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jct JOURNAL OF COATINGS TECHNOLOGY

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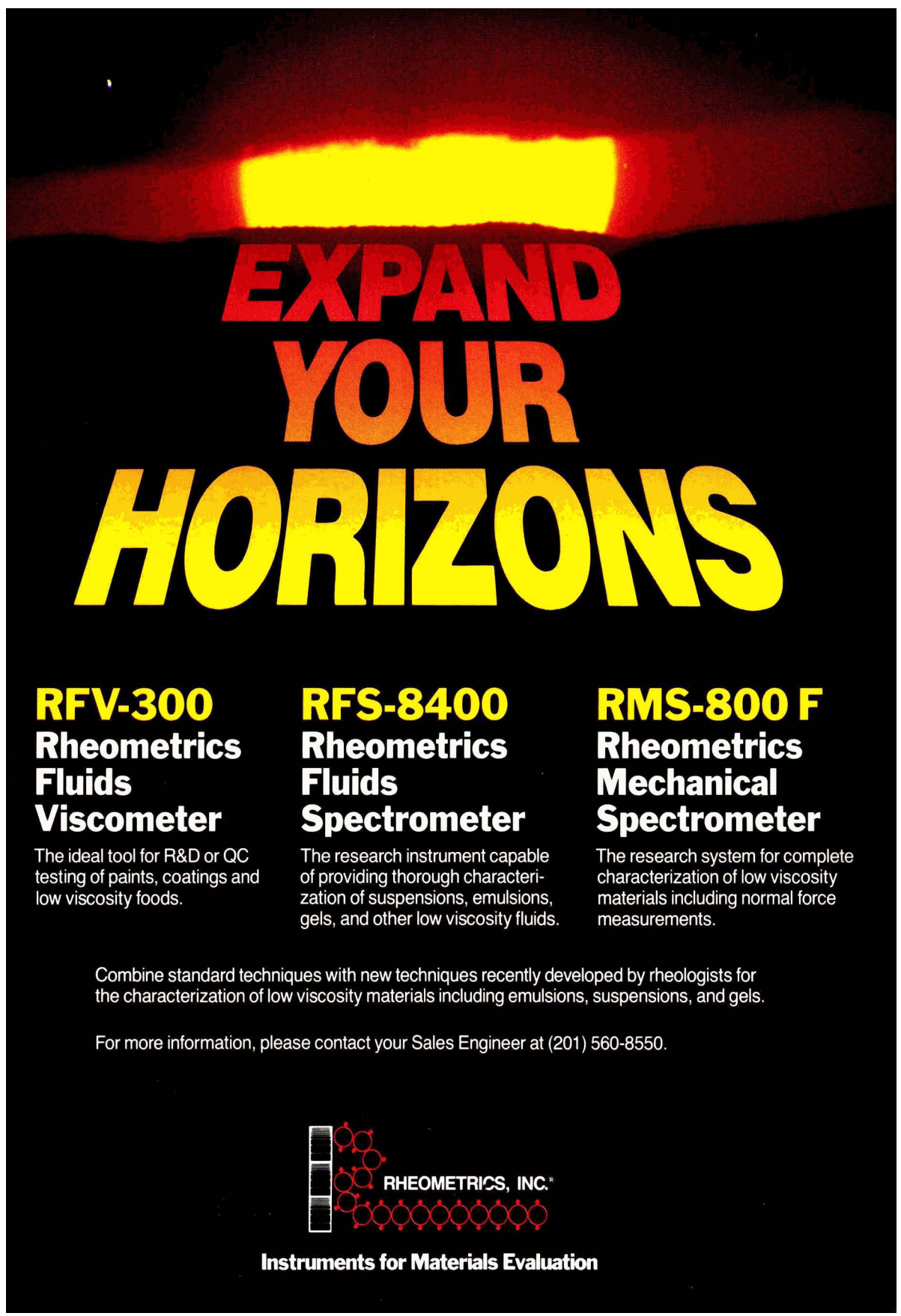

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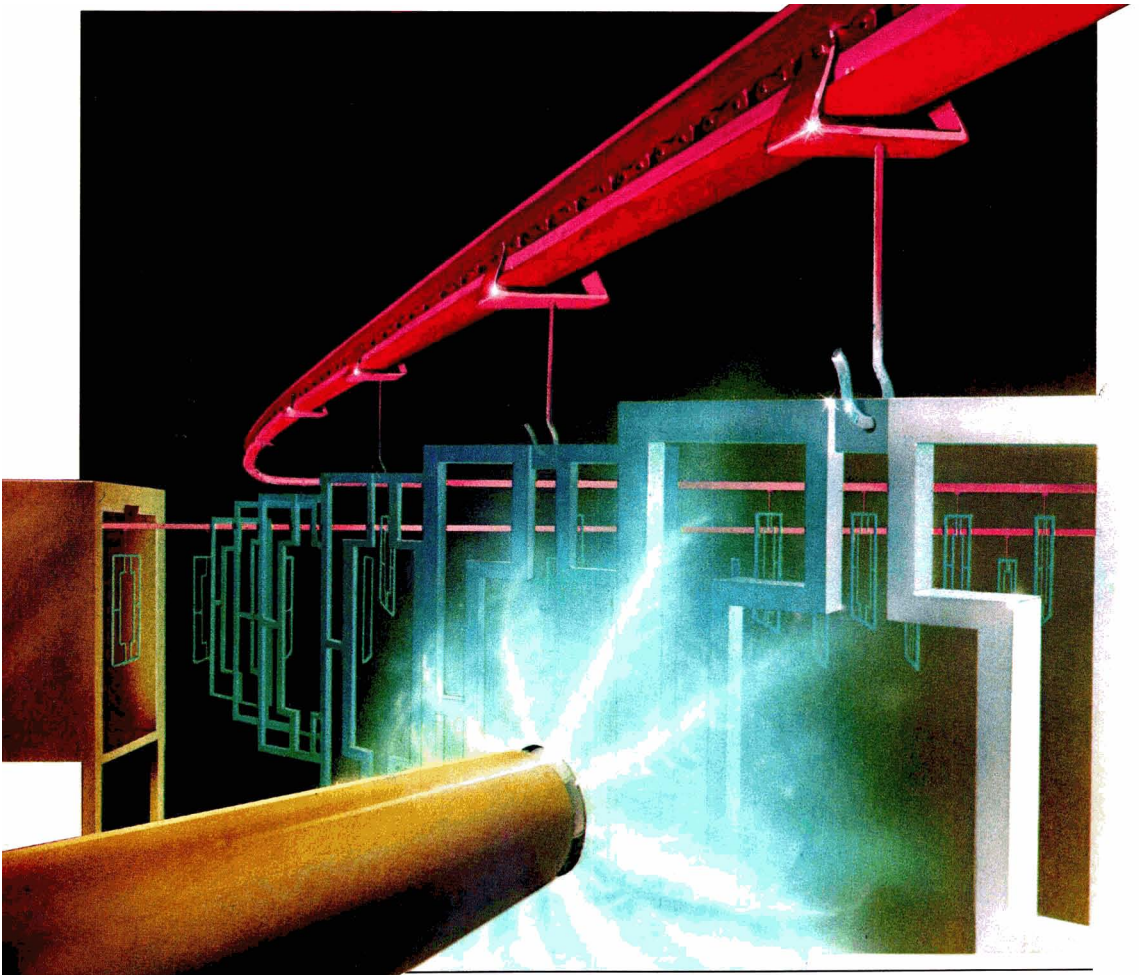

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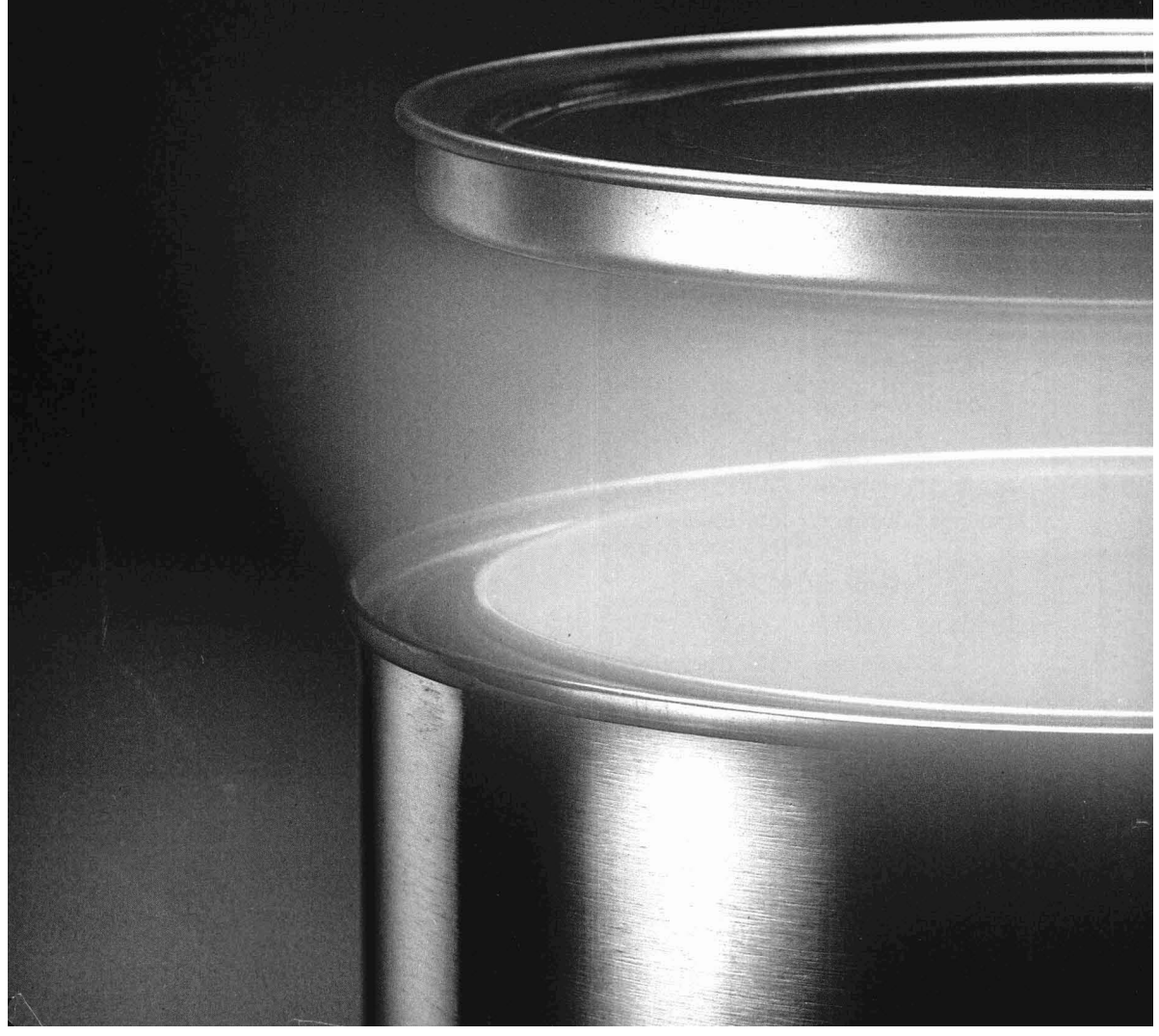

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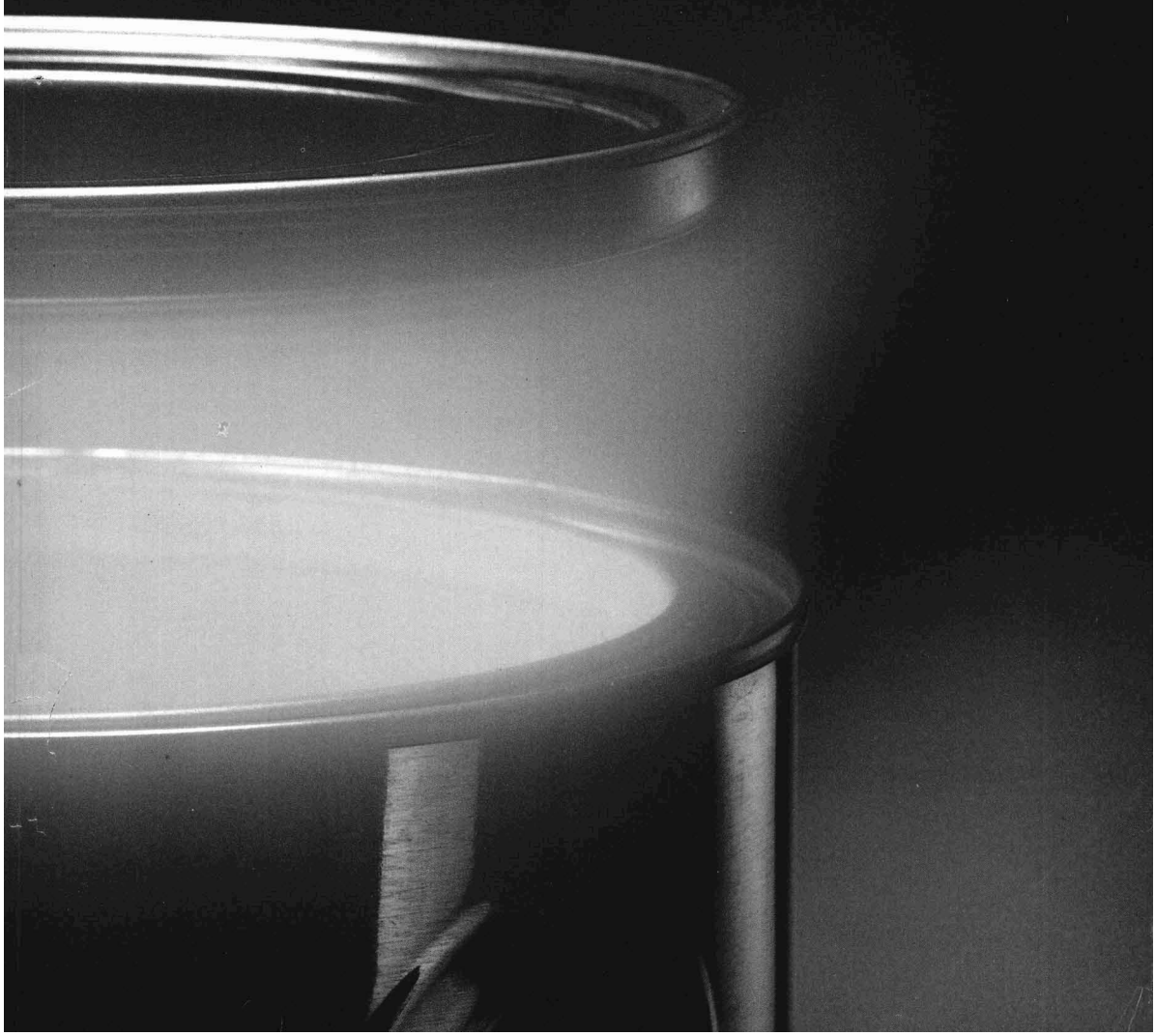

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Comment

Thanks to the Paint Show

When the Board of Directors approved President Joe Bauer's creation of three new Federation committees, it was plain that the Board shared the view of many other members: that the Federation should devote more of its attention and funding to the membership, to education, and to its role in the coatings industry.

Professional Development

The nine-member group aims to provide for: the professional development of the membership through continuing educational activities (short courses, seminars) and the attraction of technical talent into the coatings industry (local Society/University interactions, summer co-op employment). F. Louis Floyd, of the Cleveland Society, is Chairman.

Federation Chairs

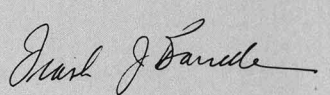
Loren Hill, of the New England Society, heads this five-member committee which is studying the possibilities and ramifications of Federation sponsorship of "Chairs" or "Professorships" at various universities. The benefit of such funding would be a stronger commitment by the university to the coatings industry and following that, an increase in the number of well-trained entry level employees. This program, however it develops, is in addition to the current scholarships awarded to the University of Detroit, Eastern Michigan University, Kent State University, University of Missouri-Rolla, North Dakota State University, and the University of Southern Mississippi.

Future Planning

Past-President Neil S. Estrada, of the Golden Gate Society, chairs the five-member committee which has been charged to: make future plans for the personnel requirements of the Federation and investigate any activities that the Federation should undertake to assist in the development of the coatings industry.

The continuing success and growth of the annual Paint Industries' Show enables the Federation to establish these committees, to fully support them in their deliberations, and to fund any of their programs approved by the Board of Directors.

To benefit the membership and education—that's why the Paint Show was established in the first place—way back in 1932.



Frank J. Borrelle,
Executive Vice-President

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Abstracts of Papers in This Issue

SLIME FILMS ON ANTIFOULING PAINTS. SHORT-TERM INDICATORS OF LONG-TERM EFFECTIVENESS—M.G. Robinson, B.D. Hall, and D. Voltolina

Journal of Coatings Technology, 57, No. 725, 35 (June 1985)

A method for an initial evaluation of the biological effectiveness of antifouling paints is described. It involves exposing test surfaces for three to four months under natural marine conditions and examining the resulting slime film by scanning electron microscopy or optical microscopy. The presence of a large number of diatom species indicates that the paint is not biologically effective and will not prevent macrofouling. If species diversity is significantly reduced, and especially if the population consists of known toxin-tolerant species, the paint warrants further testing. Slime film biomass is not a good measure of the effectiveness of antifouling paints. The paint should be applied over the same undercoat as in the potential working situation.

KINETICS OF CROSSLINKING USING BLOCKED REAGENTS AND BLOCKED CATALYSTS—W.J. Muizebelt

Journal of Coatings Technology, 57, No. 725, 43 (June 1985)

The kinetics of curing under the influence of blocked reagents and blocked catalysts are considered in this paper.

Blocked reagents dissociate thermally yielding a species which participates in a crosslinking reaction. Either the deblocking reaction or the crosslinking reaction may be rate-determining.

Experiments with Desmodur N (a trifunctional isocyanate) blocked with benzyl methacrylo hydroxamate (BMH) indicate that deblocking and crosslinking occur at comparable rates.

A theoretical model was developed describing the action of blocked catalysts in a crosslinking reaction. Results of experiments using oxime tosylates for the curing of high solid polyester/melamine coatings are generally in line with this model. The crosslinking shows an induction period and S-shaped curves are observed.

VISCOSITY OF OLIGOMER SOLUTIONS—Z.W. Wicks, et al.

Journal of Coatings Technology, 57, No. 725, 51 (June 1985)

Viscosity and glass transition temperature, T_g, of solutions of oligomeric butyl and methyl methacrylates (OBMA and OMMA) were determined. The dependence of T_g on concentration of solutions of OBMA in ethyl benzene and *m*-xylene over the entire concentration range was found to follow equation (10).

$$\frac{1}{T_g} = \frac{w_s}{T_{gs}} + \frac{w_o}{T_{go}} + K w_o w_s \qquad (10)$$

Temperature dependence of viscosity of OBMA in *m*-xylene was found to follow WLF (Williams, Landel, and Ferry) equation (1) over the whole range of concentration.

$$\ln \eta = \ln \eta_{T_r} - \frac{c_1 (T-T_r)}{c_2 + (T-T_r)} - \ln \frac{\rho_r T_r}{\rho T} \qquad (1)$$

Solutions other than pure solvent and those at low concentration followed simplified forms of the WLF equation such as (18) satisfactorily.

$$\frac{1}{34.5 - \ln \eta} = \frac{1}{A} + \frac{B}{A(T-T_g)} \qquad (18)$$

This equation provides a better model of viscosity as a function of temperature than an Arrhenius type expression, not only for these oligomer solutions, but, based on calculations using literature data, also for a wide range of other oligomers and oligomer solutions. T_g's and viscosities at equal concentration and temperature of OBMA and OMMA solutions in *m*-xylene increase as molecular weight increases and at comparable molecular weight are higher for OMMA than OBMA. Although T_g of OBMA solutions increased as T_g (or estimated T_g) of a series of solvents increased, viscosities of the solutions did not always show a corresponding increase. This, along with the system dependence of A and B in equation (18), indicates that while $(T-T_g)$ is an important factor controlling viscosity, it is not the only factor.

Addition of a power factor to the denominator of Erickson's equation, equation (19), leads to significantly improved predictions of relative viscosity as a function

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Abstracts of Papers in This Issue (continued)

of concentration for both these oligomer solutions and Erickson's.

$$\ln \eta_r = w_o/(k_1 - k_2 w_o + k_3 w_o^2) \qquad (19)$$

Intrinsic viscosity is properly calculated by dividing the inverse of constant k_1 in equation (19) by solution density at $w_o = k_1$. Intrinsic viscosities (and ln η of solutions) of OBMA in *m*-xylene increase with approximately the half power of $\bar{M}_n$, possibly indicating theta conditions. However, intrinsic viscosities of both OBMA and OMMA solutions in *m*-xylene decrease with increasing temperature. Also, intrinsic viscosity of OBMA solutions is solvent dependent.

Viscosity of oligomer solutions in the range of interest for high solids coatings depends upon oligomer and solvent viscosity, oligomer and solvent T_g, solvent-oligomer interaction, temperature, and concentration. Further studies are needed to understand the relative importance of these factors and interactions between them.

NEW ISOINDOLINE PIGMENTS FOR HIGH QUALITY APPLICATIONS—J. von der Crone

Journal of Coatings Technology, 57, No. 725, 67 (June 1985)

In recent years, numerous patents claiming novel chemical structures, improved synthetic procedures, and new compositions of matter have been published on isoindoline pigments.

A special group of pigments, obtained by the condensation of diiminoisoindoline with cyanoacetarylides, is now reported. Synthesis, influence of substituents on pigment properties, and crystal structures are discussed.

The potential and limits of such pigments are illustrated by means of examples chosen from this group. Attention is particularly drawn to the outstanding pigment properties in general, as well as to the manifold possibilities of application, including styling in paints.

Papers to be Featured in the July Issue

"Water-Borne Corrosion Inhibitive Primers Evolve to be Competitive"—Golden Gate Society for Coatings Technology, Technical Committee

"Selection of a Spatial Sampling Procedure for Evaluating the Defect Area of a Coated Steel Panel"—D. Bentz and J.W. Martin, National Bureau of Standards

"Effect of Solvent Choice on Water-Based Coating/Wood Interactions"—P. Lucas and L. Smith, State University of New York

"Physico-Chemical Factors in the Performance of Polymer-Metal Bonds"—S. Ponce, et al., Ecole Polytechnique

"Reactions of N(2-Hydroxyethyl) Amido Compounds"—Z.W. Wicks, Jr., M.R. Appelt, and J.C. Soleim, North Dakota State University

"Barriers to Innovation in the Organic Coatings Industry"—M.A. Glaser, Consultant

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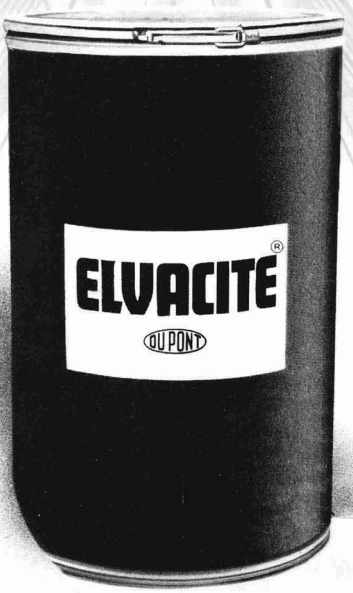

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50-Year Members of FSCT to Be Honored At 1985 AMPS in St. Louis, Oct. 7-9

The Golden Anniversary Paint Show will be held at the Cervantes Convention Center in St. Louis, MO, on October 7-9. This event will celebrate the origins and opportunities provided by this international exhibition of raw materials, equipment, and services for the paint and coatings manufacturing industry.

From its first Paint Show in 1932 until the present, the Federation of Societies for Coatings Technology has grown to encompass 26 Societies and 7,000 members, some of whom have contributed to the advancement of the Federation for the past 50 years. Recognition of these 50-year members will highlight the upcoming event in St. Louis.

At the 1985 Annual Meeting and Paint Show, all 50-year members in attendance will be given complimentary registration and luncheon tickets for themselves and their spouses. They will be honored at the luncheon, to be held on October 9. The Federation congratulates these members, listed below, as they join in the celebration of their own "Big 50."

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ALMA GOOEL
BERT GOOEL
Standard Detroit Paint Co.
8255 Lyndon
Detroit, MI 48238

CARL C. HOWSON
16 Heather Circle
Port Angeles, CA 98362

RICHARD O. INNES
O.G. Innes Corporation
10 E. 40th Street
New York, NY 10016

LEWIS P. LARSON
68 13th Ave.
Columbus, OH 43201

CHARLES H. LEVINE
61 Beaumont Street
Brooklyn, NY 11235

SIDNEY B. LEVINSON
20-B John Adams Court
Cranbury, NJ 08512

ROBERT W. MATLACK
443 Crescent Ave.
Moorestown, NJ 08057

LOREN B. ODELL
416 Crestwood Dr.
Houston, TX 77007

CHARLES OPPERMANN
9848 S. Damen Ave.
Chicago, IL 60643

ED PETERSON
Peterson Paint Co.
P.O. Box 311
Pueblo, CO 81002

WILLIAM ROSE
3360 Mathieson Drive, N.E.
Atlanta, GA 30305

ELIAS SINGER
Troy Chemical Corporation
One Avenue L
Newark, NJ 07105

HERMAN J. SINGER
300 Winston Drive
Cliffside Park, NJ 07010

G.O. STEPHENSON
2241 Palestra #14
St. Louis, MO 63146

ED WANDERMAN
8901 Shore Rd.
Brooklyn, NY 11209

DuPont DBE The safe, low- alternative to

COMPARISON OF REPRESENTATIVE PRICES OF SLOW ESTER-TYPE SOLVENTS*

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

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

DuPont DBE is a safe solvent. As the chart shows, it's also the lowest-cost high-boiling solvent in the ester class. For over a decade, DuPont DBE has been substituted for E-series glycol ether acetates by paint and coatings manufacturers. Here's why reformulating with blends of DuPont DBE solvent could be your most cost-effective route:

Low-cost blending. You minimize costs by making DuPont DBE your retarder solvent when you reformulate with P-series glycol ether acetates. The chart below shows how blends of DuPont DBE can reduce the impact of solvent replacement.

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

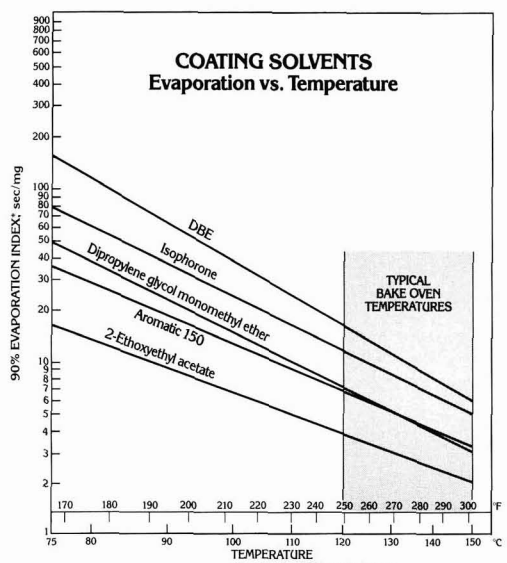
	EPOXY ENAMEL		HIGH SOLIDS POLYESTER		ACRYLIC LACQUER	
SOLVENT	**OLD FORMULA (WT. %)**	**REPLACEMENT* FORMULA (WT. %)**	**OLD FORMULA (WT. %)**	**REPLACEMENT* FORMULA (WT. %)**	**OLD FORMULA (WT. %)**	**REPLACEMENT* FORMULA (WT. %)**
DuPONT DBE	–	15	–	12	–	5
PM ACETATE	–	25	–	38	–	8
MEK	–	–	47	50	26	26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

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

**Replacement formulas are offered for experimental purposes only.*

solvent: cost EE acetate.

Comparable evaporation rates. Du Pont DBE is a slow-evaporating solvent that can improve gloss and leveling, reduce orange peel and minimize solvent popping. Its evaporation rate increases more rapidly than other slow solvents as temperature increases. And at bake-oven temperatures the evaporation rate of Du Pont DBE is comparable to other solvents, as shown in the graph.



*90% EVAPORATION INDEX = $\frac{\text{seconds for 90\% weight loss}}{\text{milligrams of sample}}$
(measured under N_2 flow of 50 ml/min.)

Low toxicity. No human health problems have been reported to date from the use of dibasic esters.

Wide application. Du Pont DBE's solvency for many polymers accounts for its use in a wide range of coatings, both conventional and high-solids. These uses include steel and aluminum coil coatings, interior and exterior can coatings and in finishes for appliances and metal furniture. Du Pont DBE is also used as a solvent in automotive acrylic lacquer and enamel topcoats, urethane trim paints, and polyester and epoxy primers.

Call toll free. Dial 800-527-2601 for help in reformulating with Du Pont DBE. We can also put you in touch with your local Chemcentral, Houghton, Inland Leidy or Gage Products Company distributor for information on low-cost, safe Du Pont DBE solvent.

DU PONT
REG. U.S. PAT. & TM. OFF.

Constituent Society Membership Figures Based on the 1983-1984-1985 Year Books

	1983				1984				1985			
	Active	Assoc.	Other	Total	Active	Assoc.	Other	Total	Active	Assoc.	Other	Total
Baltimore	128	78	11	217	130	75	16	221	135	77	13	225
Birmingham	147	—	9	156	156	—	10	166	158	—	9	167
Chicago	626	168	14	808	644	195	15	854	678	219	17	914
C-D-I-C	124	36	8	168	130	36	10	176	123	40	8	171
Cleveland	287	57	38	382	281	54	36	371	257	50	36	343
Dallas	95	26	2	123	90	35	4	129	73	34	5	112
Detroit	195	33	10	238	208	46	11	265	247	52	14	313
Golden Gate	184	32	5	227	182	52	7	241	180	56	6	242
Houston	112	46	13	171	106	50	13	169	109	50	13	172
Kansas City	66	30	12	108	69	33	10	112	80	33	10	123
Los Angeles	358	176	55	589	344	169	53	566	345	164	62	571
Louisville	119	62	6	187	113	58	7	178	113	55	4	172
Mexico	83	29	—	112	83	29	—	112	77	61	—	138
Montreal	157	124	26	303	149	99	20	268	137	72	16	225
New England	139	84	26	249	139	62	21	222	160	69	21	250
New York	405	123	26	554	389	132	37	558	382	149	37	568
Northwestern	138	45	15	198	149	39	10	198	156	52	13	221
Pacific N.W.	136	52	9	197	154	47	11	212	175	54	6	235
Philadelphia	161	66	35	262	151	82	32	265	164	77	23	264
Piedmont	96	63	3	162	101	75	6	182	98	71	7	176
Pittsburgh	134	32	10	176	138	27	9	174	132	28	11	171
Rocky Mountain	60	—	4	64	62	—	5	67	65	—	4	69
St. Louis	64	35	13	112	61	32	14	107	71	35	7	113
Southern	279	213	20	512	287	200	21	508	277	193	18	488
Toronto	204	165	1	370	203	142	3	348	246	161	5	412
Western N.Y.	66	10	5	81	63	12	5	80	70	15	5	90
TOTALS	4563	1785	372	6720	4582	1781	386	6749	4708	1867	370	6945

Does business stress cause high blood pressure?

Stress on the job is a real problem for most of us. Many people think high-pressure jobs cause high blood pressure.

Scientists and doctors aren't sure if stress causes high blood pressure. But one thing is for sure: *anybody*, no matter how they react to stress, can have high blood pressure.

If you have high blood pressure, you can control it—with medication, weight control, less salt, and whatever else your doctor tells you to do, every day.

No matter what you do for a living... keep on living.

High blood pressure. Treat it and live.

National High Blood Pressure Education Program,
National Heart, Lung, and Blood Institute,
U.S. Department of Health and Human Services

In coatings, we have the colors to match your bright ideas.

When high-performance pigmentation is critical, SCM Pigments sets the tone with titanium colors.

Our Ti Yellow™ and Golden® pigments for coatings give you excellent weatherability, heat and light stability, and color uniformity

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SCM CORPORATION
BALTIMORE, MARYLAND 21230

And our color experts will give you technical help that's backed by 60 years of experience and quality. Available to you in our labs or at your facility.

One bright idea deserves another. So get more information on SCM Pigments' titanium colors. Call our toll-free customer service number: 1-800-638-3234.

1984 Marks Record Year for Coil Coatings Industry

The coil coating industry placed a record-shattering year into the books in 1984.

A total of 4.17 million tons of coil coated steel and aluminum were consumed in 1984, according to estimates by the National Coil Coaters Association (NCCA). This was the highest figure in the industry's history, surpassing the previous record of 4.06 million tons set in 1978.

The 1984 record represented a 10.0% increase over 1983 figures. And this gain came on top of a 22.5% increase in 1983 compared to 1982.

Industry shipments of coil coated steel pushed 12.5% ahead of 1983 totals, according to NCCA estimates.

Coil coated aluminum deliveries slipped 3.4% over the same period, NCCA figures indicate.

The NCCA compiles these estimates of total shipments based on actual shipment tonnage reported to the association by both member and non-member companies. Statistics are calculated for North American markets (U.S., Canada, Mexico) only.

The extended economic recovery was the major factor in the impressive 1984 figures, NCCA member executives agreed. But in certain areas, coated coil continued to demonstrate increased market penetration because of its cost-saving features.

The coil coated steel marketplace was once again dominated by the transportation segment during 1984. A key factor was shipments to the automotive industry; carmakers posted yet another strong increase in their use of steel with coil coated, weldable zinc-rich primers. Auto manufacturers use the coil coated steel extensively for corrosion protection in body panels of cars, light trucks and vans. This one product category increased 16.7% in 1984.

Building products, the second-most-important industry user of coil coated steel, registered more moderate gains for the year. Shipments to the appliance industry also increased modestly—although NCCA member-reported tonnage of coil coated steel used in refrigerators and freezers has increased nearly 75% since 1982.

Other coated steel end-use markets which gained markedly during 1984 were roof decking, residential siding and roofing, and lighting products. NCCA members reported over 100,000 tons shipped to this last market for the first time in 1984.

Coil coated aluminum relied on its traditional bulwark industries, building products and containers and packaging, for its 1984 performance. The light metal in coil coated form held its own in 1984 despite intense pressure from competitive materials in both markets.

Metal cans, ends, and tabs again consumed extensive square footage of coil coated aluminum (which weighs only one-third as much as steel on average), and this remained the number one end-use market for aluminum coil coaters.

A variety of residential building products, including siding, roofing, soffit, awnings and mobile homes led the building products segment of coil coated aluminum's market picture. And reported tonnage shipped to the truck and trailer market rebounded strongly from a weak 1983 performance.

The coil coating industry has demonstrated sustained, rapid growth in tonnage shipped—interrupted only by major national recessions—since the NCCA began collecting statistics in 1962. Total estimated North American tonnage in that initial year was only 464,000 tons.

Coated coil is sheet steel or aluminum in coil form with organic paint or other finishes applied on automated, high-speed lines operating at speeds up to 900 feet per minute. The coated metal is delivered to users ready to fabricate, with no further finishing required.

Coated coil users thus gain major savings by eliminating paint lines and spray booths, energy-intensive curing ovens and expensive pollution control equipment of all types. Customers also benefit from more efficient production, increases in available plant space and extremely precise application of the coating with little or no waste, NCCA members assert.

Union Carbide Expands Latex Plant in Puerto Rico

The UCAR Emulsion System Div., of Union Carbide Corp., Danbury, CT, has completed an expansion and modernization project at its latex plant in Puerto Rico. Located in Bayamon, P.R., the plant has been enlarged to permit the relocation of personnel responsible for those services previously performed at other locations on the island.

To modernize the plant, computer equipment has been installed. The new system will assist the accounting and billing departments by providing automated inventory control and material costing. In the technical service laboratory, the system will serve an integral part in the firm's expanded quality assurance program.

Ferro Acquires Spanish Coatings Manufacturer

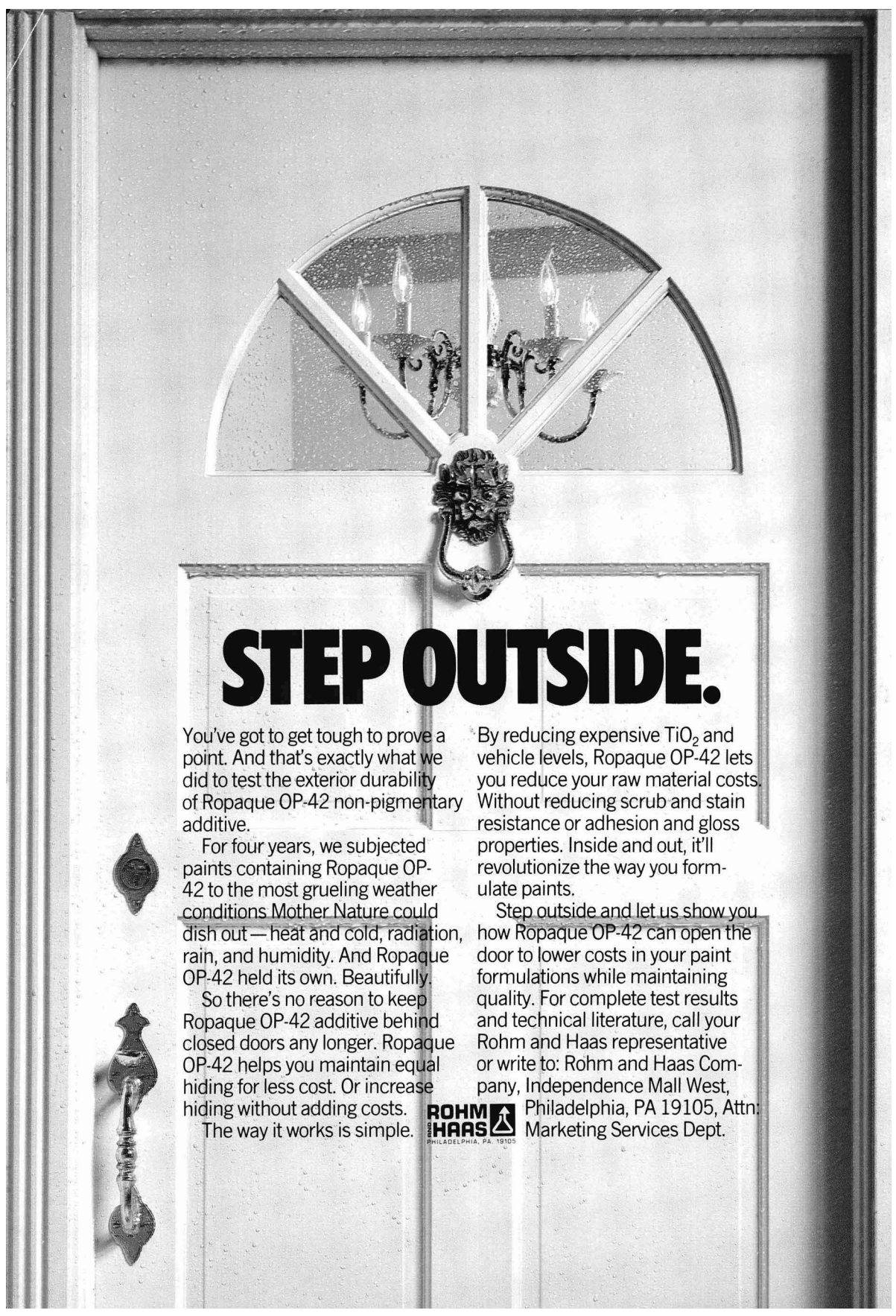
Ferro Corporation, Cleveland, OH, has purchased Inmapin Priba, S.A. of Madrid, Spain. Inmapin Priba is the largest powder coatings manufacturer in Spain. Adolph Posnick, Ferro's President and Chief Executive Officer, stated, "This acquisition, coupled with our powder coatings expansion activities in the U.S. and other areas of the world, emphasizes Ferro's commitment to being a worldwide supplier of organic powder coatings, as well as traditional inorganic coatings."

Special Discount Fares Available from TWA To Annual Meeting

Special arrangements have been made with TWA to offer discounted fares within the U.S. to/from St. Louis, MO, for the October 7–9 Annual Meeting and Paint Show at the Cervantes Convention Center. These special fares are available only when you call the unlisted toll-free number (800-325-4933); in Missouri only, call (800-392-1673). Be sure to ask for lowest fare available! You must give the FSCT Convention number which is:

#9911077

IF YOU
WANT PROOF
THAT ROPAQUE®
OP-42 CAN
STAND UP TO
MOTHER
NATURE...



STEP OUTSIDE.

You've got to get tough to prove a point. And that's exactly what we did to test the exterior durability of Ropaque OP-42 non-pigmentary additive.

For four years, we subjected paints containing Ropaque OP-42 to the most grueling weather conditions Mother Nature could dish out — heat and cold, radiation, rain, and humidity. And Ropaque OP-42 held its own. Beautifully.

So there's no reason to keep Ropaque OP-42 additive behind closed doors any longer. Ropaque OP-42 helps you maintain equal hiding for less cost. Or increase hiding without adding costs.

The way it works is simple. By reducing expensive TiO_2 and vehicle levels, Ropaque OP-42 lets you reduce your raw material costs. Without reducing scrub and stain resistance or adhesion and gloss properties. Inside and out, it'll revolutionize the way you formulate paints.

Step outside and let us show you how Ropaque OP-42 can open the door to lower costs in your paint formulations while maintaining quality. For complete test results and technical literature, call your Rohm and Haas representative or write to: Rohm and Haas Company, Independence Mall West, Philadelphia, PA 19105, Attn: Marketing Services Dept.

ROHM AND HAAS
PHILADELPHIA, PA. 19105

PPG to Open Environmental Sciences Center

PPG Industries, Inc., Pittsburgh, PA, has agreed to purchase Gulf Oil Corp.'s Life Sciences Center. The 63,000-sq ft center located in O'Hara Township will be used to expand the Coatings and Resins Group's activities in the field of environmental safety and health.

The group's environmental and safety affairs department will add approximately 10 persons to its staff of 44, and carry out four basic functions: industrial hygiene, toxicology assessment, safety and loss prevention, and environmental engineering and control. Included in these functions are developing odor controls, evaluating the ecological effects of new materials, and developing product labeling and control systems for manufacturing operations.

The new facility is expected to aid in PPG's future coatings-related growth through advanced product technology while protecting the health of employees, customers, and safeguarding the environment.

Air Products Increases Pressure Emulsion Capacity

Air Products and Chemicals, Inc., Allentown, PA, has increased its vinyl acetate-ethylene and ethylene-vinyl chloride pressure emulsions capacity by 40% at two of its facilities.

To meet the growing demand for the company's water-based products, the Calvert City, KY, and South Brunswick, NJ, plants have been expanded. Incorporated into the expansion are computer-based process control technology designed to provide a higher degree of product consistency and to increase the energy efficiency of the production process.

Air Products also operates polymer emulsion plants in Elkton, MD; Cleveland, OH; and City of Industry, CA.

Thibaut & Walker Enlarges Production Facilities

Thibaut & Walker, Newark, NJ, has expanded its production facilities as part of an ongoing program to increase productivity and customer service capability yet maintain low operating costs.

The expansion centers on the addition of a 12,000-gallon urethane reactor and includes new 20,000-gallon agitated storage tanks. These developments are estimated to increase storage capacity for finished products by 50%. A high-capacity truck-loading facility has also been included in the expansion.

Viking Pump Is Subsidiary Of Houdaille Industries

Viking Pump Division of Houdaille, Cedar Falls, Iowa, has become a separate, wholly-owned subsidiary of Houdaille Industries, Inc. Under its new corporate name of Viking Pump-Houdaille, Inc., the firm will conduct all of the operations formerly conducted by the Viking Pump Division of Houdaille.

THE BIG

50 1985

Fiftieth Anniversary of the Paint Industries' Show

CERVANTES CONVENTION CENTER
ST. LOUIS, MISSOURI OCTOBER 7-9

We'd like to teach your employees to tie you up, douse you with water, and pound you on the back.

It could save your life. And severe bleeding, chemical burns and choking are just three of the emergencies we can prepare you for.

Our courses can familiarize your employees with almost every phase of workplace safety, from simple accident prevention to first aid and CPR.

We're the American Red Cross. We're working hard to keep everyone working hard.

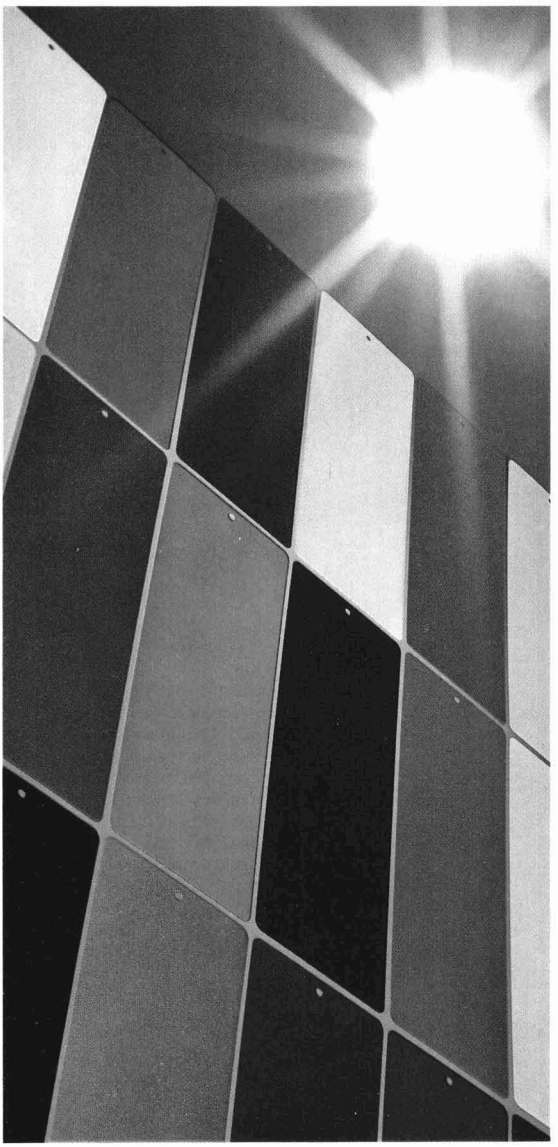
American Red Cross

We'll help. Will you?

A Public Service of This Magazine & The Advertising Council

In sunlight, coatings can die in months

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



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

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

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

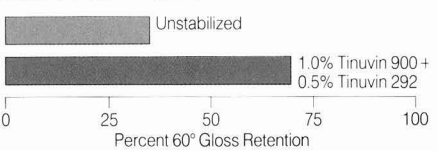
The first class is the hindered amine light stabilizers (HALS), such as Tinuvin® 292, Tinuvin 144 and Tinuvin 079L. They protect gloss and help prevent loss of physical properties that may result in chalking, erosion, water permeability and checking.

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

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

Percent 60° Gloss Retention After Two Years of Exposure

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



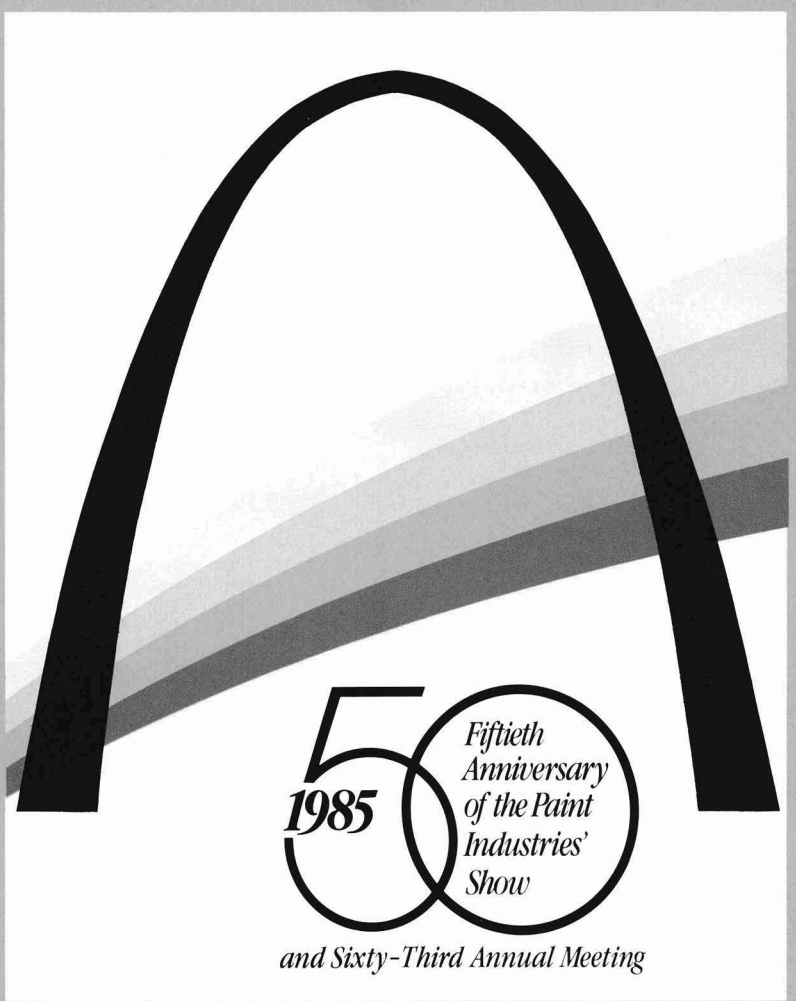
Manufacturers of maintenance, coil and powder coatings can now formulate their products to yield substantially longer service life.

