jct JOURNAL OF COATINGS TECHNOLOGY

JCTAX 55 (702) 1-88 (1983)

JULY 1983

N,N-dimeth

oxymethyl-

N',N',N'',N''-te

tramethylm

elamine(I)

Model Compound for Melamine Formaldehyde Resins

FSCT
61st Annual Meeting
48th
Paint Industries' Show
MONTREAL
October 12-14, 1983

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JULY 1983

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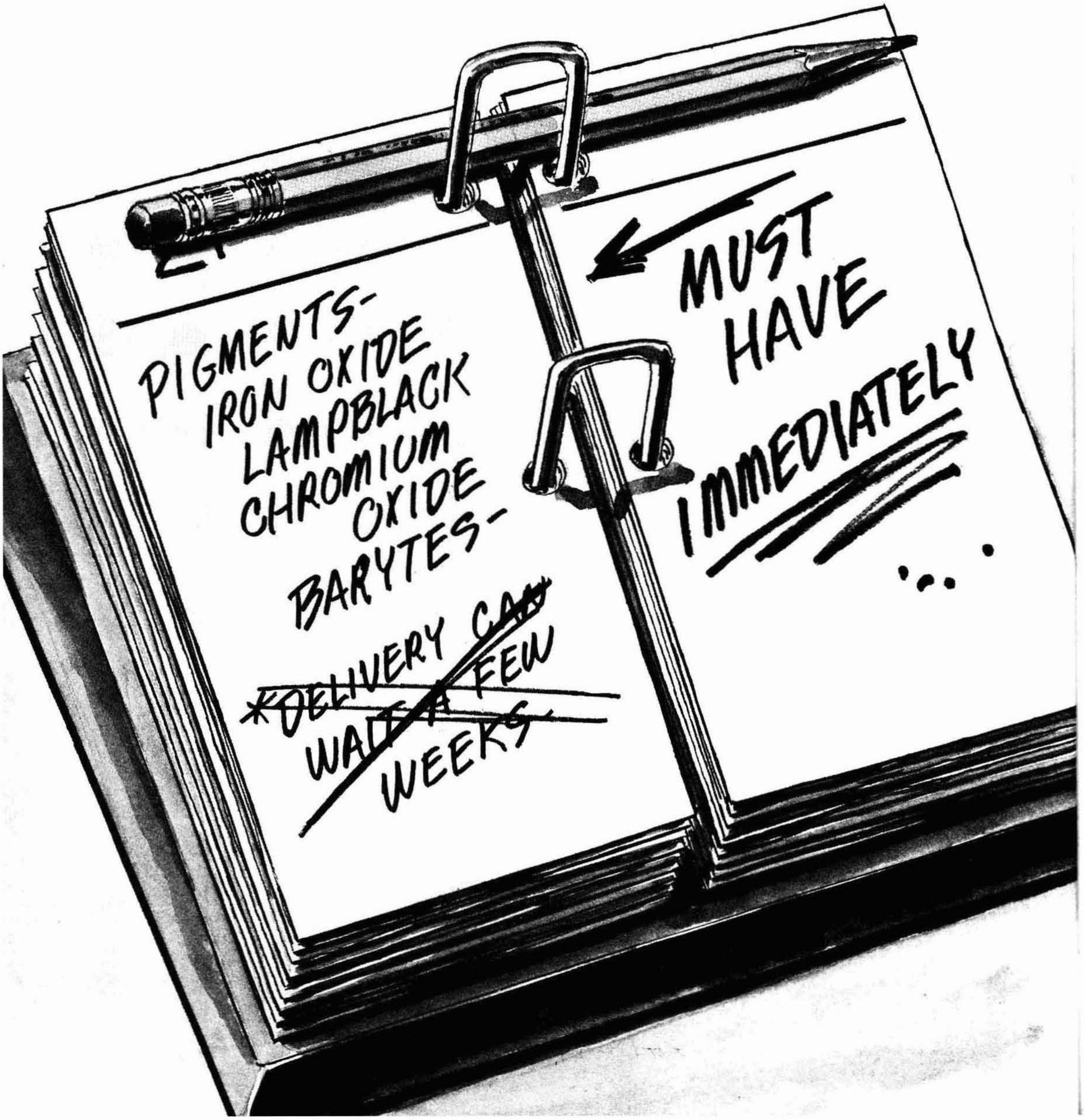
THE JOURNAL OF COATINGS TECHNOLOGY (ISSN 0361-8773) is published monthly by the Federation of Societies for Coatings Technology, 1315 Walnut St., Philadelphia, PA 19107. Phone: (215) 545-1507.

Second class postage paid at Philadelphia, PA and at additional mailing offices, POSTMASTER: Send address changes to JOURNAL OF COATINGS TECHNOLOGY, 1315 Walnut St., Philadelphia, PA 19107.

Subscriptions: U.S. and Canada—1 year, $20; 2 years, $37; 3 years, $52. Europe (Air Mail)—1 year, $40; 2 years, $77; 3 years, $112. Other countries—1 year, $30; 2 years, $57; 3 years, $82.



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LAMPBLACK
CHROMIUM
OXIDE
BARYTES-
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THE JOURNAL OF COATINGS TECHNOLOGY is published monthly by the Federation of Societies for Coatings Technology at 1315 Walnut St., Philadelphia, Pa. 19107. Phone: (215) 545-1506.

Annual dues for Active and Associate Members of the Federation of Societies for Coatings Technology is $15.00. Of this amount, $10.00 is allocated to a membership subscription to this publication. Membership in the Federation is obtained through prior affiliation with, and payment of dues to, one of its 26 Constituent Societies. Non-member subscription rates are:

	U.S. and Canada	Europe (Air Mail)	Other Countries
1 Year	$20.00	$ 40.00	$30.00
2 Years	$37.00	$ 77.00	$57.00
3 Years	$52.00	$112.00	$82.00

When available, single copies of back issues of the JOURNAL OF COATINGS TECHNOLOGY are priced as follows: $3.00 each for current calendar year issues; $4.00 each for all other issues.

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A Guide for Authors is published in each January issue.

The JOURNAL OF COATINGS TECHNOLOGY is available on microfilm from University Microfilms, a Xerox Co., Ann Arbor, Mich. 48106.

The Federation of Societies for Coatings Technology assumes no responsibility for the opinions expressed by authors in this publication.

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Comment

Safety Net

"Upon opening the Ball Mill, a sudden flash occurred as the overheated solvent was ignited by static electricity, etc."

In the coatings industry, our readers must have encountered numerous situations similar to the above which fall into the category of safety. These incidents may be minor or major and may have resulted in injury or loss of life and property damage. The net effect is that an action is taken to prevent the repetition of the accident or to learn the cause of the mishap.

Information from such occurrences are valuable teaching tools for others and could be a valued resource to JCT readers concerned with safety in the industry.

We urge our readers who have knowledge of safety related items to submit them to JCT for publication. If the response is appropriate we could provide an ongoing department dedicated to alert readers to this important facet of the coatings industry.

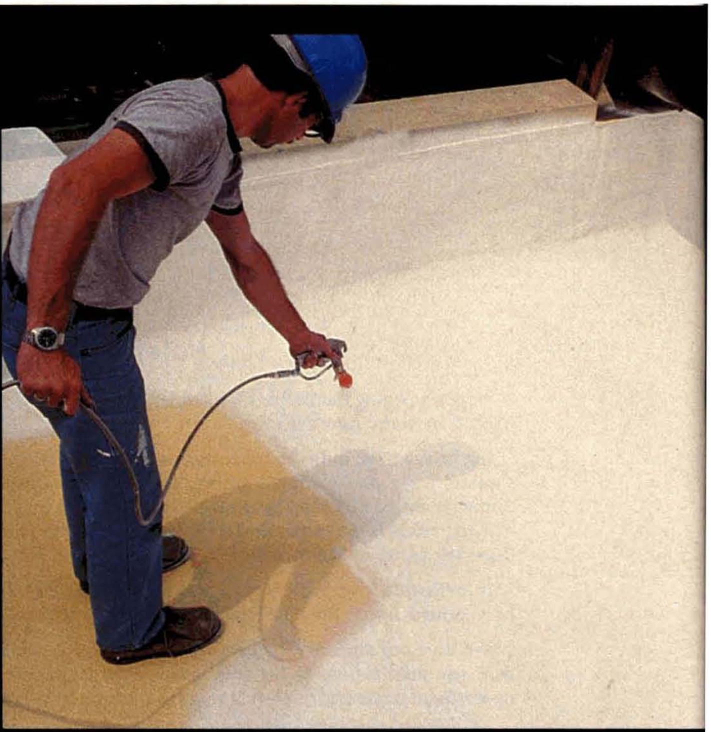
May we hear from you?

Thomas J Miranda

Thomas J. Miranda,
Technical Editor

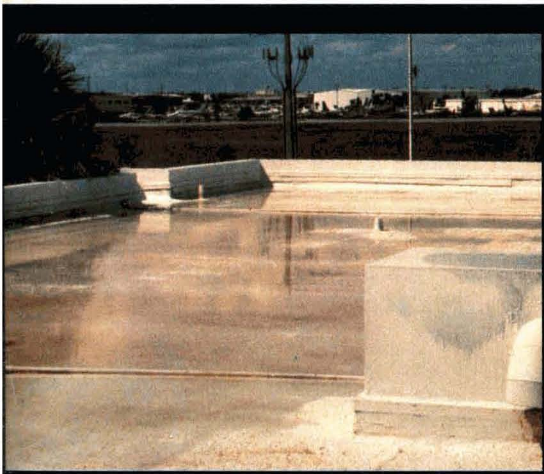
ACRYLIC BREAKTHROUGH

DO THE ROOFING

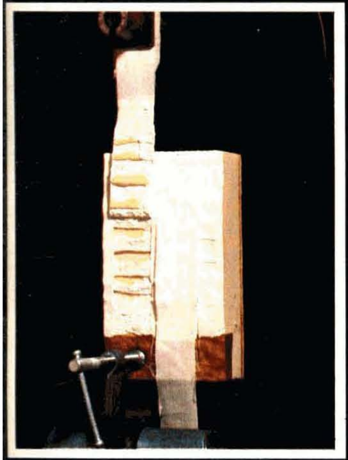


The reflective roof mastic based on RHOPLEX EC technology was spray applied on a roof in a heavy industrial environment in Pennsylvania. The photo at the lower right shows the same roof more than one year later.

INDUSTRY A BIG ACRYLIC FAVOR



This roof in Florida demonstrates how a roof mastic based on RHOPLEX EC-1895 acrylic retains its initial integrity under ponded water conditions.



The foam actually tore during peel adhesion testing on an Instron unit documenting adhesion of the mastic based on RHOPLEX EC-1791 acrylic.

Offer Roof Mastics Based on New RHOPLEX™ EC Technology

Introducing RHOPLEX® EC-1895 emulsion for superior resistance to water ponding conditions and RHOPLEX EC-1791 emulsion for superior adhesion to polyurethane foam. Acrylics from RHOPLEX EC technology are well known for:

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- **Long lasting flexibility even at low temperatures**
- **Superior dirt pick-up resistance and reflectivity**

RHOPLEX EC-1685 acrylic is the standard, when water ponding and adhesion are not problems.

For flat roofs with potential water ponding problems, the new RHOPLEX EC-1895 acrylic helps to keep bulk water from passing through the protective coating. Acrylics are known as breathers, not water vapor barriers. They transmit low levels of water vapor through the coating and thereby resist the formation of vapor traps in the building.

Another new roof mastic polymer, RHOPLEX EC-1791 provides elastomeric coatings with superior wet and dry adhesion to low and high density polyurethane foam.

Do your roofing contractors the favor of providing a seamless coating based on RHOPLEX EC technology. It goes on faster than sheets, eliminates hot tars, and reduces customer call backs. This saves time and money.

All you need to do to profit from this business opportunity is to combine your paint formulating know-how with RHOPLEX EC technology. Find out more about the product-line of acrylic polymers designed specifically for roof mastics. Write to Rohm and Haas Company, Marketing Services Department, Independence Mall West, Philadelphia, PA 19105.

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Federation News

Seminar on Computer Basics Among Program Highlights Scheduled for 1983 Annual Meeting in Montreal

A full-day seminar on computer basics for the coatings industry is among the highlight presentations announced for the 1983 Federation Annual Meeting, October 12–14, at Place Bonaventure, Montreal, Canada.

Program Chairman Peter Hiscocks, of C-I-L Paints, Inc., Toronto, said that the seminar on Thursday, October 13, is designed to answer basic computer-related questions and introduce the novice to affordable computer solutions to typical coatings problems.

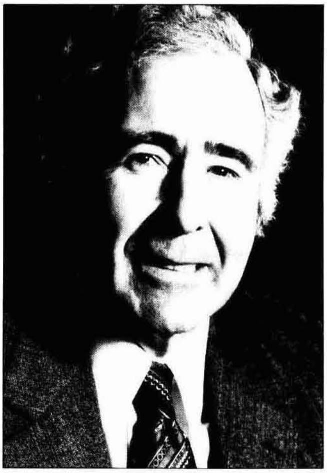
Topics to be covered included: turnkey computer systems for small and large paint companies; software for generating computerized formulations and batch ticket preparation; electronic spreadsheets vs. financial modeling software; color control with the small computer; personal computers for inventory systems; software available for microcomputers; the micro paint lab; and lab management software.

Computer hardware will be available for "hands-on" learning demonstrations and problem solving.

Other Program Highlights

The program, geared to the theme, "Knowledge Applied Profitably," will also feature the following presentations:

- Keynote Address by Jean-Marc Chaput, well-known Canadian motivational speaker.
- Joseph J. Mattiello Lecture by Frederick K. Daniel, founder of Daniel Products Co. and noted authority in the field of dispersion technology, who will speak on "The Obstacle Course from Mill Base to Finished Coating," (see May JCT).
- Seminar on air-dry aqueous binders for anti-corrosive coatings, with speakers from various supplier firms discussing current technology to help fulfill market needs.
- Manufacturing session on ways to improve profitability through efficient recycling and reclamation techniques.
- Environmental control session on how governmental health and safety regulations affect the coatings industry.
- Roon Awards competition papers.
- Constituent Society papers.
- Papers presented on behalf of affiliated overseas organizations (FATIPEC, Oil & Colour Chemists' Association, and Scandinavian Paint and Varnish Federation).



**Keynote Speaker
Jean-Marc Chaput**

These presentations will be supplemented by papers addressing the various aspects of the program theme.

Paint Show

To be held concurrently with the Annual Meeting, the 1983 Paint Show will be the largest in Federation history. Over 170 supplier companies have reserved booth space for displaying their products.

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

Exhibit hours will be 11:30 am to 5:30 pm on Wednesday, October 12; 9:30 am to 5:30 pm on Thursday, October 13; and 9:30 am to 3:00 pm on Friday, October 14.

Headquarters Hotel

The Queen Elizabeth will serve as headquarters hotel. Other hotels with blocks of rooms set aside for the Annual Meeting are the Hotel Bonaventure, L'Centre Sheraton, Regence Hyatt Montreal, Chateau Champlain, Hotel Meridien Montreal, Ramada Inn, Holiday Inn Downtown, and the Mt. Royal.

Room Reservations

All requests for rooms and suites must be sent to the Federation office on the official housing form which has been mailed to all members and is included in this issue (see pages 20–28). Additional housing forms are available from the Federation headquarters office.

Registration Fees

Advance registration fees are available for $50 for members and $65 for nonmembers. Fee for spouses activities is $35 in advance.

Once again there will be a special registration fee of $25 each for retired members and their spouses. This applies to advance registration only.

On-site registration fees will be $60 full time and $40 one day for members. Nonmember fees will be $75 full time and $50 one day. Spouses registration will be $45 on-site.

Registration forms were mailed to all members in April and are included in this issue (see pages 20–28).

Federation Luncheon

The Annual Federation Luncheon will be held on Friday at the Queen Elizabeth Hotel.

Presentations will be made to the recipients of the George Baugh Heckel Award (outstanding individual who has contributed to the advancement of the Federation) and the Flynn Awards (firms judged to have the best exhibit booths in the 1983 Paint Industries' Show). Announced will be the winners of the Annual Meeting awards, including the winning entries in the Roon Awards Competition.

Featured entertainment will be Dave Broadfoot, Canada's premier comedian and satirist. Winner of the many media awards for his performances, *Variety* calls him "Canada's Bob Hope." Appearing before 120 conventions a year, Mr. Broadfoot has given command performances before President Reagan and Her Majesty Queen Elizabeth.

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Filler	Lbs/ Gallon*	Cost/ Gallon Index**	Hegman	60° Gloss	85° Sheen
Celite 499	1.0	100	4	12	24
Celite White Mist®	1.0	190	5	15	32
Hydrogel	0.9	400	5½	16	30
Precipitated Silica	0.9	290	5½	20	34
Calcium Carbonate	3.0	70	6	18	55
Talc, Micronized	2.5	160	6	20	52
Amorphous Silica	2.75	130	6	28	48
Delaminated Clay	2.75	120	5½	26	58
Calcined Clay	2.5	150	5½	19	50

*Addition is to high gloss white alkyd enamel mixed 5 minutes in disc disperser. Initial enamel reading: 89° gloss, 94° sheen.

**Index uses Celite 499 as base = 100. Ratios based on 10,000 lb. shipments FOB NY, December, 1982.

Manville

Abstracts of Papers in This Issue

MODEL COMPOUND FOR MELAMINE FORMALDEHYDE RESINS—Z.W. Wicks, Jr. and D.Y.Y. Hsia

Journal of Coatings Technology, 55, No. 702, 29 (July 1983)

N,N-dimethoxymethyl N′,N′,N″,N″-tetramethylmelamine (I) has been synthesized as a model compound for melamine-formaldehyde resins. It is thermally stable and reacts only slowly with water or alcohol without catalyst. In the presence of catalyst and water it undergoes condensation reactions leading to the formation of a cyclic trimer. The reaction was shown to be reversible. (I) reacts rapidly with tetrahydrofurfuryl alcohol in the presence of catalyst to form the mono and then diether substitution products. Preliminary kinetic data indicate that the catalyzed reaction with alcohol does not follow either first or second order kinetics. It is possible that both S_N1 and S_N2 reactions occur.

SOME ASPECTS OF THE GRAFT POLYMERIZATION OF METHACRYLATES ONTO CARBON BLACK SURFACES BY BUTYLLITHIUM—K. Ohkita, N. Nakayama, and T. Ohtaki

Journal of Coatings Technology, 55, No. 702, 35 (July 1983)

Graft polymerization of methyl and butyl methacrylates onto carbon black surfaces was investigated using butyllithium or carbon black-butyllithium complex as an initiator. Methyl methacrylate differed from styrene in that it had faster initiation and negligible butyllithium-carbon black interaction. Presence of an HAF black treated with sodium borohydride gave a homopolymer with lower intrinsic viscosity than that with an untreated black. Methacrylate grafted onto a color black by the carbon black-butyllithium complex resulted in a gel containing carbon particles. When an excess of butyllithium beyond that required for complex formation was used, there was a decrease in gel fraction as well as in grafting ratio. A mechanism for gelation is proposed.

APPLICATION OF QUASIELASTIC LASER LIGHT SCATTERING FOR CHARACTERIZATION AND QUALITY CONTROL OF COLLOIDAL DISPERSIONS—F.B. Malihi, T. Provder, and M.E. Koehler

Journal of Coatings Technology, 55, No. 702, 41 (July 1983)

Recent applications of quasielastic laser light scattering (QELS) have demonstrated the potential of this relatively new technique for the characterization of charged colloidal particles. QELS provides a precise and rapid means for the determination of particle size and electrophoretic mobility of dilute colloidal suspensions. The information on the particle size and surface charge as obtained by QELS, makes it a valuable tool to study the hydrodynamic and electrostatic properties of synthetic polymers as they relate to the dispersion stability and rheology of water-borne coatings. Such investigations may be applied to a wide variety of systems including latexes, water-soluble polymers, micelles, microemulsions, and colloidal aggregates.

In this paper the applications of QELS to the characterization of some types of dispersions and emulsions used in the coatings industry are discussed. Specific examples include the analysis of particle size and size distribution of latex suspensions, the study of particle growth during emulsion polymerization, measurement of the degree of expansion of carboxylic latexes during neutralization, and the study of electrophoretic mobility of functional latex particles.

(Continued)

Papers to be Featured in the August Issue

Acrylic Copolymer Oligomers for High Solids Coatings Resins—D. Rhum, of Pfizer, Inc., and P.F. Aluotto, of Inmont Corp.

Design of Water-Borne Coatings for the Corrosion Protection of Steel.
Part III: Effect of Surfactants in an Aqueous Air Dry Coating—New England Society for Coatings Technology

Gadgets and Gimmicks—Southern Society Annual Meeting

Improved Analysis of Static Panel Immersion Testing Results—A.M. Becka, of David W. Taylor Naval Ship Research and Development Center.

Isocyanatoethyl Methacrylate: A Heterofunctional Monomer for Polyurethane and Vinyl Polymer Systems—M.R. Thomas, of Dow Chemical U.S.A.

Structure-Property Relationships for Radiation Curable Coatings—A. Priola, F. Renzi, and S. Cesca, of ASSORENI, Polymer Research Laboratories

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Compare the Spectrogard Automatch™ System with other color matching systems. You'll find there's no comparison. The Spectrogard Automatch System is the most significant innovation in computer color matching in recent years. It offers advanced capabilities not available on other systems.

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The Spectrogard Automatch System is a complete color matching system with formulation, batch correction and quality control capabilities. It has been designed with you—the Colorist—in mind:

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Abstracts of Papers in This Issue (Continued)

CHEMISTRY OF HIGH SOLIDS ALKYD/REACTIVE DILUENT COATINGS—D.B. Larson and W.D. Emmons

Journal of Coatings Technology, 55, No. 702, 49 (July 1983)

Pressures have increased in recent years to reduce both solvent emissions and thermal curing requirements for organic coatings. A practical response is a system based on oil modified alkyds formulated with methacrylate reactive diluents which function initially as solvents but then convert to binder. Basic requirements of a reactive diluent include low volatility, toxicity, and odor with good compatibility and stability. Dicyclopentenyloxyethyl methacrylate is a vinyl monomer that has these properties. Another requirement is rapid conversion during film formation, a process strongly opposed in thin films by oxygen inhibition of free radical polymerization. Proper coating design controls the oxygen content in the film by rapidly increasing film viscosities to slow oxygen diffusion rates and by efficiently scavenging oxygen using alkyd unsaturation. Keeping reactive diluent levels in the 15–30% range is a key part of the design. Fully complying high solids systems are achieved by further constraining the system to use low molecular weight alkyds. Model systems and practical high solids alkyd/reactive diluent coatings illustrate design principles and rationalize observed coating properties.

STUDIES ON THE ESTERIFICATION OF TRIMELLITIC ANHYDRIDE. IMPLICATIONS FOR COATING POLYMERS—W. Riddick

Journal of Coatings Technology, 55, No. 702, 57 (July 1983)

The esterification of trimellitic anhydride (TMA) with three hydroxyl-functional reactants was observed at various reaction temperatures. Results from the reaction with 2-ethylhexanol showed that TMA monoester was formed initially, and all the carboxyl groups of the monoester and diester species had approximately equal reactivity during the remainder of the reaction. The resulting distributions of TMA monoester, diester, and triester were found to be independent of reaction temperature and dependent only on extent of TMA reaction. Results from the reactions with diethylene glycol and a polyester diol led to similar conclusions. Practical implications of the results pertaining to coating polymers are explored.

• • •

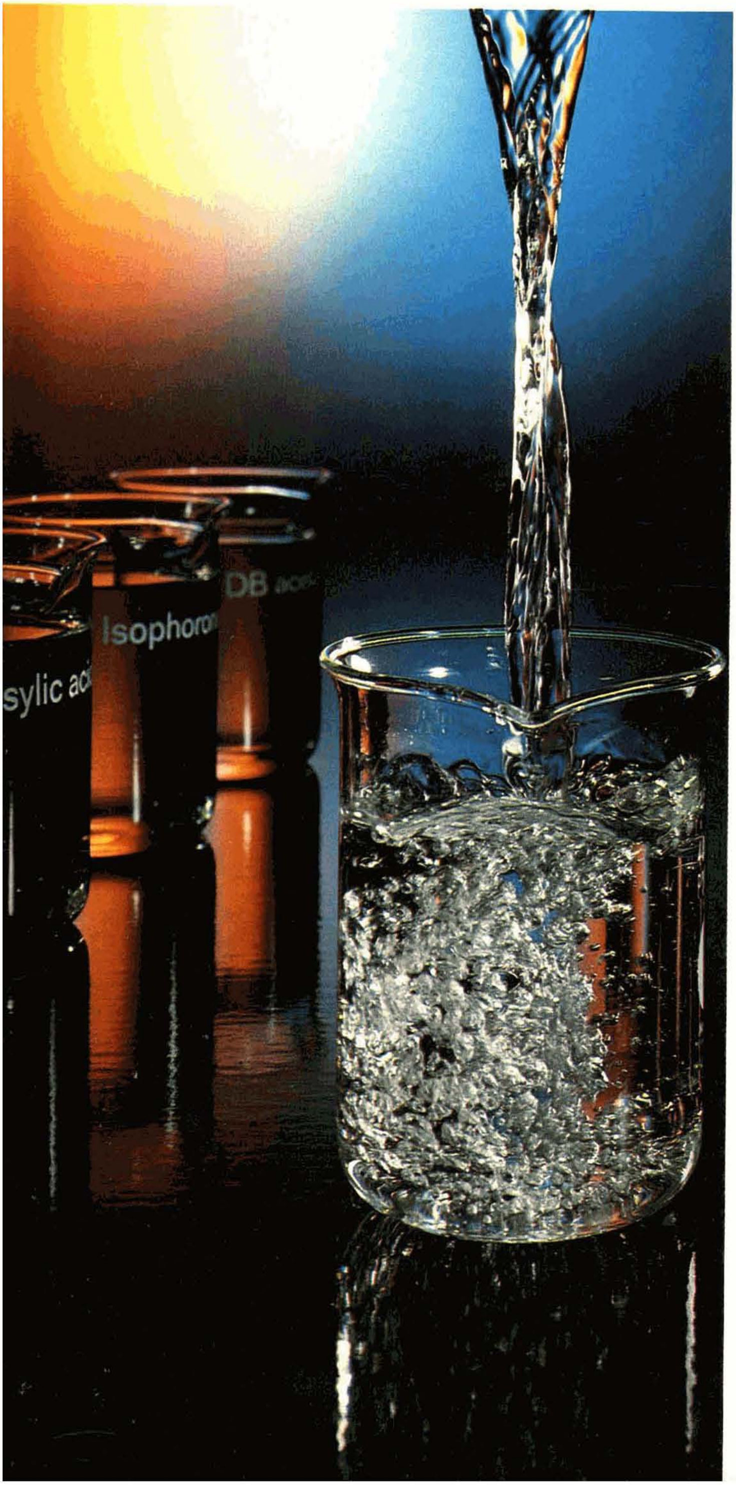
September 1983 JCT Convention Issue

The 1983 Annual Meeting and Paint Industries' Show of the Federation of Societies for Coatings Technology will be held at Place Bonaventure Montreal, Que., Canada, October 12–14.

The September issue will contain a special section featuring . . .

- Technical Program Sessions
- Abstracts of the Papers to be Presented at the Annual Meeting
- List of Paint Industries' Show Exhibitors and their Booth Numbers
- List of Exhibitors by Product/Services Classification
- Floor Plan
- Other Pertinent Convention Information

Switch to DuPont DBE*...get major savings in solvent costs.



Coatings formulators have saved up to 45% with this readily available solvent.

Try Du Pont's Dibasic Ester solvent in place of glycol ethers, glycol ether acetates, and high-boiling ketones and aromatics to develop paint formulations that produce high performance finishes... formulations that can result in major savings in solvent costs.

You'll discover that DBE is compatible with most common resin systems. Also, Dibasic Ester solvent can give you higher solids formulations without adversely affecting paint rheology. In fact, DBE has been found to improve the rheology of high solids coating systems by reducing viscosity and improving leveling properties. This makes DBE suitable for a wide range of industrial and automotive coatings.

Along with cost reduction and versatility, DBE solvent is exempt from Rule 442 (photochemically nonreactive) and can be stored indoors without restriction.

Get assistance in your R&D evaluation.

Let us show you how you can use Du Pont's DBE in your coating application. We can help you formulate a DBE blend tailored to fit your existing paint system or a new paint line. For further information and samples, write: Du Pont Company, Room G39261, Wilmington, DE 19898.

*Mixed dibasic esters of adipic, glutaric and succinic acids.

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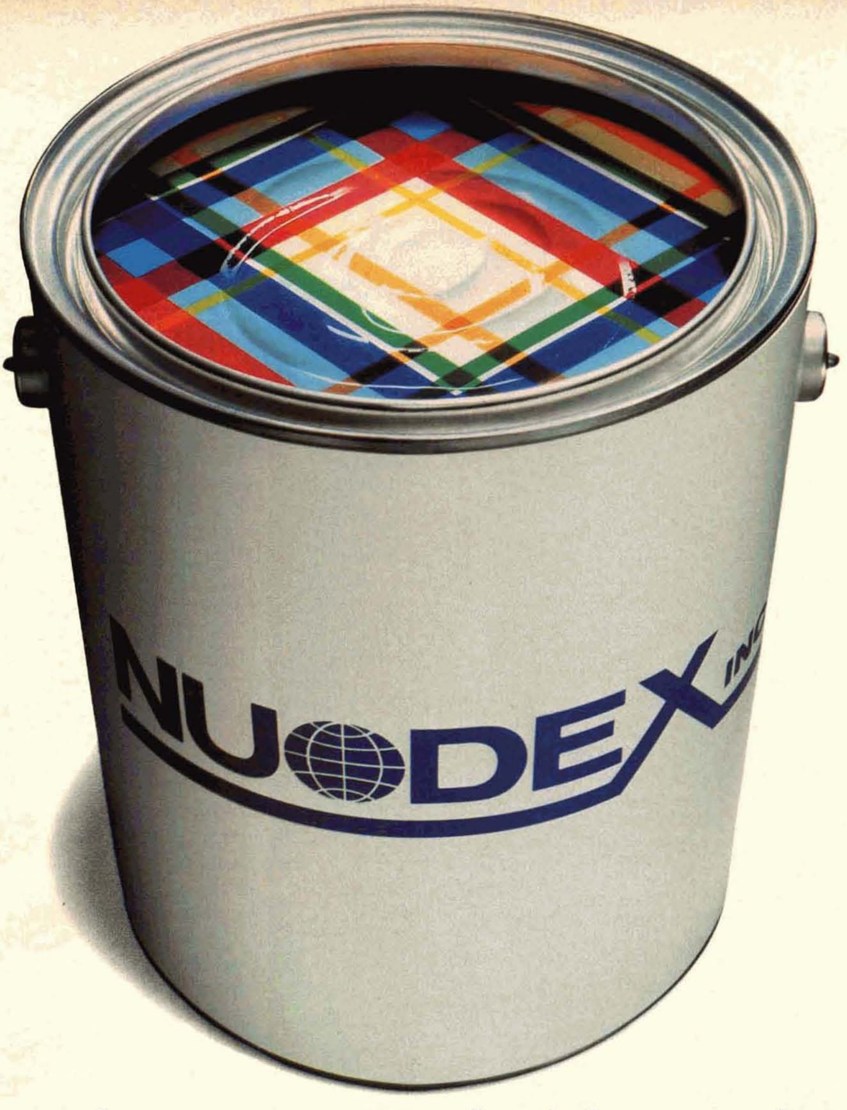

NUODEX INC

Government and Industry

EPA Issues Effluent Guidelines and Pretreatment Standards Affecting Synthetic Resin Producers

The U.S. Environmental Protection Agency (EPA) has issued its proposed *effluent limitation* guidelines and pretreatment standards for the Organic Chemicals, Plastics and Synthetic Fibers Category as required by the Clean Water Act in the March 21st *Federal Register.* These regulations when issued in final form will limit the discharge of toxic effluents into the waters of the United States and into publicly-owned treatment works from facilities that produce organic chemicals, plastics and synthetic fibers.

Included under the Plastics subcategory are process wastewater discharges generated during the manufacture of synthetic resins. Facilities generally included in this subcategory are identified by SIC 2821. Companies that manufacture synthetic resins and are either a direct or indirect discharger of resin process wastewater will be affected by this rulemaking.

The Organic Chemicals and Plastics and Synthetic Fibers Category covers a large and diverse group of facilities. The majority of the process wastewater is being generated by about 1500 facilities whose entire production relates solely to organic chemicals, plastics or synthetic fibers, i.e., facilities of the many major polymer manufacturers. Other facilities in this category are secondary producers, with organic chemicals, plastics or synthetic fiber products being ancillary to their primary production. This is the case with many companies who produce paint and coatings resins for their own consumption or for only limited commercial sale. These facilities, though not major generators of process wastewater, still may face significant impact from these regulations. Preliminary estimates by EPA indicated that the overall cost of compliance with these regulations for the entire Organic Chemical, Plastic and Synthetic Fiber Category to be close to $750 million per year, with an initial capital investment of $1.7 billion. The full extent of the economic impact of these regulations is not known at this time.

Companies which manufacture synthetic resins should review the proposed rule to determine its impact on their operations. A complete copy of the 40-page proposed rulemaking and a copy of the EPA Technical Development Documents can be obtained directly from EPA by contacting E.H. Forsht, Senior Project Officer, at (202) 382-7135 or Denise Beverly, Distribution Officer, at (202) 382-7115; Effluent Guidelines Division, 401 M Street, S.W., Washington, DC 20460.

The National Paint and Coatings Association Water Quality/Waste Management Task Force has formed a Special Work Group to evalute these proposed regulations and formulate the NPCA response.

For additional information on this proposal and on the activities of the Special Work Group, please contact Bob Nelson, Associate Director, Environmental Affairs, at NPCA headquarters, (202) 462-6272.—NPCA *Coatings*

Mearl Corp. Celebrates 50th Year

The Mearl Corp., New York, NY, producer of pearlescent and iridescent pigments, is celebrating its 50th Anniversary Year.

In 1933, the corporation was founded by Harry Mattin and Francis Earle. Its first product was pearl essence extracted from the scales of herring; the pigment was used in the production of simulated pearls, nail enamels, and custom automobile finishes.

During World War II, Mearl produced fire fighting foam for the Navy and Air Force, which it developed from the waste products of the fish scale operation.

After the war, the firm was involved in the production of synthetic pearl pigments, which found wide application in plastics. These pigments made possible the manufacture of simulated pearl buttons, and essentially replaced buttons made from natural shells.

In 1963, the firm established its research and development laboratory in Ossining, NY, and expanded its product lines.

Continuing research and development has created titanium-mica pigments with unique color effects.

Extender and Filler Pigment Consumption To Reach $1.2 Billion in U.S. by 1987

In 1982, the total volume of extender and filler pigments consumed in seven key industries reached 6.8 million tons valued at over $750 million according to a recently completed survey. While the effects of the recession continued to impact individual markets in 1982, causing moderate declines for such major pigments as calcium carbonate and kaolin, total demand is forecast to reach $1.2 billion by 1987.

The paint industry is the second largest end user with a consumption of $120 million.

The consumption of extender and filler pigments among the seven key industries tracked in this survey is expected to surpass $1.2 billion by 1987. Although the paper industry will continue to be the largest end market for extender and filler pigments, the paint industry, which is heavily dependent on automotive and housing applications, will experience an accelerated growth in consumption over the next two years from its present depressed level.

Extender and filler pigments are comprised of four major mineral groups and several individual minerals and specialty chemicals. Kaolin clay products account for over 25% of all the extender and filler pigments used in paint. Calcined kaolin contributes to hiding power in architectural coatings, is a cost effective titanium dioxide extender, and accounted for over 50% of the dollar volume of kaolin consumed in paint. Air-floated kaolin is expected to enjoy the highest rate of growth in adhesives applications as the industry continues to move toward water-base formulations.

Calcium carbonates, including ground, superfine, and precipitated types, are expected to show the highest rate of growth among all products. A major contributor to this growth is the conversion to alkaline papermaking systems. Still, a significant portion of the growth will come from increased sales to drywall compound manufacturers as the housing market improves.

Silica products, including fumed, gel, ground, and precipitated silicas, find application primarily as flatting agents in paint and as fillers in plastics and rubber. Fumed silica products are expected to exhibit the highest rate of growth as their use in specialty adhesives and sealants increases. Talc products and other specialty pigments represent the balance of sales.

The U.S. market for extender and filler pigments was supplied by about 100 companies in 1982. The report analyzes in detail the leading 16 suppliers, which together account for over 80% of the products sold, and provides information on 30 smaller suppliers with sales of $0.5 million or more. According to the survey, the leading supplier of extender and filler pigments was J. M. Huber, which competed in nine product categories in 1982, and accounted for 14% of the total dollar sales.

The report, *Extender and Filler Pigments 1983—A Continuing Business Review* represents the second edition of a three-year service designed to monitor developments in these large and changing markets. The second edition analyzes in detail the use of extender and filler pigments in paint, adhesives, and putty, caulks, and sealants. Volume Two is based on 127 interviews with consumers, suppliers, trade associations, and government agencies. Results of the second phase of the survey are currently available on subscription only from Charles H. Kline & Co., Inc., 330 Passaic Ave., Fairfield, NJ 07006.

CMA Urges Modifications to Clean Water Act

A representative of the Chemical Manufacturers Association appeared before the Senate Environmental Pollution Subcommittee to urge adoption of an amendment that would modify requirements that industry install the next level of pollution control technology called for in the Clean Water Act—known as Best Available Technology (BAT).

R.C. Forney, an Executive Vice-President of the duPont Co. and representative of CMA, said that the Clean Water Act must begin to reflect the success the nation has achieved in cleaning up its waterways and that industry be given the "statutory flexibility" it needs to meet the clean water goals that remain. Those goals are designed to provide the nation with good, clean water—not with seeing how much technology industry can be required to install, said Mr. Forney. Improvement in water quality is the means by which we should measure success, he stressed.

Mr. Forney pointed out to the Subcommittee that the new, across-the-board technical requirements may not be needed in all cases. Existing pollution control systems, known as Best Practicable Technology (BPT), have resulted in significant benefits. He noted that recent studies, including a study conducted by CMA in cooperation with the Environmental Protection Agency, have shown that existing control technology has done a better-than-expected job of controlling pollution.

What was not expected, Mr. Forney said, was (its) effectiveness in controlling priority (toxic) pollutants. He noted that the joint CMA/EPA study, which was begun in 1980, found that existing biological treatment technology removes toxic organic pollutants to effluent concentrations at or below the accepted detection limit of 10 parts per billion (ppb).

The success of current technology on priority pollutants raises questions regarding the need for across-the-board application of BAT.

The chemical industry, Mr. Forney said, is not attacking the concept of BAT. Rather, the modification of BAT should be allowed on a case-by-case basis, with the burden of proof on the company involved to demonstrate that existing technology is doing an effective job of controlling pollution.

This demonstration, Mr. Forney told the panel, would have to be based on good analytical and aquatic toxicological evidence, and the final decision on whether BAT is needed would be in the hands of the permitting agency.

Lacquer and Chemical Corp. Acquired by Ell-Bee Chemical

Ell-Bee Chemical Co., Inc., East Hanover, NJ, has announced the acquisition of Lacquer and Chemical Corp., Brooklyn, NY.

