. Product descriptions, illustrations, special accessories, and a temperature conversion table are included in the publication. For copies, contact Paul N. Gardner Co., Inc., P.O. Box 10688, Pompano Beach, FL 33061.

MSDS Software

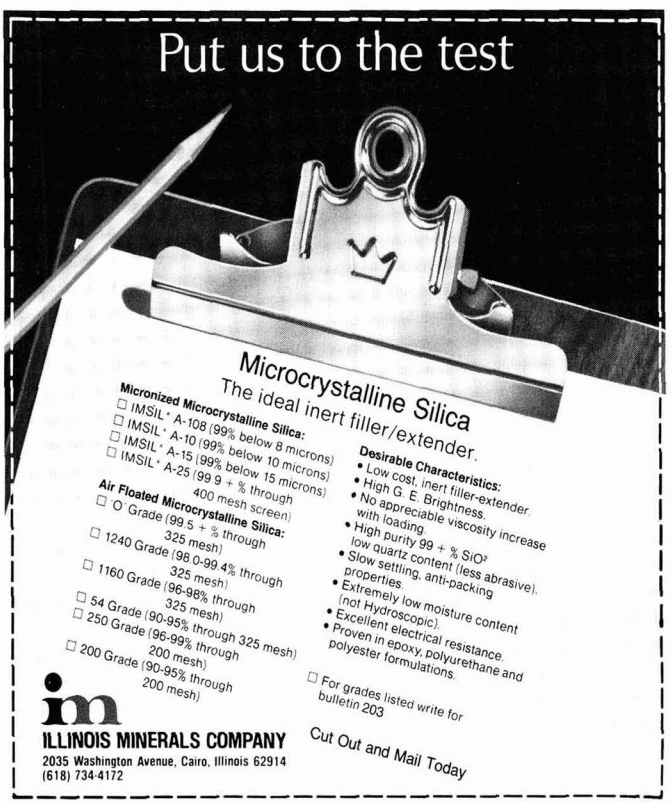
A new formula modeling system for IBM PCs is detailed in a four-color applications bulletin. The formula-based package automatically generates Material Safety Data Sheets and provides control of all laboratory calculations. For copies of the bulletin, write to Parallax Computer Corp., 3490 Route 1, Bldg. 19-1, Princeton, NJ 08540.

Meters and Sensors

A complete line of electronic measuring instruments is featured in a 16-page guide. Applications, product descriptions, and prices are included. For copies, contact Paul N. Gardner Co., Inc., P.O. Box 10688, Pompano Beach, FL 33061.

Emulsion

Application information on a recently introduced polyvinyl alcohol-stabilized vinyl acrylic emulsion is now available. The emulsion reportedly performs equally well in high quality trade sales paints and in high PVC contractor paints. Obtain further details from Reichhold Chemicals, Inc., Emulsion Polymer Div., P.O. Box Drawer K, Dover, DE 19903.



CORROSION PREVENTION BY PROTECTIVE COATINGS

Written by
Charles G. Munger

Published by
National Association
of Corrosion Engineers
Houston, TX
511 pages
$60.00: members;
$75.00: non-members

Reviewed by
John Nelson
Whirlpool Corp.
Benton Harbor, MI

This book concerns itself with corrosion prevention by protective coatings from the corrosion point of view. It has 19 chapters which can be grouped into four areas. The first area concerns the fundamentals of coating corrosion, of organic coating characteristics and formulations, and of corrosion resistant organic and zinc coatings. The second involves structural design, the substrate, surface preparation, and coating applications. The third area includes coating failures, repair, and maintenance and the fourth area covers safety, coating specifications, inspection, testing, and coating uses. Woven in between these areas are a chapter on coatings for concrete and a chapter on coatings for cathodic protection. The book is well written with many excellent illustrations, figures and tables. Mechanisms of corrosion and coating failure are given. Methods of writing coating specifications and of doing coating inspections are included. The examples range from the basic fundamentals to problems in plant coating applications.

