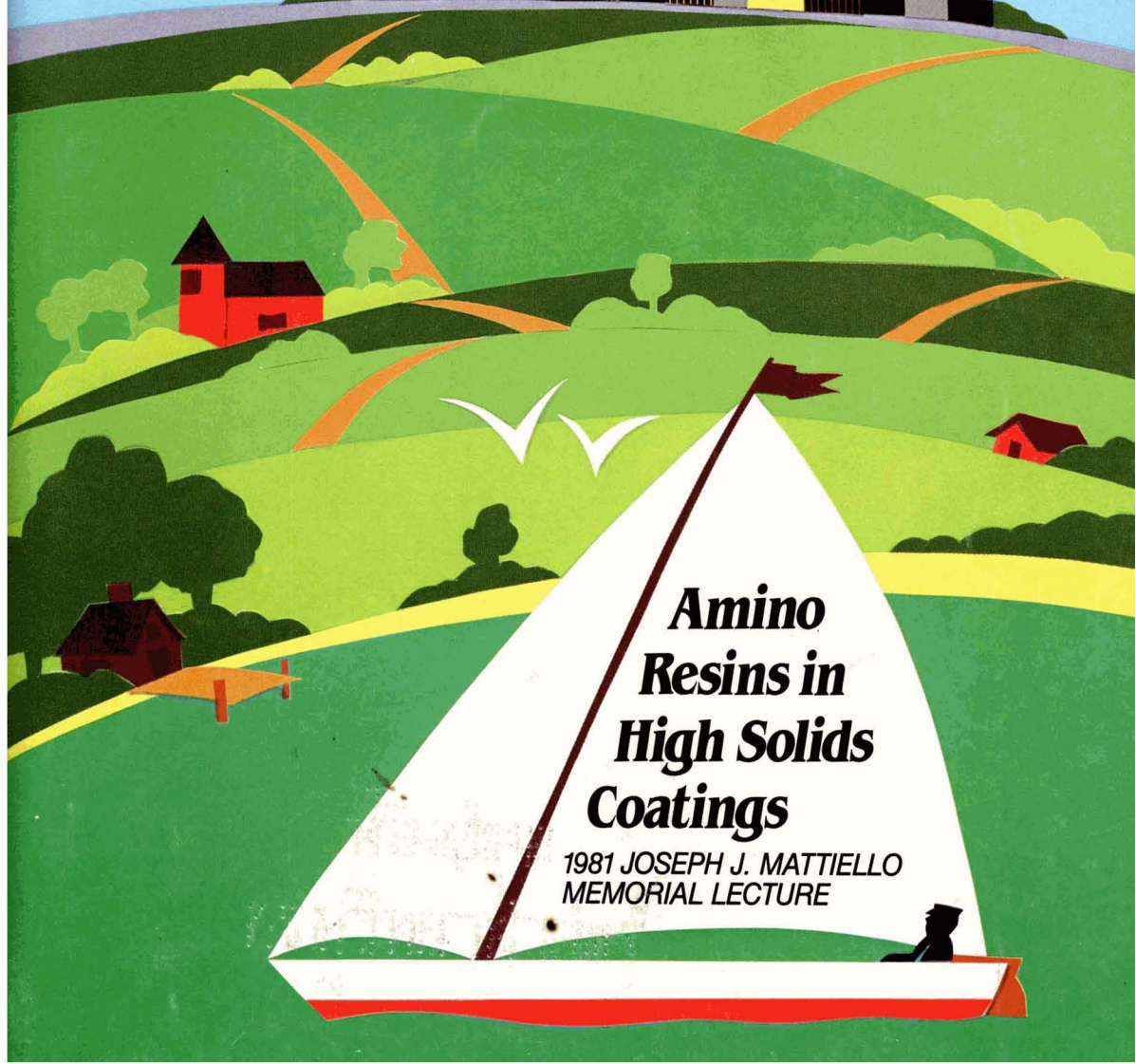


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## **Amino Resins in High Solids Coatings**

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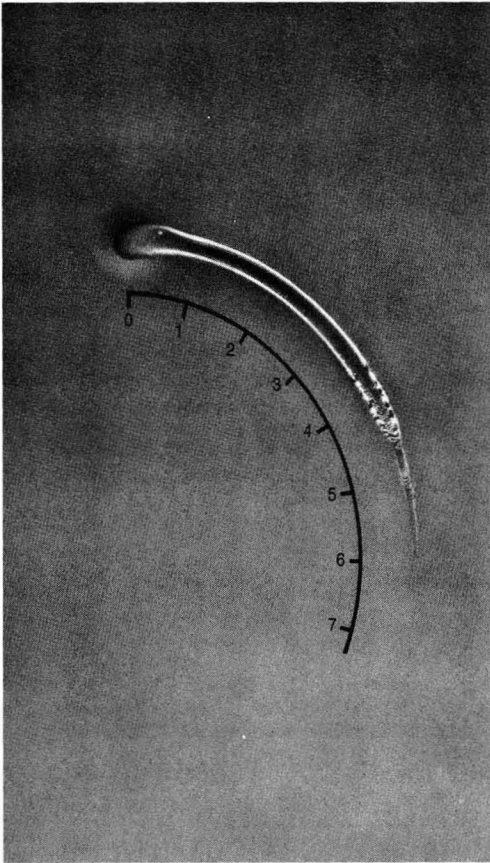
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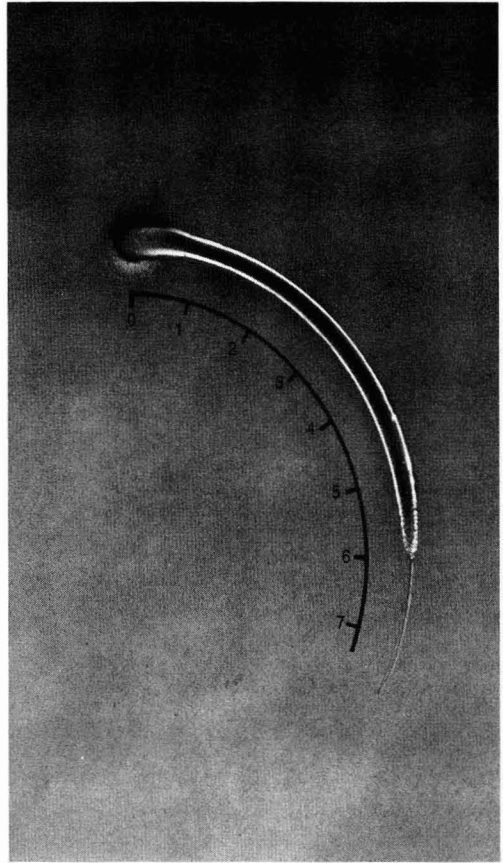
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JOURNAL OF  
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Comment	7	Support Your Local Sheriff
	8	1982 Paint Industries' Show in D.C.
Abstracts	12	
Government and Industry	14	1982 Golden Impeller Award
	21	Proceedings of the Paint Research Institute No. 149—Report of the Research Director—R.R. Myers
	26	Amino Resins in High Solids Coatings—W.J. Blank
	43	Kinetics of Thermal Dissociation of Blocked Isocyanate Cross-linkers—Detroit Society for Coatings Technology
	53	Time-Lapse Spectroscopic Investigation of Alkyd and Linseed Oil Cure—J.H. Hartshorn
	63	Determination of Lead in Paint By Energy Dispersive X-ray Fluorescence Spectrometry—G.S. Kuntz and R.L.R. Towns
Society Meetings	70	
Future Society Meetings	76	
Elections	77	
Membership Anniversaries	77	
Meetings/Education	79	Officers Elected at 46th Southern Society Annual Meeting
People	83	
	84	Bob English Honored as 'Senior Statesman'
Obituary	84	
Literature	85	
Coming Events	89	
	92	Humbug From Hillman