The business will be transferred from its current location to a new manufacturing plant recently constructed at Ell-Bee's headquarters. The new facility incorporates modern coatings technology and includes a development/customer service laboratory.

Ell-Bee is involved in the production of nitrocellulose solutions, lacquers, wood stains, and specialty industrial coatings. Lacquer and Chemical produces high technology coatings for computer applications as well as specialty industrial enamels.

Committee Activities

FSCT Corrosion, Manufacturing Committees Meet; Discuss Annual Meeting Program Presentations

Recent, separate meetings of the Corrosion and Manufacturing Committees of the Federation produced tentative plans for Committee-sponsored programming at the 1983 Federation Annual Meeting, to be held in Montreal, Quebec on October 12-14.

The following are highlights from these two meetings:

Corrosion

Meeting in New York City on March 15, the members of the Committee discussed proposed topics for a Committee-sponsored Annual Meeting session. They agreed to develop a program on the topic "Air-Dry Aqueous Binders for Anti-Corrosive Coatings." The subject would be covered in two, two-hour sessions, providing adequate time for presentations and open-forum participation by the audience.

Speakers from various resin manufacturers will present information on current technology to help fulfill market needs through a comparison of air-dry aqueous binders in anti-corrosive coatings. Committee members were assigned companies to contact for participation.

The Committee reaffirmed its interest in encouraging Society Technical Committees to pursue project work on corrosion and its control. The following are among the topics Committee members feel would be worthwhile and promising for Society Technical Committee work:

- Determination of moisture vapor permeability as a tool to predict corrosion inhibition of water-based coatings.
- Effect of degree of dispersion on performance of corrosion-resistant pigments.
- Evaluation of performance of proprietary, conversion coatings as corrosion-inhibiting products when used over rusty steel.
- Employment of multi-environment testing to facilitate prediction of corrosion inhibition.

These and additional project recommendations were presented and discussed by the Chairman at the April 28-29 meeting of the Federation Technical Advisory Committee and Society Technical Committee Chairmen.

The next meeting of the Corrosion Committee was tentatively scheduled for Wednesday morning, October 12, in Montreal, in conjunction with the 1983 Federation Annual Meeting.

Members of the FSCT Corrosion Committee are: *Chairman,* Saul Spindel, D/L Laboratories, New York, NY; Jay Austin, Halox Pigments, Hammond, IN; Dean M. Berger, Gilbert Associates, Inc., Reading, PA; Alex Chasan, U.S. Navy, Annapolis, MD; Gary W. Gardner, Porter Paint Co., Louisville, KY; Thomas Ginsberg, Union Carbide Corp., Bound Brook, NJ; Sidney Levinson, D/L Laboratories, New York, NY; Richard Max, Synkote Paint Co., Elmwood Park, NJ; Horace Philipp, Sherwin-Williams Co. of Canada, Montreal, Que.; Lothar Sander, Amchem Products Co., Ambler, PA; and Armand J. Stolte, N L Industries, Inc., Hightstown, NJ.

Also attending were Roy Brown, FSCT Technical Advisor, and Tom Kocis, FSCT Director of Field Services.

Manufacturing

The committee met in St. Louis on March 23. They reviewed the Manufacturing Committee-sponsored seminar on computers in the coatings manufacturing process and plant tour held during the 1982 Annual Meeting in Washington, D.C.

The success of the plant tour (Duron, Inc., Beltsville, MD), held for Committee members and Society Manufacturing Committee Chairmen, led to discussions of a proposed similar effort in conjunction with the 1983 Annual Meeting in Montreal. Prospective Montreal-area firms will be contacted. The tour might involve both a raw materials supplier and a coatings facility.

The committee adopted the topic "Reclaim Your Profits—Improved Profitability Through Efficient Cleaning, Recycling, and Reclamation Techniques," for the Committee-sponsored 1983 Annual Meeting presentation. Suggested subjects included tank cleaning, solvent recovery, and waste disposal.

The Kansas City Society offered for review their newly-revised slide-tape program on "Sand Mill Operation." The program was well received and a number of suggestions were made for refinement. The Society hopes to have the program available for showing at the Montreal Annual Meeting.

Meanwhile, the Houston Society continues with its efforts to update and revise its production of "Cartridge Straining." The Baltimore Society is considering developing a presentation on "Vibrating Strainers."

The Committee suggested several topics for Society consideration for A/V programs: Horizontal Mill; Filling Equipment; Computer Control; Conveying Equipment; and Safety Training.

The Committee reaffirmed its desire to stimulate interest in manufacturing-type activities at the Society level. A Manufacturing Committee presentation at a Society monthly meeting was suggested as effective. Good topics to consider for such a presentation are: solvent recovery, dust collectors, and roofing maintenance. Committee members volunteered to compile a list of such presentations made over the last few years as a source, and to maintain liaison with the National Fire Protection Association.

Kansas City Society reported on the results of a survey they conducted on Filtration Procedures Used in Resin Manufacture. Approximately 30 firms responded (out of 240 queried). The report will be written and submitted for publication in the JOURNAL OF COATINGS TECHNOLOGY.

The next meeting of the Manufacturing Steering Committee was tentatively scheduled for Wednesday morning, October 12, in Montreal, in conjunction with the Federation Annual Meeting.

Members of the FSCT Manufacturing Steering Committee are: *Chairman,* Richard E. Max, Synkote Paint Co., Elmwood Park, NJ; Michael O. Beatty, Athey Paint Co., Baltimore, MD; Cleties Crowe, Crowe Industrial Coatings, Stone Mountain, GA; Thomas Currens, Sherwin-Williams Co., Garland, TX; Thomas J. Daly, Process Design Assoc., Inc., Chicago, IL; George E. Finn, Burn-Zol Div., W. Dennis, MA; Donald Fritz, Superior Varnish & Drier Co., Merchantville, NJ; Robert S. Hogg, Preservative Paints Co., Seattle, WA; Larry Kytasaari, Tnemec, Inc., Kansas City, MO; Alun G. Morris, L.V. Lomas Chemical Co. Ltd., Mississauga, Ont.; Richard Mundinger, 3M Co., St. Paul, MN; Louie Sanguinetti, Jasco Chemical Corp., Mountain View, CA; Joseph P. Walton, Jr., Jamestown Paint & Varnish Co., Jamestown, PA; and John Wood, Cook Industrial Coatings, Detroit, MI.

Federation of Societies for Coatings Technolog

1983

61st ANNUAL MEETING
48th PAINT INDUSTRIES' SHOW

Federation of Societies for Coatings Technology
YE OLDE PAINT STONE 1737

MONTRÉAL

Place Bonaventure

October 12, 13, 14

1983 Annual Meeting
Paint Industries' Show
October 12, 13, 14
Place Bonaventure
Montreal, Quebec, Canada

TO OUR MEMBERS AND FRIENDS:

As the fifth Canadian President of the Federation, it is a distinct pleasure and privilege for me to invite you to the first Annual Meeting and Paint Industries' Show to be held in my native country.

The two host Societies—Montreal and Toronto—are eagerly awaiting this event and have been very busy in making arrangements so that you will have an enjoyable and memorable visit to the beautiful city of Montreal.

The Paint Show, which we expect will be the biggest ever, will be held in one of Canada's top showplaces, Place Bonaventure. This magnificent building, truly a city within a city, sits on top of another city within a city—Place Ville Marie.

Program sessions will be geared to the theme of the meeting, "Knowledge Applied Profitably." The selection of papers, workshops, and seminars being arranged by Peter Hiscocks and his Program Committee will send you home with a pocketful of ideas to put to profitable use in your companies.

On behalf of the 800 Canadian members associated with the Montreal, Toronto, Detroit, Northwestern, and Pacific Northwest Societies . . . Bienvenue a Canada.

A C Boyce

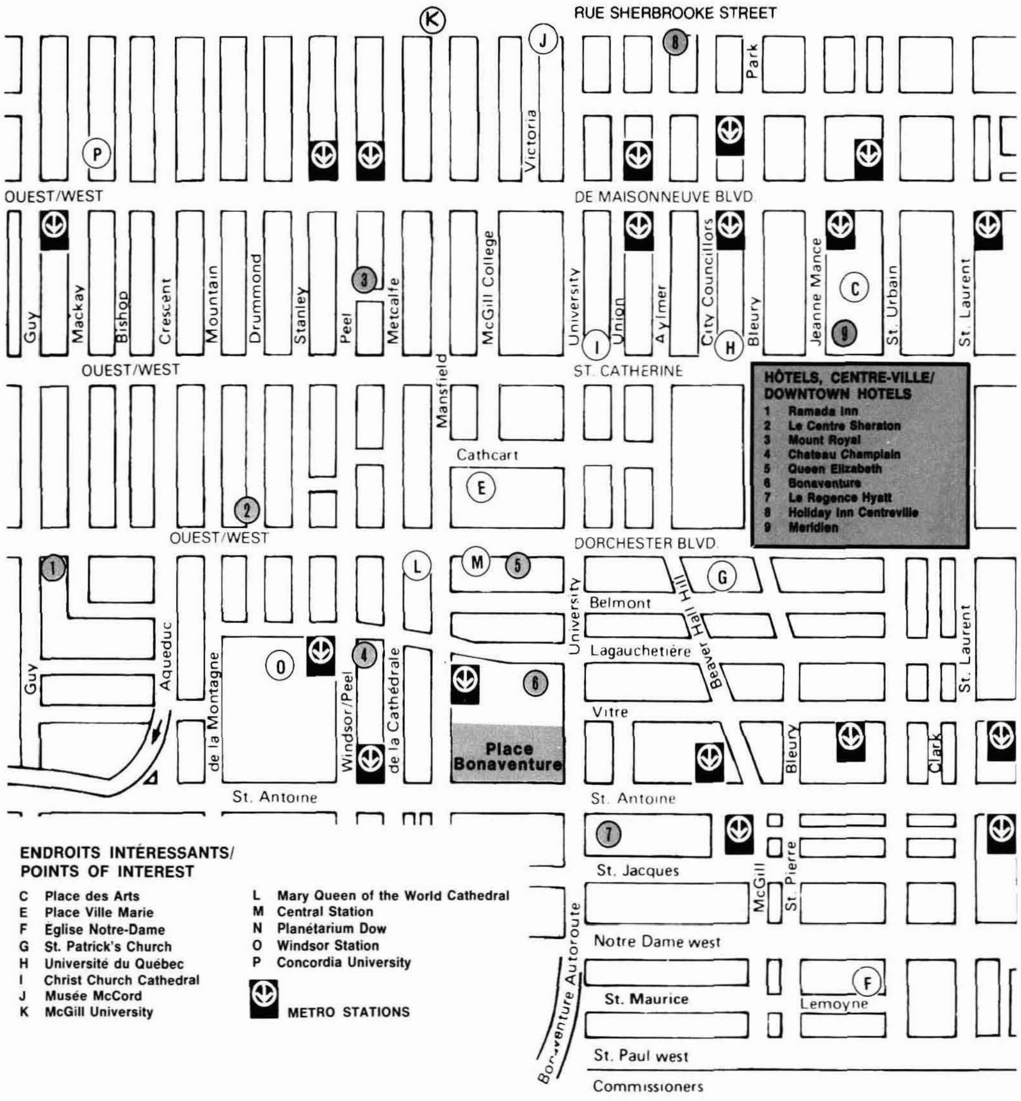
A. Clarke Boyce
President
Federation of Societies
for Coatings Technology

HOTEL INFORMATION AND RATES

All Rates are in Canadian Funds

Hotel	Singles	Doubles Twins	Parlor & 1 Bedroom	Parlor & 2 Bedrooms	Parlor & 3 Bedrooms
QUEEN ELIZABETH*	$79	$89	$208/220/246 271/280	$376/382/305 420	$544/635
HOTEL BONAVENTURE*	$95	$105	$250/400	$475	$525
L'HOTEL CENTRE SHERATON	$83	$96	$200/426	$430	
REGENCE HYATT MONTREAL	$83	$95	$164/220/330 480/600	$184/220/330 480/600	
CHATEAU CHAMPLAIN	$84	$96	$210/275	$375	
HOTEL MERIDIEN MONTREAL	$78	$88			
RAMADA INN	$59	$69			
HOLIDAY INN DOWNTOWN	$68	$74	$105		
MT. ROYAL	$53	$63			

*Requests for accommodations at either the Queen Elizabeth or the Bonaventure will be limited to five rooms per company. A parlor counts as one room.



FSCT 1983 ANNUAL MEETING AND PAINT INDUSTRIES' SHOW
PLACE BONAVENTURE, MONTREAL, QUEBEC, CANADA
OCTOBER 12, 13, 14 (Wednesday, Thursday, Friday)

APPLICATION FOR HOTEL ACCOMMODATIONS

MAIL TO:
Fed. Socs. Coatings Tech.
1315 Walnut St.—Dept. H
Philadelphia, PA 19107

All reservations will be processed by the Montreal Convention and Visitors Bureau.

Please indicate below the type of accommodations desired and choice of hotels. Assignments will be made in accordance with prevailing availability. Three to four weeks after mailing this application you will receive a confirmation from the hotel to which you have been assigned.

To make any inquiries regarding hotel reservations, please observe the following procedure:

Prior to Receiving a Confirmation: Phone the Housing Supervisor of the Montreal Convention and Visitors Bureau. (514-871-1129).

After Receiving a Confirmation: Phone the Reservations Dept. of the hotel. (Phone numbers are given in this brochure).

All reservations will be held until 6:00 p.m. and none can be guaranteed after September 12.

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

CHOICE OF HOTELS:
1st
2nd
3rd
4th

NAMES AND ADDRESSES OF ROOM OCCUPANTS AND DATES OF ARRIVAL/DEPARTURE

Type of Room	Name	Address	Dates Arrive	Dates Depart

Please Type Additional Reservations on a Separate Sheet and Attach to This Form

SEND CONFIRMATION FOR ALL RESERVATIONS TO:

Name______________________________

Company______________________________

Address______________________________

City________________ State or Province________________

Country________________ Mailing Code________________

Note: Requests for accommodations at either the Queen Elizabeth or the Bonaventure will be limited to five rooms per company. A parlor counts as one room.

MONTRÉAL

THE CITY

There's no place like Montreal. Here you'll discover a blend of the best of French and English, of European and North American ways. Montreal is the second largest French-speaking city in the world, and wholly cosmopolitan.

The old and new co-exists comfortably in Montreal, and for all its respect for tradition, this is one of the world's best planned cities. Montreal is a major international business center, a major university center, and the largest inland port in the world. And, it is also a city of arts, home to La Place des Arts, one of the world's best-known entertainment centers.

The "City Below" is Montreal's answer to downtown congestion. Instead of building skyscrapers in the air and chaos on the ground, Montreal put into action an idea first suggested more than 500 years ago by Leonardo da Vinci—a multilevel city which separates people from traffic so that both can move freely: cars and trains through separate tunnels and free-ways, pedestrians through their own enclosed, air-conditioned streets.

The City Below stretches eight miles beneath the avenues of Montreal. Brief, silent (rubber-tires) Metro rides lead to boutiques, department stores, hotels, restaurants, theatres, art galleries and more.

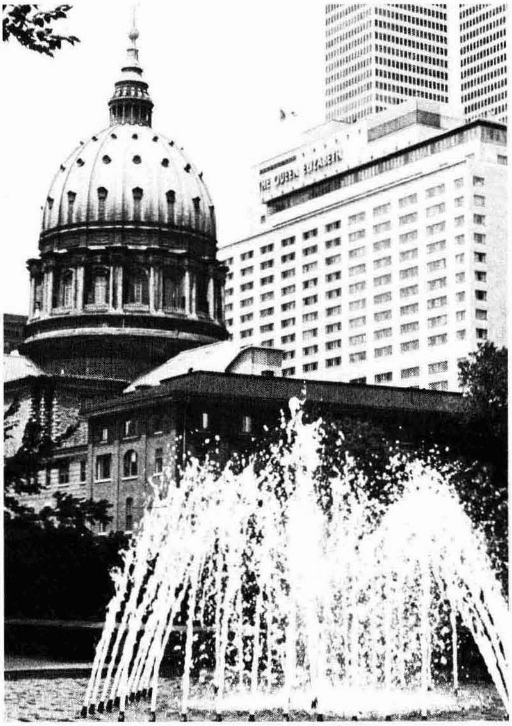
OLD MONTREAL

People frequently portray Montreal as "a little bit of Europe in North America." This description is particularly applicable to that part of the city known as "Old Montreal," the site of the early colonization of the region. The first permanent settlement of Montreal was established by the French explorer, De Maisonneuve in 1642. The early history of French Canada is preserved through the existence of many of the original structures, as well as statues and plaques commemorating the exploits of early explorers, settlers, and military figures.

A stroll through Old Montreal is a treat for even the most casual observer. Some of the oldest and most beautiful architecture in North America is to be found in this quarter.

WEATHER

During the month of October, Montreal's climate ranges from a low of 46° to a high of 57°. Medium to heavy-weight apparel will keep you comfortable. Autumn draws out the best in Mother Nature. Bring your camera to capture the "gold rush" in thick autumn foliage.



Place Bonaventure

AIR TRANSPORTATION

Two major airports serve the city: Dorval, located 16 km (10 miles) from downtown, which handles flights to and from all points in Canada and the U.S.; Mirabel, some 55 km (35 miles) from downtown, for international passenger traffic.

THINGS TO DO

Montreal's 5,000 restaurants, of which more than 100 are internationally rated, serve dishes and specialties from all corners of the earth, making Montreal a gourmet's paradise.

Exquisite French cuisine, wholesome French Canadian dishes and quick snacks are readily available throughout the city, providing quality, variety and convenience.

Vegetarians and health food enthusiasts will find that Montreal leads the way in the preparation and service of specialty foods. Kosher and seafoods also number among Montreal's specialties. The city's culinary shops and delicatessens offer meats, cheeses and other specialties from all over the world.

Montreal has justly earned its reputation as a "city of the world," where life is "a la Quebecoise," at an American pace, with a touch of European charm. It is stimulating, exciting and truly cosmopolitan.

GETTING THERE

If you are a citizen or permanent resident of the U.S., you can cross the Canadian border and return without any difficulty or delay. Passports or visas are not required. Native-born U.S. citizens should carry identification papers such as a birth, baptismal or voter's certificate, and proof of residence. Naturalized U.S. citizens should carry a naturalization certificate or other evidence of citizenship.

After 48 hours in Canada, U.S. residents may take home, duty free, $300 worth of goods for personal and household use. These must accompany the traveler. Family members traveling together may combine their personal exemptions.

PLACE BONAVENTURE

Place Bonaventure is a city within a city. It's a trade city: marketplace of the world's buyers and sellers, showplace for the merchandise of many nations and a rich source of information on trade and tourism.

It covers a six-acre multilevel site, containing not only the Exhibition Hall, where Paint Show exhibits will be on display, but a busy, bustling, boutique-filled Shopping Concourse, complete with stores, bars, restaurants, movies, entertainment, activities and services. And, atop the Place Bonaventure complex, is Hotel Bonaventure with its rooftop gardens.

1983 ADVANCE REGISTRATION

FEDERATION OF SOCIETIES FOR COATINGS TECHNOLOGY

1315 Walnut St., Philadelphia, PA 19107

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Office Use Only

Date received ____________

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Please fill out this form and mail with a check in the correct amount (made payable to the FSCT) to the Federatio address shown above. All checks must be payable in U.S. Funds, and in U.S. Banks. Any that are not will be returned Deadline date for advance registration is September 9. None will be accepted after that date.

INDUSTRY REGISTRATION FEES:

A ☐ **MEMBER** **$50.00**

Please name the Society in which you are a paid-up member:

____
Society

B ☐ **NON-MEMBER** **$65.00**

G ☐ **SPECIAL FEE FOR RETIRED MEMBERS** **$25.00**

____
Society

INFORMATION FOR REGISTRATION BADGE:

NICKNAME

FIRST NAME

LAST NAME

BUSINESS AFFILIATION

STREET

CITY

STATE *(U.S. only,*

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Model Compound For Melamine Formaldehyde Resins

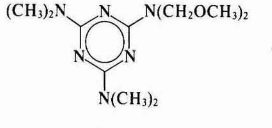
Zeno W. Wicks, Jr. and David Y.Y. Hsia[†]
North Dakota State University*

N,N-dimethoxymethyl N′,N′,N″,N″-tetramethylmelamine (I) has been synthesized as a model compound for melamine-formaldehyde resins. It is thermally stable and reacts only slowly with water or alcohol without catalyst. In the presence of catalyst and water it undergoes condensation reactions leading to the formation of a cyclic trimer. The reaction was shown to be reversible. (I) reacts rapidly with tetrahydrofurfuryl alcohol in the presence of catalyst to form the mono and then diether substitution products. Preliminary kinetic data indicate that the catalyzed reaction with alcohol does not follow either first or second order kinetics. It is possible that both S_N1 and S_N2 reactions occur.

INTRODUCTION

Melamine-formaldehyde resins are the most widely used crosslinkers in industrial baking coatings. While there have been many studies[1-13] of the reactions and possible reaction mechanisms of the resins, they have been indirect studies. Due to the high functionality and mixed compositions of the resins, it has not been possible to identify the structures or rates of formation of triazine containing products. Rather, studies have been based on changes in infrared spectra,[1,7] analysis of volatile by-products,[10,12,13] measurements of rates of cure of films, and film properties.[3] The only model compound study reported used methoxymethyl dibutyl amine as a model.[2] The difference in structure from melamine-formaldehyde resins is so great that it is not a satisfactory model.

An appropriate model compound could provide a means for increasing the understanding of reactions of the resins. After considering a variety of possibilities, N,N-dimethoxymethyl-N′,N′,N″,N″-tetramethylmelamine (I) was selected.



(I)

EXPERIMENTAL

Infrared spectra were determined with a Perkin-Elmer 137 Spectrometer. NMR spectra were determined with a Varian EM-390 MHz Spectrometer using $(CH_3)_4$ Si as internal standard. Elemental analyses were run by Galbraith Laboratories, Inc., Knoxville, TN. Mass spectra were obtained at an ionizing voltage of 70 eV using the solid sample inlet system of a MAT CH5-DF Mass Spectrometer equipped with a PDP-11 computer. Gel Permeation Chromatography experiments were done with a Waters Model 501 Unit with a differential refractometer detector using Microstyragel columns at a flow rate of 1 mL/min.

Synthesis of 2-Amino-4,6-bisdimethylaminotriazine

Initially cyanuric chloride was reacted with anhydrous ammonia.[14,15] A solution of 180 g (1 mole) of cyanuric chloride in 400 mL hot acetone was chilled in a dry ice bath to −40°C while rapidly stirring. Anhydrous ammonia was passed into the suspension at such a rate that the temperature did not exceed 0°C. After the exotherm, the reaction temperature dropped back to −40°C and addition of ammonia was stopped. The reaction mixture was poured onto crushed ice, filtered, and washed with cold acetone to remove water. The crude 1-amino-3,5-dichlorotriazine was used immediately. It was added

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slowly at 0°C into a 40% aqueous solution of dimethyl amine (4.1 moles). An exothermic reaction ensued. After the addition was complete, the reaction mixture was stirred at room temperature for five hours. The precipitate was filtered, washed with water, and then dried overnight under vacuum. The yield of crude product was 82% based on cyanuric chloride and the m.p. was 226°C. The product was recrystallized from aqueous methanol to give white crystals m.p. 229°C (lit[16] 230°C). NMR ($CDCl_3$) δ (ppm) 3.1 (s,12H,$N(CH_3)_2$), 4.8 (broad singlet, 2H,NH_2).

Synthesis of N,N-dimethoxymethyl-N′,N′,N″,N″-tetramethylmelamine (I)

A mixture of 54.6 g (0.3 mole) of 2-amino-4,6-bisdimethylaminotriazine, 243 g of 37% aqueous formaldehyde solution (3 moles) and 1.5 g of NaOH were placed in a 500 mL flask equipped with a condenser, thermometer, and stirrer. The flask was heated in a mineral oil bath while rapidly stirring the reaction mixture until a clear solution resulted. In various batches, this required a temperature of 66 to 72°C. The reaction flask was removed from the oil bath and cooled in an ice water bath. The cold solution was extracted with 400 mL of methylene chloride. The methylene chloride extract was washed twice with 500 mL of water then dried over anhydrous K_2CO_3. The methylene chloride extract (containing the dimethylolated product) was added to 272 g (8.5 moles) of methanol. The resulting solution was made acidic by adding 9.4 g of concentrated nitric acid and stirred at room temperature for 24 hours then 4.2 g of NaOH was added. The reaction mixture was extracted twice with 200 mL of methylene chloride. The methylene chloride layer was washed three times with 100 mL of water then dried over anhydrous K_2CO_3. The solvent was stripped off with vacuum. The crude yield was 92% of a product with a melting point of 35–45°C. The product was recrystallized twice from carefully dried n-heptane. Gel permeation chromatography (GPC) of a THF solution of the recrystallized product showed the presence of a small amount of dimer ($V_r = 35.1$ mL). Most reaction studies were done with this product. Some product was further purified by vacuum sublimation. The product was white crystals, m.p. 52.8-53°C. NMR ($CDCl_3$):δ ppm 3.1 (s,12H,$N(CH_3)_2$); 3.35 (s,6H,OCH_3); 5.17 (s,4H,NCH_2O), Anal. Calcd. for $C_{11}H_{22}N_6O_2$; C, 48.87; H, 8.20; N, 31.09. Found: C, 48.93; H, 8.27; N, 31.30. GPC $V_r = 37.1$ mL.

Synthesis of N,N-ditetrahydrofurfuryloxymethyl-N′,N′,N″,N″-tetramethylmelamine

2-Amino-4,6-bisdimethylaminotriazine was reacted with formaldehyde and the resulting dimethylol compound was reacted with tetrahydrofurfuryl alcohol (THFA) by a procedure identical with that used to synthesize (I). The crude product was a viscous liquid. It was not further purified. NMR ($CDCl_3$): δ ppm 1.65-2.0 (multiplet, 8H,-CH_2CH_2 in THF ring); 3.1 (s,12H,$N(CH_3)_2$); 3.5-3.6 (d,4H,-CH_2-O-CH_2); 3.6-4.2 (multiplet, 6H,-CH_2-OCH in THF ring); 5.27 (s,4H,-NCH_2O). GPC ($V_r = 35.0$ mL).

Reaction of (I) with Water

A solution of 3.19 g (1.18×10^{-2} mole) of (I) in 18.11 g of dry ethylene glycol diethyl ether (EGDE) was put in a 50 mL three-neck flask equipped with a magnetic stirrer, a condenser with a $CaCl_2$ drying tube on top, a rubber septum, and a thermometer. The solution was stirred and the flask heated in an 122°C oil bath to 117°C. Then 0.21 mL (1.18×10^{-2} mole) of water was injected through the rubber septum by syringe. An EGDE solution of *p*-toluene sulfonic acid containing 0.0273 g/mL (as determined by titration) was prepared and 1.17 mL of this solution was injected into the reaction flask. The solution was refluxed for four hours. After about one-half hour, a precipitate began to form. The amount of precipitate increased with time. After four hours, the reaction flask was cooled to room temperature. The precipitate was collected by filtration and washed with 100 mL of ethyl ether. The precipitate was slurried in 300 mL of cold ether for four hours then collected by filtration and dried. The product was shown to be 1,3,5-[tris-2-(4,6-bisdimethylamino-1,3,5-triazinyl)]-hexahydrotriazine, III, m.p. >250°C. NMR ($CDCl_3$):δ ppm 3.1 (s,36H,$N(CH_3)_2$), 5.7 (s,6H,NCH_2N). Molecular weight calcd. 582.; m/e mass spectrometry 582., IR (KBr) cm^{-1} 3000, 2850. Anal. Calcd. for $C_{24}H_{42}N_{18}$: C, 49.47; H, 7.26, N, 43.27. Found: C, 49.11; H, 7.34; N, 43.07. G.P.C. $V_r = 33.7$ mL. Yield based on (I) varied in four runs from 66–90%.

Reaction of (I) with Tetrahydrofurfuryl Alcohol

Under conditions similar to those given for the reaction with water, (I) was reacted with 2 moles of THFA in the presence of p-TSA. Samples were withdrawn periodically by syringe, weighed, neutralized with NaOH, and diluted volumetrically to make a 1% solution in tetrahydrofuran (THF). The THF solutions were used for GPC analysis. Even samples taken one minute after injection of the p-TSA solution into the refluxing reaction mixture showed a second GPC peak with $V_r = 36.1$ mL which is appropriate for the mono-THFA mono-methyl ether. Within 15 minutes a further peak appeared at $V_r = 35.1$ mL. This retention volume equals that for the di-THFA ether which was synthesized directly. NMR spectra were consistent with formation of THFA ethers.

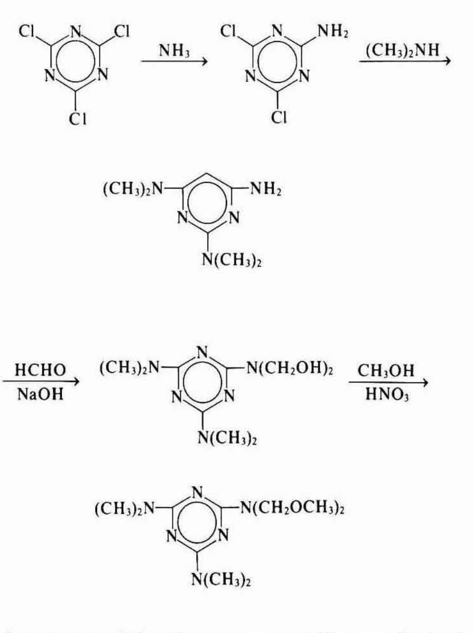
Kinetic Studies of the Reaction of (I) with THFA

A solution of 1.42 g (5.25×10^{-3} mole) of (I) 0.99 g of polystyrene ($\bar{M}_w = 2{,}100$), 1.08 g (1.05×10^{-2} mole) of THFA and 7.97 of carefully dried *p*-dioxane (total volume 11 mL) was put in a 50 mL three-neck flask equipped with a thermometer, rubber septum, condenser topped with a $CaCl_2$ drying tube, and a magnetic stirrer. The flask was heated in an oil bath to 88 ± 0.5°C. An initial sample of 0.25 mL was taken immediately before injecting 0.076 mL of a 0.0309 g/mL p-TSA solution in *p*-dioxane. Further samples were taken periodically. The samples were immediately cooled, weighed, and diluted with sufficient THF to make a 1% solution. Samples were injected into the GPC. Heights of the peak of (I) at $V_r = 37.2$ mL were normalized relative to the peak height of the polystyrene internal standard and to the peak height of the zero time sample after correcting for the volume of p-TSA solution injected. Since when one mole of (I) disappears, two equivalents of $-CH_2OCH_3$ disappear, the figures for disappearance of (I) were divided by two.

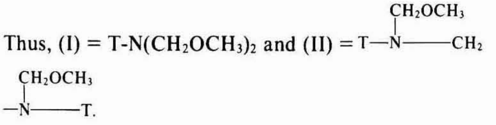
RESULTS AND DISCUSSION

Model Compound Synthesis

Cyanuric chloride was reacted at low temperature with one mole of ammonia followed by the reaction with two moles of dimethylamine then successively with formaldehyde and methanol. After two recrystallizations from n-heptane



and vacuum sublimation, very pure (I) was obtained. Since the yield of this highly purified product was low, much of the work was done with once recrystallized material. This contained a small amount of dimer (II). For simplicity in later structures, the tetramethyldiaminotriazinyl group will be designated with the symbol T.

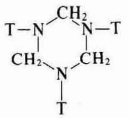

Thus, (I) = $T\text{-}N(CH_2OCH_3)_2$ and (II) $= T{-}\underset{}{\overset{CH_2OCH_3}{\overset{|}{N}}}{-}CH_2{-}\overset{CH_2OCH_3}{\overset{|}{N}}{-}T.$

Thermal Stability

A solution of (I) in xylene was refluxed (138°C) for 34 hours. No reaction occurred.

Reaction with Water

When a solution of (I) containing an equimolar amount of water was refluxed for three hours, the peak height of the GPC peak of (I) was reduced about 5%. On the other hand, when a solution of (I) in ethylene glycol diethyl ether (EGDE) with an equimolar amount of water and 1% (based on weight of (I)) of p-toluenesulfonic acid (p-TSA) was refluxed (122°C), a precipitate slowly formed. After four hours, the precipitate was collected by filtration. It was found to be the cyclic trimer (III). The structure of (III) was established by NMR and elemental analyses. The molecular ion in mass spectrometry occurred at m/e = 582 which agrees with the molecular weight of trimer (III). In repeat runs, the yield of (III) varied from 66 to 90% based on (I).



(III)

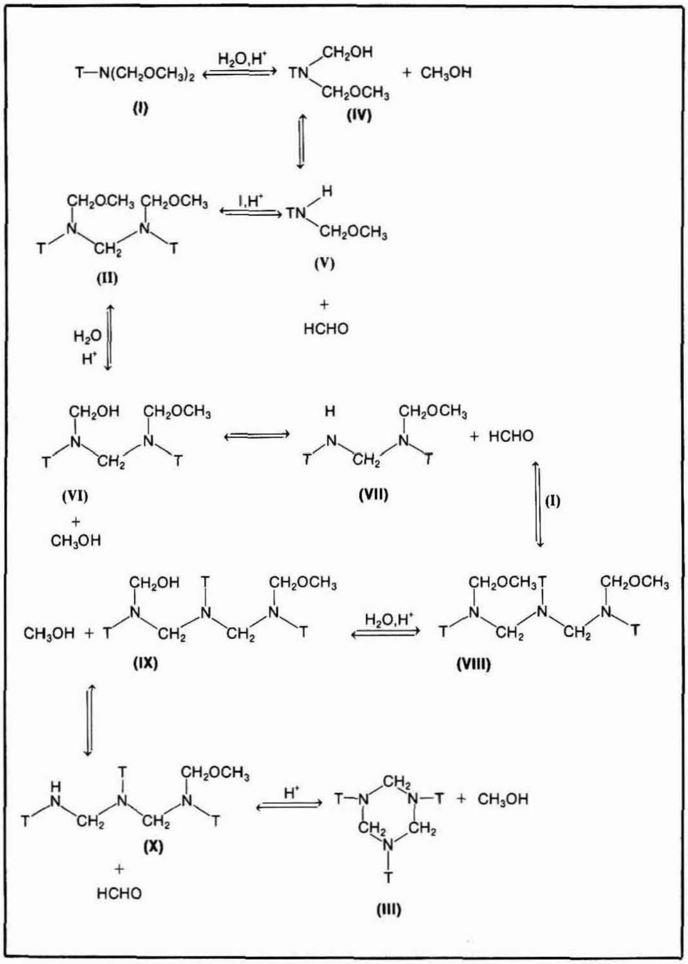
Formation of such a cyclic trimer structure has not been previously reported from melamine-formaldehyde resins. Marvel, et al.[17] suggested that cyclic trimers were formed in the reaction of urea with formaldehyde, but the evidence was indirect.

Samples at intermediate times were analyzed by GPC and NMR. As would be expected the products were mixtures. Even immediately after adding p-TSA to the refluxing solution of (I) in EGDE and water, GPC analyses showed a decrease in (I) ($V_r = 37.2$ mL) and appearance of a new peak at $V_r = 35.1$ mL. This retention volume is approximately what would be expected for dimer (II). As time proceeded, the concentration of (I) continued to fall and a new broad peak with shorter retention volume ($V_r = 32.1$ to 34.1 mL) grew. The retention volume of cyclic trimer (III) is 33.7 mL. This broad peak could include cyclic and linear trimer and higher oligomers. The NMR spectrum of (I) shows sharp singlets at δ ppm = 3.1 ($N\text{-}(CH_3)_2$); 3.35 ($-O-CH_3$); and 5.17 ($-N-CH_2-O-$). Cyclic trimer (III) has only sharp singlets at δ ppm = 3.1 ($-N-(CH_3)_2$) and 5.7 (cyclic-N-CH_2). The early samples from the reaction with water showed a new peak at 5.28 ppm. The ratio of protons at 5.17 and at 3.35 to those at 3.1 dropped as the reaction proceeded. This is consistent with formation of dimer (II) (and higher linear oligomers).

A possible reaction pathway for the formation of cyclic trimer (III) is shown in Scheme 1. Formation of a six-membered ring would favor conversion of linear trimer (X) to cyclic trimer (III) rather than continued oligomerization.

If the initial products, (IV) and (V), were present in significant concentrations, one would expect peaks (or shoulders) to appear in the GPC at larger retention volumes than that of (I). Even in the beginning of the reaction, no such peaks or shoulders were observed. (IV) and (V) would be expected to be highly reactive and hence, present only in low concentrations.

When the amount of water was decreased to 0.2 moles per mole of (I), the amount of (III) recovered after four



Scheme I

hours refluxing decreased to a 26% yield. If the calculation is based on water instead of model compound, the yield of trimer was 130%. Condensation of methoxymethylmelamine resins with elimination of methylal has frequently been proposed in the literature. Conceivably this reaction also occurred in addition to the reactions proposed. An alternative

$$2-\overset{|}{N}-CH_2OCH_3 \longrightarrow -\overset{|}{N}-CH_2-\overset{|}{N}- + (CH_3O)_2CH_2$$

explanation is that additional water and methylal can be formed by the reaction of the methanol and formaldehyde which are being generated. This water could then hydrolyze (I) leading to formation of additional trimer. Even when no water

$$HCHO + 2\ CH_3OH \overset{H^+}{\rightleftharpoons} (CH_3O)_2CH_2 + H_2O$$

was added to the reaction, after four hours, a 14% yield of trimer was isolated. While this may indicate formation of $-\overset{|}{N}-CH_2-N-$ bonds without water, removing all of the water from hygroscopic materials such as are involved in this reaction and preventing any from entering the reaction would be difficult if not impossible.

In Scheme 1, all reactions are indicated to be reversible. It has been assumed that formation of $-\overset{|}{N}-CH_2-N-$ bonds is not reversible, but no experimental evidence has been provided.[11] To test the reversibility, a suspension of 0.55 g (0.94×10^{-3} mole) of pure cyclic trimer (III) in 12 mL of EGDE and 0.77 g (0.24×10^{-1} mole) of methanol and 0.34 g (0.11×10^{-1} mole) of paraformaldehyde with 0.0055 g of p-TSA was refluxed for four hours. GPC analysis of the reaction product showed peaks at the same retention volumes as (I) and (II). The amount of conversion was small but a slow reaction rate would at least partly be due to the low solubility of (III) even in hot EGDE.

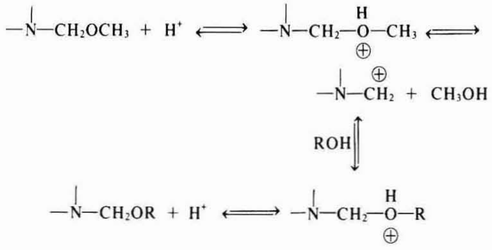
Reaction of (I) with Tetrahydrofurfuryl Alcohol

Tetrahydrofurfuryl alcohol (THFA) was selected as a model alcohol because of its distinctive NMR spectrum and because the change in molecular weight would be expected to permit separation of the mono-THFA monomethyl ether from the dimethyl ether (I).

In the absence of catalyst, no reaction between (I) and equal equivalents of THFA occurred in three hours at 100°C. On the other hand, in the presence of 1% (based on weight of (I)) p-TSA, (I) reacts rapidly with THFA. Based on GPC retention volume and NMR, the initial product is the mono-THFA monomethyl ether, but after only short reaction times the di-THFA ether also forms (identification of the di-THFA ether was based on GPC retention volume of directly synthesized di-THFA ether). Slowly a lower retention volume fraction appeared, showing that oligomers were also forming.