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

CIBA–GEIGY

FEDERATION OF SOCIETIES FOR COATINGS TECHNOLOGY

Housing and Advance Registration Forms



CERVANTES CONVENTION CENTER
ST. LOUIS, MISSOURI OCTOBER 7-9

1985 ADVANCE REGISTRATION

FEDERATION OF SOCIETIES FOR COATINGS TECHNOLOGY

1315 Walnut St., Philadelphia, PA 19107

C U V	Office Use Only
	Date received _____
	Amount $ _____
	Check No. _____

Please fill out this form and mail with a check in the correct amount (made payable to the FSCT) to the Federation address shown above. All checks must be payable in U.S. Funds. Any that are not will be returned. DEADLINE DATE FOR ADVANCE REGISTRATION IS SEPTEMBER 6. NONE WILL BE ACCEPTED AFTER THAT DATE.

A $10.00 charge will be made for cancellations received prior to September 6. No refunds will be made after that date.

INDUSTRY REGISTRATION FEES:

A ☐ MEMBER $50.00

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

_____
Federation Constituent Society

B ☐ NON-MEMBER $65.00

G ☐ SPECIAL FEE FOR RETIRED MEMBERS $25.00

_____
Federation Constituent Society

INFORMATION FOR REGISTRATION BADGE:

NICKNAME

FIRST NAME LAST NAME

BUSINESS AFFILIATION

STREET

CITY STATE *(U.S. only)*

MAILING ZONE COUNTRY (OTHER THAN U.S.)

BUSINESS CLASSIFICATION DATA FOR THE ABOVE REGISTRANT:

YOUR COMPANY (CHECK ONE BLOCK ONLY)

AA ☐ Manufacturers of Paints, Varnishes, Lacquers, Printing Inks, Sealants

BB ☐ Manufacturers of Raw Materials

CC ☐ Manufacturers of Equipment and Containers

DD ☐ Sales Agent for Raw Materials and Equipment

EE ☐ Government Agency

FF ☐ Research/Testing/ Consulting

GG ☐ Educational Institution Library

HH ☐ Paint Consumer

JJ ☐ Other

YOUR POSITION (CHECK ONE BLOCK ONLY)

KK ☐ Management/ Administration

LL ☐ Manufacturing and Engineering

MM ☐ Quality Control

NN ☐ Research and Development

PP ☐ Technical Sales Service

QQ ☐ Sales and Marketing

RR ☐ Consultant

SS ☐ Educator/Student/ Librarian

TT ☐ Other

SPOUSES REGISTRATION AND INFORMATION FOR REGISTRATION BADGE:

D ☐ SPOUSE $35.00

SPECIAL FEE FOR THE SPOUSES OF RETIRED MEMBERS ONLY:

H ☐ $25.00

NICKNAME

NAME

ADDRESS

CITY STATE

MAILING ZONE COUNTRY (OTHER THAN U.S.)

TICKETS FOR FEDERATION LUNCHEON, WEDNESDAY, OCTOBER 9 (@ $20.00)

Z ☐ NUMBER REQUIRED: _____ $20.00 EACH.

A CHECK IN THE AMOUNT OF:

$ _____

IS ENCLOSED

Plan to Attend the

Federation Annual Awards Luncheon

at the

Sheraton St. Louis Hotel • October 9th

Featuring Speaker

JIM VALVANO

Head Basketball Coach • North Carolina State University

Tickets—$20 per person

In advance or at Registration during Annual Meeting

FSCT 1985 ANNUAL MEETING AND PAINT INDUSTRIES' SHOW
CERVANTES CONVENTION CENTER, ST. LOUIS, MISSOURI
OCTOBER 7, 8, 9 (Monday, Tuesday, Wednesday)

APPLICATION FOR HOTEL ACCOMMODATIONS

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

Please indicate below the type of accommodations requested and choice of hotels. All reservations will be processed by the Housing Bureau of the St. Louis Convention & Visitors Bureau. Hotel assignments will be made in accordance with prevailing availability. The Housing Bureau will send you an acknowledgment, noting the hotel to which you have been assigned. (Bureau telephone # 314-421-1166). The confirmation of your reservation will come to you directly from the hotel, to whom you must direct all inquiries (phone numbers in this brochure).

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

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 OF ROOM OCCUPANTS AND DATES OF ARRIVAL DEPARTURE

Type of Room	Name	Dates Arrive	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 _____

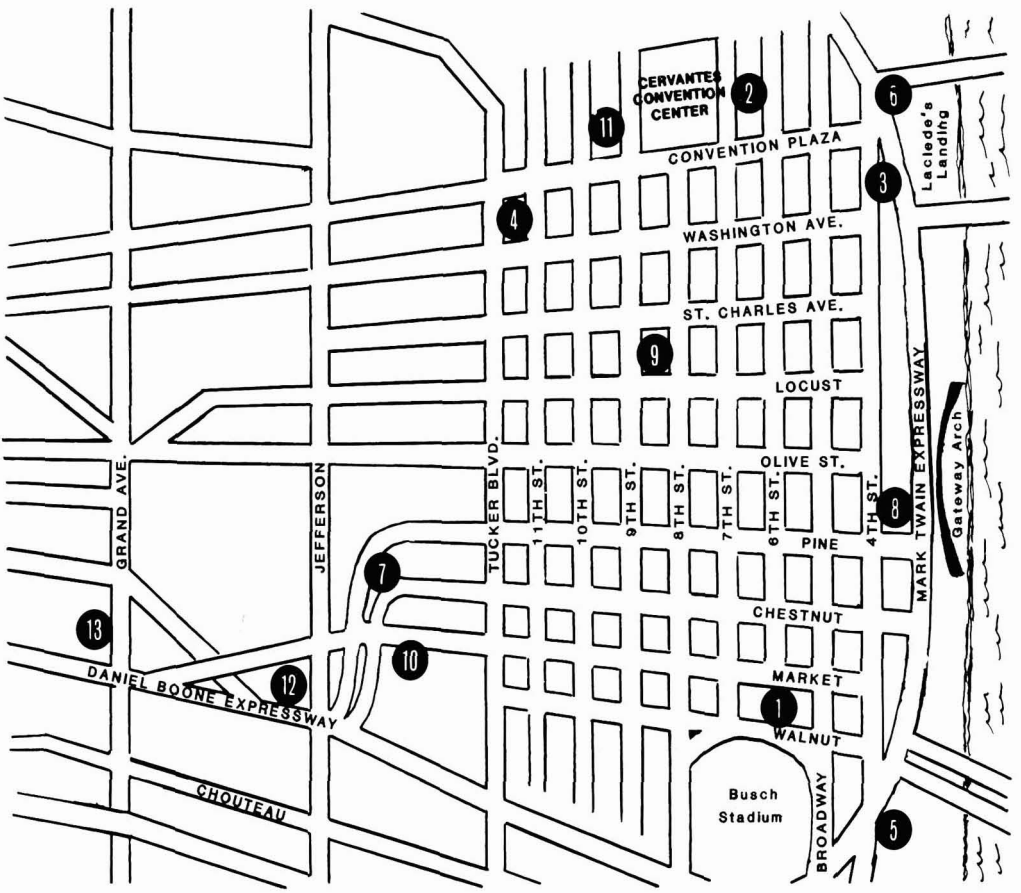
Telephone _____

Note: Requests for accommodations at the Marriott or Sheraton will be limited to five rooms per company. A parlor counts as one room.

No. on Map	Hotel	Singles	Doubles/ Twins	Parlor & 1 Bedroom	Parlor & 2 Bedrooms
(1)	†*Marriott Pavilion*	$74	$86	$185–$295	$225–$375
(2)	†Sheraton St. Louis	74	86	275	
(3)	Bel Air Hilton	72	84	175	350
(4)	Best Western St. Louisian	46	54		
(5)	Clarion	72	84	195–315	325–405
(6)	Embassy Suites	80	90		
(7)	Holiday Inn Market St.	54	61		
(8)	Holiday Inn Riverfront	70	80		
(9)	Mayfair	59	69		
(10)	Omni—Union Station	83	96	250 & up	375 & up
(11)	Radisson St. Louis	65	81	110	380–450
(12)	Rodeway Inn	48	54		
(13)	University Plaza	49	55		

* All room rates are subject to a City and State Tax of 9.75%.

† Requests for accommodations at the Marriott Pavilion or the Sheraton St. Louis will be limited to five rooms per company. A parlor counts as one room. Additional reservations will be assigned to other cooperating hotels.



FEDERATION OF SOCIETIES FOR COATINGS TECHNOLOGY
1985 ANNUAL MEETING AND PAINT INDUSTRIES' SHOW
CERVANTES CONVENTION CENTER, ST. LOUIS, MISSOURI
MONDAY THROUGH WEDNESDAY, OCTOBER 7, 8, 9

The Combined Annual Meeting and Paint Industries' Show Is a Major Educational Activity of the Federation. This Significant Coatings Industry Event Consists of Three Days of Technical Program Sessions and Exhibits, Running Concurrently.

"COATINGS RESEARCH AND DEVELOPMENT: TODAY'S INVESTMENT IN TOMORROW"

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Show hours will be: Monday—12:00-5:30; Tuesday—9:30-5:30; Wednesday—9:30-3:00.

FEDERATION BOARD TO MEET ON SUNDAY

The Federation's Board of Directors meeting will be held on Sunday, October 6, in the Marriott Hotel.

HOTELS AND RESERVATIONS

Thirteen hotels have reserved blocks of rooms for the Federation. A map showing the location of the hotels and a schedule of rates are included in this brochure.

All reservations will be processed by the St. Louis Convention and Visitors Bureau. The Bureau will send you an acknowledgment, noting the hotel to which you have been assigned. (Bureau telephone # 314-421-1166). The confirmation of your reservation will come directly from the hotel, to whom you must direct all inquiries.

Phone numbers of the hotels (Area Code 314) are:

Marriott Pavilion	421-1776	Holiday Inn Riverfront	621-8200
Sheraton St. Louis	231-5100	Mayfair	231-1500
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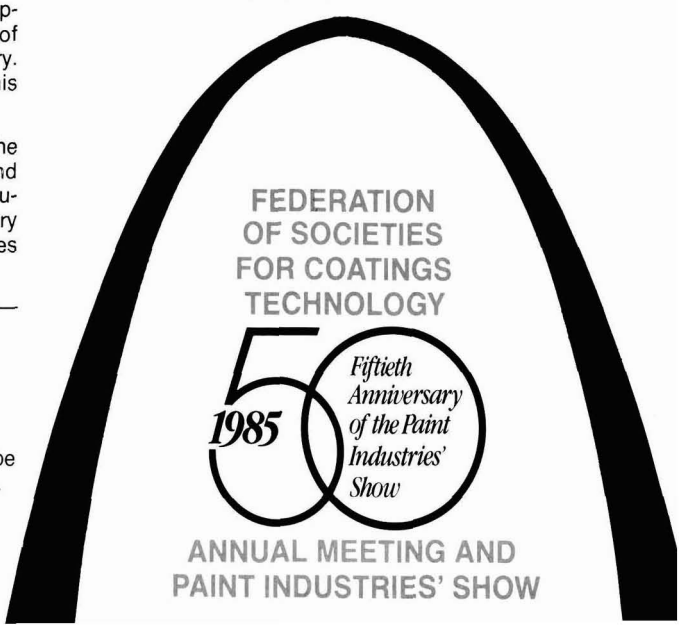
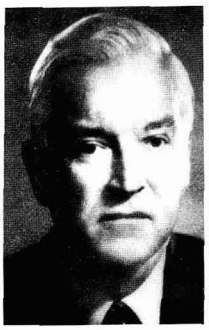


JOHN P. McANDREWS, OF DUPONT, WILL GIVE KEYNOTE ADDRESS

The Federation is pleased and honored to announce that John P. McAndrews, Group Vice-President—Finishes & Fabricated Products, DuPont Co., will present the Keynote Address at the Monday morning opening session. Mr. McAndrews joined the DuPont Co. in 1947 as a chemical engineer at the Marshall Lab. in Philadelphia. In succeeding years he advanced through several posts within the F & FP Dept. He was transferred to Remington Arms in 1966 and elected President in 1979. He was named Vice-President of the F & FP Dept. in 1982 and assumed his present position in 1983.



ANNUAL FEDERATION LUNCHEON TO BE HELD ON WEDNESDAY

The Annual Federation Luncheon will be held on Wednesday in the Sheraton. It will feature a guest speaker and the presentation of the Paint Show, Heckel, Roon, and other awards.

SPOUSES ACTIVITIES TO BEGIN WITH MONDAY AFTERNOON SOCIAL

A get acquainted wine and cheese social in the Convention Center will open the Spouses Program on Monday afternoon. There will be continental breakfasts on Tuesday and Wednesday morning and a special tour with lunch on Tuesday.

TWA NAMED OFFICIAL AIRLINE FOR ANNUAL MEETING AND PAINT SHOW

The Federation has appointed TWA as the official airline for the convention in St. Louis. For flights within the United States, phone the toll-free number: 800-325-4933 (in Missouri 800-392-1673). You must give the Federation's account number: 9911077. Be sure to ask for the lowest fare available. Applicable dates of travel for the special fare are October 4–12, 1985.

HOST COMMITTEE

The Federation's two Societies in Missouri (St. Louis and Kansas City) will serve as the official hosts for the Annual Meeting and Paint Show. The General Chairman is Howard Jerome, of Spatz Paint Industries, Inc. Subcommittee Chairmen are: Spouses—Mrs. Howard (Gene) Jerome; Program Operations—John Folkerts, of Future Coatings; Registration Area—Joseph W. Wrobel, Jr., of Ciba-Geigy Corp.; Information Services—William Reckel, of Amteco, Inc.; and Liaison—Nick Dispensa, of Davis Paint Co.

PROGRAM COMMITTEE

Joseph A. Vasta, of the DuPont Co., F&FP Dept., Wilmington, DE, is the Chairman of the Federation's Program Committee. The Vice-Chairman is Percy E. Pierce, of PPG Industries, Inc., Allison Park, PA. Other members of the committee are: Taki Anagnostou, of AKZO (Wyandotte) Paint Products, Inc., Troy, MI; William H. Ellis, of Chevron Research Co., El Segundo, CA; Richard Eley, of Glidden Coatings & Resins Div., Strongsville, OH; Stanley LeSota, of Rohm and Haas Co., Spring House, PA; John S. Ostrowski, of Valspar Corp., Minneapolis, MN; David Richie, of Midland Div., Dexter Corp., Waukegan, IL; and James Lee, of Lanchem Corp., E. St. Louis, IL.

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Slime Films on Antifouling Paints

Short-Term Indicators of Long-Term Effectiveness

Maurice G. Robinson, Beverley D. Hall, and Domenico Voltolina
Royal Roads Military College*

A method for an initial evaluation of the biological effectiveness of antifouling paints is described. It involves exposing test surfaces for three to four months under natural marine conditions and examining the resulting slime film by scanning electron microscopy or optical microscopy. The presence of a large number of diatom species indicates that the paint is not biologically effective and will not prevent macrofouling. If species diversity is significantly reduced, and especially if the population consists of known toxin-tolerant species, the paint warrants further testing. Slime film biomass is not a good measure of the effectiveness of antifouling paints. The paint should be applied over the same undercoat as in the potential working situation.

INTRODUCTION

Until recently the major goal of manufacturers of marine paints has been the prevention of macrofouling on submerged surfaces and little attention has been given to the formation of the primary slime film (bacteria, microalgae, protozoa, and their cellular exudates). This film develops even on the most effective antifouling paints[1-3] and precedes the development of macrofouling on unpainted surfaces and on ineffective antifouling paints. It is now recognized that elimination of this film would be of direct economical and functional significance, since its formation increases the frictional resistance of a ship's hull with an accompanying increase in fuel consumption.[4-7] Most of the present day studies on primary slime film development attempt to understand the physiological processes involved in the resistance of these microfouling organisms to the toxic environment represented by an antifouling-painted surface.[2,8,9]

The intent of this work was to establish a relationship between either the development of the slime film (biomass) or its species composition and the subsequent attachment of macrofouling organisms. Standard tests for antifouling paints involve suspension of painted panels (minimum surface area 465 cm^2 per side) at depths of 0.3 to 3m and examination at monthly intervals for a minimum period of one year.[10,11] Since measurable slime films are formed in a few months, if a relationship between either slime film biomass or species composition and subsequent macrofouling could be established, the initial screening of experimental formulations could then be reduced to three or four months. For this purpose we tested two paints with very effective antifouling properties as well as some which are known to fail and on which barnacle settlement has been observed.

MATERIALS AND METHODS

Our experiments began with a 12 month study (Stage 1—December 1, 1982 to December 1, 1983) on the qualitative composition of the populations developing on test aluminum surfaces painted with one of three antifoulants: a cuprous oxide vinyl paint (Canadian Government Specifications Board (CGSB) 1-GP-123, see *Table* 1) henceforth designated as Cu_2O; and two commercial paints, one a vinyl paint employing a minimum of 10–15% of tributyltin fluoride (TBTF) and the other a self-polishing copolymer (SPC) utilizing tributyltin and copper (form and concentrations unknown) as the active toxic ingredients. All have been used on Canadian vessels. While Cu_2O and SPC are known to be very effective, the use of TBTF has recently been discontinued to a large extent due to its failure to prevent

*Dept. of Chemistry, F.M.O. Victoria, B.C. VOS 1BO Canada.

Table 1—Composition of Standard Cuprous Oxide Paint And Experimental Formulations (% wt)

Ingredient	Standard Cuprous Oxide	Experimental Formulations	
	1-GP-123	Lab Black #3	Lab Black #6
Cu_2O	70.3	20	0.0
Cu_2S	0.0	20	40.7
Rosin WW	10.5	7.3	7.3
di-octylphthalate	2.4	2.2	2.2
MIBK	8.1	23.0	23.0
Toluene	5.5	15.7	15.7
Antisettling agent	0.5	0.5	0.0
Vinyl resin (VYHH)	2.7	7.3	7.3
Carbon black	0.0	3.8	3.1

barnacle attachment (T. Foster, private communication). In the second stage (August 29, 1983 to January 29, 1984) we added qualitative and quantitative observations on acrylic plastic surfaces painted with the above paints. Finally, additional tests were run between October 19, 1983 and February 19, 1984 (Stage 3). During this stage both qualitative and quantitative observations were made on aluminum surfaces painted with the above paints, and three additional paints. Two of these were experimental formulations: Lab Black #3 (LB3), which utilizes as toxins equal parts of Cu_2O and Cu_2S, and Lab Black #6 (LB6) which utilizes only Cu_2S (*Table* 1). These are currently being tested by DREP* and indicate possible failure. The third was a commercially available wax (AW), with bis-(tributyltin)oxide (12%) as the toxic component. Doubts exist as to the effectiveness of this wax.

For the first and third stages, round aluminum discs (alloy 60-61) 25 mm in diameter × 2mm thick, with a small hole offset to one side were machined, sandblasted, cleaned, and spray coated with an anticorrosive aluminum vinyl paint (CGSB 1-GP-122). Twenty-four discs were used for each of the paints being tested and 24, sprayed only with aluminum vinyl (Al), were used as controls. Material and size of the discs were chosen as convenient for mounting in the scanning electron microscope (SEM) used for these studies. For the second stage we made use of acrylic plastic plates (10 cm × 10 cm × 0.3 cm) with a hole on one side, which underwent the same treatment as the aluminum discs except that no anticorrosive aluminum vinyl was applied as an undercoat. The controls were acrylic plastic plates painted with aluminum vinyl. Plates and discs were grouped according to treatment, attached with nylon screws to racks coated with the same paint being tested and submerged at an average depth of three meters from the stationary dock at Grant Knoll in Canadian Forces Base, Esquimalt, B.C., Canada. Once a month two discs and/or plates, selected at random, were removed from each rack for further examination.

For SEM studies the discs were immediately transferred to a coplin jar containing 2% glutaraldehyde in 77% seawater, fixed for 1.5 hours, rinsed several times with distilled water, dehydrated through an ethanol series to 100% ethanol, and critical-point dried through liquid CO_2. Since ethanol damages SPC paint, the SPC-treated discs were rapidly frozen in liquid nitrogen-cooled isopentane after the water rinse, and freeze-dried overnight in a Labconco freeze-drier. According to our observations the two drying methods gave comparable results on all other paints. All samples were sputter-coated with gold and examined with a JEOL 35 scanning electron microscope.

Within one hour of collection, the acrylic plastic plates for Stage 2 were divided into equal sections, one of which was used for chlorophyll *a* analysis, the other for dry weight measurement and for qualitative studies. The covering slime was removed with a rubber scraper and collected in filtered seawater. Samples for chlorophyll analysis were filtered in dull light onto 0.45 μm pore size Gelman filters with a few drops of $MgCO_3$ suspension, folded, wrapped in aluminum foil and frozen. Within two months they were processed according to Strickland and Parsons.[12] Chlorophyll *a* concentrations were calculated using Jeffrey and Humphrey's[13] equations.

Samples for dry weight and qualitative analysis were preserved with Lugol's solution. Within two months the samples were washed with distilled water and diluted to a known volume. A known aliquot was removed and stored in 33% HNO_3 for cell counts. The remaining sample was filtered onto predried (80°C for 1.5 hours), preweighed

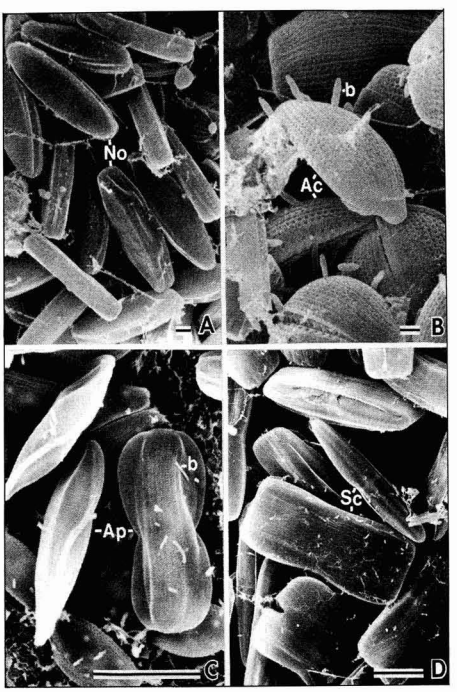

Figure 1—Diatom species tolerant to antifouling paints. (A) *Nitzschia ovalis* (No); (B) *Amphora coffeaeformis* (Ac); (C) *Amphiprora paludosa* (Ap); (D) *Stauroneis constricta* (Sc). See also Figures 2A, 4A. b, bacterium. Bar A, B, 1 μm; C, D, 10 μm

*Defence Research Establishment Pacific (DREP), CFB Esquimalt, F.M.O. Victoria, B.C., Canada.

Gelman A-E filters. The filters were redried at 80°C for 2.5 hours, reweighed, and the biomass determined by difference. For qualitative studies of the diatom populations a minimum of 500 cells was identified for each sample using an optical microscope.

For the third stage, procedures were as above except that all samples were freeze-dried and the biomass was measured only as number of cells per unit surface. After SEM examination the slime from each disc was scraped off with a scalpel blade into a known volume of 33% HNO_3 and known aliquots were counted using an inverted microscope.

As an indication of the effectiveness of the paints, we chose to use an index which measured the relative numerical importance of the diatom species present on our test surfaces. The data obtained suggested that the dominance indices, δ_1 and δ_2, described in Travers[14] would be adequate for our aims, as both underline the importance of the dominant species without considering the rare ones, the presence of which in our populations was probably due to contamination from ropes or surrounding surfaces, rather than to real adhesion to the test paints.

RESULTS

Stage 1

This consisted of qualitative observations protracted for 12 months on the composition of the communities developing on the paints mentioned below.

CONTROL: After one month, the community growing on our controls was already quite complex, with many bacteria, at least 10 species of diatoms, one species of protozoa, and two more algal species. The number of species continued to increase until month 4 (April), with the relative abundance of each species varying from month to month. Barnacles appeared between April and May. At this time the community was composed of two layers: the lower layer consisted of at least 15 species of attached pennate diatoms, early stages of an ectocarpoid alga, and tiny barnacles; the upper one consisted of large filamentous diatoms, at least six species of protozoa, and an ectoproct species. After May, SEM studies of the controls were impossible due to the size of the barnacles.

TRIBUTYLTIN FLUORIDE (TBTF): After one month, only a dense bacterial layer had developed, but one month later most of the surface was covered with a single layer of at least 10 species of pennate diatoms, as well as a protozoan and a non-diatom algal species. Until April, the biomass developing on TBTF seemed less than that on Al vinyl or Cu_2O, but the species diversity was comparable to that of Al vinyl and much greater than that of Cu_2O and SPC. Barnacles settled on TBTF between April and May and grew very rapidly, making further SEM examination impossible.

CUPROUS OXIDE (Cu_2O): Even after two months, only a very dense bacterial layer had developed. By April, dense continuous colonies of three species of diatoms: *Nitzschia ovalis* Arnott, *Amphora coffeaeformis* Agardh, and *Amphiprora paludosa* var. *hyalina* (Eulenstein ex Van Heurck) Cleve (*Figure* 1A,B,C) had formed a slime layer 100–1000μm thick. An amoeboid protozoan grazer was also present. After five months two other diatom species *Stauroneis constricta* (Ehrenberg?) Cleve (*Figure* 1D) and *Achnanthes brevipes* Agardh (*Figure* 2A) began multiplying. There was evidence of sloughing of the slime film as it approached a thickness of 1000μm. This left sparsely settled areas which were recolonized by the same five species. By September, an unidentified species of *Nitzschia,* nematodes, three protozoan species, and an unidentified arthropod completed the community. This paint prevented barnacle settlement.

Figure 2—(A) *Achnanthes brevipes* (Ab), a tolerant diatom species. s, stalk; (B-D) Fouling after three months. (B) Control, aluminum vinyl (Al); (C) Tributyltinfluoride antifouling paint (TBTF); (D) Antifouling wax (AW). Bar, 10 μm

SELF-POLISHING COPOLYMER (SPC): After two months, only small colonies of *A. paludosa* were present. This species continued to dominate while the cell density of *A. coffeaeformis* gradually increased until the end of the study. By April, the slime layer was still very patchy and thin (maximum thickness approximately 20 μm). Vorticellid protozoans and the diatom *S. constricta* were present by May. Barnacles did not settle on this paint.

Stage 2: Biomass Development

As expected, the controls supported a higher biomass than any of the paints tested; the biomass, whether measured as chlorophyll *a* (*Figure* 3A) or as dry weight

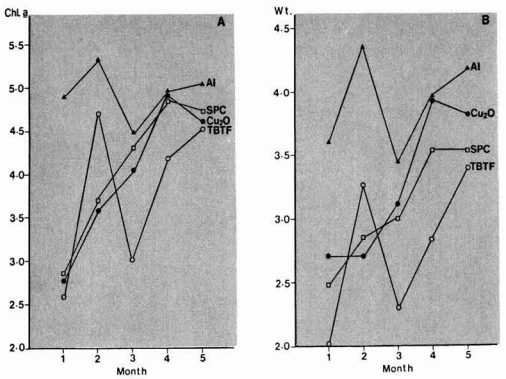

Figure 3—Biomass development from Sept. 19, 1983-Feb. 19, 1984. (A) Chlorophyll *a*, as $\log_{10}$ (Chl *a*, $\mu g \cdot m^{-2}$). (B) Dry weight, as $\log_{10}$ (Dry Weight, $mg \cdot m^{-2}$)

(*Figure* 3B), increased rapidly over the first two months, decreased drastically during the third, and increased again for the next two months. Of the other paints, only TBTF showed a comparable trend, albeit with far lower values. Both Cu_2O and SPC showed slow growth up to month 3 and a sudden increase during the fourth month which was attenuated during the fifth month. Significant variations in biomass, and particularly the reduced growth or decline in November/December, could be caused by seasonal effects and grazing by zooplankton, protozoa, and molluscs. These general factors appeared to affect all three paints and the control. We expected to see a direct relationship between the development of the slime film and subsequent failure of a paint leading to macrofouling. This was not observed. To the contrary, a higher biomass was generally found on the two most effective paints (SPC and Cu_2O) than on TBTF, the paint known to fail.

Table 2 summarizes the results of our observations on the qualitative composition of the respective diatom populations. We decided not to utilize the data of month 1 for both Stage 2 and Stage 3 due to the very sparse populations present on all paints. Under these circumstances, occasional species, which might settle on painted surfaces but fail to colonize them, would assume undue importance and contribute a wide and unwarranted variability to the data.

All antifouling paints were effective in reducing the number of species which colonized the test surfaces, with fairly high and statistically equal percentages of the total population being represented by a maximum of two species. However, TBTF seemed to allow a higher diversity, with the number of species varying between eight and 15, while both Cu_2O and SPC painted surfaces hosted a maximum of four diatom species. It seems noteworthy that the dominant species (*N. ovalis*) on the copper-based paint was different from the two species which dominated the populations on the paints which used tin (or tin and copper) as their toxic principle, suggesting lack of tin tolerance for this species.

Stage 3: Qualitative and Quantitative Population Studies

Tables 3 and 4 and *Figures* 2 and 4 summarize the results of this final part of our research. In *Table* 3, the means of the indices δ_1 and δ_2 for the last three months are given, and compared to those of the controls. *Table* 4 sums up our SEM observations and classifies the species found into three categories, according to their ability to withstand the toxic substances present in antifouling paints. This classification, clearly provisional and in need of further laboratory studies, is partly based on the results of our observations both under natural conditions and in the laboratory, and partly on data available in the literature.[1,15-18] The most tolerant diatoms (MT) are those which were consistently found in the slime film formed on the most effective paints we tested, on ships' hulls observed in the local drydocks, and which our preliminary laboratory tests have proven to be resistant to copper, tin, or both. *Nitzschia ovalis, A. coffeaeformis* and *A. paludosa* make up this group. The second category, tolerant diatoms (T), contains those species (*S. constricta, A. brevipes,* and *Synedra investiens* Wm. Smith, *Figure* 4A), which appeared on effective paints after about five months. They probably depend, for their tolerance, on the previous existence of a slime film and on methods of attachment such as stalks (*Figure* 2A) or apical mucous pads (*Figure* 4A), which allow them to minimize their contact with the painted surface. Finally, the third group, non-tolerant diatoms (NT), consists of all those species which were found only on controls and ineffective antifouling paints in these waters.

Both tables show that most of the paints are effective in increasing the dominance of selected species and reducing the numbers of species settled. However, an evident gradation exists, from the very effective Cu_2O on which

Table 2—Means and Confidence Limits (95%) for the Dominance Indices (δ_1 and δ_2), Dominant Species (D.S.), And Number of Species Found, for the Experiments in Stage 2

Paint	δ_1	t vs Al	δ_2	t vs Al	D.S.	# Species
Al	35.4 ± 16.0	/	49.3 ± 20.2	/	None	26-34
Cu_2O	96.8 ± 6.6	***	98.1 ± 5.9	**	*N. ovalis*	2-4
SPC	61.2 ± 9.5	*	98.5 ± 2.2	**	*A. paludosa, A. coffeaeformis*	2-4
TBTF	76.2 ± 11.5	**	90.1 ± 9.9	**	*A. paludosa, S. constricta*	8-15

The significance of the comparison of the mean δ_1 and δ_2 (two-tailed t test) to those of the control are also indicated (*** 0.001; ** 0.01; * 0.05).

Table 3—Means and Confidence Limits (95%) for the Dominance Indices (δ_1 and δ_2), Mean Monthly Biomass (as $\log_{10}$ N cells/½ disc), and Comparison of Means (t test) to Control and Cu_2O for Experiments in Stage 3

Paint	δ_1	t vs Al	t vs Cu	δ_2	t vs Al	t vs Cu	Log N	t vs Al	t vs Cu
Al	22.7 ± 10.0	/	***	34.3 ± 18.3	/	***	6.288 ± 0.315	/	*
Cu_2O	94.0 ± 23.7	***	/	99.7 ± 1.4	***	/	7.599 ± 1.699	*	/
SPC	78.3 ± 12.3	***	—	99.0 ± 2.5	***	—	6.973 ± 1.681	—	—
LB3	59.7 ± 2.9	***	**	89.0 ± 18.7	***	+	6.714 ± 0.588	+	—
LB6	58.3 ± 45.3	*	*	83.3 ± 8.8	***	**	6.558 ± 0.428	+	+
TBTF	36.7 ± 49.7	—	**	53.7 ± 68.7	—	*	5.918 ± 2.520	—	+
AW	31.7 ± 10.0	+	***	54.0 ± 12.4	*	***	5.660 ± 0.363	**	**

t test significance: *** 0.001; ** 0.01; * 0.05; + 0.1.

one species (*N. ovalis*) represents 94% of the total population, to the almost ineffective TBTF and AW, for which almost 50% of the diatom population is made up by a large number of species, only slightly smaller than that of the controls (*Table* 3).

Given these results, we also compared all paints to the one which seemed the most effective in reducing the number of species settled. Cuprous oxide paint was chosen, being the one for which the population could be considered practically monospecific. The results of this comparison are also given in *Table* 3, and show that SPC may be considered as effective as Cu_2O paint. Of the rest, only LB3 seemed to possess, albeit to a lesser degree, comparable antifouling properties. However, the lower significance of the tests and the higher number of colonizing species, some of which pertain to our "Non Tolerant" group, would seem to make this a poor candidate for the replacement of the more effective SPC and Cu_2O paints.

In *Table* 3 we also provide an indication of the mean monthly biomass, expressed this time as $\log_{10}$ of the number of cells per half disc for each of the paints tested, and compare these to the biomass found on the Al vinyl and Cu_2O paints. Most of the data are only different at very low probability levels, and in fact considerable overlap exists when confidence limits are considered. However, it seems clear that the most effective paints (SPC and Cu_2O) are those with the higher mean cell densities, while the control and least effective paints are those characterized by the lower cell densities.

An alternate and qualitative approach to establishing the effectiveness of the paints is a visual examination (light or electron microscope) of the slime film. *Figures* 2 and 4 show SEM micrographs of the slime films which have developed after three months on the six paints and on the control. Even without any knowledge of diatom taxonomy, it is clear that the diatom diversity is high on the control, on AW and on TBTF, whereas SPC, Cu_2O, LB3 and LB6 have only a few species present.

DISCUSSION

From the results obtained during this research it is clear that a mere measurement of biomass cannot be taken as a good indication of the antifouling properties of a paint, at

Table 4—Summary of S.E.M. Observations (Stage 3)[a]

	Al	TBTF	AW	LB6	LB3	Cu_2O	SPC
Month 1							
# Diatom sp.	14(1MT,13NT)	8(2MT,2T,4NT)	4(2MT,1T,1NT)	4(1MT,2T,1NT)	4(3MT,1NT)	(1)[b]	3(2MT,1T)
Dominant sp.	None	None	None	Sc(T)	No(MT)	None	Ac(MT)
Month 2							
# Diatom sp.	10(2T,8NT)	8(2MT,2T,4NT)	12(2MT,1T,9NT)	7(2MT,2T,3NT)	6(2MT,2T,2NT)	3(3MT)	3(2MT,1T)
Dominant sp.	None	Sc(T)	Ac(MT)	Ac(MT)	Ap(MT)	No(MT)	Ac(MT) Ap(MT)
Month 3							
# Diatom sp.	18(2MT,2T,14NT)	18(2MT,2T,14NT)	14(1MT,2T,11NT)	5(2MT,3T)	4(2MT,2T)	3(2MT,1T)	2(2MT)
Dominant sp.	None	None	None	Si(T)	Ac(MT)	No(MT)	Ac(MT) Ap(MT)
Month 4							
# Diatom sp.	17(1MT,1T,15NT)	15(2MT,3T,10NT)	12(2MT,2T,8NT)	3(1MT,2T)	6(2MT,3T,1NT)	3(2MT,1T)	3(2MT,1T)
Dominant sp.	None	None	None	Si(T) Ac(MT)	None	No(MT) Ac(MT)	Ac(MT) Ap(MT)

(a) MT, most tolerant; T, tolerant; NT, not tolerant. Ac, *Amphora coffeaeformis*; Ap, *Amphiprora paludosa*; No, *Nitzschia ovalis*; Sc, *Stauroneis constricta*; Si, *Synedra investiens*.
(b) Single individuals of several species: much debris on sample and most cells associated with this.

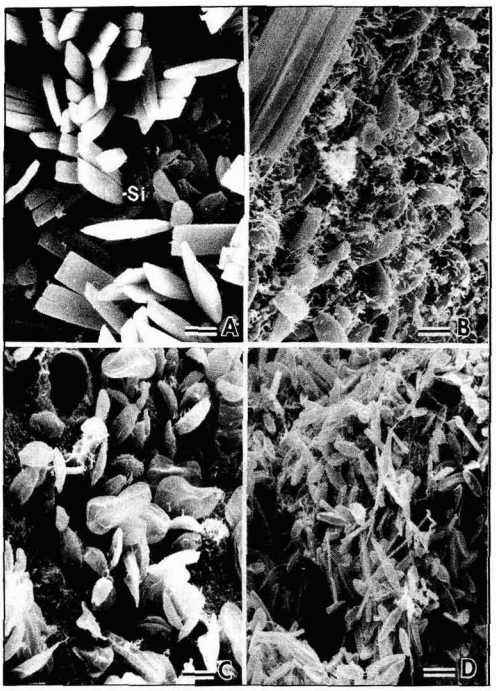

Figure 4—Fouling after three months (cont.). (A) Lab Black #6 antifouling paint (LB6). Si, *Synedra investiens*, a tolerant diatom species. (B) Lab Black #3 antifouling paint (LB3); (C) Self-polishing copolymer antifouling paint (SPC); (D) Cuprous Oxide vinyl antifouling paint (Cu_2O). Notice the reduced number of diatom species compared to the control (2B). Bar, 10 μm

least when considering microfoulers. While measurement of biomass in terms of cell numbers, rather than cell volumes or cell carbon, etc., may have some limitations, it should be kept in mind that the most likely result of any effective paint is that of eliminating a large number of sensitive species, thus favoring the growth of the few tolerant species which, through lack of competition, can utilize undisturbed all the space available. Cell dimensions, type of growth, amount of extra-cellular products, and various other biological characteristics of the species selected will determine the biomass present on the treated surface.

It is therefore the quality (i.e., species composition), not the quantity, of the microflora which can be more successfully used to indicate the potential effectiveness of a paint. Especially important is the study of species succession in these communities. Many paints may be extremely effective in the short term, but their toxicity may be later reduced, which will be inevitably indicated by the presence of a higher number of microalgal species (*Table* 4). Thus, if after three to four months the diatom population of a painted surface consists of very few species, and especially if these are among those recognized as tolerant to the toxic component(s), then this paint will warrant further investigation. Those paints recognized as being insufficiently selective (i.e., allowing a high species diversity) may be discarded.

It has recently been established that 10–12 replicate panels are required to characterize the time of failure at the 95% confidence level for any given coating.[19] This number of panels is required since the distribution of barnacle larvae is inhomogeneous and the statistical probability of a barnacle larva settling on a given plate has to be taken into account. Further, barnacle spawning and settlement are restricted to a specific two to three month period in any year, and for some locations in a given year may, on occasion, not occur. Such considerations are not relevant for our purposes. Diatoms have a much more normal distribution, are found year round, and reproduce (divide) on average every one to two days, building up a significant population in a short time. One or two small discs, such as we have used, are sufficient to obtain a population index over any three to four month period during the year, resulting in a significant reduction in the testing time and processing costs.

An interesting by-product of this research is the apparently contradictory sets of results concerning the effectiveness of TBTF. This observation is consistent with the information we received from members of the local boating community. While some describe it as a good antifouling agent, others maintain that such paint is completely ineffective. Such disparity of opinions is only apparent: used on fiberglass or plastic the paint gives, as we described, results comparable to those of the best antifouling paints we tested, while if applied on metal hulls with an anti-corrosive aluminum vinyl undercoat it quickly loses its toxic properties and allows macrofoulers to settle.

SUMMARY

Effective antifouling paints are those which prevent or at least significantly delay the attachment of macrofouling organisms to underwater marine surfaces. These paints also modify the slime film which always forms on any underwater surface.

Slime film formation on surfaces painted with various antifouling paints was studied to see if there was a relationship between either its biomass or species composition and any subsequent development of macrofouling organisms. As a result of these investigations, we propose a method which may be used to rapidly evaluate the effectiveness of antifouling paints.

Slime films are allowed to form under natural conditions on painted surfaces exposed for three to four months and are then examined by optical or scanning electron microscopy. If the number of species settled is low, and these include those species known to be toxin-tolerant, then the paint should resist the attachment of macrofoulers and is worthy of further testing. On the other hand, if species diversity is high the paint is likely to fail.

Measurement of the biomass of the slime film does not give a good indication of the effectiveness of the paint.

One paint we tested exhibited very different antifouling properties which depended on the substrate and/or

undercoat used. We suggest that new paints be tested over the same undercoat as in the potential working situation.

ACKNOWLEDGMENT

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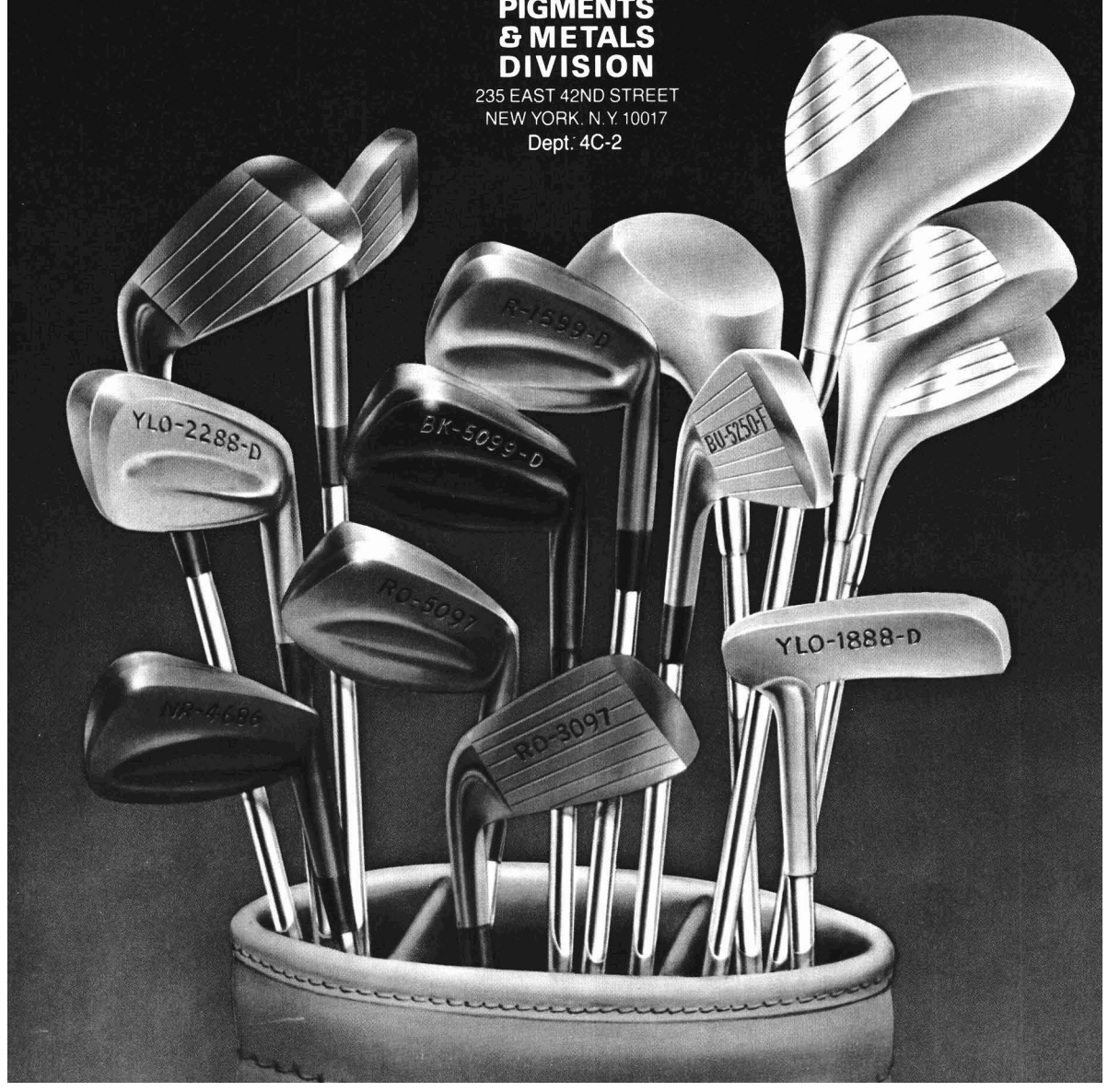

Kinetics of Crosslinking Using Blocked Reagents and Blocked Catalysts

W.J. Muizebelt
Akzo Research*

The kinetics of curing under the influence of blocked reagents and blocked catalysts are considered in this paper.

Blocked reagents dissociate thermally yielding a species which participates in a crosslinking reaction. Either the deblocking reaction or the crosslinking reaction may be rate-determining.

Experiments with Desmodur N (a trifunctional isocyanate) blocked with benzyl methacrylo hydroxamate (BMH) indicate that deblocking and crosslinking occur at comparable rates.

A theoretical model was developed described the action of blocked catalysts in a crosslinking reaction. Results of experiments using oxime tosylates for the curing of high solid polyester/melamine coatings are generally in line with this model. The crosslinking shows an induction period and S-shaped curves are observed.

INTRODUCTION

Blocked reagents and blocked catalysts offer a number of advantages when applied in coating formulations. Free isocyanates are too reactive to achieve a good package stability in polyurethane systems and they are sensitive to moisture. By making use of blocked isocyanates,[1,2] these problems can be overcome and also the toxic hazard can be reduced.

Free acid or amine-blocked catalysts generally lead to too high a conductivity for electrostatic spraying and give problems with respect to adsorption to pigment. Covalently blocked acids offer a solution in this case.[3]

Both blocked reagents and blocked catalysts are designed to give a satisfactory pot-life of one-package coating formulations, while simultaneously maintaining a good curing speed.

In a paper on the action of latent reagents, catalysts and initiators,[4] Pappas and Hill state that, in order to maximize pot-life and minimize curing temperature the deblocking reaction should exhibit a large activation energy. Since this leads to a slow rate, compensation is required by a high activation entropy. The high activation energy of the deblocking reaction will cause maximal difference in reaction rate at curing, respective storage temperatures if the deblocking reaction determines the overall rate, i.e., if it is the rate-determining step. This is only possible in the case of blocked reagents and not in the case of blocked catalysts or initiators.[5]

It is the purpose of this paper to describe both blocked reagents and blocked catalysts in curing reactions, showing the difference in approach required for the two systems. The factors involved in the kinetics of the crosslinking reaction which determines the curing speed and pot-life at various temperatures will be discussed.

REACTION SCHEMES

The crosslinking reactions under the influence of blocked reagents (BR) and blocked catalysts (BC) are schematically presented in *Figure* 1. The dissociation reactions of BR or BC are in principle of the same type (deblocking, reaction 1). The reagent (R) then reacts with some low molecular weight resin or monomer (M) to give a polymer or product (P). The catalyst (C) also leads to conversion of M into P but it does not participate in the reaction like R.

For the sake of clarity the crosslinking reactions are represented as a single elementary step 2. Although we

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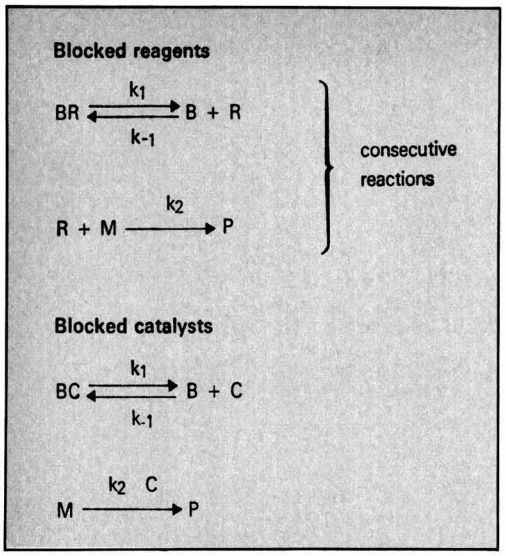


Figure 1—Reaction schemes

realize that this overlooks the complexity of the crosslinking reactions, it is necessary to demonstrate the difference in action between blocked reagents and blocked catalysts. Besides, it will be shown that experimental observations can be interpreted on the basis of the schemes presented in *Figure* 1.

Reduced to the simple schemes in *Figure* 1 it can be seen that the system of blocked reagents forms a set of consecutive reactions,[6] which is not the case in the system of blocked catalysts. The main reason for this difference is the fact that the catalyst (C) is not consumed in step 2.