21 APR 1982

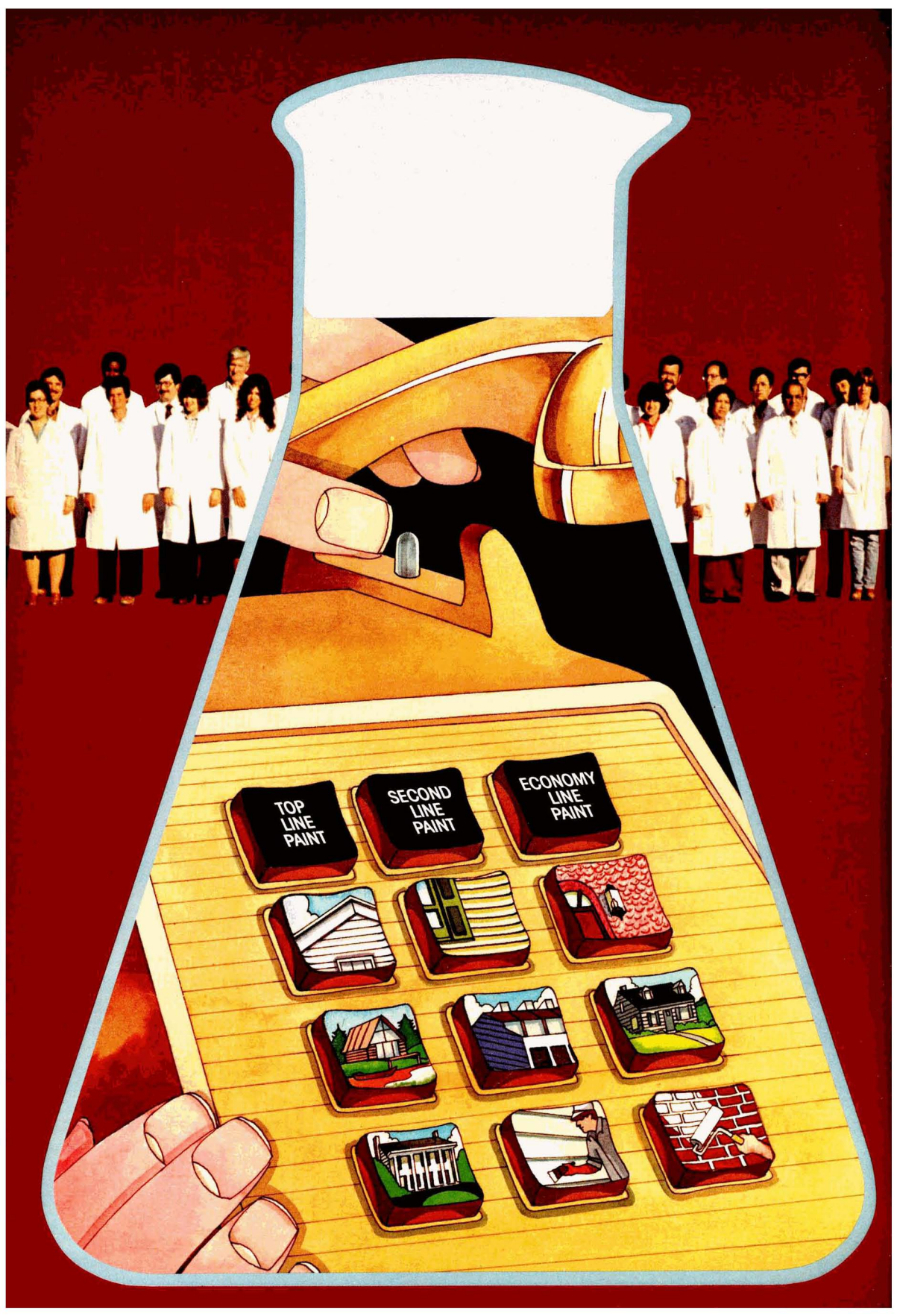


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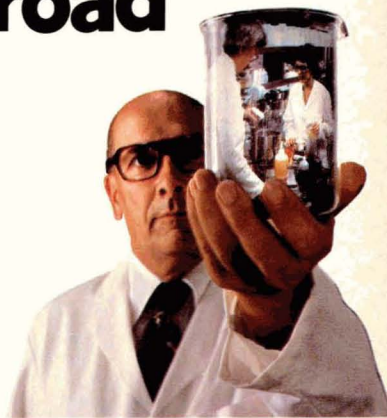


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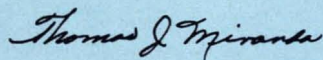
During the late 70's the regulatory climate achieved its zenith at a great cost to industry, the tax payer, and the American economy. The Coatings Industry became so involved in responding to legislation that Research and Development funds were siphoned away to satisfy government regulations.

Presently, however, we have seen a turnabout with the appointment of responsible administrators in agencies which affect our industry. These people are attempting to restore a reasonable balance between achievement of standards and rules compatible with our economic system.

One example is the work currently being done at the Environmental Protection Agency where the atmosphere has changed from an adversary position to one of cooperation between industry and government.

So if you have administrators or legislators at the local or national level who are doing a good job, why not let them know now. If we don't write, they will surely be hearing from single interest groups and assume that they reflect the mood of our industry.

As they say in the Old West—"Support your local Sheriff."



Thomas J. Miranda,  
Technical Editor

# 1982 Paint Industries' Show in D.C. Approaches Record Set at 1981 Event

Over 153 supplier firms will feature their exhibits at the 47th Paint Industries' Show of the Federation of Societies for Coatings Technology at the Sheraton Washington Hotel in Washington D.C., November 3-5. To be held in conjunction with the 60th Annual Meeting of the Federation, the Paint Show will offer attendees almost 37,000 sq. ft. of the latest developments in the industry, with 98% of exhibit space currently reserved.

The Paint Show is the only national

exhibit of raw materials and equipment used in the formulation, testing, and manufacture of paints and related coatings. Exhibitors will have on hand their top technical personnel to discuss product innovations and advances in paint manufacturing technology.

Show hours will be noon to 5:30 p.m. on Wednesday, November 3; 9:30 a.m. to 5:00 p.m. on Thursday, November 4; and 9:30 a.m. to 4:00 p.m. on Friday, November 5.

## Housing and Reservations

The Sheraton Washington Hotel will serve as headquarters hotel. Other hotels with blocks of rooms set aside for the Annual Meeting are the Shoreham, Washington Hilton, and the DuPont Plaza.

All requests for rooms and suites must be sent to the Federation office on the official form which will be mailed to all members this month and will also be included in future issues of the JCT.

## 1982 Paint Show Exhibitors

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Cosan Chemical Corp.  
Custom Chemical Co.