Kinetics of Reactions of (I) with Alcohol

It has been proposed that the specific acid catalyzed reactions of melamine-formaldehyde resins proceed via a carbonium ion.[5,11]

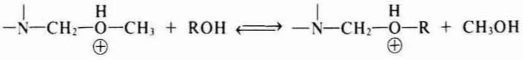


$$-\overset{|}{N}-CH_2OCH_3 + H^+ \rightleftharpoons -\overset{|}{N}-CH_2-\underset{\oplus}{\overset{H}{O}}-CH_3 \rightleftharpoons -\overset{|}{N}-\overset{\oplus}{C}H_2 + CH_3OH$$

$$\Big\Updownarrow \text{ROH}$$

$$-\overset{|}{N}-CH_2OR + H^+ \rightleftharpoons -\overset{|}{N}-CH_2-\underset{\oplus}{\overset{H}{O}}-R$$

Holmberg[9] pointed out that this S_N1 mechanism predicts that the rate of crosslinking would be independent of -OH concentration and structure. However, it has been shown that the rate of cure of films depends on -OH concentration[9] and that primary alcohols react more rapidly than secondary alcohols.[6] Holmberg[9] suggested that the reaction must proceed by a S_N2 mechanism.



$$-\overset{|}{N}-CH_2-\underset{\oplus}{\overset{H}{O}}-CH_3 + ROH \rightleftharpoons -\overset{|}{N}-CH_2-\underset{\oplus}{\overset{H}{O}}-R + CH_3OH$$

By following the disappearance of model compound (I) with different concentrations of alcohol, it should be possible to distinguish between these two mechanisms. The disappearance of (I) was followed as a function of time by GPC using polystyrene ($\overline{M}_w$ = 2,100) as an internal standard. The reaction of (I) with THFA in the presence of 1% p-TSA at 122°C proceeded too rapidly to follow the initial rate of reaction. Therefore, studies were done in *p*-dioxane at 88°C. The solution of (I) and THFA in *p*-dioxane was heated to 88°C then a solution of p-TSA was injected. Time zero was set as the time of catalyst injection. The reaction was studied with equal equivalents of THFA and (I) (that is, with 2 moles of THFA per mole of (I)) and with three equivalents of THFA per mole of (I) (that is, 6 moles of THFA per mole of (I)). The disappearance of (I) was followed with time. Since when 1 mole of THFA reacts with 1 mole of (I), two equivalents of (I) disappear, the disappearance data were divided by two. Data shown in *Table* 1 is the average of two runs where A_0 = initial concentration of (I) and A is concentration at time t.

Table 1—Disappearance ($1-A/A_0$) of (I) as a Function of Time For 1:1 and 1:3 Equivalents of (I) to THFA

Conc. in equiv/Liter	1:1	1:3	1:3/1:1
[I]	0.91	0.91	
[THFA]	0.91	2.74	
[p-TSA]	0.0071	0.0071	
Time (min)			
2	0.039	0.057	1.46
5	0.065	0.110	1.69
10	0.101	0.157	1.55
15	0.128	0.199	1.55
20	0.154	0.226	1.47
25	0.175	0.258	1.47
30	0.190	0.280	1.47

If the reaction were first order, the ratio of 1:3/1:1 would be 1 and if the reaction were second order, the ratio would be 3. As can be seen in *Table* 1, the ratio is approximately 1.5 at all time intervals. The extreme ratios comparing individual pairs of data points at all times were 1.38 to 1.83. Perhaps both S_N1 and S_N2 reactions are occurring. This would lead to an intermediate dependence on concentration. However, the ratio of fractions of (I) disappearing would then be expected to change with time.

The kinetics of the system are too complex to understand based on only disappearance of one reactant. The reactions are reversible, the rate of reaction of the second THFA molecule may be different from the rate with the first molecule, self condensation reactions may be occurring especially since the presence of traces of water is likely even though attempts were made to avoid it. Reasonable understanding of the system would at least require determination of the rate of disappearance of THFA, of the rate of appearance of the mono-THFA and di-THFA ethers in addition to the rate of disappearance of model compound. This was found to be infeasible by GPC analysis.

SUMMARY

N,N-dimethyoxy-N′,N′,N″,N″-tetramethylmelamine has been synthesized as a model compound for studying the reactions of melamine-formaldehyde resins. The model was found to be thermally stable showing no reaction when refluxed in xylene (138°C) for 34 hours. It reacted only very slowly with water or tetrahydrofurfuryl alcohol at 100°C in the absence of catalyst. With p-TSA and water, self condensation reactions occurred leading to the formation of a cyclic trimer. The self condensation reaction was found to be reversible. With THFA in the presence of p-TSA, the model compound reacted rapidly to form first, the mono-THFA monomethyl ether, then the di-THFA ether. Preliminary kinetic data suggests that the reaction is neither first nor second order but

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DAVID YINGYANG HSIA received a B.S. Degree in Chemistry from National Chung Hsing University in 1974 and a M.S. Degree in Polymers and Coatings from North Dakota State University in 1982. He then joined the Automobile OEM Department of United Technologies—Inmont as a Development Chemist. At present, he is involved in Research and Development in Industrial Protective Coatings.

perhaps reacts by both S_N1 and S_N2 mechanisms. GPC does not permit one to follow all the factors necessary for a more complete study of kinetics and mechanisms. In view of the commercial importance of reactions of melamine-formaldehyde resins, it is hoped that others will carry on studies of the reactions of what appears to be an appropriate model compound.

ACKNOWLEDGMENT

The financial support of the Fabrics and Finishes Department of E. I. duPont de Nemours is gratefully acknowledged. The authors also wish to thank Dr. S. Peter Pappas and Dr. Werner Zimmt for their suggestions and advice.

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Some Aspects Of the Graft Polymerization of Methacrylates Onto Carbon Black Surfaces by Butyllithium

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Graft polymerization of methyl and butyl methacrylates onto carbon black surfaces was investigated using butyllithium or carbon black-butyllithium complex as an initiator. Methyl methacrylate differed from styrene in that it had faster initiation and negligible butyllithium-carbon black interaction. Presence of an HAF black treated with sodium borohydride gave a homopolymer with lower intrinsic viscosity than that with an untreated black. Methacrylate grafted onto a color black by the carbon black-butyllithium complex resulted in a gel containing carbon particles. When an excess of butyllithium beyond that required for complex formation was used, there was a decrease in gel fraction as well as in grafting ratio. A mechanism for gelation is proposed.

INTRODUCTION

Functional groups, such as quinonic oxygen, hydroxyl and carboxyl groups, on carbon black surfaces, are reportedly known to react with butyllithium to give an active complex.[1,2] This complex can initiate graft polymerization of monomers with a positive e value, such as acrylonitrile and methyl methacrylate, onto carbon black. In the case of monomers with a negative e value, such as styrene and isoprene,[2,3] the complex does not function as an initiator.

When the same procedure was applied to a color black, the grafting ratio surpassed 200% and the resulting product formed a stable dispersion in organic solvents such as acetone. However, under certain reaction conditions, more than 50% of resulting polymer was found to be a gel containing carbon black. The presence of a gel would be a disadvantage in the use of the material as a black pigment for plastics or resins. Therefore, the reaction conditions needed to avoid gelation and the mechanism of gelation were investigated.

EXPERIMENTAL

Carbon Black and Reagent

FW-200 was chosen as a model color black, and an HAF black, Philblack O (N-330), was used for comparison purposes. Their characteristics were previously described.[2,3] A 15% hexane solution of butyllithium (Wako Pure Chemical Industries, Ltd.) was used as the initiator, and its effective concentration was checked by double titration.[4,5] The method of purification of monomers and solvents has been described.[2,3]

Degree of Grafting and Gel Fraction

Polymerization of vinyl monomers in the presence of carbon blacks with butyllithium, preparation of a complex by the reaction of carbon black with butyllithium, and the condition of graft polymerization utilizing the complex have been reported.[2,3] All reactions were carried out in nitrogen atmosphere at 0°C. The only difference from previous studies[6] was the method of determining grafting ratio.

As previously reported,[6] when free radical initiators are used to graft-polymerize monomers onto carbon black, the resulting dispersions are not stable when

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This article was translated from *Shikizai Kyokaishi, 54,* No. 5, 263–268 (1981), and is reprinted here by permission of the Japan Society of Colour Material.

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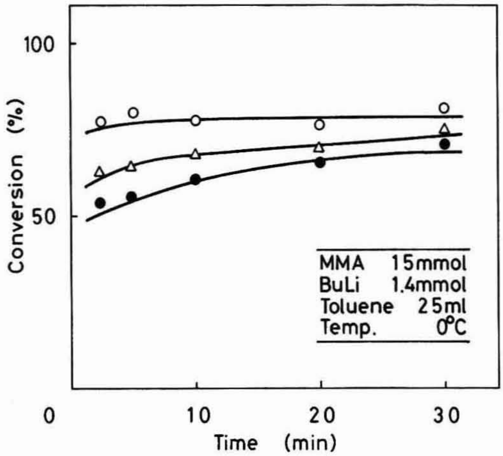

Figure 1—Polymerization of methyl methacrylate (MMA) catalyzed by n-butyllithium (BuLi): ○, in the absence of carbon black; ●, in the presence of Philblack 0 (0.6 g); △, in the presence of Philblack 0 (0.6 g) treated with sodium borohydride solution

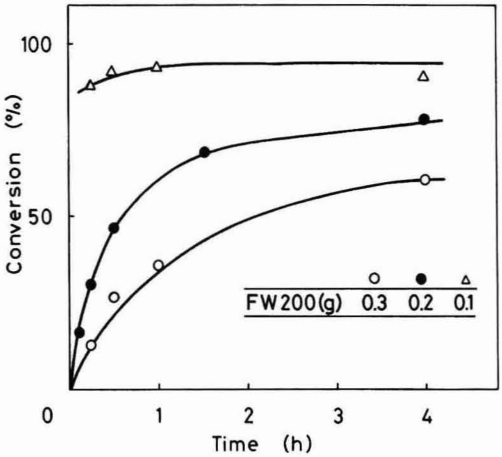

Figure 3—Anionic polymerization of methyl methacrylate (MMA) by carbon black - n-butyllithium (CB-BuLi) complexes: Conditions - MMA, 15 mmol; BuLi used for the preparation of CB-BuLi complexes, 1.4 mmol; toluene, 15 mL; temperature, 0° C

thermally dried. This tendency was greater when the amount of homopolymer formed was smaller. The separation of homopolymer from carbon black-graft-polymer was effected by Soxhelt extraction using a filter, such as Toyo Filter Paper No. 84. However, when methyl methacrylate or n-butyl methacrylate was graft polymerized onto a carbon black by a carbon black-butyllithium complex, the resulting product formed a stable dispersion in organic solvents such as acetone and tetrahydrofuran. These dispersions readily passed through filter paper leaving a carbon black-containing gel on the filter. From weight differences, the amount of gel or gel fraction was determined. Since both filtrate and gel contained carbon black, the calculated gel fraction is an apparent value.

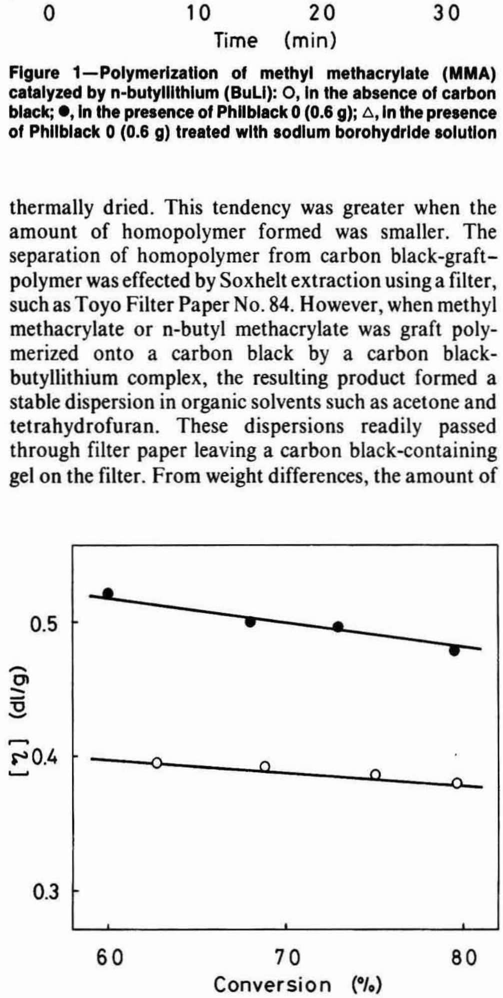

Figure 2—Change of intrinsic viscosity of homopolymer with conversion (%): ●, formed in the presence of Philblack 0 (0.6 g); ○, formed in the presence of Philblack 0 (0.6 g) treated with sodium borohydride solution. Polymerization recipes were given in *Figure* 1

In this investigation, control samples of homopolymers free from gel and carbon black grafted with polymers, such as poly(methyl methacrylate), were prepared to determine the conditions that preclude formation of a gel-like product[2] when heating. It was found that by drying reaction products at 50 ~ 60° C for 24 hours in an infrared oven, it was possible to separate homopolymer by Soxhlet extraction using benzene as solvent and Toyo Filter Paper No. 84. Extractions were carried out for periods as long as 100 hours. No carbon black particles were found in the filtrate, and the homopolymer was recovered by precipitation with methanol. The grafting ratio was calculated from the difference between the weights of total polymer and homopolymer formed and the weight of carbon black used.

RESULTS AND DISCUSSION

Polymerization of Methyl Methacrylate In the Presence of Carbon Black

When a hexane solution of butyllithium is added to a mixture of carbon black and a vinyl monomer under agitation, butyllithium is consumed by reaction with functional groups on the carbon black surface. When styrene is used graft polymerization is hindered. When methyl methacrylate or n-butyl methacrylate is used, polymerization takes place in preference to the reaction of butyllithium with the carbon black surface.[2,3]

The effect of Philblack O untreated and treated with sodium borohydride to convert quinonic functions to hydroxyl groups on the polymerization of methyl methacrylate is described in *Figure* 1. In this study, the

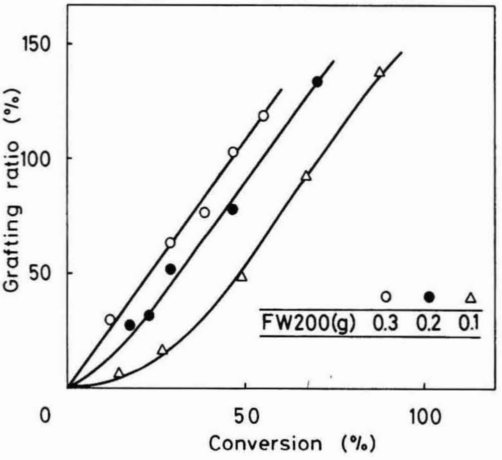

Figure 4—Dependence of grafting ratio on conversion (%): Conditions - methyl methacrylate, 15 mmol; n-butyllithium used to react with carbon black surface, 1.4 mmol; toluene, 15 mL; temperature, 0° C. Percent conversion vs time (h) curves were given in *Figure* 3

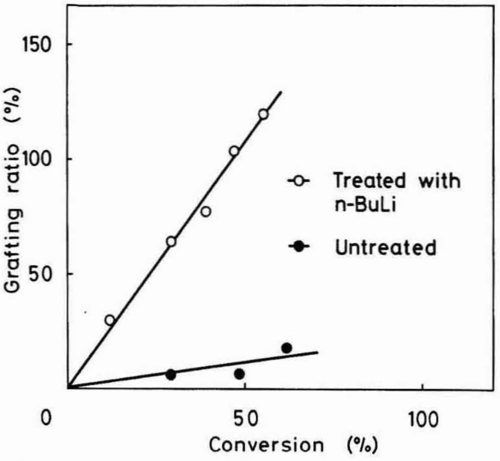

Figure 5—Percent conversion vs grafting ratio: Conditions—methyl methacrylate (MMA), 15 mmol; FW-200, 0.3 g; n-butyllithium (BuLi) used for the treatment of carbon black surface (○) or added to the mixture of carbon black and MMA as catalyst (●), 1.4 mmol; toluene, 15 mL; temperature, 0° C

method of Studebaker[7,8] for reduction of surface groups was used, and as previously reported,[3] hydrogen uptake was 0.019 wt% for Philblack O. Hydrogen consumption varies depending on the type of quinonic oxygen,[3] but approximately 1.14×10^{20}/g of oxygen containing groups were to be reduced. On the other hand, there are 0.77×10^{20}/g of hydroxyl groups originally present according to Puri, et al.[9] From these the total number of hydroxyl groups on reduced carbon black surfaces can be estimated to be approximately 1.9×10^{20}/g. The interaction of butyllithium or polymer anion with carboxyl groups can be ignored, since no appreciable amount of carboxyl groups are present on this carbon black.[10,11]

From the data in *Figure* 1, it is apparent that the conversion of methyl methacrylate is lower than 100% even in the system that did not contain carbon black. This is probably due to formation of the methyl ester of α-methylheptanoic acid and its dimer and of dibutylisopropenyl carbinol from the reaction of methyl methacrylate with butyllithium as pointed out by Kawabata, et al.[12]

Since no gel containing carbon black was formed under the conditions given in *Figure* 1, a tetrahydrofuran dispersion of the reaction product was subjected to centrifugation at 10^4 rpm for four hours. Methanol was then added to the supernatant solution to precipitate homopolymer. It was purified by reprecipitation and dried at 50° C under reduced pressure. Intrinsic viscosity of the homopolymer was determined in chloroform solution at 25° C by means of a Ubbelohde viscometer. Intrinsic viscosity of homopolymers obtained from both experiments with treated and untreated carbon black is plotted against conversion in *Figure* 2.

The data in *Figure* 2 indicate that carbon black with a high concentration of surface hydroxyl groups tends to decrease the degree of polymerization of methyl methacrylate. However, as described in *Figure* 1, such a carbon black has less retardation of polymerization than an untreated carbon black.

When styrene is used the reduction of surface groups affects neither the rate nor the degree of polymerization.[3] This is in contrast to the effect shown with methyl methacrylate or n-butyl methacrylate. But the rate of polymerization is slower and the intrinsic viscosity, i.e., the degree of polymerization, of homopolymer is larger than those without carbon black. These results can be accounted for by the lowering in catalyst concentration

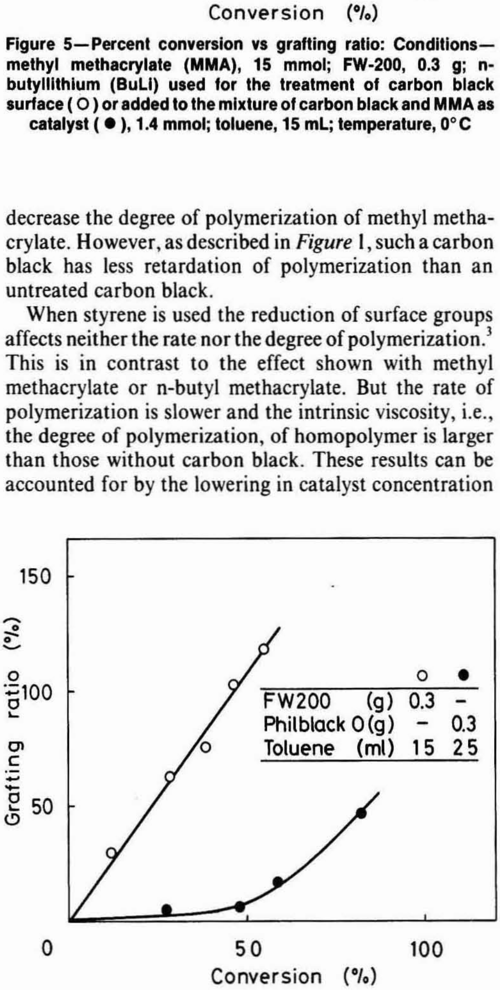

Figure 6—Dependence of grafting ratio on conversion (%): Conditions - methyl methacrylate, 15 mmol; n-butyllithium used for the preparation of carbon black - butyllithium complex, 1.4 mmol; temperature, 0° C

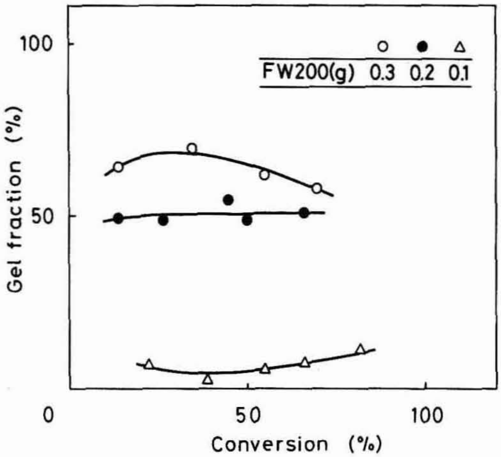

Figure 7—Percent conversion vs gel fraction: Conditions—methyl methacrylate, 15 mmol; n-butyllithium used for the reaction with carbon black surface, 1.4 mmol; toluene, 15 mL; temperature, 0° C

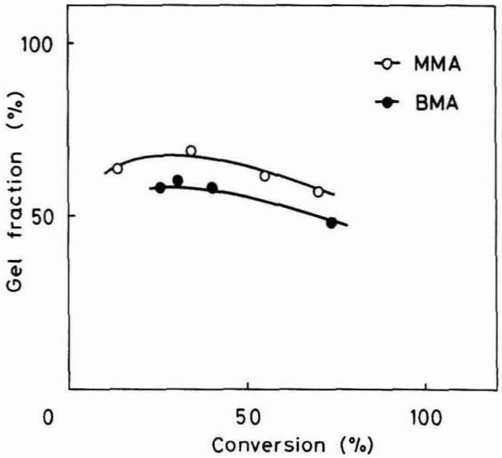

Figure 8—Percent conversion - gel fraction curves: Conditions - methyl methacrylate (MMA) or n-butyl methacrylate (BMA), 15 mmol; FW-200, 0.3g; n-butyllithium used for the preparation of carbon black-butyllithium complex, 1.4 mmol; toluene, 15 mL; temperature, 0° C

as the result of the reaction of butyllithium with oxygen-containing groups on the surface of carbon black taking place in preference to the polymerization of styrene.[3] Moreover, grafting of polystyryl anions onto carbon black surfaces is hindered and the complex formed by butyllithium and carbon black also is not active in graft polymerization of styrene.[2] With methyl methacrylate, initiation of polymerization is presumed to take place in preference to the reaction of butyllithium with oxygen-containing surface groups, such as hydroxyl groups. The fact that even after the reduction of carbon black surfaces, graftpolymer is still formed[3] and that the degree of polymerization of homopolymer is lower than that from the polymerization in the presence of untreated carbon black, as in *Figure* 2. This suggests the possibility of the reaction of polymer anion with hydroxyl groups resulting grafting products or termination of polymerization.

Graft Polymerization By Carbon Black-Butyllithium Complex

Conversion as a function of time is given in *Figure* 3 for the polymerization of methyl methacrylate in the presence of 0.1 ~ 0.3 g of FW-200 treated with 1.4 mmol of butyllithium in n-hexane.[2,3] Since the amount of butyllithium used is constant in these experiments, more butyllithium is left unreacted when less carbon black is used. As a result, more homopolymer is formed, thus indicating a higher rate of polymerization. The phenomenon will be discussed when the result in *Figure* 4 is examined.

As previously reported,[2] no catalyst is left unreacted when less than 0.25 mmol of butyllithium is used for 0.1 g of FW-200. The result is confirmed by the fact that polymerization of styrene does not take place with the above-mentioned mixture.[2] Thus, when 1.4 mmol of butyllithium is used with 0.1 g of carbon black, about 1.2 mmol of the initiator is left unreacted.

The data in *Figure* 3 indicate that the rate of polymerization is higher when more unreacted butyllithium is present. The phenomenon indicates that the rate of propagation of polymer anion initiated by phenoxylithium on the surface is much lower than the rate of polymerization of methyl methacrylate initiated by butyllithium. This is clear when the result are compared with those in *Figure* 1 with a different time scale.

Conversion Versus Grafting Ratio

The conditions of polymerization are the same both in *Figure* 3 and *Figure* 4. Studying the relationship between conversions and grafting ratio, the latter is found to be lower at a given conversion when larger amounts of butyllithium are left unreacted. That also means a lower degree of grafting is obtained when more homopolymer is formed.

In *Figure* 5, FW-200 treated with butyllithium is compared to untreated carbon black. In this experiment, 0.3 g of FW-200 treated with 1.4 mmol of butyllithium is added to methyl methacrylate without further addition of catalyst. In the experiment with untreated FW-200, 1.4 mmol of butyllithium is added to a mixture of carbon black and methyl methacrylate. The conversion is higher but there is a lower degree of grafting in the latter system. The results indicate that polymerization of methyl methacrylate takes place in preference to the reaction producing active sites such as phenoxylithium functions on carbon black.[13]

In *Figure* 6, conversion vs grafting ratio is compared using Philblack O and FW-200. Philblack O has only one-sixth the surface area of FW-200 as measured by the

BET method. It is known that 0.78 mmol of butyllithium reacts with 1 g of Philblack O.[3] Therefore, the amount of butyllithium fixed to the surface of an amount of carbon black is much smaller for Philblack O compared to FW-200, and as a result more butyllithium is left unreacted in the case of Philblack O to give a lower degree of grafting.

Conversion and Gel Content

In graft polymerizations using carbon black-butyllithium complex as the catalyst, acrylonitrile gives no gel,[2] but, on the other hand, with methyl methacrylate or butyl methacrylate, more than 50% of polymer formed is often converted into a gel containing carbon black.

In *Figure* 7, the relationship between conversion and gel fraction is described for the polymerization of methyl methacrylate when an excess amount of butyllithium over that needed to form carbon black-butyllithium complex is used under the same reaction condition as given in *Figure* 3. In this series of experiments, 1.4 mmol of butyllithium is used to treat various amounts of carbon black. Accordingly, when a smaller amount of carbon black is used, more butyllithium is left unreacted. This results in more homopolymer formation and lower gel content.

The gel formed during the polymerization swells readily in tetrahydrofuran or in acetone, but it is not dissolved even when heated. A loose network structure involving carbon black is thought to be formed by the reaction of polymer anions grown from carbon black surfaces[13] or the reaction of polymer anion with the surface of carbon black. The fact that under the reaction conditions, where a large amount of homopolymer is formed, the amount of gel tends to decrease, explaining the mechanism of gelation.

In the experiment with a small amount of carbon black, such as the one with 0.1 g of carbon black in *Figure* 7, the resulting graftpolymer passes through filter paper with homopolymer when subjected to Sohxlet extraction with tetrahydrofuran. When conversion is high, the amount of gel left in the filter after extraction is generally smaller than that of the polymer grafted onto the surface of the carbon black. The situation is reversed when conversion is low, as an apparent value including carbon black was used for the calculation of gel fraction. The experimental conditions for *Figure* 4 and *Figure* 7 are the same, but when they are compared quantitatively, there is a contradiction that in the experiment with a large amount of carbon black the difference between the amount of gel and of carbon black used is still larger than the amount of polymer grafted onto carbon black surfaces. This is a matter of reproducibility of experiments, and it indicates that with a large amount of carbon black most the graft polymer tends to form gel containing carbon black.

In *Figure* 8, methyl methacrylate and n-butyl methacrylate are compared. Ethyl methacrylate also tends to form gel in the same manner as other methacrylates.

CONCLUSION

When butyllithium is added to a mixture of carbon black and methyl methacrylate, initiation of polymerization takes place in preference to complex formation of carbon black with butyllithium. Therefore, there is a possibility of termination or grafting due to the interaction of polymer anion with oxygen-containing surface groups of carbon black. The grafting ratio is usually low in such reactions and the reaction product does not contain gel. On the other hand, in the systems in which methyl methacrylate or n-butyl methacrylate is added to carbon black-butyllithium complex, the degree of grafting is usually high and carbon black-containing gel is formed. When an excess of unreacted butyllithium is present with the carbon black-butyllithium complex, the rate of polymerization is promoted with an increase in the amount of butyllithium. In this case, formation of homopolymer is also increased while gel content and grafting ratio are both decreased.

From these results, it is clear that the degree of grafting onto carbon black surfaces can be controlled by the amount of butyllithium added in the form of complex. It is evident that the rate of graft polymerization of methyl methacrylate initiated by carbon black-butyllithium complex is much lower than those of polymerization initiated by butyllithium.

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Application of Quasielastic Laser Light Scattering For Characterization and Quality Control Of Colloidal Dispersions

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Recent applications of quasielastic laser light scattering (QELS) have demonstrated the potential of this relatively new technique for the characterization of charged colloidal particles. QELS provides a precise and rapid means for the determination of particle size and electrophoretic mobility of dilute colloidal suspensions. The information on the particle size and surface charge as obtained by QELS, makes it a valuable tool to study the hydrodynamic and electrostatic properties of synthetic polymers as they relate to the dispersion stability and rheology of water-borne coatings. Such investigations may be applied to a wide variety of systems including latexes, water-soluble polymers, micelles, microemulsions, and colloidal aggregates.

In this paper the applications of QELS to the characterization of some types of dispersions and emulsions used in the coatings industry are discussed. Specific examples include the analysis of particle size and size distribution of latex suspensions, the study of particle growth during emulsion polymerization, measurement of the degree of expansion of carboxylic latexes during neutralization, and the study of electrophoretic mobility of functional latex particles.

INTRODUCTION

The measurement of particle size and size distribution is an important component of quality control in water-borne coatings systems. Particle size and size distribution control the rheology of dispersions as well as the mechanical strength and optical quality of the final film.[1] An exhaustive review of various methods for the determination of particle size and size distribution has been reported by Collins and coworkers.[2] Microscopy, sedimentation, light scattering (dissymmetry and turbidity measurement), and electrozone sensing are among the major techniques routinely used for particle size analysis. A number of these methods require extensive calibration, long analysis time, and considerable operator skill. Furthermore, the range of size and type of particles that can be analyzed are limited for each technique. Such limitations, to a great extent, can be overcome by using the relatively new technique known as Quasielastic Laser Light Scattering (QELS).

Since its introduction over a decade ago, QELS has found widespread application in material science, biology, and physical chemistry of fluids and interfaces. The majority of applications have been concerned with the study of diffusional and translational motion of particles in dilute suspension. In contrast to conventional light scattering methods which are concerned with the average intensity of scattered light, QELS is concerned with the spectral content and the time dependent intensity fluctuations of scattered light which are driven by the Brownian motion or translational motion of the scatterers in the suspending medium.

The key parameter determined by QELS is the diffusion coefficient which can be related to the particle size. The advantages of QELS include rapid analysis time (a few minutes), precision, and wide dynamic range of particle size (i.e., 10 to 3000 nanometers). In addition, minimal sample preparation is required and only the refractive index and viscosity of dispersing media are needed. Furthermore, as will be discussed later, the instrumentation can be configured to conduct electrophoresis measurements which permits the simultaneous analysis of particle size and mobility.

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Presented in part at the 25th Technical Conference of the Cleveland Society for Coatings Technology, March 23, 1982, Berea, OH.

Glossary

A_o:	background signal level
C(t):	Intensity autocorrelation function
$\underline{D}$:	translational diffusion coefficient (cm^2/sec)
$\bar{D}$:	z-average diffusion coefficient (cm^2/sec)
D_i:	diffusion coefficient of each fraction (cm^2/sec)
$\underline{d}$:	particle size (nanometer, nm)
$\bar{d}_n$:	number average particle size (nm)
$\bar{d}_w$:	weight average particle size (nm)
$\bar{d}_z$:	z-average particle size (nm)
$\bar{d}_{QELS}$:	QELS-average particle size (nm)
d_i:	particle size of each fraction (nm)
E:	electric field (volts/cm)
G(Γ):	distribution function of decay rates
k:	Boltzmann constant
K:	scattering vector
M_i:	molecular weight of each fraction
n:	refractive index
Q:	polydispersity factor
T:	temperature (° k)
V:	particle velocity (cm/sec)
W_i:	weight fraction
$\underline{\Gamma}$:	decay rate (sec^{-1})
$\bar{\Gamma}$:	mean decay rate (sec^{-1})
η:	viscosity (poise)
λ_o:	wavelength of laser light (nm)
ϵ:	dielectric constant
μ:	electrophoretic mobility ($cm^2/volt\text{-}sec$)
μ_2:	second moment of the distribution function of decay rates
$\Delta\tau$:	oscillation period (sec)
θ:	scattering angle
ζ:	zeta potential (millivolts)

Principles of QELS and Methods of Data Analysis

Figure 1 is a schematic representation of the apparatus required for particle size analysis by QELS. A beam of light is focused onto a dilute suspension of particles and the scattered intensity is measured at some angle θ by a detector. The analysis of the intensity variations due to the diffusion of scattering particles requires the use of specific detection techniques (i.e., Optical Mixing[3]) and subsequent digital autocorrelation of the detected signal.

The theoretical basis of QELS has been discussed in a series of recent reviews on the subject.[3-5] A summary of the data analysis methods used in our laboratory is briefly discussed.

Particle Size Analysis
Monodisperse Particle Systems

The information obtainable from the experiment is C(t), the autocorrelation function of the scattered light intensity. C(t) is the product of the intensity at a given time and a delayed version of the intensity averaged as a function of delay time. For monodisperse particle systems in homodyne detection mode,[3] C(t) is a single exponential as shown in *Figure* 2a and is defined as

$$C(t) = A_o + A\exp(-2DK^2t), \tag{1}$$

where D is the Z-average translational diffusion coefficient (cm^2/sec), A_o is the background signal level, A is a constant depending on the optical design and K (cm^{-1}), the scattering vector, is an optical constant given by

$$K = \frac{4\pi n}{\lambda_o}\operatorname{Sin}\theta/2, \tag{2}$$

n is the refractive index of the medium, λ_o is the scattering wavelength in vacuum (cm), and θ is the angle of observation of the scattered light.

For a spherical particle in dilute suspension, the translational diffusion coefficient (D) can be related to the particle diameter d (cm) through the Stokes-Einstein equation[6]

$$D = \frac{kT}{3\pi\eta d}, \tag{3}$$

where η is the viscosity of suspending medium (poise), T is the temperature (° K), and k is the Boltzmann constant.

Polydisperse Particle Systems

For polydisperse particle systems, the autocorrelation function becomes a sum of exponentials. A number of methods have been proposed for data interpretation of polydisperse systems.[7-9] For continuous size distributions, the simplest approach is the method of Cumulants as suggested by Koppel.[7] The function $\ell n[C(t)]$ is fit to a second order polynomial

$$[\ell n[C(t)] - A_o]^{1/2} = -\bar{\Gamma}t + \frac{1}{2!}\mu_2 t^2 + \cdots \tag{4}$$

$$Q = \mu_2/(\bar{\Gamma})^2, \tag{5}$$

where Q is the variance of the distribution function and a measure of polydispersity, μ_2 is the second moment, and $\bar{\Gamma}$ the mean decay rate, is defined in terms of average diffusion coefficient $\bar{D}$ by

$$\bar{\Gamma} = \bar{D}K^2 \tag{6}$$

$\bar{D}$ is the Z average diffusion coefficient which can be defined in terms of weight fraction (w_i), molecular weight (M_i), and diffusion coefficient (D_i) of the individual particle components of the polydisperse system.

$$\bar{D} = \frac{\Sigma_i^m w_i M_i D_i}{\Sigma_i^m w_i M_i}. \tag{7}$$

Again, using the Stokes-Einstein relation an average particle size $\bar{d}$ is obtained by,

$$\bar{d}_{QELS} = \frac{\Sigma_i^m w_i d_i^3}{\Sigma_i^m w_i d_i^2}, \tag{8}$$

where d_i is particle size of each fraction. In general

$$\bar{d}_w < \bar{d}_{QELS} < \bar{d}_z, \tag{9}$$

where $\bar{d}_w$ and $\bar{d}_z$ are weight- and Z-average particle sizes, respectively.

For multimodal particle size distributions, the correlation function is the sum of exponentials, each with a decay rate proportional to the average diffusion coefficient of a size mode. To analyze C(t) in this case, one can use a nonlinear regression technique to extract individual diffusion coefficients, and, hence, the average particle sizes. This approach, due to the limitations in the

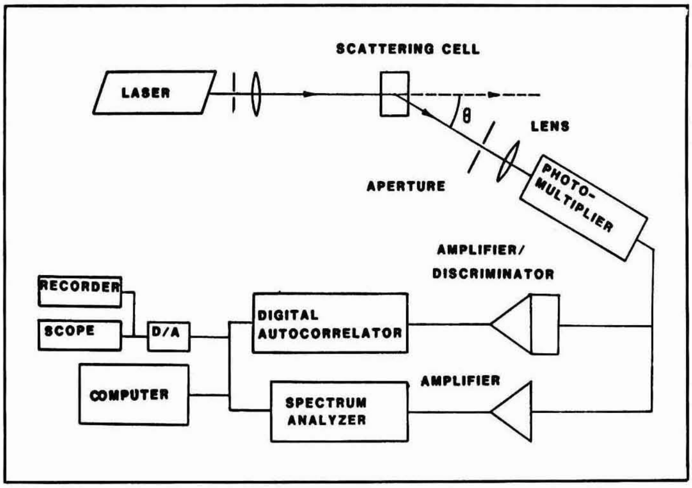

Figure 1—Schematic diagram of a quasielastic laser light scattering spectrometer

signal-to-noise ratio of C(t), often is limited to bimodal size distributions. The autocorrelation function in this case takes the following form:

$$C(t) = A_o + [A_1\exp(-2D_1K^2t) + A_2\exp(-2D_2K^2t)]^2 \qquad (10)$$

where the diffusion coefficients D_1 and D_2 are related to the average particle sizes d_1 and d_2 according to equation (3).

Recently, a more extensive study of polydisperse systems has been reported by Chu and coworkers.[9] They proposed a histogram method for analyzing the non-exponential decay profile of the correlation function. In this method, the normalized distribution function of the decay rates, $G(\Gamma)$, is approximated by an equally segmented histogram in Γ-space. This histogram is then determined so as to minimize the difference between the computed C(t) and the experimental one with respect to the histogram parameters. The histogram method is applicable to the analysis of polydispersity in systems with unimodal, bimodal, or a multimodal distribution of particle size.