In a system of consecutive reactions one of the steps may be rate-determining. If step 2 is fast as compared with both step 1 and the reverse step −1, R will react away as soon as it has formed and it will not attain a significant concentration. The rate of formation of P will then be equal to the deblocking rate of BR (see *Figure* 2). Similarly, if step 2 is slow and preceded by a fast equilibrium 1, the crosslinking is rate-determining.

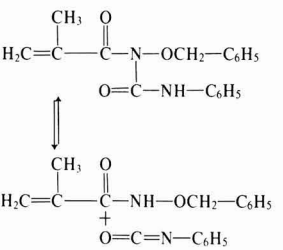
A different approach is required in the case of blocked catalysts. Here the deblocking reaction cannot be the rate-determining step in the crosslinking, which may be illustrated as follows: in the beginning the rate of deblocking is relatively fast, but because little catalyst has been formed as yet, the rate of crosslinking is still slow. As the deblocking reaction proceeds, its rate will drop because starting material concentration diminishes. However, the rate of crosslinking increases since there is now more catalyst. There is no simple relation between the rates of deblocking and crosslinking. An induction period and an S-shaped curve are the result.

This system will be dealt with later.

BLOCKED REAGENTS

Deblocking BMH/Desmodur N

Kinetic studies were reported[7] on the system phenylisocyanate blocked with benzyl methacrylo hydroxamate (BMH):



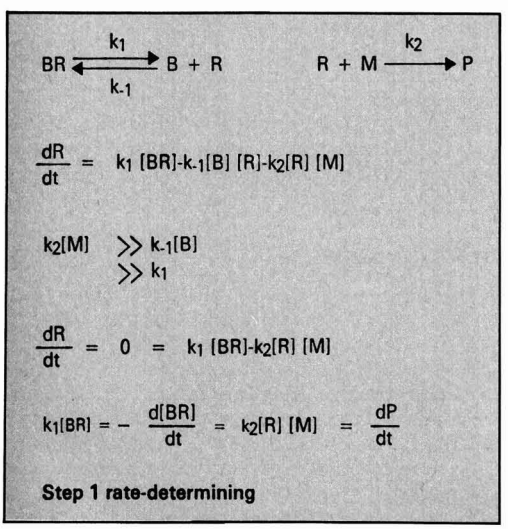


Figure 2—Blocked reagents

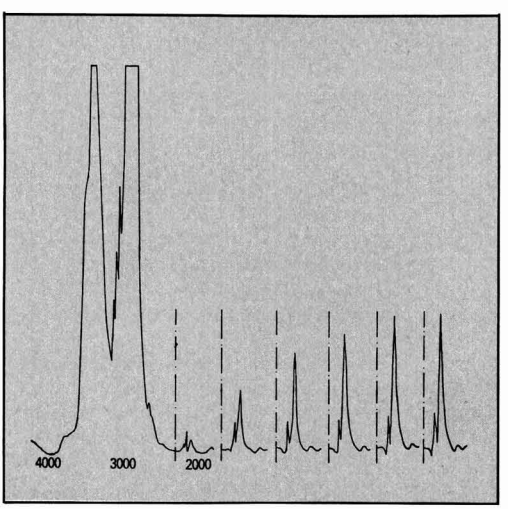
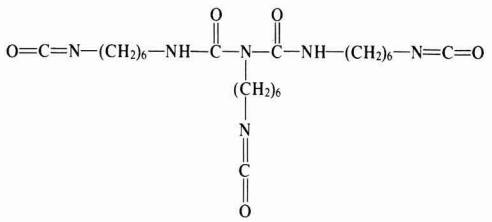


Figure 3—Dissociation of blocked isocyanate followed by infrared

However, the actual isocyanate used is Desmodur N consisting largely of a trifunctional derivative:



We have therefore performed a study on the deblocking of BMH-blocked Desmodur N using infrared spectrometry as the analytical tool. For this purpose a thin film of BMH/Desmodur N* was formed between two KBr discs, which was then placed in a heated cell in the infrared spectrometer (Perkin Elmer 983).

After three minutes the sample had reached temperature equilibrium and spectra were recorded in the range 4000-2000 cm^{-1} at regular time intervals. The isocyanate peak at 2260 cm^{-1} increased (*Figure* 3) until the equilibrium value. These observations were made at a number of temperatures in the range 85.5°–191°C. From the data the dissociation rate constant k_1 as well as that of the reverse reaction (k_{-1}) could be calculated.

Above 104°C equilibrium was reached too rapidly to permit the determination of rate constants and only equilibrium constants K_1 could be determined (*Table* 1).†

The activation energy for the deblocking reaction calculated from k_1 at 85.5° and 104°C is 21.5 kcal/mole. ΔH and ΔS values for the equilibrium constant K_1 were determined from the slope and the intercept of a plot of log K_1 vs. 1000/T, respectively (*Figure* 4).

Table 1—Kinetics and Equilibrium BMH + Desmodur N

$$BI \underset{k_{-1}}{\overset{k_1}{\rightleftharpoons}} B + I$$

T (°C)	K_1 (mole/l)	$k_1 \times 10^5$ (s^{-1})	$k_{-1} \times 10^5$ ($l\ mole^{-1}\ s^{-1}$)
85.5	0.078	6.8	88
104	0.188	30	160
125	0.526		
156	1.90		
175	3.68		
191	6.32		

ΔH = 13.9 kcal/mole
ΔS = 33 e.u.
Ea_1 = 21.5 kcal/mole

Crosslinking Reaction

From the data in *Table* 1 one may determine the time required for the deblocking reaction to reach equilibrium at the curing temperature of 130°C. By extrapolating the results of the measurements at 85.5° and 104°C, it is estimated that this time will be about 4-5 minutes.

This is in agreement with observations made on the curing of an OH-functional coating containing Desmodur N blocked with, among others, BMH. The concentration of the intermediate isocyanate was followed as a function of time by means of Fourier Transform Infrared Spectrometry. The temperature was raised to 130°C in about eight minutes. It is seen in *Figure* 5 that the concentration of free isocyanate reaches a maximum in about five minutes, followed by a rather rapid decrease. Most of the isocyanate is consumed in about 10 minutes. This implies that crosslinking occurs at a rate comparable with the deblocking reaction and that neither of these steps is rate-determining.

BLOCKED CATALYSTS

Deblocking Oxime Tosylates

REACTION KINETICS: A recent paper describes crosslinking of high solid coatings under the influence of oxime tosylates** as blocked catalysts.[3]

Here, we will report additional details of the deblocking reaction, insofar as required for a quantitative consideration of the kinetic aspects of the crosslinking reaction.

It was reported that the deblocking is a first order reaction resulting in a Beckmann fragmentation or

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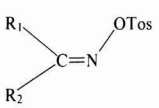

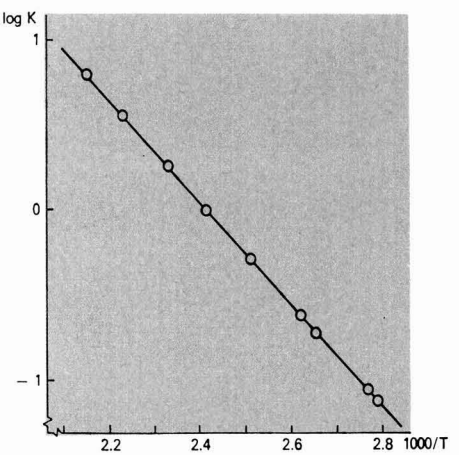

Figure 4—Determination of ΔH and ΔS

* The actual equivalent weight of Desmodur N was determined by titration as 216 g/eq. NCO. An excess BMH was used. Concentrations: [BMH] = 2.21 mole/L; Desmodur N[NCO] = 1.99 mole/L.

† The rate constant k_1 (in particular) and k_{-1} for the dissociation of BMH/Desmodur N are considerably smaller than those reported for BMH/phenylisocyanate[7] at 80° and 90°C. Also the equilibrium constant K_1 is smaller in the case of Desmodur N. A likely explanation, especially for the difference in equilibrium constants, may be found in the fact that phenylisocyanate will be stabilized by resonance due to conjugation of the π electrons of the isocyanate group with those of the benzene ring. This will lead to a larger dissociation of blocked phenylisocyanate than in the case of an aliphatic isocyanate such as Desmodur N.

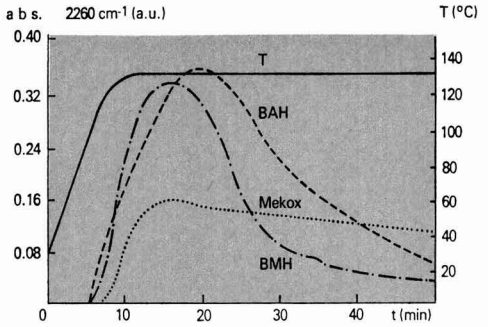

Figure 5—Free isocyanate concentration as a function of time for Desmodur N blocked with three different groups reacting in a coatings system as followed by Fourier Transform Infrared

rearrangement, dependent on the nature of the groups R_1 and R_2. Decomposition rate constants were determined in mixtures of dioxane and neopentyl alcohol. Measurements at various temperatures resulted in an Arrhenius plot from which activation parameters were calculated and half life of the blocked acid as a function of temperature was indicated.[3]

The reaction rate is only slightly dependent on the alcohol concentration. The deblocking rate constant k_1 may be thought to receive contributions from a component that is zero order in NPA and another which is first order in NPA:

$$k_1 = k_1^0 + k_1^1 [NPA]$$

The magnitudes of k_1^0 and k_1^1 were determined from the intercept resp. the slope of a plot of k_1 vs [NPA]:

$$k_1^0 = 39 \times 10^{-5} s^{-1}$$

$$k_1^1 = 16 \times 10^{-5} l.mole^{-1} s^{-1}$$

Alternatively, one might interpret the deblocking as a first order reaction only (zero order in NPA), whereby the slight effect of NPA is considered as a solvent effect.

DEBLOCKING IN A COATING: The decomposition rate of the blocked acid in the actual coating may be somewhat different from the rates determined in the model solvent NPA/dioxane because of the different environment (OH-functionality).

One way to follow the deblocking reaction in the coating is by monitoring the conductivity, which will change due to the liberation of acid. This was done at 35° C and 50° C. The experimental points in *Figure* 6 show the conductivity of a coating containing benzil oxime tosylate (BOT), plotted as a first order process. The infinite reading is the conductivity of a coating containing an equivalent amount of free p-TSA. The dashed line indicates the change, which may be expected on the basis of the decomposition rate in neopentyl alcohol/dioxane obtained by extrapolation of the Arrhenius plot for BOT to lower temperatures (see Figure 1 in reference 3).

Three conclusions are apparent from *Figure* 6: (1) the decomposition rate of BOT measured in NPA/dioxane is meaningful for the rate in the actual coating; (2) the Arrhenius plot may be extrapolated to lower temperatures; and (3) half lives of blocked acids may be determined from this plot at curing as well as at storage temperatures.

A remaining question is to what extent half lives of blocked acids determine the curing times and the pot lives of the coatings.

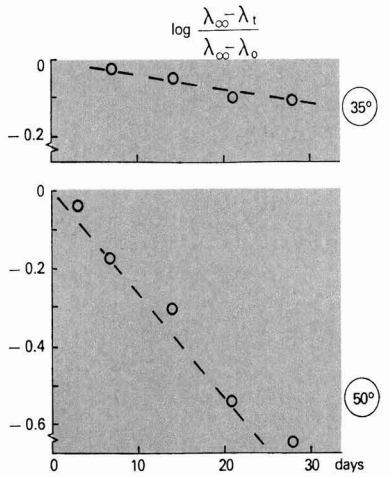

Figure 6—Decomposition of BOT in coating

Crosslinking

THEORY: The main crosslinking reaction taking place at curing temperatures is a transetherification reaction between the crosslinking reagent hexamethoxymethylmelamine (HMMM) and a diesterdiol, during which methanol is liberated[3] (*Figure* 7).

The kinetics of crosslinking under the influence of blocked catalysts are indicated in *Figure* 8. The blocked

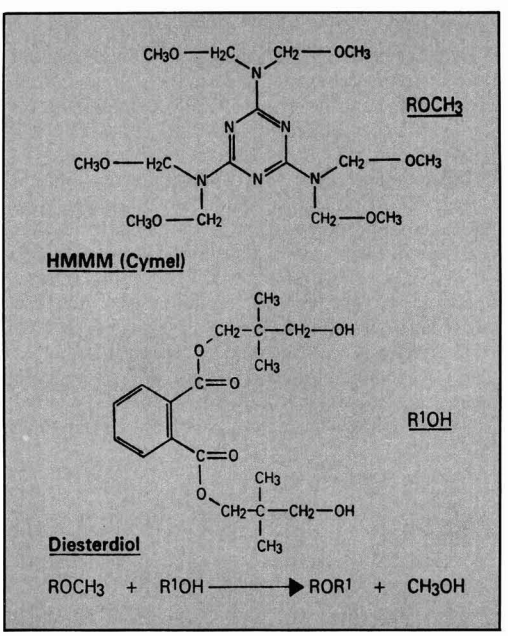

Figure 7—Crosslink reaction

catalyst decomposes according to a first order reaction. If the crosslinking is first order in M and catalyzed by C, the concentration of M decreases according to the formula on the bottom line of *Figure* 8.

The conversion into P as a function of time is depicted graphically in *Figure* 9. The crosslinking rate constant k_2 and $[BC_0]$ are arbitrarily set at 1. The decomposition rate constant is varied from 20 to 0.01. When deblocking is very fast (k_1=20), the situation is virtually identical to a case in which free catalyst is used. When the deblocking reaction gets slower there is an induction period during which a certain amount of catalyst is liberated. The longer the induction period the slower the crosslinking process proceeds. However, there is no proportionality. Differences in deblocking rates are only partially reflected in the times required for a certain conversion, even when the deblocking is slow as compared to the crosslinking.

Figure 10 shows similar plots for a second order crosslinking reaction. Since the rate now drops quadratically with the concentration of starting material, it takes much more time to achieve a complete conversion.

VISCOSITY INCREASE DURING STORAGE: *Figure* 11 shows the increase in viscosity (measured with a flow cup) of a coating composition in a closed vessel under the influence of p-TSA and BOT. Though the reaction occurring may be different from that shown in *Figure* 8 (no methanol escape), the patterns obtained are roughly those of *Figure* 9 or 10. With p-TSA, doubling of the viscosity takes place in about one hour. With BOT, an induction period occurs and doubling of viscosity now takes three hours. Half life of BOT is four hours at 90°C.

At 35°C, a similar behavior is found. Doubling of the viscosity takes four weeks (p-TSA) and 17 weeks (BOT). Half life of BOT is 10 weeks at 35°C.

At room temperature the viscosity does not increase very much in the presence of BOT. This behavior is unexpected since the half life of BOT at 22°C of somewhat over a year is only slightly longer than the duration of the experiment. Also conductivity measurements indicate that BOT decomposes, but apparently it does not lead to a significant viscosity increase.

$$BC \xrightarrow{k_1} B + C \qquad M \xrightarrow{C\,k_2} P$$

$$\frac{d[C]}{dt} = k_1[BC] = -\frac{d[BC]}{dt}$$

Integration yields:

$$[C_t] = [BC_0]\,(1-e^{-k_1 t})$$

$$-\frac{d[M]}{dt} = k_2\,[M]\,[C_t]$$

$$= k_2\,[M]\,[BC_0]\,(1-e^{-k_1 t})$$

Integration yields:

$$\ln\frac{[M]}{[M_0]} = \frac{k_2}{k_1}\,[BC_0]\,(1-e^{-k_1 t}-k_1 t)$$

Figure 8—Kinetics of crosslinking

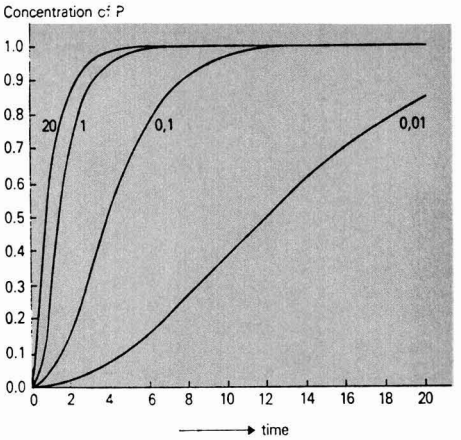

Figure 9—Concentrations of P as a function of time, k_2 = 1, k_1 = variable. First order model

CURING EXPERIMENTS: The reaction occurring in a thin film is different from that taking place in a closed vessel, because now methanol can escape. By following the methanol production it is possible to monitor the curing reaction. This was done by covering a small plate with some 50 mg (accurately weighed) coating formulation, yielding a layer of about 100 μm thickness. The plate was placed in an oven at 130°C and a moderate flow of helium passed over it.

Volatiles were condensed in a cold trap cooled with liquid nitrogen. The amount of methanol collected after some time was determined by gas chromatography, using ethanol as an internal standard.

The coating formulation consisted of pure HMMM and diesterdiol in a 1:2 molar ratio. The ratio $-OCH_3/-OH$

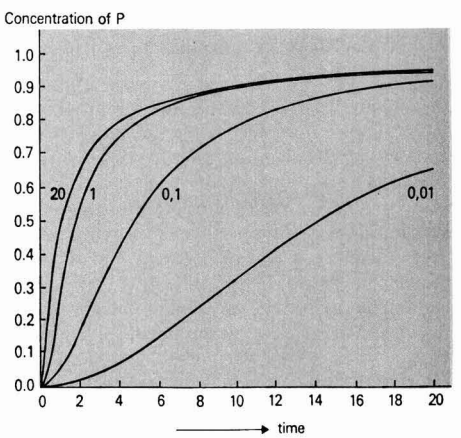

Figure 10—Concentration of P as a function of time. Second order model

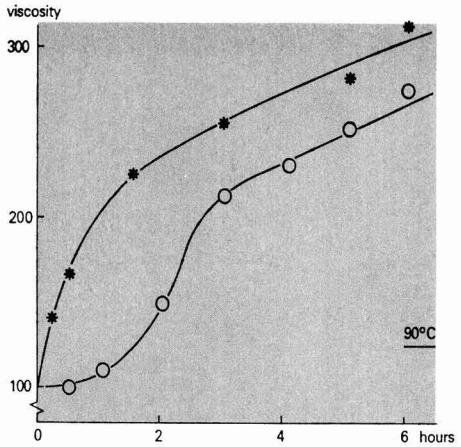

Figure 11—Viscosity vs time at 90°C

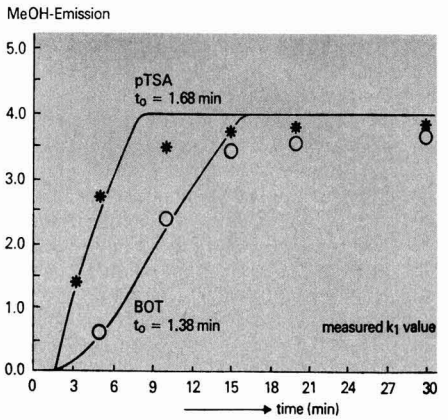

Figure 12—MeOH emission at 130°C

is therefore 6:4 and the maximum amount of methanol which can be produced is 4 moles/mole HMMM. Catalyst was provided in an amount of 2 moles% per mole diesterdiol.

Experimental results are given in *Figure* 12. The lines were calculated by means of a curve-fitting program with k_2 (the crosslinking rate constant) and t_0 (warming-up time for the film) as variables. The measured value of the deblocking rate constant k_1 was used. It was assumed that the crosslinking reaction is first order in HMMM and independent of ROH, as proposed by Bauer and Budde.[8]

It can be seen that the lines fit the experimental points well up to about 3 moles per mole HMMM. The t_0 values are in good agreement with the warming-up time, which was independently determined by means of a thermocouple. The maximum amount of 4 moles methanol is not reached even after 30 minutes of curing. This may point to a second order crosslinking reaction (compare *Figure* 10). However, this may as well be caused by diffusional limitations due to the increasing viscosity of the hardening film.

Deblocking Equilibrium

The catalyst p-TSA is formed in the decomposition of oxime tosylates in an irreversible reaction. However, the use of blocked catalysts need not be limited to systems in which the catalyst is liberated in a relatively slow irreversible reaction.

Another possible reaction mechanism may involve deblocking of the catalyst in a dynamic equilibrium with an equilibrium constant K_1. Under the conditions mentioned in *Figure* 13 (e.g., $K_1 \ll 1$), the apparent activation energy for the crosslinking reaction is now increased by half the dissociation enthalpy of the blocked catalyst.

In *Figure* 14 the theoretical effect of the relevant system, based on computer calculations, is indicated. Temperature dependence of the overall reaction is given in the form of Arrhenius plots assuming $E_{a2} = 15$ kcal/mole and $\Delta H_1 = 25$ kcal/mole. Eight different values of K_1 (each differing by a factor 10) have been assumed. For small values of K_1 the slope of the Arrhenius plot yields an activation energy of 27.5 kcal/mole. For larger values of K_1 the slope diminishes and approaches the same value as in the case of a free catalyst (e.g., 15 kcal/mole in the example given).

The mode of action described above might be encountered in the case of blocked amines,[9] e.g., urea derivatives dissociating into free amine catalyst and isocyanate:

$$R{-}NH{-}\underset{\underset{O}{\|}}{C}{-}NR_2 \rightleftharpoons R{-}N{=}C{=}O + HNR_2$$

$$BC \underset{k_{-1}}{\overset{k_1}{\rightleftharpoons}} B\ (inert) + C$$

$$M \xrightarrow{C\ k_2} P$$

$$K_1 = \frac{k_1}{k_{-1}} = \frac{[B]\,[C]}{[BC]}$$

$$\text{if } K_1 \ll 1 \rightarrow [BC] \approx [BC_0]$$

$$[B] = [C]$$

$$[C] = \sqrt{K_1[BC_0]}$$

$$\frac{d[P]}{dt} = k_2\,[C]\,[M] = k_2 K_1^{1/2}\,[BC_0]^{1/2}\,[M]$$

$$k_2 K_1^{1/2} = A_2\, e^{\frac{\Delta S_1}{2R}}\, e^{\frac{-(E_{a2} + 1/2 \Delta H_1)}{RT}}$$

Figure 13—Dissociation of blocked catalyst equilibrium

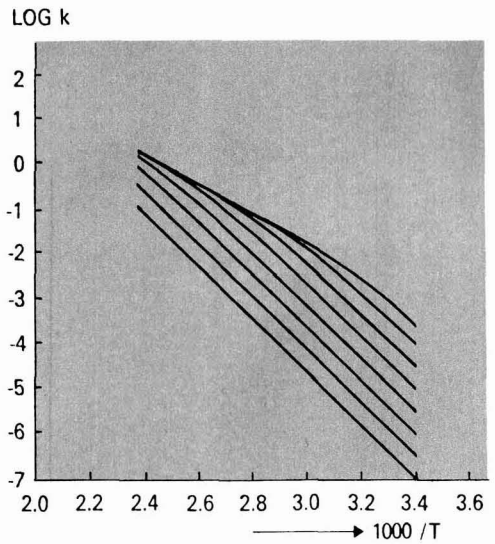

Figure 14—Arrhenius plots

These amines are used as a catalyst in the curing of powder coatings.

DISCUSSION

Apart from other effects, the action of blocked reagents and blocked catalysts in a crosslinking reaction has a number of kinetic implications with consequences for pot-life and curing speed of a coating.

Firstly, blocked reagents may increase the temperature dependence of the reaction so that longer pot-lives are possible without appreciable loss of curing speed. The effect is most notable if the deblocking has a high activation energy and is slow enough to be rate-determining. This is precisely the situation referred to by Pappas and Hill.[4] The effect is less pronounced if the deblocking reaction is relatively faster as has been observed in the case of BMH blocked Desmodur N. If the activation energy for deblocking exceeds that for crosslinking the deblocking may be rate-determining at low temperature and not at curing temperature, however.

The action of blocked catalysts is somewhat more complex. The occurrence of an induction period implies a slow crosslinking, particularly in the beginning of the reaction. This means that in general pot-life (low conversions) will be enhanced whereas curing time (high conversions) increases only slightly. The effect of a high activation energy for the deblocking reaction plays a secondary role here since the deblocking rate influences the crosslinking only partially. Nevertheless this effect contributes to the effect due to the induction period. The combined effect of induction period and high activation energy makes blocked catalysts an interesting system from a kinetic viewpoint, with useful practical implications with respect to pot-life and curing speed.

SUMMARY

The kinetics of crosslinking under the influence of blocked reagents and blocked catalysts are considered both theoretically and experimentally. Deblocking followed by crosslinking forms a set of consecutive reactions in the case of blocked reagents and either step may be rate-determining. Experiments with Desmodur N blocked with benzyl methacrylo hydroxamate demonstrate that deblocking and crosslinking occur with comparable rates, however.

Crosslinking under the influence of blocked catalysts generally leads to an induction period and S-shaped curves. Equations are derived to describe the kinetics. Differences in deblocking rate are only partially reflected in differences in crosslinking rate. This implies that half life of the blocked catalyst is only one of the factors determining pot-life and curing speed. Curing experiments with high solid polyester/melamine coatings containing benzil oxime tosylate as blocked catalyst are generally in line with the theory developed.

ACKNOWLEDGMENT

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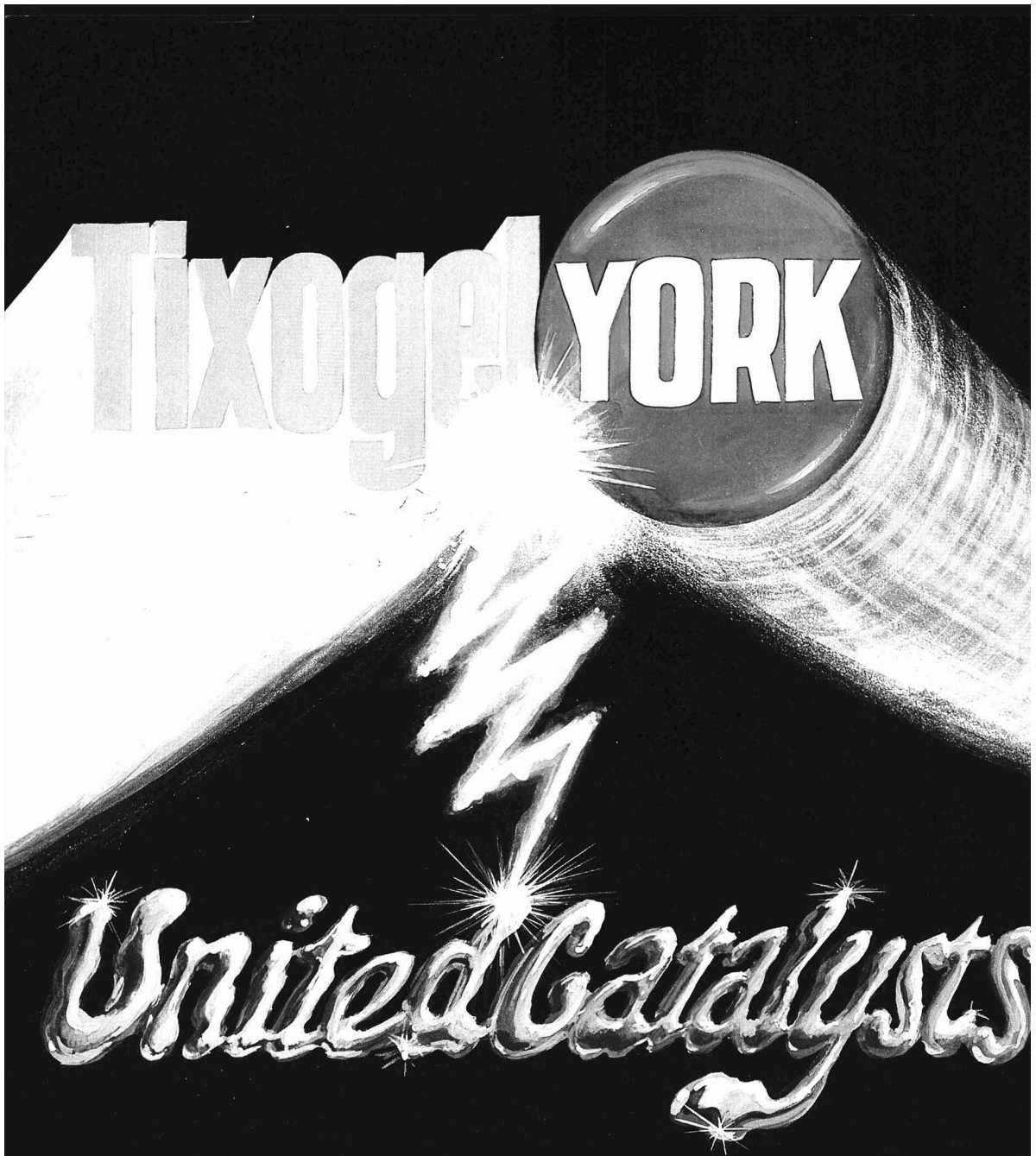

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Viscosity of Oligomer Solutions

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North Dakota State University*

Viscosity and glass transition temperature, T_g, of solutions of oligomeric butyl and methyl methacrylates (OBMA and OMMA) were determined. The dependence of T_g on concentration of solutions of OBMA in ethyl benzene and *m*-xylene over the entire concentration range was found to follow equation (10).

$$\frac{1}{T_g} = \frac{w_s}{T_{gs}} + \frac{w_o}{T_{go}} + K w_o w_s \qquad (10)$$

Temperature dependence of viscosity of OBMA in *m*-xylene was found to follow WLF (Williams, Landel, and Ferry) equation (1) over the whole range of concentration.

$$\ln \eta = \ln \eta_{T_r} - \frac{c_1 (T\text{-}T_r)}{c_2 + (T\text{-}T_r)} - \ln \frac{\rho_r T_r}{\rho T} \qquad (1)$$

Solutions other than pure solvent and those at low concentration followed simplified forms of the WLF equation such as (18) satisfactorily.

$$\frac{1}{34.5 - \ln \eta} = \frac{1}{A} + \frac{B}{A(T\text{-}T_g)} \qquad (18)$$

This equation provides a better model of viscosity as a function of temperature than an Arrhenius type expression, not only for these oligomer solutions, but, based on calculations using literature data, also for a wide range of other oligomers and oligomer solutions. T_g's and viscosities at equal concentration and temperature of OBMA and OMMA solutions in *m*-xylene increase as molecular weight increases and at comparable molecular weight are higher for OMMA than OBMA. Although T_g of OBMA solutions increased as T_g (or estimated T_g) of a series of solvents increased, viscosities of the solutions did not always show a corresponding increase. This, along with the system dependence of A and B in equation (18), indicates that while $(T\text{-}T_g)$ is an important factor controlling viscosity, it is not the only factor.

Addition of a power factor to the denominator of Erickson's equation, equation (19), leads to significantly improved predictions of relative viscosity as a function of concentration for both these oligomer solutions and Erickson's.

$$\ln \eta_r = w_o/(k_1 - k_2 w_o + k_3 w_o^2) \qquad (19)$$

Intrinsic viscosity is properly calculated by dividing the inverse of constant k_1 in equation (19) by solution density at $w_o = k_1$. Intrinsic viscosities (and ln η of solutions) of OBMA in *m*-xylene increase with approximately the half power of $\bar{M}_n$, possibly indicating theta conditions. However, intrinsic viscosities of both OBMA and OMMA solutions in *m*-xylene decrease with increasing temperature. Also, intrinsic viscosity of OBMA solutions is solvent dependent.

Viscosity of oligomer solutions in the range of interest for high solids coatings depends upon oligomer and solvent viscosity, oligomer and solvent T_g, solvent-oligomer interaction, temperature, and concentration. Further studies are needed to understand the relative importance of these factors and interactions between them.

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INTRODUCTION

Formulation of high solids coatings with sufficiently low viscosity for application requires the use of low molecular weight polymers, i.e., oligomers. While the factors controlling the viscosity of dilute polymer solutions have been widely studied, relatively little

research on the factors controlling the viscosity of oligomer solutions has been published. The available literature has been surveyed in a recent review paper.[1]

It was proposed that an important factor controlling viscosity of oligomer solutions is the availability of free volume. This suggested that the relationship between viscosity and temperature for oligomer solutions might be represented by a Williams, Landel and Ferry (WLF) type equation widely applied to viscosity-temperature relations of polymer melts.[2] One form of the WLF equation is given in equation (1).*

$$\ln \eta = \ln \eta_{T_r} - \frac{c_1(T-T_r)}{c_2 + (T-T_r)} - \ln \frac{\rho_r T_r}{\rho T} \qquad (1)$$

Hill, et al., tested this hypothesis by applying equation (2), a simplified form of equation (1), to viscosity, temperature data from several oligomer solutions.[3]

$$\ln \eta = 34.5 - \frac{40\,(T-T_g)}{51.6 + (T-T_g)} \qquad (2)$$

In equation (2), they used T_g as a reference temperature, η_{T_g} as 10^{15} mPa·s, with the corresponding "universal values" of constants c_1 and c_2 and neglected the density correction term. They calculated T_g of an oligomer solution by substituting viscosity data for the solution at a series of temperatures into equation (2) and found that the calculated T_g was constant in most of the examples tried. T_g's of the solutions were not experimentally determined. They found that equation (2) fit the viscosity-temperature data for different concentration solutions of a high solids polyester resin and three melamine-formaldehyde resins. Calculated T_g and viscosity decreased as solution concentration decreased in line with equation (2). On the other hand, equation (2) did not lead to constant calculated T_g's when applied to viscosity-temperature data for solutions of a high solids acrylic resin or a conventional acrylic resin. These resins were higher in molecular weight. It was suggested that the higher molecular weight might permit coil formation. Possibly, thermal expansion of the coil structure could partially offset the increase in free volume with temperature resulting in less decrease in viscosity.

Erickson studied the relationship between relative viscosity and concentration of several oligomers in several solvents.[4-6] He proposed equation (3) to express the dependence.

$$\ln \eta_r = \frac{w_o}{k_1 - k_2 w_o} \qquad (3)$$

The constant, k_1, was stated to be the inverse of weight intrinsic viscosity, $[\eta]_w$. He related the weight intrinsic viscosities of solutions of an oligomer in various solvents to an oligomer-solvent interaction variable, I_v.

$$I_v = V_s^{1/2}\,\Delta\delta \qquad (4)$$

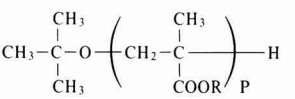
$$\Delta\delta = [4(\delta_{Do} - \delta_{Ds})^2 + (\delta_{Po} - \delta_{Ps})^2 + (\delta_{Ho} - \delta_{Hs})^2]^{1/2}$$

He concluded that weight intrinsic viscosity decreased as I_v increased until some limiting value, beyond which it increased. Presumably, when the solubility parameters of the solvent and oligomer are similar, the oligomer molecule is extended, leading to a relatively high weight intrinsic viscosity. As the difference in solubility parameter increases, decreased oligomer-solvent interaction leads to a less extended oligomer conformation and lower weight intrinsic viscosity. However, if the difference in solubility parameter becomes large enough, oligomer-oligomer interaction is favored and weight intrinsic viscosity increases.

On the other hand, Sherwin, et al., concluded from a study of high solids resins that, with the exception of carboxylic acid functional resins, solubility parameter of solvents had little effect on intrinsic viscosity.[7] They further concluded, again with the exception of carboxylic acid substituted resins, that relative viscosity of moderately concentrated resin solutions was little affected by solvent selection when concentrations were expressed in volume fractions. It is difficult to compare the work of Sherwin with that of Erickson because of units. The former calculated relative viscosity by dividing kinematic viscosity of a solution by the kinematic viscosity of the solvent rather than the usual calculation, used by Erickson, using absolute viscosities.

This paper reports the results of studies of factors controlling the viscosity of solutions of oligomeric butyl and methyl methacrylate (OBMA and OMMA). Since these oligomers have no polar functional groups, they are simpler systems than those used in coatings. They should, however, be more uniform and more easily characterized than the more complex resin systems. It was felt that such a study could provide the basis for broader future work with actual coatings resins.

OBMA and OMMA were synthesized by the anionic polymerization method of Lewis and Haggard.[8,9] So that the terminal group in all cases would be the same without having transesterification, potassium *t*-butoxide was used as the initiator and *t*-butanol was used as the molecular weight regulator (molecular weight increases as the mole ratio of monomer to alcohol increases). The general structures of the oligomers prepared were:



EXPERIMENTAL

Oligomer Preparation

The synthetic procedure was adapted from that of Lewis and Haggard.[8,9] All apparatus and materials were carefully dried and purged with oxygen-free N_2 to minimize presence of H_2O and O_2. A typical oligomerization was run as follows. A 2-L flask was charged with 4.36 g (0.0389 mol) potassium *t*-butoxide, 33.89 g (0.457 mol) *t*-butanol, and 55 mL of *m*-xylene under an O_2-free N_2 atmosphere. The alkoxide dissolved on warming to 60°C. Butyl methacrylate (BMA), 257.4 g (1.819 mol), was added dropwise over a period of 1.5 hr at a rate such that the temperature was maintained at 75 ± 5°C. The reaction mixture became

*For consistency, all equations are stated in natural logs rather than base 10 logs, all viscosities are in mPa·s, and all constants have positive signs. Equations from the literature are restated to follow these conventions.

cloudy and an orange color developed. Further *m*-xylene (100 mL) was added as needed to maintain low viscosity. On completion of BMA addition, the reaction mixture was held at 75°C for 30 min. Then a further 240.8 g (1.693 mol) of BMA were added over 1.5 hr at a rate to maintain temperature at 75 ± 5°C. During this second monomer addition, an additional 130 mL of *m*-xylene were added to maintain low viscosity. When monomer addition was complete, the temperature was held at 75°C for 45 min, then allowed to cool. The alkoxide was neutralized by addition of a slight excess of HCl, 3.4 mL (0.0408 mol) of concentrated HCl in 50 mL H_2O. The orange color disappeared. The reaction mixture was washed five times with one liter of distilled water. The organic layer, containing some emulsified water, was stripped of water and some *m*-xylene using a rotary evaporator with water aspirator vacuum while warming gently (T<60°C). The hazy solution was diluted with *m*-xylene, diatomaceous silica was added, and the solution was filtered through a glass frit filter. The clear filtrate was stripped of most of the solvent using a rotary evaporator and vacuum pump until the viscosity was such that the solution flowed only slowly. Molecular weight, by freezing point depression, was 1400. Acid content was shown to be negligible by titration, indicating no hydrolysis had occurred during oligomerization. Since no method for determination of weight average molecular weight, $\overline{M}_w$, is available, $\overline{M}_w/\overline{M}_n$ was estimated by GPC using a refractive index detector. In all cases estimated $\overline{M}_w/\overline{M}_n$ was 1.2 or less. Since the refractive index difference between oligomer and THF increases with $\overline{M}$, $\overline{M}_w/\overline{M}_n$ obtained in this manner is an upper limit. Haggard reports that such methacrylate oligomers are atactic.[9] This has been confirmed in our laboratories by C^{13} NMR of OMMA. In organolithium initiated polymerization of methacrylates at higher temperatures and especially in aromatic solvents, termination by backbiting to form a cyclic ketoester has been reported.[10] Volker, et al., report that no backbiting was observed during $KOCH_3/CH_3OH$ oligomerization of methyl methacrylate.[11] Lack of a signal in the range of 200 ppm in C^{13} NMR of our oligomers indicates the absence of a ketone carbon.

Non-Volatile Content

Various modifications of conventional methods for determining solids were found to be unsatisfactory. Either solvent loss was incomplete, leading to high solids values, or results were obviously too low, presumably due to either volatilization of low molecular weight fractions of the oligomer or possibly to depolymerization. Gel Permeation Chromatography (GPC) and Gas Chromatography (GC) using internal standards were evaluated, but inconsistent results were obtained. Thermogravimetric Analysis (TGA) was found to be satisfactory for very high solids solutions using a DuPont 951 Thermogravimetric Analyzer with a model 990 programmer starting with a concentrated oligomer solution. A 30 to 40 mg sample was used with a N_2 flow of 50 mL/min starting at 25°C and heating at 20°/min to 150°C followed by an isothermal hold. Both weight loss and first derivative of weight loss were monitored as a function of time. With high solids samples, no weight loss occurred in the initial 1 to 2 min, then solvent loss occurred. The first derivative increased with time to a maximum, then decreased to a low constant level while weight loss continued at a very slow rate. Solvent loss was taken to be the weight loss up until the time that the first derivative became constant. Precision was ±0.2% by weight of oligomer. For verification, several *m*-xylene solutions were also analyzed by UV. Virtually identical results were obtained.

Molecular Weights

Initially, GPC was used to determine molecular weight using the separate peaks of dimer through pentamer for standardization. However, refractive index and UV absorption change as a function of degree of polymerization in the oligomer range so that detector response is not constant as a function of molecular weight as it is with polymers. Hence, molecular weights cannot be accurately determined by GPC. Therefore, freezing point depression in benzene was used to determine number average molecular weight, $\overline{M}_n$. Apparent $\overline{M}_n$ was determined at a minimum of five low concentrations. Least squares analysis of a plot of ln $\overline{M}_n$ vs grams of solute per gram of solvent was used to calculate $\overline{M}_n$ extrapolated to zero concentration. No method is currently available for determination of weight average molecular weight, $\overline{M}_w$, of oligomers. GPC, with polystyrene standards, was used to estimate $\overline{M}_w/\overline{M}_n$, less than 1.2. Since refractive index difference between OBMA or OMMA and the carrier solvent, tetrahydrofuran, increases with increasing molecular weight, the $\overline{M}_w/\overline{M}_n$ obtained by GPC with refractive index detection is an upper limit.

Density

Densities were determined over a temperature range of 15–40°C using a Paar DMA-46 Digital Density Meter. Solution densities were determined as a function of concentration. Data were found to fit equation (5) with r^2 correlation factors of >0.999 with random scatter in residual plots [$\rho_{obs} - \rho_{pred}$) vs w_s].

$$\rho = \rho_s + K_1 w_s + K_2 w_s^2 \quad (5)$$

Density as a function of temperature could only be determined over a range of 15-40°C with the available density meter. Viscosities in some cases were measured as a function of temperature up to 80°C. The densities needed to calculate absolute viscosity were calculated using equation (6).

$$\rho = \rho_{T_r} - c_1(T\text{-}T_r) - c_2(T\text{-}T_r)^2 \quad (6)$$

High correlation factors (r^2>0.999) were obtained. The limited number of data points precluded the use of cubic term in the equation as is used over wider temperature ranges.[12] Results with and without a cubic term with International Critical Tables data for *m*-xylene showed that, over the range of 15-80°C, the

cube term is significant only in the fourth figure after the decimal point. Density data are given in the Appendix.

Viscosity Determination

In initial studies, a Ferranti-Shirley cone and plate viscometer was used to determine viscosity as a function of shear rate. Over a range of 1 to 1,000 sec^{-1}, viscosity was independent of shear rate. Since the solutions were Newtonian, viscosities were determined with Cannon-Fenske capillary viscometers standardized with Cannon Viscosity Standards. Temperature was controlled using a water bath controlled to ±0.03°C. Viscosities were converted into absolute viscosity by multiplying by density. Data are given in the Appendix.

For the studies of the effect of solvent structure on viscosity, the OBMA was prepared in benzene. As much benzene as possible was removed in a rotary evaporator at 80°C under high vacuum. The desired solvent was added, the system warmed at 80°C under vacuum. The process was repeated three times to assure that all benzene was removed and only the desired solvent remained.

Glass Transition Temperatures, T_g

T_g's were determined with a DuPont 910 Differential Scanning Calorimetry (DSC) Module (910) with a Model 990 Programmer. Sealed aluminum pans were used. Extrapolated onset of the transition was taken as T_g. Since initial runs at a range of heating rates showed that 10°/min gave results equal to lower rates, 10°/min was selected as the standard heating rate. Samples for determination of the T_g of solvent free oligomer, T_{go}, were prepared using a sample in a DSC pan heated until all solvent was lost in a TGA furnace as described in determination of non-volatile content. The sample was removed from the TGA and the DSC pan was sealed for use in determining T_g by DSC.

Most of the studies were done with *m*-xylene solutions, but some work was done with *o* and *p*-xylene and ethyl benzene. Only in the case of ethyl benzene was it possible to determine T_g's of solvent alone (T_g 120°K, 111-122°K lit.[13]) and solutions at all concentrations. In other cases, lower concentration solutions at least partially crystallized during the cooling below the expected T_g. When complete crystallization had occurred, no T_g type transition was observed, only a melting point transition. When partial crystallization had occurred, a T_g transition, at higher temperature an exotherm indicating further crystallization and, at a still higher temperature, the endotherm resulting from melting were observed. If there had been partial crystallization during cooling, the area under the exotherm of crystallization was less than the area above the endotherm of melting. In such cases, T_g data were discarded because they may represent the T_g of some indeterminate concentration solution after part of the solvent had crystallized out. In the case of moderate to high concentration solutions, no crystallization or melting was observed, only the T_g transition. In the case of *m*-xylene alone, partial crystallization occurred during cooling of the solvent, but the T_g (125°K) is still valid since no change in composition resulted from partial crystallization. The minimum concentration at which T_g could be determined increased with increasing molecular weight of the OBMA. T_g of OBMA-3600 *o*-xylene solutions could only be determined at concentrations of $w_o = 0.4$ or higher. The minimum concentration at which T_g of OMMA solutions in *m*-xylene could be determined was higher than for OBMA solutions of comparable molecular weight. Both *o*- and *p*-xylene crystallized completely during cooling and T_g of the solvents alone could not be determined. The minimum concentration at which T_g of OMMA solutions in *p*-xylene could be determined was 0.6. T_g values are given in the table in the Appendix.

Statistical Analyses of Data

The Statistical Analysis System (SAS) from the SAS Institute, Cary, NC, was used. The GLM regression analysis subroutine was used for linear models and initial estimates for non-linear analyses. The NLIN non-linear optimization subroutine was used for curved response models.

RESULTS AND DISCUSSION

Temperature Effects

DEPENDENCE OF T_g ON CONCENTRATION: It was found that equations (7)[14,15] and (8),[3,16,17] which have been used at high oligomer or polymer concentrations, did not fit the data. Equation (7) predicts a linear plot of T_g vs w_s. An actual plot showed substantial curvature.

$$T_g = T_{go} - kw_s \tag{7}$$

$$T_g = T_{go} + Cw_s + C'w_s^2 \tag{8}$$

Non-linear regression analysis of data with equation (8) gave reasonably high correlation factors, but there was a definite curved pattern in residual plots. Another T_g mixing equation, commonly used to predict T_g's of copolymers, is the Fox equation (9).[18]

$$\frac{1}{T_g} = \frac{w_1}{T_{g1}} + \frac{w_2}{T_{g2}} \tag{9}$$

Data did not fit equation (9); however, addition of an interaction term as shown in equation (10) represented the data well. Equation (10) can be rearranged to equation (11).

$$\frac{1}{T_g} = \frac{w_s}{T_{gs}} + \frac{w_o}{T_{go}} + k_3 w_o w_s \tag{10}$$

$$T_g = 1/(k_1 - k_2 w_o - k_3 w_o^2) \tag{11}$$

where $k_1 = 1/T_{gs}$, which is more easily analyzed. Equation (11) gave r^2 correlation factors of >0.999 for all data with standard errors of T_g less than ± 4°C. With all *m*-xylene solutions and the ethyl benzene solutions studied, equation (11) was applicable over the whole range of concentration including solvent free oligomer and oligomer free solvent. The T_g data for the solutions in *o*- and *p*-xylene over the limited concentra-

tion ranges available also fit equation (11) well, but the lowest concentrations at which T_g could be determined were $w_o = 0.4$ and 0.6, respectively. In these cases, T_g predictions should be made only within the range of data. Due to experimental error in T_g determination, predicted T_g's from equation (11) are used whenever T_g values are needed in later calculations. Both experimental and predicted T_g values are given in the Appendix.

TEMPERATURE DEPENDENCE OF VISCOSITY: It has been suggested that the temperature dependence of coatings resin solutions and oligomers follows an Arrhenius type relationship such as shown in equation (12).[7,19,20]

$$\ln \eta = \ln A + \frac{E_v}{RT} \tag{12}$$

While equation (12) gave r^2 correlation factors of 0.96 or higher for the temperature dependence of viscosity of *m*-xylene solutions of OBMA-2200, plots of residuals showed a definite curved pattern, indicating that it is not a satisfactory model.