The book is excellent and informative in what is covered, however, not all corrosion-preventive coatings are covered. The focus is on organic coating and zinc-containing coatings. Electroplated (except zinc), aluminized, anodized, and sputtered coatings are not included. Examples given in the book are oriented to industrial manufacturing facilities, power industry, transportation industries, and marine service.

This book would be of value to anyone wanting a better understanding of coatings and coating corrosion fundamentals. Anyone interested or working in the area of paint formulation to paint application would find this book an excellent reference. Anyone charged with the responsibility of maintenance of painted surfaces would find this book a jewel.

WEATHERING OF POLYMERS

Written by
Anthony Davis
and David Sims

Published by
Elsevier Applied Science
Publishers
London and New York
$52.50, 294 pages

Reviewed by
Ray A. Dickie
Ford Motor Co.
Dearborn, MI

This book provides an overview of work on the weathering of bulk plastics up to about 1981. Surface coatings appear to have been systematically excluded from consideration, and as a result the book will be of limited interest to coatings scientists.

The first half of the book provides a general survey of the environment (including a separate chapter on ultraviolet radiation), discussions of weathering trials and artificial weathering methods for plastics, an overview of characterization methods, and a discussion in general terms of photo-oxidation and stabilization. The balance of the book is devoted to specific classes of polymeric materials (engineering polymers, polyvinyl chloride, vinyl polymers, polyurethanes, rubbers, and composites). Given the breadth of the subject matter and the relatively modest size of the book (294 pp.), much of the discussion is necessarily not very thorough. Extensive references to the literature are given.

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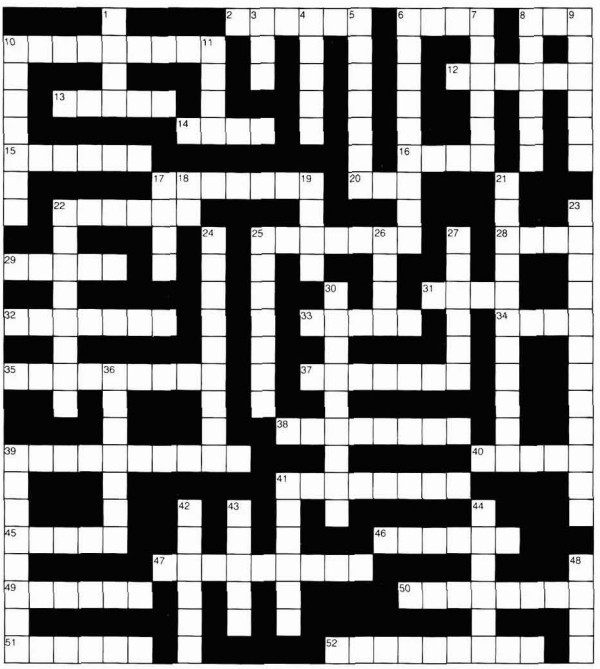
by Earl Hill



Solution to be published in November Issue

No. 8

ACROSS

2. Plastics hardness measurement
6. Critical volume (Abr.)
8. Flash Point measurement (Abr.)
10. Gaseous cation, mainly pigments (Chem.)
12. Gutta-percha substitute (gum)
13. Int. Chemistry organization (Abr.)
14. C_____ board
15. China clay
16. A bad impression
17. Metal. Goes with 10 Across
20. Total reflectance measurement (Abr.)
22. Phenolic nut constituent
25. To grow in the lab (Biol.)
28. Unique lacquer type
29. Deficient in clarity
31. Type of mill
32. How one makes things
33. Shine (syn.)
34. British association (Abr.)
35. Reaction vessel (Chem.)
37. Light diffusion
38. Liquid aldehyde (Chem.)
39. Lemongrass oil
40. Used in wood finishing
41. C_____ ratio
45. Lake pigment intermediate (red)
46. Water color pigment, usually brown
47. Holds things & stuff
49. Element used in lab testing (Anal.)
50. B_____ oil; semidrying (uncommon)
51. Originally known for selling driers
52. Soluble cellulose nitrate