Electrophoretic QELS

In the presence of an electric field, E, the scatterers move with a velocity, V. The autocorrelation function C′(t), in this case will be an exponentially damped cosine function as shown in *Figure* 2b, with a period, $\Delta\tau$, defined as[10-13]

$$\Delta\tau = \frac{2\pi}{KV\cos(\theta/2)}, \qquad (11)$$

where K is defined in equation (2) and the velocity, V (cm/sec), can be expressed in terms of the electric field, E (volt/cm), times the electrophoretic mobility, μ (cm^2/volt.sec), of the particles

$$V = \mu \cdot E \qquad (12)$$

Combining equations (2), (11), and (12) gives the working equation of the autocorrelation function

$$\Delta\tau = \frac{\lambda}{\mu E n \sin\theta} \qquad (13)$$

The quantity $2\pi/\Delta\tau$ (sec^{-1}) is known as the "Doppler shift" which is the drift in the frequency of the scattered light compared to the incident light.[10] The zeta potential ζ (volt) is then calculated from the Smoluchowski equation,[14]

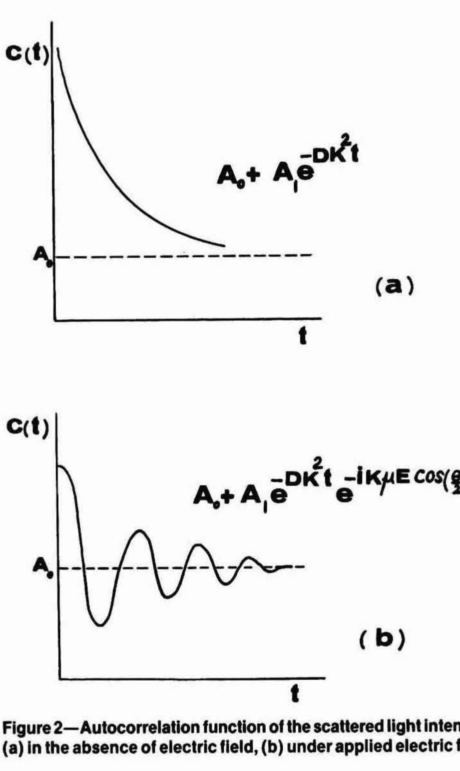

Figure 2—Autocorrelation function of the scattered light intensity (a) in the absence of electric field, (b) under applied electric field

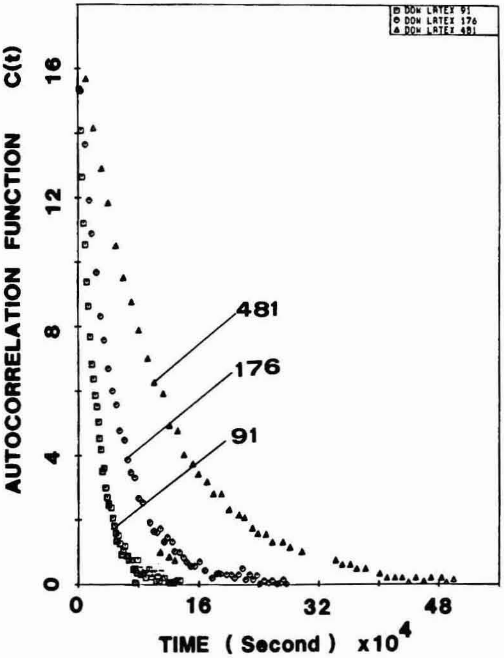

Figure 3—Composite autocorrelation functions for polystyrene latexes with different particle size

$$\zeta = \frac{4\pi\eta\mu}{\epsilon}, \quad (14)$$

where ϵ and η are the dielectric constant and viscosity of the medium, respectively. Equation 14 applies to systems with large ionic strength where the particle size is very much larger than the ionic atmosphere surrounding it. In the case of systems with small ionic strength, one should use the Huckel equation,[15]

$$\zeta = \frac{6\pi\eta\mu}{\epsilon}. \quad (15)$$

A more elaborate conversion of the electrophoretic mobility to zeta potential has been suggested by Wiersema[16] in order to account for relaxation and retardation effects arising as consequence of the ionic atmosphere.

EXPERIMENTAL

Materials

The latexes investigated include four monodisperse polystyrene latexes* with nominal sizes of 91 nm, 109 nm, 176 nm, and 481 nm, as determined by Transmission Electron microscopy by the vendor (samples A thru D).

Table 1—Particle Size Analysis of Latex Particles by QELS

Sample	Nominal Size (nm)	$\bar{D}$(×10^9 cm²/sec)	$\bar{d}$(nm)	Q
A	91[a]	47.2	93.3	0.05
B	109[a]	38.6	111	0.08
C	176[a]	23.3	185	0.06
D	481[a]	9.60	497	0.09
E	205[a]	20.0	214	0.08
F	273[a]	15.1	285	0.05
G	361[a]	11.1	386	0.07
H	—	7.95	536	0.14

(a) Measured by transmission electron microscopy (by Dow Diagnostics).
(b) Measured by turbidity analysis (Reference 20).

Also used were three monodisperse polymethyl methacrylate latexes (Samples E thru G) prepared in our laboratory by seeded emulsion polymerization techniques.

A polydisperse styrene-ethylacrylate latex containing 7% methacrylic acid was used in Electrophoretic-QELS studies.

Sample Preparation

Aqueous dispersions of latex and other systems used for QELS measurements were prepared by dilution in distilled deionized water. For particles with the size sensitive to the buffer conditions of the dispersing media and also for the case of electrophoretic mobility measurements, samples should be diluted with serum extracted from the original concentrated dispersions. Depending on the size of the particles and the cell geometry, concentrations of 2×10^{-3} to 2×10^{-2} percent (w/v) total solids were used. Acceptable concentration ranges vary according to the scattering characteristics of the sample.[17] As a guideline, polystyrene latex spheres of diameter 100 nm to 500 nm can be satisfactorily measured in the concentration range of 10^{-3}% to 10^{-2}% (w/v), (Higher concentrations correspond to the smaller particle sizes).

In general, samples should be free of dust and contaminations. However, the requirements for filtration vary with the scattering angle. For wide scattering angles (i.e., $\theta > 90$), a precision of ±3% or better can be obtained without filtration for latex samples.

The choice of scattering angle depends on the particle size and polydispersity and is determined from the Mie scattering theory.[17] For polydisperse systems, consideration must be given to the interference effects associated with the angular dependence of the scattering intensity.[18] Such effects result in the apparent small average particle size at large angles ($\theta > 40$).

Instrumentation

Quasielastic light scattering was carried out using a photon correlation spectrometer. The system consisted of a Specta-physics 120 He-Ne laser source producing 15 mW at a wavelength of 623.8 nm. The scattered light was detected by an ITT FW-130 photomultiplier (PMT).

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*Obtained from Dow Diagnostics, Indianapolis, IN.

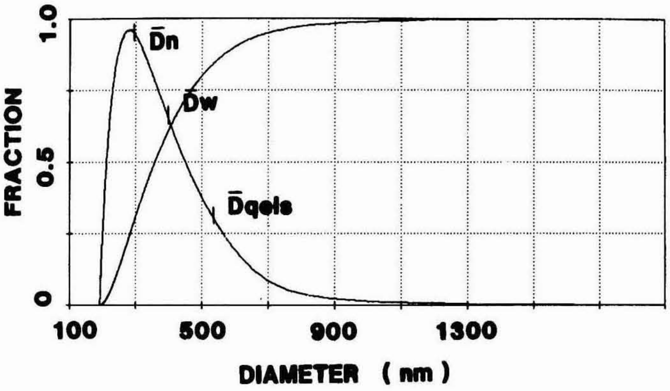

Figure 4—Weight differential and integral particle size distribution of latex (H) obtained by disc centrifuge photosedimentometery

The signal from the PMT was amplified before autocorrelation using an amplifier/discriminator, Pacific Photometric model 3262/ad4. The autocorrelation function was analyzed with a Saicor 42A, 100 channel digital autocorrelator.

Currently, more advanced digital correlators are commercially available (e.g., MALVERN model M-2000, Malvern Scientific Corp., Ronkonkoma, NY, or the LANGLEY-FORD model 1096, Langley-Ford Instruments, Amherst, MA). Such correlators provide more channels, hence, improved analytical accuracy required for characterization of polydisperse samples. A Tektronix Model 5103 oscilloscope provided the visual inspection of C(t) during the data aquisition period (typically 2 to 5 minutes). The data aquisition and reduction was performed using a MINC-11 microcomputer (Digital Equipment Corp.).

For particle size analysis, cylindrical glass cells 2.5 cm in diameter with 30 mL volume were used. For electrophoresis measurements, a sample cell, designed by Ware[19] and coworkers, was used which permits the application of the electric field to the sample. The cell can be cooled to avoid the adverse effects of Joule heating. A switching power supply (Kepco) was used which permits the application of an electric field of up to 80 volts/cm.

RESULTS AND DISCUSSIONS

Particle Size Analysis of Latexes

Figure 3 shows the composite autocorrelation functions, C(t), for three monodisperse polystyrene latex samples, with nominal particle sizes of 91 nm, 176 nm, and 481 nm. The initial slope of C(t), which is proportional to the diffusion coefficient, increases with a decrease in size. As mentioned earlier, in the case of monodisperse samples C(t) is a single exponential with a decay time constant proportional to the diffusion coefficient of the particle.

Table 1 summarizes the results of particle size analysis of a series of latex samples. The data analysis is done by the cumulant method which results in an average particle size ($\bar{d}$) and a polydispersity factor (Q). For monodisperse samples (A–G), good agreement between average size by QELS and nominal values is obtained as indicated by *Table* 1. Furthermore, the Q factor is generally less than 0.1 for monodisperse latexes. The higher the Q value the larger is the width of size distribution.

Table 1 also indicates a comparison of particle size analysis by QELS with that of turbidity analysis[20] for Samples E–G. In general, the particle sizes obtained by QELS are larger than that of turbidity analysis. The difference between the two depends on the polydispersity of the sample. This is to be expected due to the higher moment size average determined by the QELS method compared to the weight average size obtained from turbidity measurements. The differences in average size between the two methods range from 4% for small particles up to 8% for the largest particles.

Sample H is a commercial polydisperse acrylic latex used in the formulation of interior house paints. The particle size distribution for this sample, as obtained by disc centrifuge photosedimentometery (DCP),[21] is shown in *Figure* 4 which also compares the average particle size obtained by QELS (536 nm) with the calculated weight average ($\bar{d}_w = 401$ nm) and number average ($\bar{d}_n = 294$ nm) particle sizes from DCP data. Again, the large average particle size from QELS reflects the higher moment size average by this method. The Q factor of 0.14 obtained from QELS data corresponds to the polydispersity value of $\bar{d}_w / \bar{d}_n = 1.6$ calculated from disc centrifuge data.

Analysis of Bimodal Size Distribution

A mixture of two standard polystyrene latexes has been used to generate bimodal size distributions. In this case, the autocorrelation function C(t) is the sum of two exponentials [equation (10)] with decay rates corresponding to the average diffusion coefficient of each component of the mixture. With a reasonable signal-to-noise ratio in the raw data, a nonlinear regression algorithm can be used to analyze the individual decay rates from the measured correlation function.

In our study, a mixture of two standard polystyrene latexes with nominal particle sizes of 109 nm and 234 nm has been used. The concentration of the 109 nm latex in the mixture was 10 times that of 234 nm latex. The

Table 2—Particle Size Measured by QELS During Emulsion Polymerization

Reaction Time (min)	Average Particle Size (nm)
15	85.7
30	105
45	125
60	138
75	150
90	185
105	190
120	170

nonlinear regression analysis resulted in the following average particle sizes: $\bar{d}_1 = 115$ nm; $d_2 = 280$ nm.

Comparison of the results with the nominal values of the latexes present in the mixture shows reasonable agreement within the resolution of the method. However, the effectiveness of this technique to a great extent is dependent on the relative size and concentration of each component present in the mixture.

Particle Size Growth During Emulsion Polymerization

Quasielastic light scattering is particularly well suited to the monitoring of particle formation and growth throughout the course of emulsion polymerization.[22-23] The advantage of QELS over other techniques is that QELS permits study of the initial stage (particle formation) on the reaction during which small particles ($d < 100$ nm) are formed. Furthermore, the concentration of the system need not be known. This permits the possibility of real time particle size measurement, utilizing an on line flow-thru cell. Such a configuration is attractive for process control applications.

A semicontinuous emulsion polymerization of an acrylic copolymer latex was studied throughout the course of the monomer feed. Samples were taken from the batch at 15 minute intervals up to two hours of monomer feed. The samples were then diluted and the particle size was measured by QELS. The size measurements were performed at 120° scattering angle and at a temperature of 22° C. The results are illustrated in *Table* 2 and *Figure* 5. *Figure* 5 indicates that the average particle size increases, consistent with at least some growth of particles formed early in the reaction. Although the final stage of the reaction has not been studied, there is an indication of a plateau, in particle size, after about 100 minutes. Furthermore, dividing the total volume of polymer by the volume of an average particle, calculated from the average diameter, gives an estimate of the total number of particles present at a given reaction time. A more accurate estimate of the number of particles may be obtained from QELS analysis combined with the determination of the total solids for each sample.

Such analysis can be used to relate the number of particles generated during the course of polymerization to synthetic and process parameters (i.e. soap concentration, initiator level, monomer feed rate, etc.), in order to study the reaction kinetics.[24-25] In addition, one can detect anomalous behavior in particle growth such as aggregation or the generation of a large particle size mode, during the polymerization process.

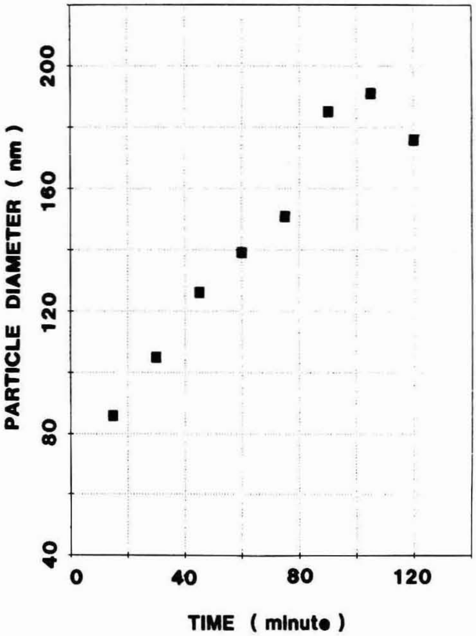

Figure 5—Average particle size vs time during emulsion polymerization

Expansion of Carboxylic Latexes Upon Neutralization

Carboxylic monomers are included in latex polymerization recipes in order to control the stability and the viscosity of latex suspensions. This is achieved by means of adjusting the subsequent neutralization level through addition of different amounts of an organic amine.

During the neutralization process the carboxyl groups concentrated near the particle surface are ionized and, therefore, impart electrostatic charge to the particle. Furthermore, the particle expands due to the unfolding of the carboxylated polymer chains, thus increasing the viscosity of the system.

The QELS technique has been used to study the expansion process of such latexes. The sample used was an acrylic latex containing about 5% methacrylic acid. An organic amine was used to neutralize the latex to various levels up to 100% neutralization. Results of this study are shown in *Figure* 6. Before neutralization the latex sample had an average size of 162 nm. As indicated in *Figure* 6, the particles initially expand up to about 60% neutralization. A maximum change in the average size of about 10% was reached at 60% neutralization. The decrease in size after the maximum point may be due to the coiling of the polymer chains at the surface, caused by the reduction of the interaction between charged polymer

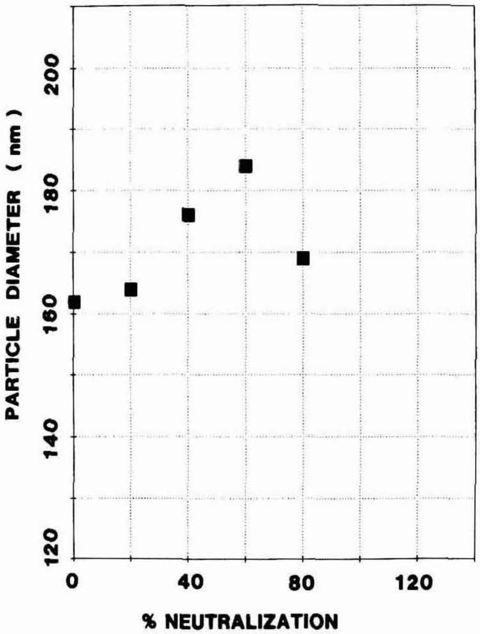

Figure 6—Expansion of a carboxylic latex upon neutralization

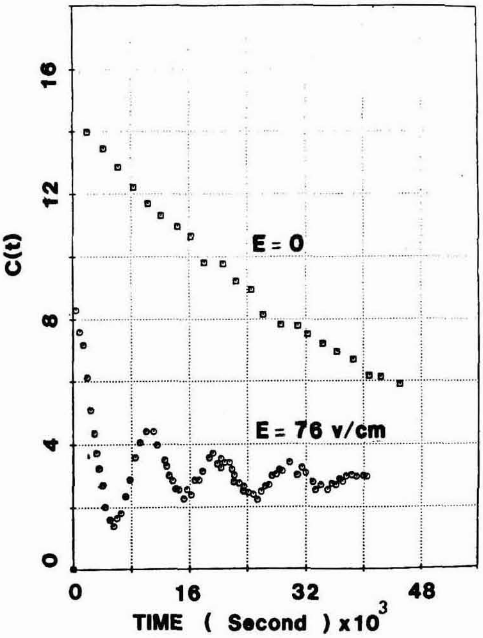

Figure 7—Autocorrelation functions obtained in electrophoretic-QELS experiment. (a) Electric field = 0, (b) Electric field = 76 volts/cm

segments. One should note that *Figure* 6 indicates only the change in the average particle size versus percent neutralization. For polydisperse systems, the degree of swelling as measured by QELS method is influenced primarily by the expansion of larger particles as indicated by equation (8). Such studies, combined with rheological analyses, permit one to study the mechanism of expansion, and the changes in morphology of latex particles upon neutralization.

Electrophoretic QELS

As mentioned earlier, an important advantage of the QELS technique is the ability to determine the electrophoretic mobility of particles.[10-13] *Figure* 7 shows the two autocorrelation functions obtained in the electrophoretic-QELS experiment. An acrylic latex containing 7% methacrylic acid was used in this experiment.[13] The heterodyne detection mode was implemented at 15° scattering angle. With no electric field (E = 0), the autocorrelation function follows a single exponential from which the latex particle size, 256 nm, was determined.

In the presence of an electric field (E = 76 volt/cm), the autocorrelation function is an exponentially damped cosine wave. The frequency of oscillation is proportional to the velocity of particles. The higher the electric field, the larger the particle velocity, hence the faster the oscillation occurs as indicated by equations (12) and (13). From the frequency of oscillations, the electrophoretic mobility and the zeta potential can be calculated. For the example shown, the frequency of oscillation was 104 sec^{-1}. From this value the electrophoretic mobility of 2.53×10^{-4} cm^2/volt-sec and the zeta potential of −48 millivolts was determined. This high negative zeta potential is well above the onset of flocculation (i.e., −20 mv),[26] which is an indication of the good stability of the latex under the experimental conditions (i.e., pH of 5.9 and specific conductivity of 1.48 micromhos/cm).

The potential of QELS compared to the classical methods, i.e., microelectrophoresis, lies in the precision and speed of analysis. The simultaneous determination of mobility and particle size available by the QELS method provides a valuable tool to study the electrostatic stability, flocculation phenomena, and to assess the degree of dispersion of particles in an aqueous system.

CONCLUSIONS

The applications of the QELS technique reported in this paper demonstrate the potential of this method as an attractive characterization tool for studying colloidal properties of dispersions. Although the focus of this article has been on latex suspensions, the capabilities of QELS as reported by other research groups cover a wide range of systems of interest to coatings industry.

The measurement of particle size and particle size distribution by QELS has been applied to a variety of systems including latexes,[32] micelles,[27-28] microemulsions,[29] and colloidal aggregates.[30] In addition, QELS has been used to study particle nucleation and growth in emulsion and dispersion polymerization,[22-25] to study

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aggregation processes,[13] and to investigate particle expansion and swelling as applies to latex functionality.[31]

The combination of electrophoresis with QELS permits the simultaneous measurement of diffusion coefficients and the electrophoretic mobilities of dispersions.[10-13] This method appears to have great potential in industrial work involving charged suspensions (such as electrocoat paint suspensions). It also can be used as a probe to study the stability and rheology of dispersions of charged colloidal particles.

Recent advances in the microelectronics and optical systems have made possible the design of a less expensive and integrated instrument readily available to the chemist as a powerful analytical tool for characterization of coatings systems.

ACKNOWLEDGMENTS

The authors wish to thank Professor Alex Jamieson, of Case Western Reserve University, for helpful comments and for providing the use of the instrumentation for this study.

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Chemistry of High Solids Alkyd/Reactive Diluent Coatings

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Pressures have increased in recent years to reduce both solvent emissions and thermal curing requirements for organic coatings. A practical response is a system based on oil modified alkyds formulated with methacrylate reactive diluents which function initially as solvents but then convert to binder. Basic requirements of a reactive diluent include low volatility, toxicity, and odor with good compatibility and stability. Dicyclopentenyloxyethyl methacrylate is a vinyl monomer that has these properties. Another requirement is rapid conversion during film formation, a process strongly opposed in thin films by oxygen inhibition of free radical polymerization. Proper coating design controls the oxygen content in the film by rapidly increasing film viscosities to slow oxygen diffusion rates and by efficiently scavenging oxygen using alkyd unsaturation. Keeping reactive diluent levels in the 15–30% range is a key part of the design. Fully complying high solids systems are achieved by further constraining the system to use low molecular weight alkyds. Model systems and practical high solids alkyd/reactive diluent coatings illustrate design principles and rationalize observed coating properties.

INTRODUCTION

In recent years enormous pressure has been placed on the coatings industry to reduce both solvent emissions and the thermal energy required for cure of organic coatings. An attractive tool to achieve this objective is the development of a reactive solvent-dilute which can function as a solvent in the formulation of the coating, but which during the cure process (preferably at ambient or force dry temperatures) is converted to an integral part of the film. Such a material must be characterized by low volatility, low toxicity, low odor, and a solubility parameter such that it is a solvent for and compatible with a wide variety of resinous film formers. It should be reasonable in cost and should enhance film build, resistance properties, and mechanical properties of high solids coatings of which it is a key component. A number of approaches to identification of a suitable reactive-diluent have been described in the literature,[1] the most interesting of which is the vinyl dioxolane system developed by the duPont Company.[2] None of these systems have had, at least to date, the necessary balance of cost/performance for commercialization. A notable exception is Nature's reactive diluent, linseed oil, which is used in a variety of ways even though it has major viscosity, compatibility, and durability limitations.

PROPERTIES OF DPOMA

An attractive route to reactive diluents should be found in properly designed vinyl monomers, since a whole body of knowledge exists for converting solvent-like vinyl monomers to polymers by free-radical polymerization processes. This approach was investigated by ICI,[3] among others, using commercially available monomers. Unfortunately, the commercial monomers are not fully satisfactory for various reasons, such as too high volatility, package instability, toxicity, extreme oxygen inhibition of cure, and poor compatibility with other resins in polymerized form. Some of the commercial monomers are re-examined in this study, but focus is on an acrylic monomer especially designed for reactive diluent use, namely, an autoxidizable methacrylate which can be cured at ambient temperature by oxidatively initiated polymerization.

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

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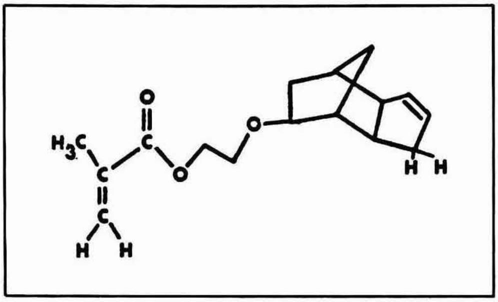


Figure 1—Structure of dicyclopentenyloxyethyl methacrylate (DPOMA)

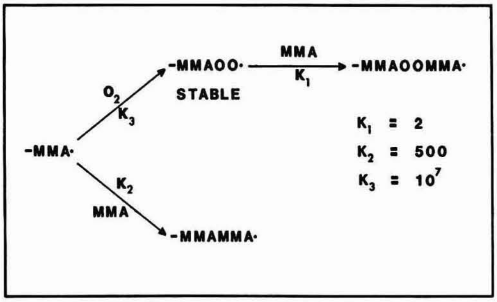


Figure 2—Important rate constants for polymerization of methyl methacrylate in presence of oxygen

Some thirty years ago, Bruson synthesized dicyclopentyl methacrylate from the addition of methacrylic acid to dicyclopentadiene.[4] He also noted that this ester, in the presence of a cobalt compound and oxygen, was converted to a hard insoluble film at ambient temperature. Recent examination of dicyclopentyl methacrylate as a reactive diluent has shown that it is of some interest in this context; however, it is too volatile for use in many systems and, even worse, it has a penetrating, persistent and atrocious odor. Accordingly, our attention was focused on dicyclopentenyloxyethyl methacrylate (*Figure* 1), which is designated as DPOMA throughout this paper.

Typical physical properties of DPOMA are summarized in *Table* 1. The monomer does have an odor but it is mild and has generally been considered inoffensive. The significant elements of *Table* 1 include the low viscosity, high boiling point, and a solubility parameter of 8.6. Accordingly, DPOMA is a good solvent for, and compatible with, most common organic film formers. In practical terms, DPOMA is not volatile under air drying cure cycles or at 180°F (82°C) for 30 min. At higher temperatures, evaporation of monomer competes with cure, but with appropriate reactive resins, it may be cured at temperatures exceeding 250°F (121°C) without noticeable monomer loss.

A very important consideration in the use of any reactive diluent is its toxicity. The acute toxicity profile is summarized in *Table* 2, suggesting that the acute toxicity of this monomer is in fact lower than many commonly used organic solvents.

CURE CHEMISTRY OF DPOMA

DPOMA contains both a polymerizable methacrylate double bond and an allylic group which can serve as a free radical source in the presence of oxygen and conventional metallic driers. It is, therefore, capable not only of homopolymerization to a solid state within coating vehicles, but also of interpolymerization with unsaturated resins that may comprise all or part of the vehicle, e.g., drying oil alkyds, fumarate polyester, polyfunctional acrylates.

The monomer is highly stable in the absence of metallic drier and active free radical sources, but it may polymerize in bulk in as little as two days in the presence of driers such as cobalt salts and adventitious oxygen. Excellent formulation stability of systems containing both DPOMA and cobalt may be achieved, however, by addition of low levels of volatile oxime, e.g., methyl ethyl ketone oxime. In thin films the oxime evaporates and normal cure is obtained. The use of oximes as antiskinning agents in alkyd paints is well known. In DPOMA systems containing cobalt, however, the function of oxime is that of a volatile stabilizer and without it these systems have limited stability. Presumably, the oxime complexes the cobalt drier and renders it inactive prior to film formation.[5]

In the presence of cobalt drier, DPOMA reacts with oxygen and in a 24–36 hour period at room temperature is converted to a hard, brittle insoluble solid (Knoop hardness number of 15). The amount of oxygen consumed per mole of DPOMA varies considerably with

Table 1—Typical Physical Properties of DPOMA

Appearance	Clear liquid
Color	100–300 APHA
Viscosity	15–19 cps (25°C)
Density	1.064 g/cm^3 (25°C)
Refractive index	1.496 (22°C)
Boiling point	350°C at 760 mm
Solubility parameter	8.6 (cal/cm^3)$^{1/2}$
Flash point (Pensky-Martens CC)	>200°F
Cured film hardness (KHN)	15
Cure shrinkage	8.7%
Glass temperature (homopolymer)	40–50°C
Inhibitor	50 ppm Hydroquinone

Table 2—DPOMA Toxicity Summary

Acute oral (rats)	$LD_{50} > 5$ g/kg ("practically non-toxic")
Acute dermal (rabbits)	$LD_{50} > 5$ g/kg ("practically non-toxic")
Eye irritation (rabbits)	Non-irritating
Skin irritation (rabbits)	Draize = 1–2
Acute inhalation (rats)	4 hr. $LC_{50} > 2.8$ mg/L ("practically non-toxic")
Ames mutagenic test	Non-mutagenic
Skin sensitization (guinea pig)	Not a sensitizer

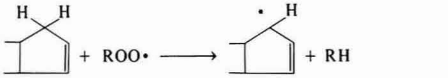
Table 3—Effect of Multifunctional Reactive Diluent on Early Dry Properties[a] and Stability

Reactive Diluent(s)	Set[a,b] Time (hrs)	Through[a,c] Dry (hrs)	Surface[a,d] Tack (hrs)	Viscosity (poise)[e,f]		
				Initial	3 Days 140°F	10 Days 140°F
20 DPMOA	1.7	3.0	>18<36	4.8	5.2	5.5
10 DPOMA/10 TMPTMA	1.5	2.4	<18	4.8	6.2	8.2
20 TMPTMA	1.5	1.8	<18	4.9	Gel	—

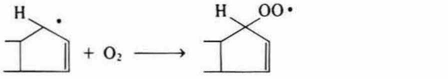
(a) 60 High solids medium soya alkyd/20 very long linseed alkyd/20 reactive diluent//0.06% cobalt/0.06% calcium/0.15% MEK oxime.
(b) Set-to-touch.
(c) Circular recorder.
(d) Zapon Tack Tester, 300 g/5 sec.
(e) ICI cone and plate Viscometer, 10^4 sec^{-1} shear rate.
(f) 20 PVC TiO_2 paints based on clears of footnote (a).
(g) ~ 1.3 mil films.

the geometry of the sample. In experiments with larger surface area/volume ratios more oxygen is taken up. In a typical thick film experiment *ca.* 6 moles of oxygen react per 100 moles of monomer.

The detailed structural chemistry of the DPOMA cure is a complex question. Almost certainly, however, the process is initiated by an adventitious radical acting on the allylic function of the monomer and this generates the chain reaction described by equations (1)-(3). Equations (4) and (5) are the typical one electron transfer reactions observed from cobalt and organic hydroperoxide.



(1)



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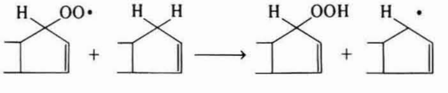

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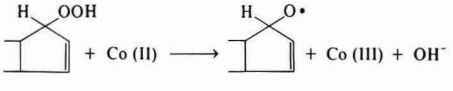

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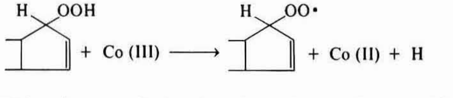
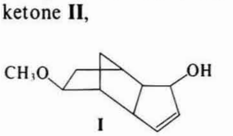
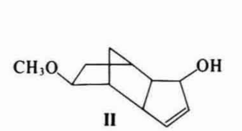


(5)

This picture of the chemistry is consistent with the autoxidation of cyclopentene which has been studied in detail.[6] It is also supported by our examination of the autoxidation of the model compound dicyclopentenyl methyl ether where the unsaturated alcohol **I** and ketone **II**,



I



II

(the expected decomposition products of the corresponding hydroperoxide) were identified as the only volatile products of the cobalt catalyzed oxidation.

The radical flux generated by this autoxidation results in polymerization of the methacrylate functionality of the monomer. The growing methacrylate radical chain is, however, intercepted by oxygen to form a terminal peroxy radical before it can attain high molecular weight. Indeed, if sufficient oxygen is present a methacrylate/oxygen alternating copolymer is the major product formed. The polymeric peroxide is remarkably stable but is obviously undesirable in terms of achieving maximum coating properties. The quantitative relationships[7] governing methacrylate polymerization in a partial pressure of oxygen are described in *Figure* 2. It is quite evident that the great reactivity of oxygen toward carbon free radicals is the dominant factor when oxygen is present.

Data obtained in our laboratories from elemental analysis, hydroperoxide titrations, and spectroscopic (nmr) evaluations have shown that in thin films at ambient temperature oxygen copolymer is the primary product. However, in thick films, at elevated temperature (60° C or higher), or in the presence of an effective oxygen scavenger such as drying oil or alkyd, methacrylate homopolymerization is the dominant process. Crosslinking also occurs during the polymerization since addition of the various radicals to the allylic unsaturation is possible.

Table 4—Effect of Soft Reactive Diluents On Early Drying Properties[a,e]

Reactive Diluent(s)	Set Time[b] (hrs)	Through Dry[c] (hrs)	Surface Tack[d] (hrs)
20 DPOMA	2.1	4.3	24
20 Stearyl Methacrylate (SMA)	3.1	5.1	>9 Days
10 DPOMA/10 SMA	2.2	4.0	2-5 Days

(a) 80 Long soya/20 reactive diluent//0.06% cobalt/0.06% calcium/0.15% MEK oxime.
(b) Set-to-touch.
(c) Circular recorder.
(d) Zapon Tack Tester, 300 g/5 sec.
(e) ~ 1.3 mil films.

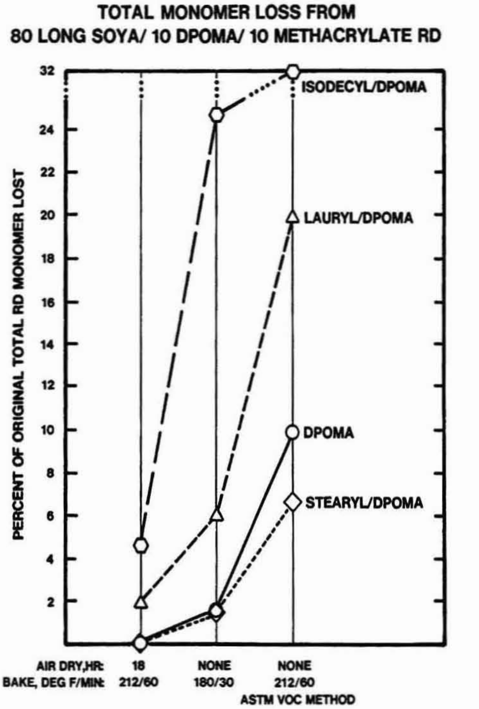


Figure 3—Volatility of DPOMA and DPOMA/RD (50/50) blends in formulation with alkyd

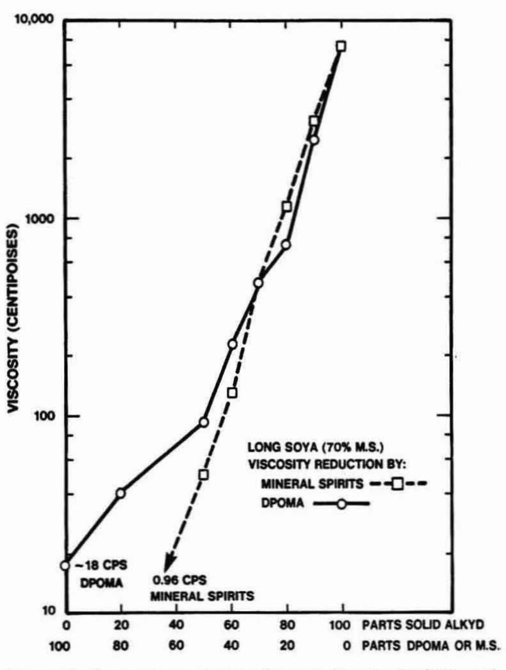


Figure 4—Comparison of viscosity reduction by DPOMA and mineral spirits. Ordinate represents replacement of alkyd solids by either DPOMA or MS

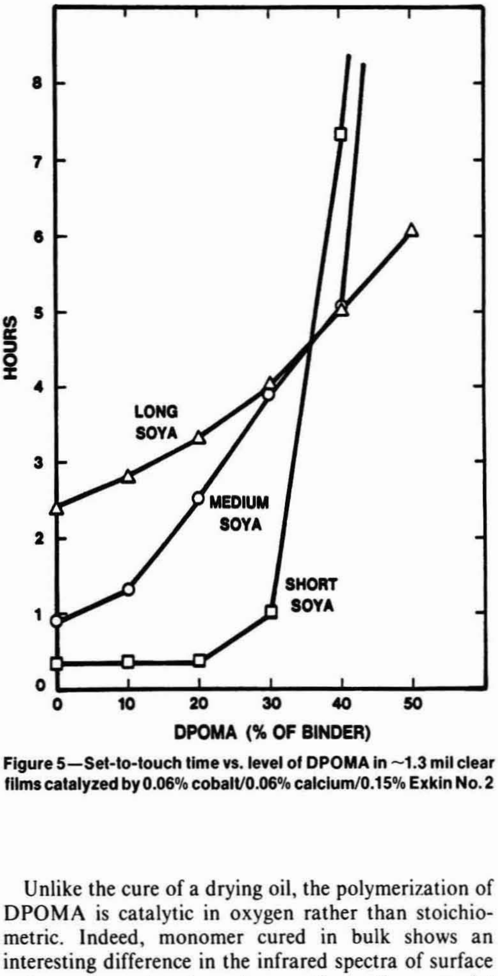


Figure 5—Set-to-touch time vs. level of DPOMA in ~1.3 mil clear films catalyzed by 0.06% cobalt/0.06% calcium/0.15% Exkin No. 2

Unlike the cure of a drying oil, the polymerization of DPOMA is catalytic in oxygen rather than stoichiometric. Indeed, monomer cured in bulk shows an interesting difference in the infrared spectra of surface polymer versus interior polymer. The surface material has absorption characteristics of hydroxyl and carbonyl groups generated by the autoxidation. Interior polymer shows the typical spectrum of DPOMA homopolymer prepared in the absence of oxygen. In practical terms, this also means that DPOMA does not have the through-dry problem characteristic of so many alkyds. Finally, it should be noted that, even though the DPOMA polymerization is initiated by oxygen in the presence of cobalt, there are indications of oxygen inhibition of cure. A film, particularly during the early stages of dry, may exhibit surface tack; however, this disappears since the autoxidation-polymerization mechanism ultimately overcomes this problem.

ALKYD/REACTIVE DILUENT

DPOMA is an excellent solvent for many coating resins, and it can, therefore, be used broadly to raise application solids. The chemistry of DPOMA obviously suggests its use as a partial solvent replacement in drying oil alkyds for air pollution compliance.

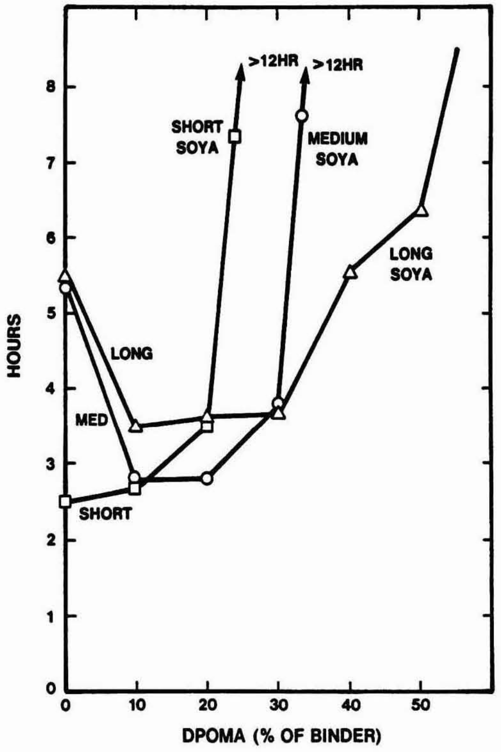


Figure 6—Through-dry vs. level of DPOMA in conventional soya alkyds for ~ 1.3 mil clear films catalyzed by 0.06% cobalt/0.06% calcium/0.15% Exkin No. 2

Drying characteristics of early experimental paints containing high DPOMA levels (>40%) versus the unmodified control reveal interesting and characteristic elements of DPOMA cure chemistry. Early dry or set time is extended by monomer plasticization of the resin prior to its cure. This "fly paper" stage renders such coatings unsatisfactory. However, polymerization of the DPOMA eliminates the plasticizing effect of the monomer and the overall effect is only a slight extension of the dry-to-touch time. In addition, the through-dry time is greatly reduced. Accelerated weathering data indicates a nominal decrease in durability as measured by gloss retention, and the economics of these high level modifications of alkyds with DPOMA are impractical. Also, at these high levels of DPOMA, the coatings, in contrast to the pure alkyd controls, are hard and brittle. Accordingly, attention was focused on optimization of low levels (10–20%) of alkyd modification where the cost premiums of the system are small and where it is still possible to achieve significant advantages in solids, film build, through-cure, and resistance properties.