Applicability of the simplified WLF equation used by Hill, et al.,[3] equation (2), was tested by calculating solution T_g for OBMA-2200 solutions in *m*-xylene as was done by Hill, et al. Constant T_g was not obtained. In polymer melts, it has been shown that the "universal constants" used in equation (2) are system dependent.[14] Therefore, equation (13) was tested treating the "constants" as adjustable parameters,

$$\ln\eta = 34.5 - \frac{A(T\text{-}T_g)}{B + (T\text{-}T_g)} \tag{13}$$

but keeping the other simplifications in equation (2). Equation (13) was shown by non-linear regression analysis to give high correlation coefficients ($r^2 > 0.99$) for viscosity dependence on temperature of *m*-xylene solutions of OBMA-2200 over a wide range of concentration ($w_o = 0.15$ to 0.8). Plots of residuals were random. However, it was found for low concentration solutions ($w_o < 0.15$) and for pure *m*-xylene, that, even though high correlation factors were obtained, plots of residuals vs temperature were curved. Also, over most of the range, both constants A and B increased with concentration. Based on the relationship proposed in the literature between constants A and B and free volume, one would expect that constant A would increase with concentration and constant B would decrease.[14]

The full form of the WLF equation, equation (1), using the lowest experimental temperature as the reference temperature and including the density term was evaluated by non-linear regression analysis. Very high correlation factors were obtained ($r^2 > 0.9999$), residual plots were random, and c_1 increased with concentration while c_2 decreased. The equation fit the data over the whole range of concentration from *m*-xylene alone to the highest concentration tested ($w_o = 0.803$). Dropping the density correction term reduced the correlation factor very slightly, but did not make a significant difference in the fit. These results indicate that the problem with the use of equation (13) may result from the assumption that the viscosity at T_g is 10^{15} mPa·s. Experimental determination of viscosity at T_g is difficult and inaccurate as is determination of density at T_g for very low T_g materials. Equation (1) can be restated as equation (14), using T_g as the reference temperature.

$$\ln \eta = \ln \frac{\eta_{T_g}}{\rho_g} + \frac{A(T\text{-}T_g)}{B + (T\text{-}T_g)} - \ln \frac{T_g}{\rho T} \tag{14}$$

Data on viscosity, temperature, and T_g of OBMA-2200 were used in nonlinear analysis to calculate A, B, and η_{T_g}/ρ_g. Fit was excellent over the whole range of concentration. Constants A and B changed oppositely in the directions expected with concentration. However, the calculated values for η_{T_g}/ρ_g were irrationally low for the lower concentration solutions ($w_o < 0.6$), η_{T_g} as low as 300 mPa·s for *m*-xylene alone, assuming $\rho_g = 1$. The calculations were based on viscosities determined over a temperature range of 228 - 353°K (15-80°C) while the lower concentration solutions had T_g values as low as 125°K. Perhaps with the errors involved in determining viscosity as a function of temperature, the very low values of η_{T_g}/ρ_g result from attempting to extrapolate to temperatures too far below the experimental range. It is also possible that non-linear analysis using three adjustable parameters with only nine data points gives erroneous results. Further data points over a wider temperature range will be required to differentiate between these possibilities and the possibility that equation (14) is not a satisfactory model over a wide temperature range.

From this work is appears that equations of the WLF form do give good predictions of viscosity, except at low concentrations, at least within the range of experimental temperatures. Therefore, $(T\text{-}T_g)$ is an important factor controlling viscosity. However, as evidenced by the lack of constancy of A and B, it is not the only factor. One cannot calculate viscosity from $(T\text{-}T_g)$ alone.

Another equation, most commonly called the Vogel equation, from which the various WLF equations can be derived, has also been used to describe temperature dependence of viscosity.[14,21]

$$\ln\eta = \ln C + D/(T\text{-}T_\infty) \tag{15}$$

As would be expected, equation (15) fits the data comparably to equation (13) without the assumption that $\eta_{T_g} = 10^{15}$ mPa·s. Equation (15) has what might be called an "advantage" in that η_{T_g} does not explicitly appear in the equation so it is not evident that there is a problem. Equation (14) seems preferable to us since at least T_g can be determined experimentally while T_∞ cannot.

A modification of the Vogel equation, shown in equation (16), has been said to give an improvement in fit of data.[14] It also seems attractive since it appears to combine the concept of an activation energy for flow with a free volume effect. From equation (16), equation (17) can be derived.

$$\ln \eta = \ln C + \frac{E_v}{RT} + \frac{D}{(T\text{-}T_\infty)} \tag{16}$$

$$\ln \eta = \frac{E_v}{R}\left(\frac{1}{T} - \frac{1}{T_g}\right) + \ln \eta_{T_g} - \frac{A(T\text{-}T_g)}{B + (T\text{-}T_g)} - \ln \frac{\rho_g T_g}{\rho T} \tag{17}$$

Table 1—Viscosity as a Function of Temperature Of Epon Resin 828.[a] T_g = 259.6°K

T (K)	η(mPa·s)
298	15,300
304	7,450
309	3,680
314	2,000
333	300
373	36
423	17.7

(a) Data are averages of readings of data points on a graph[27] by four people.

Equation (17) was tested with the data from *m*-xylene and several OBMA-2200 solutions in *m*-xylene over a wide range of concentrations. Since too few data points were available to permit calculation of four adjustable parameters by non-linear analyses, η_{T_g} was assumed to be 10^{15} mPa·s. While r^2 correlation factors were high (>0.9999), the values of E_v were widely varied including some with negative activation energies. It would be of interest to test this equation with much larger data sets over a wide range of concentrations, but this initial attempt did not look promising.

EMPIRICAL USE OF WLF EQUATION (13): As noted earlier, an Arrhenius relationship, equation (12), did not fit the data for OBMA solutions. Since equation (12) has been recommended for prediction of viscosities of coatings resin solutions as a function of temperature,[20] it was decided to test it with literature data. A linear form of equation (13), equation (18), was also evaluated.

$$\frac{1}{34.5 - \ln \eta} = \frac{1}{A} + \frac{B}{A(T\text{-}T_g)} \qquad (18)$$

With all data tested, equation (12) gave curved residual plots indicating that ln η is not linear with 1/T. The residuals may be small when narrow temperature ranges are used, but even then the response is not linear. On the other hand, it was found that equation (18) does give linear plots with high correlation (r^2>0.99) for all examples. Several sets of data for various molecular weight oligomeric styrene melts[15,21,22] were tested. High correlation factors were obtained for data on 1,3,5-tri-α-naphthylbenzene[23] and α-phenyl-o-cresol,[24] but patterns were evident in residual plots. It was found that some of the transition points in these patterns corresponded to temperatures at which changes had been made in the method of viscosity measurement. This suggests that the patterns may be artifacts of measurement technique rather than the failure of equation (18) to be a satisfactory model. (Utracki[25] proposed that the data for these low molecular weight compounds fit a step series of Arrhenius relationships, but apparently did not recognize that several of his "steps" occurred at temperatures where changes in method of viscosity measurement had been made.) Since viscosity-temperature data were available for the Cannon Viscosity Standards,[26] T_g's of these standards were determined. (Data are given in the Appendix.) The data for these mineral oils and polybutenes also fit equation (18). The data of Hill, et al.[3] were also tested with equation (18). Since experimental T_g data were not available, the average T_g which they had calculated for each of their solutions using equation (2) was used. In all cases, including the acrylic resins which did not give constant T_g with equation (2), equation (18) gave high correlation factors. While the values of A and B were calculated, they are not given since they probably have no physical significance when η_{T_g} is assumed to be 10^{15} mPa·s.

An example of the comparison of the use of equations (12) and (18) using data for Epon® Resin 828 given in *Table* 1 is shown in *Figure* 1. In order to compare the two equations on the same graph, values predicted by use of the two linear equations were used to plot ln η as a function of T and the experimental data points were plotted.

These data were selected as an example since viscosity values were available over a wide range of temperatures (25-150° C). The apparent activation energy for flow, E_v, in equation (12), is 14 kcal/mole, the correlation factor, r^2 = 0.94. A plot of residuals vs T is clearly curved. Most commonly Arrhenius type relationships have been claimed when viscosities have been determined over a limited temperature range. If only the five data points from 25 to 60°C in *Table* 1 were used, the E_v was lower (5.6 kcal/mole) and the correlation factor was very high (0.999). The r^2 correlation factor with equation (15) using data over the whole range of 25-150°C is 0.994. The values for constants A and B are 35 and 16, respectively, very different from the "universal values" of 40 and 51.6 in equation (2).

FACTORS AFFECTING T_g: Since T_g is an important factor affecting the viscosity of oligomer solutions at some temperature of interest, in designing high solids coatings systems factors affecting T_g of the solution in addition to concentration should be considered. *Table* 2 shows the T_g and viscosities of solutions of different molecular weight OBMA's and OMMA's at w_o = 0.6. In both cases, the higher molecular weight oligomer gives a solution with higher T_g and higher viscosity. The difference between the T_g's and viscosities of the OBMA and the OMMA solutions makes it evident that oligomer structure is also a significant factor in both T_g and viscosity.

Another potential factor affecting T_g of an oligomer solution is the effect of solvent T_g and solvent-oligomer interaction on T_g. In an attempt to test the effect of solvent T_g on solution T_g and viscosity, solutions of OBMA ($\bar{M}_n$ = 3,600) in *o*-, *m*-, and *p*-xylenes and ethyl benzene were studied. These solvents were selected since their solubility parameters are very similar.[28] It might be expected that solvent-oligomer interaction effects on T_g and viscosity would also be similar. On the other hand, the melting points of the four solvents are very different: ethyl benzene, 178° K; *m*-xylene, 225° K; *o*-xylene, 248° K; and *p*-xylene, 286° K.[29] Even though Angell, et al.[13] have pointed out that the approximation

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that $T_g = 2/3\ T_m$ is a poor approximation, it would be expected, based on melting points, that T_g of the solvents would increase in the order ethyl benzene, *m*-xylene, *o*-xylene to *p*-xylene. Unfortunately, it was found that only the T_g's of ethyl benzene (120° K) and *m*-xylene (125° K) could be experimentally determined by DSC. This is in line with the statement of Turnbull and Cohen that only liquids with $T_b/T_m > 2.0$ readily form glasses.[30] It was found that, using the fastest feasible means of cooling the DSC cell, *o*- and *p*-xylene crystallized before the temperature was below the possible T_g, making it impossible to determine T_g. In *Table* 2, the T_g's of the solutions of OBMA-3600 in the four solvents increase in the order anticipated. The viscosities of the solutions in ethyl benzene, *m*-xylene and *o*-xylene increase in the same order. However, the viscosity of the *p*-xylene solution is the lowest of the four in spite of the fact that its T_g is the highest. It would be of interest to determine viscosities of *p*-xylene solutions of OBMA as a function of temperature to see whether the values of c_1 and c_2 in the WLF equation are significantly different from those of *m*-xylene solutions at the same concentrations. In any case, this is another example of what has been said earlier, that solution T_g is a factor controlling oligomer solution viscosity, but not the only factor. Thus solvent T_g may be one factor affecting T_g and viscosity of oligomer solutions, but some other factor(s) is also involved.

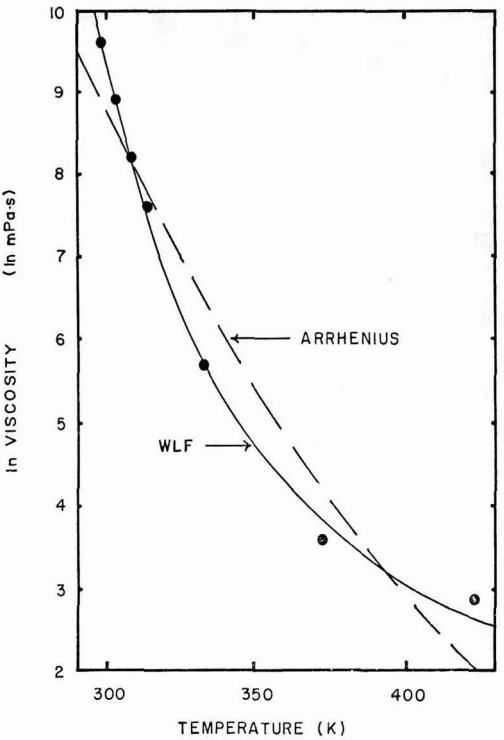

Figure 1—Predicted ln η of Epon 828 as a function of T by equations (12) and (18) showing experimental data points

Dependence of Relative Viscosity on Concentration

When viscosity data as a function of concentration were analyzed using Erickson's equation (3),[4-6] it was found that while correlation factors were relatively high ($r^2 > 0.96$), plots of residuals were clearly curved showing it was not a good model. A variety of other equations was tested. It was found that addition of a second order term to the denominator of Erickson's equation, as in equation (19), gave higher correlation factors ($r^2 > 0.999$) and much more nearly random scatter of the residual plots.

$$\ln \eta_r = w_o/(k_1 - k_2 w_o + k_3 w_o^2) \qquad (19)$$

Relatively consistently, equation (19) leads to predictions of relative viscosity at low concentrations ($w_o > 0.15$) which are slightly higher than experimental values. A possible explanation for this can be seen by considering the transformation of equation (19) to equation (20).

$$\ln \eta_r = \frac{w_o}{k_1 w_s + \dfrac{w_o}{\ln \eta_o - \ln \eta_s} - k_3 w_o w_s} \qquad (20)$$

It can be seen in equation (20) that the interaction term ($k_3 w_o w_s$) maximizes at $w_o = w_s = 0.5$. There is no evident reason why the interaction should maximize at this concentration. This suggests that the final term in the denominator of equation (19) should be some power other than 2 depending upon the concentration of largest interaction. This hypothesis was tested by allowing this exponent to be a fourth adjustable parameter in nonlinear regression analysis. The r^2 correlation factors were found to be even higher than when the exponent had a value of 2 and residuals were completely random, including values at low oligomer concentrations. However, even with 30 point data sets, the standard errors in k_3 and the exponent were so large that no significance can be placed on the values calculated. Further evaluation of this hypothesis will require more data points (preferably with smaller experimental errors).

In order to test the empirical utility of equation (19), it was utilized with Erickson's data.[5] Much higher correlation factors were obtained with equation (19), $r^2 > 0.9999$, than with his equation (3), $r^2 > 0.96$, and plots of residuals were much more nearly random. Thus, although equation (19) is not an exact model for viscosity dependence on concentration of oligomer

Table 2—Solution T_g and Viscosity (25°C) at $w_o = 0.6$

Oligomer	Solvent	T_g (K)	η(mPa·s)
OMMA-1000	*m*-xylene	187	76
OMMA-1600	*m*-xylene	192	1060
OBMA-1100	*m*-xylene	165	12.5
OBMA-3600	ethyl benzene	160	84
	m-xylene	169	103
	o-xylene	175	126
	p-xylene	184	82

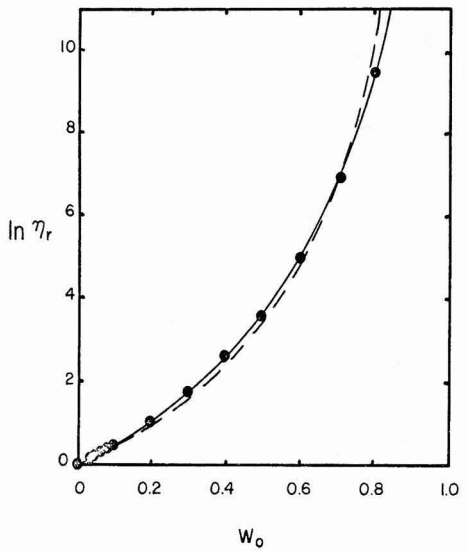

Figure 2—ln η_r vs w_o for OBMA-3600/*p*-xylene at 25°C from equation (3) and equation (19). [---Equation (3), —Equation (19), ln η_r (Observed)]

solutions, it is a better representation than equation (3). The applicability of the two equations is compared in *Figure* 2. The curves are based on values predicted by equations (3) and (19) for ln η_r at 25°C for *p*-xylene solutions of OBMA-3600 as a function of concentration.

Intrinsic Viscosity Effects

Erickson showed that k_1 in equation (3) is the inverse of weight intrinsic viscosity, $[\eta]_w$.[5] Constant k_1 in equation (19) is also the inverse of $[\eta]_w$. Weight intrinsic viscosity represents the grams of oligomer solution per gram of oligomer of isolated oligomer molecules at the limit of ln η_r/w_o as w_o approaches zero. (It is not unitless as stated by Erickson.[5]) It is more useful to compare intrinsic viscosities, as is done with polymer solutions, since they represent the hydrodynamic volume swept out by the oligomer and associated solvent molecules. The units of $[\eta]$ are mL of oligomer solution/gram of oligomer. Erickson[5] and Patton[20] state that weight intrinsic viscosity can be converted to intrinsic viscosity by dividing by solvent density. This does not seem appropriate. For example, if $k_1 = 0.25$, then $[\eta]_w = 4.0$ g/g. This means that the concentration of oligomer in the isolated oligomer molecule and associated solvent is 0.25, i.e., k_1. Therefore, $[\eta]$ should be calculated by dividing $[\eta]_w$ by the density at $w_o = k_1$. It should be noted that if concentrations in equations (3) and (19) were expressed in grams of oligomer/mL of solution, the corresponding k_1 would be the inverse of the intrinsic viscosity obtained by dividing weight intrinsic viscosity by solvent density rather than by the density of the isolated oligomer molecule and its associated solvent. In polymer solutions, the difference is small since the intrinsic viscosities are high (usually 40 mL/g or higher) so that the difference between solvent density and the density of the dilute solution at $w_o = 1/[\eta]_w$ is small. However, when intrinsic viscosities for oligomers are determined this way, as was done for example by Sherwin, et al.,[7] intrinsic viscosities should be corrected for this density effect.

Table 3 shows k_1, $[\eta]_w$ and $[\eta]$ for the oligomer solutions studied. The standard errors of k_1 are given. There is no statistically valid method for calculating standard error of the inverse of a function. Approximations of the error ranges of $[\eta]_w$ can be calculated by taking the inverses of the upper and lower limits of the standard error range of k_1. As would be expected, the $[\eta]$ of OMMA solutions is higher than that of OBMA solutions of approximately the same molecular weight. With both OBMA and OMMA, as would be expected, $[\eta]$ increases with molecular weight. The data for the five different molecular weight OBMA solutions in *m*-xylene at 25°C were used in the Mark-Houwink equation (21) to determine the values of constants *a* (0.49) and K (0.094).

$$[\eta] = K\,M^a \qquad (21)$$

The correlation factor r^2 was 0.883. With the limited number of data points and the possible error range of M, caution should be expressed about the reliability of the figures. It is of interest, however, to note that the value of *a* closely approaches 0.5, the usual value of *a* for polymers under theta conditions. This is in line with the general statement that oligomer solutions behave as if they are at theta conditions independent of solvent.[31] The value of K (0.094) corresponds to K_θ for PBMA in the literature of 0.038 mL/g.[31]

As can be seen in *Table* 3, $[\eta]$ of both OBMA and OMMA solutions in *m*-xylene decrease with increasing temperature. This is a surprising result since polymer systems (except those with very strong solvent-polymer interactions) generally show an increase in $[\eta]$ with an increase in temperature.[31] Data for PMMA in *m*-xylene from the literature,[32] also shown in *Table* 3, show an increase in $[\eta]$ with temperature. The $[\eta]$ of PBMA in *m*-xylene was determined and found to be independent of temperature, within experimental error, over the range of 20-40°C. No explanation for the decrease in $[\eta]$ with temperature has been devised. The dependence of $[\eta]$ on temperature indicates that 25°C in *m*-xylene are not theta conditions for OBMA-2200 even though *a* in equation (21) is approximately 0.5.

Further indications that theta conditions may not be met can be seen by the change in $[\eta]$ when *m*-xylene is compared with the other solvents at 25°C. Intrinsic viscosity decreases in the order *o*-xylene, *m*-xylene, ethyl benzene, *p*-xylene. Mangaraj, et al., showed that $[\eta]$ of polymer solutions in solvents of similar structure decreases as $V_s^{1/2}(\delta_p - \delta_s)$ increases.[33] Using total solubility parameters from Hoy[28] for the four solvents and calculating that of OBMA using his group constants (9.27), $V_s^{1/2}(\delta_p - \delta_s)$ values for the four systems were calculated: *o*-xylene, 2.31; *m*-xylene, 4.33; ethyl benzene, 4.77; *p*-xylene, 4.90. Thus, the order

corresponds to that reported by Mangaraj, et al., for polymer solutions. Again, caution should be exercised in interpreting the data. The differences in $[\eta]$ and in solubility parameter are relatively small. In order to compare with Erickson's proposed relationship between $[\eta]_w$ and his I_v,[5] calculation of three dimensional solubility parameters for the four solvents was attempted using Hoy's method.[28] Unfortunately, this could not be done for either ethyl benzene or *o*-xylene since one of the steps in the calculation resulted in needing to take the square root of a negative number. Erickson's data for his "acrylic I" in a variety of solvents[5] were reanalyzed using equation (19). Plots were made of both $[\eta]_w$ and $[\eta]$ against his I_v values. While the same trend that he reported was present, addition of error bars to the data points in both dimensions raises some doubt about the significance of the trend.

Viscosities of Moderately Concentrated Solutions

To compare the effect of various factors on relative viscosity and viscosity of moderately concentrated solutions, viscosities of solutions at $w_o = 0.6$ were calculated using equation (19). The results are also given in *Table* 3. The facts that viscosity decreases with temperature following a WLF type relationship and that OMMA solutions have higher viscosities than similar molecular weight OBMA solutions have already been discussed. Viscosity of OBMA solutions increases with molecular weight (with the exception of the small reversal between 2,100 and 2,200). Calculation showed that ln η depends upon the 0.52 power ($r^2 = 0.86$) of molecular weight. This value is not statistically significantly different than the dependence of $[\eta]$ on M.

It was pointed out earlier that the increasing order of viscosity at 25°C and T_g of solutions of OBMA is ethyl benzene, *m*-xylene, *o*-xylene, but that, while the *p*-xylene has the highest T_g at $w_o = 0.6$, its viscosity is the lowest of the four. Due to the differences in solvent viscosity, the order of relative viscosity at $w_o = 0.6$ is not the same as the order of viscosities. In decreasing order of relative viscosities, they are: *o*-xylene > *m*-xylene > *p*-xylene > ethyl benzene. Absolute viscosities, but not relative viscosities, decrease as $V_s^{1/2}$ $(\delta_o - \delta_s)$ increases.

Calculated Viscosities of Solvent Free Oligomers

By setting $w_o = 1$ in equation (19), viscosities of the solvent free oligomers can be calculated. Since this involves the reciprocal of the difference between small numbers, considerable error in the calculated values can be anticipated. Unfortunately, there is no statistical test

Table 3—Intrinsic Viscosities and Viscosities Of Oligomer Solutions at $w_o = 0.6$

Oligomer $\bar{M}_n$	Solvent	T (°C)	k_1	$[\eta]_w$	$[\eta]$	η_r $w_o = 0.6$	η(mPa·s) $w_o = 0.6$
OBMA-1100	*m*-xylene	25	0.375 ± 0.015	2.67	2.91	22	13
OBMA-1400	*m*-xylene	25	0.345 ± 0.025	2.90	3.19	42	24
OBMA-2100	*m*-xylene	25	0.247 ± 0.004	4.05	4.49	135	78
OBMA-2200	*m*-xylene	15	0.232 ± 0.007	4.31	4.76	179	116
OBMA-2200	*m*-xylene	20	0.238 ± 0.006	4.20	4.65	165	101
OBMA-2200	*m*-xylene	25	0.241 ± 0.006	4.15	4.62	131	75
OBMA-2200	*m*-xylene	30	0.246 ± 0.006	4.07	4.54	108	59
OBMA-2200	*m*-xylene	35	0.249 ± 0.006	4.02	4.49	96	49
OBMA-2200	*m*-xylene	40	0.253 ± 0.006	3.95	4.45	83	40
OBMA-2200	*m*-xylene	50	0.263 ± 0.009	3.80	4.31	66	29
OBMA-2200	*m*-xylene	60	0.271 ± 0.008	3.69	4.21	54	22
OBMA-2200	*m*-xylene	80	0.289 ± 0.008	3.46	4.00	38	13
OBMA-3600	*m*-xylene	25	0.226 ± 0.004	4.42	4.94	164	103
OBMA-3600	*o*-xylene	25	0.211 ± 0.005	4.73	5.22	169	126
OBMA-3600	*p*-xylene	25	0.236 ± 0.003	4.23	4.73	142	82
OBMA-3600	ethyl benzene	25	0.233 ± 0.004	4.29	4.78	140	84
OMMA-1000	*m*-xylene	15	0.331 ± 0.004	3.02	3.19	240	156
OMMA-1000	*m*-xylene	25	0.347 ± 0.005	2.88	3.05	132	76
OMMA-1000	*m*-xylene	40	0.366 ± 0.014	2.73	2.92	63	31
OMMA-1600	*m*-xylene	25	0.230 ± 0.003	4.27	4.64	1850	1060
PBMA-73500	*m*-xylene	20	—	—	55	—	—
PBMA-73500	*m*-xylene	25	—	—	54	—	—
PBMA-73500	*m*-xylene	40	—	—	56	—	—
PMMA-940000	*m*-xylene[a]	40	—	—	55	—	—
PMMA-940000	*m*-xylene[a]	50	—	—	62	—	—
PMMA-940000	*m*-xylene[a]	60	—	—	71	—	—
PMMA-940000	*m*-xylene[a]	70	—	—	80	—	—

(a) Data from reference 32

Table 4—T_g and ln η_o (25°C) of Solvent Free Oligomers (T_g calcd. with equation (11)) (ln η_o calcd. with equation (19))

Oligomer	T_{go} (K)	ln η_o (mPa·s)
OBMA-1100	228	10.32
OBMA-1300	235[a]	--
OBMA-1400	236	11.50
OBMA-2100	258[a]	14.60
OBMA-2200	257	14.83
OBMA-3600	263	16.99
OMMA-1000	270	28.02
OMMA-1600	—	43.10

(a) Experimental values.

that permits estimating a standard error. The results of such calculations are shown in *Table* 4 and 5. Also given are the T_g's of the solvent free oligomers.

As would be expected (see *Table* 4), viscosity of the solvent free oligomer at 25°C increases with molecular weight. It is generally found that below the critical molecular weight for entanglement, M_c, ln viscosity of polymer melts shows first to second order dependence on ln molecular weight and above M_c dependence is on the 3.4 power.[14] The slope of a plot of ln η_o vs ln M of the OBMA samples is approximately 6. While the accuracy of the data cannot be high, clearly the dependence of ln viscosity on ln molecular weight is very strong. This presumably results from the rapid change in T_g over this range in M as compared to the slower change in T_g with M for polymers with molecular weights near to M_c. This suggestion is in line with the even greater dependence of viscosity on molecular weight for the two OMMA samples. (T_g could not be determined for the OMMA-1600 since a solvent free sample could not be obtained and T_g of its solutions could only be determined for a narrow range of concentrations.) The data for viscosity of solvent free OBMA as a function of temperature given in *Table* 5 were analyzed using WLF equation (1) neglecting the density term. The r^2 correlation factor was 0.99987 with a random scatter of residuals.

SUMMARY

Glass transition temperature of ethyl benzene and solutions of OBMA in ethyl benzene could be determined by DSC at all concentrations. Available equations for calculating the dependence of T_g on concentration were found to be poor models. Equation (10), or its equivalent (11), were found to fit experimental data over the whole range from $w_o = 0$ to 1.

$$\frac{1}{T_g} = \frac{w_s}{T_{gs}} + \frac{w_o}{T_{go}} + k_3 w_o w_s \qquad (10)$$

While the T_g of *m*-xylene could be determined, the T_g of dilute solutions of OBMA in *m*-xylene could not be determined. Equation (10) still served as a satisfactory model for the whole range of concentrations. As $\bar{M}_n$ of the OBMA increased, the minimum w_o at which T_g could be determined increased. OMMA solutions in *m*-xylene required still higher concentrations for T_g determination. In the cases of *o*- and *p*-xylenes, T_g could be determined only of relatively high concentration solutions of OBMA.

The temperature dependence of viscosity of OBMA in *m*-xylene was found to follow the full form of the WLF equation (1) using the lowest experimental temperature as reference temperature.

$$\ln \eta = \ln \eta_{T_r} - \frac{c_1 (T-T_r)}{c_2 + (T-T_r)} - \ln \frac{\rho_r T_r}{\rho T} \qquad (1)$$

While simplified forms of the WLF equation were not as good models, they can be useful empirical relationships except for dilute solutions. For example, equation (13) fits data for a wide range of oligomers and oligomer solutions very well and is a much better empirical relationship than Arrhenius type equations.

$$\frac{1}{34.5 - \ln \eta} = \frac{1}{A} + \frac{B}{A\,(T-T_g)} \qquad (13)$$

The fact that "universal constants" such as in equation (2) are not appropriate for all oligomers and oligomer solutions indicates that although $(T-T_g)$ is an important factor controlling the viscosity of oligomer solutions, it cannot be the only factor.

While Erickson's equation is not a satisfactory model for the dependence of relative viscosity on concentration, addition of a second order term to the denominator as in equation (19) substantially improves the fit of the data.

$$\ln \eta_r = w_o/(k_1 - k_2 w_o + k_3 w_o^2) \qquad (19)$$

While equation (19) is not an exact model, it is a useful empirical relationship which provides quite close correlation between observed and predicted data for all the systems studied in this work and Erickson's data.

Constant k_1 in equation (19) is the reciprocal of weight intrinsic viscosity. Weight intrinsic viscosity is properly converted into intrinsic viscosity by dividing by density of an oligomer solution at a concentration of $w_o = k_1$. Mark-Houwink constant *a* for OBMA solutions *m*-xylene was found to be 0.49. This is in line with literature statements that oligomer solutions behave as if they are at theta conditions in any solvent. However, intrinsic viscosity of OBMA solutions was found to be dependent on solvent structure and was found to decrease with increasing temperature. These results indicate that *m*-xylene at 25°C is not a theta

Table 5—Calculated ln η_o of Solvent Free OBMA-2200 As a Function of Temperature

T (°C)	ln η_o (mPa·s)
15	16.52
20	15.38
25	14.83
30	13.88
35	13.23
40	12.61
50	10.81
60	9.96
80	8.09

condition for OBMA. Further work with a wider range of oligomers, molecular weights, solvents and temperatures will be required to understand the relationships.

Viscosity of solutions in the range of concentration of interest in high solids coatings was found to depend upon oligomer T_g (which depends on both $\overline{M}_n$ and structure), solvent T_g and solvent-oligomer interaction, and, of course, temperature, concentration and solvent viscosity. However, further studies are essential to understand the relative importance of the factors and interactions between them. The effect of polar substituents such as OH and COOH will be of particular importance. Such an understanding would make a significant contribution to the efficiency of formulating high solids coatings.

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APPENDIX

Sample	w_o (g/g)	T±0.03 (°C)	Density	Exp'tl calcd by eq. 4 or 5	T_g (K)	Exp'tl calcd by eq. 10	Ln η (mPa·s)	ln η_r
***m*-xylene**	—	15	0.8685	0.8685	125	125	−0.4326	0
	—	20	0.8641	0.8641	—	—	−0.4950	—
	—	25	0.8598	0.8600	—	—	−0.5549	—
	—	30	0.8554	0.8554	—	—	−0.6101	—
	—	35	0.8510	0.8510	—	—	−0.6639	—
	—	40	0.8466	0.8466	—	—	−0.7170	—
	—	50	—	0.8377	—	—	−0.8245	—
	—	60.1	—	0.8287	—	—	−0.9100	—
	—	80.5	—	0.8103	—	—	−1.0803	—
OBMA1100	0.011	25	0.8615	0.8615	—	—	−0.5278	0.0271
(*m*-xylene)	0.017	25	0.8622	0.8623	—	—	−0.5108	0.0441
	0.028	25	0.8637	0.8637	—	—	−0.4789	0.0760
	0.056	25	0.8675	0.8675	—	—	−0.4003	0.1546
	0.083	25	0.8713	0.8712	—	—	−0.3176	0.2373
	0.110	25	0.8752	0.8750	—	—	−0.2323	0.3226

Sample	w_o (g/g)	T±0.03 (°C)	Density	Exp'tl calcd by eq. 4 or 5	T_g (K)	Exp'tl calcd by eq. 10	Ln η (mPa·s)	ln η_r
	0.116	25	0.8758	0.8758	—	—	−0.2146	0.3403
	0.137	25	0.8789	0.8787	—	—	−0.1394	0.4155
	0.228	25	0.8922	0.8916	—	—	0.1965	0.7514
	0.337	25	0.9072	0.9075	—	—	0.6728	1.2277
	0.441	25	0.9230	0.9232	—	—	1.2924	1.8473
	0.535	25	0.9385	0.9379	—	—	2.0203	2.5752
	0.542	25	0.9396	0.9389	—	—	2.0710	2.6259
	0.668	25	0.9553	0.9592	170	173	3.0003	3.5552
	0.733	25	0.9715	0.9699	183	181	4.0314	4.5863
	0.784	25	0.9798	0.9784	188	188	4.9406	5.4955
	0.878	25	0.9942	0.9944	203	203	6.7083	7.2632
	0.967	15	1.0174	1.0174	219	220	10.3500	—
	—	20	1.0136	1.0137	—	—	9.7330	—
	—	25	1.0099	1.0100	—	—	9.1686	9.7235
	—	30	1.0064	1.0063	—	—	8.6774	—
	—	35	1.0026	1.0026	—	—	8.2029	—
	—	40	0.9990	0.9990	—	—	7.7574	—
	1.000	—	—	—	229	228	—	—
OBMA1400	0.024	25	0.8628	0.8635	—	—	−0.4922	0.0627
(*m*-xylene)	0.116	25	0.8767	0.8772	—	—	−0.1688	0.3861
	0.227	25	0.8935	0.8943	—	—	0.3169	0.8718
	0.334	25	0.9098	0.9111	—	—	0.8971	1.4520
	0.437	25	0.9269	0.9277	—	—	1.6334	2.1884
	0.538	25	0.9418	0.9442	157	158	2.4655	3.0204
	0.543	15	0.9543	0.9542	—	159	2.7935	—
	—	20	0.9501	0.9502	—	—	2.6293	—
	—	25	0.9462	0.9451	—	—	2.4906	3.0443
	—	30	0.9423	0.9422	—	—	2.3546	—
	—	35	0.9382	0.9382	—	—	2.2482	—
	—	40	0.9341	0.9341	—	—	2.1214	—
	—	50	—	0.9260	—	—	1.8892	—
	—	60.2	—	0.9177	—	—	1.6799	—
	—	80.3	—	0.9011	—	—	1.3040	—
	0.567	25	0.9499	0.9490	—	—	2.8226	3.3775
	0.610	25	0.9549	0.9562	—	—	3.2036	3.7585
	0.706	25	0.9745	0.9725	180	178	4.6540	5.2089
	0.751	25	0.9834	0.9805	184	184	5.3505	5.9054
	0.800	25	0.9912	0.9890	197	192	6.3637	6.9186
	0.810	15	1.0006	1.0006	193	194	7.3604	—
	—	20	0.9967	0.9968	—	—	6.9645	—
	—	25	0.9930	0.9930	—	—	6.6258	7.1783
	—	30	0.9893	0.9893	—	—	6.3071	—
	—	35	0.9855	0.9855	—	—	6.0589	—
	—	40	0.9817	0.9817	—	—	5.7613	—
	—	50	—	0.9742	—	—	5.2159	—
	—	60.2	—	0.9666	—	—	4.7300	—
	—	80.3	—	0.9516	—	—	3.9080	—
	0.863	25	0.9967	1.0001	203	203	7.5583	8.1134
	0.896	25	1.0049	1.0060	205	210	8.0598	8.6147
	0.941	25	1.0126	1.0141	216	220	9.8063	10.3612
	0.980	—	—	—	234	230	—	—
	1.000	—	—	—	237	236	—	—
OBMA2100	0.005	25	0.8602	0.8601	—	—	−0.5259	0.0171
(*m*-xylene)	0.012	25	0.8610	0.8612	—	—	−0.4976	0.0454
	0.016	25	0.8618	0.8618	—	—	−0.4829	0.0601
	0.023	25	0.8624	0.8628	—	—	−0.4668	0.0762
	0.027	25	0.8634	0.8635	—	—	−0.4447	0.0983
	0.030	25	0.8640	0.8641	—	—	−0.4216	0.1214
	0.033	25	0.8645	0.8645	—	—	−0.4095	0.1335
	0.039	25	0.8652	0.8655	—	—	−0.3871	0.1559
	0.077	25	0.8718	0.8716	—	—	−0.2083	0.3347
	0.125	25	0.8795	0.8795	—	—	0.0296	0.5726
	0.164	25	0.8861	0.8859	—	—	0.2469	0.7899
	0.203	25	0.8922	0.8924	—	—	0.4762	1.0192
	0.262	25	0.9027	0.9025	—	—	0.8713	1.4143
	0.316	25	0.9118	0.9117	—	—	1.2641	1.8071
	0.343	25	0.9163	0.9164	—	—	1.4839	2.0269
	0.393	25	0.9252	0.9252	—	—	1.9344	2.4774
	0.427	25	0.9312	0.9312	—	—	2.2418	2.7848
	0.478	25	0.9405	0.9405	—	—	2.8034	3.3464

Sample	w_o (g/g)	T±0.03 (°C)	Density	Exp'tl calcd by eq. 4 or 5	T_g (K)	Exp'tl calcd by eq. 10	Ln η (mPa·s)	ln η_r
	0.529	25	0.9498	0.9497	—	—	3.3878	3.9308
	0.578	25	0.9589	0.9587	—	—	4.0378	4.5808
	0.595	25	0.9617	0.9619	—	—	4.3360	4.8790
	1.000	—	—	—	258	—	—	—
OBMA3600	0.013	15	0.8705	0.8706	—	125	−0.3820	—
(*m*-xylene)	—	20	0.8659	0.8662	—	—	−0.4464	—
	—	25	0.8616	0.8621	—	—	−0.5046	0.0503
	—	30	0.8573	0.8575	—	—	−0.5616	—
	—	35	0.8529	0.8531	—	—	−0.6173	—
	—	40	0.8485	0.8488	—	—	−0.6702	—
	—	50	—	0.8399	—	—	−0.7718	—
	—	60.1	—	0.8310	—	—	−0.8662	—
	—	80.4	—	0.8142	—	—	−1.0368	—
	0.030	15	0.8731	0.8733	—	126	−0.3062	—
	—	20	0.8686	0.8689	—	—	−0.3711	—
	—	25	0.8643	0.8647	—	—	−0.4323	0.1226
	—	30	0.8600	0.8602	—	—	−0.4910	—
	—	35	0.8555	0.8558	—	—	−0.5475	—
	—	40	0.8511	0.8515	—	—	−0.6012	—
	—	50	—	0.8427	—	—	−0.7020	—
	—	60.1	—	0.8340	—	—	−0.8005	—
	—	80.35	—	0.8151	—	—	−0.9783	—
	0.084	15	0.8818	0.8819	—	128	−0.0612	—
	—	20	0.8772	0.8775	—	—	−0.1306	—
	—	25	0.8729	0.8733	—	—	−0.1943	0.3606
	—	30	0.8687	0.8689	—	—	−0.2605	—
	—	35	0.8642	0.8645	—	—	−0.3193	—
	—	40	0.8598	0.8603	—	—	−0.3778	—
	—	50	—	0.8518	—	—	−0.4889	—
	—	60.1	—	0.8433	—	—	−0.5918	—
	—	78.95	—	0.8261	—	—	−0.7712	—
	0.108	15	0.8855	0.8857	—	128	0.0616	—
		20	0.8810	0.8813	—	—	−0.0127	—
		25	0.8767	0.8771	—	—	−0.0794	0.4755
		30	0.8725	0.8727	—	—	−0.1453	—
		35	0.8681	0.8683	—	—	−0.2086	—
		40	0.8637	0.8642	—	—	−0.2694	—
		50	—	0.8558	—	—	−0.3838	—
		60.1	—	0.8474	—	—	−0.4920	—
	—	80.4	—	0.8289	—	—	−0.6874	—
	0.150	15	0.8923	0.8926	130	130	0.2977	—
	—	20	0.8879	0.8882	—	—	0.2184	—
	—	25	0.8838	0.8840	—	—	0.1397	0.6946
	—	30	0.8795	0.8797	—	—	0.0697	—
	—	35	0.8751	0.8753	—	—	0.0056	—
	—	40	0.8708	0.8712	—	—	−0.0607	—
	0.220	15	0.9038	0.9042	—	134	0.7341	—
	—	20	0.8994	0.8998	—	—	0.6437	—
	—	25	0.8953	0.8956	—	—	0.5640	1.1189
	—	30	0.8910	0.8914	—	—	0.4839	—
	—	35	0.8867	0.8871	—	—	0.4040	—
	—	40	0.8824	0.8831	—	—	0.3343	—
	—	50	—	0.8750	—	—	0.1964	—
	—	60.1	—	0.8670	—	—	0.0702	—
	—	80.35	—	0.8474	—	—	−0.1567	—
	0.254	—	—	—	135	135	—	—
	0.268	15	0.9126	0.9121	—	136	1.0526	—
	—	20	0.9082	0.9078	—	—	0.9544	—
	—	25	0.9039	0.9035	—	—	0.8588	1.4138
	—	30	0.9000	0.8995	—	—	0.7800	—
	—	35	0.8957	0.8951	—	—	0.6959	—
	—	40	0.8914	0.8911	—	—	0.6143	—
	0.271	15	0.9124	0.9127	—	136	1.0936	—
	—	20	0.9082	0.9084	—	—	0.9911	—
	—	25	0.9039	0.9041	—	—	0.8956	1.4505
	—	30	0.8997	0.9000	—	—	0.8063	—
	—	35	0.8954	0.8957	—	—	0.7229	—
	—	40	0.8914	0.8917	—	—	0.6480	—
	—	50	—	0.8837	—	—	0.4888	—
	—	60.1	—	0.8758	—	—	0.3477	—

Sample	w_o (g/g)	T±0.03 (°C)	Density	Exp'tl calcd by eq. 4 or 5	T_g (K)	Exp'tl calcd by eq. 10	Ln η (mPa·s)	ln η_r
	—	78.95	—	0.8582	—	—	0.113	—
	0.278	—	—	—	137	137	—	—
	0.443	—	—	—	148	148	—	—
	0.444	15	—	0.9425	—	149	2.6682	—
	—	20	—	0.9383	—	—	2.5147	—
	—	25	—	0.9341	—	—	2.2659	2.9208
	—	30	—	0.9302	—	—	2.2486	—
	—	35	—	0.9260	—	—	2.1225	—
	—	40	—	0.9219	—	—	1.9958	—
	0.456	15	0.9448	0.9446	148	150	2.9748	—
	—	20	0.9407	0.9404	—	—	2.7920	—
	—	25	0.9366	0.9363	—	—	2.6393	3.0349
	—	30	0.9326	0.9324	—	—	2.4913	—
	—	35	0.9285	0.9285	—	—	2.3718	—
	—	40	0.9243	0.9241	—	—	2.2332	—
	—	50	—	0.9160	—	—	1.9782	—
	—	60.2	—	0.9077	—	—	1.7418	—
	—	80.3	—	0.8904	—	—	1.3388	—
	0.486	—	—	—	156	152	—	—
	0.586	—	—	—	164	163	—	—
	0.657	15	0.9813	0.9810	171	172	5.7133	—
	—	20	0.9773	0.9770	—	—	5.4169	—
	—	25	0.9734	0.9731	—	—	5.1551	5.7100
	—	30	0.9696	0.9693	—	—	4.9241	—
	—	35	0.9656	0.9653	—	—	4.6835	—
	—	40	0.9616	0.9607	—	—	4.4689	—
	—	50	—	0.9519	—	—	4.0608	—
	—	60.1	—	0.9422	—	—	3.7029	—
	—	80.35	—	0.9309	—	—	3.0906	—
	0.701	—	—	—	178	179	—	—
	0.705	15	0.9897	0.9899	—	180	6.7650	—
	—	20	0.9856	0.9859	—	—	6.4215	—
	—	25	0.9819	0.9821	—	—	6.0499	6.6048
	—	30	0.9782	0.9783	—	—	5.7597	—
	—	35	0.9742	0.9744	—	—	5.5200	—
	—	40	0.9704	0.9697	—	—	5.2626	—
	0.724	15	0.9940	0.9935	183	183	7.1362	—
	—	20	0.9902	0.9896	—	—	6.7768	—
	—	25	0.9862	0.9858	—	—	6.4458	7.0007
	—	30	0.9825	0.9820	—	—	6.1369	—
	—	35	0.9786	0.9782	—	—	5.8598	—
	—	40	0.9744	0.9733	—	—	5.5940	—
	—	50	—	0.9640	—	—	5.0921	—
	—	60.1	—	0.9536	—	—	4.6405	—
	—	78.95	—	0.9413	—	—	3.9390	—
	0.803	15	1.0079	1.0084	196	198	9.0097	—
	—	20	1.0040	1.0045	—	—	8.5453	—
	—	25	1.0003	1.0008	—	—	8.1151	8.6701
	—	30	0.9966	0.9971	—	—	7.7391	—
	—	35	0.9929	0.9933	—	—	7.3757	—
	—	40	0.9862	0.9882	—	—	7.0333	—
	—	50	—	0.9783	—	—	6.4032	—
	—	60.1	—	0.9670	—	—	5.8400	—
	—	80.2	—	0.9271	—	—	4.8987	—
	0.807	—	—	—	204	199	—	—
	0.838	—	—	—	206	206	—	—
	0.860	15	—	1.0194	207	211	10.8758	—
	—	20	—	1.0157	—	—	10.3104	—
	—	25	—	1.0121	—	—	9.7238	10.2787
	—	30	—	1.0083	—	—	9.2484	—
	—	35	—	1.0047	—	—	8.8336	—
	—	40	—	0.9992	—	—	8.4310	—
	0.951	—	—	—	242	238	—	—
	1.000	—	—	—	256	257	—	—
OBMA2200 (*m*-xylene)	0.020	25	0.8626	0.8626	—	—	−0.4620	0.0861
	0.060	25	0.8691	0.8691	—	—	−0.2784	0.2698
	0.080	25	0.8721	0.8724	—	—	−0.1803	0.3679
	0.100	25	0.8758	0.8757	—	—	−0.0726	0.4756
	0.200	25	0.8924	0.8924	—	—	0.5188	1.0670
	0.300	25	0.9095	0.9096	140	—	1.2149	1.7631

Sample	w_o (g/g)	T±0.03 (°C)	Density	Exp'tl calcd by eq. 4 or 5	T_g (K)	Exp'tl calcd by eq. 10	Ln η (mPa·s)	ln η_r
	0.400	25	0.9283	0.9274	152	—	2.0844	2.6326
	0.500	25	0.9455	0.9458	154	—	3.2108	3.7590
	0.600	25	0.9642	0.9647	162	—	4.5880	5.1362
	0.700	25	0.9836	0.9839	191	—	6.3026	6.8508
	0.799	25	1.0038	1.0034	202	—	8.7275	9.2756
	0.931	—	—	—	236	—	—	—
	1.000	—	—	—	264	—	—	—
OBMA3600	0.000	25	0.8567	—	—	—	−0.5058	0.0000
(*p*-xylene)	0.030	25	0.8612	—	—	—	−0.3886	0.1172
	0.037	25	0.8623	—	—	—	−0.3581	0.1477
	0.060	25	0.8661	—	—	—	−0.2536	0.2522
	0.080	25	0.8693	—	—	—	−0.1590	0.3468
	0.101	25	0.8728	—	—	—	−0.0545	0.4514
	0.200	25	0.8897	—	—	—	0.5008	1.0066
	0.300	25	0.9073	—	—	—	1.1969	1.7028
	0.400	25	0.9252	—	—	—	2.0268	2.5327
	0.503	25	0.9443	—	—	—	3.1268	3.6326
	0.602	25	0.9639	—	183	—	4.5020	5.0079
	0.710	25	0.9855	—	195	—	6.4770	6.9828
	0.804	25	1.0038	—	207	—	8.9822	9.4880
	0.975	—	—	—	257	—	—	—
OBMA3600	0.000	25	0.8748	—	—	—	−0.2957	0.0000
(*o*-xylene)	0.020	25	0.8775	—	—	—	−0.2058	0.0899
	0.040	25	0.8805	—	—	—	−0.1109	0.1848
	0.080	25	0.8865	—	—	—	0.0862	0.3819
	0.200	25	0.9049	—	—	—	0.8020	1.0977
	0.300	25	0.9208	—	—	—	1.5282	1.8239
	0.399	25	0.9370	—	153	—	2.3979	2.6936
	0.500	25	0.9532	—	157	—	3.4965	3.7922
	0.600	25	0.9703	—	176	—	4.8752	5.1709
	0.693	25	0.9830	—	193	—	6.4052	6.7009
	0.798	25	1.0041	—	211	—	8.9619	9.2576
	0.968	—	—	—	247	—	—	—
OBMA3600	0.000	25	0.8617	—	122	—	−0.4620	0.0000
(ethyl-benzene)	0.020	25	0.8649	—	—	—	−0.3930	0.0690
	0.058	25	0.8700	—	—	—	−0.2194	0.2426
	0.100	25	0.8778	—	124	—	−0.0100	0.4520
	0.200	25	0.8941	—	126	—	0.5596	1.0217
	0.300	25	0.9113	—	130	—	1.2413	1.7033
	0.400	25	0.9290	—	—	—	2.0844	2.5465
	0.498	25	0.9457	—	144	—	3.0910	3.5531
	0.600	25	0.9654	—	156	—	4.4998	4.9618
	0.698	25	0.9385	—	176	—	6.1964	6.6585
	0.802	25	1.0045	—	204	—	8.8622	9.3242
	0.928	—	—	—	230	—	—	—
OMMA1000	0.020	15	0.8728	—	—	—	−0.3696	0.0627
(*m*-xylene)	—	25	0.8640	—	—	—	−0.4878	0.0587
	—	40	0.8508	—	—	—	−0.6597	0.0536
	0.049	15	0.8794	—	—	—	−0.2718	0.1605
	—	25	0.8706	—	—	—	−0.3975	0.1490
	—	40	0.8575	—	—	—	−0.5727	0.1406
	0.060	15	0.8819	—	—	—	−0.2319	0.2004
	—	25	0.8731	—	—	—	−0.3638	0.1826
	—	40	0.8599	—	—	—	−0.5413	0.1721
	0.100	15	0.8913	—	—	—	−0.0747	0.3576
	—	25	0.8825	—	—	—	−0.2083	0.3382
	—	40	0.8694	—	—	—	−0.4035	0.3099
	0.200	15	0.9152	—	138	—	0.4055	0.8378
	—	25	0.9065	—	—	—	0.2390	0.7855
	—	40	0.8934	—	—	—	−0.0020	0.7113
	0.300	15	0.9407	—	141	—	1.0473	1.4796
	—	25	0.9318	—	—	—	0.8242	1.3706
	—	40	0.9189	—	—	—	0.5188	1.2321
	0.350	15	0.9536	—	146	—	1.4469	1.8792
	—	25	0.9450	—	—	—	1.1787	1.7251
	—	40	0.9320	—	—	—	0.8416	1.5549

Sample	w_o (g/g)	T±0.03 (°C)	Density	Exp'tl calcd by eq. 4 or 5	T_g (K)	Exp'tl calcd by eq. 10	Ln η (mPa·s)	ln η_r
	0.400	15	0.9669	—	—	—	1.9155	2.3478
	—	25	0.9581	—	—	—	1.5953	2.1418
	—	40	0.9452	—	—	—	1.1694	1.8827
	0.437	15	0.9772	—	—	—	2.3418	2.7741
	—	25	0.9685	—	—	—	1.9879	2.5343
	—	40	0.9558	—	—	—	1.5151	2.2285
	0.500	15	0.9948	—	170	—	3.1822	3.6145
	—	25	0.9861	—	—	—	2.7147	3.2611
	—	40	0.9734	—	—	—	2.0425	2.7559
	0.600	15	1.0246	—	187	—	5.0689	5.5012
	—	25	1.0161	—	—	—	4.3694	4.9159
	—	40	1.0034	—	—	—	3.4468	4.1602
	0.700	15	1.0559	—	206	—	7.8240	8.2564
	—	25	1.0474	—	—	—	6.7464	7.2929
	—	40	1.0350	—	—	—	5.3566	6.0699
	0.813	—	—	—	230	—	—	—
	1.000	—	—	—	274	—	—	—
OMMA1600 (*m*-xylene)	0.020	25	0.8650	—	—	—	—	0.0871
	0.088	25	0.8819	—	—	—	—	0.4090
	0.125	25	0.8915	—	—	—	—	0.6231
	0.153	25	0.8986	—	—	—	—	0.7902
	0.160	25	0.9007	—	—	—	—	0.8426
	0.170	25	0.9033	—	—	—	—	0.9122
	0.173	25	0.9041	—	—	—	—	0.9331
	0.184	25	0.9068	—	—	—	—	1.0175
	0.188	25	0.9079	—	—	—	—	1.0407
	0.201	25	0.9115	—	—	—	—	1.1890
	0.203	25	0.9121	—	—	—	—	1.1457
	0.204	25	0.9124	—	—	—	—	1.1658
	0.208	25	0.9133	—	—	—	—	1.1886
	0.220	25	0.9165	—	—	—	—	1.3050
	0.237	25	0.9212	—	—	—	—	1.4479
	0.241	25	0.9223	—	—	—	—	1.4607
	0.242	25	0.9227	—	—	—	—	1.5064
	0.248	25	0.9243	—	—	—	—	1.5219
	0.254	25	0.9260	—	—	—	—	1.5666
	0.256	25	0.9265	—	—	—	—	1.6585
	0.263	25	0.9285	—	—	—	—	1.6615
	0.285	25	0.9347	—	—	—	—	1.8989
	0.286	25	0.9349	—	—	—	—	1.9265
	0.293	25	0.9367	—	—	—	—	1.9797
	0.296	25	0.9376	—	—	—	—	2.0610
	0.303	25	0.9397	—	—	—	—	2.1079
	0.313	25	0.9424	—	—	—	—	2.1067
	0.314	25	0.9428	—	—	—	—	2.2260
	0.326	25	0.9461	—	—	—	—	2.3551
	0.356	25	0.9548	—	—	—	—	2.6929
	0.359	25	0.9557	—	—	—	—	2.7783
	0.408	25	0.9698	—	—	—	—	3.3232
	0.409	25	0.9712	—	—	—	—	3.5026
	0.421	25	0.9739	—	—	—	—	3.6718
	0.593	25	1.0269	—	—	—	—	7.2449
	0.713	25	1.0664	—	—	—	—	11.9337

List of Symbols Used

E_v — Activation Energy for Viscous Flow (kcal/mol)
$\overline{M}_n$ — Number Average Molecular Weight
$\overline{M}_w$ — Weight Average Molecular Weight
T_b — Boiling Point
T_g — Glass Transition Temperature
T_{go} — Glass Transition Temperature of Solvent Free Oligomer
T_{gs} — Glass Transition Temperature of Solvent
T_m — Melting Point
T_r — Reference Temperature
w_o — Weight Fraction Oligomer
w_s — Weight Fraction Solvent
δ — Solubility Parameter
δ_o — Solubility Parameter of Oligomer
δ_s — Solubility Parameter of Solvent
η — Absolute Viscosity (mPa·s)
η_o — Viscosity of Solvent Free Oligomer
η_r — Relative Viscosity (η/η_s)
η_s — Viscosity of Solvent
η_{T_g} — Viscosity at T_g
η_{T_r} — Viscosity at T_r
$[\eta]$ — Intrinsic Viscosity (mL/g)
$[\eta]_w$ — Weight Intrinsic Viscosity (g/g)
ρ — Density (g/mL)
ρ_g — Density at T_g
ρ_r — Density at T_r
ρ_s — Solvent Density

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In recent years, numerous patents claiming novel chemical structures, improved sythetic procedures, and new compositions of matter have been published on isoindoline pigments.