DOWN

1. Metallic salt (Chem.)
3. Color value (Abr.)
4. C_____ing (film defect)
5. Not solid (Pl.)
6. Assemblage of components
7. Putty mixer
8. Pigment color
9. Stick pigment
10. Ripple (not the wine)
11. Common alcohol (Chemist's abr.)
17. Quercus suber
18. Light metal symbol (Chem.)
19. To smallify
21. Critical company function
22. Reddish lead mineral
23. Fish oil acid
24. Two phenols
25. Artistic technique
26. Handy at company picnic (food)
27. Applies paint
30. Alkyd ingredient
36. Natural anti-oxidant
39. All together now
41. Protective cap (Arch.)
42. Film characteristic
43. A kind of concrete
44. Cresylic acid component
48. E _____ grain

Coming Events

FEDERATION MEETINGS

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

(Oct. 29-30)—Meeting of Technical Advisory Committee with Society Technical Committee Chairmen. Madison, WI.

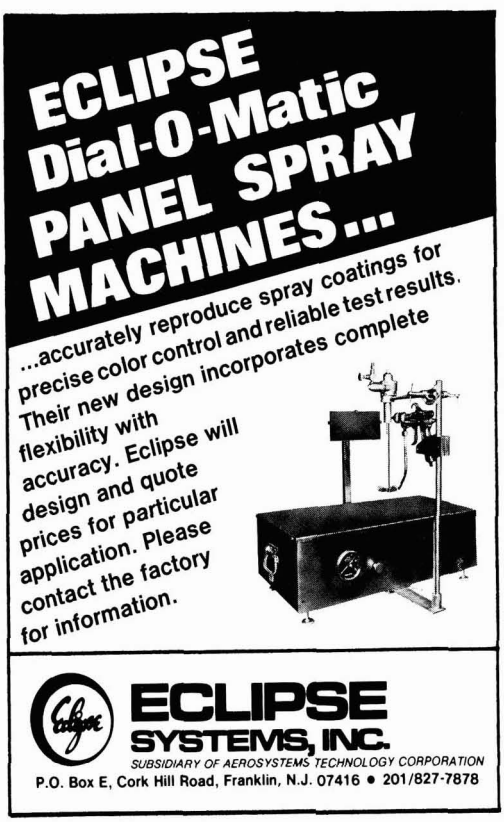
1986

(May 13-16)—Federation "Spring Week." Seminar on 13th and 14th; Society Officers on 15th; and Board of Directors on 16th. Sheraton Station Square, Pittsburgh, PA.

(Nov. 5-7)—64th Annual Meeting and 51st Paint Industries' Show. World Congress Center, Atlanta, GA.

SPECIAL SOCIETY MEETINGS

(Nov. 5-6)—Symco '85. 15th Annual Chicago Society Coatings Symposium, "The Right Stuff." Knickers Restaurant, Des Plaines, IL. (Lonnie U. Haynes, Chairman, Hercules, Incorporated, 300 E. Schuman Blvd., One Energy Center, Suite 260, Naperville, IL 60540).



1986

(Apr. 2-5)—Southern Society. Annual Meeting. DeSoto Hilton Hotel, Savannah, GA. (Ronald R. Brown, Union Chemicals Div., P.O. Box 26845, Charlotte, NC 28213).

(Apr. 9-11)—Southwestern Paint Convention of Dallas and Houston Societies. Wyndham Hotel, near Houston Intercontinental Airport, Houston, TX. (Mike Winters, Ribelin Distributors, Inc., 7766 Blankenship, Houston, TX 77055).