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Draiswerke, Inc.  
Drew Chemical Corp.  
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Freeport Kaolin Co.  
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Hooker Chemicals & Plastics Corp.  
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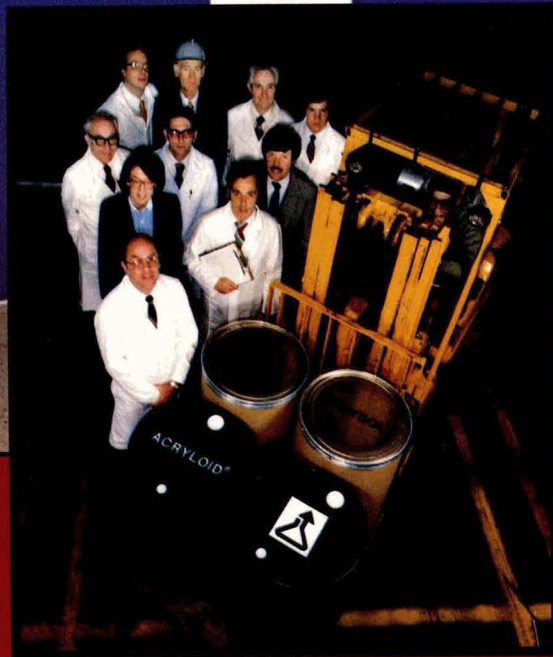
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# Abstracts of Papers in This Issue

## AMINO RESINS IN HIGH SOLIDS COATINGS—W.J. Blank

Journal of Coatings Technology, 54, No. 687, 26 (Apr. 1982)

The decrease in molecular weight and functionality required to increase the application solids of coating systems has a profound effect on the network formed during the crosslinking process with amino resins.

Because of reduced functionality the gel point of high solids coating systems takes place at increased conversion of functional sites; furthermore, higher conversion of functional groups is necessary to achieve the required properties. The amount of functional groups available and the functionality of an amino resin will depend on the structure of the resin, its degree of alkylation, methylolation, degree of polymerization, and the type of catalysis used. Amino resins catalyzed by a weak acid catalyst have potentially far fewer reactive sites available than fully alkylated or strong acid catalyzed resins. Additionally, general acid catalyzed amino resins have a tendency to self-condense. This competing reaction further reduces the amount of functional groups available for crosslinks. Therefore, for most high solids coatings only fully alkylated amino resins can give the combination of high application solids with excellent performance. Partially alkylated amino resins responding to general acid catalysis are predominately suitable for medium solid content and when performance does not require a highly crosslinked network.

## KINETICS OF THERMAL DISSOCIATION OF BLOCKED ISOCYANATE CROSSLINKERS—Detroit Society for Coatings Technology

Journal of Coatings Technology, 54, No. 687, 43 (Apr. 1982)

Novel isocyanurate crosslinkers containing blocked isocyanate groups were synthesized and the kinetics of thermal dissociation were studied by isothermal thermogravimetric analysis in air and nitrogen. Isocyanurate crosslinkers containing aromatic and aliphatic isocyanates were blocked with methyl-ethyl ketoxime and 2-ethylhexanol. The activation energies and frequency factors were measured and a method of calculating dissociation temperatures is proposed.

## TIME-LAPSE SPECTROSCOPIC INVESTIGATION OF ALKYD AND LINSEED OIL CURE—J.H. Hartshorn

Journal of Coatings Technology, 54, No. 687, 53 (Apr. 1982)

The air-drying mechanism of a conventional soya alkyd and of a linseed oil has been investigated by a new technique, Time-Lapse Infrared Spectroscopy. This technique is capable of assessing not only the reactions taking place during polymer cure but when and in which order they occur. Each of these materials was found to follow similar though different oxidation routes. Both are initiated by oxygen via hydroperoxide formation; however, their generation/decomposition rates and their oxidation and crosslinking paths differed markedly. Several competing reactions are identified. Some of these predominate in the alkyd case while others prevail in the oil. These may be divided into three phases: an inhibition period, a relatively fast initiation, and crosslinking. The observed reactions are compared with some previously reported drying mechanisms.

## DETERMINATION OF LEAD IN PAINT BY ENERGY DISPERSIVE X-RAY FLUORESCENCE SPECTROMETRY—G.S. Kuntz and R.L.R. Towns

Journal of Coatings Technology, 54, No. 687, 63 (Apr. 1982)

Available methods for the determination of lead in paint require lengthy sample preparation and may neither provide positive identification of the element nor address the matrix effects without a significant number of additional measurements. An X-ray fluorescence spectrometric method of quantitation using the fundamental parameters "pure element behind a thin sample" technique is described. This approach accounts for both calibration and matrix correction while requiring relatively simple sample preparation. Minimum detection limits ( $3\sigma$ ) are 0.2 and 0.4 ppm at the Lead  $L\alpha$  and  $L\beta$  lines, respectively, with a relative standard deviation of approximately four percent.



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Molyorange/BON Control	\$1.50

### RED B—SOLVENT & AQUEOUS

Pfizer Red (R-1599D)	27.3 lb.
Permanent Red	16.4 lb.
TiO <sub>2</sub>	9.7 lb.
Pigment Level 53.4 lb./100 gal.	
Pigment Cost/Gallon	\$1.80
Molyorange/Per Red Control	\$1.86

### GREEN—SOLVENT & AQUEOUS SYSTEMS

Pfizer Yellow (YLO-2288D)	34.7 lb.	Pigment Level	50.0 lb./100 gal.
Phthalo Green	14.3 lb.	Pigment Cost/Gallon	\$1.45
TiO <sub>2</sub>	1.0 lb.	Med. Chrome Green Control	\$1.30



## MINERALS, PIGMENTS & METALS DIVISION

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For Information by Phone Call 215-253-6261, Ext. 307

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- Send me tech data on your implement enamel formulae.
- Have a Pfizer representative phone for an appointment.

Name & Title \_\_\_\_\_

Company \_\_\_\_\_

Address \_\_\_\_\_

City \_\_\_\_\_ State \_\_\_\_\_ Zip \_\_\_\_\_

Phone \_\_\_\_\_ Best Time \_\_\_\_\_

## Kline's Economic Indicators Predict Little Growth in Paint Industry Through 1986

Market conditions and product mix changes indicate that there will be essentially no growth in the industry over the next five years, according to the new sixth edition of the *Kline Guide to the Paint Industry*. Sales of the paint industry were \$8.3 billion in 1981, a 9.2% increase over 1980. Units, however, grew only 2% between 1980 and 1981, down from the 2.3% per year rate experienced since 1976. Unit sales are estimated to grow only 0.1% a year reaching 1,045 million gallons in 1986.

Historically, paint shipments fluctuate with the economy and new construction. It is due to the sharp decrease in housing starts, rising costs of raw materials, and the maturity of the paint industry itself that no growth is expected. In fact, dollar shipments of product finishes will decrease 7.8% annually between 1981-1986. Solvent-based product finishes are declining in their share of the market, making way for water-based, high-solids grades, and powder coatings. Also, application efficiency is increasing with the use of such methods as electrostatic systems and electro-dipping. Therefore, real growth of product finishes is offset by reducing transfer waste.

Architectural coatings will see the most substantial unit growth of 3.0% annually, this due to the continued

**Table 1—Estimated Shipments of the U.S. Paint Industry 1980, 1981, and 1986**

	1980	1981	1986	Average Annual Growth Rate 1981-1986
<b>\$ Million</b>				
Architectural coatings .....	\$3,641.2	\$3,930.0	\$4,450.0	3.5%
Product finishes-OEM .....	2,418.5	2,565.0	1,710.0	-7.8
Special purpose coatings .....	1,576.2	1,890.0	2,085.0	2.0
Total .....	\$7,635.9	\$8,385.0	\$8,335.0	-0.1%
<b>Million gallons</b>				
Architectural coatings .....	529.5	545.0	630.0	3.0%
Product finishes-OEM .....	298.2	300.0	200.0	-7.8
Special purpose coatings .....	191.4	195.0	215.0	2.0
Total .....	1,019.1	1,040.0	1,045.0	0.1%

Note: Forecasts in constant 1981 dollars.