A special group of pigments, obtained by the condensation of diiminoisoindoline with cyanoacetarylides, is now reported. Synthesis, influence of substituents on pigment properties, and crystal structures are discussed.

The potential and limits of such pigments are illustrated by means of examples chosen from this group. Attention is particularly drawn to the outstanding pigment properties in general, as well as to the manifold possibilities of application, including styling in paints.

INTRODUCTION

In recent years, numerous patents have been published on isoindoline compounds. As early as 30 years ago, patents were taken out by German firms on isoindoline dyestuffs.[1,2] The technical importance of this interesting class of products was realized much later. In the seventies, the pigment application of biscondensation products of diiminoisoindoline with methylene active compounds was patented by BASF.[3] These patents include C.I. Pigment Yellow 139 (*Figure* 1). Although this molecule has a very simple structure—it is the biscondensation product of diiminoisoindoline with barbituric acid[4]—it was necessary to develop special procedures to obtain the product in the right pigmentary form. Not only the chemical purity, but also the particle form and size of the pigment were of importance and hence needed to be improved.[5,6] Depending on its particle size, the pigment can be very brilliant and transparent or opaque with a high durability (*Figure* 2).

The success of such a simple product, which can be synthesized from easily available raw materials in good yield, evidently triggered extensive activity in this field of chemistry. The chemical possibilities of varying the shades and properties of these dyestuffs are indeed manifold. Much work in this direction has been done by different research groups. For the sake of illustration, three examples from the patent literature are mentioned (*Table* 1).

The formulae represent three different classes of isoindoline structures. The first class has a symmetrical molecule with methylene active compounds. The brown pigment is described in the same patent applications as Pigment Yellow 139. Durability and solvent fastness are high.

The possibility of synthesizing asymmetrical structures leads to a multiplication of the number of new chemical

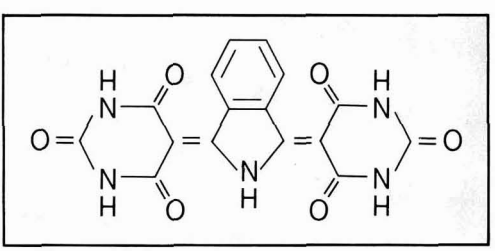

Figure 1—C.I. Pigment Yellow 139

Presented at the 62nd Annual Meeting of the Federation of Societies for Coatings Technology in Chicago, IL, October 25, 1985.

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Table 1—Examples of Different Classes of Isoindoline Pigments from Literature

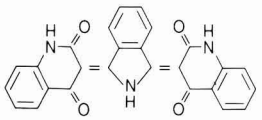
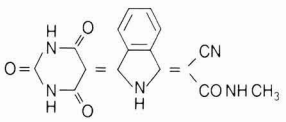
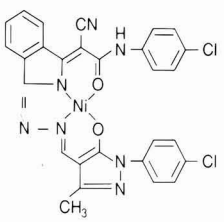
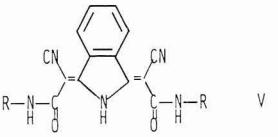
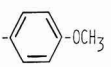
pigment	shade	reference
	brown	(3)
	yellow	(7, 8)
	red	(9)

Table 2—Influence of Substituent R on the Pigments Shades



V

R	SHADE IN 0.2% CONCENTRATION IN PLASTICIZED PVC
	YELLOW
	REDDISH YELLOW
	SCARLET
	BROWN

compounds. The second pigment in *Table* 1 has a very bright yellow shade. As the durability is only borderline, it cannot be used in high quality paints. We have found an additional interesting application for it. Because of its special properties, it is suitable for photoelectrophoretic imaging processes.

A third class consists of metal complexes. Based on isoindoline nucleus, we have synthesized a number of brilliant red metal complexes in our research laboratories. The pigment shown in *Table* 1 has a very high standard with respect to general pigment fastness properties.

Following, a group of isoindoline pigments obtained by the condensation of diiminoisoindoline with cyanoacetarylides is presented in more detail.

BISCONDENSATION OF DIIMINOISOINDOLINE WITH CYANOACETARYLIDES

Synthesis and Raw Materials

In *Figure* 3, the synthesis is described. Under mild conditions, a semi-condensed intermediate (III) is

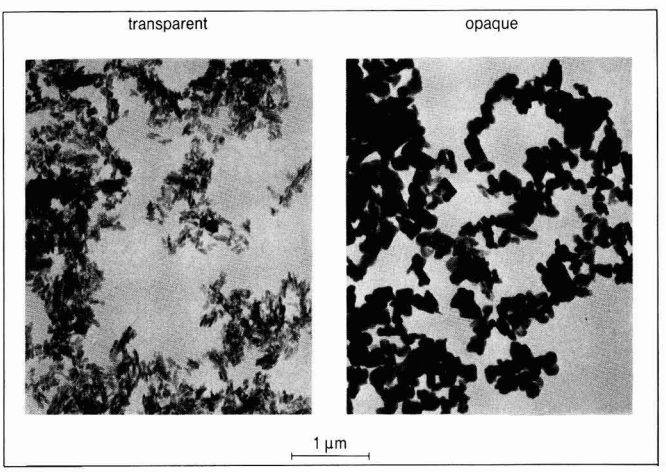


Figure 2—Photomicrograph of a Transmission Electron Microscopy of C.I. Pigment Yellow 139. Transparent form with high color strength, opaque form with good durability

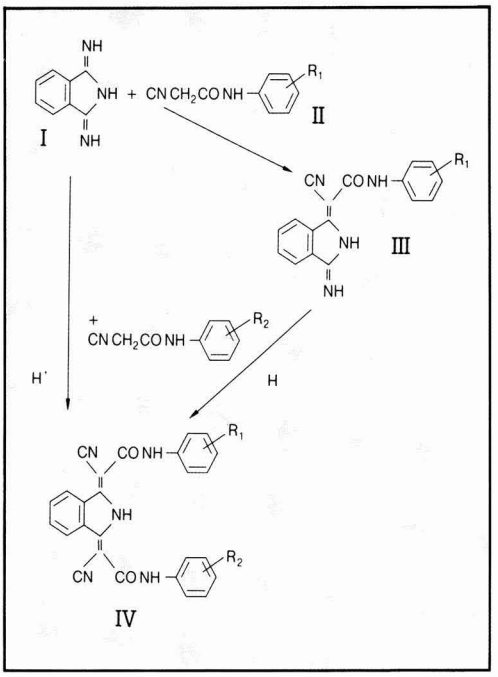


Figure 3—Synthesis of bis-methine-isoindoline pigments

obtained in very high yield and pure chemical form. This product can be isolated or condensed direct with a second methylene active compound (II) in the presence of an acid. We have synthesized a large number of new products by this reaction.

For the synthesis of pure asymmetrical compounds, it is better to first isolate the semi-condensed intermediate (III). The technical procedure for the preparation of isoindolines of this type is very simple: It is either a one- or a two-step process in which the required reaction temperature is moderately elevated and conventional solvents are used. The raw materials are easily available; the starting material for diiminoisoindoline (I) is phthalonitrile. The cyanoacetarylides (II) are prepared from corresponding amines and cyanoacetic acid derivatives.

Properties

When synthesizing new pigments belonging to isoindoline compounds of type (V) (*Table* 2), we were first surprised by the wide variety of colors obtained. Upon alteration of the substituent R, the shade of the pigments can change from yellow, through orange and red, to brown. In *Table* 2 this effect is demonstrated.

The influence of the substituent R can hardly be adequately explained by the well-known rules of chromophoric groups applying to soluble dyestuffs. We dissolved the pigments in DMF and measured their absorption spectra. Nearly all of the pigments presented in *Figure* 3 have a maxima at wavelengths of 495 and 498 nm and corresponding molar extinction coefficients of 26,000 to 29,500. These differences are negligible in comparison with the variation in the colors of the pigments observed in the solid state.

X-ray Structure and Analysis

To learn more about crystal structure, we have made a three-dimensional X-ray structure analysis of the symmetrical scarlet pigment (VI) shown in *Figure* 4. The single crystals were obtained by crystallization in dimethylsulfoxide. Crystal data: Monoclinic, $P2_1/n$, a = 3,862 Å; b = 30,014 Å; c = 19,153 Å; $\beta = 91.60°$; Z = 4.

The structure was solved by direct methods (Mulfan 77 system of programs). The H-atoms could not be localized. A first result is shown in *Figure* 4.

This allows us to gain some idea of the structure of the molecule with the steric arrangement of the atoms and the bond lengths. The illustrated molecule is not completely planar. One p-chloroanilide group is twisted out of the plane by an angle of 28° C (*Figure* 5). Unfortunately, we have crystal structure evidence for one symmetrical pigment only. The major problem in such investigations is posed by the difficulty of growing good and large enough single crystals. We hope that we shall soon succeed in obtaining an X-ray analysis of an asymmetrical pigment.

Crystal Modification

The importance of crystal structure is demonstrated in particular by the strong influence which different crystal

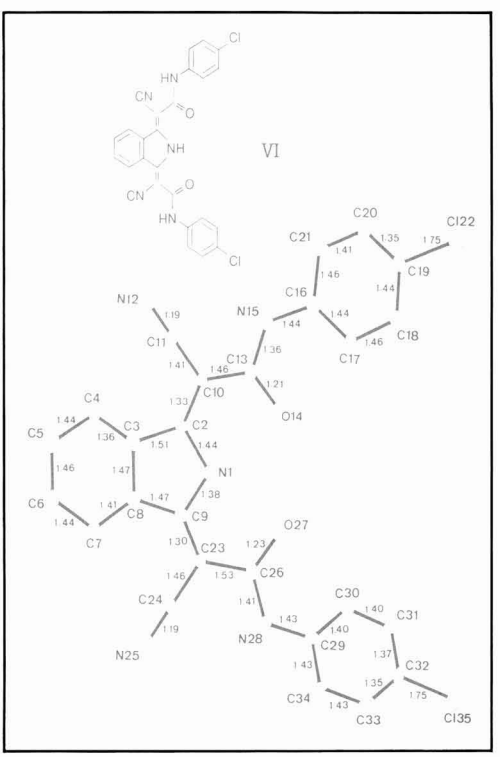


Figure 4—X-ray analysis of Pigment Scarlet VI. Steric arrangement of the atoms and bond angles

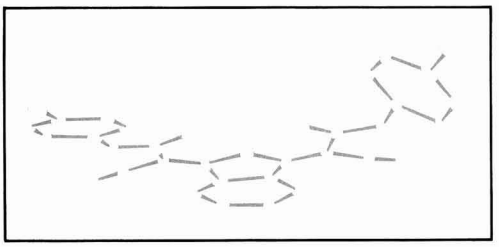


Figure 5—X-ray analysis. One p-chloroanilide group is twisted out of the plane by an angle of 28°C

Table 3—Influence of Crystal Modification on Shade

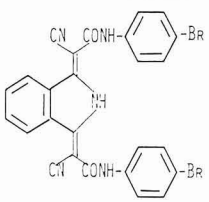
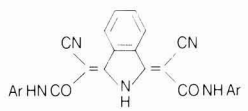
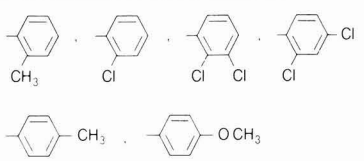
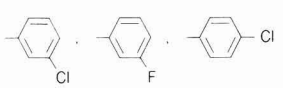
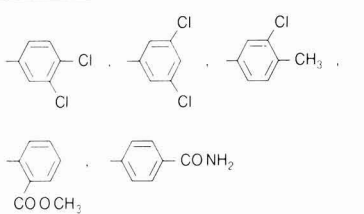
Pigment	Phase	Shade
	α	YELLOW
	β	SCARLET

Table 4—Influence of Substituents on Migration in PVC



Migration	Ar
strong	
some	
poor or no	

modifications can have on pigment properties. *Table* 3 shows the structural formula of a pigment which exists in two modifications. The synthesis of the pigment in standard solvents leads to the yellow modification. In certain solvents, such as DMF, one observes an alteration of the crystal habitus, which is accompanied by a color change. When testing the two modifications in pigment applications we noticed that the scarlet form is much more interesting. Whereas the scarlet compound has excellent fastness properties, the yellow one migrates and has poor light stability.

H-bridge Formation

Another very interesting fact which can be deduced from the X-ray analysis is the kind of H-bridge formation. *Figure* 6 shows two molecules which are held together by H-bridges.

Figure 7 represents a two-dimensional arrangement of several molecules. The distance of 3.0 to 3.2 Å clearly indicates that the intermolecular forces of the H-bridges are not very powerful. In *Figure* 8 the overlapping of two molecules may be seen.

Bleed Resistance

Such intermolecular forces are in turn responsible for another remarkable property of these isoindolines, namely insolubility. Depending on the substituent on the arylide group, we have either bad or good solvent fastness. *Table* 4 contains a selection of substituted anilides showing great differences in migration in PVC. In general, the solvent fastness decreases with substituents in the orthoposition, but an exception is the carboxylic ester group. Good migration fastness was achieved with

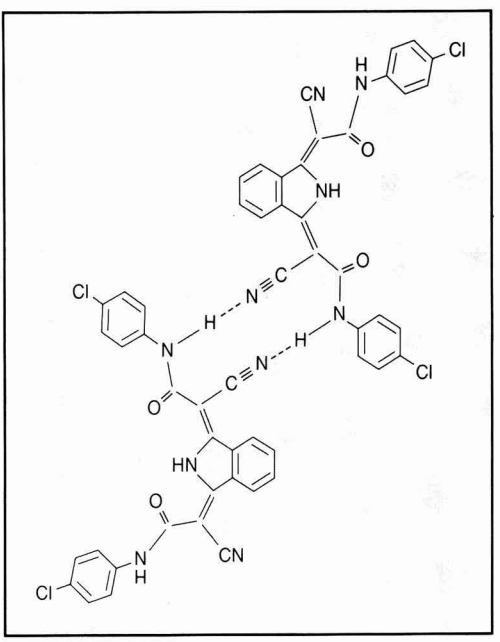


Figure 6—Formation of H-bridges

symmetrical products carrying two substituents in the meta- or para-position or with polar groups. Surprisingly, we found that simple asymmetrical compounds also can have good migration fastness. Two examples are shown in *Figure* 9. This is contrary to the experience that asymmetrical molecules are more soluble.

Together with the already described scarlet VI, the two orange pigments VII and VIII (*Figure* 9) exhibit a high durability.

High Quality Applications of Orange Pigment (VII)

The orange pigment (VII) is in many ways remarkably superior to the scarlet pigments of *Table* 4, and therefore efforts were especially concentrated on this pigment. Owing to the pigment's extraordinary weather-fastness, the main focus of interest has been on high quality applications, e.g., automotive finishes. In the final part of this paper, mention will be made of what has been done to evaluate the most interesting fields of application for this new pigment.

The outstanding outdoor durability of the orange pigment leads to good effects in opaque white reductions and metallic finishes. It was compared with well-known commercial orange pigments (*Table* 5).

As a result of its yellowish hue, brightness, and high color strength, this pigment (VII) has exclusive properties which cannot be imitated by others. Compared with the commercial products, it has outstanding color strength, which makes it an economical pigment for near-masstone applications. Satisfactory hiding power is displayed by TiO_2 combinations containing up to 50% colored pigment. Because of this high color strength it is possible to generate strong shades even at low pigment/binder ratios. As a consequence, it is possible to produce with this pigment bright shades with good gloss and good rheological properties in this shade area.

In the metallic application field, a number of styling proposals can be made. In combination with inorganic pigments, such as transparent iron oxide or carbon black, attractive shades result. With carbon black we have, for example, the styling possibility of creating a yellowish-

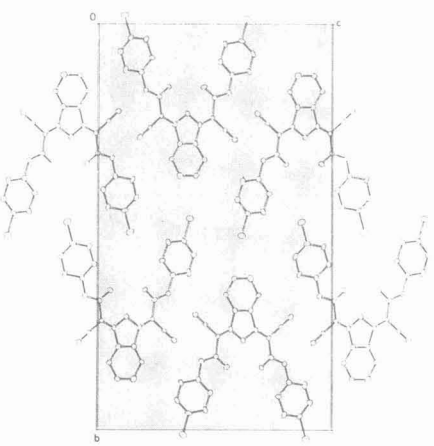


Figure 7—Two-dimensional arrangement of several molecules

Table 5—Commercial Orange Pigments Selected as Standards of Comparison

C. I. Pigment	chemical class
Orange 36	Monoazo
Orange 060	Monoazo
Orange 043	Perinon
Orange 64	Tetrachloro-isoindolinon

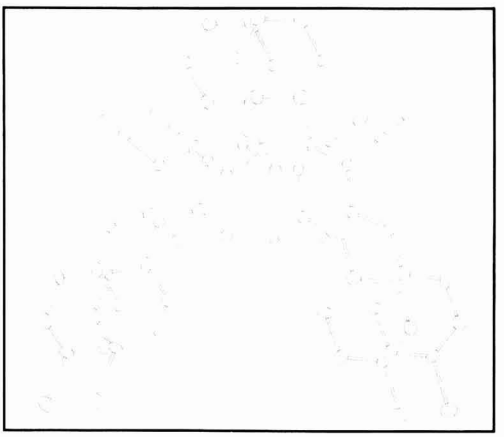


Figure 8—Overlapping of two molecules

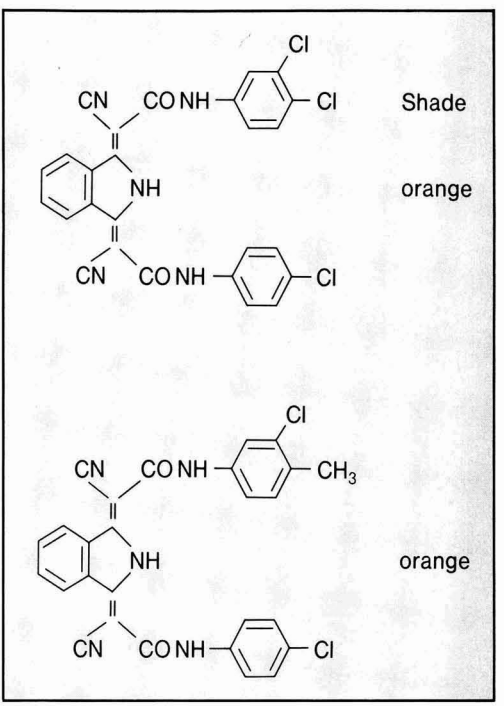


Figure 9—Asymmetrical orange pigments with good migration fastness in PVC

brown. As regards combinations with organic pigments, the new orange also has been tested together with isoindolinone yellow and quinacridone, perylene and anthraquinone reds. We think this opens up a wide scope for stylings: it is a challenge to imagination and creativity.

The durability in most interesting fields of application has been tested in two paint systems, namely in alkyd melamin and thermosetting acrylic enamels. First results from accelerated weathering demonstrated an excellent weather resistance of the new orange pigment. These results were confirmed by 12 and 24 months' weathering in Florida.

CONCLUSIONS

The present class of dyestuffs has turned out to be a rich source of several new pigment structures with clean and fast shades. It is not easy to surpass the high standard of organic pigments today. To evaluate the advantages of new products, specifically directed work has to be done in the field of application. Nevertheless, there is still room for genuine progress. This has been demonstrated by a new orange isoindoline pigment with outstanding weather resistance in paints for high quality applications.

ACKNOWLEDGMENT

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Errata

In the article, "Effects of Catalysts on the Kinetics of the Water-Toluene Diisocyanate Reaction," published in the March issue of the JCT, the authors state that the catalyst, Dabco,® was *obtained* from Eastman Chemical Co. Dabco, however, is a registered Tradename of its producer and owner, Air Products and Chemicals, Inc.

OPEN FORUM

What a Paint Formulator Should Know About Emulsion Polymerization

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The McCloskey Corporation*

Introduction

The tremendous growth in latex paint sales throughout the world has been brought about by a number of factors. Some of these factors are the availability of new raw materials; advances in emulsion polymerization techniques; consumer acceptance due to easier cleanup; the rising cost of hydrocarbon-based solvents; rising insurance cost and, of course, government regulations. A better understanding of the mechanism of polymerization has been achieved and this, in turn, has helped the latex manufacturer tailor-make latexes for specific applications.

Key Components in Emulsion Polymerization

It is relatively simple to make an emulsion in water by vigorous agitation, but it separates fairly soon after stirring ceases. A stabilizing system is required to prevent this from happening. This usually takes the form of surfactants and/or colloids, which are essential both during and after polymerization. Another feature is a means to convert the monomers to polymers (initiator). Other minor components are buffers and chain transfer agents, among others.

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An enormous number of monomers are available and suitable for emulsion polymerization. They are characterized by their ability to be polymerized by free radical addition polymerization.

Surfactants are a special class of materials which are capable of forming micelles. A micelle is an aggregate of surfactant where the water loving (hydrophilic) portions are oriented towards the water phase and the hydrophobic portions are oriented inwardly. When monomer is present the micelle is swelled with monomer.

The number of micelles and their size have a large bearing on the final latex properties. Initiators are materials that start the polymerization of monomer to polymer by generation of free radicals.

Water is required as the dispersing medium and as a heat-transfer medium during polymerization and as the solvent for surfactant and initiators. Water quality is important as dissolved salts lead to flocculation of latex particles.

Polymerization in Emulsion Systems

A typical simplified formula for an ionic stabilized latex is as follows:

Monomers	100
Ionic Surfactants	1
Non Ionic Surfactants	1
Water	100
Potassium Persulphate	2
Buffer	

The components are emulsified in the reaction vessel. Prior to reaction there are monomer molecules in solution, micelles containing monomer molecules, surfactant in solution, initiator in solution, and stabilized monomer droplets.

In the nucleation stage (*Figure* 1), surfactant in solution and dispersed monomer droplets approximately 10 microns stabilized by surfactant are present. Also present are free radicals generated in solution. There are a number of possible sites for polymerization. The significant sites depend upon the relative rates involved. The most likely mechanism involves free radicals or small growing chains which are formed in solution entering a micelle and propagating polymerization there.

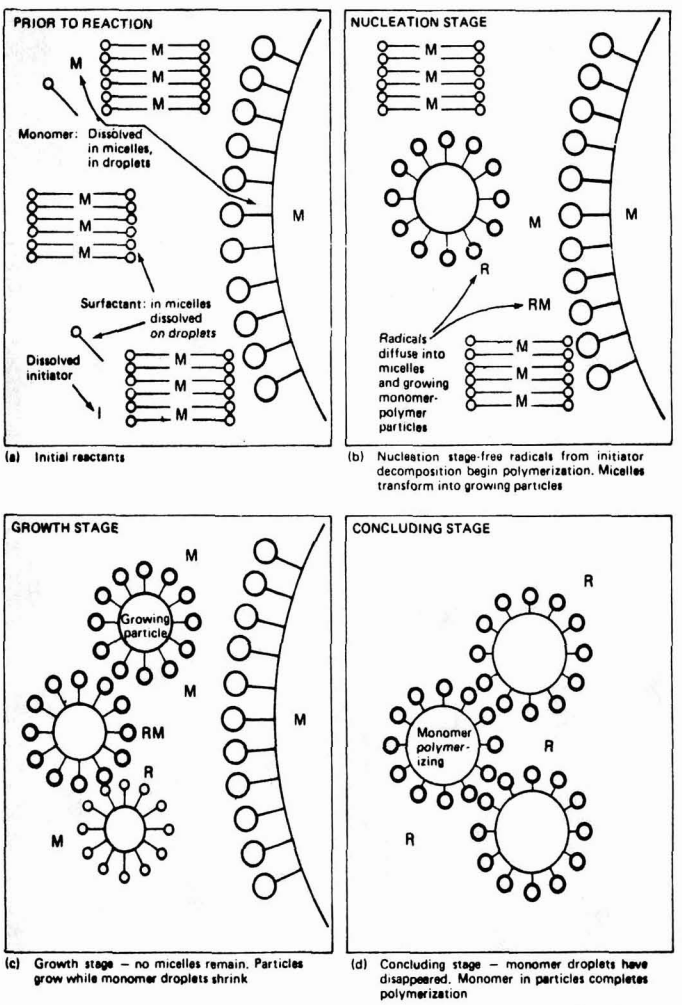
More monomer diffuses from the monomer droplets into solution and then into the micelle particles, which become ionically stabilized particles. As new surfaces form on the particles, ionic surfactants diffuse from micelle containing no polymer to absorb onto it. Soon all the micelles disappear, turning into latex particles or diffused onto the surface of these particles. At this stage it is probable that few new particles form. This stage occurs at about 15–20% conversion in a typical vinyl acetate/acrylate system.

In the *growth stage*, further polymerization takes place in the existing particles being initiated by absorbed free radicals or by small growing chains. No micelles

remain and, of course, no new particles are formed. The monomer droplets act as reservoirs of monomer and disappear towards the end of the polymerization.

When the monomers polymerize, a large amount of heat is generated. The heat of the exotherm is readily dispersed by heat transfer to water. Since the polymer particles are relatively non-interacting, the viscosity remains low up to about 60% solids content. Thus, latex particles can be depicted as having absorbed surfactants on the surface—giving the particle an electric charge: a negative charge in the case of an anionically stabilized latex; a postive charge in the case of a cationically stablized latex and, of course, no charge in the case of a nonionically stabilized latex. The presence of a charge causes an electrostatic repulsion between the particles. The counterions form a diffuse layer around the particles maintaining overall electrical neutrality. Nonionic surfactants can supplement the ionic stabilization by forming a steric barrier. But this barrier is of insufficient thickness to give stability by itself.

Addition of water soluble polymers to ionically stabilized latex can cause flocculation of the particles by simultaneous absorption onto more than one particle, causing poor flow. This causes thickening, but the identity of the particles is not lost (for example, thickening a latex with hydroxyethylcellulose).



(a) Initial reactants

(b) Nucleation stage-free radicals from initiator decomposition begin polymerization. Micelles transform into growing particles

(c) Growth stage – no micelles remain. Particles grow while monomer droplets shrink

(d) Concluding stage – monomer droplets have disappeared. Monomer in particles completes polymerization

Figure 1—Stages of polymerization. (Reprinted from "Modern Surface Coatings," Vol. 1, p. 162, 1983, TAFE Educational Books, Sydney, Australia. Authors: Oil and Colour Chemist Association of Australia)

Effect of Monomer Composition

Some of the monomers used commercially in emulsion polymerization are shown in *Table* 1. They are divided into various classes.

Types of Monomers

Polymers made from VAM, a hard monomer, can show poor water resistance and are liable to saponification. They are low in cost and give good all-around performance, especially when copolymerized with acrylates.

Styrene is another hard monomer. Homopolymer gives hard brittle films at elevated temperatures. Styrene is most useful when plasticized with soft acrylic monomers, making products which have outstanding water and alkali resistance and high gloss.

Methylmethacrylate is another hard monomer. MMA copolymers are highly resistant to water, alkalis, acids, and oils, UV radiation, and degradation due to weathering.

Esters of acrylic acid are examples of soft monomers. The most commonly used are ethylhexylacrylate and butyl acrylate. The longer the side chain, the greater the flexibilizing effect. Ethylene, another soft monomer, imparts good alkali resistance when polymerized with VAM.

Specific monomers are used in small amounts to impart specific properties (e.g., alkali thickenable, reactive with melamine formaldehyde resins, self cross-linking, chalk adhesion, wet adhesion, grain crack resistance, reaction with isocyanates, epoxies, etc.).

Effect of Monomer Composition *On Film Properties*

Minimum film forming temperature is the minimum temperature at which a polymer emulsion will form a continuous film.

For paint use many commercially available latexes have relatively high minimum film forming temperatures (10–15°C). Thus it is necessay to include temporary plasticizers or coalescents such as Texanol® (Eastman Chemical), PM acetate (Arco Chemical), butyl cellosolve (Union Carbide), tributoxy ethyl phosphate, etc.

These plasticizers act by swelling the latex particles much like gasoline swells rubber and makes it sticky and soft.

Effect of Particle Size

The size of latex particles depends on the water phase and the method of manufacture. Fine particle size emulsions have

Table 1—Monomers Used in Emulsion Polymerization

Chemical Type	Hardening	Flexibilizing	Special Effects
Vinyl esters	Vinyl acetate	Vinyl propionate Vinyl versitate	
Acrylic or Methacrylic esters	Methylmethacrylate	Ethyl acrylate Butyl acrylate 2 ethyl hexyl-acrylate Butyl methacrylate	Acrylic acid Glycidyl meth. Acrylate Hydroxyl ethyl Methacrylate Acrylamide Dimethyl amino ethyl methacrylate Acrylamide
Fumaric/Maleic esters		Dibutyl maleate Dioctyl fumarate	
Hydrocarbons	Styrene Vinyl toluene	Butadiene Ethylene Isobutylene	
Miscellaneous	Acrylonitrile Vinyl chloride Vinylidine chloride		

sizes less than 1 micron, while course particle size emulsions have sizes from 1-3 microns.

Particle size and particle size distribution play an important role in the performance characteristics of emulsions and derived products.

Anionic surfactants tend to give finer, more uniform particle size emulsions. However, foaming can be a problem.

Emulsion viscosity—Viscosity is independent of molecular weight, unlike solution polymers such as alkyds. It is affected to some extent by particle size, but composition of the water phase and resin solids content also have a considerable influence.

Gloss—Fine particle size emulsions tend to have better clarity and gloss.

Pigment binding—The finer the particle size of an emulsion, the greater the pigment binding power and, therefore, the higher the critical PVC. This is caused by better packing efficiency. Thus, in pigmented films the smaller latex particles are better able to coalesce around pigment particles. The reduced polarity of VAM/acrylic copolymers leads to finer particle size emulsions (compared with homopolymers). Thus, pigment binding properties are improved.

Freeze/Thaw Stability—Latexes are liable to damage via freezing and subsequent thawing because the surfactants become less efficient. F/T stability is considerably influenced by amount and type of colloids and surfactants present. Finer particle size emulsions tend to have decreased stability (greater surface area).

Film Formation

Latex particles form films by coalescence of the individual particles. As the water evaporates, the resin particles are crowded together and capillary forces overcome repulsive forces. The resin particles come into contact with each other and the polymer particles deform and diffuse. As the particle size becomes smaller these forces become greater. Effective coalescence will not occur below a critical temperature known as the Minimum Film Forming Temperature (MFFT). After particles have coalesced it is several days before final equilibrium is reached, depending on the fate of surfactants.

The presence of solvent (in water phase) has no significant effect on evaporation rate. The evaporation behavior after film formation is influenced by the partitioning of the solvent between the resin and the water phases. Thus, the initial evaporation rate may be enhanced by the presence of solvent but final water loss may well be retarded.

Properties Required of a Latex

A key property required of a latex is chemical stability. The chemical stability of latexes to multivalent ions should be good. Generally, latex containing an ionic surfactant will break when polyvalent ions are added. For this reason, many latexes prepared are based on nonionic surfactants usually with a small amount of anionic present for latex stability.

The next property in importance is mechanical stability. Latex should be able to withstand 10 to 20 minutes in a blender without coagulum formation.

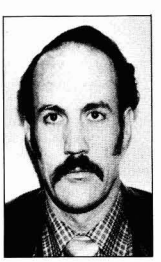
In regard to shelf stability, latex should not rise or fall on viscosity in storage.

The formulation of a freeze/thaw stable latex is difficult. The usual approach is to increase the protective colloid and/or surfactant levels. The addition of such compounds as glycols may also be used. However, these methods involve compromise and usually result in decreased water resistance of the polymer film.

The latex should have low foaming properties, which assist both paint manufacturer and paint application. The latex should be stable over a pH range and the strength of a polymer should be directly proportional to its molecular weight. Therefore, the toughest paint films are formed by latex polymers having high molecular weights. Higher molecular weights also improve the resistance of films to greases, oils, acids, and alkalis. The molecular weight of a latex polymer has a profound effect on the scrub resistance of paint made from the polymer.

Generally, the inherent resistance to weathering in optimum formulations as

DR. BRUCE HENSHAW has held the position of Corporate Director for Research and Development at The McCloskey Corporation since 1982. He has worked for 14 years in the field of polymer chemistry as applied to surface coatings, printing inks and adhesives, and has specialized in the areas of acrylic and modified acrylic solutions and emulsion. He is a co-inventor of five U.S. patents. Dr. Henshaw holds both a B.S. with honors and a Ph.D. in physical organic chemistry from the University of Western Australia. He is a member of The Royal Australian Chemical Institute, American Chemical Society, The Oil and Color Chemists Association of Australia and the Pacific Northwest Society for Coatings Technology.



measured by durability factors decreases in the order MMA, BMA, MMA/BA, MMA/EA. However, polymers based on MMA, BMA, EA, and BA are all classed as inherently durable.

Viscosity is usually measured using a Brookfield Viscometer with a given spindle and rpm speed. However, one should remember that the viscosity of a latex is very dependent upon its method of measurement. The shear rate or spindle speed must be specified. In some cases the sample history also needs to be specified.

The particle size can vary from 0.05 to 3 microns. The actual measurement of latex particles is difficult and can really be best accomplished using electron microscope, or ultra centrifugation techniques.

However, if one visually examines a latex, one can get an idea about the particle size. For example, a small fine particle size latex generally has a bluish tinge. Films formed from such a latex generally are very tightly formed. A large particle size opaque white latex generally gives better flow. However, it is not coalesced as tightly. Generally, for a trade sales latex, a combination of particle sizes is best. This leads to latexes with bimodal and trimodal particle size distribution.

Minimum Film Forming Temperature of the latex obviously determines its application. For example, a gloss latex would need to have a higher MFFT than a latex used in an exterior flat application.

Acid is generally present in most acrylic latex and styrene acrylic latex. It promotes flow and leveling, affects particle size, adhesion, calcium ion stability and freeze/thaw stability. A small amount of acid in the polymer backbone can dramatically affect the viscosity of a latex on neutralization. It also dramatically affects freeze/thaw stability when in neutralized form.

Conclusion

Techniques of emulsion polymerization, as stated at the outset, have advanced dramatically over the years and contributed to the manufacture of tailor-made latexes for specific applications. By understanding the mechanism of polymerization and the key components—as well as the various stages—necessary for the process, paint formulators can enhance product properties and performance. This in turn will result in what we believe will be continued growth in the development and sales of latex paints worldwide.

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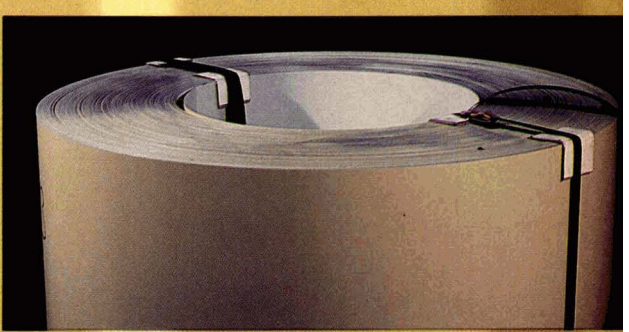

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These subcommittee reports are for the use of the membership of ASTM Committee D-1 in continuing its work and for the interest of readers in the activities of ASTM Committee D-1. The reports are not official actions of ASTM and may not be quoted as representing any action or policy of the American Society for Testing and Materials.

January 1985 Subcommittee Reports Of ASTM Committee D-1

The January meeting of ASTM Committee D-1 on Paint and Related Coatings and Materials was held on January 20–24, 1985 at the Hotel Marriott in Orlando, FL. In the three and one-half days preceding the final session and general meeting of Committee D-1, 125 members and guests met in 148 scheduled meetings of D-1 subcommittees and working task groups. The present membership of Committee D-1 is 489.

Memorial Resolutions were presented on the passing of John H. Calbeck and S. Leonard Davidson.

Special Highlight of the meeting was a seminar on "The Regulatory Process and the Paint Industry," which was held in two, three-hour sessions on Monday and Tuesday. Speakers included J.F. Martonik, Deputy Director, Health Standard Program, OSHA; A.I. Roberts, Associate Director, Office of Hazardous Materials Regulation, DOT; C.M. Jacobson, Compliance Officer, Regulatory Management Division, CPSC; M. Straus, Chief, Waste Identification Branch, EPA; and J.C. Berry, Chief, Chemical Application Section, EPA.

Highlights

The following items of major interest to the coatings industry warrant special emphasis:

Karl Fischer Method for Water Determination—Sub. D01.21.54 has shelved further work on the use of the Hydranal reagent in this method since it was determined that there was no improvement in the method using this reagent. It was found that sample homogeneity or solubility in the solvent used is a primary source of error.

Flocculation—Sub. D01.24.27 is seeking techniques for determining degree of flocculation. Anyone having information on a method that might be evaluated should contact the chairman of Sub. D01.24.

Munsell Color System—Sub. D01.26.02 is revising D 1535, "Specification of Color by the Munsell System," to change the table of luminous reflectance factor and Munsell Value to an absolute basic, rather than the relative to MgO white standard.

New Epoxy Resin Task Group—Sub. D01.33 has formed a new task group which plans to work on a method using head space analysis to determine epichlorohydrin content.

Organo-Tin Release Rates—Sub. D01.45.08 is a new task group established to develop a reliable method for determination of release rates of organo-tins from anti-fouling paints.

Condition Assessment—Sub. D01.46.10 is a new group set up to develop a guide for assessing the condition of the existing paint on a structure, and the condition of the underlying substrate in order to provide a sound basis for maintenance painting.

Zinc-Rich Coatings—Sub. D01.48 has been dissolved and will revert to a task group of Sub. D01.46.

Wood Building Products—Sub. D01.52 has limited its scope to building products and is seeking new members from the Mill Work manufacturers.

Chronic Toxicity Laws—Sub. D01.57 is seeking input on how to organize to handle individual State bills on chronic toxicity.

New Standards

New D-1 standards since the May 1984 meetings of D-1 approved by the ASTM Committee on Standards in the months shown (subcommittee jurisdiction):

JUNE 1984

D 4366-84, "Method for Hardness of Organic Coatings by Pendulum Damping Tests" (Sub. D01.35)

D 4367-84, "Test Method for Benzene in Hydrocarbon Solvents by Gas Chromatography" (Sub. D01.35)

D 4370-84, "Test Methods for Acid and Base Milliequivalent Content of Electrocoat Bath Samples" (Sub. D01.21)

AUGUST 1984

D 4399-84, "Test Method for Measurement of Electrical Conductivity of Electrocoat Baths" (Sub. D01.21)

D 4400-84, "Test Methods for Sag Resistance of Paints Using a Multinotch Applicator" (Sub. D01.42)

OCTOBER 1984

D 4414-84, "Practice for Measurement of Wet Film Thickness of Organic Coatings by Notched Gages" (Sub. D01.23)

NOVEMBER 1984

D 4415-84, "Test Method for Determination of Dimer in Acrylic Acid" (Sub. D01.35)

D 4416-84, "Specification for Acrylic Acid" (Sub. D01.35)

D 4417-84, "Test Method for Field Measurement of Surface Profile of Blast Cleaned Steel" (Sub. D01.45)

JANUARY 1985

D 4449-85, "Method for Visual Evaluation of Gloss Differences Between Surfaces of Similar Appearance" (Sub. D01.26)

D 4450-85, "Test Method for Analysis of Zinc Hydroxy Phosphite Pigment" (Sub. D01.21)

D 4451-85, "Test Method for Pigment Content of Paint by Low Temperature Furnace Ashing" (Sub. D01.21)

Future Meetings

July 7-10, 1985—San Diego, CA (Town & Country Hotel)
January 12-16, 1986—New Orleans, LA (Sheraton New Orleans)
June 15-19, 1986—Philadelphia, PA (Franklin Plaza)

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DIVISION 1 ADMINISTRATIVE

SUBCOMMITTEE D01.06 INTERNATIONAL LIAISON

J.C. Weaver, Chairman

ISO/TC 35 symbolizes Technical Committee TC 35 on Paints and Varnishes, headquartered at NNI in the Netherlands, of the International Standards Organization, headquartered in Geneva, Switzerland. TC 35's secretary, Dr. P.J.H.A.M. van de Leemput recently succeeded Mr. J.A. (Hans) de Wilde, retired, a stalwart member of D-1, widely remembered for his editorial prowess in D-1. TC 35, organized in 1952, is parallel to D-1 in overall scope, in international membership and in diversity of standards developed. TC 35's 158 standards, as of the 1981 classified listing by Mr. de Wilde, continues to be printed in the backs of ASTM Standards Volumes 6.01, 6.02, and 6.03, along with similar listings of test methods of U.S. Federal Standard 141b (141c, 1984 is now issued).

World-wide growth in multinational corporations and in international trade does transcend the historic regional foci of ASTM and ISO, and of the national boundaries. These classic restraints are best circumvented at task group levels, as recognized in ASTM's Committee Regulation 3.3.1. Annual republication of those ASTM Volumes 6 on paints, etc., with those ISO listings included, spread about 10,000 copies world-wide annually with convenient listings of both sets of standards.

Illustrations of commonality: (1) ASTM D 1078 on solvent distillation to dry point, in frequent world-wide use, was adapted into ISO 4626; (2) D 1544 on the 1933 Gardner Color Standards (quantified in the 1970's only in one laboratory in Cleveland, OH) was adapted into ISO 4630; while, (3) ISO 1522 on Koenig and Persoz pendulum hardness was adapted into D 4366-84 after a Sub. D01.23 task group round-robin for precision.

D-1 can participate officially in ISO/TC only via ANSI, the American National Standards Institute, in New York, NY. Two-way passage of voluminous paper flow only via ANSI is too great a burden on top of D-1's large paper flow to be borne by a D-1 volunteer or his, or her, employer in the absence of special funds to pay an ANSI or ASTM staff member, or a volunteer, for handling ISO paint papers. D-1 is, therefore, only an "O" (observer) member, not a "P" (participating) member in the ISO/TC 35.

However, H.E. Ashton, of the National Research Council in Ottawa, Canada, is an able, stalwart participant in both D-1 and TC 35. D-1's "O" membership need not bar or impede direct communication between American or European members of task groups in D-1 and TC 35 for earlier, parallel development or revision of standards in their respective editorial and style formats. Any D-1 member may communicate on these mutual interests directly with the TC 35 secretary, Dr. P.J.H.A.M. van de Leemput, Nederlands Normalisatie-instituut, Kalfjeslaan 2, P.O. Box 5029, 2600 GB Delft, Netherlands, concerning any of the working groups in the following TC 35 organization list.

ISO/TC 35

SCOPE: Standardization in the field of paints, varnishes and related products including raw materials.