(Apr. 29-May 1)—"Advances in Coatings Technology" Conference sponsored by the Cleveland Society for Coatings Technology. NASA, Lewis Research Center, Cleveland, OH. (Dr. Rosemary Loza, Standard Oil Co. (Ohio), 4440 Warrensville Center Rd., Cleveland, OH 44128).

(May 1-3)—Pacific Northwest Society, Annual Symposium. Marriott Hotel, Portland, OR. (Gerald A. McKnight, Rodda Paint Co., 6932 S.W. Macadam Ave., Portland, OR 97219).

(May 15-16)—New England Society. Symposium: "Launching the New Revolution—Compliance for the 21st Century." (Maureen Lein, Davidson Rubber Co., Industrial Park Dr., Dover, NH 03820).

(May 22)—Birmingham Club. Symposium: "Miracle '86." Strathallan Hotel, Birmingham, England. (David Heath, Holden Surface Coatings Ltd., Bordesley Green Rd., Bordesley Green, Birmingham B9 4TQ, England).

1987

(Feb. 23-25)—Western Coatings Societies' Symposium and Show, Monterey Convention Center, Monterey, CA. (Barry Adler, Royell, Inc., 1150 Hamilton Ct., Menlo Park, CA 94025).

OTHER ORGANIZATIONS

(Oct. 21-23)—Symposium on "New Chemistry for Product Diversification and Specialization in Coatings." Sponsored by Eastern Michigan University. Ann Arbor, MI. (Alan Green, Technology Services Center, Eastern Michigan University, 150 Sill Hall, Ypsilanti, MI 48197).

(Oct. 21-25)—"Physical Testing of Paints & Coatings" Short Course sponsored by the University of Missouri-Rolla, Rolla, MO. (Prof. James O. Stoffer, Dept. of Chemistry, University of Missouri-Rolla, Rolla, MO 65401).

(Oct. 27-30)—Fire Retardant Chemicals Association Fall Conference. Pinehurst Hotel, Pinehurst, NC. (James D. Innes, FRCA, 851 New Holland Ave., P.O. Box 3535, Lancaster, PA 17604).

(Nov. 4-5)—"Painting Processes: Industrial Paint Application Technology" Short Course sponsored by Kent State University, Kent, OH. (Carl J. Knauss, Kent State University, Chemistry Dept., Kent, OH 44242).

(Nov. 4-6)—"Estimating for Painting Contractors and Maintenance Engineers" Short Course sponsored by the University of Missouri-Rolla, Rolla, MO. (Prof. James O. Stoffer, Dept. of Chemistry, University of Missouri-Rolla, Rolla, MO 65401).

(Nov. 4-6)—National Paint and Coatings Association. 98th Annual Meeting. Hilton Hotel, New Orleans, LA. (Karen Bradley, NPCA, 1500 Rhode Island Ave. N.W., Washington, D.C. 20005).

(Nov. 6-8)—National Coil Coaters Association Fall Technical Meeting. Hyatt Regency Dearborn, Dearborn, MI. (Tony Carroll, NCCA, 1900 Arch St., Philadelphia, PA 19103).

(Nov. 13-15)—"Nuclear Power Coatings Inspection." Seminar sponsored by KTA-Tator, Inc., Pittsburgh, PA. (Bill Corbett, KTA-Tator, Inc., 115 Technology Dr., Pittsburgh, PA 15275).

(Nov. 15-17)—38th National Decorating Products Association Show. McCormick Place, Chicago, IL. (Lillian Smysor, NDPA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132).

(Nov. 18-19)—Seminar on "Fine Particles and Fillers—Overlooked Opportunities." Sheridan Park Conference Center, Mississauga, Ont., Canada. (H.G. McAdie, Ontario Research Foundation, Sheridan Park Research Community, Mississauga, Ont., Canada, L5K 1B3).

(Nov. 18-21)—"Introduction to Coatings Technology" Short Course sponsored by Kent State University, Kent, OH. (Carl J. Knauss, Kent State University, Chemistry Dept., Kent, OH 44242).