Source: Estimates by C. H. Kline & Co., based on data derived from "Current Industrial Reports, M28F".

growth of do-it-yourself paints (DIY). Both dollar shipments and units for special purpose coatings are expected to grow at 2.0% annually between 1981-1986 (Table 1).

### Profitability

The new *Paint Guide* cites CITIBANK data indicating that the industry earned 12.2% on stockholders' equity in 1980—1.1 percentage points more than in 1979. Paint industry return on investment has improved considerably over the last decade, dropping slightly in only 1975 and 1978. However, they continue below the returns for all manufacturing. Margin on sales fell below all manufacturing levels in 1963 and currently are at 3%, 2 percentage points below that for all manufacturing.

### Company Concentration

U.S. sales of the paint industry are relatively concentrated in a small number of firms. Sales by the 30 leading paint producers totaled \$4.6 billion in 1980, or 60% of the industry total of \$7.6 billion. The five leading producers—Sherwin-Williams, PPG, Du Pont, Glidden (SCM), and Mobil, accounted for 28% of total shipments, including exports. These companies were also ranked in the top five in 1978, with PPG and Du Pont exchanging second and third positions.

The new sixth edition of the *Paint Guide* is available at \$97 a copy with discounts available for quantity purchases of three or more from Charles H. Kline & Co., 330 Passaic Avenue, Fairfield, N.J. 07006.

### Call for Nominees For Golden Impeller Award

The Golden Impeller Award, for outstanding service to the coatings industry in the field of dispersion technology, will be presented again this year at the Annual Meeting of the Federation, in Washington, D.C., November 3-5.

Persons wishing to submit nominations for the award, which is sponsored by Morehouse Industries, should contact by May 1 the Chairman of the Board of Judges, Michael W. Malaga, Seegott, Inc., 190 E. Washington St., Chagrin Falls, OH 44022.

Other board members include: Dr. Zeno Wicks, Jr., of North Dakota State University; Fred K. Daniel, of Daniel Products Co.; Dr. Herman Larson, of Lan Chem Resin Co.; and Dr. Seymour Hochberg, retired from duPont Co.

Previous winners include Hugh F. Purcell, Mr. Daniel, Temple C. Patton, and Dr. Hochberg.

### FSCT Educational Committee Offers 1982 Edition of Coating Courses Guide

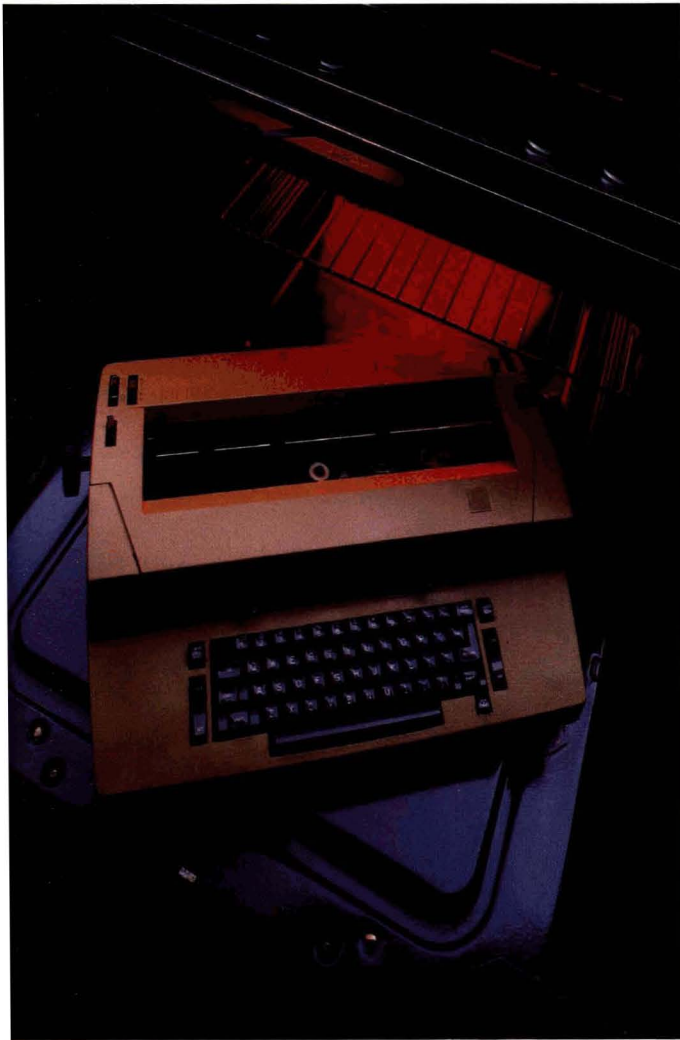
Publication of the 1982 "Guide to Coatings Courses, Symposia, and Seminars," compiled by its Educational Committee, has been announced by the Federation of Societies for Coatings Technology.

Based on information supplied by the Constituent Societies, the Guide includes a variety of coatings educational offerings listed by geographic region and Society. The Educational Committee updates the listing annually to reflect current programs, curricula, etc.

To obtain free copies of the 30-page Guide, write to Educational Committee (Coatings Courses), c/o Federation of Societies for Coatings Technology, 1315 Walnut Street, Suite 832, Philadelphia, PA 19107.



# Thanks to our acrylic resins, typewriter manufacturers can get out of the baking business.



So can manufacturers of snowmobiles. Or garden tractors. Or automobiles. Or motor homes. Because urethane coatings based on our acrylic resin systems cure at room temperature. Or they can be brought to "handling" hardness at as little as 150°F for 10 minutes and finish curing outside the oven.

That makes them a great problem-solver for coating products that can't take heat. And a great energy-saver even for products that can.

And Henkel acrylic resin based coatings offer other advantages as well. They provide twice the coating life of alkyds. They match or surpass the adhesion and performance of other leading urethanes. But they cost less than a polyester/isocyanate system, because they take less of the high cost isocyanate component.