SC 1—Terminology
SC 2—Pigments and extenders—DIN (West Germany)
WG 1—Chromate and phosphate pigments
WG 3—Chrome oxide green pigments
WG 4—Cadmium pigments
WG 5—Aluminum pigments
WG 6—Solubility of dyestuffs
WG 7—Dispersability
WG 8—Zinc dust pigments
SC 9—General test methods for paints and varnishes—BSI (UK)
WG 1—Accelerated weathering
WG 2—Humidity
WG 3—Drying time
WG 4—Hiding power
WG 6—Gloss
WG 7—Indentation
WG 8—Rapid deformation
WG 11—Evaluation of degradation of painted surfaces
WG 14—Toxic content
WG 15—Fire retardance
WG 16—Powder coatings
WG 17—Instrumental color measurement
WG 18—Salt spray corrosion tests
WG 19—Washability
WG 20—Abbreviated sampling procedures
SC 10—Test methods for binders for paints & varnishes—W. Germany
SC 12—Preparation of steel substrates before painting—UK
WG 1—Surface profile
WG 2—Surface cleanliness
WG 3—Surface preparation methods
SC 13—Drying oils—BCS (Sri Lanka)

SUBCOMMITTEE D01.08 ENVIRONMENTAL CONCERNS

J.J. Brezinski, Chairman

A proposed scope was approved for submission to Sub. D01.90 as follows: "To assess the need for, recommend the initiation of, and coordinate the timely development and effective utilization of test methods and standards relating to all environmental matters pertaining to current and potential federal and state regulations to insure that methods of compliance and the impact on the regulated community are understood. Compatibility with ASTM Society will be effected through association with the Environmental Coordinating Committee."

Accountabilities were assigned to assess the need for new test methods and standards at appropriate federal and state regulatory bodies. These include:

Air Quality—E.A. Praschan, H. Fujimoto
Water Quality—D.O. Lawson
Solid Waste—R.J. Connor
Toxic Substances, General—J.J. Brezinski

J.J. Brezinski will represent the subcommittee in relations with the Environmental Coordinating Committee.

SUBCOMMITTEE D01.13 CONSUMER AFFAIRS

R.S. Shane, Chairman

The subcommittee met on October 12, 1984 at ASTM Headquarters. Members of Committee F-15 on Consumer Product Standards and of the Delaware Valley Consumer Sounding Board were present. Of special note was the personal input of Mrs. Lee Gray, Chairman of F-15's Subcommittee on Consumer Participation and Director of the Office of Consumer Affairs of the U.S. Dept. of Commerce.

The chief issue that emerged was a strong desire for informative labeling of architectural paints, both by the Government and the consumers present. A proposed draft standard was submitted by W.V. Moseley, Jr. It was voted to submit this draft to Sub. D01.90 with a recommendation that it be referred to an appropriate standards-writing subcommittee with the assurance of help from the Sub. D01.13 and F-15. This recommendation has been referred to Sub. D01.42 by the D-1 chairman.

Future meetings of Sub. D01.13 will be held once a year with F-15 and twice a year with D-1. The next meeting will be in San Diego in July, 1985.

At the D-1 meeting in January, Moseley announced that the State of Virginia is withdrawing its paint labeling suggestions from ASTM.

SUBCOMMITTEE D01.15 LECTURES & SYMPOSIA

H.A. Wray, Chairman

As a result of a discussion on future committee programs, a symposium was approved to be held in June of 1987 in Michigan, in conjunction with the D-1 meeting, on Automotive Finishing and Refinishing. It was also discussed that there may be a possibility of a tour of some automotive facility. Another idea proposed was to canvass the committee for ideas for a seminar to cover standards affiliations in the coatings industry, both new and old. In addition, volunteers will be requested to prepare and present a talk on their suggested idea. Thus the seminar will present topics of interest to the committee.

Special thanks were noted to the individuals who presented material at the seminar on "The Regulatory Process and the Paint Industry."

SUBCOMMITTEE D01.18 EDITORIAL

H.E. Ashton, Chairman

D01.18.02, Indexing, S. LeSota, Chairman, met with D. Savini of the ASTM Editorial staff present, to discuss indexing of the ASTM *Book of Standards.* All members are recording proposed changes on 3 x 5 index cards. These cards will be organized, edited, and submitted to H. Shupak at Headquarters every six months.

SUBCOMMITTEE D01.94 AWARDS & MEMORIALS

H.K. Hammond III, Chairman

A candidate has been chosen for the 1985 Gardner Award. A candidate had still not been selected for the Pearce Award at meeting time, but a tentative one was under consideration.

No candidates had been received for the Certificates of Appreciation at meeting time, but several were submitted later. For this award a brief statement must be submitted by a subcommittee chairman supporting each nomination and giving the name and address of the candidates company superior.

Two D-1 members have been selected by the Society Award of Merit Committee to receive this award, but since these persons had not as yet been officially notified their identities were withheld.

Two D-1 members, John H. Calbeck and S. Leonard Davidson, are known to have passed away since the last meeting. Memorial resolutions on their passing were prepared for reading at the D-1 meeting.

DIVISION 20 RESEARCH AND GENERAL METHODS

SUBCOMMITTEE D01.20 SAMPLING, STATISTICS, ETC.

H.E. Ashton, Chairman

The chairman reported that the corrected copy of D 3980 had been sent to ASTM Headquarters and it should appear in the 1985 *Book of Standards.* A revision to accommodate changes previously discussed will be submitted to letter ballot. Since definitions will not be part of the revision, this part of D 3980 will be sent to the Committee on Terminology. C.J. Sherman said that he had not made any progress in preparing a computer program based on D 3980 because computer time was not available. However, a new user-friendly computer has been purchased by his employer and, with the assistance of his son, he hoped to have a program ready for the next meeting. The chairman will contact the ASTM vice-president for information resources to establish how this computer program could be made available through ASTM.

The chairman will review Specification D 3924 and Practice D 3925 and, if they are acceptable, will submit them to concurrent ballot for reapproval. It was agreed that the D-1 rating practice, now in the D-1 Handbook, will be put in suitable format for inclusion in the *Book of Standards* and submitted to ballot.

J.C. Weaver mentioned that accreditation of paint testing laboratories might become a subject for consideration by Sub. D01.20.

SUBCOMMITTEE D01.21 CHEMICAL ANALYSIS OF PAINTS AND PAINT MATERIALS

R.W. Scott, Chairman

D01.21.13, Coordination of VOC Standards and Information, J.C. Weaver, Chairman, reported now that D 3960, "Practice for Determining Volatile Organic Content (VOC) of Paints and Related Coatings," has been revised, the balloting should be expedited as soon as possible to get the method in the 1986 ASTM *Book of Standards.* During 1985 there will be more changes required. These will be made and will be incorporated into D 3960 as needed.

EPA personnel participation is needed in Sub. D01.21 to generate a symbiotic interplay to determine what type of test methods are needed, and how this subcommittee with its experts can address the needs of the government regulatory bodies and industry.

Besides Sub. D01.08 on Environmental Concerns, there is an ASTM Coordinating Committee on Environmental Matters which has been reformulated annually. This committee decided it had to revise its mission again in how it relates to 130 other ASTM Committees and especially to D-19 (water) and D-22 (air), and others. This committee now is eager to approach the EPA from top to bottom, so that EPA and ASTM coordinate better. This fits in with comments made by EPA personnel that ASTM should go higher in the EPA hierarchy if it wants EPA personnel on its committees. Apparently other ASTM committees such as D-19 and D-22 are being accommodated with heavy EPA representation. D-1 has not done its homework. Plans are to work through this ASTM committee.

The use of "affiliate" membership in D-1 for EPA personnel was discussed. Since EPA travel funds are limited, it was suggested Sub. D01.21 hold an interim meeting at Research Triangle Park, NC, on subjects of current interest, such as transfer efficiency, volume nonvolatile, VOC precision statements, determining water in coatings, etc.

It is the goal of Sub. D01.08 to generate a better relationship with EPA and other government regulatory hierarchy. For this reason it was suggested that, in place of Sub. D01.21, Sub. D01.08 should approach the US-EPA at Research Triangle Park first to ascertain what is going on and what interest is prevalent now and in the very near future.

D01.21.22, Analysis of Electrocoat Bath Samples, W.B. Van DerLinde, Chairman, reported that two test methods, D 4370, "Test Method for Acid and Base Content of Electrocoat Baths," and D 4399, "Test Method for Measurement of Electrical Conductivity of Electrocoat Baths," have been approved as standards.

The test method to determine the pH of electrocoat baths and the one to determine the nonvolatile and pigment content of electrocoat baths are ready for Society ballot.

Round-robins will be initiated immediately for the new procedures for solvent determination and ionic contamination determination of electrocoat baths. Samples of commercial electrocoat paints will be used for these round-robins. The samples will consist of two cationic and two anionic electrocoat paints. For the solvent determination, the samples may need spiking with additional solvents to cover the range of solvents commonly used in electrocoat paints.

Ionic contaminations can be added to these paints for their determination. For practical reasons, the ionic contaminations should be separated into cationic and anionic determinations. The cations can be determined by an atomic absorption method, while individual methods can be used for each anionic contaminant. Ion chromatography is an alternate method of analysis for ionic contaminants. The possibilities will be explored for the next meeting.

D01.21.23, Thermoplastic Traffic Marking, J. O'Brien, Sr., Chairman, reported that the round-robin for the chemical analysis of thermoplastic traffic markings has been initiated. As yet, no results are available.

As a suggestion to expedite future round-robins, it was proposed that the physical and chemical tests be separated and run as separate entities. Each test method could be developed individually and then all combined into a "Recommended Practice." This is the path being taken by Sub. D01.21.22 for its electrocoat bath samples. In addition, the collaborators would not be burdened with a whole series of physical and chemical tests to be run on numerous samples as presently done. This matter will be discussed at the next meeting. The collaborators will be contacted immediately to determine whether the total round-robin can be completed by the next meeting. H. Fujimoto (Inmont), H.D. Swafford (Glidden), and R.W. Scott (Sherwin-Williams) stated they would be interested in participating in the chemical analysis portion of future round-robins.

D01.21.42, AAS Analysis of Pigment, W.V. Moseley, Jr., Chairman, reported that "Standard Method of Test for the Determination of Titanium Dioxide Content of Pigment Recovered from Whole Paint by Atomic Absorption Spectrometry" received a negative in the last concurrent ballot, which was found to be editorial and was withdrawn. The method will be revised to reflect A.M. Snider's comments and those regarding Para. ↓1.4. It was felt that his comments on Para. 11.5, in which he questioned what to do after the sample is filtered, is adequately answered in the first part of the sentence.

When this method was balloted, the "Precision and Bias" statements were inadvertently left out. A precision statement generated by H.E. Ashton was found acceptable and will be incorporated, along with some editorial changes, into the revised method which will then be submitted for a second D-1 ballot.

D01.21.46, X-Ray Analysis of Pigment, A.M. Snider, Jr., Chairman, focused discussions on two methods which have undergone round-robin investigation. One method is "Standard Method for Titanium Dioxide in Paint by X-Ray Fluorescence (XRF)." The other is "Standard Method for Pigment Identification and Quantitative Determination in Paint Films by X-Ray Diffraction (XRD)."

Average observed titanium dioxide content for each of four paint samples reported by three laboratories which participated in the XRF round-robin, was discussed along with the between lab statistics. The lack of results from duplicate individual runs by the same operators prevented the calculation of single operator, or within laboratory, precision. H. Fujimoto will attempt to locate detailed individual data from the participants. If the detailed data can be located, the chairman will determine the needed precision statements. If the data is inadequate, another round-robin will be initiated with fresh paint samples.

The data collected from the four labs which participated in a round-robin for a general XRD method show an undesirable spread. Results were pretty well scattered. Comments received will be reviewed by the chairman in an attempt to determine how the method can be refined further and the procedure made more explicit. After revision, another round-robin will be initiated. Collaborators with X-Ray equipment are needed. Contact A.M. Snider at 412-487-4500.

D01.21.47, Analysis of Calcium Boro Silicate, J. Austin, Chairman, reported that a new proposed standard, "Analysis of Calcium Boro Silicate (now D 4487)," passed a concurrent ballot with no negatives. Comments received from J. Fetsko on the need for a "Significance and Use" statement and from H.A. Wray on the need to use a capital "L" when referring to Litres, will be added to the standard as soon as possible.

D01.21.48, Analysis of Zinc Hydroxy Phosphite, L. DiCarlo, Chairman, reviewed the new standard method for this analysis (D 4450) and agreed that no further revisions were required. It will appear in the 1985 *Book of Standards.*

D01.21.53, Trace Levels of Monomers in Paints, J. Benga, Chairman, discussed comments and corrections received as a result of a Sub. D01.21 ballot at the meeting in Montreal in May. Now that the revised method with the recalculated precision statements is ready for D-1 ballot, it will be expedited as soon as possible.

It was agreed that there is a need for a method to determine trace isocyanates. An HPLC method is being revised into ASTM format. The first draft should be ready for review and round-robin testing shortly. The area of concentration will be below 1000 ppm. Commercial samples will be obtained from Mobay and/or Spencer Kellogg. J.J. Brezinski will call Dr. Foss at Spencer Kellogg to obtain samples. Methods were solicited for determining other isocyanates, such as methyl isocyanate and hexamethylene diisocyanate.

Mr. Brezinski stated that government regulations may require the determination of low levels (ppm) of toxic compounds such as formaldehyde and vinyl chloride monomers. He recommended that Sub. D01.21 develop suitable methods to fill these needs immediately.

The method to analyze for free formaldehyde is already under study by J.H. Smith in Sub. D01.33 (Varnish and Resins . . .). Should trace amounts of formaldehyde be developed under the resin group or under Sub. D01.21? It was felt that Sub. D01.21's interest would be to look at free formaldehyde in paint products and not in the pure amine-formaldehyde type resin.

D01.21.54, Revision of D 4017, Water in Paints by Karl Fischer Titration Method, K. Leavell, Chairman, reviewed and discussed precision statements developed from the data obtained from the first round-robin using Hydranal reagent on three paint systems (water-based can coating, architectural trade sales paint, and an electrocoat product). Analysis of the laboratories' results indicated the data obtained was of equal precision to previous regular Karl Fischer (KF) titration results.

A second round-robin was initiated in an attempt to refine the method. Samples tested for their water content were a water-based can coating, a water-reducible automotive topcoat and an architectural trade sales paint. Examination of the data shows two outliers which appear to indicate significant problems using the Hydranal reagent. Sample homogeneity, or solubility in the solvent (methanol) used, is a prime source of error. Since the Hydranal reagent is not an improvement over the KF reagent, no further work will be done by this task group. A report on the round-robin study will be written and footnoted in D 4017.

D01.21.55, Halohydrocarbons in Paints, C. Niemi, Chairman, met to resolve the one D-1 ballot negative and four sets of comments received on a concurrent ballot (D0102 84-2). Attempts to contact the individuals who submitted the negative and comments were unsuccessful. It was decided to discuss the negative and to take any appropriate and necessary action to expedite the method's inclusion as an ASTM Standard.

The negative from F.M. Raffo, considering all the facts, was found to be not persuasive by a vote of 17-0-0. The negative voter will be sent an explanation of the action taken.

Comments from J.J. Brezinski, A.H. Landrock, F.M. Raffo, and H.E. Ashton will be incorporated into the method where appropriate. It will then be submitted to Society ballot. The method is designated D 4457.

D01.21.56, Revision of D 3960-81, Standard Practice for Determining VOC of Paints and Related Coatings, M.E. Sites, Chairman, reviewed a revised copy of D 3960 and made a number of changes and additions, mainly editorial, to the document. This revised method will be submitted to concurrent Sub. D01.21/D-1 ballot.

The possibility of including a precision statement, which was generated in two round-robins, in calculating the VOC of solvent-reducible and water-reducible coatings, was discussed. It was decided to leave it out since Standard Practices do not require precision statements. The question of whether a precision statement could be added as an appendix will be investigated.

The intent to reference the Hydranal method for determining water in paints as a means to improve precision on water-reducible coatings has been shelved since round-robin results with Hydranal showed poorer precision than those obtained with D 4017 and D 3792.

D01.21.80, Exploratory Analytical Research, K. Leavell, Chairman, covered several topics as follows:

(1) Quantachrome's gas pycnometer for determining volume nonvolatile is being used by one member's lab. The only inconvenience reported was that a fairly large sample, 3–5 grams, of dried film is required. Film density tends to be on the low side, but the speed with which the result is obtained (minutes) is superior to the two to three hours required with D 2697. At the next meeting, a comparison will be made vs the conventional stainless steel disk method.

(2) Interest was generated in running anions and cations in electrocoat ultrafiltrate samples using ion chromatography. W.B. Van DerLinde will initiate this activity in Sub. D01.21.22. Four labs volunteered to participate in the round-robin.

(3) FTIR was discussed as a method for running hydroxyl numbers of resins. The instrument has been found useful for running fairly high hydroxyl numbers, greater than 50, with improving accuracy the higher the hydroxyl number. Very low hydroxyl numbers are rarely practical by infrared.

(4) The Coulter's N-4 Particle Size Analyzer has been found useful for latices, NAD's, and some pigment dispersions. Until more members acquire the instrument, round-robin testing is not feasible.

(5) R.W. Scott brought to the attention of the members the Quintel Corp.'s Computrac Moisture Analyzer which automatically measures loss-on-drying; it automatically calculates, calibrates and weighs. Since the instrument appears oriented to measure moisture loss using an open coil heater, the members felt it would be dangerous to use on solvent-reducible coatings. In addition, only one sample could be run at one time.

SUBCOMMITTEE D01.22 HEALTH AND SAFETY

H.A. Wray, Chairman

D01.22.01 on Precautionary Statements reported that the proposed D-1 Policy, "Precautionary Hazard Statement Use in ASTM D-1 Documents and Publications," had passed a satisfactory ballot with the resolution of the negative. The task group recommended that negative ballot be declared not persuasive.

The subcommittee agreed with the task group by unanimous vote (7-0-0). The task group also proposed and the subcommittee agreed that the title of this document should be changed to a "Guide" and that it be submitted to a concurrent Sub. D01.22/D-1 ballot.

The subcommittee approved a revision of D 1310, "Test Method for Flash Point of Liquids by Tag Open-Cup Apparatus," to correct an error in the cup. The corrected method will be submitted to concurrent Sub. D01.22/D-1 ballot.

It was agreed that D 3630, "Practice for Determining Constituents Classified as Hazardous Contained in Protective Coatings," be submitted to both Sub. D01.21 and Sub. D01.22 to determine its usefulness and need for revision.

Likewise, the "Bibliography of Health and Safety Related Items" now appearing in the D-1 Handbook will be submitted to D-1 to determine its usefulness and need for revision and whether it should be made into a Guide Standard maintained in the Handbook or withdrawn from the Handbook.

The ASTM Coordinating Committee for Flash Point and Related Properties (CCFP) recommended that D 3278, "Test

Method for Flash Point of Liquids by Setaflash Closed-Cup Apparatus," be revised to include n-butanol as a reference standard for flash point at a temperature approximately the classification point for flammable liquids. A concurrent Sub. D01.22/D-1 ballot will be conducted to approve the inclusion of this reference standard in D 3278. The Coordinating Committee is studying additional reference standards for this method.

Sub. D01.22 members will cooperate with the CCFP in a round-robin to check the precision of D 4206, "Sustained Burning Test by the Setaflash Open-Cup Apparatus."

The chairman reported that the CCFP was working on a flash point manual, temperature devices to be used in the Setaflash to replace mercury in glass thermometers in this instrument, and to furnish additional round-robin results for submittal to DOT for use of D 4206 in regulations.

SUBCOMMITTEE D01.23 PHYSICAL PROPERTIES OF APPLIED PAINT FILMS

M.P. Morse, Chairman

D 823, "Application of Uniform Films on Test Panels," is being revised by changing the apparatus section of Method B to permit the use of any dipcoater meeting the range of withdrawal rates specified. The sources of two commercially available dipcoaters will also be given.

D01.23.10 on Adhesion, H.E. Ashton, Chairman, is developing a cross-hatch/tape test suitable for testing thick films. The use of a 2-3 mil multi-blade cutter is being investigated, as is the use of a template/utility knife procedure. Some film tearing has been experienced with these techniques and some procedural changes are being made to minimize this problem. D 2197, "Adhesion of Organic Coatings," is being revised by eliminating the use of the Arco Microknife, which is no longer manufactured.

D01.23.11 on Wet Film Thickness, H.A. Ball, Chairman, has made some revisions to D 1212, "Wet Film Thickness Measurements with the Interchemical and Pfund Gages," to clarify some terminology. The revision will be submitted to D-1 letter ballot.

D01.23.14 on Hardness, Mar and Abrasion Resistance, M.P. Morse, Chairman, has revised D 1474, "Indentation Hardness," to qualify statements made in the Significance and Use section. This will be submitted to D-1 ballot. A questionnaire will be distributed to D-1 members to determine how mar resistance of coatings is defined and how it is presently being measured. Depending upon the information received, the group will initiate a program to develop an instrumental method for measuring mar resistance.

D01.23.15 on Slip Resistance, M.P. Morse, Chairman, has drafted a new method for measuring surface static friction of coatings that will be submitted to D-1 letter ballot. An interlaboratory test will be conducted to determine the precision of the method. Future activities will include the development of a method for measuring dynamic static friction of coating surfaces.

D01.23.16 on Water Vapor Permeability, M.P. Morse, Chairman, has made a major revision of D 1653, "Moisture Vapor Permeability of Organic Coating Films," by providing procedures for wet and dry cup determinations under several environmental conditions. This revision will be submitted to D-1 letter ballot.

D01.23.18 on Flexibility, M.P. Morse, Chairman, has made some minor revisions in D 1737, "Elongation of Coatings by Cylindrical Mandrel," and in D 522, "Elongation of Coatings by Conical Mandrel." These will be submitted for D-1 letter ballot. As a future activity, these two methods will be combined into one method that will be titled, "Flexibility of Organic Coatings by Mandrel Tests."

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

C.K. Schoff, Chairman

D01.24.19, Viscosity by Efflux Cups, C.K. Schoff, Chairman, discussed D 4212, "Viscosity by Dip Type Cups." The method has been broken into two separate documents, one for Zahn cups, the other for Shell cups. After slight modification, the methods will be submitted for Sub. D01.24 ballot and circulated to Sub. D01.56 on Printing Inks, because of their interest in these cups. D 4212 will be balloted for withdrawal on condition that the two methods will replace it. After these have cleared Sub. D01.24 ballot they will be submitted to D-1 ballot as a group.

It was decided that a new round-robin for precision data was necessary for the Zahn cup method. The present statement is based on data taken with a single set of cups that were circulated to the cooperators. Therefore, there is no true reproducibility data and the value for reproducibility in the precision statement is much better (lower) than it should be. The new round-robin will involve testing of three paints and two inks with the No. 2 and 4 Zahn cups (the most commonly used). For the tests it is hoped that new cups can be borrowed from the Gardner-Neotech Div. of Pacific Scientific and from Paul N. Gardner Co.

D01.24.20A, Low Shear Viscosity, discussed the proposed method for determination of viscosity at low shear rates by the relaxation technique. It was decided to prepare the draft for Sub. D01.24 ballot after determining whether the computer program from the recent publication on the relaxation technique can be made public and placed on file at ASTM Headquarters. It also was suggested that the draft be sent to Mr. Temple Patton for review since the original idea for the technique and the original publication were both his. His template method will also be included.

D01.24.20B, D 2196 Viscosity by Brookfield, D. Howard, Chairman, discussed the completed draft of the new precision statements for D 2196 and the need for reapproval or revision of the method (due in 1986). Suggestions were made for corrections and additions to the precision statements. It was decided to submit D 2196 and the revised precision statements for concurrent Sub. D01.24 and D-1 balloting.

D01.24.26, Electrical Properties of Liquid Paints and Paint Materials (Including Solvents), heard that the chairman had resigned and the outline for the method on "Measurement of Electrical Resistance of Liquid Paint and Paint Materials" had not progressed any further. However, the Ransburg Corp. has shown considerable interest in becoming involved and should be able to help considerably, both from the standpoint of the proposed method and for providing information on the best use of the Ransburg Tester, the most common device for measuring resistance in the paint industry. It is hoped to have a draft for the d.c. method and more information on testing in time for the San Diego meeting in July. A number of companies and individuals have expressed interest in this work and wish to cooperate in any interlaboratory testing.

D01.24.27, Flocculation, discussed the slightly modified outline of the proposed practice for testing the degree of flocculation. Since there appears to be a real

need for such a practice, work will continue. The chairman will do a literature search for techniques for determining degree of flocculation and will carry out the previously promised survey of D-1 members for interest in a flocculation practice and include a request for information on techniques currently used by D-1 members. Individuals in the group will review articles in the literature and methods for evaluating *liquid* paint, ink and paste specimens for flocculation will be discussed at the July meeting in San Diego. Techniques for liquid specimens will be given initial emphasis because of the lack of ASTM methods or other published information in this area. More methods exist in the area of appearance of dry films (tint strength, hiding, gloss, color/color difference, etc.).

D01.24.28, Evaporation Rates, reviewed D 3538, "Evaporation Rates of Volatile Liquids by Shell Thin Film Evaporometer," which is up for reapproval in 1986. The fact that a new automatic evaporometer was to be unveiled later this year by Quintel Corp., of Tempe, AZ, was mentioned. The chairman will have an opportunity to test a prototype of the instrument and will report his findings. There is a good chance that this new evaporometer will be added to D 3539 and the title of the method changed to cover use of evaporometers in general. This is a future project, however, and D 3539 will be submitted for reapproval this year with minimal changes other than the addition of a Significance and Use section.

SUBCOMMITTEE D01.26 OPTICAL PROPERTIES

C.J. Sherman, Chairman

D01.26.02, Color Measurement, C.S. McCamy, Chairman, discussed thoroughly a revised draft of D 3134, "Selecting and Defining Color and Gloss Tolerances of Opaque Surfaces and Evaluating Conformance." Changes will be incorporated and a new draft will be balloted by Sub. D01.26.

As a result of a Sub. D01.26 ballot on D 2244, "Instrumental Evaluation of Color Difference of Opaque Materials," comments received will be included in a revision prior to submitting to D-1 ballot.

D 1535, "Specification of Color by the Munsell System," will be revised to change the table of luminous reflectance factor and Munsell Value to an absolute basic, rather than relative to magnesium oxide white standard. The revision will be submitted to a concurrent Sub. D01.26/D-1 ballot.

D01.26.06, Hiding Power, L. Schaeffer, Chairman, will revise D 2805, "Hiding Power of Paints," as a result of comments received on a Sub. D01.26 ballot. It will then be submitted for concurrent Sub. D01.26/D-1 ballot.

D01.26.11, Gloss and Goniophotometry, M.P. Morse, Chairman, reported that a negative and several editorial comments were received on the latest ballot on D 523, "Specular Gloss." The portion of the negative concerning the name of the method was found not persuasive, but the portion dealing with the definition of specular gloss and luminous reflectance factor was persuasive. A new draft incorporating the changes agreed upon will be submitted to concurrent Sub. D01.26/D-1 ballot.

D01.26.23, Reflectance of Horizontal Coatings, N. Johnson, Chairman, will submit for concurrent Sub. D01.26/D-1 ballot a revision of D 4061, "Retroreflectance of Horizontal Coatings," with updated terminology and reference. A new round-robin with defined sample holder and entrance angle setting techniques has been proposed. Participants are solicited.

D01.26.24, Tinting Strength, C.J. Sherman, Chairman, reported that a negative and several editorial comments were received on the balloting of D 332, "Tinting Strength of White Pigments." The portion of the negative dealing with the equation to calculate tinting strength was found to be not persuasive, but the other portions were persuasive. A new draft incorporating these comments will be submitted to a concurrent Sub. D01.26/D-1 ballot.

D 2745, "Instrumental Tinting Strength of White Pigments," received two negatives and several editorial comments. The portion concerning the equation to calculate tinting strength was found not persuasive, but the rest was persuasive. A revision including these changes will be submitted to concurrent Sub. D01.26/D-1 ballot.

D 3964, "Standard Practice for the Selection of Coating Specimens and their Preparation for Appearance Measurement," will be submitted for concurrent Sub. D01.26/D-1 ballot for reapproval.

SUBCOMMITTEE D01.27 ACCELERATED TESTS FOR PROTECTIVE COATINGS

E.A. Praschan, Chairman

On the latest D-1 ballot a revision of D 659, "Evaluating Degree of Chalking of Exterior Paints," received six negatives. Three of these were found to be, in part, not persuasive by a vote of 9-0-0. However, because of the substantial number of recommended editorial changes in the other negatives (found to be persuasive) and comments on this item, it will be revised and reballoted at the D-1 level. The negative voters will be contacted to explain this action.

D01.27.02, Water Tests, G.W. Grossman, Chairman, discussed the results of the recent Sub. D01.27 ballot on revisions of four water methods (D 870, D 1735, D 2247, and D 2366). For the most part the negatives and comments received on these proposed revisions were found persuasive. The chairman will revise these standards for reballoting by Sub. D01.27. In the case of D 2366, "Accelerated Testing of Moisture Blister Resistance of Exterior House Paints on Wood," the revisions proposed on the ballot were very extensive. It was felt that it would be better to propose a new and broader practice for condensation testing, which would include D 2366 and lead to the eventual withdrawal of the current D 2366.

D01.27.04, Light and Water Exposure Apparatus, S.M. Totty, Chairman, discussed two negatives and several comments received on the 84-1 Subcommittee ballot on the proposed standard practice for Conducting Tests on Paint using Fluorescent UV-Condensation Apparatus. Most of the comments received will be included in the proposed practice before it is submitted to D-1 ballot. One negative had been withdrawn previously. The other by B. Appleman was found, in part, to be not persuasive by a vote of 9-0-0 in the Sub. D01.27 meeting. Dr. Appleman will be contacted to explain the reason for the rejection.

D01.27.09, Corrosion, R.L. Williamson, Chairman, discussed work done previously by SAE on Scab corrosion cycle testing. It was agreed that the group will conduct a round-robin on Scab corrosion cycles using automotive systems. In addition to laboratory cycle tests the system will be exposed to various Florida methods used to produce this corrosion as well as "on vehicle" and Arizona proving ground tests. It is planned to have the laboratory cycle test results and a first draft of the proposed standard completed in time for the July 1985 meeting.

D01.27.10, Accelerated Outdoor Weathering, M.P. Morse, Chairman, reported that the exposure study of 12 automotive coatings comparing EMMA n.t.w. (night time wetting) at DSET Laboratories in Arizona to Florida's 5

degree S. Black Box is underway. The study also includes a modified EMMA n.t.w. cycle (higher panel temperatures), 5 degrees S. plywood-backed rack and 5 degrees S. open rack exposures. Since one year Florida results will not be available until after the July meeting the group will not meet until the January 1986 meeting.

D01.27.14, Filiform Corrosion. F.W. Lutze, Chairman, is attempting to determine if the formation of filiform corrosion can be accelerated over the rate obtained by the current method D 2803, by increasing the temperature of the humidity cabinet. General Motors and Parker will conduct some screening tests to evaluate the effect of several variables prior to starting round-robin testing.

D01.27.16, Chalking, J.S. Robbins, Chairman, discussed the results of the committee ballot on a revision of D 659. The summary of this meeting is given earlier at the start of these minutes.

D01.27.17, Evaluation of Weathering Effects, A.S. Allen, Chairman. The latest draft of a revision of D 660, "Evaluation of Checking," was reviewed by the chairman. Based on comments received during the discussion, the revision will be rewritten and be presented at the July meeting before submitting it for D-1 ballot.

E.A. Praschan announced he will be stepping down as Sub. D01.27 chairman and that R.L. Williamson, of Parker Company, will be the new chairman.

SUBCOMMITTEE D01.28 BIODETERIORATION

D.L. Campbell, Chairman

D01.28.01, Package Stability, W.B. Woods, Chairman, discussed the revision of D 2574, "Resistance of Emulsion Paints in the Container to Attack by Microorganisms." A round-robin will begin to develop data for the "Presumptive Challenge Test of Preservatives to Prevent Microbial Spoilage of Paints in the Container."

D01.28.02, Rapid Determination of Enzyme Presence, D.P. Leipold, Chairman, reported that results of the last round-robin with four cooperators showed a reasonably reproducible pattern which may lead to a satisfactory method.

Prior to the last round-robin it will be determined whether a non-solvent such as propylene glycol can be used to provide good dispersion of the CMC 4H1 without effecting enzyme activity.

In the next round-robin the conditions during dispersion and the elapsed time between sample preparation and viscosity measurement will be controlled closely. A new sample of the CMC 4H1 will be provided. Two paints will be tested, a vinyl acrylic at pH 7.5 and an acrylic at pH 9.0.

The potential value of this procedure for determination of very low levels of enzyme in raw materials was discussed.

D01.28.04, Resistance of Paint Films to Attack by Algae, W.B. Woods, Chairman, reported that Nuodex and Vanderbilt have expressed a willingness to participate in future round-robins. A.R. MacGillivray suggested that the scope of the task group be expanded to include algae and lichens.

D01.28.05, Recoating Mildewed Surfaces, D.L. Campbell, Chairman, reported that Draft 6 of the new "Standard Guide for Determining the Presence of and Removing Microbial (Fungal or Algal) Growth on Paint and Related Coatings" will be balloted by Sub. D01.28 members in early 1985.

DIVISION 30 PAINT MATERIALS

SUBCOMMITTEE D01.31 PIGMENT SPECIFICATIONS

C.W. Fuller, Chairman

An editorial comment by K. Hancock on the Zinc Hydroxy Phosphite specification was accepted. A negative by W.C. Johnson was voted not persuasive (7-1-2), because it was felt that it was not necessary to include pH, soluble salt level, and buffering action in order to describe a pigment.

The standards published since May 1984 were reviewed. It was decided that D 719, the analysis of D 604, should be left in the Applicable Document section of D 604, "Specification for Diatomaceous Silica Pigment."

Editorial comments by H.E. Ashton and A. Landrock on D 83, "Specification for Red Lead," were accepted, as was an editorial comment on D 475, "Specification for Pure Para Red Toner." D 3720 was added to D 476, "Specification for Titanium Dioxide Pigments," as an applicable document.

An editorial correction by A. Eickhoff pointed out that D 1028 in D 604 should be D 1228. It was agreed that D 604 should remain singular.

The specs from 1984 and 1985 will be mailed to the membership for comments to be returned to C.W. Fuller with a copy to D.H. Ruddick.

D 1199, "Specification for Calcium Carbonate Pigments," will be submitted in modified form by R. Roland to reflect the addition of a Type VI.

W.C. Johnson will draft a standard practice for selecting pigments for use in anticorrosive primers.

SUBCOMMITTEE D01.33 VARNISH & RESINS, INCLUDING SHELLAC

C.M. Winchester, Chairman Pro Tem

The subcommittee expressed regret on the resignation of Chairman A.C. Abbott, Jr. and appreciation for his years of service to the subcommittee.

The formation of a new task group for Epoxy Resins was announced. It will be chaired by Robert A. Orr, of Dow Chemical. It is planned to work on a new method using head space analysis to determine epichlorohydrin content of epoxy resins. D 1652, "Method for Determination of Epoxy Equivalent Weight in Epoxy Resin," and D 1726, "Method for Determination of Hydrolyzable Chlorides in Epoxy Resins," also will be revised.

Task group chairmen are still needed for urethane, alkyd and emulsion groups.

D01.33.01, Varnish, H.A. Ball, Chairman, reported that the editorial comments from the concurrent Sub. D01.33/D-1 ballot on Draft #4, D 154, "Guide to Testing Varnishes," had been incorporated. D 154, as revised, will be submitted to Society ballot.

D01.33.23, Phenolic Resins, H.D. Marshall, Chairman, reported that an informal ballot had been conducted of task group members and industry representatives on a proposed Guide for Testing Phenolic Resins and 10 new test methods. Comments received will be incorporated and second drafts will be balloted by Sub. D01.33 before the July meeting.

D01.33.24, Nitrogen Resins, J.H. Smith, Chairman, reported that laboratory testing of a proposed method for determination of free formaldehyde in nitrogen resins was underway. When the procedure has been optimized a round-robin will be organized.

D01.33.25, PVC and PVB Resins, J.J. Brezinski, Chairman, reported the "Standard Guide for Poly (Vinyl Chloride) Resins" has been designated D 4368. The SPI has an ongoing project to develop a test method for vinyl chloride monomer in vinyl packaging materials sensitive to one part per billion. Committee D-20's Section 15.08 on Vinyl Chloride Monomer and Resins will be contacted about improving instructions for head space analysis of vinyl chloride monomers in PVC resins now in D 3749.

SUBCOMMITTEE D01.35 SOLVENTS, PLASTICIZERS AND CHEMICAL INTERMEDIATES

L.R. Thurman, Chairman

D01.35.10, Solvents-Hydrocarbons and Ketones, S.A. Yuhas, Jr., Chairman, will prepare five standards for Sub. D01.35 ballot: D 329, D 1133, D 1353, D 1363, and D 1722. D 1722, "Test for Water Miscibility of Water Soluble Solvents," was revised by the addition of a proposed Significance and Use statement and a Precision and Bias statement.

An item of new business, D 1364, "Test Method for Water in Volatile Solvents (Fischer Reagent Titration Method)," was discussed. There is a need to update this method to allow the use of commercially available reagents of the pyridine type described in the reagents section and to allow use of non-pyridine reagents which have become available. An editorial revision will be submitted for ballot, reflecting the availability of these reagents. In the future there will be a need to develop precision data for these available reagents.

D01.35.20, Reactive Monomers—Vinyl Monomers and Aldehydes, T.H. Golson, Chairman, will prepare four standards for Sub. D01.35 ballot: D 1078, D 3541, D 3547, and D 3548. A discussion was held on the joint effort of Sub. D01.35 and Sub. D01.22 to resolve the precautionary statement language. Sub. D01.22 will put the "proposed policy" into the format of an ASTM Standard Guide and reballot.

New standards for methyl acrylate and acetaldehyde will be proposed at the next meeting.

D01.35.30, Chemical Intermediates, J.R. Morrison, Chairman, will prepare 11 standards for Sub. D01.35 ballot: D 331, D 1612, D 1719, D 3546, D 3620, D 3622, D XXXX Specification for Propylene Glycol Monomethyl Ether, D XXXX Test for Purity of Propylene Glycol Monomethyl Ether, D XXXX Specification for Dipropylene Glycol Monomethyl Ether, D XXXX Test for Purity of Dipropylene Glycol Monomethyl Ether, and D 1258 section 6.4.

An Ad Hoc Committee was appointed to finalize a gas chromatographic analytical method for 2-ethylhexanol.

D 4052, "Test Method for Determining Specific Gravity," was evaluated and judged to be acceptable for Sub. D01.35 standards.

D01.35.40, Plasticizers and Ester Solvents, R.L. Smith, Chairman, will prepare 12 standards for Sub. D01.35 ballot: D XXXX Specification for n-Butyl Acetate, D XXXX Specification for Ethyl Acetate, withdrawal of D 303, D 3126, D 3729 (n-butyl acetate), withdrawal of D 302, D 3127, D 3727 (ethyl acetate), D 2634, D 3130, D 3540, and D 3545.

D01.35.50, Liaison. Lucille P. Field was appointed chairman of this group. Chairmen of the other four task groups will compile a list of all specifications not under the jurisdiction of Sub. D01.35, which will be reviewed at the next meeting to establish further action.

New Business—A proposal was made to issue D 1078 as a joint standard with IP-195 (both entitled, "Distillation Range of Volatile Organic Liquids"). A comparison will be made and a recommendation issued.

A review and summation of Karl Fischer water test methods will be conducted. The suitability or usefulness to Sub. D01.35 standards will be determined.

Consideration is being given to a special publication specific to solvents—specifications and test methods.

DIVISION 40 PAINT PRODUCTS APPLIED ON SITE

SUBCOMMITTEE D01.41 CRITERIA FOR THE PURCHASE OF COMMERCIAL PAINTS AND COATINGS

J.C. Weaver, Chairman pro tem

Reassessment continues on the scope, role and plans for Sub. D01.41, with little agreement among diverse interests across the three main classes of D-1 members.

D01.41.01, Title and Scope Revision, J.C. Weaver, Chairman, noted the diversity of opinions across federal government departments and among various states on (1) general questions on primary technical composition versus performance specifications and (2) secondary contractual elements such as Part B Form of ASTM Specifications: B9 Inspection, B20 Rejection and Rehearing, B21 Certification,—Quality Assurance, et al. Competition was noted between large volume central contracts on unbranded specification paints versus small decentralized purchases of widely available branded or trademarked paints.

D01.41.02, D 3927, Standard Guide for State and Institutional Purchasing of Paint, W.V. Moseley, Jr., Chairman, discussed returns on an early 1984 letter ballot and on a December 11, 1984 draft on D 3927's Weighted Cost sections, and asked for more time to resolve differences. ASTM's five-year revision rule requires early D-1 action on reballoting D 3927-80.

D01.41.05, Latex Flat Wall Paint Specification, I.V. Bloom, Chairman, discussed Sub. D01.41 ballot comments and negative votes on Draft No. 2, which had deleted some of the compositional requirements of Draft No. 1, while emphasizing performance tests and also including contractual sections: 4-Ordering Information, 10-Rejection and Rehearing, 11-Certification, 12-Container Labeling and Marking and 13-Packaging. More time was needed for resolution of comments. An interim meeting was planned (later set for May 7, 1985 at NPCA Headquarters in Washington, D.C.).

SUBCOMMITTEE D01.44 TRAFFIC COATINGS

R.L. Davidson, Chairman

The development of a specification for thermo-plastic is well underway. A new series of photographs that better depict a traffic line failure range is being prepared. The report of the retro-reflectance workshop last fall will soon be available.

SUBCOMMITTEE D01.45 MARINE COATINGS

L.S. Birnbaum, Chairman

D01.45.05, Algae Control, C.D. Stevens, Chairman, reviewed Draft #3 of the proposed "Standard Test Method for

Evaluation of Antifouling Coating Algal Control Efficacy" prepared by the chairman. This draft incorporated suggested changes on preconditioning, panel spacing, panel cleaning, and rating scheme. It was agreed to incorporate some additional modifications recommended during the discussion into Draft #4, which will then be forwarded to the ASTM Editorial Staff for review prior to the next meeting.

D01.45.06, Dynamic Testing, D. Laster, Chairman. In the absence of the chairman, the meeting was chaired by L. Birnbaum. Copies of the latest draft of "Standard Test Method for Marine Anti-Fouling Systems under Hydrodynamic Flow" prepared by Laster (and designated at this meeting as Draft #1, dated 1/23/85) were circulated. ASTM Editorial Staff comments on this draft were presented for approval, and the document was reviewed for further corrections and additions. The group agreed upon several revisions and approved the submission to Sub. D01.45 ballot.

Copies of a schematic for High Velocity Flow channel apparatus for dynamic testing of antifouling systems was circulated. This was furnished by T. Dowd who advised that the Navy would like Sub. D01.45 to consider this technique for development of an alternative standard for dynamic testing. Since Dowd was not present to provide additional details, it was agreed to defer action on this request until the next meeting. The Navy will be requested to send a representative to the next meeting to provide details and answer questions.

D01.45.07, Antifouling Rating, C. Perez, Chairman, circulated results of exposure studies underway since March 1982 using the Miami Marine Research rating scheme. C.D. Stevens will be provided with a set of photographs from this series showing a range in AF performance. A description of the rating method has been furnished to Stevens by Miami Marine.

Battelle has furnished a description of the Battelle rating method and a set of photographs showing a range in performance. The photographs provided were of proprietary paint test panels. T. Herlihy explained that he did not utilize the Sub. D01.45 panels because he was unable to cover the lower performance range. After considerable discussion, it was agreed that Battelle and Miami Marine Research would resolve the selection of photographs of exposed panels and send them to Stevens. Stevens will review his efforts to prepare photos of exposed panels by reviewers for precision and bias data when using the Battelle, The Miami Research, and the present D 3623 schemes.

D01.45.08, Organo-Tin Release Rate, R. Gangi, Chairman, met after the conclusion of the Sub. D01.45 meeting. This is a new working group established to develop a reliable method for determination of organo-tin release rates from organo-tin antifouling paints. The results of an Ad Hoc group meeting held in Arlington, VA on 1/7/85 to define the scope and objectives were reported.

A number of methods were circulated including protocols from the Navy (NSRDC, Annapolis), International Paint, M&T, and Ameron. It was agreed that a number of variables could affect results and must be considered in the development of a standard including substrate, paint conditioning, temperature, salinity, slime and pH. P. Schatzberg indicated that based on Navy studies in San Diego harbor and other Navy harbors, low release rates in AF paints for use by the Navy was essential to avoid a significant impact on the environment and that the Navy planned to determine in-situ release rates and concentrations in harbors to verify impact. The chairman indicated that he would probably convene a group meeting prior to the July 1985 meeting.

D01.45 Meeting—It was announced that F-25.02 was scheduled to meet in New Orleans 2/13/85 to 2/15/85 and that it was requested that Sub. D01.45 members be added to the F-25.02 mailing list.

A test method prepared by Laster in ASTM format for blister resistance of anticorrosive hull paints was distributed with a verbal request from the Navy that it be used as a basis for an ASTM standard. The subcommittee agreed that a need for a blister resistance test existed, but questioned whether this draft document was the proper basis for the test. In view of the absence of a Navy spokesman on this test, action on the request was deferred until the July meeting.

A test method for cathodic disbondment prepared by International Paint Co. was distributed in connection with the development of an ASTM standard for this property. Also distributed was the test developed by the Navy and described in the drafts of the proposed DoD performance specification for antifouling paint. It was pointed out that this method was also included in ASTM F-25.02 drafts of performance specifications for anticorrosive and antifouling paints. Action on this item was deferred pending receipt of a written response from members (to be included in the distribution of the minutes) regarding their interest and willingness to participate.

SUBCOMMITTEE D01.46 INDUSTRIAL PROTECTIVE PAINTING

K.A. Trimber, Chairman

D01.46.02, Surface Preparation, K.A. Trimber, Chairman, reported that the new standard for profile measurement developed by the task group has been approved by COS as of November 14, 1984. It is designated D 4417-84, "Test Method for Field Measurement of Surface Profile of Blast Cleaned Steel." It addresses the use of the Keane-Tator Surface Profile Comparator. Testex Press-O-Film tape and depth micrometer.

D 2200, "Pictorial Surface Preparation Standards for Painting Steel Surfaces," was due for reapproval in 1984. A modified copy was included in the 84-3 committee ballot. One negative (Sub. D01.46 and D-1) was received from F. Lichtenstadter and comments from three other voters. The comments were resolved through editorial changes. The negative, which stated that it should be made more clear that photograph BSa2 does not comply with the SSPC-SP6 definition of Commercial Blast due to the presence of mill scale, was found to be persuasive and "Note 2" was changed editorially to address this concern. The changes were accepted and withdrawn by the negative voter by telephone.

D01.46.03, Repainting, R. Wakefield, Chairman, reported that, as a result of the discussion of the negatives and comments held at the May 1984 meeting, a revised draft of the new "Test Method for Field Identification of Coatings" will be prepared (Draft 4) by March 15, 1985 for another Sub. D01.46 ballot.

SSPC has expressed interest in cooperating in this group's work. They are currently working on the compatibility of coatings used for repainting. It was agreed that this Draft 4 will be circulated to SSPC for comments, and that the SSPC compatibility work likewise will be circulated to Sub. D01.46. If SSPC finds a need for the development of test methods to verify compatibility, it is expected that such methods will be developed by this task group.

D01.46.04, Pull-Off Adhesion, A. Cunningham, Chairman, reviewed the three negatives and 14 comments from the Sub. D01.46 ballot of October 1984 on Draft 7 of proposed "Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers." The negatives were resolved. The changes, including those resulting from the comments, will be

included in Draft 8 which will be submitted to a concurrent Sub. D01.46/D-1 ballot.

D01.46.07, Inspection, R. Martell, Chairman, had an inspection list circulated prior to the meeting which addressed quality items that an inspector must examine during surface preparation and coating application. The checklist was reviewed and additions made. The checklist will be appended to a newly revised draft of D 3276, "Standard Guide for Painting Inspectors." Both the Guide and the checklist will be submitted to Sub. D01.46 ballot in the near future. The plans are to review all comments at the July meeting and to submit the Guide to D-1 ballot in the Fall of 1985.

D01.46.10, Condition Assessment, M. McKnight, Chairman, discussed the scope of the task group and defined it as follows: "Develop a guide for assessing the condition of the existing paint on a structure, and the condition of the underlying substrate. The objective of the assessment is to provide a sound basis for determining maintenance painting procedures. Subjects to be addressed include inspection protocol, condition standards, rating of coating and substrate, surface deterioration, and characterization of existing coating."

A checklist of the information to be included in the condition surveys will be circulated to Sub. D01.46 members by May 15, 1985. SSPC has agreed to cooperate as they are currently working in this area. It is expected that this ASTM standard will be followed for the collection of the data, and the SSPC method followed for the evaluation of the data and for making decisions on the amount of repainting to be done, system selection, etc.