(Dec. 4-6)—Short Course on "Advanced Chemical Coatings for Technical Managers." Sponsored by Eastern Michigan University. Ann Arbor, MI. (Alan Green, Technology Services Center, Eastern Michigan University, 150 Sill Hall, Ypsilanti, MI 48197).

(Dec. 9-10)—Short Course on "Introduction to Paint Job Estimating." Sponsored by Eastern Michigan University. Ann Arbor, MI. (Alan Green, Technology Services Center, Eastern Michigan University, 150 Sill Hall, Ypsilanti, MI 48197).

(Dec. 11-13)—Short Course on "Update on Coatings for Painting Contractors." Sponsored by Eastern Michigan University. Ann Arbor, MI. (Alan Green, Technology Services Center, Eastern Michigan University, 150 Sill Hall, Ypsilanti, MI 48197).

1986

(Feb. 9-12)—"The Colors of History: Identification, Re-Creation, Preservation" Conference sponsored by Inter-Society Color Council. Colonial Williamsburg, VA. (T.G. Webber, 1722 Forest Hill Dr., Vienna, WV 26105).

(Feb. 24-27)—Steel Structures Painting Council Annual Meeting and Symposium. Peachtree Plaza Hotel, Atlanta, GA. (Harold W. Hower, SSPC, 4400 Fifth Ave., Pittsburgh, PA 15213).

(Mar. 4-6)—"Coatings Inspection of Chemical Plants." Seminar sponsored by KTA-Tator, Inc., Pittsburgh, PA. (Bill Corbett, KTA-Tator, Inc., 115 Technology Dr., Pittsburgh, PA 15275).

(Mar. 10-14)—"The Basic Composition of Coatings" Short Course sponsored by the University of Missouri-Rolla, Rolla, MO. (Prof. James O. Stoffer, Chemistry Dept., University of Missouri-Rolla, Rolla, MO 65401).

(Mar. 11-13)—Electrocoat/86. Sponsored by *Products Finishing* Magazine. Drawbridge Inn, Ft. Mitchell, KY. (Tom Robison, Products Finishing, 6600 Clough Pike, Cincinnati, OH 45244).

(Mar. 17-21)—Annual Meeting of Chemical Coaters Association. Georgia International Convention and Trade Center, Atlanta, GA. (CCA, P.O. Box 241, Wheaton, IL 60189).

(Mar. 26-28)—"Bridges and Highway Structures Coating Inspection." Seminar sponsored by KTA-Tator, Inc., Pittsburgh, PA. (Bill Corbett, KTA-Tator, Inc., 115 Technology Dr., Pittsburgh, PA 15275).

(Apr. 5-6)—Workshop on "Size-Exclusion Chromatography." Sponsored by ACS Div. of Polymeric Materials: Science and Engineering. New York, NY. (Theodore Provder, Glidden Coatings & Resins, Div. of SCM Corp., 16651 Sprague Rd., Strongsville, OH 44136).

(Apr. 6-11)—"Advances in Size-Exclusion Chromatography" Symposium. Sponsored by ACS Div. of Polymeric Materials: Science and Technology. New York, NY. (Theodore Provder, Glidden Coatings & Resins, Div. of SCM Corp., 16651 Sprague Rd., Strongsville, OH 44136).

(Apr. 7-11)—"Paint Formulation" Introductory Short Course sponsored by the University of Missouri-Rolla, Rolla, MO. (Prof. James O. Stoffer, Dept. of Chemistry, University of Missouri-Rolla, Rolla, MO 65401).

(Apr. 9-16)—"Surface Treatment '86" sponsored by the World Center for Industrial Technology. Hannover Fairgrounds, Hannover, West Germany. (Hannover Fairs USA Inc., P.O. Box 7066, 103 Carnegie Center, Princeton, NJ 08540).