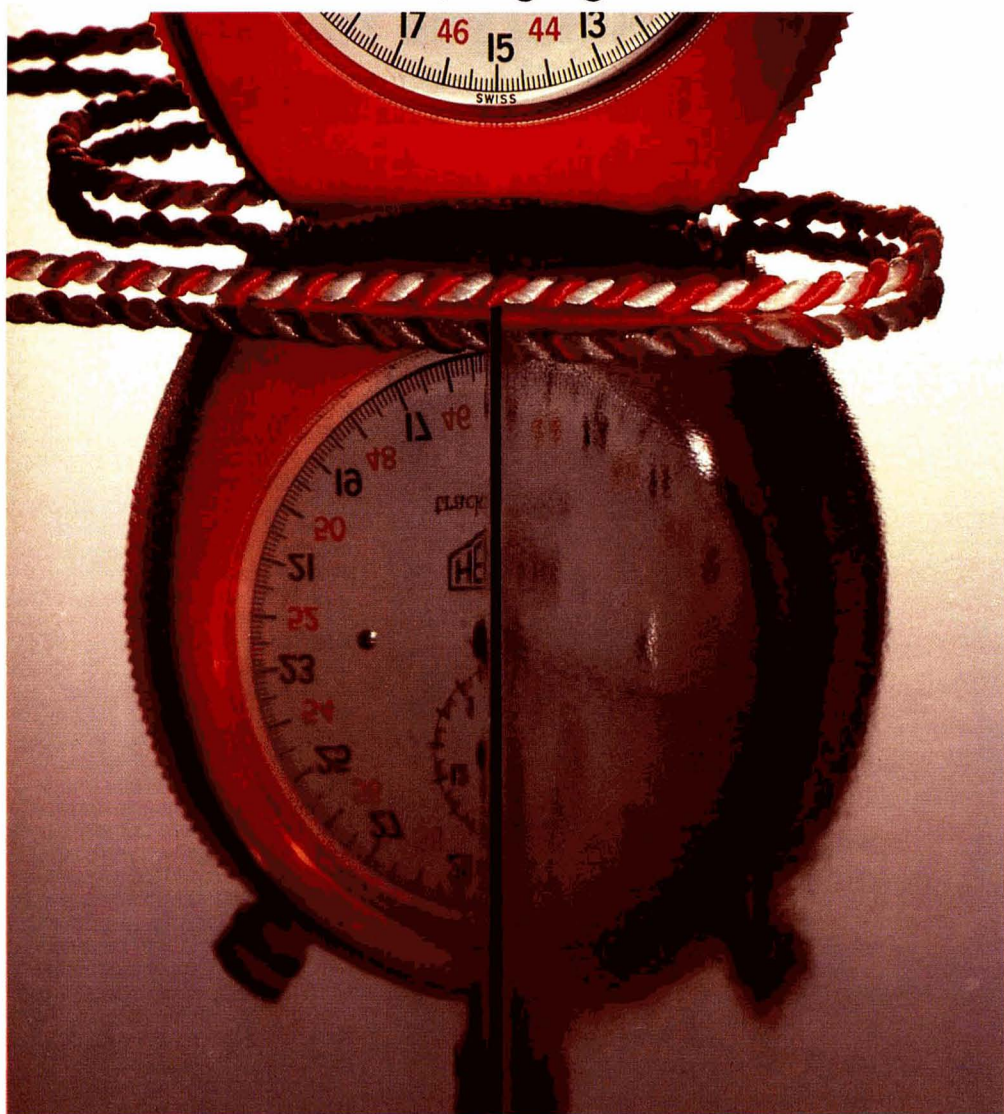
Henkel offers a line of acrylic systems based on three primary acrylic resins. G-CURE 867 acrylic resin provides unsurpassed gloss retention and excellent chemical resistance. G-CURE 868 acrylic resin is softer, more flexible, and can be used with harder isocyanates or melamines. G-CURE 869 acrylic resin based coatings are fast drying, making them particularly effective for factory applications and wood finishes.

If coatings based on Henkel acrylic resins can do so much for manufacturers of typewriters, garden tractors, snowmobiles, automobiles and motor homes, imagine what they can do for your customers' coating problems. For more information about Henkel resin capabilities, write Resins Division, Henkel Corporation, 4620 West 77th St., Dept. JCT 42, Minneapolis, MN 55437.

## Resins Division



# Which fast-drying, water-borne polymer is really high gloss?



## New Joncryl 537.

## "State of the art."

Joncryl 537 is a remarkable new water-borne emulsion polymer for industrial coatings. It's the first truly high-gloss, fast-drying product of its kind. The reflection in the photo above compares Joncryl 537 to a conventional "high-gloss" emulsion polymer. As you can see, Joncryl 537 has gloss and depth of image to take the state of the art several steps forward.

Joncryl 537 doesn't take second place in drying time and handleability, either. It air-dries tack-free in under one hour, with 80% of ultimate hardness in just 24 hours. Plus, it can be applied by any common method on most substrates.

It's made to be easy to use, with low foaming characteristics for wide formulating latitude. It's an excellent pigment wetter, and can often replace grinding vehicles. High quality manufacturing standards give you batch-to-batch consistency. And it's competitively priced.

Joncryl 537 is only one in the broad, innovative line of polymer products from Johnson Wax. Write or call for more information on Joncryl 537, or our complete line: Specialty Chemicals Group, Worldwide Innochem Operations, Johnson Wax, Racine, WI 53403, Telephone: 414/631-3789.

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SPECIALTY CHEMICALS

**We do much more than floors.**

Two New Pigments  
with High  
Fastness Properties

# HEUCOTRON® Red 23

# HEUCOTRON® Yellow 5



**Higher industrial hygienic protection**  
**Increased SO<sub>2</sub>-resistance**  
**Increased Hiding Power**  
**Increased brilliancy and colour depth**

### Two New Pigments with High Fastness Properties

HEUCOTRON-Yellow 5 and HEUCOTRON-Red 23 have excellent fastness to light, weathering and SO<sub>2</sub>, and possess more brilliant colour shades than other comparable pigments.

### Industrial Hygienic Security

The special merit of HEUCOTRON-Yellow 5 is the low acid soluble lead content of less than 1% according to DIN 55975 (pigment/hydrochloric acid = 1/1.000).

According to project ISO 6713 second edition 1981 (pigment/hydrochloric acid = 1:15) this means an acid soluble lead content of 1% for both pigments.

This considerable reduction of the acid soluble lead content in both pigments results in a corresponding increase of industrial hygienic security and furthermore these pigments do not have to be marked with the St. Andrews Cross.

### SO<sub>2</sub>-Test

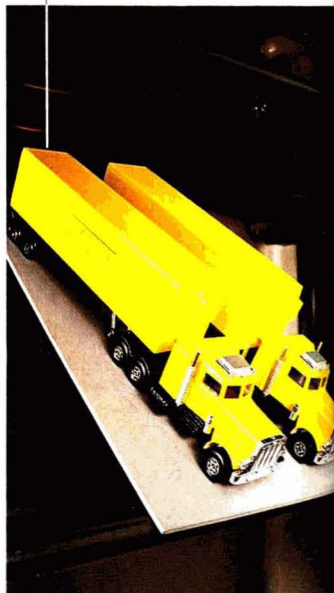
To test for durability panels were coated with an alkyd-melamine resin varnish containing 20% for HEUCOTRON-Yellow 5 and 15% for HEUCOTRON-Red 23, then tested in accordance with DIN 50018 with 2 litres SO<sub>2</sub> in a Kesternich apparatus.

HEUCOTRON-Yellow 5 and HEUCOTRON-Red 23 were compared with high performance pigments from the highly stabilized series.

The Kesternich test has shown that HEUCOTRON-Yellow 5 and HEUCOTRON-Red 23 possess significantly better resistance to SO<sub>2</sub> than other pigments.

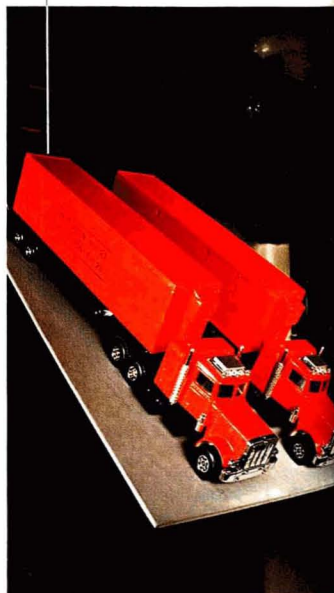
Because of this as well as the excellent fastness to light, weathering and heat HEUCOTRON-Yellow 5 and HEUCOTRON-Red 23 are particularly suitable for use in high quality paint systems.