In developing useful formulations of alkyds with reactive diluents, the first issue is proper choice of the reactive diluent itself. Acrylate esters are generally undesirable because of less favorable toxicity profiles and because they react with the oxime stabilizers which are critical for development of stable one-package systems. Multifunctional methacrylates such as trimethylolpropane trimethacrylate provide a good cure balance but they are not sufficiently stable for one-package systems even in the presence of oxime (*Table* 3). Higher alkyl methacrylates such as stearyl or lauryl methacrylates are stable, but excessively soften the films[3] and frequently suffer from slow surface cure (*Table* 4). Moreover, lauryl methacrylate is too volatile under VOC testing conditions (*Figure* 3). Accordingly, DPOMA seems a reasonable choice for this application. Even though its viscosity of 18

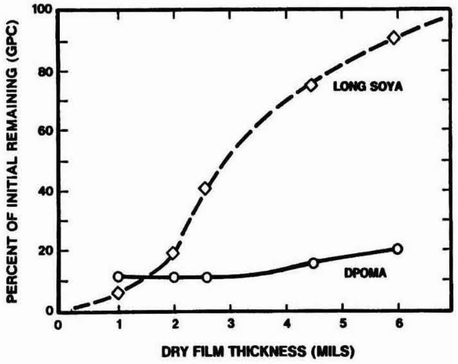


Figure 7—Conversion of conventional soya alkyd vs. DPOMA in 20 PVC (TiO_2) formulation of 80 alkyd/20 DPOMA//0.09% cobalt/0.22% zirconium/0.23% Exkin No. 2

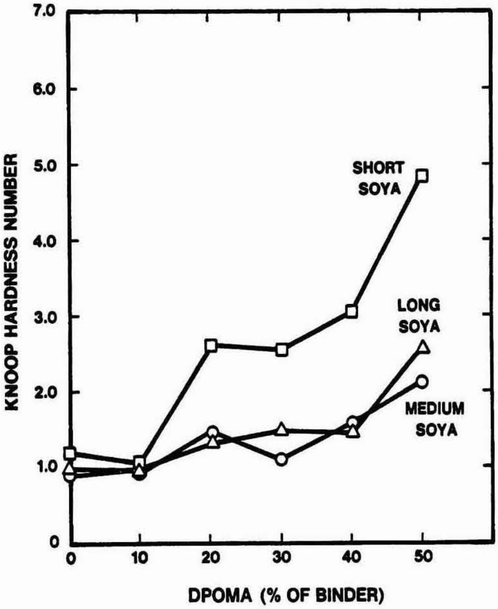


Figure 8—Film hardness vs. level of DPOMA in conventional soya alkyds for ~ 1.3 mil two-week-old clear films catalyzed by 0.06% cobalt/0.06% calcium/0.15% Exkin No. 2, Bonderite 40 substrate

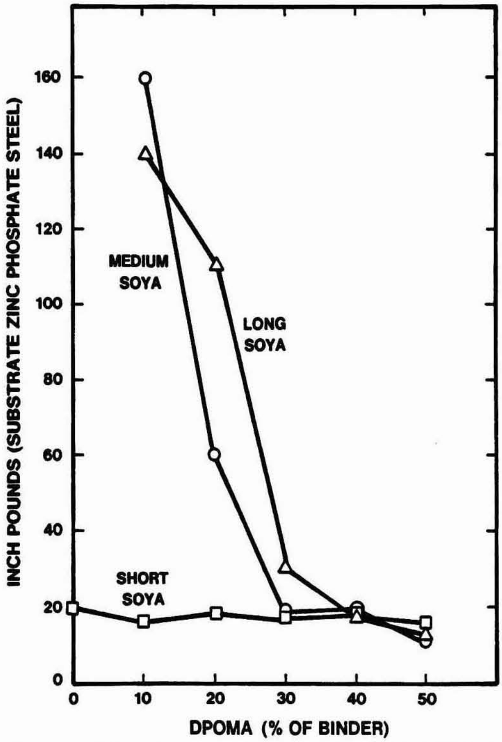


Figure 9—Direct impact resistance vs. level of DPOMA in conventional soya alkyds for ~ 1.3 mil two-week-old clear films catalyzed by 0.06 colbalt/0.06% calcium/0.15% Exkin No. 2, Bonderite 40 substrate

cps is greater than the viscosity of conventional solvents, it provides excellent viscosity reduction, quite comparable to mineral spirits, at high solids, as shown in *Figure* 4.

The most difficult problem associated with alkyd/monomer formulations is optimization of cure speed so that set times and through-cure are in satisfactory ranges. *Figure* 5 summarizes the effect of DPOMA on the set time of short, medium, and long soya oil alkyds. With the hard short oil alkyd, the monomer's plasticizing effect is not noticeable in terms of extended set times, until levels greater than 30% are used. With the medium and long oil alkyds the set time is extended, but at levels of monomer of 20% or under the effect is tolerable. Through-cure, as measured by film deformation of the circular recorder, is more interesting. Short oil alkyd DPOMA combinations show extended dry, probably because the oxygen scavenging effect of the short oil is insufficient to establish the anaerobic conditions in the film necessary for rapid monomer polymerization. With medium and long oil alkyds, however, through-cure is significantly improved by DPOMA modification, particularly at the 20% level (*Figure* 6). This is a major virtue of these systems particularly in air-dry and force-dry factory applications.

The superior through-dry of reactive diluent modified alkyds is related to the plasticizing effect of reactive diluent before conversion. More oxygen can enter the film which leads to faster alkyd crosslinking than in the unmodified alkyd. Concomitantly, the reactive diluent is coreacting with alkyd. Analytical studies using liquid chromatography indicate that reactive diluent conversion begins at about the set-time and is almost complete by through dry.

Even for reactive diluent modified alkyds, film thicknesses can be reached where the films are undercured in their lower portions; behavior very typical of alkyds. Oxygen reaction with the film is again responsible, but in this case, the film skins over before the film interior can react with enough oxygen to cure the alkyd. However, under these anaerobic conditions the reactive diluent can cure. *Figure* 7 is based on HPLC data and dramatizes this situation in detail. For the soya alkyd under air dry conditions, uncured alkyd increases dramatically as film thickness increases beyond ~ 2 mils. In strong contrast, the cure of DPOMA is virtually independent of film thickness.

Table 5—Use of Oils to Improve Flexibility of DPOMA Coating Systems^{a,e}

Reactive Diluent	Surface Tack^{b} (hrs to pass)	Impact Resistance^{d} (inch-lbs) Direct	Reverse	Tukon Hardness^{d} (KHN)	Pencil Hardness^{d} Before	After Gasoline^{c}
100 DPOMA	22-48	120	28	2.4	HB	HB
75 DPOMA/25 Soy bean oil	48-72	>160	100	1.6	2B	2B
75 DPOMA/25 Linseed oil	48-72	>160	110	1.7	2B	2B
75 DPOMA/25 Dehydrated castor oil	48-72	>160	140	1.6	B	2B
50 DPOMA/50 Soy bean oil	80-96	>160	>160	1.1	2B	2B
50 DPOMA/50 Linseed oil	80-96	>160	150	1.2	2B	2B
50 DPOMA/50 Dehydrated castor oil	48-72	>160	>160	1.2	2B	2B
No reactive diluent	30-46	>160	>160	1.5	2B	3B

(a) 20 PVC TiO_2, 80 medium oil alkyd/20 reactive diluent.
(b) Zapon, 300 g.
(c) 5 minute soak.
(d) One month properties.
(e) ~ 1.3 mil films.

Table 6—Alkyd Characteristics and Estimated DPOMA Level Needed for High Solids Paint

Oil[a] Length	% Solids	Solvent	Viscosity (G-H)	Comment	GPC Molecular Weight[b] M_w	M_n	M_w/M_n	Est.[c] % DPOMA to Give "J" Viscosity at 70% Solids[d]
Short	50	Blend	Y-Z1	General purpose	92,000	3100	30	45
Medium	50	OMS[e]	Z-Z2	" "	106,000	3300	32	45
Long	70	OMS	Y-Z2	" "	47,000	3800	12	26
Medium	80	CA[f]	Z3	High solids, chain stopped	25,000	2400	10	$\leqslant 10$

(a) Soya.
(b) pMMA calibration, low MW column; values for classification purposes only.
(c) See footnote (b).
(d) A "J" viscosity at 70% solids in general gives a high solids gloss paint (250–350 g l organic volatiles).
(e) Odorless mineral spirits.
(f) Cellosolve acetate.

Table 7—Comparison of Experimental DPOMA Modified Paints With Commercial Paints

Formulation	Volatiles (g/liter)	Viscosity KU	Viscosity Poise	Dry Properties Set (hrs)	Dry Properties Through Dry[c] (hrs)	10 Day Air Dry Hardness Tukon (KHN)	Hardness Pencil	Gasoline Resistance (15 min)	Alkali Resistance[a] (% Film Left) 15 min	2 hr	Flexibility 3/8" Mandrel	Reverse Impact[b] (in-lb)	Gloss 60°	Gloss 20°
A	244	83	3.7	2.7	5.0	1.4	B	2B	65%	45%	Pass	3	85	73
B	300	82	4.0	1.8	4.8	2.5	H	F	90	58	"	18	90	72
C	279	87	7.4	0.9	6.0	1.6	B	2B	62	55	"	<1	89	74
Commercial maintenance	438	86	3.8	2.0	5.0	1.5	2B	4B	0	0	"	25	85	56
Commercial equipment	500	79	2.6	0.3	>6<18	2.7	HB	3B	0	0	"	50	91	80

Formulation A: 20 PVC, TiO_2, 60 Long Soya/20 Very Long Linseed/20 DPOMA
" B: 20 PVC, TiO_2, 60 High Solids Med. Soya/20 Very Long Linseed/20 DPOMA
" C: 20 PVC, TiO_2, 40 High Solids Med. Soya Chain Stopped/20 Very Long Linseed/20 DPOMA/20 Methacrylate Modified Alkyd

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(a) 3% NaOH
(b) Substrate zinc phosphate steel
(c) Circular Recorder

Table 8—Spray Characteristics and Film Properties of High Solids Alkyd/Reactive Diluent Coatings: 5 PVC Yellow Iron Oxide (3 Pfizer YLO 1889D/1 R-960 TiO_2) Force Dry at 180°F/30 Min. at 1.5 Mil Over S-412 Steel[a]

ID	Spray Characteristics Viscosity #4 Ford (sec)	Viscosity ICI (poise)	Solids (%)	VOC (lb/gal)	9-Day Film Properties Gloss 60°/20°	Hardness Konig (cycles)	Hardness Tukon (KHN)	Impact (inch-lb) Dir./Rev.	Flexibility (Mandrel) 3/8"	Flexibility (Mandrel) 1/8"	Gasoline Resistance[b] Before/After
A	22	0.7	56	3.8	95/84	21	1.3	25/6	P	P	3B/6B
B	38	1.7	63	3.3	94/84	28	1.8	50/12	P	P	2B/5B
C	32	1.7	66	3.0	92/72	54	3.0	23/2	P	F	2B/2B
D	19	1.0	38	5.2	90/72	30	1.6	12/<1	P	P	3B/<6B

A-40 High Solids Chain Stopped Med. Soya/20 Acrylic Modified Alkyd/20 V. Long Linseed Alkyd/20 DPOMA.
B-40 High Solids Chain Stopped Med. Soya/20 Monomer Modified Alkyd/20 V. Long Linseed Alkyd/15 DPOMA/15 TMPTMA.
C-40 High Solids Chain Stopped Med. Soya/20 Styrene Modified Alkyd/20 V. Long Linseed/15 DPOMA/5 TMPTMA.
D-Commercial Alkyd Based Machinery Coating.

(a) All pass thumb twist and Zapon (300 g) after force dry
(b) Pencil hardness after 15 min. gasoline soak

Table 9—Gloss Retention for 45 TiO_2[a]/55 Binder[b] Based on Long Soya Alkyd

			Percent Gloss Retention						
			QUV[c]		Weather-ometer[d]	100% Humidity	Outdoor Exposure[e]		
Formulation	Gloss Angle	Initial Angle	140 Hrs	240 Hrs	520 Hrs	670 Hrs	3 Mos	6 Mos	Gloss After 6 Months Exposure
Long Soya	60°	85	100+%	74%	87%	95%	100%	100%	89
85 Long Soya/15 DPOMA	"	85	100	61	76	94	100	87	73
Commercial Control[f]	"	81	—	7	37	52	83	82	71
Long Soya	20°	76	61	18	51	81	87	72	55
85 Long Soya/15 DPOMA	"	75	60	12	40	72	75	40	30
Commercial Control	"	61	—	2	7	21	52	43	26

(a) Rutile.
(b) 23% phthalic anhydride, 70% solids in mineral spirits.
(c) Fluorescent UV lamp excitation.
(d) Xenon arc excitation.
(e) 45° South in Newton, Pa.
(f) 37 TiO_2 (rutile)/63 binder (tall oil alkyd).

Mechanical properties were also examined as a function of monomer modification level. As *Figure* 8 indicates, hardness of medium and long oil alkyds is not significantly influenced by monomer levels under 40%. In all systems, gasoline and alkali resistance are upgraded. Impact resistance (*Figure* 9) does decrease but at levels up to and including 20%; a significant amount of impact resistance still remains for medium and long oil alkyds. If alkyd choice does not provide sufficient flexibility, the use of oils to replace some reactive diluent is feasible, if some compromise in surface tack is acceptable (*Table* 5). Soft methacrylates such as lauryl or stearyl are unsatisfactory as flexibilizers because of long surface tack times (*Table* 4) and severely mottled films surfaces at critical film thicknesses in the 1-1.5 mil range.

The selection of proper alkyds for development of high solids alkyd DPOMA systems is critical. If a high solids formulation is arbitrarily defined in the clear coatings as a "J" viscosity at 70% solids, the amount of reactive diluent required to achieve this solids level is indicated in *Table* 6. The conventional short and medium oil alkyds require too much reactive diluent. In actual practice, alkyd blends and high solids alkyds offer attractive systems. If moderate dry time is required (>2 hours set) a long oil alkyd can be utilized with 20% DPOMA. Solids of this type of formulation can readily be increased by use of up to 20% very long oil alkyd (some at 100% solids) without significant sacrifice in set time. A high solids fast-dry formulation (set<2 hours) is conveniently based on 40-60% medium oil alkyd (sometimes chain stopped), 20% very long oil alkyd, and (optionally) 20% styrenated or methacrylated alkyd again with 20% DPOMA. The monomer modified alkyd is useful in improving fast dry because of its high Tg and improved lacquer dry. Typical high solids formulations of the types described above are summarized in *Table* 7 and compared to commercial maintenance and equipment formulations. In general, the DPOMA systems have somewhat longer set times, much higher solids and film build, good brushing qualities, improved through dry and resistance properties, and some decrease in flexibility. Formulations which force dry at 180° F/30 min and spray at VOC's less than 3.5 lb/gal are also possible (*Table* 8). Durability of these, measured by gloss retention (*Table* 9), is typically slightly less than the alkyd controls but is in the same range as typical commercial products.

SUMMARY

Modification of alkyds by vinyl monomers that act as reactive diluents gives high solids coatings with improved through-dry, alkali resistance, and gasoline resistance. Dicyclopentenyloxyethyl methacrylate, an acrylic monomer designed specifically as a reactive diluent, illustrates the use of such monomers as alkyd modifiers. Successful formulations must keep reactive diluent levels in the 15-30% range. Alkyds must be chosen which provide sufficient oil length in order to achieve an acceptable cure rate, provide sufficient hardness for acceptable set times, and have a low enough molecular weight to allow high solids formulations.

ACKNOWLEDGMENT

We would like to acknowledge the major contributions of K. Nyi, P. Sperry, R. Wiersema, J. Shelley, R. Novak, and J. Dixon to the work described herein.

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Studies on the Esterification Of Trimellitic Anhydride

Implications for Coating Polymers

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The esterification of trimellitic anhydride (TMA) with three hydroxyl-functional reactants was observed at various reaction temperatures. Results from the reaction with 2-ethylhexanol showed that TMA monoester was formed initially, and all the carboxyl groups of the monoester and diester species had approximately equal reactivity during the remainder of the reaction. The resulting distributions of TMA monoester, diester, and triester were found to be independent of reaction temperature and dependent only on extent of TMA reaction. Results from the reactions with diethylene glycol and a polyester diol led to similar conclusions. Practical implications of the results pertaining to coating polymers are explored.

INTRODUCTION

Trimellitic anhydride (TMA) is a trifunctional aromatic acid anhydride used as an intermediate in the formation of coating polymers such as polyesters. The structures of TMA and its various ester derivatives are shown in *Figure* 1. When esterified to completion, in triester form, TMA serves as a source of branching in a polymer chain. When partially reacted onto a polymer, in monoester or diester form, it contributes free, unreacted carboxyls which can take part in thermoset reactions and which can impart water dispersibility upon neutralization.

TMA's trifunctionality and its combination of anhydride and carboxyl functionality make it a more versatile raw material than most other commonly used acids. Because of this versatility, questions arise as to the best way to design and process coating polymers using TMA. Can TMA be reacted exclusively into monoester form or diester form? Does steric hindrance impede the formation of triester? Is reaction temperature a major factor in determining which reaction products result?

These questions and others are only partly answered by the available literature on TMA derivatives.[1] Work presented here uses chemical analysis in an attempt to further characterize TMA reactivity. In brief, the distribution of TMA mono–, di–, and triester was observed as a function of reaction temperature, extent of reaction, and type of hydroxyl-functional reactant. The results help resolve some of the issues regarding the nature of polymers employing TMA.

EXPERIMENTAL

Trimellitic Anhydride Esterification

Reactions of TMA with an alcohol, a diol, and a linear, hydroxyl-functional polyester were observed. Selected as representative compounds were 2-ethylhexanol (2EH), diethylene glycol (DEG), and a low molecular weight polyester diol made from neopentyl glycol, adipic acid, and isophthalic acid. Formulations are shown in *Table* 1. No esterification catalysts were used.

All reactions were carried out in two-liter flasks with nitrogen sparge and agitation. Steam-jacketed partial condensers and water-cooled total condensers were used with DEG and polyester diol batches. A Dean-Stark trap and total condenser were used with all 2EH batches to remove water and return condensed 2EH to the reaction flask.

The formulations in *Table* 1 were reacted in various batches at different temperatures. These batches are shown in *Tables* 2 and 3. Formulation A, for example, was reacted in separate batches at seven different temperatures, to evaluate the effects of temperature on reaction products. For most of the batches, the raw materials were mixed at room temperature and then

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Presented at the 60th Annual Meeting of the Federation of Societies for Coatings Technology in Washington, DC, November 3, 1982.

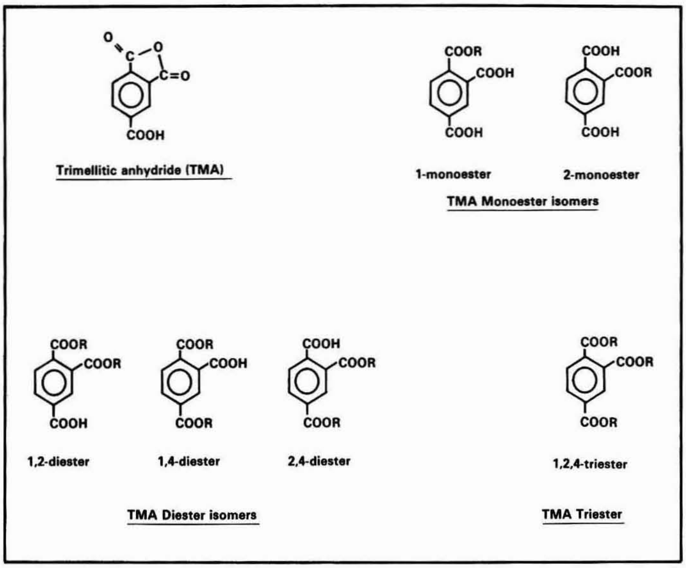

Figure 1—Structures of trimellitic anhydride and its ester derivatives

heated to the desired temperature, with at least some esterification occurring during the heat-up period. In all cases, once the desired temperature was reached, that temperature was maintained for the remainder of the reaction. With each batch, samples were withdrawn at various intervals after the desired temperature was reached. Acid numbers were measured, and the samples were retained for analysis.

Chromatographic Analysis

Samples from the TMA/2EH batches were analyzed by esterification gas chromatography (EGC). The TMA/DEG and TMA/polyester reaction products, being polymeric in nature, were not analyzed. The EGC method used is based on esterification of carboxylic acid salts with trimethylphosphate (TMP). A mixture of TMP (5 mL), pyridine (2 mL), and the TMA reaction product (0.5 g) was heated to 145°C for 30 min during which time unreacted carboxyls were converted to methyl esters.

The ester samples were then separated by gas chromatography, with a silicone stationary phase column. A temperature gradient was used, whereby column temperature during separation increased from 175°C to 300°C at a rate of 8°C per minute. Theoretical response factors relative to triphenylmethane were used to convert peak areas to relative compound weights.

RESULTS AND DISCUSSION

A typical chromatogram of a TMA/2EH sample is shown in *Figure* 2. Every compound shown in *Figure* 1, except for the trimellitic anhydride, has a corresponding peak in the *Figure* 2 chromatogram. Chromatographic analysis not only separated mono–, di–, and triester species from each other, but also separated the monoester isomers from each other and the diester isomers from each other. Two types of distributions—those of mono–, di–, and triester and those of monoester isomers and diester isomers—were therefore quantified by the chromatographic data. Analysis of the TMA/2EH samples showed how both types of distributions varied with reaction temperature and also extent of reaction.

Distributions of TMA Monoester, Diester, and Triester

The mono–, di–, and triester distribution results for 15 TMA/2EH samples are shown in *Table* 4. The following observations are noted from the data:

INITIAL REACTION: The predominant initial reaction of TMA was the opening of the anhydride ring to produce monoester. This would be expected, given the high reactivity of an anhydride group compared to that of a carboxyl group. Since sample 15-1 was withdrawn very early during the esterification reaction, at a low extent of reaction, its analysis in *Table* 4 best shows the effect of this initial reaction.

TRIMELLITIC ACID: Analysis of many samples showed higher than expected levels of trimellitic acid. Under the conditions of excess hydroxyl used here, it was assumed that trimellitic anhydride would convert almost entirely to monoester during the initial stage of the reaction, leaving no trimellitic anhydride or trimellitic acid.

Significantly lower levels of trimellitic acid were found if the analytical sample preparation procedure was modified. When pyridine was replaced with tetramethylguanidine, and the reaction with TMP was carried out at 100°C instead of 145°C, only 1.7%, 0.2%, and 0% trimellitic acid were found, respectively, in samples 15-1, 15-3, and 15-4. These levels are contrasted with 7.7%, 1.5%, and 0.7% levels shown in *Table* 4. It is felt that a side reaction occurring during the sample preparation reaction significantly affected trimellitic acid levels. Actual levels before the preparation reaction were probably lower than those shown in *Table* 4. Since the side reaction had little effect on the relative levels of mono-, di-, and triester, the modified procedure was not carried out on all the samples. The relative levels of mono-, di-, and triester shown in *Table* 4 are believed to be valid.

DIESTER AND TRIESTER FORMATION: TMA diester and triester species began forming simultaneously in the early stages of the TMA/2EH reaction, even at temperatures as low as 100°C and 120°C. Reaction rates were slow at these low temperatures, but given time, substantial levels of diester and triester did build up, resulting in a distribution of monoester, diester, and triester species. Sample 16-2, for example, withdrawn from the flask after 11 hours at 120°C, contained 47% diester and 30% triester along with 21% monoester.

INDEPENDENCE OF TEMPERATURE: Distributions of TMA mono-, di-, and triester were independent of reaction temperature and dependent only on acid number (or extent of reaction) of the samples analyzed. In other words, the extent of reaction alone was enough to completely define a sample's distribution of esters. This point is illustrated by *Table* 5, which groups *Table* 4 data by acid number similarity of the samples. Samples 16-2, 15-3, and 17-2, for example, had similar TMA ester distributions despite substantial differences in reaction temperatures. The three samples had similar acid numbers.

The chromatographic mono-, di-, and triester results are summarized in *Figure* 3, which depicts the distributions as a function of average extent of TMA reaction. Average extent of reaction is defined here as the average number of esterified sites per TMA molecule. An extent of reaction of 2.0, for example, refers to TMA being in diester form on average, with 67% of the TMA acid equivalents having been esterified. Trimellitic acid levels were omitted from the *Figure* 3 graph due to their relative insignificance and due to their dependence on the type of analytical procedure employed. The distributions of all 15 analyzed samples fell along this graph. It is clear that

Table 1—Formulations

	Parts by Weight		
	A	B	C
2-Ethylhexanol	70.9		
Diethylene glycol		52.8	
Linear polyester diol[a]			85.2
Trimellitic anhydride	29.1	47.2	14.8
	100.0	100.0	100.0
% Excess hydroxyl	20	35	37
Acid number at 1/3 acid equivalents reacted	170	276	86
Acid number at 2/3 acid equivalents reacted	87	144	44

	Parts by Weight
(a) Neopentyl glycol	50.0
Adipic acid	23.4
Isophthalic acid	26.6
	100.0

Ingredients reacted to a 5 acid number; 530 theoretical number-average molecular weight.

Table 2—TMA/2-Ethylhexanol Batches

Batch number	14	16	15	17	18	19	21
Formulation	A	A	A	A	A	A	A
Time needed to reach reaction temperature, hr[a]	0.4	0.7	1.0	0.8	1.1	1.3	2.0
Reaction temperature, °C	100	120	140	160	180	200	220
Total time at reaction temperature, hr	6.6	12.3	5.8	5.7	3.8	3.8	4.0
Final acid number	152	78	68	30	17	10	4.6
Extent of trimellitic anhydride reaction, at final acid number[b]	1.22	2.11	2.23	2.67	2.80	2.89	2.95

(a) Equal to the number of hours that esterification took place at temperatures lower than the designated reaction temperature.

(b) Average number of esterified sites per TMA molecule, calculated on the basis of final acid number. Sample calculation:

For Formulation A at a 152 acid number,

$$\text{Yield} = \frac{\text{charge wt.} - \text{water off at zero acid number}}{1 - \dfrac{18\,(152)}{56{,}100}} = 993.9 \text{ g (based on 1,000 g charge)}$$

$$\text{Acid equivalents remaining} = \frac{(993.9 \text{ g})\,(152 \text{ mg KOH/g})}{56{,}100 \text{ mg KOH/acid eq.}} = 2.69 \text{ equivalents}$$

Reacted acid equivalents = charged acid eq. − remaining acid eq. = 1.86 equivalents

Extent of TMA reaction = reacted TMA acid eq./moles TMA charged = 1.86/1.52 = 1.22

Table 3—TMA/Diethylene Glycol and TMA/Polyester Diol Batches

	TMA/DEG				TMA/Polyester Diol
Batch number	22	23	24	25	98
Formulation	B	B	B	B	C
Time needed to reach reaction temperature, hr[a]	0.5	0.5	0.6	1.5	0.1
Reaction temperature, °C	140	160	180	200	180
Total time at reaction temperature, hr	12.9	12.6	5.7	2.0	6.2
Final acid number	112	58	57	60	30.8
Extent of trimellitic anhydride reaction, at final acid number[b]	2.23	2.61	2.62	2.60	2.30

(a) Equal to the number of hours that esterification took place at temperatures lower than the designated reaction temperature. In batch 98, TMA was added to the hydroxyl-functional material at 170°C.

(b) Average number of esterified sites per TMA molecule, calculated on the basis of final acid number.

the distributions were independent of reaction temperature.

Isomeric Distributions Of TMA Monoester and Diester

Assuming that the initial reaction of TMA with hydroxyl compounds involves only the opening of the reactive anhydride ring, then two monoester isomers are possible: 1-monoester and 2-monoester (referring to the 1 and 2 positions on the trimellitic anhydride molecule).[1] Subsequent to monoester formation, diester formation can potentially result in three isomers: 1,2-diester, 1,4-diester, and 2,4-diester. These isomeric structures are shown in *Figure* 1. The chromatogram in *Figure* 2 indicates that all five isomers mentioned appeared analytically as separate peaks, along with the single 1,2,4-triester peak. Following the chromatographic baseline from left to right, the triester peak first appears, followed by the three diester isomeric peaks, followed by the two monoester isomeric peaks. The compounds actually eluted in the reverse order.

Table 6 shows the monoester and diester isomeric distributions for each of the 15 samples analyzed. The relative amounts of monoester isomers and of diester isomers did not vary greatly from one sample to another. The isomeric distributions were, therefore, relatively independent of both reaction temperature and extent of reaction. Predicted relative volatilities of the isomers lead to a guess that the 1- and 2-monoesters were present in a 60/40 ratio, respectively, and the 1,2-, 2,4-, and 1,4-diesters were present in a 53/22/25 ratio, respectively.

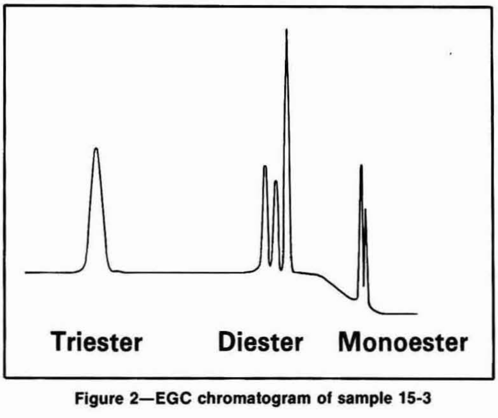

Figure 2—EGC chromatogram of sample 15-3

Relative Reactivity of TMA Carboxyls

The mono-, di-, and triester data and the isomeric data lead to speculation that the carboxyl groups belonging to various TMA/2EH reaction products may have approximately equal reactivity. This hypothesis, if true, would imply that any carboxyl reactivity differences arising from steric or electronic effects are negligible in this reaction. To test the hypothesis, an investigation was undertaken to determine the mono-, di-, and triester distributions and the isomeric distributions that would result from an assumption of equal reactivity.

MONO-, DI-, AND TRIESTER DISTRIBUTIONS: A computer program was used to simulate the esterification of TMA with alcohol and to generate distributions of mono-, di-, and triester as a function of average extent of reaction. A "Monte Carlo"-type study was used.[2] This type of study involves the use of synthetic sampling—by drawing random values from statistical distributions—to simulate the behavior of some physical process. For the study, it was assumed that the initial reaction of TMA is its conversion to 100% monoester through the opening of the anhydride ring. Thus, 100% monoester was used as the starting point for the model. It was also assumed that at any point during the subsequent reaction, all carboxyls in the system have equal reactivity and, therefore, equal probability of reacting with alcohol molecules. This would mean that each of the seven different types of carboxyls belonging to the five mono- and diester isomers shown in *Figure* 1 have equal reactivity.

The computer results are summarized by the graph in *Figure* 4. The graph is symmetrical around a vertical line passing through the point on the x axis representing two reacted sites per TMA molecule. Implicit in the assumptions used for the computer model is the idea that the results depicted in *Figure* 4 are independent of the ratio of TMA and alcohol reactants. That ratio determines only the maximum extent of TMA reaction possible.

The computer-generated graph in *Figure* 4 is quite similar to the graph in *Figure* 3 showing actual chromatographic results.

Table 4—Chromatographic Analysis of TMA/2-Ethylhexanol Reaction Products

					Molar Distribution, as Percent					
Sample Designation	**Batch No.**	**Reaction Temperature**	**Reaction Time[a]**	**Measured Acid No.**	**Trimellitic Acid**	**Monoester**	**Diester**	**Triester**	**Chromatographic Extent of Reaction[b]**	**Acid Number Extent of Reaction[c]**
14-1	14	100°C	6.6 hr	152.4	3.5	69.6	24.4	2.5	1.26	1.22
16-1	16	120	3.8	131.8	3.1	49.7	38.9	8.3	1.52	1.47
16-2	16	120	11.0	84.8	1.3	21.4	47.2	30.1	2.06	2.03
15-1	15	140	−0.3	173.1	7.7	88.4	3.9	—	0.96	0.96
15-2	15	140	1.2	133.6	3.3	50.4	38.4	7.9	1.51	1.45
15-3	15	140	3.8	86.8	1.5	21.9	48.2	28.4	2.04	2.01
15-4	15	140	5.8	67.5	0.7	13.8	44.6	40.9	2.26	2.23
17-1	17	160	0	142.7	3.5	57.5	33.7	5.3	1.41	1.34
17-2	17	160	1.2	88.7	1.3	22.9	47.9	27.9	2.02	1.98
17-3	17	160	5.7	29.7	—	1.8	26.0	72.2	2.71	2.67
18-1	18	180	0	100.3	1.6	29.0	48.0	21.4	1.89	1.85
18-2	18	180	3.8	17.4	—	1.0	16.2	82.8	2.82	2.80
19-1	19	200	0	67.5	0.5	13.6	44.8	41.1	2.27	2.23
19-2	19	200	3.8	10.1	—	—	9.8	90.2	2.90	2.89
21-1	21	220	0.3	18.4	—	1.2	17.1	81.7	2.81	2.79

(a) Elapsed time between achievement of designated reaction temperature and extraction of sample from batch. Sample 15-1 was pulled at 125°C, before the ultimate reaction temperature was reached. Samples 17-1, 18-1, and 19-1 were pulled upon achievement of the designated temperature.

(b) Average number of esterified sites per TMA molecule, as calculated from chromatographic molar distribution.

(c) Average number of esterified sites per TMA molecule, as calculated from measured acid number of sample.

ISOMERIC DISTRIBUTIONS: As a further test of the hypothesis of equal carboxyl reactivity, the isomeric distributions which would result from this hypothesis were investigated. *Figure* 5 shows reaction schemes for TMA monoester formation and diester formation, based on this assumption of equal reactivity.

The monoester formation scheme in *Figure* 5 is based on actual chromatographic results showing a 60/40 ratio (on average) of the two monoester isomers. Although *Figure* 5 indicates that the 1-monoester is the favored isomer, this has not actually been determined. Using this 60/40 ratio as the monoester starting point for diester formation, the scheme shows the diester isomeric distribution that would result if each of the monoester and diester carboxyls had equal reactivity. The resulting distribution would be 50% 1,2-diester, 30% 1,4-diester, and 20% 2,4-diester. This 50/30/20 ratio compares rather well with the 53/25/22 ratio found on average experimentally, shown in *Table* 6. If the starting point for diester formation in *Figure* 5 had been 60/40 2-monoester/1-monoester instead of vice versa, then the resulting diester isomeric ratio would still be 50/30/20, but in this case the 30 and 20 would refer to 2,4-diester and 1,4-diester, respectively.

The computer study and the isomeric distribution study offer convincing support for the theory of approximately equal carboxyl reactivity in the reaction between TMA and 2EH. The model based on this hypothesis closely approximated the isomeric distributions and the mono–, di–, and triester distributions found experimentally in the TMA/2EH systems.

Validity of Chromatographic Results

The validity of the TMA/2EH chromatographic results is supported by the following observations:

Table 5—TMA/2-Ethylhexanol Chromatography Results—Grouped by Similarity of Measured Acid Numbers of Samples

			Molar Distribution, as Percent			
Sample Designation	**Reaction Temperature (°C)**	**Measured Acid Number**	**Trimellitic Acid**	**Monoester**	**Diester**	**Triester**
16-1	120	131.8	3.1	49.7	38.9	8.3
15-2	140	133.6	3.3	50.4	38.4	7.9
16-2	120	84.8	1.3	21.4	47.2	30.1
15-3	140	86.8	1.5	21.9	48.2	28.4
17-2	160	88.7	1.3	22.9	47.9	27.9
15-4	140	67.5	0.7	13.8	44.6	40.9
19-1	200	67.5	0.5	13.6	44.8	41.1
18-2	180	17.4	—	1.0	16.2	82.8
21-1	220	18.4	—	1.2	17.1	81.7

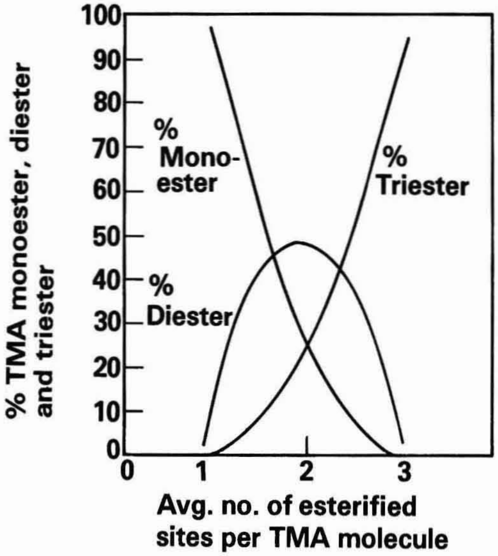

Figure 3—Chromatographic distribution of TMA esters vs extent of TMA reaction

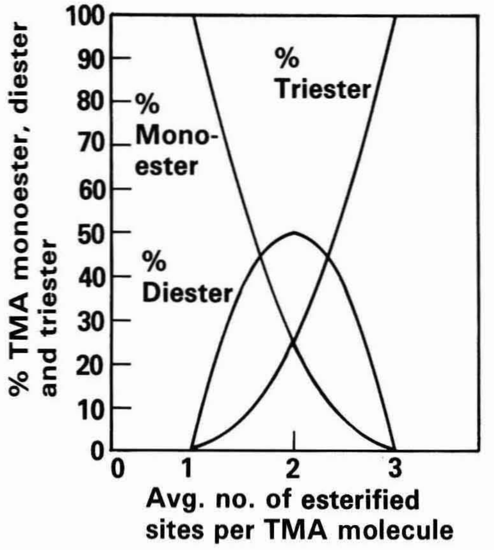

Figure 4—Computer-generated distribution of TMA esters vs extent of TMA reaction—based on an assumption of equal reactivity of carboxyls

EXTENT OF REACTION CORRELATION: There was in general good correlation between average extents of reaction calculated from sample acid numbers and average extents of reaction calculated from chromatographic results. This correlation is shown in the last two columns of *Table* 4. Thus, for example, it is unlikely that TMA monoester or diester esterified further with excess 2EH during chemical treatment of the samples for esterification gas chromatography.

Table 6—TMA/2-Ethylhexanol Chromatography Results—Monoester and Diester Isomeric Distributions

Sample Designation	Monoester Isomeric Distribution, as Percent, Isomer #1[a]	Isomer #2	Diester Isomeric Distribution, as Percent Isomer #1[a]	Isomer #2	Isomer #3
14-1	41	59	51	21	28
16-1	39	61	51	21	28
16-2	35	65	54	19	27
15-1	43	57	47	23	30
15-2	40	60	50	22	28
15-3	39	61	52	22	27
15-4	40	60	53	21	26
17-1	42	58	50	22	28
17-2	42	58	51	22	26
17-3	18	82	58	20	22
18-1	43	57	51	23	26
18-2	50	50	60	20	20
19-1	45	55	52	24	25
19-2	—	—	61	20	19
21-1	43	57	55	23	22
mean	40	60	53	22	25
std. deviation. .	7.2	7.2	3.9	1.4	3.2

(a) Isomers are numbered in their order of elution.

ACID NUMBER PROGRESSION: *Figure* 6 is a plot of acid number vs time for Batch 17 (160° C) in the TMA/2EH series. At an 87 acid number, TMA was in diester form on average. The fact that acid numbers less than 87 could be measured indicates that some triester definitely formed. This finding supports the presence of triester in chromatographic results.