(Apr. 14-15)—ASTM Symposium on "Testing of Metallic and Inorganic Coatings," Chicago, IL. (Teri Carroll, ASTM Standards Development Div., 1916 Race St., Philadelphia, PA 19103).

(May 5-9)—"Physical Testing of Paints and Coatings" Short Course sponsored by the University of Missouri-Rolla, Rolla, MO. (Prof. James O. Stoffer, Dept. of Chemistry, University of Missouri-Rolla, Rolla, MO 65401).

(May 13-15)—11th Annual Powder & Bulk Solids Conference/Exhibition. O'Hare Exposition Center, Rosemont, IL. (Patricia Dickinson, Show Manager, c/o Cahners Exposition Group, Cahners Plaza, 1350 E. Touhy Ave., P.O. Box 5060, Des Plaines, IL 60017).

(May 14-15)—Surfex '86. Oil and Colour Chemists' Association Exhibit. Harrogate, England. (OCCA, Priory House, 967 Harrow Rd., Wembley, Middlesex, England HAO 2SF).

(May 19-22)—"Basic Microcomputer Programs for Coatings" Short Course sponsored by the University of Missouri-Rolla, Rolla, MO. (Prof. James O. Stoffer, Dept. of Chemistry, University of Missouri-Rolla, Rolla, MO 65401).

(June 10-16)—CHINAPLAS 86. International Exhibition Centre, Beijing, China. (Kallman Associates, 5 Maple Court, Ridgewood, NJ 07450).

(June 15-18)—60th Colloid and Surface Symposium. Georgia Institute of Technology, Atlanta, GA. (Symposium Chairman, M.J. Matteson, School of Chemical Engineering, Georgia Institute of Technology, Atlanta, GA 30332).

(July 3-6)—Oil and Colour Chemists' Association Australia. 28th Annual Convention. The Estate, McLaren Vale, South Australia. (OCCAA, 6 Wilson Ave., Felixstow, South Australia 5090, Australia).

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WACKER-CHEMIE GMBH **18-19**

'Humbug' from Hillman

Chris George of Columbia Chemicals, Canada Ltd., is searching for an answer to the weighty problem that follows. Those of our readers who can take the time from busy schedules to solve his dilemma should write to him—not to me.

Chris' Problem

Three pigment salesmen who had neglected to book accommodations for the St. Louis Convention found themselves in a dilemma. It seems that all the rooms in St. Louis were taken except for one. After some deliberation, it was decided that they would share the room.

They were told that the rate was $30 for the night (*Where did they find that flea bag? Ed.*). Each paid ten dollars. A little while later, the clerk discovered that he should have given them the convention rate, which was $25 (*Breakfast included?*).

Finding it difficult to divide the $5 among the three, he decided to return $1 to each and pocket the $2 balance for himself. In effect, each salesman had now contributed $9 as his portion. That totaled $27 plus the $2 pocketed by the clerk came to $29. What happened to the last dollar?

Some folks save string, some save baseball cards and Roy Tasse has collected issues of Reichhold's "By Gum." Roy kindly sent me a copy published in December 1959 and one going all the way back to 1944. For me, they struck a poignant note of nostalgia, seeing names like Charlie O'Connor, Weinmann, Wampner, El Murray, Ed Raswyck and Sid Lauren. Sid, of course, is still stirring the paint pots but seeing his name in the 1959 issue reminded me how long some of us stubbornly hang in there.

For you, I have chosen some comments and bits of humor featured in those old issues (more in later columns). Here is a particular piece that delighted me which was included in an article written by Charlie O'Connor in 1939. Charlie, who later was Honorary Chairman of RCI, questioned, "How chemists got into the paint business anyway". He wrote—

Take the case of the paint and varnish company selling a complete line of architectural and industrial finishes. The owner is a former salesman or he inherited the business from Dad, who was a salesman and allergic to chemists. He employs a one-man chemical clinic, not because it will do any good, but to give his place a strong pungent smell of authority.