D 610, "Standard Method for Evaluating Degree of Rusting on Painted Steel Surfaces," is due for reapproval in 1985. An editorially revised copy (adapting it to the new ASTM format) will be submitted for concurrent Sub. D01.46/D-1 ballot this Spring.

D01.46 Wrap-Up—D01.48 on Zinc-Rich Coatings met jointly with Sub. D01.46 in this session. Sub. D01.48 stated that its chairman, D. Kinder, could no longer be active in the subcommittee and that its scope was so limited that it was felt no longer necessary to act as a separate subcommittee. A request from Sub. D01.48 was approved to recombine with Sub. D01.46. The Executive Subcommittee has also approved this move. It will become Sub. D01.46.11 on Zinc-Rich Coatings, chaired by R. Wakefield.

SUBCOMMITTEE D01.47 MASONRY TREATMENTS

R.S. Shane, Chairman

The comment and the negative on the ballot on the proposed scope of the subcommittee were withdrawn. Further discussion on the scope ensued and it was recognized that amendments are always possible. It was decided to ask all D-1 members to suggest topics for Sub. D01.47 task groups and to ask for volunteers to work in these task groups. Anyone submitting suggestions or volunteering should contact the subcommittee chairman. It was suggested that problems for task groups should be small-sized problems capable of solution in reasonable time, e.g., condition assessment, cleaning specifications by material, etc.

SUBCOMMITTEE D01.48 ZINC-RICH COATINGS

D.C. Kinder, Chairman

The meeting was chaired by R.H. Wakefield in the absence of D.C. Kinder who has submitted his resignation as chairman.

A motion was made and passed unanimously to dissolve Sub. D01.48 and to reform as a task group of Sub. D01.46 on Industrial Protective Painting. Sub. D01.46 had already voted to accept this task group. It will be designated Sub. D01.46.11 and will be chaired by R.H. Wakefield. A one-hour meeting will be scheduled for the July 1985 meeting of D-1, at which time the scope of the task group activity will be presented.

The members listed on the Sub. D01.48 roster (57) will be polled by mail as to whether they wish to retain membership in the Zinc Rich Task Group, D01.46.11.

L. Smith presented a summation of work done by Georgia Tech on development of a solvent-rub test to evaluate the cure of ethyl silicate-based inorganic zinc-rich coatings. Smith will write a draft ASTM standard for this test for distribution and review prior to the July meeting. The chairman will advise Committee D-33, whose members have been working on cure tests of the Sub. D01.46.11 activity.

It was reported that the status of the proposed circle (bullet hole) test procedure is unchanged from the last meeting. A round-robin test protocol to evaluate Draft 2 of the test method has not been developed. The chairman will attempt to draft the round-robin protocol prior to the next meeting.

DIVISION 50 PAINTS FOR FACTORY APPLICATION

SUBCOMMITTEE D01.52 FACTORY-COATED WOOD BUILDING PRODUCTS

S.B. Schroeder, Chairman

Results of balloting on several items and some other methods now under study were discussed.

On D 2793, "Standard Test Method for Block Resistance of Organic Coatings on Wood Substrates," negatives were cast by C.M. Winchester and H.E. Ashton. Although there were some editorial comments and concerns, the primary deficiency noted, and found persuasive, was the lack of a precision statement, Mr. Ashton suggested that D 2793 be combined with D 3003, "Standard Test Method for Pressure Mottling and Blocking Resistance of Organic Coatings on Metal Substrates." This suggestion was approved unanimously. It was further decided that the chairman would pursue this combination of methods with Sub. D01.53, which has jurisdiction over D 3003.

A one-year extension has been given to D 2921, "Standard Method for Qualitative Tests for the Presence of Water Repellents and Preservatives in Wood Products," in the hope that a new task group on Mill Work could be formed in time to address this method. However, the first Mill Work task group meeting was not attended by the Mill Work manufacturers. One more attempt will be made by the chairman and C. Peterson to obtain input from members of the National Woodwork Manufacturers Association prior to the July meeting when a decision will be made to either rewrite or discontinue D 2921.

The chairman produced a tentative Significance and Use statement for D 3719, "Standard Test Method for Dirt Collection on Coated Exterior Panels," which was approved unanimously. The revised method will be submitted for concurrent Sub. D01.52/D-1 ballot.

J. DeVedo volunteered to review D 2336, "Standard Practice for Specifying Properties from Liquid Through Cured State for Coatings Factory Applied to Wood Products," prior to the July meeting.

J. DeVedo and the chairman volunteered to review D 2691, "Standard Methods for Microscopical Measurement of Dry Film Thickness of Coatings on Wood Products," with analytical experts at their respective companies.

D01.52B, Hardboard, S.B. Schroeder, Chairman, discussed previous and new data submitted by J. Tooley which appears to show serious reproducibility problems with the AHA "protection and interaction test." This method is a simple technique intended to measure and quantify the degree of wetting and swelling induced in hardboard siding by the action of surfactant components in trade sales latex paints. It was decided to wait until the present AHA round-robin is completed and additional data is available before making a decision to attempt writing a standard for this material.

Other means of measurement of surfactant effects on hardboard were also discussed. The chairman volunteered to explore the possible adaptation of a standard salt spray chamber to provide a wet exposure on coated hardboard.

D01.52.13, Prefinished Siding, K. Kruse, Chairman, reported that the response to the industry survey for test method needs was disappointing, but some information was received. Prior to the July meeting the chairman will review this information and determine whether existing ASTM methods satisfy the stated needs. Some of the tests presently being run in the industry appear to be similar to ASTM methods.

The chairman will also review past data on the DSET Lab's "EMMAQUA" test and report at the July meeting. A standard for this test may be justified.

Other Business—It was announced that the suggested change in scope and title of Sub. D01.52 has been approved. The title has been changed to "Factory Coated Wood Building Products."

SUBCOMMITTEE D01.53 COIL COATED METAL

R.A. Cassel, Chairman

Sub. D01.53 met as a committee of the whole with the main topic for discussion, once again, the lack of working members. This was the reason for having no task group meetings. All business will be handled at the subcommittee level until sufficient help is available to reactivate task groups. With respect to recruiting, J.C. Weaver suggested contacting E.A. Praschan to determine any automotive interest in coil-applied systems.

S.B. Schroeder, chairman of Sub. D01.52, met with Sub. D01.53 to discuss the possibility of combining D 2793, "Block Resistance of Organic Coatings on Wood Substrates," and D 3003, "Pressure Mottling and Blocking Resistance of Organic Coatings on Metal Substrates." Schroeder will put together a draft of a joint document prior to the next meeting in July.

L. Hamilton and C. Milburn agreed to review D 3363, "Film Hardness by Pencil."

SUBCOMMITTEE D01.55 FACTORY-APPLIED COATINGS ON PREFORMED PRODUCTS

F. Zurlo, Chairman

D01.55.01, Coatings for Wood Products, F. Zurlo, Chairman, approved the scope modification and inclusion of specification details for test panels in D 1211, "Temperature Change Resistance of Clear Nitrocellulose Lacquer Films Applied to Wood." The method will be submitted to concurrent Sub. D01.55/D-1 ballot.

Methods D 2571, D 3023, D 2620, D 3459, and D 2091, which are due for reballoting in 1986, were reviewed. Significance and Use statements were added to the latter three. All five methods will be submitted for technical review to four corporate laboratories with expertise in wood finishes. The intent is to have the reviewed methods, with any attendant comments, available for subcommittee review and editing at the July meeting. Following that meeting the methods will be submitted for appropriate balloting.

Strategy was agreed upon to encourage additional participation in Sub. D01.55 by representatives from the manufacturers of factory-applied coatings for wood products.

D01.55.07, Industrial Water-Reducible Coatings, F. Zurlo, Chairman, reported that the "Guide for Testing Industrial Water-Reducible Coatings" has been edited and is ready for Society ballot.

With the completion of this guide, the task group has no other outstanding project. If none develop, dissolution will be considered.

SUBCOMMITTEE D01.56 PRINTING INKS

J.M. Fetsko, Chairman

D01.56.01, Fineness of Grind, J. Cichon, Chairman, distributed a summary of round-robin results and a proposed precision statement for D 1316, "Test for Fineness of Grind of Printing Inks by the Production Grindometer." It was noted that reproducibility of scratch endpoints was markedly improved by treating four readings as a single determination and by deleting premixes from the statistical analysis. The text of the test method will be revised prior to reballoting.

D01.56.02, Lightfastness of Prints, A. Mulvihill, Chairman, reported on progress with exposure of prints to sunlight in various locations of the USA. As expected, rate of color degradation was greater in July than in December. In addition, halftone prints on coated paper changed faster than the corresponding solid and uncoated paper prints, respectively. Attempts are also being made to develop accelerated exposure conditions which will rate the prints in the same order as did actual light exposure.

D01.56.04, Viscosity of Paste Inks, J.M. Fetsko, Chairman, reported that D 4040, "Viscosity of Printing Inks by the Falling Rod Viscometer," is up for review in 1986. It was suggested that the method be expanded to include the "n" value and apparent yield stress.

D01.56.06, Ink Tack, C. Shepard, Chairman, reported that the new test method, "Apparent Tack of Printing Inks by the Inkometer," will appear as D 4361 in Part 06.01 of the 1985 *Book of Standards.*

D01.56.09, Tinting Strength, W. Riedel, Chairman, distributed a summary of round-robin results and a proposed method to minimize bias in visual tinting strength tests. In response to reports that the use of a blue tinted base for yellow colorants sometimes gives erroneous results, the subcommittee agreed to conduct a new round-robin in order to test the suitability of TiO_2 and ZnO white bases for evaluating the relative strength of yellow dispersions.

D01.56.10, Water Uptake of Litho Inks, G. Bien, Chairman, distributed a summary of round-robin data and a proposed test method for measuring water uptake as a function of time utilizing the Duke Tester. A discussion ensued on the preferred method for adding water and for removing unemulsified water and the need for controlling test temperature. Besides revising the method accordingly, statistical analysis must be performed on the test data.

D01.56.11, Nonvolatile Content of Printing Inks, B. Blom, Chairman, distributed copies of the proposed test method, which now covers NVM of heatset, as well as liquid printing inks. After minor changes are incorporated, the method can be submitted for concurrent Sub. D01.56/D-1 ballot this Spring.

D01.56.12, Viscosity of Liquid Inks, C. Shepard, Chairman, reported that two current methods, D 4212, "Viscosity by Dip Type Viscosity Cups," and D 2196, "Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer," are quite satisfactory for liquid printing inks. Any work in this area will be conducted in cooperation with the responsible subcommittee, Sub. D01.24.

D01.56.13, Ink Mileage, B. Blom, Chairman, reported that plans to correlate laboratory test results with ink mileage obtained during production press runs have to be deferred until a new representative from a major printing house is appointed to the Task Force. The subcommittee suggested that, in the meantime, a study be conducted to compare mileages of emulsified and unemulsified offset inks.

D01.56.14, Drying of Heatset Inks, A. Urdea, Chairman, reported that four companies participated in an exploratory round-robin in which prints prepared to a standard density were tested for set-off and rub resistance after passing through the Sinvatrol Tester. The subcommittee agreed that, in the next round-robin, the endpoint would be set-off, not rub resistance.

D01.56.15, Specific Gravity of Printing Inks, G. Bien, Chairman, having reviewed D 891, "Specific Gravity of Industrial Hydrocarbons and Related Materials," and D 1475, "Density of Paint, Varnish, Lacquer, and Related Products," considered the two existing ASTM methods to be very well written and quite adequate for printing ink use. Therefore, no work in this area need be conducted by Sub. D01.56.

D01.56.16, Abrasion of Gravure Inks, J. Hart, Chairman, reported that due to changes in corporate personnel, no work has been conducted to investigate methods for abrasion of rotogravure inks. The pursuit of this subject will be postponed until further notice.

SUBCOMMITTEE D01.57 ARTISTS' PAINTS

J.T. Luke, Chairman

A Planning Meeting was held on January 19, 1985 to give a general update of the situation in California and other states concerning state labeling laws for chronic toxicity and to review the proposal to include alkyds under D 4302 and D 4303. Upcoming meetings were also discussed. A meeting is scheduled in conjunction with the annual Inter-Society Color Council which will be held in Pittsburgh on April 14–16. The subcommittee will also meet with D-1 in San Diego in July with a special meeting scheduled at the Town and Country Hotel on Monday morning, July 8, concerning labeling for chronic toxicity and California's new law. State of California officials concerned with these matters have been invited to speak at this meeting.

Chairman Luke is seeking input on how to organize to handle individual state bills on chronic toxicity. Laws are pending in several states. This will be discussed in July.

A discussion followed on details of changes proposed in D 4303.

D01.57.01, Preparation of Samples for Colorimetric Determination, T. Pamer, Chairman, reported that "Preparation of Films of Artists' Paste Paints for Colorimetric and Other Measurements," written by H.W. Levison, had been held up because of lack of complete hiding in the acrylic drawdowns. Most, but not all, oils were opaque when drawn down at 6 mils, but many acrylic paints were not. I. Shack passed out templates with a 10 mil aperture to participants in an upcoming round-robin drawdown of acrylic paints.

Luke reported that a mathematical method to adjust for the effect of the substrate on measurements of semi-opaque specimens has been suggested by R. Berns. If this proves to be accurate it would be an alternative to thick films.

D01.57.02, Lightfastness of Pigments, H.W. Levison, reviewed a proposed revision of D 4303. The revision of 10.3.1 is: Expose one specimen, inland, outdoors under glass to sunlight in Southern Florida below 27 degrees latitude following Method 1 and expose the second specimen indoors to simulated daylight following either Method 2 or 3.

D01.57.03, Tinting Strength, T. Pamer, Chairman, issued a report on the work done on Phase I of a proposed tinting strength (TS) standard. Phase I deals with a method to compare TS of several prepared tube paints. Data on seven new paints was presented and that from previous work on two paints was reviewed in a new format.

Overall, the test method of Ruth Johnston-Feller's looks very promising. Results indicated that a range of 20–55% R at the wavelengths of maximum absorption, might be too broad for some colors. Subcommittee members thought a range of 35–45% for all paints would be practical and more accurate.

The question of which K/S factor (R.B. or G.) should be used when calculating TS for certain pigments is also being studied. It was suggested that additional paints to be included in an information table for the proposed method as follows: cadmium yellow light, yellow ochre, black, siennas, umbers, and white.

It was decided that since the chance of incompatibility between different artists' paint lines is small, a standard acrylic mixing white should be required, rather than requiring that the same brand of white be used as the paint being tested. The tinting strength of artists' whites varies widely. A. Spizzo agreed to work on a volume/weight conversion method for this proposed test method.

D01.57.04, Specification for Artists' Paints, A. Spizzo, Chairman, reviewed the proposed changes to D 4302 that were discussed in the Planning Meeting. Some additional changes were proposed so that natural resin/oil paints can be included in the standard. All of these proposed changes are detailed in the official minutes of the subcommittee.

D01.57.07, Physical Properties, H.W. Levison, Chairman, reported that the paper entitled, "Yellowing of Paint Vehicles in an Indoor Environment," prepared by H.W. Levison, will be dropped as a proposed standard. This paper will now be prepared for publication as a journal article.

The paper entitled, "The Adhesion and Cohesion of Normal Films of Artists' Acrylic Emulsion and Oil Tube Paints," prepared by H.W. Levison, using a Semimicro System 2000 Adhesion Testing Apparatus, was recommended to be incorporated in a standard. Sub. D01.57 members were asked to read it again and comment.

D01.57.09, Watercolors, E.T. Vonderbrink, Chairman, heard reports from three members working on this project:

I. Shack is working on using an air brush for applying watercolor to paper. He is having problems getting a uniform film because of spitting of the airbrush, but will continue to work on the technique for application.

R. Johnston-Feller is not happy with the air brush application because the quantity of paint deposited is not known. She is working on a drawdown method for applying uniform films. Two paints were tested by drawing down 4–10% dilutions on #42 Whatman filter paper to achieve 40% reflectance. For these few paints the method had a repeatability of 95%. Work will be continued on this method.

T. Vonderbrink is working on air brushing 1 volume watercolor/19 volumes water on 200 lb. cold pressed paper. He circulated a report and showed panels of four different watercolors air brushed on

2″ × 6″ panels at four different reflectances (30%, 40%, 50%, 60%). Six colorimetric readings were taken top to bottom on each panel at one inch intervals. Delta E's were calculated for each reading and compared to the average. Areas of nonuniformity could be seen.

The subcommittee thought the 40% reflectance was a good visual color for testing. Data indicates that the larger the area brushed, the more the color variation. Since only a small panel (2″ × 2″) is needed for testing, it is possible to get a test panel of uniform color by air brushing a large area to get 40% plus/minus 5% R, and checking by instrumentation, for a uniform area. When a uniform area is found, it can be cut out and used as the panel for testing. Additional work will be done on air brushing a large area and obtaining colorimetric data on a 2″ × 2″ area of uniform color.

D01.57.10, Consumer Evaluation, J.T. Luke, Chairman, circulated a report, "Lightfastness Test for Artists' Materials." This report gives supplies needed and a test method that can be used by a person desiring to gauge the lightfastness of artists' materials which are displayed behind glass. The test uses the ISO Blue Wool Standard cards as controls for the amount of exposure. The method can be used by an artist to evaluate his own materials, but if the results are to be communicated to others, a rating system is needed. Results of a round-robin indicate that three or more individuals will have to rate exposed samples to achieve repeatable ratings.

Input was requested as to what terms should be used to characterize the three broad lightfastness categories into which materials will be placed. The terms "durable," "moderately durable," and "fugitive" were proposed as possible ratings for the materials after testing.

DIVISION 60
PAINT APPLICATION

SUBCOMMITTEE D01.61
PAINT APPLICATION TOOLS

F.B. Burns, Chairman

D01.61.01, Paint Brushes, T.J. Sliva, Chairman, announced that materials will be sent out in March to begin two paint brush characterization test round-robins. One of these methods is to determine the paint discharge characteristics of a brush, and the other is intended to determine the paint film leveling characteristics of a brush. Seven cooperators agreed to participate. The chairman plans to complete testing before the next meeting.

D01.61.02, Paint Rollers, J.F. Price, Chairman, announced that the first round-robin test on Discharge Characteristics of a Roller Cover was in progress. To date three of the seven cooperators had returned results. A discussion by the group led to a proposal for a task group on "pressure-fed" roller covers. Emil Martin, of PPG, agreed to be chairman of this new group to be formalized at the next meeting.

ASTM Committee D-1 Staff Liaison is Brent Backus. ASTM is located at 1916 Race St., Philadelphia, PA 19103.

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BIRMINGHAM ..... FEB.

"UV Radiation Wavelength"

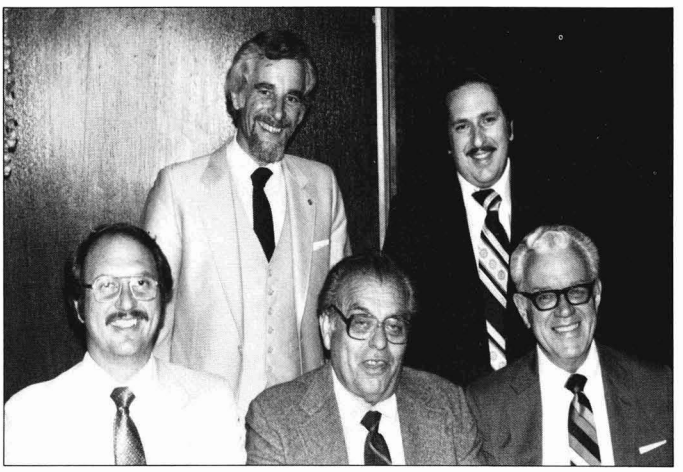
Dr. L.A. Simpson, of B.T.P. Tioxide Ltd., presented a lecture entitled, "THE SIGNIFICANCE OF WAVELENGTH OF UV RADIATION ON DURABILITY."

Dr. Simpson examined the effect of UV wavelength on the durability factors of gloss, chalking, and mass loss in TiO_2 pigmented systems. He began by explaining that the spectral distribution of low UV wavelengths determines the extent to which photochemical and photocatalytic degradation of a binder occurs.

Comparing photochemical and photocatalytic degradation, Dr. Simpson stated that photochemical degradation occurs at the binder surface while photocatalytic degradation results in binder breakdown at sites immediately surrounding pigment particles, which degrade due to UV absorption. He also explained that in alkyd-based paints, exposed between 390 and 290nM, the degree of photochemical degradation was found to increase as wavelength decreased, whereas the degree of photocatalytic degradation remained constant. Where only photocatalytic degradation occurs, poor gloss results, and if only photochemical degradation occurs a rougher surface is produced. If both types of degradation occur, the paint has a better gloss and is less rough than where each occur singly. Mass loss is constant for both, added Dr. Simpson.

To reproduce the UV wavelengths found outside, the lamps used in exposure test equipment should emit a similar spectral distribution, stated Dr. Simpson. The closest match to summer sunlight is the xenon arc, followed by the carbon arc. He added that exposure of alkyds at 310nM using an xenon arc, or at 390nM using a carbon arc, produced results very similar to outside exposure. The results of natural weathering mirrored those of the carbon and xenon arcs.

In closing, Dr. Simpson discussed variations in gloss readings. Large variations in gloss readings after weathering in machines using different tube or lamp types has been proven to be a result of the different UV spectral emittance of the tubes. He stated that when different resin types are artificially weathered at lower wavelengths, gloss readings are erroneous when compared with the results of natural weathering. Mass loss measurements, however, show closer reproducibility, concluded Dr. Simpson.



1984-85 OFFICERS OF LOS ANGELES SOCIETY. (Left to right): Treasurer—Henry Kirsch; Society Representative—Jan Van Zelm; Vice-President—Mike Gildon; Secretary—Ray DiMaio; and President—Earl Smith

Q. How is mass loss measured?

A. Using stainless steel panels of 3 × 6 inches, coated by brush or spray, and stoved (where necessary), and sent to Florida for exposure and measurement.

Q. Can UV absorbers be used in paints to reduce degradation?

A. No. This is impracticable as they also cause pigment flocculation and reduce gloss.

DAVID M. HEATH, *Secretary*

CHICAGO ..... MAR.

"Air Dry Water-Borne Primers"

Al Heitkamp, of Cargill, Inc., addressed the topic of "AIR DRY WATER-BORNE PRIMERS FOR METAL SUBSTRATES," and Hugh Smith, of Sun Chemical Corp., presented an "ENVIRONMENTAL UPDATE."

R.L. KLEINLEIN, *Secretary*

CLEVELAND ..... MAR.

"Rheology"

Carl Knaus, of Kent State University, discussed "RHEOLOGY FOR THE PAINT FORMULATOR."

Rheology is the science of the deformation and flow of matter, explained Dr. Knaus. The rheological property which is most familiar is viscosity. He then enumerated several types of viscometers in common use: the rotating paddle, efflux cups, falling ball types, and bubble tubes. All are valuable tools but are of limited value in the study of rheological properties, stated Mr. Knaus. They work best with strictly Newtonian liquids and were designed to give a one point reading under closely controlled conditions, he added.

Instruments have been developed to measure other properties such as flow, sag, impact resistance, adhesion, and roll spattering. Dr. Knaus ended his discussion by stating that today many of these

Northwestern Society President Rich Johnson, of Cargill, Inc., (right) presents Lowell Wood, of Frost Paint, with the Society Honorary Member plaque

OFFICERS OF NEW YORK SOCIETY for 1984-85. (Left to right): Secretary—Kenneth De Paul; Vice-President—Raymond P. Gangi; President—Michael Iskowitz; Treasurer—John W. Burlage; and Society Representative—Saul Spindel

rheological properties are routinely measured in control labs.

MADELYN K. HARDING, *Secretary*

GOLDEN GATE ..... MAR.

"Paint and Profit"

Barry Adler, of Royelle, Inc., Society Representative to the Federation, was presented with a Twenty-Five Year pin.

Certificates from the Federation were presented to members of the Technical Committee who worked on the award-winning paper, "Water-Borne Corrosion-Inhibitive Primers Evolve to Be Competitive," presented by Bob Athey, in October at the Paint Show.

Albert J. McMichael, of Universal Color Dispersions, described the "SENSIBLE WAY TO PAINT AND PROFIT."

KARL SAUER, *Secretary*

LOS ANGELES ..... MAR.

Past-Presidents' Night

Earl Smith, Society President, introduced all the Past-Presidents that were present and named those not in attendance. Those present included: Lloyd Haanstra (1983-84); Romer E. Johnson (1982-83); Jan Van Zelm (1981-82); Donald I. Jordan (1980-81); Albert Seneker (1979-80); Fred Croad (1978-79); Robert F. Koperek (1977-78); Gerald L. West (1976-77); Kenneth O'Morrow (1975-76); Robert McNeill (1974-75); William H. Ellis (1971-72); Trevellyan Whittington (1969-70); and Albert Aronow (1964-65).

Past-President Lloyd Haanstra gave an environmental report concerning the Rule 1113 change of 380 grams per liter down to 250 and what may happen if the industry does not do something to stop it. Five people were selected to do detailed work on the reasons why the industry cannot live with the 250 level.

Mr. Haanstra also told of a recent Architectural Task Force meeting. He emphasized the need for industry funding of the Task Force. He added that the Air Resources Board needs a survey to learn where the coatings industry is today and if past regulations had any effect at all on air pollution.

Dick Sutherland reported that the 1985 Western Coatings Societies Symposium, held Feb. 26-Mar. 1, was very successful with 3000 registrants and 170 exhibits.

Al McMichael, of Universal Color Dispersions, spoke on "THE SENSIBLE WAY TO PAINT AND PROFIT."

Mr. McMichael discussed the economic and sales advantages in manufacturing paint using purchased dispersions vs in-house composite grinding. He pointed out that dispersion suppliers are now, more than ever, in the service industry. In closing, Mr. McMichael said that the objective will be to substantiate areas of mutual opportunity for both the paint manufacturer and the dispersion supplier.

RAY DIMAIO, *Secretary*

LOUISVILLE ..... MAR.

"Wetting and Dispersing Agents"

Society President Ed Thomasson announced the nomination of Larry Pitchford as Secretary for 1985-86.

Robert W. Vash, of Byk Chemie USA, spoke on "WETTING AND DISPERSING AGENTS AND HOW THEY AFFECT PRODUCTIVITY."

According to Mr. Vash, pigments arrive at a manufacturing facility in one of three states—primary particles, secondary or flocculents, and tertiary particles or aggregates. The goal of dispersing/wetting agents is to make it easier through mechanical or chemical means to separate pigment particles and to insert "shock absorbers" between particles so they can't run back together. One method of accomplishing this, explained Mr. Vash, is to absorb or displace moisture or air off the surface of the particles and to attach something to the particle that is compatible with the continuous media.

Mr. Vash then discussed five classes of wetting agents: anionic, cationic, electroneutral, amphoteric, and non-ionogenic. The benefits derived from the use of these wetting/dispersing agents include maximum dispersion, maximum color, maximum tint strength, maximum anti-settling, improved hiding, improved gloss, reduced solvent popping, reduced air entrapment and reduced flotation of pigment.

Mr. Vash closed by describing the difference between flotation and flooding.

K.R. HYDE, *Secretary*

1984-85 OFFICERS OF ROCKY MOUNTAIN SOCIETY. (Left to right): Society Representative—James E. Peterson; President—Larry Lewandowski; Vice-President—Carwin Beardall; Treasurer—Marcy S. Baugh; and Secretary—Craig Hansen

NEW ENGLAND ..... MAR.

"Duologue"

A moment of silence was observed in memory of Gus Hermann and Charlie Parker, both of whom recently passed away.

Richard L. Connors, of Anna Maria College, addressed the topic of "DUOLOGUE."

According to Mr. Connors, duologue is when two people are talking but not listening to each other. "We do not listen . . . we talk" not dialogue, but duologue, said Mr. Connors. He explained that it is a personal distortion of the other person's world to fit our own. Actuality is nothing but each person's perception of it.

In business, managers become good communicators when they know their self-worth and have integrity.

Mr. Connors closed by stating if a situation is not productive, the key to changing it is communication.

KEVIN A. MULKERN, *Secretary*

NEW YORK..... JAN.

"Epoxy Resins"

Ronald S. Bauer, of Shell Development Co., presented an overview of "EPOXY RESINS."

Dr. Bauer specifically addressed recent advances being made in the area of new epoxy resin coatings and some of the new resins.

KENNETH J. DEPAUL, *Secretary*

NORTHWESTERN ..... MAR.

"Quality Symposium"

Lowell Wood, of Frost Paint and Oil Corp., was announced as a Federation Honorary member for his years of service to the organization.

Four individuals addressed the subject of "QUALITY" at the Society's symposium/meeting.

Ron Hamann, of 3M, discussed "MANAGING CHANGE THROUGH ANNUAL QUALITY IMPROVEMENT PLANS" and Dave Twedt, of Cargill, Inc., spoke on "MANAGEMENT LEADERSHIP, THE CORNERSTONE TO QUALITY IMPROVEMENT." "THE JOURNEY TO ZERO DEFECTS" was presented by Burt Thulin, of Tennant Co., and Wally Meyer, of McWhorter Chemicals, demonstrated that "QUALITY IS MORE THAN JUST IN VOGUE."

LARRY BRANDENBERGER, *Secretary*

OFFICERS OF THE MEXICO SOCIETY for 1984-85. (Seated, left to right): Vice-President—Angel Ruiz; President—Antonio Pina; Secretary—Rosa Maria Rojas; and Treasurer—Cuauhtemoc Pereda. (Standing): Jose Luis Padilla; Mauricio Esquivel; Gabriel Cicero; Rene Cruz; and Miguel Guevara

PIEDMONT..... JAN.

"Low VOC Polyurethane Coatings"

Carol Niemi, of Dow Chemical U.S.A., discussed "LOW VOC POLYURETHANE COATINGS FOR STRUCTURAL PLASTICS."

Ms. Niemi's presentation dealt with compliance coatings formulated with 1,1,1 trichloroethane and methelyne chloride to help achieve compliance with various clean air act regulations.

STEVE LASINE, *Secretary*

PITTSBURGH ..... APR.

"Cellosolve Acetate"

Chairman of the Nominating Committee, Michael Gillen, announced the nomination of officers for the 1985-86 term. They were: President-Elect—Anthony Isacco II, of Puritan Paint & Oil Co.; Secretary—Mark Troutman, Bradley Paint Co.; Treasurer—Ed Threlkeld, Ashland Chemical Co.; Director-At-Large—Morris Berger, Gateway Paint & Chemical Co.; and Society Representative—Ray Uhlig, PPG Industries, Inc. Joseph Mascia, of Campbell Chemical Co. will serve as President for the 1985-86 term.

Ron Reedshaw, of Union Carbide Corp., spoke on the "STATUS OF CELLOSOLVE ACETATE" and the "USE OF SOLVENTS AS ADDITIVES IN HIGH SOLIDS COATINGS."

There are no direct replacements for cellosolve acetate in all applications, stated Mr. Reedshaw. He then enumerated several possibilities for some applications: primary amyl acetate; PM acetate; and MAK—all of which are faster evaporating; and butyl cellosolve acetate which is slower. Due to a need for other replacements, explained Mr. Reedshaw, over 1000 solvent molecules were screened and eight were chosen as possible alternatives. PM acetate, propyl C.A. and PE acetate (which are not in n TSCA), butyl C.A., cellosolve propionate, primary amyl acetate, and n-butyl acetate comprised this list.

Mr. Reedshaw concluded this portion of his presentation by stating that if the federal government allows a TLV of five for cellosolve acetate, it will be used. If TLV is set at one or less, then cellosolve acetate will be replaced.

The second half of Mr. Reedshaw's presentation concerned the use of solvents as additives. He stated that the properties needed are: low density, proper boiling point, electrical resistance, low surface tension, and strong solvency.

ANTHONY J. ISACCO II, *Secretary*

ROCKY MOUNTAIN ..... MAR.

"Paint and Profit"

Al McMichael, of Universal Color Dispersions, spoke on "A SENSIBLE WAY TO PAINT AND PROFIT."

CRAIG R. HANSEN, *Secretary*

ST. LOUIS..... MAR.

"Organic Pigments"

Hugh Smith, of Sun Chemical Co., presented a lecture on "ORGANIC PIGMENTS."

Dr. Smith discussed the history and basic structures of organic pigments, as well as their growth in the coatings industry. He also described the classification into the five chemical groups, the changes that have been made, and how they have been accomplished.

MERLE D. HELD, *Secretary*

Elections

CHICAGO

Active

GIERKE, MARK A.—DeSoto, Inc., Chicago Heights, IL.
GIERMAN, CHUCK—DeSoto, Inc., Chicago Heights.
HAMIDANI, ABDULLAH—The Enterprise Cos., Wheeling, IL.
JACHIM, FRANK M.—Sherwin-Williams Co., Chicago, IL.
MARBRY, DAVID L.—Morton Chemical Co., Woodstock, IL.
POLLOCK, DAVID J.—W.E. Phillips Inc., Arlington Heights, IL.
PROSKEN, CAROL—Ace Hardware Corp., Matteson, IL.
SARA, PAUL S.—McWhorter, Inc., Carpentersville, IL.
TORPE, BILL—Dupli-Color Products Co., Elk Grove Village, IL.

Associate

ALLYN, JOHN—Whittaker, Clark & Daniels, Willowbrook, IL.
BISHOP, BILL—Cargill, Inc., Carpentersville, IL.
BRAINARD, G. DOUGLAS—Sun Chemical Corp., Naperville, IL.
LAWRENCE, RONALD A.—Donald R. Fitzgerald Co., Chicago, IL.
PHILLIPS, WILLIAM G.—BASF Wyandotte Corp., Appleton, WI.
TAIT, ROBERT W.—Union Carbide Corp., Chicago.
WINTERBURN, CHARLES H.—ArcoGraph Inc., La Grange, IL.

Educator/Student

BREITBEIL, FRED W. III—De Paul University, Chicago, IL.

GOLDEN GATE

Active

MATSON, ROBERT K.—Bay Area Air Quality Management District, San Francisco, CA.
REZNIK, DAVID—Tri/Valley Growers, Menlo Park, CA.
SHAFER, RANDY—International Paint Co., South San Francisco, CA.
WHALEY, DON—T.V.G. Container Div. Inc., Fremont, CA.

Associate

GOLDING, HARRY J.—Carmona Chemical Co., San Francisco, CA.
HOSOKAWA, MARVIN—Ishihara Corp., USA, San Francisco.
MOSS, ROSEMARY—Carmona Chemical Co., San Francisco.
POMPEO, BILL—American Cyanamid Co., Yorba Linda, CA.
RASMUSSEN, TOM—TCR Industries, Inc., Emeryville, CA.

KANSAS CITY

Active

BERTRAND, MERYL M.—Pratt & Lambert Inc., Wichita, KS.
HANN, CHARLES S.—Pratt & Lambert Inc., Wichita.
VARINA, DONALD R.—Hydrozo Coatings Co., Lincoln, NE.

Associate

HANNA, MARC W.—Solvent Supply Co., Riverside, MO.
WOOLSEY, DONALD K.—Thompson-Hayward Chemical Co., Kansas City, KS.

Retired

BARR, JOHN W.—Kansas City, MO.
HUTSLER, THOMAS E.—Kansas City.
OLIVER, HARRY—Shawnee Mission, KS.

LOS ANGELES

Active

DRISKO, RICHARD W.—Naval Civil Engineering Lab., Port Hueneme, CA.
FUNG, HARLEY—Benjamin Moore & Co., Commerce, CA.
GEORGE, DONALD J.—W.W. Henry Co., Huntington Park, CA.
GUEST, ALLEN—Swedlow, Inc., Garden Grove, CA.
JOHNSON, ROBERT A.—Consultant, Carson, CA.
MARTIN, EDWARD J.—Chemical Technology Labs, Inc., Lynwood, CA.
MUKAMAL, HAROLD—Swedlow, Inc., Garden Grove.
NELSON, WAYNE D.—Universal Paint Corp., Tempe, AZ.
ONSTEIN, BILL—Las Palmas Nursery, Compton, CA.
ROMERO, ROBERTO—Chemical Technology Labs, Inc., Lynwood.

Associate

BAK, JOANN P.—Rohm and Haas Co., Los Angeles, CA.
BOLLENBACH, DENNIS—S.C. Johnson & Son Inc., Los Angeles.
BOORN, GRAHAM M.—Stay and Day Paint Materials Co., Los Angeles.
DONOHOE, THOMAS D.—Pacific Coast Chemical, Los Angeles.
EMERSON, CLINT—Cargill, Inc., Lynwood, CA.
FINKLE, LARRY—U.S. Container Corp., Vernon, CA.
FISCHER, FRED—Diamond Shamrock Chemicals Co., Richmond, CA.
GODDARD, LAURAN—McKeeson Chemical Co., Commerce, CA.
HOLMES, BRIAN A.—Union Chemicals Div., La Mirada, CA.
JACKSON, ROBERT B.—Dow Chemical USA, City of Industry, CA.
JAKOSH, ROBERT C.—Spencer Kellogg Div., Long Beach, CA.
MERTZ, DIANA—Pacific Coast Chemicals, Vernon.
MORRILL, GAY A.—Hercules Incorporated, Walnut Creek, CA.
OBERSTAR, RONALD J.—Harshaw/Filtrol Partnership, Irvine, CA.
RUDOLPH, EDWARD—Pacific Dispersions Co., Inc., Cudahy, CA.
SAMPSEL, KATHLEEN—Triad Sales Co., Anaheim, CA.
STULL, AARON—Pacific Coast Chemicals, Los Angeles.
WEISSE, KARL H.—Pacific Dispersions, Cudahy.

NEW YORK

Active

RICHTER, RUDY—Con-Lux Coatings, Inc., Edison, NJ.
WAYNOR, HUGO—Pyrolac Corp., East Brunswick, NJ.

Associate

FELDMAN, JOSEF—EM Chemicals, Hawthorne, NY.
LEE, WILLIAM—Degussa Corp., Teterboro, NJ.
NAULTY, ROB—Colloids, Inc., Chatham, NJ.
OLLEY, PETER H.—EM Chemicals, Hawthorne.

NORTHWEST

Active

DREYER, JEFFREY H.—Valspar Corp., Minneapolis, MN.
TOBIAS, MICHAEL A.—Valspar Corp., Minneapolis.

PACIFIC NORTHWEST

Active

GNATOWSKI, JAN M.—Industrial Science Laboratory Inc., Richmond, B.C.

Associate

NORWALK, M.A.—Precision Cast Parts, Portland, OR.

KANSAS CITY SOCIETY OFFICERS for 1984-85. (Left to right): Treasurer—Steven D. Johnson, of Cook Paint & Varnish Co.; Secretary—Jerry P. Hefling, of Loctite Corp.; Society Representative—Norman Hon, of Cook Paint & Varnish; Vice-President—Gene Wayenberg, of Tnemec Co.; and President—Steve Bussjaeger, of Davis Paint Co.

SCHAFFER, RONALD J.—Armak Chemicals Ltd., Edmonton, Alberta.

PITTSBURGH

Active

BOYD, DONALD W.—PPG Industries, Inc., Allison Park, PA.

GRUBBS, H.C.—Mobay Chemical Corp., Pittsburgh, PA.

KENNEDY, COLLEEN J.—Mobay Chemical Corp., Pittsburgh.

RUSSELL, RANDALL L.C.—Ball Chemical Co., Glenshaw, PA.

SURESH, MATT—National Polymers Inc., Bethel Park, PA.

TANSKI, PAUL—PPG Industries, Inc., Springdale, PA.

Associate

KELLEY, DONALD J.—Whittaker, Clark & Daniels, McKees Rocks, PA.

SHEEHAN, PATRICK M.—Durr Marketing Assoc., Inc., Pittsburgh, PA.

WESTERN NEW YORK

Active

CLARE, MARY JANE—Pratt & Lambert, Inc., Buffalo, NY.

DAVIS, MARLENE L.—Spencer Kellogg Div., Buffalo.

FROMWILLER, JOHN K.—Spencer Kellogg Div., Cheektowago, NY.

HAKE, KARL L.—Spencer Kellogg Div., Buffalo.

HARDY, JOANNE R.—Pierce & Stevens, Buffalo.

LOMBARDO, KAREN—Spencer Kellogg Div., Buffalo.

STEFFAN, MICHAEL A.—Pratt & Lambert, Inc., Buffalo.

WIGDORSKI, ROBERT M.—Pierce & Stevens, Buffalo.

YOUNG, KELLY A.—Spencer Kellogg Div., Buffalo.

Associate

TIERNEY, WILLIAM—W.D. Tierney Inc., Cleveland, OH.

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People

William H. Ellis, a Past-President of both the Federation and the Los Angeles Society, retired May 1, 1985, after 35 years of service with Chevron Research Co., El Segundo, CA. For most of those years he was responsible for solvent research and technical service for the Chevron Corp. He was also a pioneer in working with regulatory agencies in solvent air pollution legislation.

Mr. Ellis was President of the Los Angeles Society in 1971–72 and General Chairman of the Western Coatings Societies Symposium and Show in 1974. After ten years on the Federation's Board of Directors, he was elected an officer and served as President in 1980–81.

Mr. Ellis has published several papers in the JCT, two of which were prize-winners in the Roon Awards competition: "Precise Control of Solvent Blend Composition During Evaporation"—Fourth Prize, 1971; and "Comparative Solvent Evaporation Mechanisms for Conventional and High Solids Coatings"—First Prize, 1981.

Clifford K. Schoff has been named to the position of Scientist at the Coatings and Resins Div. of PPG Industries, Inc., Allison Park, PA. Dr. Schoff is President of the Pittsburgh Society and Membership Secretary-Treasurer of the ASTM Committee D-1.

At the 101st Annual Convention of the Painting and Decorating Contractors of America, **George M. Lukas,** President of Southwest Painting, Inc., was elected President-Elect of the PDCA. Other officers elected included: President—**Lawrence E. Gove;** Vice-President—**Martin A. Berger;** Director of the Treasury—**Jon Leuteneker;** and Directors—**Richard Grund** and **William F. Johnson.**

Pratt and Lambert, Wichita, KS, has announced three promotions in the firm's Industrial Coatings Div. **Bill Ellison** has moved to the position of Operations Manager of the Wichita Div., **Tom Jewell** advanced to Group Leader of the Buffalo Industrial Coatings Laboratory, and **Dan Bernard,** Technical Director of the Wichita Div., has assumed additional responsibilities for the Buffalo Div. Mr. Bernard is a member of the Kansas City Society.

W.H. Ellis

R. Stanziola

W.L. Kallal, Jr.

G.E.F. Brewer

Ralph Stanziola, a founder and Executive Vice-President of Applied Color Systems, Inc., Princeton, NJ, has resigned from the firm to pursue interests in teaching, research, and consulting in the field of color science. Maintaining an affiliation with ACS as a consultant, Mr. Stanziola will assist the ACS Applications Support Group in the research and development of solutions to complex color control problems and will also conduct customer training seminars. Mr. Stanziola is a member of the Philadelphia Society. In 1981, he received the Armin J. Bruning Award for outstanding contribution to the science of color in the field of coatings technology.

Henley and Company, New York, NY, has added **William L. Kallal, Jr.,** to its sales force. Headquartered in Henley's Distribution Center in Elk Grove Village, IL, Mr. Kallal will serve as a Sales Representative for the midwest territory. Mr. Kallal is a member of the Chicago Society.

H.H. (Tex) Morris has retired from his post as Vice-President, Research and Development, for Freeport Kaolin Co., Gordon, VA, to reactivate the consulting firm of SSI Consultants, Macon, GA. Mr. Morris is a member of the Southern Society.

Bryan F. Knudsen has joined VIP Enterprises, Inc., Miami, FL, as Technical Services Manager. Mr. Knudsen's responsibilities will include developing distributor and representative training programs as well as handling product and application recommendations, specifications, and problems.

The Association for Finishing Processes of the Society of Manufacturing Engineers has honored **Dr. George E.F. Brewer** by awarding him the status of "Certified Manufacturing Engineer through Eminence in the Field of General Finishing." Dr. Brewer is a member of the Detroit Society.

The Inter-Society Color Council has elected three new members to its Board of Directors. **Jack J. Hsia, Justin L. Renilson,** and **Wolfgang Walter** will serve three year terms. Retiring members from the ISCC Board are **Dr. Fred W. Billmeyer, Jr., Rolf G. Kuehni,** and **Richard D. Ingalls.** Dr. Billmeyer is a member of the New York Society.

The Lord Corp., Erie, PA, has appointed **Charles Boehner** as Technical Sales Representative in the Industrial Coatings Div. Based in the St. Louis office, Mr. Boehner will be responsible for sales in the midwest territory.

Macbeth, a division of Kollmorgen Corp., Newburgh, NY, has promoted **Kenneth M. Briot** to Western Regional Sales Manager. Mr. Briot joined Macbeth in 1984 as a Manufacturers Representative.

Victor M. Willis is the Manager of Technical Services for the Paints Div. of Ace Hardware, Oak Brook, IL. Mr. Willis is a Past-President of the Chicago Society and has served on the Federation's Board of Directors. He is currently Chairman of the MMA Award Committee. [In the March JCT, we erroneously reported that Mr. Willis was a technical sales representative of Ace. Sorry, Vic.—*Ed.*]

To strengthen their market position, the Sun Chemical Corp., Cincinnati, OH, has realigned its Pigments Division. **Jack Gormley** has been appointed to the newly created position of Business Development Manager and **Peter A. Lewis** will succeed Mr. Gormley as Dry Color Business Manager. Both Mr. Gormley and Mr. Lewis are members of the CDIC Society. **Alan Boles** will assume responsibility as District Manager for the Southern Region. In addition, **Bobby J. Peaks** has joined the division as Southwestern District Manager, and **Kenneth C. Drilling** has been appointed as a Sales Representative.

Stephen R. Sides has joined the staff of the National Paint and Coatings Association, Washington, DC. Mr. Sides will serve as Manager of Health Affairs in the association's technical division. **Patrick J. Hurd,** who previously held the position, has been promoted to Legal Coordinator, Health and Safety.

Harvey C. Johnson has been named the Northern Regional Sales Manager with the Coatings Division of Ferro Corp., Cleveland, OH. Mr. Johnson will be responsible for sales of powder coatings in the northern region of the United States.

As part of its corporate expansion and realignment, Nalco Chemical Co., Oak Brook, IL, has announced the following organizational changes: **David E. Jackson** to the position of Executive Vice-President, U.S. Operations; **Keith V. Davis**—Executive Vice-President, Operations Staff; **C. Allen Smith**—Group Vice-President and President of a new Energy Chemicals Div.; **Edward J. Mooney**—Group Vice-President and President of the company's restructured Industrial Div.; **Roderick E. Eddy**—Senior Vice-President, Corporate Development; **W. Steven Weeber**—Vice-President, Marketing and **James F. Lambe**—Vice-President, Human Resources.

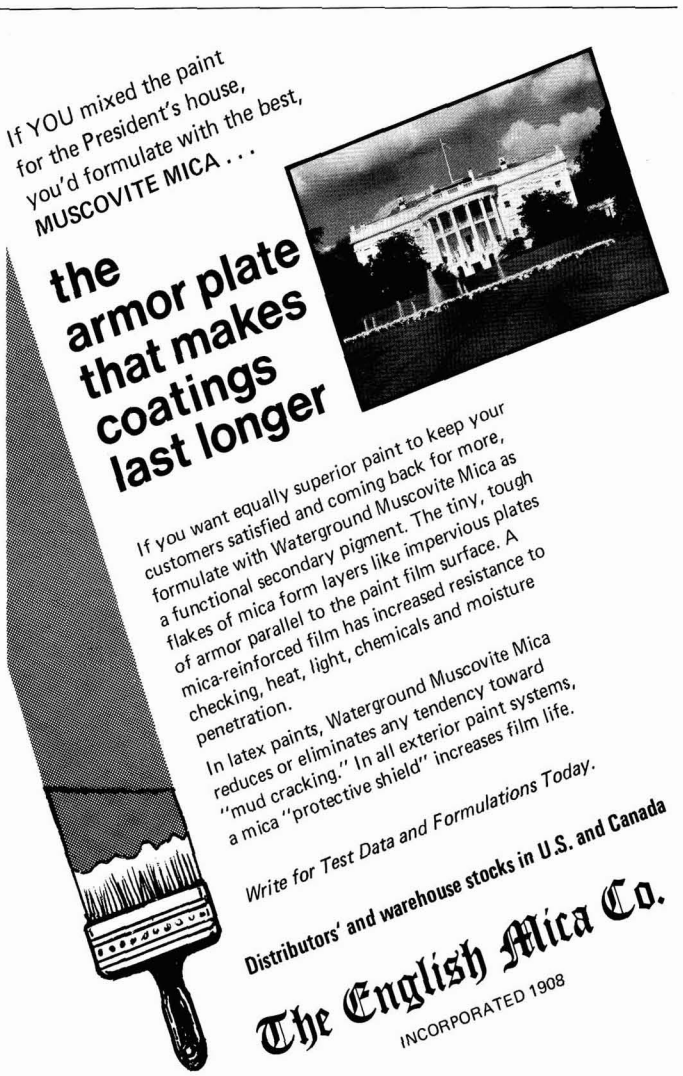
James V. Gates has assumed the office of Chairman of the Board and Chief Executive Officer of Mobile Paint Mfg. Co., Mobile, AL. **Robert A. Williams** is President and Chief Operating Officer of the firm.