If the acid number drop during monoester formation could have been accurately monitored and included in *Figure* 6, then the total graph would have shown a very rapid drop in acid number followed by the more gradual drop that was actually monitored. This change in reaction rate would have resulted from the difference in reactivity between the TMA anhydride ring and monoester carboxyls. The inflection in the curve would have occurred at about a 170 acid number, at which point monoester had been formed on average and diester was just beginning to form. The fact that no large changes in reaction rate occurred elsewhere in the curve supports the concept of equal carboxyl reactivity for this reaction. Since the reaction rate did not drop at an 87 acid number, for example, diester carboxyls could not have been substantially less reactive than monoester carboxyls. The simultaneous formation of diester and triester following monoester formation is therefore implied. *Figure* 6 shows that the reaction rate continually drops as the reaction proceeds, but only gradually, and this gradual drop is only a result of a declining concentration of carboxyls and hydroxyls.

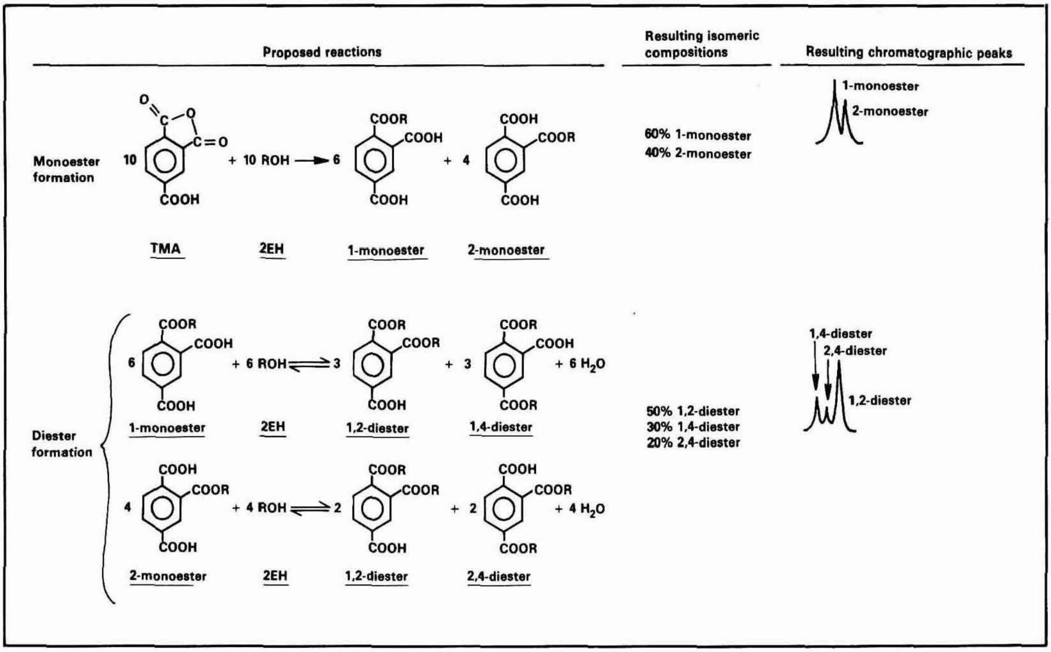

Figure 5—Conversion of TMA to monoester and diester isomers as suggested by the concept of equal carboxyl reactivity

TMA/Diethylene Glycol Reaction

Due to possible differences in steric hindrance and other factors, it could not be assumed that the TMA/2EH results discussed would also apply to polymers such as those formed by the TMA/DEG reaction. The four TMA/DEG batches described in *Table* 3 were prepared so that TMA reactivity could be characterized under circumstances different from those in the 2EH reaction.

The TMA/DEG reaction products could not be chromatographically analyzed to show how TMA reacted. As with the 2EH batches, however, certain conclusions could be drawn from the reaction progression as monitored by acid number measurement. *Table* 3 shows that each of the four DEG batches was able to be taken to an average extent of reaction greater than 2.0 reacted sites per molecule. Some TMA triester therefore was formed in each batch. More specifically, since the 140°C batch was taken to a 2.23 extent of reaction, at least 23% of the TMA molecules were in triester form by the end of the reaction. The 200°C batch, reacted to a 2.60 extent of reaction, contained at least 60% TMA triester by the time the reaction was terminated.

A plot of acid number vs time for the 180°C TMA/DEG batch is shown in *Figure* 7. At a 144 acid number, a 2.0 extent of reaction was reached on average. As with the other DEG batches, no major change in reaction rate is seen at a 2.0 extent of reaction or elsewhere on the graph. It can be assumed then that TMA triester began forming early in the course of the reaction instead of suddenly appearing during the later stages.

The behavior of TMA in the DEG batches was, therefore, qualitatively similar to its behavior in the 2EH batches. Significant levels of triester did form, and likely began forming shortly after monoester formation, even at low reaction temperatures. As with the 2EH reaction, TMA carboxyls appeared to have approximately equal reactivity. It appears that any additional steric hindrance effects caused by polymer formation in the DEG batches did not seriously impede the formation of TMA triester.

TMA/Polyester Diol Reaction

To more closely approximate the conditions under which a carboxyl-functional coating polymer is made, the reaction of TMA with a polyester diol was examined. The polyester, formed from neopentyl glycol, adipic acid, and isophthalic acid, was reacted to a low acid number as shown in *Table* 1. By using a linear polyester, the potential for gelation during the reaction with TMA was reduced. The TMA and polyester were reacted together in one batch, at 180°C, as shown in *Table* 3. The resulting polymer could be used in a water-reducible coating.

Figure 8 shows a plot of acid number vs time for the TMA/polyester batch. As with the DEG and 2EH reactions, acid numbers were easily reached at which TMA triester was definitely forming. Also, as with the DEG and 2EH reactions, there were no sudden changes in reaction rate during the portion of the reaction that was monitored. Once again, TMA carboxyls appeared to have approximately equal reactivity.

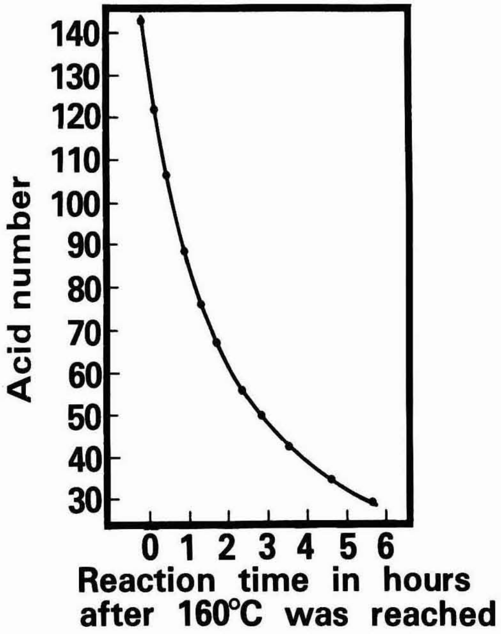

Figure 6—Acid number progression for batch 17: TMA/2-ethylhexanol at 160° C

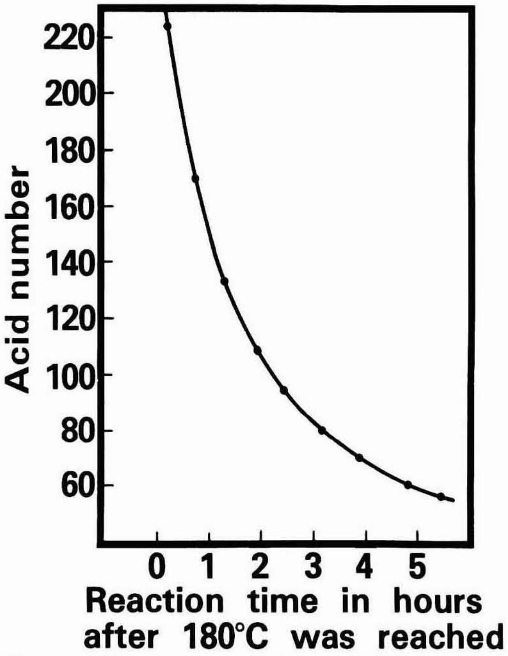

Figure 7—Acid number progression for batch 24: TMA/diethylene glycol at 180° C

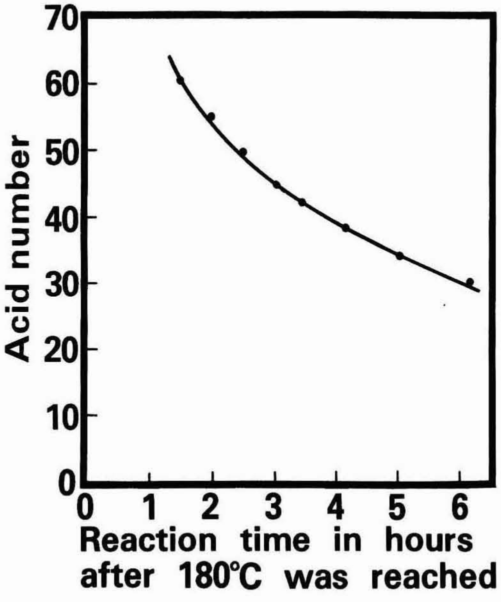

Figure 8—Acid number progression for batch 98: TMA/polyester diol at 180° C

Some Practical Implications of Results

The various results presented here lead to many practical implications regarding coating polymers based on TMA. Some of these implications are discussed here.

Monoester Formation: It should be easy to employ TMA in monoester form in a coating polymer. The TMA monoester species should predominate if the reaction can be stopped after the anhydride ring has opened. This can be accomplished for typical coating alkyds and polyesters by adding TMA to the base resin at 170–180° C, holding at that temperature for 15 to 30 min, cooling, and thinning when possible. The 170–180° C temperature is generally high enough to keep the base prepolymer fluid and mobile, and it is high enough to melt the TMA, facilitating fast reaction (TMA melts at 168° C). The 15 to 30 min reaction period should convert all the TMA to monoester, without forming too much di- or triester.

Triester Formation: By approaching a zero acid number, it is also possible to achieve close to 100% TMA triester.

Diester Formation: It may be difficult through conventional processing to prepare a polymer which employs TMA strictly in diester form. A distribution of mono-, di-, and triester will more likely result. However, it is conceivable that proper selection of raw materials and processing procedure could enable much higher levels of diester to be obtained than the levels found in work presented here. Hydroxyls attached to bulky alkyl groups, for example, might be able to impede triester formation through steric hindrance. Diester levels could rise far above 50% with the 1,4- and 2,4- diester isomers predominating. In a situation such as this, TMA's carboxyls would not have equal reactivity.

STAGING OF TMA ADDITION: Even under conditions of equal TMA carboxyl reactivity, the resin formulator is not necessarily restricted to TMA ester distributions of the type in *Figure* 3. More complex distributions can be achieved by splitting the TMA charge into two or more stages. For example, by charging TMA in two stages, reacting the first stage to a low acid number, and reacting the second stage to a higher acid number, TMA can be used for the dual purpose of providing polymer branching and also contributing carboxyl functionality to the polymer. The resulting ester distribution can deviate widely from distributions in *Figure* 3. A 50/50 triester/monoester combination is one possibility.

CARBOXYL/COSOLVENT REACTION: The fact that TMA carboxyls can react at low temperatures suggests the possibility of reaction between polymer carboxyls and cosolvent hydroxyls upon thinning of a water-reducible polymer. In fact, the author has studied the phenomenon of n-butanol cosolvent reacting with TMA carboxyls of a water-reducible polyester upon thinning at 150°C. The potential for reaction depends partly on the reactivity of the cosolvent involved. For example, ethylene glycol monobutyl ether was judged less reactive than n-butanol. Ultimate film properties could be affected by this phenomenon of cosolvent reaction.

GELATION: Another practical implication involves the gelation characteristics of a TMA-based polymer. To the extent that the 2EH esterification results apply to a coating polymer, then the gelation characteristics contributed by TMA to the polymer are more dependent on extent of TMA reaction than on reaction temperature. Limiting the reaction temperature to 180°C, for example, would not necessarily prevent eventual gelation of the polymer.

SUMMARY

The data presented here were gathered in an attempt to characterize TMA esterification behavior as a function of such variables as reaction temperature and type of hydroxyl reactant. The study of TMA's reaction with 2-ethylhexanol generated a large amount of informative data. Results from the reactions of TMA with diethylene glycol and with a polyester diol were more limited in scope, but they did imply that the 2-ethylhexanol esterification results can carry over to polymer formation. Two primary conclusions were drawn regarding these three reactions:

(1) The predominant initial reaction of TMA with the hydroxyls was the opening of the anhydride ring to produce TMA monoester.

(2) As TMA monoester formed and the reaction continued, the carboxyl groups of the various TMA derivatives had approximately equal reactivity.

It is emphasized that the data presented here were generated from a limited number of reaction types. Only three types of hydroxyl reactants were evaluated, and such conditions as excess carboxyl as opposed to excess hydroxyl were not included in the evaluation. Consequently, the conclusions drawn cannot be presumed to apply to all sets of reaction conditions. The current work has nevertheless helped resolve major issues on TMA reactivity, and it leads to a better understanding of the nature of TMA-based coating polymers.

ACKNOWLEDGMENTS

The advice and support of Tom Czajka, Bob Engelhardt, Al Wilson, and Bert Harker, of Amoco Chemicals Corp., are gratefully acknowledged. The author also wishes to thank George W. Powers and Eric Ziegel, of Standard Oil Co. (Indiana), for their supervision of the analytical and computer work, respectively, carried out for this study.

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Society Meetings

BIRMINGHAM .... MAR.

"The Analytical Laboratory In a Modern Paint Company"

Dr. Stan Bryan, of Donald Macpherson, discussed "THE ROLE OF THE ANALYTICAL LABORATORY IN A MODERN PAINT COMPANY."

Dr. Bryan outlined the important roles played by the analytical laboratory in support of the technical functions. He described the types and catagory of analytical laboratories, the most familiar consisting of two burettes and a card table.

The three essential operations of any paint analysis were, according to Dr. Bryan, separation, identification, and quantification. The modern laboratory often has classical equipment like balance, centrifuge, oven and furnace, as well as atomic absorption spectrophotometry, infra-red spectrophotometry, and gas-liquid chromatography. Dr. Bryan detailed this equipment.

Dr. Bryan discussed the basic pattern underlying analysis in the paint company, and illustrated with real examples. Raw materials, wet and dry paint, and other problems relating to all the departments of a company right through to customer service were detailed.

Dr. Bryan concluded with a discussion of costs versus benefits.

Q. Is there any equipment available for the routine measurement of particle size in liquid dispersions?

A. No, but I believe this will come as part of the drive to optimize pigment use.

Q. Many companies cannot afford large amount of instruments. What would you choose on a limited budget?

A. Infra-red spectrophotometry—the more sophisticated machines will approach a complete analysis on-machine.

Q. Is it for an internal laboratory to provide data on environmental questions?

A. No. An external laboratory should be used for the element of confidence and to a lesser extent special expertise.

Q. Do you charge the individual section of the company for work done?

A. Yes, by the hour.

Q. How do you present solvent to a capillary core without contaminating it?

A. We use a packed column, the newer versions of which are cleanable.

D. H. CLEMENT, *Secretary*

CLEVELAND .... APR.

"Annual Technical Conference"

The society's Education Committee sponsored its 26th Annual Technical Conference entitled, "Advances in Coatings Technology," at the NASA Lewis Research Center, Cleveland, OH.

Lawrence Ross, Director of the Space Directorate at Lewis Research Center was the speaker for Wednesday's evening dinner program He described some of the activities, programs, and projects with which the Center is concerned. Currently, the Center is involved in the space shuttle program. According to Mr. Ross, their next step is the Centaur Project.

Dr. Geoffrey D. Parfitt, of Carnegie-Mellon University, gave a special guest lecture Thursday morning. The subject of his talk was "Stabilization of Pigment Dispersions by Adsorbed Polymers."

Dr. Parfitt explained that the thickness of adsorbed layers and conformation of adsorbed macromolecules are important parameters. He reviewed current knowledge relative to stabilization of dispersions of titanium dioxide and polymer latexes.

The Outstanding Speaker Award was presented to Clifford K. Schoff, of PPG Industries, Inc. He presented a paper co-authored with Percy E. Pierce on "The Effects of Surface Tension and Viscosity on Surface Defects in Coatings."

A tour of a portion of the Lewis Research Center gave some insight into the activities involved in space research.

RAYMOND PODLEWSKI, *Secretary*

KANSAS CITY .... APR.

"Changing Technologies For Industrial Coatings"

The Educational Committee report was presented by Karl King. He stated that he and other committee members had judged the local Science Fair and had selected three entries representative of the coatings industry. These winners will be honored at the May meeting.

The Technical Committee announced its Annual Meeting presentation entitled, "A Performance Comparison of Exterior Flat Finishes on Harboard Siding," which will be given by Roger Haines. Mr. Haines presented the paper in rough draft form with slides to the membership, asking for suggestions and ideas to improve his presentation.

Richard Hong, of Spencer Kellogg Div. of Textron, Inc., gave a talk entitled, "CHANGING TECHNOLOGIES FOR INDUSTRIAL COATINGS."

Mr. Hong discussed that the basis of his presentation was a questionnaire which his company had sent to 2,000 coatings manufacturers. Three-hundred and four responses were received, slightly over 15%. Mr. Hong noted that usually only 10% respond to a questionnaire of this type.

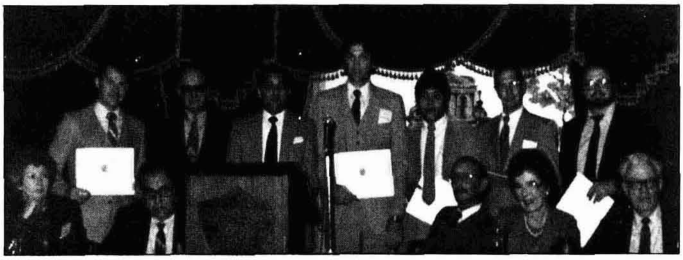
The questionnaire asked what manufacturers are doing currently, how they are meeting current regulations, plans of future compliance coatings, and problems which have to be overcome with the new technologies.

According to Mr. Hong, the survey showed 85% of the responders working on or currently supplying low VOC (volatile organic compounds) coatings. Mostly all compliance coatings produced are high solids or water-reducible systems. The polymers of choice in both water-borne and high solids air dries are alkyds and acrylics. They made up over 80% of the usage with urethanes and epoxies making up the rest. In bake systems, polyester resins appear at 20% in water-borne systems and at over 35% in high solids. Alkyds make up about 1/3 of the usage in both water-borne and high solids bake enamels. Acrylics claim 35% of water-borne and 30% of the high solids bake systems.

Mr. Hong showed slides of technologies and polymers of choice for coating plastic and also of two-pack systems.

The questionnaire asked for problems with water-borne coatings. The problems listed by most respondents were with: (1) dry time; (2) stability, and (3) application. Problems with high solids most named were: (1) application; (2) rheology, and (3) solids level.

When asked if they would consider "reactive diluents" to lower the VOC of



A.F. Voss/American Paint & Coatings Journal Award Certificates were presented to members of the LASCT Technical Committee for its first prize presentation of "Reactive Silane Modified Pigments II: A Designed Experiment in Silanized Talc/Latex Formulations" which was given at the 1982 Annual Meeting. Members of the Technical Committee include (standing left to right): Bud Jenkins, Richard Nording, *Chairman*, Dodwell De Silva, Carl Thompson, Gil Mislang, Al Seneker, and James Hall. Not present was Robert Athey, who presented the paper. Others attending the meeting were (left to right): Barbara Gildon; Secretary—Michael Gildon; President—Romer Johnson and his wife Kay; and Treasurer—Earl Smith

coatings, 76% of the respondents said "yes." When asked the same question about chlorinated solvents, only 34% said "yes," stated Mr. Hong.

A question which asked about future problems facing the coatings industries brought the following responses: (1) government regulations, (2) profit, (3) new technologies, (4) waste disposal, (5) slow growth industry, (6) lack of proper polymers, (7) end user education, and (8) poor economic conditions.

Mr. Hong showed several current formulations by generic name which are used in high solids and water-borne systems. The slides showed dry times, VOC contents, viscosity, etc.

Q. What solvents will be used to replace the ethylene glycol ether (EE) and ethylene glycol ether acetates (EE acetate)?

A. A couple of solvents are now being tested. They include ethylene glycol monopropyl ether (EP) and propylene glycol methyl ether (PM) and acetates of both of these.

Q. Can you recoat high solids enamels after overnight dry?

A. The S-K short oil alkyd has a critical recoat time, but no recoat problems have been found with the medium oil high solids alkyd resin enamels.

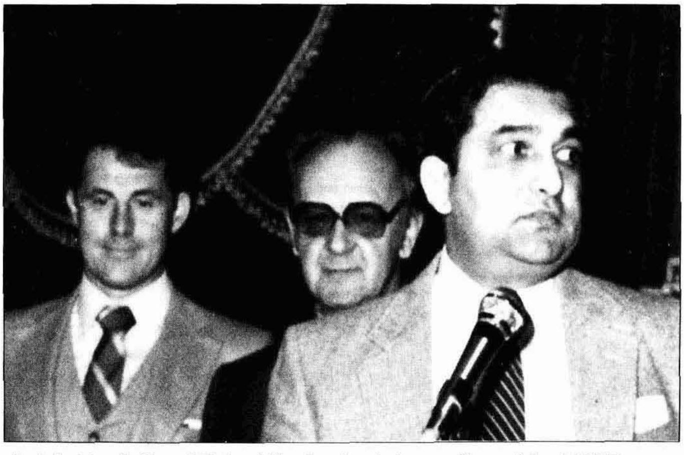
Q. What is the future of reactive diluents?

A. If the diluent were stable and stayed in the film without hurting film properties, they would be good. Unfortunately, most of the reactive diluents are not stable.

Q. Do you think chlorinated solvents are the answer for low VOC coatings?

A. Although the solvent companies say 1,1,1-trichloroethane and methylene chloride are non-hazardous, most people are afraid of them. If they do rise in usage, it will probably only be temporary.

GENE A. WAYENBERG, *Secretary*



Bud Jenkins (left) and Richard Nording (center), co-authors of the LASCT paper entitled, "Color Change with Change of Viewing Angle," which was presented at the 1982 Annual Meeting, were recognized for their merit by LASCT Technical Committee Chairman Dodwell De Silva

LOS ANGELES .... APR.

"Formulating Solvent-Based Compliance Coatings"

This meeting being Bosses Night, President Romer Johnson welcomed all bosses present.

A moment of silence was observed for Ralph Lieberman, a retired executive from Sinclair Paint and a past-president of the Los Angeles Paint and Coatings Association, who passed away recently.

Lloyd Haanstra, Environmental Chairman, reported on a consultation meeting with the staff members from different districts and ARB. The purpose of the meeting was to discuss exempt categories of Rule 1113. These exempt categories are due to expire in September of 1983, but the various districts are considering to recommend their extension to September, 1984. There are various limits suggested for categories of exempt paints. Mr. Haanstra stated that they are also changing some of the definitions which were not exactly right. In addition, there may be some additional categories for which they will probably seek exemptions. These categories cover specialty flats, quick dry primers, and enamel undercoaters. The meeting also discussed different limits for various categories for 1989. There are public hearings planned prior to changing any of the limits.

Dr. Henry George, retired society member, discussed "FORMULATING SOLVENT-BASED COMPLIANCE COATINGS."

Dr. George covered the formulation of solvent-based coatings over a wide range of metal coatings. Both air dry and baking coatings were covered, with slides shown to cover the formulations. Most of the formulations used covered the use of chlorinated hydrocarbon solvents to achieve the VOC limits necessary to comply with the latest air quality rules.

Q. Why does the home state of Dow not accept the use of chlorinated hydrocarbons as exempt?

A. This is being worked on, and will probably soon be accepted.

Q. Does the Stearic Acid on aluminum powder protect the aluminum from the chlorinated solvent?

A. It protects it for a short while.

Q. Do we expect the price of chlorinated hydrocarbons to go down?

A. No.

Q. What is the worst problem encountered with coatings made with 1-1-1 trichlorethane?

A. Reluctance by customers to use chlorinated solvents as safe.

Q. Were flash paints given in the talk open cup or closed cup?

A. They are both similar.

Q. Why do some air pollution districts in California now allow the 1,1,1, exemption to the Rule 1107?

A. It is up to the district to allow it or not.

Q. Does the stabilizer do anything else? Does reclaiming remove the stabilizer? How do you overcome this?

A. Distilling removes stabilizer. Do not use this distilled material.

Q. In high humidity areas using 1, 1, 1 TRI as the solvent system, what problems would the applicator be faced with?

A. The system may dry too fast, with a little ropy or orange peel.

Q. In aluminum based paints can methylene chloride be successfully substituted without risk?

A. Methylene chloride is probably too fast to use in most paints.

Q. The formulas covered in the talk are based on alkyds. Has any work been done with other vehicles such as chlorinated rubber or vinyls?

A. No, we have not.

MICHAEL M. GILDON, *Secretary*

NEW YORK .... MAR.

"Computers In the Coatings Industry"

Richard Max, of Synkote Paint, discussed "COMPUTERS IN THE COATINGS INDUSTRY."

The need for a computer was discussed. Mr. Max said that most companies have done without computers for a long time, but some advantages are found by investing in a computer. These included speed and accuracy. He defined a computer as a complex filling and data retrieval system with the added function of calculating and colating.

Mr. Max explained the file structure which is broken down into various categories. These categories are set up so that information can be rapidly and logically obtained. Formula explosion, formula implosion, and raw material costs can be done rapidly, said Mr. Max. Many computers can do word processing, mailing and customer list maintenance, spread sheet options, and office and business functions. Computers can check inventory, commissions, profit margins, and many other functions.

Computer hardware, which centers around a central processing unit (CPU), was explained by Mr. Max. This is the "brain" of the computer. Most CPU's have a small amount of resident memory. This memory is measured in thousands of bytes. To communicate with the CPU, some sort of external equipment is needed. In most cases this consists of a keyboard and a Cathode Ray Tube (CRT) and/or a printer. Much special equipment is also available, said Mr. Max.

Software was then discussed by Mr. Max. There are two types: (1) the operating system software which is supplied by the equipment manufacturer and (2) application software, which allows the computer to perform the functions

that you want performed. The operating system software is not modifiable. Because of this, a program that works in one computer may not work in another. Application software can be of several types. The packaged software is the most economical, said Mr. Max. There are some limitations with this type. The customized software is the most costly but should do anything and everything you want a computer to do. Inherent dangers in custom programs include guarantees by programmers than cannot be filled. Program bugs are to be expected, stressed Mr. Max. A cross between packaged and custom programming is called adaptive programming. Basically, it is a modification of an existing package. Cost is in between custom and package programs.

Factors to consider when selecting a computer were discussed. What functions do you want to computerize now as well as those functions to be computerized in the future? Not all hardware need be bought in the beginning, it can be added on. Leasing equipment is another approach, said Mr. Max. Expandability should be planned in.

Seeking professional help is important, said Mr. Max. Check vendors and contracts especially from software vendors. Look out for hidden costs. Check out repair records of vendors. Get manuals on usage. Check availability of package programs.

Implementation was explained. Mr. Max suggested that a thorough shake down be done before actual usage. Plan a gradual implementation since many people feel threatened by computers. A good idea is to run parallel systems (computer and by hand) in the beginning. Expect some breakdowns. Repeated breakdowns or errors should not be tolerated. Do not change forms from those which were done by hand.

Evaluation is important, stressed Mr. Max. If the job is being done at a cost effective price, do not worry about obsolescence. Pick a time frame for the useful life of the system and use it in your cost analysis. Plan for expansion, he said.

Using the computer for color was explained. Mr. Max said that programs for color are not generally compatible with business computers. If modified, too many compromises must be made. It is generally better to buy a separate color unit.

Q Halon fire extinguishers were mentioned. Why is this type needed?

A. This type leaves no residue; other types do. Hence, there is no damage to the equipment from an extinguished small fire.

Q. In purchasing a computer, considering the advances in technology, how do you determine what company you should go to and how do you select the right model?

A. This is like any other purchase, except most purchasers are not familiar with the item being purchased. One important item is to get impartial professional help. Another is to know just what you want to do with a computer. There are a myriad of other factors as well.

Q. When seeking professional help, are there consultants for the paint industry only?

A. Yes there are but not too many.

Q. Does it pay to hire a full-time software specialist?

A. Maybe. It depends on what you want. In most cases you only need a consultant.

MICHAEL ISKOWITZ, *Secretary*

NEW YORK .... APR.

"Comparison Between Ink and Paint"

Don Fritz, of Superior Varnish Co., discussed the "COMPARISON BETWEEN INK AND PAINT."

Mr. Fritz related the different types of inks to their nearest paint type. News and comic ink is made of carbon black and mineral oil, hence, it is similar to an oil wood stain. Metal deco ink is like a coil coating. Overprints are varnishes, said Mr. Fritz. Rotogravure is most like lacquer stains. Flexographic ink comes in two types: solvent flexo and water flexo. They are most like oil and water stains. Letterpress and lithograph inks are both like alkyd enamels.

Mr. Fritz stated that his presentation will center around letterpress and lithographic inks. Most inks can be broken down into two types: liquid and paste ink.

According to Mr. Fritz, liquid inks are pure solutions of oils, resins, and alkyds in strong solvent. They are low viscosity solutions or dispersions of resins in fast evaporating solvents. Paste inks, said Mr. Fritz, are high viscosity solutions of resins in weak, slow evaporating solvents. If a printer wants good drying, he must have fast solvent release, stated Mr. Fritz. Thus, paste inks must be formulated very close to the solution point of the resin in the solvent. Varnish resin solubility must be such that there is no resin kickout during application, but there must be a quick evaporation of solvent after application, stressed Mr. Fritz.

A semi-gloss enamel was compared to a quicksetting ink. Mr. Fritz detailed the typical formula needed for a heat setting ink: 12% pigment, 55% varnish or alkyd, 33% solvent, and no drier or extender. A typical formula for a quickset ink would be almost identical to the heatset, only driers are added. Again, no extender would be used, said Mr. Fritz. By comparison, an alkyd semi-gloss enamel would be: 12% pigment, 15% extender, 25% alkyd or varnish, 47% solvent, and 1% drier. From these numbers it is obvious the inks are operating at higher total solids with lower P.V.C. This low P.V.C. is due to the high vehicle solids, said Mr. Fritz.

The manufacture of ink vs. paint was then compared. According to Mr. Fritz, both employ high speed dispersion, but inks need finer dispersion than paint. Therefore, most inks are put through three roller mills. Grind is measured on a zero to 20 scale, whereas paint is measured on a zero to eight scale. Cleanliness is also very important. There must not be any undissolved resin in the ink.

Properties of inks were discussed. The most important properties are tack, set, penetration, and holdout. Tack is a measure of the transfer. It is the most important property of ink. The lower the ink's viscosity, the lower the tack.

Mr. Fritz discussed solvents used in inks and paints. In many cases this is comparing mineral oil to mineral spirits 66/3. Mineral oil has a much higher boiling range. The KB of mineral oil is 26 vs. 33 for mineral spirits. Flash Point of mineral oil is 220° F vs. 106° F for mineral spirits.

The characteristics of paste ink vs. paint was detailed by Mr. Fritz. Viscosity of paste ink is 200 poise vs. 10 poise for paint. Coverage of the ink is 1500 square feet per pound vs. 500 square feet per gallon for paint. Characteristics include: rheology—ink is plastic, paint is thixotropic; wet film—inks are applied to 0.5 mils vs. 4.0 mils for paint; and dry film—0.4 mils for ink, 1.5 mils for paint.

MICHAEL ISKOWITZ, *Secretary*

PACIFIC NORTHWEST .. MAR.

"Epoxy Resin Technology"

"RECENT DEVELOPMENTS IN EPOXY RESIN TECHNOLOGY" was discussed by Dr. Ron Bauer, of Shell Development Co.

Dr. Bauer spoke on recent developments in epoxy resin technology, particularly in the water-borne and high solids areas. Water-borne epoxy coatings have been developed for use in electrodeposition through reactions of the resin with para amino benzoic acid to solubilize them. Although not considered as effective as the solvent-carried 2-component epoxy systems, ambient cured waterborne epoxy coatings have been developed. High solids low bake coatings are possible by increasing the epoxy chain length to promote flexibility. For ambient cure high solids coatings, the ketimine curing agents are preferred, but high film thicknesses are difficult, stressed Dr. Bauer.

Weatherable epoxy resins have lower solution viscosities than regular epoxy resins, permitting higher resin solids in the coatings. Dr. Bauer advised a 70% volume solids level be maintained to restrict the reactivity encountered at higher levels, with the accompanying decrease in pot-life.

WILLIAM SHACKELFORD, *Secretary*

PIEDMONT .... APR.

"Five Keys to Loss Prevention In the Paint and Coatings Operations"

Jeff Fleming, of the Safety and Loss Prevention Dept. of Hercules, Incorporated, presented "FIVE KEYS TO LOSS PREVENTION IN THE PAINT AND COATINGS OPERATIONS."

Mr. Fleming stated that because of the industry we are in, handling large quantities of flammable materials, fires are inevitable. By implementing the five keys to loss prevention, the frequency of a fire occurring and the size of the loss when a fire does occur can be greatly minimized. The five keys are:

(1) Chemical Characteristics: By knowing the chemical characteristics of the raw materials, finished goods and even cleaning materials (characters such as molecular weights, vapor densities, flash points, flammable limits, ignition temperatures, and toxicities), facilities can be designed for fire prevention and safety.

(2) Segregation: Reactive materials such as nitrocellulose, amines, and caustics, should be segregated. By separating storage areas from production areas by distances and by fire walls, the extent of damage in case of a fire is minimized.

(3) Control: If the flammables and the fuel load could be kept under control or small, the chance of extinguishing a fire is very favorable. Installation of safety features such as self-closing valves, panic buttons, and interlocking flow switches with sprinkler systems, could help keep fuel loads small. In other words, they stop feeding the fire in case one is started.

(4) Eliminating Sources of Ignition: The three legs of a fire triangle are air, fuel, and heat. By eliminating one leg of this triangle, fires could be prevented, said Mr. Fleming. While removal of the air or the fuel leg is not always possible or economical, that leaves the heat leg which represents all the sources of ignition to be eliminated. The ignition sources are static electricity, static spray, struck spark, electrical, chemical reaction, and heat. Static electricity is usually related to improper handling of plastic materials such as plastic pails, shrink wraps and even plastic liners inside paper bags. Since plastics cannot be easily grounded, the use of these materials must be removed from ignitable sources as far as possible. Static spray is caused by liquid falling in space that can break up into droplets. This can be eliminated by filling from the bottom of the vessel or directly the stream to the side of the tank. Struck sparks are caused by metal to metal contact. Use of nonferrous tools can minimize struck sparks. Electrical arc can cause fire. Therefore, explosion proof electrical equipments must be periodically checked and maintained. Reactive chemicals must be segregated and the

employee must be trained to handle these materials. Overheated motors, hot bearings, and open light fixtures are some heat sources that can ignite flammable mixtures, so check to remove these heat sources from your inventory of flammables, said Mr. Fleming.

(5) Fire Protection: Fire protection must be based on the materials you are handling. Automatic sprinklers, CO_2 system, dry chemical system, and wheeled extinguishers all must be properly maintained and their use practiced by employees periodically.

When all these keys of fire protection are practiced, chances of your facility becoming a statistic in case of a fire will certainly be minimized, stated Mr. Fleming.

Philip Wong, *Secretary*

PITTSBURGH .... MAY

"SSPC—Changing Role and New Trends in the Technology of Painting Steel Structures"

The following slate of officers was elected for the 1983–84 year: President—Michael Gillen, of Van Horn, Metz & Co., Inc.; President-Elect—Clifford Schoff, PPG Industries, Inc.; Secretary—Joe Mascia, of Campbell Chemical Co.; and Treasurer—Ray Chlodney, of Puritan Paint & Oil Co.

John Keane, Executive Director of the Steel Structures Painting Council, gave a talk entitled, "SSPC—Changing Role and New Trends in the Technology of Painting Steel Structures."

Mr. Keane began his talk by pointing out that the Steel Structures Painting Council is a nonprofit research association that focuses sharply on the coating of structural steel. Its headquarters (both labs and offices) are in the Mellon Institute building of Carnegie-Mellon University.

The purposes of the SSPC are: (1) To determine best methods for surface preparation and paint application; (2) To issue specifications; (3) To do research and evaluations; and (4) To issue recommendations.

The SSPC carries out its work with a small professional staff, a number of advisory product committees composed of volunteers from industry and government, and support from a wide range of sources, including the Federation of Societies for Coatings Technology, the National Association of Corrosion Engineers, other professional and technical organizations, many paint manufacturers and users, and 25 state highway departments, stated Mr. Keane.

The major "products" of the SSPC are its publications, the best-known being the two volume "Good Painting Practice", the most recent edition.

Mr. Keane discussed some of the SSPC's current projects. He pointed out that projects always have multiple sponsors. Therefore, no single member or sponsor has to carry a heavy monetary load. He began with a description of PAGE—Performance of Alternative Coatings in the Environment, which deals with new concepts in coatings, including alternative surface preparation techniques, possible replacements for lead and chromium containing pigments, and low solvent formulations. So far, a number of interesting results has come from the project. With regard to surface preparation, wet blasting has compared favorable to dry blasting and a finer (smoother) surface profile enables coatings to perform better than does a coarse profile. None of the alternative pigments has proved superior to red lead and chromates, but zinc oxide and zinc dust/zinc oxide have come close. Attempts at reducing solvent levels have led to work on water-borne and high solids coatings, alternative solvents, and alternative vehicles. The waterborne systems have begun to approach solvent-based paints in performance, but are no better. It has been found that waterborne coatings require thicker films for adequate performance.

Mr. Keane stated that the work on surface profiles has led to a number of interesting observations and raised several questions concerning the measurement, control, and specification of surface profile. He showed Scanning Electron Microscope (SEM) pictures that illustrated a problem or defect in which small chip- or splinter-like "hackles" stick up from the surface of the steel and are a focus for failure. He also showed examples of surface debris. He pointed out that a bad profile can cause problems, but that paints can be quite forgiving. He then described the Keane-Tator surface profile comparitor which provides a series of standards to compare to a surface in order to determine the quality of its profile.

Mr. Keane discussed another project, one on painting of "weathering" steel. This special alloy was designed so that it would not need to be painted, but in highly corrosive environments even it cannot survive without additional protection. Therefore, there is considerable interest in establishing the best coating for such steel. The SSPC project has shown that certain surface preparations and zinc rich coatings can provide longer life for "weathering" steels.

Mr. Keane invited the members of the audience to attend the next SSPC Annual Meeting which will be December 5–9, 1983 in Cocoa Beach, FL. The meeting will be co-sponsored by the Federation, the Painting and Decorating Contractors Association, and the National Association of Corrosion Engineers. There also will be a tie-in with NASA and the Florida Department of Transportation. There will be a Seminar on the painting of public structures and a large number of product committee sessions.

In closing, Mr. Keane described the SSPC's plans for the future which include a broadening of services, more performance-oriented voluntary consensus specifications, more work on public structure painting and on maintenance of coatings, a membership drive, and a big push on publications. In addition, the SSPC is looking for a Director of Development to help make these plans a reality.

Q. How much of the paint on a structure is put on in the shop?

A. A first coat usually is put on in the shop, but the rest is done on the site after the structure has been completed. Most shops want to do as little painting as possible in the shop.