As the business grew, the sample-toters got into the habit of coming in off the road on Saturday and dumping a sample of some competitor's material on the chemist's desk and saying, "Give me something as good or better than that for 25¢ less a gallon or we'll lose the business of the Buncombe Bustle Corporation. All you need to do is to add more zinc oxide to H-88 Kitchen Cabinet Enamel and if that won't work try P-99 Toilet Seat Enamel with a little less plasticizer. (Chemists always appreciate this type of advice from the sales force.) And, oh, yes, gotta have it by Tuesday. I am playing golf with old man Buncombe in the afternoon to sign the order."

Then after the chemist had labored like a mountain to bring forth his mouse and the sample turned out to be a stinkeroo the salesman wept bitterly into his beer and cried that all chemists were bums and should be garrotted in some terrible way. Was the chemist to blame? Decidedly not!

This company didn't want a chemist; they wanted a Wizard of Oz.

Times haven't changed so very much after all!

A few of the By Gum quickies—

Barium: What one does to dead people.

•

Barber: A brilliant conversationalist who occasionally cuts hair.

•

Sympathy: What one girl offers another in exchange for details.

•

Chiropractor: A fellow who gets paid for doing what an ordinary guy would get slapped for.

•

Advice: The counsel old men give to young men when they can no longer set them examples.

•

The psychiatrist was examining little Willie, whose conduct had been giving his parents much concern.
"What do you like to do best, Willie?"
"I like to shoot birds."
"And what do you like to do next best?"
"I like to make slingshots to shoot birds with."
"Well, what do you like to do next best?"
"I like to take little girls' bloomers off."
Now we're getting somewhere, thought the psychiatrist.
"What do you like to do then?"
"Take the elastic out of the bloomers to make slingshots to shoot birds with."

—Herb Hillman
Humbug's Nest
P.O. Box 135
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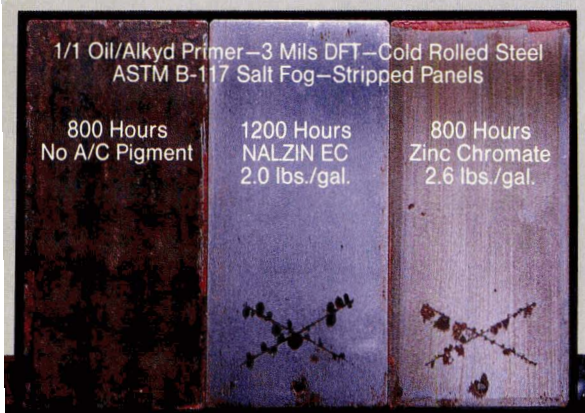