John F. Wood has been named Vice-President of Manufacturing, Akzo Coatings America, Troy, MI. Mr. Wood's responsibilities include management of the Engineering, Health and Safety Department and supervising manufacturing procedures and performance at the company's plants. Mr. Wood is a member of the Detroit Society.

The Midland Div. of The Dexter Corp., Waukegan, IL, has announced the recipients of the 1985 David L. Coffin Award, granted annually by the firm to honor and award full time technical members who have made outstanding innovations. The recipients included: **Joseph A. Chess** and **Richard W. Clope** for their work on "High Corrosion Resistant Primer for Use on Hot Dipped Galvanized Steel and Galvalume," and **Michael D. Laffoon** for his work on "Water Reducible 'F' and 'C' Enamels for Food Cans." Honorable Mentions were granted to **Jean DuBois** and **Michel Hesling,** of Dexter's French Subsidiary, Societe des Vernis Bouvet, for their work entitled, "Glass Bottle and Jar Surface Coating," and to **Dr. Daniel L. Neumann** for "Glue System for Corrugated Board."

The Midland Division has promoted **John Rybicki** to Group Supervisor, Industrial Products Group. In his new capacity, Mr. Rybicki will be responsible for organizing, administrating, and evaluating the technical programs for the Cookware/Bakeware and One Component Urethane projects.

Kevin Bosman has been added to the Analytical Services Group of the Midland Div. Mr. Bosman brings four years of experience in industrial research to his new position.



Two Hundred Attend Southwestern Paint Convention; Loren B. Odell Awarded Fifty-Year Membership Pin

More than 200 persons attended the Southwestern Paint Convention from April 10-12, in Dallas, TX. The SWPC is a joint activity of the Dallas and Houston Societies for Coatings Technology. The event was held at the Anatole Hotel, which will serve as co-headquarters for the Federation's Annual Meeting and Paint Show in Dallas, 1987.

The highlight of the closing luncheon was the presentation of the Federation's 50-year membership pen and pin to Loren B. Odell, a founder of both the Dallas and Houston Societies.

General Chairman of the SWPC was Ross Frank, of D&F Distributing, Inc. The Program Chairman was Van Falcone, of Koppers Co., and President of the Dallas Society.

Program Presentations

Program presentations were:

"Economic Recovery of Solvent Vapors"—L. Durr, of DCI Corp.

"Paint Software for Micro Computers"—S. Phelan, of DataLogix.

"Recent Developments in Water-Borne Epoxy Chemistry"—P. Williams, of Celanese Specialty Resins.

"Small Media Milling System"—L. Dombrowski, of Chicago Boiler Co.

"Effect of Molecular Weight on Performance of Cellulosic Thickeners"—D. Blake, of Dow Chemical U.S.A.

"Organo Clays"—T. Powell, Jr., of United Catalysts, Inc.

"High Solids Thermosetting Systems"—V. Calder, of S.C. Johnson & Son.

"The Sandpiper Pump"—R. Bennett, of Warren Rupp-Houdaille, Inc.

"Business Conditions in 1985"—R. Carson, of Eastman Chemical Products, Inc.

Mr. Carson was given first prize as the Best Speaker at the SWPC. Second prize went to Mr. Dombrowski.

Mr. Odell

Mr. Odell, a well-known figure in the coatings industry for a half-century, was presented the 50-year mementos by Joseph A. Bauer, Federation President, and Frank J. Borrelle, Executive Vice-President.

A native of North Dakota and a graduate of NDSU, Mr. Odell joined the coatings industry in 1935, with the Davis Paint Co., Kansas City. He joined the Kansas City Society at the time.

Future positions in the industry were: Chief Chemist and VP for Manufacturing, Pratt Paint & Varnish Co., Dallas, six years; Research Chemist, Eagle-Picher Co., Joplin, MO., two years; Technical Director, Vice-President, and President, James Bute Co., Houston, 18 years; Director of Research and Technical Administrator, Napko Corp., Houston, 15 years.

Mr. Odell retired in 1978, but continues his involvement with the industry as a Consultant.

He served in all Chairs of both the Dallas and Houston Societies, and is an Honorary Member of Houston.

In the Federation, Mr. Odell has been a member of the Executive Committee and Board of Directors, Paint Show Chairman twice, Annual Meeting Program Chairman, Meetings Committee Chairman, and Houston Society Representative to the Board.

He has been an active participant in several other organizations, such as: NPCA, ACS, NACE, AIC, ASTM, Engineers Council of Houston, Texas Institute of Chemists, Kiwanis Club, and others.

1986 SWPC

The 1986 Southwestern Paint Convention will be held at the Wyndham Hotel (near Houston Intercontinental Airport), April 9-11.

Washington Paint Technical Group Celebrated 25th Anniversary in April

The Washington Paint Technical Group highlighted "Twenty-five Years of Commitment," at its 25th Annual Symposium, held on April 15-16, in Washington, D.C. Chaired by Kenneth L. Zacharias, of NPCA, the program provided for an exchange of information between the paint and coatings industry and representatives of government agencies interested in and responsible for specification development, quality assurance, and acquisition of a broad spectrum of coatings items. The following presentations were included:

"Business Outlook for 1985"—Dr. Ronald Utt, of U.S. Chamber of Commerce.

"Automotive Coatings Directions"—Dr. J. David Norstrom, of Ford Motor Co.

"In-House Solvent Recovery"—Michael J. Schmutzer, of Disti, Inc.

"Titanate and Zirconate Coupling Agents for High Performance Coatings and Composites"—Salvatore J. Monte, of Kenrich Petrochemicals Inc.

"Tailor-Made Amino Functional Epoxy Curing Agents"—Michael Landise, of Henkel Corp.

"Reflective Floor Coatings for Aircraft Hangars"—Dr. Peter J. Hearst, of Naval Civil Engineering Laboratory.

"Effect of Anaerobic Growth in Paints"—Dr. Robert A. Opperman, of Cosan Chemical Corp.

"Regulations Affecting the Coatings Industry"—Bruce Hamill, Esq., of National Paint and Coatings Association.

"Recent Research and New Standards at SSPC"—Dr. Bernard Appleman, of Steel Structures Paint Council.

"What's Happened to Waterborne Coatings"—Nicholas Roman, of Rohm and Haas Co.

"Chlorinated Rubber Paint Systems for Structural Steel—Twelve Years Later"—Dean M. Berger, Gilbert/Commonwealth, Inc. and Rufus F. Wint, of WIND Associates.

"Use of Titanium Pigments in High Solids Coatings"—Calvin C. Tatman, of SCM Pigments.

"Fire Retardant Materials Program of NAVSEA"—John J. Tock, of Naval Sea Systems Command.

During the Symposium, WPTG President, John M. Montgomery, of Verlan Limited, presented Mr. Zacharias with the "Man of the Year" Award in recognition of his many contributions to the organization.

DePaul Univ. Institutes Masters Degree Program in Coatings Technology

The Chemistry Department of DePaul University, Chicago, IL, has organized a Master of Science degree program in coatings technology to begin in fall of 1985. The main objectives of the program are to satisfy the demand for technical professionals in the coatings industries at an advanced level and to provide an opportunity for Bachelor of Science level coatings chemists in the Chicago area to enhance their knowledge and skill for improved levels of performance and advancement in salary and rank.

Admission to the program requires admission to the DePaul Graduate School. Candidates should have earned a B.S. degree in chemistry or its equivalent. The 12 course curriculum, which will require about six quarters of effort, includes two advanced courses each in organic chemistry, inorganic chemistry and physical chemistry; three courses in polymer chemistry; and three courses in coatings technology, one of which is a coatings laboratory.

The Chemistry Department at DePaul has a full-time faculty of ten persons, two of whom have been teaching the physical chemistry of polymers and polymer synthesis for 12 years. Coatings chemists from local industries teach the specialized courses. The Chemistry Department is located on the Lincoln Park Campus and graduate courses are all taught in the evening.

The Coatings Technology Program at DePaul University has received the endorsement and active support of the Chicago Society for Coatings Technology.

For more information and for application to the Graduate School at DePaul University, write to Dr. Jurgis A. Anysas or Dr. Fred W. Breitbeil III, at DePaul University, Dept. of Chemistry, 1036 West Belden Ave., Chicago, IL 60614.

ACS Sponsors Color Technology Seminars

Applied Color Systems, Inc., Princeton, NJ, has scheduled a series of color technology seminars to be held in the U.S. and Latin America throughout 1985. Under the direction of Wes Coppock, ACS Director of Educational Services, the seminars will cover wide ranging topics of practical interest to both management and technical personnel.

Each seminar will be organized in five sessions. Session I will deal with color physics and will cover such topics as additive and subtractive color mixing; metamerism; spectrophotometry and colorimetry; and metallic and pearlescent effects. Session II will focus on color specifications, examining acceptability of color vs perceptibility, and the establishment of color quality assurance parameters.

Turbid media theory will be explored in the third session. Specific steps involved in color matching will be discussed, along with techniques for determining absorption and scattering co-efficients and the proper development and maintenance of accurate color data files in computer systems.

Session IV will deal with instrumental color control, and Session V will cover special topics such as the aesthetics of color, the effect of gloss on perceived color, and uses of computer color control systems for inventory control and batch ticketing.

The North American seminar schedule includes the following: July 1-3—Myrtle Beach, SC; Aug. 21-23—Cape Cod, MA; Sept. 30-Oct. 1—Toronto, Canada; Oct. 3-4—Montreal, Canada; and Dec. 18-20—Orlando, FL.

For additional information, contact Wes Coppock, Applied Color Systems, Inc., 2848-M Carolina Center, I-85 South, Charlotte, NC 26208.

Daniel Products Co. Holds Seminars in Far East

Daniel Products Co., Jersey City, NJ, recently held technical seminars in Singapore and Jakarta, Indonesia. Both two-day sessions were attended by principal chemists from leading paint and ink companies of Singapore, Indonesia, Malaysia, Thailand, Hong Kong, and Taiwan. The seminars, designed to reach overseas technical audiences, were organized by Harry Langeslag, of Southeast Asia for DSM Resins, BV, The Netherlands. DSM Resins is the master distributor for Daniel Products' line of pigment dispersions and specialty additives in parts of Europe, Africa, South America, and Asia.

Corrosion Short Course Offered at Lehigh Univ.

The 14th Annual Corrosion Short Course will be held at Lehigh University in Bethlehem, PA, on July 15-19.

Entitled "Corrosion and Its Control by Protective Coatings," the course will focus on corrosion principles and the mechanisms of corrosion beneath paints, metallic coatings and inorganic coatings. Additional information may be obtained by contacting Prof. Henry Leidheiser, Jr. or Dr. Richard Granata, Sinclair Laboratory #7, Lehigh University, Bethlehem, PA 18015.

CALL FOR PAPERS

Ninth Annual Meeting of The Adhesion Society

The Ninth Annual Meeting of the Adhesion Society will be held on February 9-12, 1986 at the Marriott Hotel on Hilton Head Island, SC. Session topics and organizers include:

THIN FILM ADHESION: MICROELECTRONICS TECHNOLOGY (Dr. P. Ho, IBM, P.O. Box 218, Yorktown Heights, NY 10598).

BOND DURABILITY AND SURFACE ANALYSIS (Dr. A.V. Pocius, 3M Co., Bldg. 209-1C, St. Paul, MN 55144).

EVALUATION OF ADHESION TEST METHODS (Dr. H.F. Brinson, Virginia Tech, ESM Dept., Blacksburg, VA 24061).

ADHESIVE SYNTHESIS AND FORMULATION (P.M. Hergenrogher, NASA-LaRC, M.S. 226, Hampton, VA 23665).

GENERAL PAPERS (Dr. R.A. Draughn, Medical Univ. S.C., Materials Sci. Dept., Charleston, SC 29425).

Persons interested in presenting papers in the above areas should contact the respective organizers directly. Paper titles are due July 1 and abstracts are due on October 1.

Further details on the meeting can be obtained from Program Chairman, Dr. J.P. Wightman, Virginia Tech., Chem. Dept., Blacksburg, VA 24061.

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Introduced in literature is ethyl 3-ethoxypropionate, a solvent compatible with a wide range of resins. This solvent features an evaporation rate which is suitable for both air-dried and baked coatings. For additional information, as well as sample and trial quantities, contact J.J. Jones, New Products Div., Eastman Chemical Products, Inc., P.O. Box 431, Kingsport, TN 37662.

Dispersion

A new highly pigmented water-based lampblack type dispersion is featured in a brochure. The product contains 50% pigment in a non-ionic type dispersant system and can be employed in a variety of aqueous applications. Write to Merril D. Garret, CDI Dispersions, 27 Haynes Ave., Newark, NJ 07114 for brochures and samples of Black Shield #12716.

Pumps

A redesigned, "state of the art" air distribution system focusing on optimizing air flow is the subject of a recently released brochure. The system provides up to 13% greater pumping capacity and reduced air consumption averaging over 10% across the entire performance range. For further details on the "Supersaver," contact Wilden Pump & Engineering Co., P.O. Box 845, Colton, PA 92324.

Solvents Guide

A 12-page booklet describes a wide range of solvents used in the formulation of higher solids coatings. Covered in the guide are ketones, esters, alcohols, and glycol ethers. A comparison of the characteristics of low solids and higher solids is also included. Copies of "UCAR Solvents for Higher Solids Coatings" can be obtained from Union Carbide Corp., Solvents and Coatings Materials Div., Dept. K3442, 39 Old Ridgebury Rd., Danbury, CT 06817.

Hexyl Alcohol

Featured in literature is a new proprietary primary alcohol. Reported to be an economical alternative to n-hexanol, it also has potential as a slow evaporating solvent for inks. Write to Union Carbide Corp., Solvents and Coatings Materials Div., Dept. K3442, 39 Old Ridgebury Rd., Danbury, CT 06817, for details on UCAR Hexanol.

Analyzer

A new particle size analyzer is detailed in a product bulletin. Determining particle size from 150 to 0.1 micrometers diameter using photoextinction as the detection method, this instrument provides rapid measurement of the particle size distribution of fine powders or slurries. Further information on the MicroSizer 5300 is available from Micromeritics Instrument Corp., 5680 Goshen Springs Rd., Norcross, GA 30093.

Thixotropic Agents

A full-color, 16-page brochure emphasizes the benefits of liquid and gel thixotropic additive systems. Explained in the brochure are the handling, blending, and processing advantages of the liquid and gel agents over solid systems. Suggested applications and examples are also included. For a copy of product brochure #1084 362-5R1 write: Lubrizol Corp., Diversified Products Group, 29400 Lakeland Blvd., Wickliffe, OH 44092.

Cavity Pumps

A two-page data sheet describes a new series of mini progressive cavity pumps. The pumps are suited to metering a wide range of materials as well as pumping low volumes of shear sensitive and high viscosity/high solids content materials, sludges, abrasives, and heavy pastes. For copies of the data sheet, contact Netzsch Inc., 119 Pickering Way, Exton, PA 19341.

Grinding Mills

Data sheets, outlining a series of small media grinding mills, have recently been published. The sheets describe the grinding system and list the mill's extensive applications: processing of printing inks; dyestuffs; food/pharmaceuticals; paints; pigments; magnetic coatings; minerals; and ceramics. Further details are available from Netzsch Inc., 119 Pickering Way, Exton, PA 19341.

Dispersions

Two new lines of color dispersions for industrial coatings are discussed in a product bulletin. One dispersion is compatible in solvent-borne systems, and the other is designed for water-borne systems. For technical information and samples, contact Pacific Dispersions, Inc., 4615 Ardine St., Cudahy, CA 90201.

MSDS Software

The benefits of a new software package are outlined in literature. Designed to eliminate paper work and calculations involved in meeting the reporting obligations of the OSHA Hazard Communication Standard, the package produces a comprehensive 10-section Material Safety Data Sheet in a short amount of time. Currently the package is designed for the IBM PC and DEC PDP-11 minicomputers. For additional information on the MSDS program, contact Parallax Computer Corp., P.O. Box 3357, 3490 Route 1, Building 19-1, Princeton, NJ 08540.

Goniophotometer

The application and use of a new goniophotometer are included in a brochure. The instrument allows researchers and manufacturers to study the light scattering phenomenon of materials and products in two or three dimensional analysis. The goniophotometer has uses in the paint, paper, plastics, film, audio and video tape, cosmetics and hair care, electronics, and chemicals industries. Write to HunterLab, 11495 Sunset Hills Rd., Reston, VA 22090 for further details on GP-1R.

Industrial Meters

An eight-page, two-color brochure describes the key features and benefits of industrial meters. The Illuminance Meter, Luminance Meter 1; Chroma Meter II Reflectance, Chroma Meter II Incident, and TV Color Analyzer II are discussed in the brochure. For copies, contact John T. McCasland, Minolta Corp., 101 Williams Dr., Ramsey, NJ 07446.

Storage Tanks

Cone bottom, vertical liquid storage tanks designed to withstand maximum loads and stresses are outlined in a brochure. Technical specifications on the tanks can be obtained from Certified Equipment & Mfg. Co., P.O. Box 298, Springfield, IL 62705.

Mixers

A four-page brochure describing a line of double planetary mixers is now available. Covering nine models, the brochure includes product specifications such as dimensions, void and working volumes, HP, orbit speeds, and blade speeds. Write to Premier Mill Corp., 220 E. 23rd St., New York, NY 10010 for copies of the brochure.

Grinding Mills

Data sheets outlining the full line of production size grinding mills have been published. The sheets describe the milling systems which are designed for grinding high solids and high viscosity materials containing hard to grind pigments. For copies on the John Small Media Grinding Mills contact Netzsch Inc., Grinding Dept., 119 Pickering Way, Exton, PA 19341.

Latex

Performance capabilities of a new high solids acrylic emulsion are described in a brochure. Weather-barrier coatings with good application properties and durability can be formulated with the latex because of its resistance to water. The new emulsion is also recommended for caulks, sealants and mastics. Request copies from: Union Carbide Corp., UCAR Emulsion Systems, Dept. K3442, 39 Old Ridgebury Rd., Danbury, CT 06817.

Resins Study

A brochure listing the tentative table of contents of a new techno-economic multiclient study entitled, "Tackifying Resins" has been released. The study will provide technical, economic and marketing information on tackifiers, their raw materials and end uses, major suppliers, users, and market shares. Statistical projections to 1990 will also be included. For the free brochure, contact Skeist Laboratories, Inc., 112 Naylon Ave., Livingston, NJ 07039.

Colorants

Outlined in a pamphlet are the properties of an expanded line of colorants. The colorants are available for a wide range of vehicle systems used in the automotive, textile, coating, plastics and other industries. They are also available in a complete range of solid and liquid pigments for all processing methods. Inquiries for copies should be sent to Raymond F. Wardle, Permuthane Polymers, Corwin St., P.O. Box 3039, Peabody, MA 01960.

Hazardous Waste Directory

A directory identifying 121 commercial facilities licensed to treat, store, and dispose of hazardous wastes has been published. Compiled from an annual survey conducted in February 1985, the directory offers pertinent information on each facility as well as results of a survey to determine the outlook for new facilities. Single copies are available for $75 and can be ordered from McCoy and Associates, 13131 West Cedar Dr., Lakewood, CO 80228.

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A booklet covering 16 zircoaluminate products varying in organo-functionality and solvent base has been published. All the products are added directly to the resin and/or solvent grind and no pretreatment is necessary. Further details can be obtained from Lawrence B. Cohen, Cavedon Chemical Co., Inc., P.O. Box 329, Woonsocket, RI 02895.

Hazards System

A Hazardous Materials Identification System designed by the National Paint and Coatings Association is now available. The system includes labels, wallet cards, posters, implementation manual, MSDS Guide, raw materials rating manual, and a slide/tape program for employee education and training. To order a set, contact Labelmaster, Customer Service Dept., 5724 N. Pulaski Rd., Chicago, IL 60646.

Color Control

A process image color control system has been introduced in literature. The system allows users to directly measure color images of their product. Included in the system are an optical sensor, designed for reflectance color measurement, and a data processor for direct measurement of the various instruments. For applications information, write to Hunter Associates Laboratory, 11495 Sunset Hills Rd., Reston, VA 22090.

Storage Tanks

Performance specifications and technical details of stainless steel storage tanks are outlined in a data sheet. The ribbed tanks have 14 gauge shells, cone tops, and 12 gauge cone bottoms to withstand maximum loads without rupture or spills. For further information on the Type 304 tanks, write to Certified Equipment & Mfg. Co., P.O. Box 298, Springfield, IL 62705.

Hazards Report

The National Institute for Occupational Safety and Health has recently issued a report entitled, "Recommendations for Control of Occupational Safety and Health Hazards . . . Manufacture of Paint and Allied Coating Products." The report provides guidance for protecting workers involved in the manufacture of paint and allied coating products. Send inquiries for copies to Publications Dissemination, NIOSH, 4676 Columbia Pkwy., Cincinnati, OH 45226.

Color System

An on-line color measurement system designed to measure the color and appearance of raw materials and finished products on the processing or production line is the subject of a new product bulletin. Incorporating a modulated light source and fiber optical probes, the system provides a flexible measurement system free from stray light. For detailed technical information on the Dynagard System, write to Pacific Scientific, Gardner/Neotec Instrument Div., 2431 Linden Ln., Silver Spring, MD 20910.

Viscometer System

The simplified set-ups and complete software control of a rotational viscometer provided by a new computer controlled viscometer system are described in a bulletin. The system enables researchers to perform flow curves, constant shear rate, viscosity/time, and viscosity/temperature tests easily and quickly. Various software programs are available for measurements and data evaluation. Contact Darren Bronen, Haake Buchler Instruments, Inc., 244 Saddle River Rd., Saddle Brooke, NJ 07662 for copies on the Rheocontroller.

Coatings Gauge

Performance specifications as well as special features of a coatings thickness gauge are outlined in a brochure. The gauge is designed for use in paint, electroplating, galvanizing, and metal coatings. For copies of the four-color brochure, contact Elcometer Inc., P.O. Box 1203, Birmingham, MI 48012.

Stainless Steel Tanks

A new line of stainless steel tanks and process vessels has been featured in literature. Available in a wide range of standard sizes and configurations, the tanks offer ease of cleaning and complete drainage. Contact Robert Richman, Stainless Metals, Inc., 43-49 10th St., Long Island City, NY 11101.

Black Colorant

Highlighted in a product bulletin are the anti-settling characteristics of a new black colorant for thermoplastics. Formulated for injection molding and blow molding, the colorant exhibits superior masstone and jetness. Contact James Steever, Pigment Dispersions Inc., 54 Kellogg Court, P.O. Box 412, Edison, NJ 08818 for technical data.

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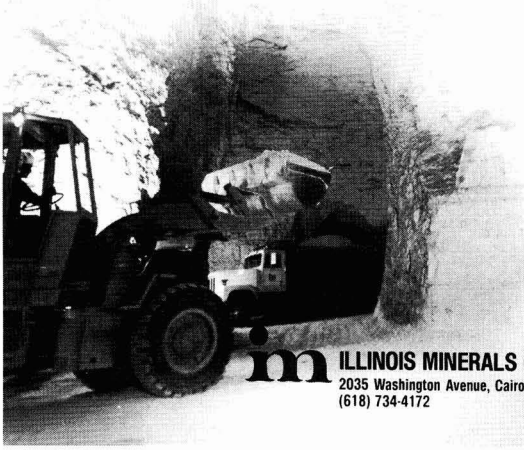

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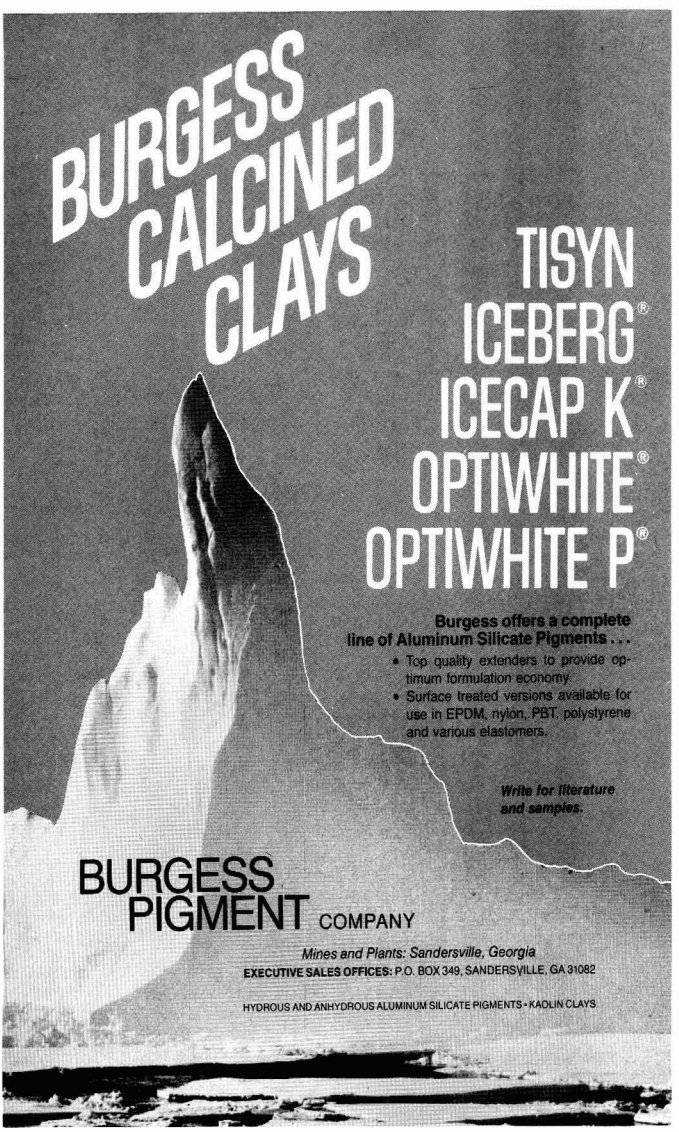
Letters to the Editor

Metric Conversion—An Idea Whose Time Has Come?

TO THE EDITOR:

In reading the article by Earl Hill on unit conversions, (JOURNAL OF COATINGS TECHNOLOGY, Open Forum, January 1985, p. 73) one is impressed primarily by the amount of effort required to manipulate so called "English" units, when the much simpler metric system is available. With the further complication of mixed metric and "English" units, the whole arrangement seems jerry-built and absurd. Useful as Mr. Hill's article might be under present circumstances, I think the kind of effort it represents would be better expended toward the conversion of our industry to the exclusive use of metric units.

LEONARD SCHAEFFER,
The Leneta Company
Ho-Ho-Kus, NJ



TO THE EDITOR:

I appreciate the comments of Mr. Leonard Schaeffer of The Leneta Co. with reference to my article on metric conversion charts.

From a personal viewpoint, I have used only metric units for about the last five years. I can now even say degrees Celsius without thinking. Although I totally agree that we should use the metric system exclusively, my experience indicates that this is not happening in a few of our labs, many of our production facilities, and with only a few exceptions, not with our ultimate customers. It was for this reason that I prepared the charts in the article with both metric and English units so that interested users would have a choice. Partly it was for an update, since previous charts of this type have been mostly all English conversions. The equations were included so that persons with an active interest in conversions could program them into a pocket calculator or personal computer for more exact results. The graphs, of course, give a good overall picture of conversion but suffer somewhat in accuracy due to their very nature, even though they were done by computer.

Overall, I think it would be very informative if someone could institute a survey to track just what degree of both "soft" and "hard" conversion is taking place in our industry—in the laboratory, marketing, production and with customers. I know of no such information. Perhaps this would be a good project for the NPCA or the FSCT.

It would also be informative to begin keeping a record of the use of both "soft" and "hard" metric conversions. Perhaps then we could see if, over a period of years in our industry, the use of the metric system is increasing, decreasing, or staying about the same. A tendency toward the last two situations would be cause for concern and could signal the need for increased action to implement conversion.

Unfortunately, to be perfectly realistic, I fear that metric conversion in the United States is a good idea whose time has not really come and whose acceptance is only problematical. We will probably all be Methuselah's before it comes about at anything close to universal acceptance.

H. EARL HILL
The Lord Corporation
Erie, PA

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Book Review

ENCYCLOPEDIA OF CHEMISTRY

Edited by
Douglas M. Considine, P.E.
and Glenn Considine

Published by
Van Nostrand Reinhold Co.
New York, NY
1082 pages, $89.50

Reviewed by
Dr. Thomas J. Miranda
Whirlpool Corp.
Benton Harbor, MI

This fourth edition of the popular encyclopedia, begun in 1956, has been completely revised. The editors have made every attempt to include new material, more than doubling the content of the previous issue. As a result, greater attention was used in selecting wordage and using more illustrations.

While the book may serve the chemical needs of chemists, it offers the opportunity to search for interdisciplinary fields, i.e., metals, energy sources, plant chemicals, waste and pollution, and a host of topics. This volume should be a useful desk reference.

ORGANIC COATINGS SCIENCE AND TECHNOLOGY VOL. 6

Edited by
Geoffrey D. Parfitt and
Angelo V. Patsis

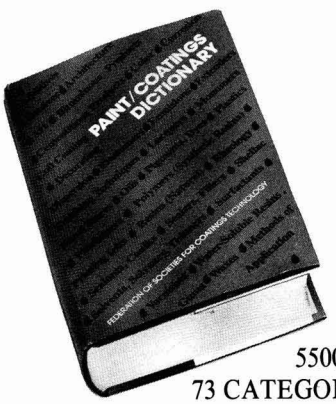
Published by
Marcel Dekker, Inc.
New York, NY
534 pages, $75.00

Reviewed by
Dr. Thomas J. Miranda
Whirlpool Corp.
Benton Harbor, MI

This volume is the sixth in the series of collected papers presented annually at the Athens Conference which features papers from the Eighth International Conference in Organic Coatings Science and Technology and includes results obtained from 24 invited and three contributed papers.

Included in this volume are the latest developments in Organic Coatings presented by leading experts in the field. Emphasis includes works on pigments, solvents, high solids, epoxies, alkyds and clear coatings. Also included are new approaches to corrosion protection and surface analytical instrumentation and its use in solving coating problems.

Like its predecessors, this book provides the coatings technologist an excellent review of the current scientific and developmental advances being made in the field. This volume is recommended for those seeking an update on coatings developments.



If you are at all involved with matters dealing with organic coatings, rush an order for this book.
Metal Finishing

Highly commended and will undoubtedly be needed in all paint libraries.
World Surface Coatings Abstracts

One's admiration grows page by page. The work involved, the care and attention to detail are truly admirable.
Australian Oil and Colour Chemists' Proceedings and News

This monumental dictionary is useful to all interested in paint, and who isn't? It is useful to veteran technologists, new scientists emerging from universities, and secretaries who need spelling guides.
ASTM, Standardization News

The book is a landmark in its field.
Chemical Marketing Reporter

One of the most important and appreciated books ever published for the paint and coatings industry.
Western Paint Industry

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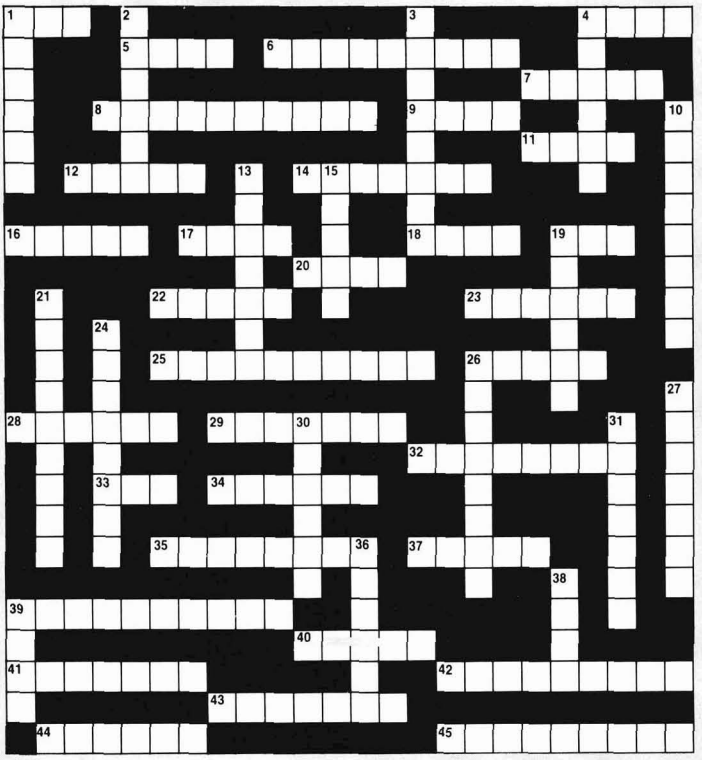
by Earl Hill

Famous Names, Famous Faces



Solution to be published in July Issue

No. 6

ACROSS

1. *Flash Point tester*
4. Known for color theory
5. Common refractometer
6. Paint scientist pioneer
7. Abrasion equipment, e.g.
8. Reflective metallic pigments
9. Classic resin tradename
11. _____ gel (T.N.)
12. Type of hardness (K_____)
14. Famous calcium carbonate
16. Curing agents (C_____L)
17. Polymers are his life (M_____)
18. _____atrol
19. Known for emulsion work
20. *Flow cup*
22. Cryptometer
23. The large family company (Ohio)
25. *Temperature person*
26. Urethane pioneer
28. Solubility Parameter pioneer
29. Let there be light
32. Classic flow control agent
33. Famous vendor (Eng. origin) (Abr.)
34. White stuff (T.N.)
35. _____ unit
37. Scientist Antoine
39. Viscosity, e.g.
40. Research institute head
41. Emulsion named for a Greek letter (T.N.)
42. Polymeric thickener *supreme* (T.N.)
43. Pigment trade name (H_____E)
44. Electrocoat pioneer (B_____)
45. Classic defoamer

DOWN

1. Heavy slippery powder/plastic
2. Known for earth pigments
3. Reagent for reducing sugars (An. Chem.)
4. Rheological theorist
10. Phila. JCT person (B_____)
13. The equipment people
15. Another type of hardness
19. Color & appearance *pioneer*
21. Calcium silicate named after him (W.H.W.)
24. Plastic can (T.N.)
26. So. European turpentine
27. Tom M_____ (JCT)
30. Type of hexagon cell
31. T_____ Hydrometer
36. The chart people
38. Well known Microknife
39. Drawdown bar

Coming Events

FEDERATION MEETINGS

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

(Oct. 7-9)—63rd Annual Meeting and 50th Paint Industries' Show. Convention Center, St. Louis, MO.

1986

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

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

SPECIAL SOCIETY MEETINGS

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

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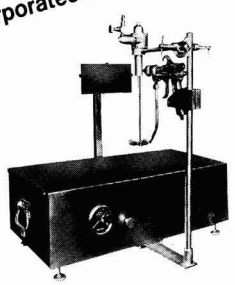

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(Apr. 2-5)—Southern Society. Annual Meeting. DeSoto Hilton Hotel, Savannah, GA. (Ronald R. Brown, Union Chemicals Div., P.O. Box 26845, Charlotte, NC 28213).

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

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

1987

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

OTHER ORGANIZATIONS

(June 26-29)—Oil & Colour Chemists' Association's Biennial Conference. Edinburgh, Scotland. (R.H. Hamblin, OCCA, Priory House, 967 Harrow Rd., Wembley, Middlesex, HAO 2SF England).

(July 4-7)—Oil and Colour Chemists Association Australia. 27th Convention. Hunter Valley, NSW, Australia. (Ted Saultry, 46 Tamarisk Ave., Glen Waverly VK.3150, Australia).

(July 7-10)—ASTM Committee D-1 on Paint and Related Coatings and Materials. Town and Country Hotel, San Diego, CA. (Brent Backus, ASTM, 1916 Race St., Philadelphia, PA 19103).

(July 8-12)—11th International Conference on Organic Coatings Science and Technology. Athens, Greece. (Dr. A.V. Patsis, Institute in Materials Science, S.U.N.Y., New Paltz, NY 12561).

(July 15-19)—Short Course on "Corrosion and Its Control by Protective Coatings." Lehigh University, Bethlehem, PA. (Prof. H. Leidheiser, Jr., Sinclair Lab. #7, Lehigh University, Bethlehem, PA 18015).

(Aug. 26-28)—Advanced Colorimetry Course. Munsell Color Science Laboratory, Rochester Institute of Technology campus, Rochester, NY. (Martha Pschirrer, Munsell Color Science Laboratory, RIT, P.O. Box 9887, Rochester, NY 14623).

(Sept. 2-4)—Federation of Scandinavian Paint and Varnish Technologists. 11th Congress. SAS Hotel Scandinavia, Oslo, Norway. (Paal Ivan, Nodest Industries A/S, Boks 500, N-3001 Drammen, Norway).

(Sept. 2-6)—Short Course on "Films and Coatings for Technology." Davos Congress Center, Switzerland. (Continuing Education Institute—Europe, Rortorpsvagen 5, S-612 00 Finspang, Sweden).

(Sept. 9-13)—Short Course on "Nitride and Carbide Coatings." LSRH Research Center, Neuchatel, Switzerland. (Continuing Education Institute—Europe, Rortorpsvagen 5, S-612 00 Finspang, Sweden).

(Sept. 16-19)—Fifth Finishing Conference and Exposition of the Association for Finishing Processes of the Society of Manufacturing Engineers. Cobo Hall, Detroit, MI. (Public Relations Dept., AFP/SME, One SME Dr., P.O. Box 930, Dearborn, MI 48121).

(Sept. 17-19)—FINISHING '85. Cobo Hall, Detroit, MI. (AFP/SME, P.O. Box 930, Dearborn, MI 48128).

(Sept. 21-24)—Canadian Paint and Coatings Assn. 73rd Annual Convention. Clevelands House, Minnett, Ont. (CPCA, 515 St. Catherine St. W., Montreal, Que., Canada H3B 1B4).

(Sept. 30-Oct. 2)—12th International Naval Stores Meeting. Westin Peachtree Plaza Hotel, Atlanta, GA. (Douglas E. Campbell, Executive Director, Pulp Chemicals Assn., 60 E. 42nd St., New York, NY).

(Oct. 8-11)—XVIth Congress of AFTPV (French Assn. of Paint and Varnish Technicians), Strasbourg, France. (Congress Secretary, B.P. 1036, 68050 Mulhouse Cedex, France).

(Oct. 14-18)—European Conference on Applications of Surface and Interface Analysis. Veldhoven, The Netherlands. (ECASIA Conference Bureau: QLT Convention Services, Keizersgracht 792, 1017 EC Amsterdam, The Netherlands).

(Oct. 15-17)—ASTM Committee B-8 on Metallic and Inorganic Coatings. ASTM Headquarters, Philadelphia, PA. (Teri Carroll, ASTM, 1916 Race St., Philadelphia, PA 19103).

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

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

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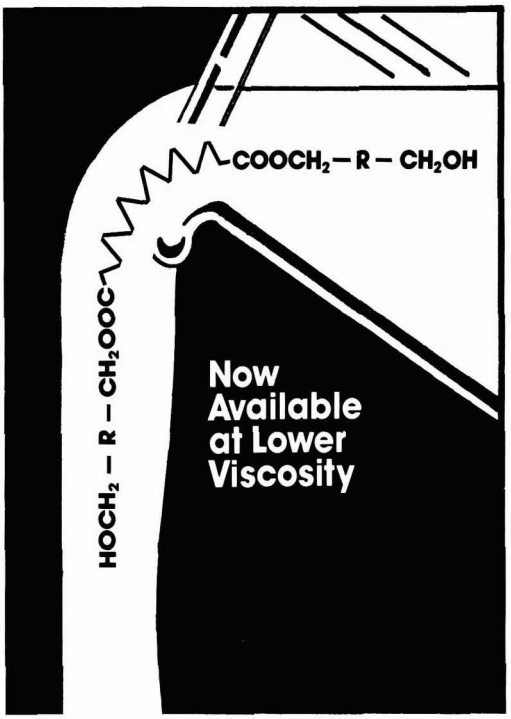

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'Humbug' from Hillman

Christopher George of Canada found some hilarious reprints from "Zen and Now, the Baby Boomers Guide to Middle Life" in the publication, *Toronto Life.* Here follows some expurgated reprints of the reprints. My easily shocked editors may have, worriedly, expurgated a few more. Your loss!

Then, you . . .	**Now, you . . .**
• Loved sex and drugs and rock and roll	• Love cheques and hugs and Manilow
• Drove Dad's car	• Drive the company car
• Drank cheap wine with good friends	• Drink good wine with cheap friends
• Bought black-light posters	• Buy Tibetan art
• Decorated with wicker	• Live with your interior designer
• Admired Abbie Hoffman	• Admit reading "Dear Abby"
• Ate steak and potatoes	• Recommend nouvelle cuisine
• Had to have The Answer	• Would rather not know
• Said you had been at Woodstock	• Pretend you don't buy oil stocks
• Cared about job placement	• Read The Book of Job
• Tried to sleep on waterbeds	• Try to sleep on futons

I Am Almost 40 When . . .

It suddenly feels right to wear a cardigan.

I use the stairs to get in and out of the swimming pool.

I can get all the news I need by reading the headlines.

I explain how to do things better than I actually do them.

A martini tastes good.

I notice a fad or a dress style come back for the third time.

I defend Joan Baez.

I tell people I'm older than I actually am just to hear how good I look for my age.

I wear shorts only around the house.

I know my pharmacist's first name.

A Role By Any Other Name

Social and political changes notwithstanding, there are some things that are a constant part of life. But now they go under different names.

When Women Did It	*Now That Men Do It Too:*
• Cooking	• Gastronomy
• Child rearing	• Parenting
• Housework	• Domestic duties
• Typing	• Word processing
• Budgeting	• Financial management

• • •

Herman Singer, a long time New York Society friend, now escaped to Florida, found these "Thoughts on the Business of Life" in Forbes Magazine.

- Expecting something for nothing is the most popular form of hope.—Arnold Glascow
- Habit is the nursery of errors.—Victor Hugo
- Sow an act and you reap a habit.—G.D. Boardman
- You can't pick cherries with your back to the tree.—J.P. Morgan
- Whilst he sits on the cushions of advantage, he goes to sleep.—Emerson

Well, Herm, I published them but, frankly, I find homilies like these somewhat nauseating. Can't you just visualize those pompous gentlemen (Morgan with a midget on his knee) exchanging the above sage pleasantries in the Exclusive Club of the Sky?

• • •

From the *Del-Chem Bulletin,* Joe Vasta spotted these—

A man and his dog sat in a theater watching a movie. When the movie ended, the dog applauded vigorously, which caused a nearby spectator to remark in amazement, "How astonishing!"

"It sure is," said the dog's owner. "He hated the book."

Off the coast of Oregon, a ship collided with a fishing boat in heavy fog. No real damage was done, but as the offending ship tried to back off, it banged into the boat again. The captain was afraid that he might have done some damage with the second blow.

"Can you stay afloat?" he shouted through a megaphone to the foundering victim.

"I guess so," called back the skipper of the boat. "Do you want to try again?"

—Herb Hillman
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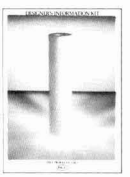

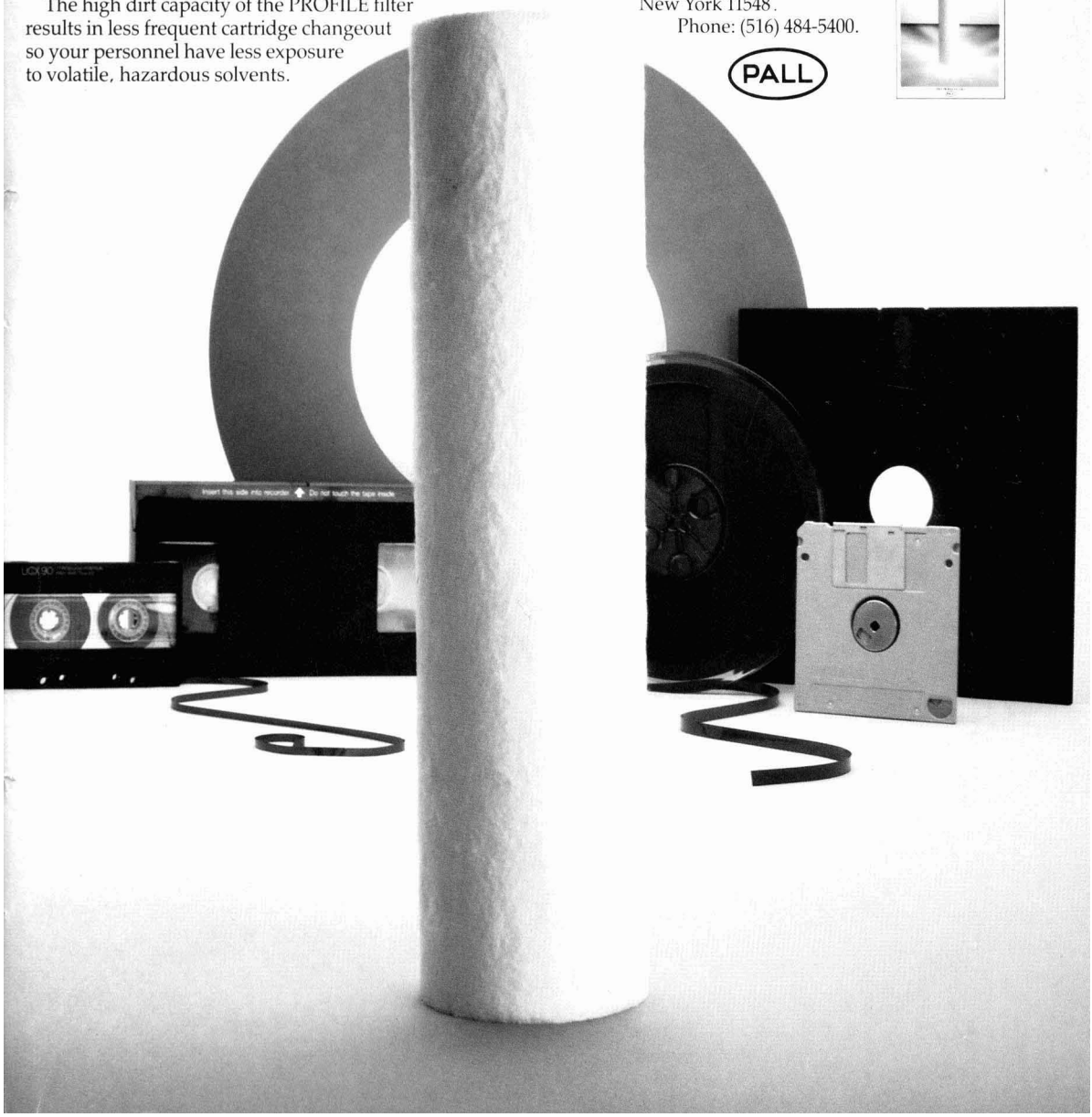

Resume' of a coatings Tribologist

Most simply defined* as "one who specializes in the control of friction and wear." The title could apply to all 80+ employees at our 100,000 sq. ft. plant. More particularly, it relates to:

+ the engineers, scientists and technicians specializing in product development and applications testing,
+ the production supervisors responsible for grind consistency and quality control,
+ the trained sales staff doing problem analysis and formulation recommendations in the field.

*from the Greek TRIBEIN - "to rub or wear"

We take polyethylene and PTFE raw materials and manufacture them into products that provide wear, mar, scuff, abrasion resistance, and other properties.

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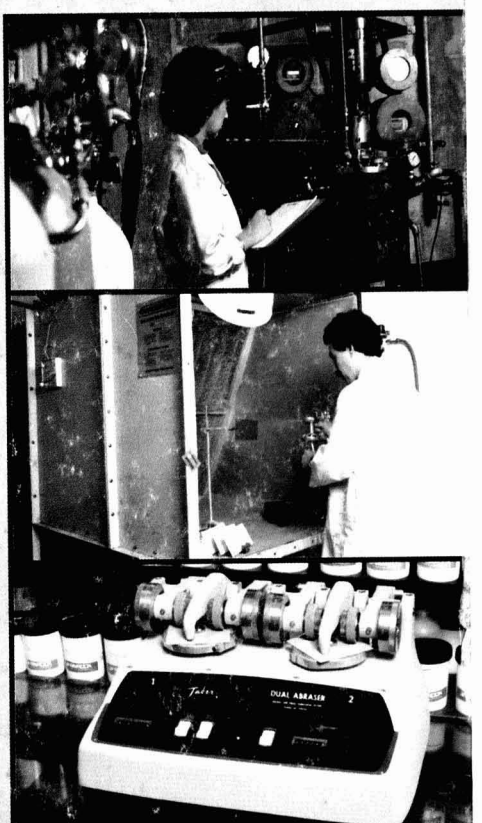

TOP: *Reactor produces developmental polyethylene waxes.* MIDDL *Samples are prepared by roller, brush, draw-down and spray applic tion techniques.* BOTTOM: *Testing is conducted for slip and frictio rub and mar, and wear and abrasion.**

*Taber Abraser shown distributed by Testing Machines, Inc.

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