Q. How does the SSPC interact with ASTM? Are these organizations competitors?

A. SSPC and ASTM try not to duplicate efforts. Many people are active in both organizations and attempts are made to coordinate activities. SSPC specifications reference ASTM methods and ASTM raw material specifications. SSPC has cooperated with ASTM on developing visual standards of surface preparation and a joint standard has been issued.

Clifford Schoff, *Secretary*

ROCKY MOUNTAIN .... APR.

"Formulating a Solvent-Based Compliance Coating"

Dr. Henry George, of Coatings Technology, International, spoke on "Formulating a Solvent-Based Compliance Coating."

Dr. George said that EPA regulations have forced the chemical coatings industry to develop coatings containing low levels of VOC. These coatings are water-based, high solids and exempt solvent systems. The exempt solvent coatings use, as the main solvent, 1,1,1-trichloroethane and or methylene chloride.

Luis O. Garcia, *Secretary*

Elections

BIRMINGHAM

Active

Brown, Joe—Capricorn Chemicals Ltd., Stratford, London, Eng.

CLEVELAND

Active

Shaffer, Robert J.—Republic Steel Research Center, Cleveland, OH.
Whitlam, Douglas A.—J.C. Whitlam Mfg. Co., Wadsworth, OH.

DETROIT

Active

Bender, Howard S.—General Motors, Warren, MI.
Burdsall, Anna M.—Inmont Corp., Hamtramck, MI.
Domas, Lawrence—Fisher Body Div., GMC, Fenton, MI.
Gessner, Carolyn G.—Inmont Corp., Hamtramck.
Gessner, Michael A.—Inmont Corp., Hamtramck.
Hunter, James E.—DeSoto, Inc., Westland, MI.
Johnston, Ron J.—Wyandotte Paint Prod., Inc., Troy, MI.
Jones, David K.—Inmont Corp., Hamtramck.
Larson, Eric—DeSoto, Inc., Westland.
Spencley, Joseph T. Jr.,—DeSoto, Inc., Westland.
Vijan, Meera—Energy Conversion Devices, Madison Heights, MI.

Associate

Gentilia, John—Union Carbide Corp., Birmingham, MI.

KANSAS CITY

Active

Loboda, Frank J.—Sinclair & Valentine Div., N. Kansas City, MO.

Associate

Beardsley, Kenneth L.—Thompson-Hayward Chemical Co., Kansas City, KS.

LOS ANGELES

Active

Aye, Elizabeth—Elixer Industries, Gardena, CA.
Irvine, John—Elixer Industries, Gardena.
Nording, Toi—Elixer Industries, Gardena.
Tai, Paul J.—Pacific Dispersions Co., Inc., Cudahy, CA.
Tamura, Joe—Specialty Finishes, Cucamonga, CA.
Zywocienski, John M.—Sinclair Paint Co., Los Angeles, CA.

Associate

Baecker, H. J.—Dow Corning Corp., Irvine, CA.
Yurtis, Philip—Polyvinyl Chemicals, Ltd., Vallejo, CA.

NEW ENGLAND

Associate

Fuge, Peter K.—OMYA Inc., Proctor, VT.

NEW YORK

Active

Doss, Emile M.—Cellomer Corp., Newark, NJ.
Gasman, Robert C.—Thibaut & Walker Co., Inc., Newark.
Rapaport, Monroe—Pyrolac Corp., Hawthorne, NJ.
Werneken, Sati—Indol Magruder, Elizabeth, NJ.

Associate

Farley, Denis P.—Neville Chemical Co., Middletown, NJ.
Sheridan, Peter J.—Angus Chemical, Denville, NJ.

SOUTHERN

Active

Esquivel, Carlos—Fuller Adhesives, Inc., Pompano Beach, FL.
Lee, Billy M.—Bruning Paint Co., Pompano Beach.
Staten, Edwina C.—W. M. Barr Co., Memphis, TN.

Associate

Bradshaw, John G.—Freeport Kaolin Co., Bay Saint Louis, MS.
Lambe, James M.—Cargill, Inc., Forest Park, GA.
Rad, Eric—Southgate Marketing Assoc., Miami, FL.

Educator and Student

Gooch, Jan W.—Georgia Tech, Atlanta, GA.

TORONTO

Active

Cheung, Lisa P.—Northern Pigment Ltd., Toronto, Ont., Can.
Coates, Randy—Technical Coatings Co., Ltd., Burlington, Ont., Can.
Fanous, Raouf—Continuous Color Coat, Scarboro, Ont., Can.
King, William W.—Stegor Consultants, Cambridge, Ont., Can.
Lui, Henry—Dominion Colour Co., Ltd., Ajax, Ont., Can.
Mar, Andrew—Color Your World, Inc., Toronto.
Van Lier, Winston R.—Lepage's Limited, Bramelea, Ont., Can.

Associate

Dottori, Ernest M.—C-I-L Chemicals Inc., North York, Ont., Can.
Greason, Eric—Industrial Colours & Chemicals, Mississauga, Ont., Can.
O'Brien, Garrick P.—Canadian Construction Controls Ltd., Waterloo, Ont., Can.
Polak, Lilly—Stepan Chemical Co., Willowdale, Ont., Can.
Steinmetz, Jean X.—Kingsley & Keith (Canada) Inc., Etobicoke, Ont., Can.

FSCT Membership Anniversaries

25-Year Members

Cleveland

Herbert Herman, Retired Member.
Charles A. Ponyik, Cook Paint & Varnish Co.
William Waltz, Universal Cooperatives, Inc.

Pacific Northwest

Win Case, Cordano Chemical Co.
John H. Daller, McCloskey Varnish Co.
Reg Giltrow, General Paints & Wallcoverings.
Thomas F. Harland, Forrest Paint Co.
Harold E. Messler, Miller Paint Co.
Jay M. Mitchell, Van Waters & Rogers, Inc.
Deryk R. Pawsey, Rohm and Haas Canada Ltd.
Paul H. Payne, Norris Paint Manufacturing Co.
Robert Sailors, Longview Paint & Varnish Co.

Technical Articles in Other Publications

Compiled by the Technical Information Systems Committee—H. Skowronska, Chairman

The periodicals cited may be obtained from the listed publisher, or may be consulted at various libraries. Will you please consult the CHEMICAL ABSTRACTS SOURCE INDEX or the UNION LIST OF SERIALS / NEW SERIALS at your nearest library to find the public and/or college-university library where these periodicals are available.

Double Liaison—Chimie des Peintures (in French)

Published by Double Liaison—Chimie des Peintures, 5 Rue Etex, 75018 Paris.

Vol. 29 No. 322-323 July-August 1982

Schultz, J., Carre, A., and Simon, H.—"Determination of Surface Energy of Polymers by Wetting"; 9-14.

Potin, Ch., Pleurdeau, A., and Bruneau, CM.—"Polymers with Biocide Properties. Bibliographic Analysis"; 15-28.

Lambrechts, P. and Van der Meeren, J.—"Hydroxyalkyl Starches as Protective Colloids in the Aqueous Emulsion Polymerisation of Vinyl Acetate"; 29-33.

Vol. 29 No. 324 September 1982

Mehalla, H. and Kern, J.M.—"Determination of Unsaturation in Aromatic Hydrocarbon Resins"; 13-18.

Calbo, L.J.—"Acid Catalysts for High Solid Content Coatings and Aqueous Systems"; 29-33.

Potin, Ch., Pleurdeau, A., and Bruneau, C.M.—"Polymers with Biocide Properties. Bibliographic Analysis. Part II"; 35-43.

Vol. 29 No. 325 October 1982

Biglieri, F. and Di Paolo, V.—"Stability of Titanium Dioxide Dispersions Tested by Means of Polymer Adsorption Measurements on the Pigment Surface"; 21-26.

Bultman, D.A. and Pike, M.T.—"Use of Fluorochemical Surfactants in Floor Polishes"; 27-35.

Piens, M.—"Electrochemical Studies on Anticorrosion Coatings. An Evolution Towards an Analysis of the Behavior of Coated Metals"; 39-45.

Van Rossum, E.C.E.—"Liability Coming from Products and its Consequences for the Paint and Ink Industry"; 52-55.

Vol. 29 No. 326 November 1982

Durand, D., Helias, P., and Bruneau, CM—"Intrinsic Reactivity and Statistical Description of Three-dimensional Polymer Condensations. Part I: Study of the Pre-gellification Phase"; 13-18.

Carter, E.—"Micaceous Iron Oxide Paints"; 19-27.

Vol. 29 No. 327 December 1982

Durand, D., Helias, P., and Bruneau, C.M.—"Intrinsic Reactivity and Statistical Description of Three-dimensional Polymer Condensations. Part II: Study of the Post-gellification Phase"; 13-21.

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Published by Curt R. Vincentz Verlag, Schiffgraben 41-43, Postfach 6247, 3000 Hannover 1.

Vol. 88 No. 11 November 1982

Dörner, K.H.—"Credit Insurance and Control"; 905-906.

Penczek, P.—"Glass Transition Temperature of Polyesterimide Paint Films"; 907-909.

Kirchmayr, R., Berner, G., Hüsler, R., and Rist, G.—"Non-yellowing Photoinitiators"; 910-916.

Breinsberger, J. and Koppelmann, J.—"Performance of Three-Coat Finishes in the Stone Impact Test"; 916-920.

Brushwell, W.—"Paints and Adhesives"; 920-924.

Raths, S.—"Wet Grinding with Micro-elements in a Double Chamber Attritor"; 925-928.

Murjahn, K.—"How Will the Market for Varnishes and Emulsion Paints Develop?"; 953-958.

Skotarek, H.J.—"In-House Solvent Recovery—Yes or No?"; 964-965.

Vol. 88 No. 12 December 1982

Stachowiak, S.A.—"Rheology of Epoxy Resin Powders"; 996-1000.

Bagda, E. and Lipp, G.—"Mechanics of Crack-Bridging"; 1000-1003.

Häring, E., Reh, J., and Sonnenberg, K.H.—"Experience with Electron Beam Curing Wheel Finishes. A New Application for a Radiation Chemistry Technique"; 1004-1009.

Wolski, W. and Wolniewicz, A.—"Processing of Iron Oxide Yellow for Manufacturing Magnetic Tape"; 1009-1017.

Anon.—"Process Computer Controlled Synthetic Resin Production Improves Reproducibility"; 1017-1021.

Brushwell, W.—"Paint Technology in the CMEA States"; 1022-1026.

Wamsley, S.H.—"European Paint Industry Now and in the Future"; 1045-1050.

Ritter, H.—"Rationalisation of Plant Operation and Environmental Protection at Herberts GmbH in Wuppertal"; 1050-1052.

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Kneifel, A.—"Stone Impact Testing of Coatings at the German Federal Railways"; 6-8.

Matejicek, A., Kaska, J., Eichler, J., and Horky, J.—"Grinding Coating Powders in Special Ball Mills"; 9-13.

Schwegmann, B.—"Toners in Finished Goods Manufacture"; 13-15.

Brushwell, W.—"Experimental Paint Samples—Tomorrow's Standard Formulations?"; 15-19.

German Standard Draft *DIN 53 167:* Testing of Paints, Varnishes and Similar Coating Materials; Salt Spray Test on Paint Coatings and Similar Coatings *and* German Standard Draft *DIN 6713:* Paints and Varnishes; Preparation of Acid Extracts from Paints in Liquid or Powder Form . . . . 20-29.

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Funke, W.—"Environmentally Acceptable Corrosion Protection with Organic Coatings—Problems and Potential"; 86-91.

Klug, L.—"Measurement of Dynamic Properties on Insulated Cold-Hardening Lacquer Films During the Curing Process"; 92-98.

Mazura, J., Eichler, J., Mach, M., and Cermak, C.—"Recording of Parameters During UV-Curing, Using the Simplex Method and Regression Analysis"; 99-102.

Mowat, D.—"DBE—A Versatile Solvent"; 102-103.

Brushwell, W.—"Environmental Control and Job Safety for Paint Production and Application"; 103-105.

German Standard Draft DIN 53 217 Part 3: Testing of Paints, Varnishes and Similar Coating Materials; Determination of Density; Immersed Body . . . 106-109.
Schnabel, W.—"Paint and Lacquer Compounding—Nozzle Technology in Mixing"; 133-134.

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Ondratschek, D. and Thomer, K.W.—"Effect of Processing Parameters on Colour Variations of Paint Films in Compressed Air Paint Spraying"; 162-166.
Besold, R.—"Novel Anti-Corrosion Systems Based on Lamellar Zinc Pigments", 167-173.
Premkumar, N., Sathyanarayana, M.N., Balakrishiva, R.S., Shirsalka, M.M., and Sivasamban, M.A.—"Factors Influencing the Adhesion of Aqueous Alkyd Resin Systems on Steel Plate. Effect of the Melamine Resin Component of the Alkyd and the Level of Pigmentation"; 174-177.
Schmidt, H.—"Formulating and Correcting Paints Inexpensively"; 177.
Brushwell, W.—"Formulation and Applications of Elastomeric Coatings"; 178-182.
Anon.—"BASF Workshop on Printing Inks"; 182-184.

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Winkler, J. and Dulog, L.—"Flocculation Characteristics of Pigments in High Solids Systems"; 236-242.
Beranek, E.—"Selection of Paint Test Methods in Relation to Their Importance in Quality Measurement"; 243-247.
Mielke, W. and Sickfeld, J.—"Thermogravimetric Investigation of Epoxy Resins"; 247-252.
Braun, F.—"Investigation of Offset Inks—Influence of Pigments on Emulsion Formation"; 253-259.
Brushwell, W.—"Papers and Technical Books on Paint Developments"; 259-264.
Heikel, A.R.—"Tinting Systems Increase in Importance"; 304-306.
Anon.—"Packaging Line for Plastic Containers at Sigma Coatings BV, Uithoorn, Netherlands"; 306-308.

Journal of the Oil and Colour Chemists' Association

Published by Oil and Colour Chemists' Association, Priory House, 967 Harrow Rd., Wembley, Middlesex, HAO 2SF England.

Vol. 65 No. 11 November 1982

Raju, K.V.S.N. and Yaseen, M.—"Effect of Part Replacement of Titanium Dioxide by Barytes on the Properties of Alkyd Paints"; 401-408.
Saxena, M.S., Jain, P.K., and Vasishtha, A.K.—"Styrene Copolymerisation of Isomerised Tobacco Seed (Nicotiana tobacum) Oil and its Alkyd"; 409-414.
Walker, P.—"Organosilanes as Adhesion Promoters for Organic Coatings. Part 1: Silanes on the Metal Surface"; 415-423.

Vol. 65 No. 12 December 1982

Jaycock, M.L. and Kayem, G.J.—"Stability and Flocculation in a Latex Paint. Part 1. Influence of Dispersants on Particle Stability"; 431-435.
Walker, P.—"Organosilanes as Adhesion Promoters for Organic Coatings. Part 2: Silanes in Paints"; 436-443.
White, A.D.—"The Future of the Refinishing Industry"; 444-447.

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Agrawal, D. and Vasishtha, A.K.—"Novolac-based Soya Epoxy Esters for Surface Coatings"; 2-5.
Caprari, J.J., Pressa, H.R., delAmo, B., and Lasquibar, C.—"High Build Paints Based on Chlorinated Rubber / Coal Tar Pitch Binders"; 6-11.
Jaycock, M.J. and Kayem, G.J.—"Stability and Flocculation in a Latex Paint. Part 2: Flocculation in the Drying Paint Film"; 11-17.
Tyler, T.J.C.—"Refinishing Over the Next Ten Years"; 17-19.

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Bravery, A.F., Barry, S., and Worley, W.—"An Alternative Method for Testing the Mould Resistance of Paint Films"; 39-43.
Manley, T.R. and Scurr, G.—"Use of DTA to Study the UV Curing of Acrylic Resins"; 43-48.
Narain, S., Bonanos, N., and Hocking, M.G.—"AC Impedance Measurements on Thick Organic Coatings on Mild Steel Substrates"; 48-52.
Sprecher, N.—"Flakeglas-filled Coatings: Past, Present, and Future"; 52-54.

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Sitaramam, B.S., Chatterjee, P.C., and Sivasamban, M.A.—"Copolymerisation of Polyunsaturated Fatty Vinyl Monomers with Styrene"; 86-87.
Mills, D.J. and Mayne, J.E.O.—"Structural Changes in Polymer Films. Part 2: Effect of the Pigment Zinc Oxide on the Electrolytic Resistance of Transition Temperature; Some Comparisons with the Pigment Iron Oxide and Red Lead"; 88-93.
Callow, L.M. and Scantlebury, J.D.—"Electrochemical Impedance in Coated Metal Electrodes. Part 5: Film Thickness"; 93-96.

Meetings/Education

Pacific Northwest Society Attracts 178 to Annual May Symposium

An attendance of 178 (128 men, 50 ladies) was recorded for the Annual Symposium of the Pacific Northwest Society, May 5–7, in Portland, OR.

Jerry McKnight, of Lilly Industrial Coatings, headed the Symposium Committee. He was assisted by: Roy Blackburn, Bruce Henshaw, Bob Howard, Jim Riedel, Scott Shannon, Gary Welp, Allison King, Steve Rearden, Dennis Lakin, Bill Cordano, John Daller, and Steve Norton, the Society President.

The technical program was comprised of the following presentations:

"Comparative Solvent Evaporation Mechanisms for Conventional and High Solids Coatings"—Bill Ellis, of Chevron Research Co.

"Two-Component Waterborne Epoxy Materials for Maintenance Coatings"—Jack Avery, of Celanese Plastics & Specialties Co.

"Practical Applications of Computer Color Matching"—Larry Backus, of Macbeth Div., Kollmorgen Corp.

"Quality and Consistency from an Easier Dispersing Rheological Additive"—Alfred Whitton, of NL Industries.

"Waterborne Acrylic and Urethane Resins in Industrial Finishes"—John Fitzwater, of Polyvinyl Chemicals.

"Effects of Metal Catalysis in Water and High Solids Systems"—Robert Middlemiss, of Mooney Chemicals.

"Horizontal Media Mills"—Myron Segal, of Premier Mill Corp.

"Propylene Based Glycol-Ethers and Acetates in Coatings"—Dr. Mark Adamchuck, of Arco Chemical Co.

"High Solids Coatings Are Here"—Robert Price, of Spencer Kellogg Div.

Highlights from the annual business meeting were:

(1) Total membership passed the 200 mark (136 Active, 56 Associate, and ten from other classes).

(2) Curt Bailey, Chairman, announced that the History and By-Laws booklet will be published in the fall.

(3) The Society will support the New England Society in the preservation of the Boston Stone.

(4) An educational course for all members from the three sections is being considered.

(5) The 1984 symposium will be held May 3–5, at the Park Hilton Hotel in Seattle.

(6) The 1985 symposium will be at the Empress Hotel, Victoria, B.C., April 25–27.

(7) The slate of officers elected for the coming year are: President—Bob Hogg, of Preservative Paint Co.; President-Elect—Ottwin Schmidt, of Shanahan's Ltd.; Secretary—Jerry McKnight, of Lilly Industrial Coatings; Treasurer—Dennis Hatfield, of J.F. Shelton Co.; and Society Representative—Deryk Pawsey, of Rohm and Haas Co. Bill Shackelford, of Gaco-Western, Inc., was reappointed Administrative Secretary.

At the annual banquet, Paul H. Payne, of Norris Paint Co., was honored with the 1983 James A. Leider, Jr. Outstanding Service Award.

The "international" sports competition was a beach ball basketball game. The Seattle Section won handily.

CALL FOR PAPERS AND DISPLAYS
Cleveland Manufacturing Committees Symposium
"Tools for Tomorrow: Increased Profitability Thru Innovative Manufacturing Concepts"
January 17–18, 1984

An invitation has been issued for companies and individuals' to participate in a joint table top show and symposium scheduled for January 17–18, 1984 to be sponsored by the Manufacturing Committees of the Cleveland Society for Coatings Technology and the Cleveland Paint and Coatings Association.

The theme of the meeting will be "Tools for Tomorrow: Increased Profitability Thru Innovative Manufacturing Concepts."

Papers or displays relating to innovations or innovative processes in materials, materials handling, processing, testing, and packaging will be of interest.

Interested persons should contact: (for table top display)—Richard Shannon Jr., Jamestown Paint & Varnish Co., 108 Main St., Jamestown, PA 16134; (for symposium papers)—Katherine Mihalak, Glidden Coatings & Resins Div., SCM Corp., D.P. Joyce Research Center, P.O. Box 8827, Strongsville, OH 44136.

Golden Gate Society Accepting Nominations For Apfel Scholarships

Nominations are being accepted by the Golden Gate Society for Coatings Technology for two Alfred Apfel Memorial Scholarships of $1,000 each at college entry level for September 1983.

To be eligible, a scholarship applicant must be of a family in which at least one member is a fully-paid member of the society or of the GGPCA. An official application and supporting documents must be submitted on or before July 15. The applicant's major or vocational interest must be demonstrated to be viable to the paint and coatings industry.

For information, contact Educational Committee Chairman, Ted Favata, Triangle Paint Co., Inc., 2222 Third St., Berkeley, CA 94710.

Philadelphia Society Sponsors 'Substrate Technology' Seminar

The Philadelphia Society for Coatings Technology sponsored a one-day seminar entitled, "Substrate Technology," on May 16, at the Philadelphia College of Textiles and Science, Philadelphia, PA.

The following topics and speakers were featured:

METALS: Robert Cassel, Amchem Products.

PLASTICS: Jim Beauregard, General Electric Co.

WOODS: Dr. Winfred Cote, State University of New York College of Environmental Science and Forestry.

CEMENT: Bryon Follin, E.I. du Pont de Nemours & Co., Inc.

MARINE SUBSTRATE: Speaker from International Paint Co.

SUBSTRATE ECONOMICS: J.W. Prane, Consultant.

Seminar Chairman was Robert C. Sonntag, of Superior Varnish & Drier Co.

SSPC to Hold 'Painting Public Structures' Dec. 7–8, Cocoa Beach, FL

The Steel Structures Painting Council will sponsor a symposium entitled, "Painting Public Structures: Practices and Economics," on December 7–8 at Cocoa Beach, FL.

The symposium is designed to address the problems created by deferred maintenance of such public structures as bridges, transportation facilities, and water and sewage plants. It will stress fundamentals of administration and technology for a protective coatings program.

Issues to be covered include establishing effective maintenance painting programs, cost-effectiveness, preparing specifications, procurement practices, quality control, selecting a contractor, surface preparation for maintenance painting, paint application procedures, inspection, and others.

A special feature of the seminar will be two papers describing the Florida Department of Transportation's Bridge Paint Qualification Test and NASA's Zinc-Rich Paint Exposure Test. On Friday, December 9, there will be a tour of test exposure sites.

A preliminary program with topics and speakers is as follows:

"Establishing a Maintenance Painting Program"—John Padberg, Port Authority of NY and NJ.

"Selecting a Contractor"—Norman Strauss, Painting and Decorating Contractors of America.

"Paint Failure Analysis"—Dr. Richard Drisko, Naval Construction Center.

"Quantitative Evaluation of Coatings Failure"—Dr. Jonathan Martin, National Bureau of Standards.

"Surface Preparation for Maintenance Painting"—John Peart, Avondale Shipyards.

"Painting Systems"—Charles Dauderman, Degraco Coatings.

"Economics of Zinc-Rich Painting Systems"—Hugh Morrow, Zinc Institute.

"Surface Preparation for Maintenance Painting"—Dr. Bernard Appleman, Exxon.

"Paint Tests for a Qualified Products List"—Richard Ramsey, Florida Dept. of Transportation.

"Cost Effectiveness of Quality Assurance Programs"—Thomas J. Bauer, Imperial Professional Coatings.

"FED/MIL Specs or Non-Government Standards for DOD Contracts?"—Lee E. Rogers, Office of the Under Secretary of Defense—Research Engineering.

"The NASA Water-Base Technology: Application and Economics"—Parke Schaffer, Jr., Inorganic Coatings, Inc.

"Preparing Effective Specifications"—G.P. Mitchell, Sigma Coatings, Inc.

"Inspection"—Gene Hult, URS Corp.

"Fusion Bonded Coatings"—Michael S. Stenko, MCP Facilities Corp.

"Concept of Painting Systems" Thomas A. Calzone, Carboline Co.

"Total Shop Painting of Structures"—Gary Tinklenberg, Michigan Dept. of Transportation.

"Abrasive Reclamation—Economics and Facilities"—H.M. Hedrick, Apache Abrasives.

"What Is Inspection Worth?"—Walter W. Kaminski, E.I. du Pont de Nemours & Co., Inc.

For more information, contact *Symposium Coordinator,* Dr. Harold E. Hower, SSPC, 4400 Fifth Ave., Pittsburgh, PA 15213.

AFP/SME Announces RAD/CUR Workshop Sept. 27–29

The Association for Finishing Processes of the Society of Manufacturing Engineers (AFP/SME) has announced a workshop on "Radiation Curing in the Printing and Converting Industry," September 27–29, 1983, at the Hershey Hotel, Philadelphia, PA.

Radiation curing, a major process technology in the printing and converting industries, encompasses electron beam (EB), ultraviolet (UV), and infra red (IR) energy for the polymerization and curing in inks, coatings, and adhesives. This applied technology will be the theme of the workshop along with in-depth coverage of economics, productivity, energy usage, and product performance.

Industry specialists will address these topics and successful practitioners will serve as group leaders of sessions that will concentrate on specific application methods and curing technologies.

Sessions include fundamentals of radiation curing, introduction to radiation curing, in-depth update of raw materials and applications, an overall report on uses of radiation curing, energy sources, application equipment, inks and varnishes, and coatings.

Chairpersons are Martin Kaufman, of Gotham Ink & Color Co., Inc., and David Davis, of Cellofilm Corp.

For further information, contact Donna Theisen, Administrator, Technical Activities Dept., SME, One SME Dr., P.O. Box 930, Dearborn, MI 48121.

ACS Coatings and Plastics Division Renamed Division of Polymeric Materials: Science and Engineering

The American Chemical Society's Division of Organic Coatings and Plastics Chemistry has changed its name to the Division of Polymeric Materials: Science and Engineering.

The Division was founded in 1924 as the Paint and Varnish Division. Over the years, the interests of the Division's members have come to include adhesives, biomedical polymers, composites, electronic application of polymers, and many other areas of applied polymer science and technology. This diversity of interest has long been evident in the symposia sponsored by the Division; the new Division name has been chosen to better reflect program content.

For many years, the Division has participated in joint and cosponsorship of symposia with other ACS Divisions including the Division of Polymer Chemistry, and has been an active participant in the Macromolecular Secretariat. These activities of the Division will not be affected by the change in name.

Proceedings of the Division's meetings have been published as preprints for over 40 years. This service to Division members and the technical community will continue. The preprint books will have a new name, *Polymeric Materials Science and Engineering,* and will continue to be sent to Division members as one of the benefits of Division membership. Back issues and library subscriptions will continue to be handled by ACS Books and Journals.

Awards administered by the Division are the Arthur K. Doolittle Award and the ACS Award in Applied Polymer Science. The Doolittle Award is given annually for the best paper presented in the Division's meetings. The ACS Award in Applied Polymer Science was established in 1981 by the Phillips Petroleum Company to recognize and encourage outstanding achievements in the science or technology of plastics, coatings, polymer composites, and related fields.

Further information on the Division, including an application for membership, can be obtained from the Division Secretary, Ray Dickie, Scientific Research Laboratories E3198, Ford Motor Co., Box 2053, Dearborn, MI 48121.

People

Saul Spindel, President of D/L Laboratories, New York, NY, was presented with the New York Society's PaVac Award "in recognition of his many years of unselfish devotion to the New York Society and his technical developments and educational efforts which have contributed to the advancement of The Protective Coatings Industry."

Presenting the award was 1983 PaVac Lecturer Dr. S. Peter Pappas, of North Dakota State University. The title of his presentation was "Low-Energy Curing, Physical Organic Chemistry and Creativity."

Mr. Spindel began his career in the coatings industry in 1947 at E.M. Wanderman & Co. From 1948 thru 1959 he worked for the Clover Leaf Paint and Varnish Co. In 1959 he joined the D/L Laboratories, then known as the David Litter Laboratories, starting as a Research Chemist, and after various promotions, attained his present position as President.

He joined the New York Society in 1958 and, shortly thereafter, became a member of its Education Committee. Subsequently he became a member of the Board of Directors of the Society and increased his participation in Society activities, serving on various committees, including the Technical Committee, Program Committee and Joint Coordinating Committee. Mr. Spindel was elected as Society Treasurer in 1976 and, after going through the chairs of office, served as President in 1979.

He has served the Federation in various capacities and is presently Chairman of the Federation Corrosion Committee as well as a member of the Technical Advisory Committee and the Nominating Committee. He has also served as a member of the Federation Finance Committee. Additionally, because he is the New York Society Representative, he is a member of the Federation Board of Directors.

He has published, as author and coauthor, over a dozen articles on the use, testing, and performance of all types of coatings and coatings materials. Additionally, he is a contributing author to the recent (1982) revision of the "Steel Structures Painting Manual."

He is active in the American Society for Testing and Materials, serving as a member of the Executive Committee of ASTM C-24. He is also a member of the National Association of Corrosion Engineers and the American Chemical Society.

S. Spindel

F. Daniel

J. R. Pickering

H. Leidheiser, Jr.

Twenty-seven years ago **Fred Daniel** abandoned a consulting business to venture into manufacturing pigment dispersions and additives for the coatings, ink, and allied industries. Five years ago he sold his company, Daniel Products Co., but remained active in its management until recently. Not being ready yet to retire to a life of technical writing, he has resumed his consulting career with emphasis on productivity improvement, process engineering, and technical or commercial management problems of the coatings industry. Mr. Daniel's office and home are located at 37 Constitution Hill, Princeton, NJ 08540. Mr. Daniel is a New York Society member.

J.R. Pickering has been re-elected President of the National Coil Coaters Association. He is Chairman of the Board at Lilly Industrial Coatings, Inc., Indianapolis, IN.

Three other officers were also reelected. They are: Vice-Presidents—**James S. Smith,** of Roll Coater, Inc., and **William A. Boyd,** of Vail Rubber Products Corp.; and Treasurer—**John H. Geyer,** of Amchem Products, Inc.

Paul H. Payne, of Norris Paint Co., Salem, OR, was presented the Pacific Northwest Society's James A. Leider, Jr. Outstanding Service Award during the Society's annual symposium on May 7. Mr. Payne, a Past-President, has been a long-time spearhead of educational activities for the Society and active in Federation educational matters as well.

Washburn-Linder Co., Inc., Framingham, MA, has announced the appointment of **Cal R. Wiegers** as Vice-President—Sales. Mr. Wiegers is a member of the New England Society.

Dr. Henry Leidheiser, Jr., Professor of Chemistry and Director of Lehigh University's Center for Surface and Coatings Research since 1968, has been named to the Aloca Foundation Professorship at Lehigh.

Dr. Leidheiser, who was recently honored with the prestigious Willis Rodney Whitney Award, presented by the National Association of Corrosion Engineers, has conducted an internationally-recognized program of study in the science of corrosion and corrosion control.

In addition to other pioneering research efforts, Dr. Leidheiser was among the first to pursue the application of impedance methods to predict the performance of painted metals and was an early leader in the use of Mossbauer spectroscopy in research directed toward a better understanding of corrosion. His use of emission Mossbauer spectroscopy for studies of passivation and for nondestructive studies of metal-polymer interfaces have been described as unique and classic applications.

Dr. Leidheiser has written more than 150 published articles and papers and is author or editor of five books. His contribution to surface and corrosion science, represented by these publications, are widely recognized and have led to a number of previous honors from organizations such as the Electrochemical Society, the Oak Ridge Institute of Nuclear Studies, the Institute of Metal Finishing, the Electroplaters' Society, and the Society of Automotive Engineers.

Advanced Coatings & Chemicals, Temple City, CA, has announced the appointment of **Dion Peralta** to Vice-President and Technical Director. An employee in the paint industry for 27 years, Mr. Peralta is a member of the Los Angeles Society.

Telford C. Wollan has been appointed Special Products Manager for the wood and general metals product lines of Reliance Universal Inc., Zion, IL. A 30-year employee of the firm, Mr. Wollan has served in both technical and sales capacities. Mr. Wollan is a member of the Chicago Society.

Also, Reliance has announced the promotion of **Victor Kurk** to the position of Technical Director. Mr. Kurk previously served as Group Leader responsible for the metal labs. He is a member of the Louisville Society.

The Flecto Co., Inc., Oakland, CA, has announced the appointment of **Alfredo J. Guevarra** to the position of Senior Chemist in the Research and Development Department. Prior to joining the firm, Mr. Guevarra had been associated with The O'Brien Corp. for 28 years. He is a member of the Golden Gate Society.

Celanese Specialty Resins, Louisville, KY, has announced the promotion of **David A. Shimp** to the position of Senior Research Associate at its Technical Center. He will be responsible for research and exploratory development for new resins systems. Since he began his career as a research chemist in 1957, Mr. Shimp has held various research and development positions with Celanese. He has published five technical articles, has written 42 technical bulletins, and has received 15 U.S. patents. Mr. Shimp is a member of the Louisville Society.

The Midland Div. of the Dexter Corp. has announced the following appointments. Promoted to the positions of Operations Managers were **Lee Soule** and **Barry E. Bromstead,** based at the Rocky Hill, CT plant and at the Waukegan, IL operations, respectively. **C.D. Mitchell** was assigned to the position of Division Controller and **Robert A. Graham** was named Sales Manager of Building Products. Mr. Graham is a Cleveland Society member.

The Color Marketing Group has elected the following officers for the 1983 year. Elected to the position of President is **Jim Brown,** of Charlotte, NC, who has been involved in the floor covering and home furnishings industries and presently owns his own firm, Jim Brown Associates. Named as Vice-President is **Joyce Davenport,** Supervisor, Color Development, for DeSoto, Inc. Ms. Davenport is a member of the Chicago Society. Everett R. Call is CMG Executive Vice-President.

John M. Hafeli has announced his retirement with Glidden Coatings & Resins, Div. of SCM Corp., after 48 years in the coatings industry. Mr. Hafeli had most recently served the firm as National Accounts Manager—Metal Decorating of Glidden Chemical Coatings. A 22-year Glidden employee, Mr. Hafeli had also been associated with Berry Brothers, Inc., and Reichhold Chemicals, Inc.

Mr. Hafeli is a Past-President of the Detroit Society and is a Society Honorary Member. He has served on the Board of the CPCA, has been a past-president and board member of the Metal Decorating Suppliers Guild, and has been associated with the National Metal Decorators Association and the American Chemical Society.

DeSoto's 1982 "Researcher of the Year" awards were presented to two research teams for their outstanding contributions to new product development.

Kenneth Jasenof, Art Jimenez, and **Edward Zychowski** received honors for their efforts in the development of Acropon II,® a coil-applied building products coating. Messrs. Jasenof and Jimenez are members of the Chicago Society.

The research team of **Robert Zable** and **Gene Michal** also received honors for their development of Aquaform®, a coil-applied enamel for beer and beverage easy open ends. Mr. Zable is a Chicago Society member.

James W. Gracie has been named Market Applications Manager of the Chemicals Div. of J.M. Huber Corp., Havre de Grace, MD. Prior to joining the firm, Mr. Gracie was associated with W.R. Grace & Co., Davison Div. He is a Baltimore Society member.

Raybo Chemical Co., Huntingdon, WV, has announced the promotion of **Howard B. Swanson Jr.** to Vice-President and Technical Director. Prior to his appointment with Raybo, Mr. Swanson was employed by Hughson Chemical Div. of Lord Corp. Mr. Swanson is a C-D-I-C Society member.

Edwin C. Hill has joined Ed Simal & Associates, Atlanta, GA, as Vice-President and Associate. Prior to his appointment, Mr. Hill had been associated with NL Industries, Inc. He is a Southern Society member.

W. Jack Young has been appointed Sales Manager for Morehouse Industries, Inc., Fullerton, CA.

PPG Industries, Inc., Pittsburgh, PA has announced several new appointments. **Malcolm G. Slaney** has been elected Group Vice-President, Coatings and Resins. Prior to his appointment, Mr. Slaney had been Vice-President and General Manager of the firm's coatings and resins div. He joined PPG in 1963 as Assistant to the general sales manager of industrial finishes and two years later became Market Manager of appliance finishes. In 1972, Mr. Slaney was appointed Manager of the resins product group, and three years later he was named Assistant Manager of industrial products. He was elected Vice-President of industrial products in 1976.

Also announced by the firm, was the appointment of **Robert D. Duncan** to the position of Vice-President and General Manager of the Industrial Chemical Div. Mr. Duncan had served as Vice-President, Industrial Chemicals—U.S.

Dr. Clifford K. Schoff, of PPG Industries, Inc., was presented with the Cleveland Society's Outstanding Speaker Award at the Society's 26th Annual Technical Conference. He was recognized for his paper, co-authored with Percy E. Pierce, of PPG, entitled, "The Effects of Surface Tension and Viscosity on Surface Defects in Coatings." Dr. Schoff is Secretary of the Pittsburgh Society.

Shamrock Chemicals Corp., Newark, NJ, has announced the appointment of **Alan I. Kalmikoff** to the position of National Sales Manager/Coatings. Mr. Kalmikoff, who has been with the firm for two years, was previously employed as Midwestern Sales Representative. He is a member of the Chicago Society.

Dr. Alfred Rudin, FCIC, professor of chemistry at the University of Waterloo, Waterloo, Ontario, Canada, was presented with the 1983 Protective Coatings Award of The Chemical Institute of Canada. The award consists of a $1,000 honorarium and a scroll and is given in recognition of outstanding contributions to the science and art of organic coatings. Dr. Rudin, a faculty member of the University since 1967, is involved in many areas of polymer research including membrane separation processes, synthetic fibers, and solid-state NMR of polymers. With colleagues at the University, Dr. Rudin is currently developing a method for accurate determination of the size and distribution of polymer molecules.

StanChem, Inc., East Berline, CT, has appointed **William A. Rains** as Vice-President and Technical Director.

Two new sales representatives have been appointed by Chemical Coatings Div. of The Sherwin-Williams Co. to serve finishers of metal substrates in the Los Angeles area. Based at the division's City of Commerce center are **Heidi Schroeder** and **Richard Stalker.** Ms. Schroeder is assigned to business development of industrial coatings for the area including independently owned "job shops." Responsible for the development of the division's general metal finishing business for original equipment manufacturers (OEM's) in the area is Mr. Stalker.

Robert A. Katherine has been named Chairman of the Board of McCloskey Varnish Co., Philadelphia, PA. Mr. Katherine has served the firm as President.

Carboline Co., St. Louis, MO, has announced the promotion of **Mike Hasser** to Corporate Director of Quality responsible for all facets of corporate activities relative to the consistent formulation, production, and sale of quality products. Mr. Hasser previously served as plant manager of the firm's Xenia, OH facility.

Nalco Chemical Co., Oak Brook, IL, has announced the following appointments. **W. Steven Weeber** has been promoted to the position of Vice-President, Nalco Europe within its International Div. Previously, Mr. Weeber was Vice-President, Marketing for the International Div. Replacing Mr. Weeber in that position is **Milford B. Harp,** previously Vice-President, Marketing, Industrial Div. **A.J. Fox** has been promoted to the position of Manager, International Executive Accounts. Prior to this appointment, Mr. Fox had served as Executive Accounts Manager in the division. Also promoted was **Earle E. Allen** to the position of Vice-President, Marketing, Industrial Div. He had previously served as Marketing Manager to Water Treatment Chemicals. Also announced by the firm was the transfer of **James L. Castle** to Market Manager, Water Clarification and Pollution Control, Industrial Div. Prior to his transfer, Mr. Castle was Regional Manager in the Water Treatment Chemicals Group.