HEUCOTRON-Yellow 5



Reference sample

HEUCOTRON-Red 23



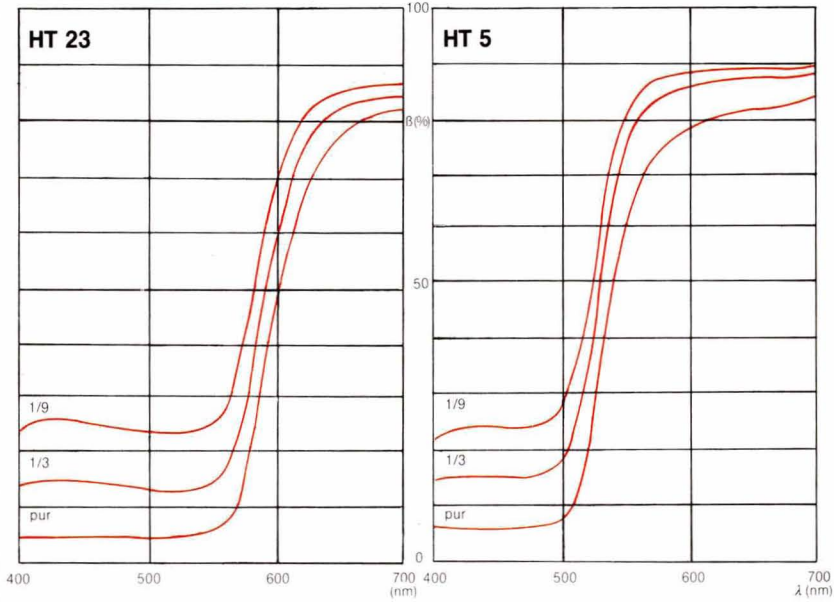
Reference sample

# HEUCOTRON® Red 23

# HEUCOTRON® Yellow 5

HEUCOTRON-Yellow 5

Colorimetric Data, Illuminant D 65, 10°



cycles Kesternich

HEUCOTRON-Red 23



cycles Kesternich

### Reply Card

Please send me a 500-g-sample each of

- HEUCOTRON-Yellow 5
- two HEUCOTRON-Standard-Chromium Yellows (medium-stabilized)
- general information on the Heubach-Group

Remarks: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

Technical Data	HEUCOTRON Yellow 5	HEUCOTRON Red 23
Bulking volume g/ml	0,4	0,7
Tamped volume g/ml	0,6	0,9
Specific gravity g/cm <sup>3</sup>	5,6	5,7
Oil absorption g/100 g	29,5	25
Hiding power (TiO <sub>2</sub> = 100)	86	160
Heat fastness	200° C	190° C
pH-value	ca. 6	ca. 6
Acid soluble Lead (DIN 55975)	< 1%	< 2%
ISO 6713	< 1%	< 1%

Fastness to:	HEUCOTRON Red 23	HEUCOTRON Yellow 5
light		
Enamel pur	8	8
1/3 ST	8	8
1/9 ST	8	8
Exposure		
Enamel, air-drying pur	4 d	4 d
1/3 ST	4 d	4 d
1/9 ST	4 d	4 d
Enamel, stoving pur	5-4 d	5-4 d
1/3 ST	5-4 d	5-4 d
1/9 ST	5-4 d	5-4 d
Solvents		
Aliph. hydrocarb.	5	5
Arom. hydrocarb.	5	5
Chlor. hydrocarb.	5	5
Esters	5	5
Ketones	5	5
Alcohols	5	5
Plasticizers	5	5
Linseed oil	5	5
Acids	4	4
Alkalis	4	4
Lime	no	no
Cement	no	no
Overspray	yes	yes



Name \_\_\_\_\_

Company + Company Address  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

## Reply Card

**Dr. Hans Heubach**

Heubachstraße 7

D-3394 Langelsheim 1

West Germany

**Dr. Hans Heubach GmbH & Co. KG**

Heubachstraße 7  
D-3394 Langelsheim 1, W. Germany  
Telephon (0 53 26) 5 20  
Telex 09 57 726 (heuba d)

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NUMBER 98

APRIL 1982

FEDERATION

# newsletter



## **FEDERATION PUBLISHES GUIDE TO TRADE PAINT QUALITY**

A Consumer Guide to Trade Paint Quality, produced to help promote an appreciation of paint quality features and an understanding of paint application, has been published by the Federation of Societies for Coatings Technology.

The guide, entitled "Know Paint Quality Before You Buy," focuses on latex interior flat paint and depicts the performance characteristics of low, average, and high-quality products.

Written and developed by the Southern Society for Coatings Technology, an affiliate of the Federation, the pamphlet is designed for point-of-purchase use to help the consumer understand what to look for in paints. Full-color reproductions illustrate the effects of paint quality features on staining and washability-durability, as well as spreading rate, application, and wet and dry hiding.

Commenting on the guide, Federation President Howard Jerome said, "The Southern Society is to be commended for developing the brochure and responding to the need for consumer information. Publication by the Federation is in keeping with the May 1981 resolution of the Federation Board of Directors to support appropriate efforts to increase consumer awareness of various qualities of architectural paints."

Cooperating with the Federation in the distribution of the guide will be the National Decorating Products Association and its member paint store dealers.

The price of the guide is 10¢ each, minimum order of 100 copies. Order from the Federation of Societies for Coatings Technology, 1315 Walnut St., Philadelphia, PA 19107.

## **ROYAL A. BROWN BECOMES CONSULTANT TO FEDERATION**

Royal A. Brown, well-known figure in the coatings industry for many years, has been retained as a consultant to the Federation of Societies for Coatings Technology. The announcement was made by Howard Jerome, Federation President.

Mr. Brown, whose title will be Technical Advisor, retired earlier this year from the National Paint and Coatings Association, where he served for 16 years, most recently as Vice-President-Technical.

In his announcement, Mr. Jerome said that Mr. Brown will be concerned with advancing the technical, educational, and manufacturing activities of the

Federation in cooperation with those committees of the organization. In this capacity, he will be responsible for developing national programs, including a Federation-sponsored seminar in the spring of 1983. He will also be called upon, from time to time, to represent the Federation in various industry liaison capacities.

Mr. Jerome added that the Federation is fortunate to obtain the services of Mr. Brown, currently a Trustee of the Paint Research Institute, noting that his extensive background and experience in both technical and administrative aspects of the industry uniquely qualify him for his Federation duties.

Mr. Brown's industry career includes service with Gilman Paint & Varnish Co., Southern Varnish Corp., Egyptian Lacquer Manufacturing Co., and the Chemical Div. of The Stanley Works, prior to joining NPCA as Technical Director in 1966. He was appointed Vice-President of the Association in 1978.

Mr. Brown will work out of his home in the Washington, D.C. area.

## **ONE HUNDRED FIFTY-FIVE EXHIBITORS SIGN UP FOR 1982 PAINT SHOW IN DC**

One hundred and fifty-five exhibitors have signed up for space in the Federation's 1982 Paint Show at the Sheraton Washington Hotel, Washington, DC, November 3-5. Show hours will be: 12:00 to 5:30 on Wednesday; 9:30 to 5:00 on Thursday; and 9:30 to 4:00 on Friday.