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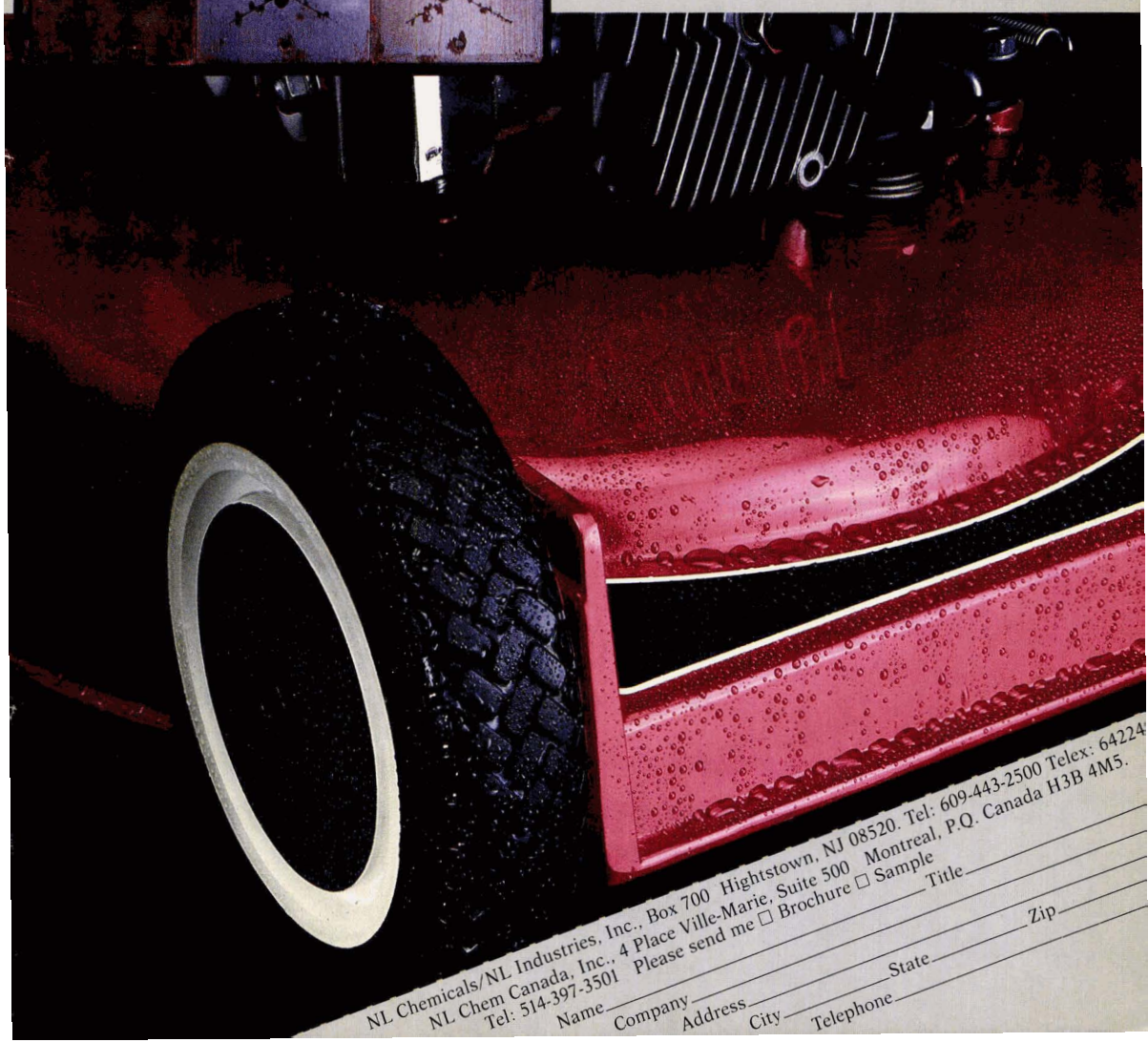

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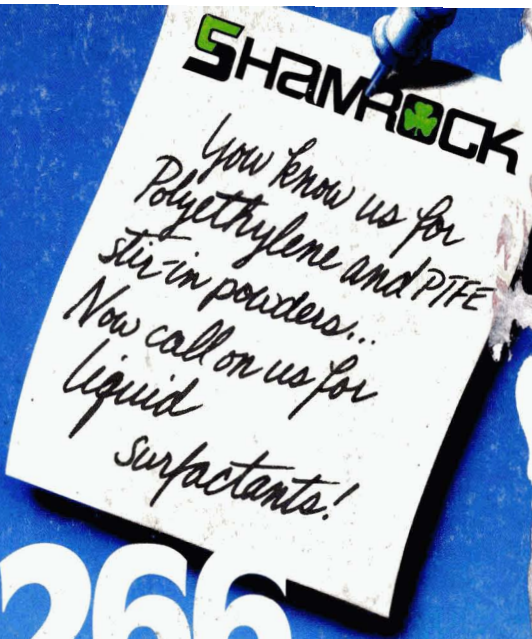

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