Robert E. Davis was elected President and Chief Operating Officer of Sun Chemical Corp., New York, NY. Prior to his appointment, Mr. Davis had been President and Chief Operating Officer of Morton Thiokol, Inc., the chemical company formed by the merger of Thiokol Corp. and Morton Norwich Products, Inc.

Air Products and Chemicals, Inc., Allentown, PA, has appointed **John M. Tacca** as Development Specialist for the company's Performance Chemicals Div.

Deane A. Anderson was appointed Vice-President of Russell Finex, Inc., Mount Vernon, NY. In addition, Mr. Anderson will continue as National Sales Manager responsible for sales, marketing, and advertising.

The appointment of **Stewart E. Gloyer** as Vice-President of Research and Development for the Humko Chemical Div. was announced by Witco Chemical Corp., New York, NY.

Henkel Corp. has named **Dr. Hans-Heinrich Guertler** as Executive Vice-President with responsibility for the Chemical Products Div.

Interstab Chemicals, Inc., New Brunswick, NJ, has appointed **Donald G. Hampson** to the position of General Manager of its PVC Additives Group, and **Borys Kostyszyn** as General Manager of Thermoset Processing Chemicals.

Earl F. Leach has joined Surface Treatments, Inc., a subsidiary of Widger Chemical Corp., as Manager of Sales to the metal forming industry.

John D. Hale has been promoted to Vice-President—Technical Operation for Kerr-McGee Chemical Corp., Oklahoma City, OK. He will be responsible for development and application of new or improved technologies in the chemical mining and processing, mineral benefication, electrochemistry, and pigment-finishing operations. An employee of the firm since 1965, Mr. Hale formerly served as Manager of its Technical Center.

A reorganization of the Chemical Manufacturers Association's Government Relations Department has been announced. In the reorganization, **Myron T. Foveauz** and **Timothy F. Burns** have been named Assistant Directors of Government Relations. Mr. Foveaux will head energy, international trade, taxation, and transportation. Mr. Burns will direct efforts in the areas of health, safety, and environmental concern.

Dave W. Baumgart has joined the sales force for Chemcentral/Toledo. He will be responsible for sales in the Northwest corner of Ohio and a portion of the Toledo territory.

B. Duane Marsh has been appointed Vice-President of Technology in the newly-formed Specialty Polymers and Composites Div. of Union Carbide Corp., Danbury, CT. Dr. Marsh joined the firm in 1967 as a Senior Engineer in the Chemicals and Plastics Div. and has held various positions, most recently serving as General Manager, Research and Development in the Carbon Products Div.

Obituary

Willard W. Vasterling, Sr., 77, Past-President of the Federation of Societies for Coatings Technology (1969–70), died on June 10.

Mr. Vasterling was associated with the paint industry for 62 years. He began his career with Phelan-Faust Paint Manufacturing Co. and was later associated with the Glidden Co. From 1935–1972, Mr. Vasterling was employed with Davis Paint Co. and served as Executive Vice-President at the time of his retirement from that company. Since 1973, Mr. Vasterling had been with the Morton Myers Co.

Mr. Vasterling served as President of both the St. Louis and Kansas City Societies. He acted as the Kansas City Society Representative to the Federation Council and was elected into Society Honorary Membership. He was also a past-president of the Kansas City Paint and Coatings Association and was awarded honorary membership into the association.

He is survived by his wife and two children.

Nancy Iran Phillips, 54, a freelance writer who covered paint industry activities in Chicago for the *American Paint & Coatings Journal,* died April 16.

Ms. Phillips was well-known by members of the Chicago Society and the CPCA. For several years she reported on the monthly meetings of both groups to readers of the APCJ. She also covered the annual SYMCO conference and other special paint industry activities.

Literature

Colorimeter

A unique tool for color measurement of products too large for a standard colorimeter is featured in new literature. Described is the XL-825 colorimeter with a remote optical sensor designed for use with robots on an assembly line or for monitoring production processes or hand-held in-plant for quality control. For information, contact Pacific Scientific, Gardner/Neotec Instrument Div., 2431 Linden Lane, Silver Spring, MD 20910.

Fog Exposure Systems

Information is available on a series of corrosive fog exposure systems designed to meet the requirements of most existing ASTM, U.S. Federal Standards, and Din Test Specification for accelerated corrosion testing. For bulletin No. 1420, write Atlas Electric Devices Co., 4114 No. Ravenswood, Ave., Chicago, IL 60613.

Liquid Level Measurement

Literature is available featuring an advanced liquid level measurement system designed with an internal microprocessor for providing local and remote indication of level measurement and an optional microcomputer operated data acquisition unit. Uses for the system are detailed as are the system's capabilities. For information, contact Krohne-America, Inc., 11 Dearborn Rd., Peabody, MA 01960.

Testing Equipment

A comprehensive range of precision engineered testing equipment essential for R&D, quality control, and field inspection of surface coatings is described in recently published literature. For information, contact Laporte (United States) Inc., distributor for Sheen Instruments of England, 411 Hackensack Ave., Hackensack, NJ 07601.

Silicone

An aminofunctional polydimethylsilicone copolymer that enhances the performance properties of detergent-resistant auto polishes and rinses is the subject of an eight-page product application bulletin recently issued. Typical uses of the silicone are detailed as are its formulations and physical properties. Copies of literature, SUI-276, are available from Union Carbide Silicones, Dept. K1476, Danbury, CT 06817.

Applications Data Bank

Information is available on a separator applications data bank designed to eliminate unnecessary time and trouble for end users in solving separation problems and to provide performance and application data on more than 700 materials. For a copy of the literature, contact SWECO, Inc., Robert W. Kenagy, 6033 E. Bandini Blvd., P.O. Box 4151, Los Angeles, CA 90051.

Meters

A new, 10-page brochure featuring a line of industrial meters is now available. Outlined are the features and benefits of five meters, as well as their accessories. In addition, easy-to-read charts and graphs offer precise technical information on each meter. For a copy, contact Minolta Corp., Industrial Meters Sales Manager, John McCasland, 101 Williams Dr., Ramsey, NJ 07446.

Viscometers

A new brochure describing the various types of viscometers has recently been published. Entitled, "When Viscosity Is Measured," the 28-page four color booklet lists the different models of viscometers and describes how they are used for measuring the fluid characteristics of substances. Information is provided on how to select the right viscometer and accessory for each scientific or individual application. Copies of the literature are available from Brookfield Engineering Laboratories, Inc., Dept. NR 43, 240 Cushing St., Stoughton, MA 02072.

Metal Finishing Products

A literature kit is available featuring a complete line of metal finishing products. Included is information on numerous maintenance/cleaning products, automotive conversion coating, cold forming and coil coating chemicals, and systems for other major industries. To obtain the literature kit, contact Surface Treatments, Inc., Marketing Dept., 13650 E. Ten Mile Rd., Warren, MI 48089.

Mica

Information is available on a complete line of muscovite mica products. Contact NYCO, P.O. Box 368, Mountain View Dr., Willsboro, NY 12996.

Wax

Literature is available featuring a micronized PTFE modified polyethylene wax with a controlled average particle size less than three microns. Discussed is the wax's easy dispersability in most coatings systems using high speed mixers or dissolvers and its recommended uses in offset, gravure, screen process, and electron cure inks. More information is available from Capricorn Chemicals Corp., Harmon Cove Towers, Secaucus, NJ 07094.

Color Meter

Literature is available featuring a portable tristimulus color comparison meter for rapid and convenient evaluation of colored materials. For information, contact Biotronics Instruments, P.O. Box 27, Newport Beach, CA 92662.

Paint Container Market

The availability of a worldwide multiclient study of penetration by plastics and composites of the one gallon paint container market has been announced. The study is highlighted by technological and economic forecasts for competing packages and raw materials, as well as sensitivity analyses, manufacturing cost estimates for each container type, and a detailed look at the structure of the paint container industry. Additionally, the research includes a market by market summary of the demand for paint containers in major world markets outside the U.S., along with consumer preference testing of new plastic cans. The study, available for $4,500, is obtainable from Phillip Townsend Associates, Inc., P.O. Box 90327, Houston, TX 77290.

Color Monitoring Systems

Information is available on an expanded line of on-line color monitoring systems, which are based on tristimulus colorimteric technology. Discussed is how the earlier systems measured the reflectance color of opaque products and how they are currently used in closed loop color control systems. Also, the new systems which are capable of measuring color of transparent and translucent sheet products by transmission are explained. For information, contact HunterLab, 11495 Sunset Hills Rd., Reston, VA 22090.

Color Tools

A literature and sales-promotion package featuring the new Cape May Victorian Color Palette is now available to dealers. The package contains various promotion pieces as well as a brochure displaying the 70 shades of the new color palette. For information, contact Fuller-O'Brien Paints, Marketing Dept., 450 E. Grand Ave., S. San Francisco, CA 94080.

Stabilizers and Antioxidants

Information is available on a multi-client, techno-economic marketing study covering stabilizers and antioxidants for plastics. The study, comprised of 418 pages, 315 tables, and referenced by an index with 2350 entries, provides an in-depth analysis on the chemistry, grades, prices, suppliers, polymer systems, volumes, projections, technological and marketing trends for 15 families of products. Designed as a tool for strategic planning and as a guide to the best market opportunities, the study is available for a subscription fee of $6,500. A free brochure, with Table of Contents, is available from Skeist Laboratories, Inc., 112 Naylon Ave., Livingston, NJ 07039.

Agents

Literature is introducing a series of seven surface modification and coupling agents which are multimetal, low molecular weight inorganic polymers in which two types of organic ligands are chemically anchored to the backbone. Formulations and application uses are detailed. For complete technical information, contact Cavedon Chemical Co., Inc., 26 Avenue C, Woonsocket, RI 02895.

Solvent

Information is available featuring propylene glycol monomethyl ether acetate (PMAc), a new high performance, low toxicity solvent. Technical data sheets are available from ARCO Chemical Co., Marketing Communications Dept., 1500 Market St., Philadelphia, PA 19101.

Dispersions

Literature is introducing three new ultra-fine surface conditioners for use in thin film applications including can coatings and metal decorating inks. For information, contact Daniel Products Co., 400 Claremont Ave., Jersey City, NJ 07304.

Air Sampling Equipment

A new 48-page catalog is available featuring a comprehensive line of air sampling equipment. More than 800 test instruments are listed as are their features, operations, and typical applications. For information, contact SKC, Inc., R.D. 1, 395 Valley View Rd., Eighty Four, PA 15330.

CLASSIFIED ADVERTISING

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Book Review

PAINT HANDBOOK

Edited by
Guy E. Weismantel

Published by
McGraw-Hill Book Co.
New York, NY
741 pgs., 1981, $37.50

Reviewed by
George R. Pilcher
Hanna Chemical Coatings Corp.
Columbus, OH

In his brief, yet concise and informative Preface, the editor clearly establishes the intended use and audience for this volume: it is "... a reference work for architects, corrosion engineers, specification writers, painting contractors, and operating companies staff engineers.... This is not a textbook; it does not cover theory.... The main emphasis is on architectural and industrial coatings that are field- or shop applied." The coatings scientist should take this *caveat* to heart; it is definitely not a book for everyone, although it should probably be well-received by its intended audience.

The *Paint Handbook* is divided into twenty chapters, each authored by one or more of 21 contributors drawn from industry, academia and the ranks of the professional consultants. The variety of topics include Paint Fundamentals, Selecting the Paint System, Surface Preparation, Application Techniques, and Economics. Naturally, the multiple authors lead to a certain variability among the chapters, although the scope of activities for the book's intended audience is covered fairly thoroughly. Some chapters, such as Surface Preparation: Part II, by K. B. Tator, are really quite excellent for both content and presentation.

It is, therefore, that much more unpleasant to report that even its strengths cannot completely compensate for its weaknesses, which include a number of misleading statements, regrettable omissions and even errors: listing orthophenanthroline (sic) as a "nonmetallic drier," stating that melamine resins which are "... synthesized from melamine, a ring compound, and formaldehyde, act much as urea resins do. However, they cure more quickly or at lower temperatures...," and listing "regular silica, which gives a rough surface..." under the Extender Section of Chapter 3, while omitting any mention of the numerous hydrogels, aerogels, crystalline, amorphous, diatomaceous, *et al,* forms of silica both available and in common usage in the same systems covered in this book. There is nothing innately wrong with limiting detailed comment based on the anticipated audience for a book such as this, in an effort to present "nuts and bolts" information—but that information should be accurate, even if it is severely limited.

Recommended, with reservations, to users of industrial maintenance coatings.

MACROMOLECULES
An Introduction to Polymer Science

Edited by
F.A. Bovey and F.H. Winslow

Published by
Academic Press
New York, NY
549 pgs., 1979, $47.00

Reviewed by
Thomas J. Miranda
Whirlpool Corp.
Benton Harbor, MI

This text arose from a need within the Bell Laboratories for a volume to accompany an introductory course. The topics treated by four other authors as well as the editors include: The Nature of Macromolecules, Formation of Macromolecules, Microstructure and Chain Conformation, Solution Properties, Morphology, Physical Behavior, Reactions of Macromolecules, and Biological Macromolecules. This text is heavy on fundamentals and provides good detail on reaction mechanisms of polymerization including coordination, emulsion and ring opening polymerization.

Chapter 4 treats the thermodynamics of polymer solutions and molecular weight determinations. Chapter 5 covers morphology, the crystalline state and viscoelastic properties of polymers. The remaining two chapters cover polymer reactions, stabilization and degradation and biological polymers.

This book is well written with little emphasis on the technology of polymers. For those requiring a text emphasizing fundamentals it is a good contribution to the list of polymer text books.

METAL FINISHING Guidebook and Directory Issue '83 Volume 81, NO. 1A

Compiled and Edited by
Metal Finishing Staff

Published by
Metals and Plastics
Publications, Inc.
Hackensack, NJ
1034 pgs., included in $21.00
monthly subscription price

Reviewed by
Franklin G. Katje
Whirlpool Corp.
Benton Harbor, MI

This text is a collection of technical papers, as well as charts and tables covering the various aspects of metal finishing. Subjects covered a range from anodizing to zinc plating.

The major areas of surface preparation, solutions, control/analysis, special procedures and equipment are covered in good detail. Special sections are provided that give a directory of associations, schools, trade names and information about suppliers of chemicals and equipment. An excellent section of tables and data sheets along with government specifications is also provided.

The reviewer found this text to be an excellent reference work for anyone working in the field of metal finishing. The subjects are well written and organized. Information is well indexed, greatly enhancing the value of this book.

The reviewer would strongly recommend this text to anyone having even remote affiliation with a metal finishing operation.

Errata

In the article, "High Solids Coatings from New Oligomers," by M.S. Chattha and J.C. Cassatta (May 1983, pp 39–46), figures 5, 6, and 7 are out of sequence. They should read:

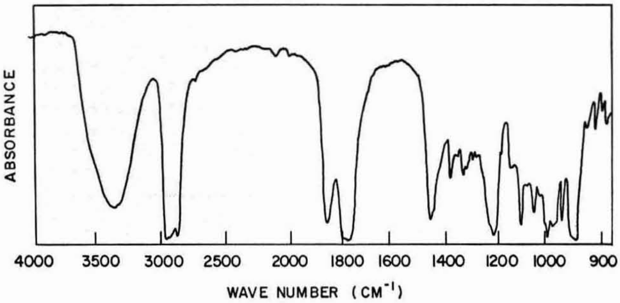

Figure 5—Infrared spectrum of mixture of 2-ethyl-1,3-hexanediol and methylhexahydrophthalic anhydride immediately after mixing at room temperature

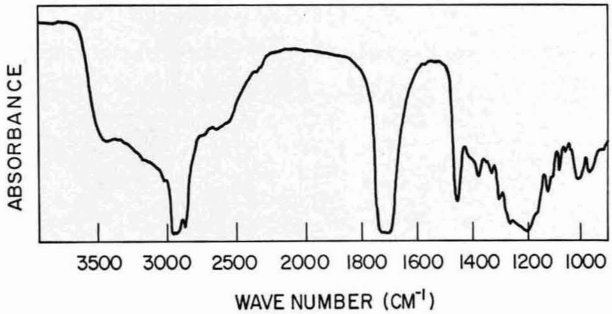

Figure 6—Infrared spectrum of the mixture described in *Figure* 1 after heating at 100°C for two hours

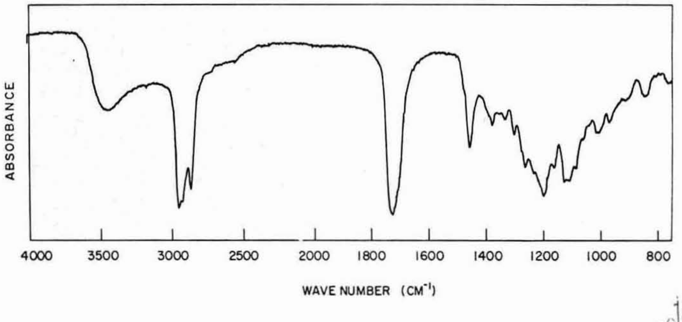

Figure 7—Infrared spectrum of the tetrahydroxy oligomer *5*

Corrected
A.J.
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Coming Events

FEDERATION MEETINGS

(Oct. 12–14)—61st Annual Meeting and 48th Paint Industries' Show. Place Bonaventure, Montreal, Quebec, Canada. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, PA 19107).

1984

(May 17–18)—Spring Meetings. Society Officers on 17th; Board of Directors on 18th. Galt House, Louisville, KY. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, PA 19107).

(Oct. 24–26)—62nd Annual Meeting and 49th Paint Industries' Show. Conrad Hilton Hotel, Chicago, IL. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, PA 19107).

SPECIAL SOCIETY MEETINGS

1984

(Jan. 17–18)—Cleveland Society for Coatings Technology. Joint Manufacturing Committee Symposium with the Cleveland PCA. "Tools for Tomorrow: Increased Profitability Thru Innovative Manufacturing Concepts."

(Mar. 7–9)—Southern Society for Coatings Technology. Fiftieth Anniversary Meeting. Surfside Hotel, Clearwater, FL. (James E. Geiger, Sun Coatings, Inc., 12295 75th St., N., Largo, FL 33543).

(Apr.)—Cleveland Society for Coatings Technology 27th Annual Technical Conference, "Advances in Coatings Technology. (Richard . Eley, Glidden Coatings & Resins Div. SCM Corp., 16651 Sprague Rd., Strongsville, OH 44136).

(Apr. 12–14)—Southwestern Paint Convention of Dallas and Houston Societies. Shamrock Hilton Hotel, Houston, TX.

(May 3–5)—Pacific Northwest Society for Coatings Technology Symposium. Park Hilton Hotel, Seattle, WA. (Robert Hogg, Preservative Paint Co., 5410 Airport Way S., Seattle, WA 98108).

1985

(Feb. 26–Mar. 1)—Western Coatings Societies Symposium and Show. Disneyland Hotel, Anaheim, CA.

(Apr. 25–27)—Pacific Northwest Society for Coatings Technology Symposium. Empress Hotel, Victoria, B.C. (Ottwin Schmidt, Shanahan's Ltd., 8400 124th St., Surrey, B.C., Canada V3W 6K1).

OTHER ORGANIZATIONS

(July 25–29)—"Microbiological Degradation" sponsored by the Gordon Research Conferences. Brewster Academy, Wolfeboro, NH. (Alexander M. Cruickshank, Director, Gordon Research Conferences, University of Rhode Island, Kingston, RI 02881).

(July 29–31)—Oil & Colour Chemists' Association, New Zealand Div., 21st Annual Convention. International Hotel, Rotorua, New Zealand. (OCCA, 1983 Convention, P.O. Box 5192, Auckland, New Zealand).

(Aug. 8–12)—Fourth International Conference on the Organometallic and Coordination of Germanium, Tin, and Lead. McGill University, Montreal, Que., Canada. (Prof. M. Onyszchuk, McGill University, Dept. of Chemistry, 801 Sherbrooke St. West., Montreal, Que., Canada H3A 2K6).

(Aug. 8–12)—"Physics and Chemistry of Coatings and Films" sponsored by the Gordon Research Conferences. Plymouth State College, Plymouth, NH. (Alexander M. Cruickshank, Director, Gordon Research Conferences, University of Rhode Island, Kingston, RI 02881).

(Aug. 15–19)—"Advances in Emulsion Polymerization and Latex Technology" Short Course. Schatzalp Berghotel, Davos, Switzerland. (Dr. Gary Poehlein, School of Chemical Engineering, Georgia Institute of Technology, Atlanta, GA 30332).

(Aug. 17–19)—"Color Control Technology" Seminar sponsored by Applied Color Systems, Inc. Wequasett Inn, Cape Cod, MA. (ACS, Inc., P.O. Box 5800, Princeton, NJ 08540).

(Aug. 22–26)—"Physics and Chemistry of Printing Inks" Short Course. Davos, Switzerland. (Dr. Mohamed S. El-Aasser, Lehigh University, Dept. of Chemical Engineering, Sinclair Lab #7, Bethlehem, PA 18015).

(Aug. 26–30)—International Colour Association's Forsius Symposium on Colour Systems. Kungalv, Sweden. (Fred W. Billmeyer, Jr., Dept. of Chemistry, MRC 217, Rensselaer Polytechnic Institute, Troy, NY 12181).

(Aug. 31–Sept. 8)—20th Commission Internationale de l'Eclairage Congress. RAI Congress Center, Amsterdam, The Netherlands. (U.S. National Committee, CIE, c/o National Bureau of Standards, Washington, D.C. 20034).

(Sept. 17–19)—"Color Control Technology" Seminar sponsored by Applied Color Systems, Inc. Atlantic City, NJ. (ACS, Inc., P.O. Box 5800, Princeton, NJ 08540).

(Sept. 18–20)—Canadian Paint and Coatings Association's 71st Annual Convention. Hyatt Regency Hotel, Montreal, Quebec, Canada. (CPCA, 515 St. Catherine St. W., Suite 825, Montreal, Quebec H3B 1B4, Canada).

(Sept. 27–28)—Pulp Chemicals Association's 10th International Naval Stores Conference and Technical Symposium. Westin Peachtree Plaza Hotel, Atlanta, GA. (Pulp Chemicals Association, 60 E. 42nd St., New York, NY 10165).

(Sept. 27–29)—"Radiation Curing in the Printing and Converting Industry" Workshop sponsored by the Association for Finishing Processes of the Society of Manufacturing Engineers (AFP/SME). Hershey Hotel, Philadelphia, PA. (Donna Theisen, Administrator, Technical Activities Dept., SME, One SME Dr., P.O. Box 930, Dearborn, MI 48121).

(Sept. 27–30)—National Coil Coaters Association Fall Technical Meeting. Hyatt Regency O'Hare, Chicago, IL. (NCCA, 1900 Arch St., Philadelphia, PA 19103).

(Sept. 28–Oct. 1)—Oil & Colour Chemists' Association's Silver Jubilee Convention and Exhibition. Southern Cross Hotel, Melbourne, Australia. (O.C.C.A.A., 1983 Pacific Coatings Convention, C/- Tioxide Australis Pty. Ltd., Private Bag 13, Ascot Vale, Victoria, 3032, Australia).

(Oct. 5–6)—"Finishing of Plastics for Functional and Decorative Purposes" Technical Conference. Decorating Division of the Society of Plastics Engineers. Hyatt Regency, Cherry Hill, NJ. (Ed Stumpek, General Electric, One Plastics Ave., Pittsfield, MA 01201).

(Oct. 11–13)—"Finishing '83" sponsored by the Association for Finishing Processes of the Society of Manufacturing Engineers. Cincinnati Convention Center, Cincinnati, OH. (Susan Buhr, AFP/SME Administrator, One SME Dr., P.O. Box 930, Dearborn, MI 48128).

(Oct. 30–Nov. 2)—National Paint & Coatings Association 96th Annual Meeting. Bonaventure Hotel, Los Angeles, CA. (Karen Welch, NPCA, 1500 Rhode Island Ave., N.W., Washington, D.C. 20005).

(Nov. 2–3)—3rd World Congress on Coatings Systems for Bridges and Steel Structures. Breckenridge Concourse Hotel, St. Louis, MO. (John A. Gordon, Jr., Conference Director, Arts & Sciences, Continuing Education, University of Missouri-Rolla, Rolla, MO. 65401).

(Nov. 2–4)—"Fundamentals of Adhesion: Theory, Practice, and Applications" Short Course. State University of New York, New Paltz, NY. (Dr. Angelos V. Patsis, Chemistry Dept., SUNY, New Paltz, NY 12561).

(Nov. 8–9)—Resins and Pigments Exhibition. Europa Hotel, London, England. (Polymers, Paints & Colour Journal, Queensway House, Redhill, Surrey, RH1 1QS, England).

(Nov. 11–13)—36th National Decorating Products Show. McCormick Place, Chicago, IL. (NDPA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132).

(Dec. 7–8)—"Painting Public Structures: Practices and Economics" Symposium sponsored by the Steel Structures Painting Council. Cocoa Beach, FL. (Dr. Harold Hower, SSPC, 4400 Fifth Ave., Pittsburgh, PA 15213).

(Dec. 12–14)—"Color Control Technology" Seminar sponsored by Applied Color Systems, Inc. Disneyworld/Epcot Center, FL. (ACS, Inc., P.O. Box 5800, Princeton, NJ 08540).

1984

(Feb. 12–15)—Inter-Society Color Council Conference. Colonial Williamsburg Lodge. Williamsburg, VA. (Fred W. Billmeyer, Dept. of Chemistry, Rensselaer Polytechnic Institute, Troy, NY 12181).

(Feb. 12–16)—14th Australian Polymer Symposium sponsored by the Polymer Div. of the Royal Australian Chemical Institute. Old Ballarat Motor Inn, Ballarat, Australia. (Dr. G.B. Guise, RACI Polymer Div., P.O. Box 224, Belmont, Vic., 3216, Australia).

(Apr.)—"Electrochemical Test Methods of the Protecting Properties of Metals Coatings" Meeting. Genoa, Italy, (Prof. P.L. Bonora, Istituto di Chimica, Fac. Ingegneria—Fiera del Mare Pad. D. 16129 Genova, Italy).

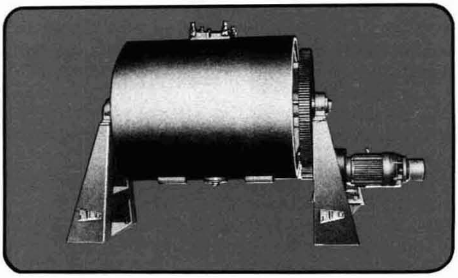
(Apr. 8–10)—Inter-Society Color Council Annual Meeting. Michigan Inn, Southfield, MI. (Fred W. Billmeyer, Dept. of Chemistry, Rensselaer Polytechnic Institute, Troy, NY 12181).

(Apr. 12–15)—"FARBE 84". Munich Trade Fair Centre, Munich, West Germany. (Kallman Associates, Five Maple Court, Ridgewood, NJ 07450).

(Apr. 24–25)—Electrocoat/84 Conference, sponsored by *Products Finishing* Magazine. Drawbridge Inn, Cincinnati, OH. (Anne Porter, Products Finishing, 6600 Clough Pike, Cincinnati, OH 45244).

(May 1–4)—Painting and Decorating Contractors of America. 100th Anniversary Meeting. New York, NY.

(Sept. 23–28)—XVIIth Congress of FATIPEC (Federation of Associations of Technicians in the Paint, Varnish, Lacquer and Printing Ink Industries of Continental Europe). Lugano, Switzerland. (C. Bourgery, Secretary General, FATIPEC, Maison de la Chimie, 28 Rue St.-Dominque, 75 Paris (7), France).



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'Humbug' from Hillman

Life in the Hereafter

After a short lifetime in the paint industry, 48 years to be exact, I happily look forward to life in the hereafter.

Hereafter, Libby and I plan to live our life in Vermont with occasional time out to see our country and perhaps parts of the world beyond. For the past year we've been living in Vermont and New York—between heaven and earth—partly in coatings and partly in maple syrup. The trial run has been great and I am ready for the hereafter in our haven (or heaven—to reach for the corny analogy). It appears that life in the hereafter will be a hell of a lot more fun for us than life in the heretofore. I'll be gleefully trading a paint bucket for a sap bucket and a paint brush for the beautiful sugar bush.

Much less than a year ago I looked with caution and suspicion at the man from the EPA. Now, I am ready to shout, "Come on man—do your stuff. Keep our Vermont air fresh and clean. Save those trout streams and wipe out acid rain."

As I face the hereafter, it is probably appropriate for me to bequeath to those I leave behind some of the goodies I have collected in my former life. So . . .

Item: To Bob Ziegler, Editor, and Tom Miranda, Technical Editor, I leave all those incomprehensible (to me) articles in the JCT, through which I struggled, out of a nagging sense of obligation to my job.

Item: To Frank Borrelle, I leave my position in the line waiting to get into breakfast at his conventions.

Item: My old copies of Abel Banov's eternally optimistic market reports in the *APCJ* I leave to all the TiO_2 marketing managers to light their way to even higher profits.

Item: I bequeath my antique and now obsolete slide rules to all those aging chemists who, like me, resist the frustration of Basic, Fortran, etc.

Item: My formulations of circa 1938 I leave to all our advertising managers who have invented far more new products than I did.

Now, more lightly burdened, I hope to continue Humbug's philosophies with the help of all our old friends and, *please,* some new ones. I shall, therefore do what I can to lessen the scientific value of this fine journal. All those who would like to join me in this humanitarian venture are urged to correspond with me at Humbug's Nest, Box 135, Whitingham, VT 05361.

• • •

L.K. Hosfeld has sent in a comprehensive report on "Professor" Paul A. Crafton who simultaneously held down three college teaching jobs—all on phony credentials. Hosfeld mentions that this character had up to 34 aliases and adds—"Just think, if he had not been caught, at age 62 he could have collected 34 Social Security checks." *Food for thought!*

Now that Howard Jerome has given up the heavy responsibilities of Federation Presidency, he has devoted his time and wisdom to collecting and composing "bon mots" for Humbug. Here are just a few of his recent observations.

- The life expectancy of a house plant varies inversely with its price and directly with its ugliness.
- Authority tends to assign jobs to those least able to do them.
- In any organization there will always be one person who knows what is going on. This person must be fired.
- The one course you must take to graduate will not be offered during your last semester.
- Circumstances can force a generalized incompetent to become competent, at least in a specialized field.
- No man's life, liberty, or property are safe while the legislature is in session.
- The organization of any bureaucracy is very much like a septic tank—the really big chunks rise to the top.

I am so pleased that another member of the Lauren family has joined our wonderful but wacky contributors. This time we have heard from Dr. Barbara Lauren. Barbara, whom I have known since she was weaned on a paint splattered bottle, has taken time from her complex legal and literary duties at Boston University to collect appropriate nonsense for Humbug.

—The minutes of a meeting of the University of Denver Senate include a "Membership Rooster."

—From an "Announcement of Vacancy, Computer Science Math" at Tabor College: "Primary teaching responsibilities will be in Computer Science, with one or two lover-level courses assigned annually."

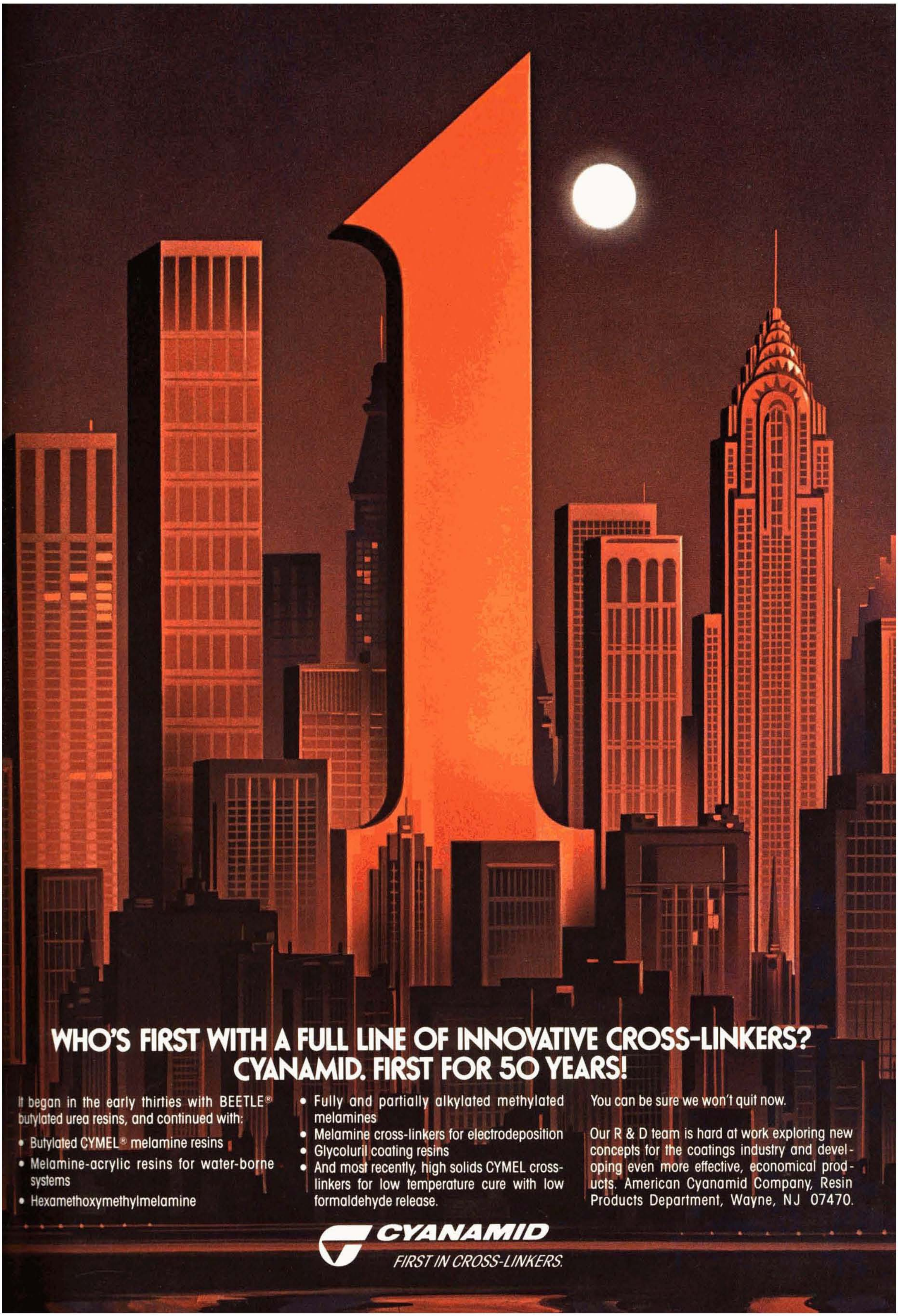
—One section of the latest annual report of the American Assembly of Collegiate Schools of Business is entitled "The Future and Beyond."

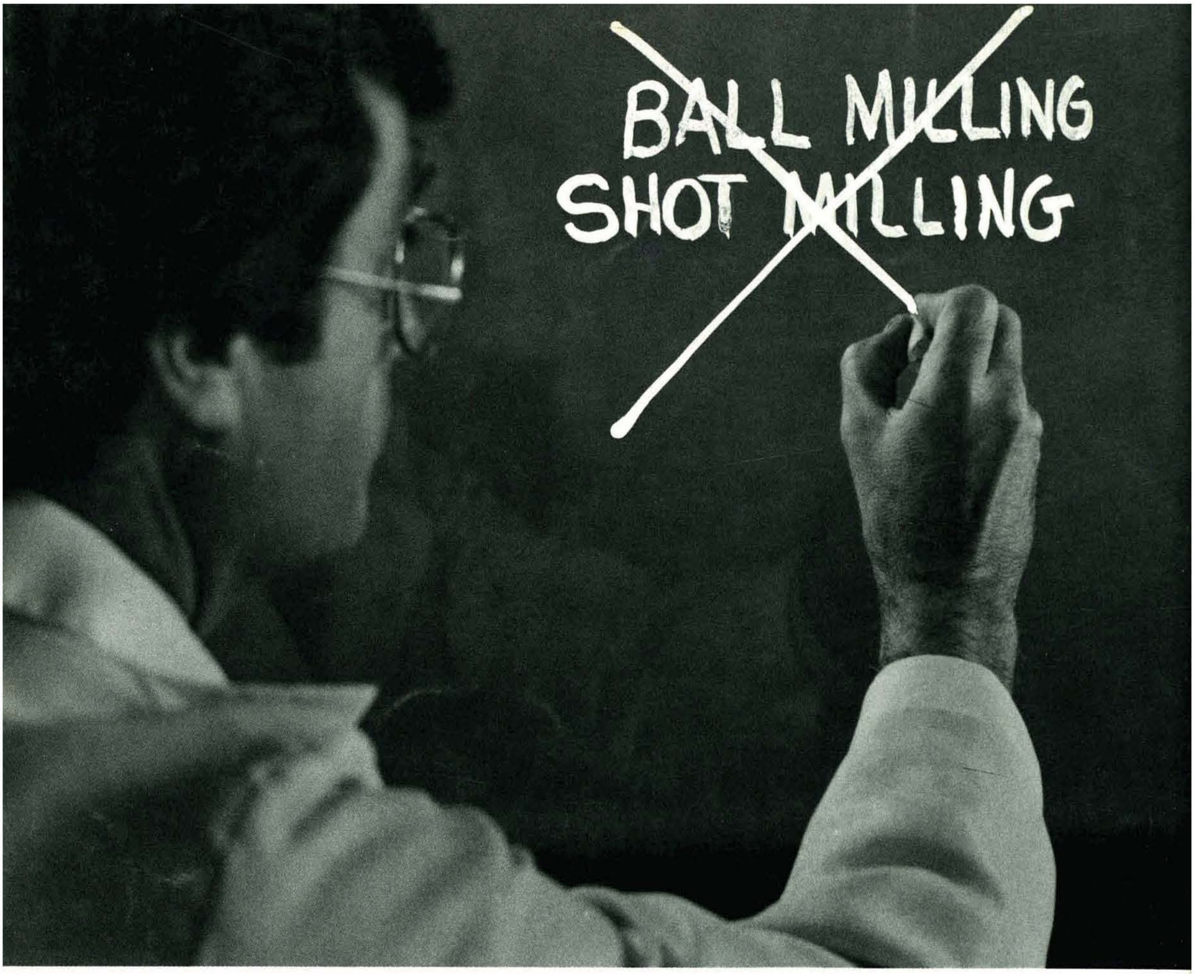
—From the minutes of a meeting of the Plant Maintenance and Energy Conservation Committee at Albany (GA.) Junior College: "Dr. Saville and Mr. Swain brought up the problems peculiar to F-Building in conservation of energy, and it was decided that F-Building would have to be treated as a special case where conservation is concerned. Problems in that building are largely the result of the position of the sun, which does not follow committee recommendations."

—The chairman of this week's opening session of the National Research Council's conference on fertility is Mercedes B. Concepcion.

—Dorothy Williams, editor of *Illini Week,* the faculty-staff paper at the University of Illinois, received a letter promoting a "career coaching workshop for women" on October 30, at which a woman will be the featured speaker and all the other speakers are women. The salutation on the letter: "Gentlemen."

—Herb Hillman

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