Aceto Chemical Co., Inc.  
ACT-Roper Plastics  
Air Products & Chemicals, Inc.  
Aluminum Co. of America  
C. M. Ambrose Co.  
American Felt & Filter Co.  
Amoco Chemicals Corp.  
Applied Color Systems, Inc.  
Armstrong Containers, Inc.  
Ashland Chemical Co., Ind. Chem. & Solv. Div.  
Atlas Electric Devices Co.

B.A.G. Corp.  
BASF Wyandotte Corp.  
Battelle Memorial Institute  
Beltron Corp.  
Blackmer Pump Div., Dover Corp.  
Brinkmann Instruments, Inc.  
Brookfield Engineering Labs., Inc.  
Buckman Laboratories, Inc.  
Burgess Pigment Co.  
Byk-Mallinckrodt USA, Inc.

Cabot Corp.  
Cargill, Inc.  
CDI Dispersions  
Celanese Chemical Co., Inc.

Celanese Plastics & Specialties Co.  
CEM Corp.  
Chemical Waste Management, Inc.  
Chicago Boiler Co.  
Clawson Tank Co.  
Color Corp. of America  
Columbian Chemicals Co.  
Commercial Filters Div.  
Continental Fibre Drum Co.  
Cosan Chemical Corp.  
Custom Chemical Co.

Daniel Products Co.  
Degussa Corp.  
Diamond Shamrock Corp.  
Diano Corp., Sub. Bausch & Lomb  
D/L Laboratories  
Dominion Colour Co. Ltd.  
Dow Chemical USA  
Draiswerke, Inc.  
Drew Chemical Co.  
DSET Laboratories, Inc.

Eastman Chemical Products, Inc.  
Ebonex Corp.  
Eiger Machinery, Inc.  
Engelhard Corp., Min. & Chem. Div.



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Filter Specialists, Inc.  
Freeport Kaolin Co.  
Fricke McCormick Engineering

GE Silicones  
Georgia Kaolin Co.  
Goodyear Tire & Rubber Co.  
W. R. Grace & Co., Davison Chem. Div.  
Graco, Inc.  
Gregory Group, Inc.

Halox Pigments  
Dr. Hans Heubach GmbH & Co. KG  
Harshaw Chemical Co.  
Harshaw Chemical Co.  
Henkel Corp.  
Hercules Incorporated  
Hilton-Davis Chemical Group  
Hockmeyer Equipment Corp.  
Hooker Chem. & Plastics Corp.  
J. M. Huber Corp.  
Hunter Associates Lab., Inc.

ICI Americas, Inc.  
Ideal Mfg. & Sales Corp.  
International Min. & Chem. Corp.  
Interstab Chemicals, Inc.

Johnson Wax

Kay-Fries, Inc.  
Kenrich Petrochemicals, Inc.

Labelette Co.  
Lancer Dispersions, Cleveland P & C Co.  
Laporte (United States) Inc.  
Liquid Controls Corp.  
Lorcon Chemicals, Inc.

3M Co.  
Macbeth Div., Kollmorgen Corp.  
Manchem Incorporated  
Manville Products Corp.  
Meadowbrook Corp.  
Merck & Co., Inc.  
Mini FIBERS, Inc.  
Modern Paint and Coatings  
Morehouse Industries, Inc.  
Myers Engineering

Nalco Chemical Co.  
Netzsch Incorporated  
Neutronics, Inc.  
Neville Chemical Co.  
NL Chemicals/NL Industries, Inc.  
NYCO, Div. of Processed Minerals, Inc.

Omya, Inc.  
Ottawa Silica Co.

Pacific Scientific Co., Gardner/Neotec  
Paint Research Institute  
Penn Color, Inc.  
Pennsylvania Glass Sand Corp.  
Pfaudler Co.  
Pfizer, Inc., MPM Div.  
Plastican, Inc.  
Polyvinyl Chemical Industries, Inc.  
PPG Industries, Inc.  
Premier Mill Corp.

Q-Panel Co.

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Reichhold Chemicals, Inc.  
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Russell Finex, Inc.

Sandoz Colors & Chemicals  
Sanyo-Kokusaku Pulp Co. Ltd.  
Semi-Bulk Systems, Inc.  
Sewell Plastics, Inc.  
Shamrock Chemicals Corp.  
Sherwin-Williams Chemicals  
Silverline Manufacturing Co., Inc.  
South Florida Test Service, Inc.  
Southern Clay Products, Inc.  
Spencer Kellogg, Div. of Textron, Inc.  
Standard Container Co.  
Sun Chemical Corp., Pigments Div.  
SWECO, Inc.  
Systech Corp.

Technology Marketing Corp.  
Tenneco Chemicals, Inc.  
Thibaut & Walker Co., Inc.  
Thiele Engineering Co.  
Troy Chemical Corp.

Union Camp Corp.  
Union Carbide Corp.  
Union Chemicals Div., Union Oil Co.  
Union Process, Inc.  
Uniroyal Chemical Co.

United Catalysts, Inc.  
Universal Color Dispersions  
University of Missouri-Rolla

R. T. Vanderbilt Co., Inc.  
Viking Pump Div., Houdaille Ind.  
Vorti-Siv Div., M&M Machine, Inc.

Wacker Chemical Co.  
Warren Rupp Co.  
Wellco Products & Itasco Ind.  
Wilden Pump & Engineering Co.  
Witco Chemical Corp.

Zeelan Industries, Inc.

## **NEWS BRIEFS FROM THE SOCIETIES**

BIRMINGHAM--The Club, which has shown steady growth over the years, enrolled its 150th member in February....Has completed an A/V "Introduction to the Paint Industry."

GOLDEN GATE--The Manufacturing Committee's Annual Seminar (June 14) will be on "Computers." An equipment display will be included...."Spectrum '83--Tomorrow's Technology: Today" will be theme of 16th Biennial Western Coatings Societies' Symposium and Show, February 23-25, 1983, at the Hyatt Regency - Embarcadero Center, in San Francisco. Ted Favata, of Triangle Coatings Co., is the General Chairman. Co-Chairman is Rob Holt, of Sherwin-Williams Co. Working with them are these sub-committee chairmen: Technical--Gordon Rook of O'Brien Corp.; Registration & Housing--Tom Dowd, of E.T. Horn Co.; Entertainment--Bud Harmon, of Borden Chemical Co.; Exhibits--John Beater, of O'Brien Corp.; Spouses' Program--Rhoda Harmon, of Harmon Associates; Treasurer--Barry Adler, of Royell, Inc.; and Publicity--Dave Waldron, of the duPont Co....Ted Favata was presented a special award at the February meeting in recognition of his many years of outstanding contributions to the coatings industry in the Bay Area.

KANSAS CITY--"Instrumentation for Quality Coatings in the 80's" is the theme of the joint meeting with St. Louis which KC will host on June 11-12....Has initiated a fund-raising project called "The First Annual Beef-A-Rama." Grand prize is a side of beef. (And where better than in KC!).

NEW ENGLAND--Four seminars will be featured at "Coatings Tech Expo '82": Manufacturing & Management; Waste Management; Technical Uses of Computers; and Advances in Coatings Formulations. A new item will be a Society/Industry exhibit of old equipment and other materials of historical interest.

NORTHWESTERN--11th Annual Symposium in March was on "Present and Future Equipment for Economical and Efficient Applications of Compliance Coatings."

PHILADELPHIA--Will sponsor Seminar on "Dispersion Technology" on May 3.

ST. LOUIS--26 high school chemistry teachers attended the 7th annual Education Night recently. Speaker was Dr. John C. Graham, Prof. of Polymer and Coatings Technology at Eastern Michigan University.

SOUTHERN--J. Robert "Bob" English, retired partner of R.T. Hopkins Co. in Atlanta, was honored as the Senior Statesman for 1982 by the Southern Society at its annual meeting in March; Bob was recognized for the many years of steadfast support he has given the Society and the coatings industry.



**HIGH SOLIDS AMINO  
CROSS-LINKING AGENTS**



**CYANAMID**

# Cyanamid Cross-Linking Agents for Coating Resins

## The 300 and 370 Series of CYMEL® Cross-Linking Agents

300	301	303	325	327	350	370	373	380	385	1116	1130
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### PROPERTIES

Structure	Melamine											
	←											→
Non-Volatile, % 45 Min. at 45°C,	98 Min.	98 Min.	98 Min.	80 ± 2	90 ± 2	98 Min.	88 ± 2	85 ± 2	80 ± 2	79 ± 2	98 Min.	98 Min.
Solvent	—	—	—	Isobutanol	—	—	Isobutanol	Water	Isobutanol	Water	—	—
Viscosity, Gardner-Holdt, 25°C	Waxy Solid	W-Z2	X-Z2	X-Z1	Z-Z3	Z2-Z5	Z2-Z4	Z-Z4	V-Z	U-W	U-Y	W-Z2
Viscosity, Poises, Approx.	—	12.8- 43.4	15.5- 43.4	14.4- 30.2	26.8- 54.6	43.4- 118.2	42.7- 74.8	28.6- 79.9	10.3- 26.3	7.8- 13.4	7.1- 19.9	12.1- 40.9
Pounds per Gallon, Approx.	10.0	10.0	10.0	9.3	9.8	10.0	9.8	10.5	9.6	10.4	9.4	9.4
Specific Gravity, g/mL	1.20	1.20	1.20	1.12	1.18	1.20	1.18	1.26	1.16	1.25	1.13	1.13
Flash Point, Setflash, °F	>200	>200	>200	112	112	>200	115	>200	115	>200	>200	>200
Molecular Size	Monomeric	Monomeric	Monomeric	Polymeric	Polymeric	Monomeric	Polymeric	Polymeric	Polymeric	Polymeric	Monomeric	Monomeric
Equivalent Weight <sup>1</sup>	130- 190	130- 190	130- 190	Note 2	Note 2	185-270	Note 2	Note 2	Note 2	Note 2	145- 220	150- 220
Free Formaldehyde, % Max.	0.5	0.5	0.5	1.0	1.0	0.5	3.0	3.0	3.0	0.5	0.5	0.5
Principal Reactive Groups	Alkoxy	Alkoxy	Alkoxy	Alkoxy- Imino	Alkoxy- Imino	Alkoxy	Alkoxy- Methylol	Alkoxy- Methylol	Alkoxy- Methylol	Methylol	Alkoxy	Alkoxy
Methylol Content	Very Low	Low	Very Low	Low	Low	Medium	High	High	High	Very High	Very Low	Very Low
Alkylation Alcohol	Methanol	Methanol	Methanol	Methanol	Methanol	Methanol	Methanol	Methanol	Methanol	Methanol	MeOH EtOH	MeOH BuOH
FDA Approved Under 175.300	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes

### SUGGESTED USES

#### Systems

	300	301	303	325	327	350	370	373	380	385	1116	1130
Water Based, Solution	●	●	●●	●	●	●	●●	●●		●	★	★
Emulsion	●	●	●●		●	●●	●	●●		●●	★	★
Electrocoating			●								●	●●
Solvent, Exempt, Non-Exempt	●	●	●●	●●	●●	★	●●		●●		★	★
High Solids	●	●	●●	●	●●	●	●		●		●●	●●
Powder	●●	●	●●									

#### Application Areas

	300	301	303	325	327	350	370	373	380	385	1116	1130
Appliance	●	●	●●	●	●●	●	●●		●●		●	●●
Automotive	●	●	●●	●●	●●	●	●		●		●	●●
Coil Coating	●	●	●●	●	●●	●	●●		●	★	●	●
Ink	●	●	●●								●	●●
Metal Decorating	●	●	●●	●	●●	●	●●		●		●	●●
Paper Coating	●	●	●●	●	●●	●	●		●		●	
Wood Conversion Finish	●	●	●●	●	●		●	★	●	★		

#### KEY

- PRIMARY CHOICE
- ALTERNATE CHOICE
- ★ RECOMMENDED FOR SPECIAL APPLICATIONS OR PROPERTIES

Cyanamid offers a broad range of alkylated amino cross-linking agents especially formulated to meet the large number of end-use requirements in the industrial coatings and related fields. They react with polymers containing carboxyl, hydroxyl, and amide functional groups and, as outlined below, vary in chemical structure according to:

- Type of amino resin - melamine, benzoguanamine, glycoluril, urea
- Type of alkylation - methanol, ethanol, butanol
- Degree of alkylation and polymerization

### CYMEL 1100 Cross-Linking Agents

### BEETLE® Cross-Linking Agents

CYMEL 1100 Cross-Linking Agents				BEETLE® Cross-Linking Agents							
1141	1156	1158	1123	1125	1170	1171	1172	55	60	65	80
85 ± 2	98 Min.	80 ± 2	Benzoguanamine		Glycoluril			Urea			
98 Min.	85 ± 2		98 Min.	85 ± 2	97 Min.	95 Min.	45 ± 2	75 ± 2	88 ± 2	98 Min.	96 Min.
Butanol	—	N-Butanol	—	2-Ethoxy ethanol	—	—	Water	Water	iso-propanol	—	—
W-Y	Z1-Z3	X-Z3	Z1-Z3	Z4-Z7	Z-Z2	Z1-Z3	<A	X-Z1	X-Z1	Z3-Z6	X-Z3
11.6-19.0	28.4-48.6	13.7-49.1	31.6-54.2	72.3-442.3	24.3-38.7	32.4-55.6	—	16.3-34.0	15.2-31.9	57.4-183.5	14.2-50.9
9.0	8.7	8.8	9.7	9.5	8.9	10.0	10.1	10.5	9.8	10.3	9.1
1.08	1.05	1.06	1.17	1.14	1.07	1.20	1.22	1.26	1.18	1.24	9.1
102	>200	117	>200	160	>200	>200	>200	>200	96	>200	>200
Monomeric	Monomeric	Polymeric	Monomeric	Polymeric	Monomeric	Monomeric	Monomeric	Polymeric	Dimer	Dimer	Polymeric
150-250	140-200	Note 2	130-190	170-220	150-230	100-140	75-100	Note 2	Note 2	Note 2	Note 2
1.0	0.5	1.0	0.5	0.5	0.5	0.5	1.5	1.0	2.0	2.0	0.5
Carboxy-Alkoxy	Alkoxy	Alkoxy-Imino	Alkoxy	Alkoxy-Carboxyl	Alkoxy	Alkoxy	Methylol	Alkoxy-Methylol	Alkoxy-Methylol	Alkoxy-Methylol	Alkoxy
Very Low	Very Low	Low	Very Low	Very Low	Very Low	Very Low	Very High	Low-Medium	High	Medium	Very Low
MeOH, EtOH	n-BuOH	n-BuOH	Methanol-Ethanol	Methanol-Ethanol	n-BuOH	MeOH, EtOH	None	Methanol	Methanol	Methanol	n-BuOH
Yes	Yes	Yes	No	No	No	No	No	Yes	Yes	Yes	Yes

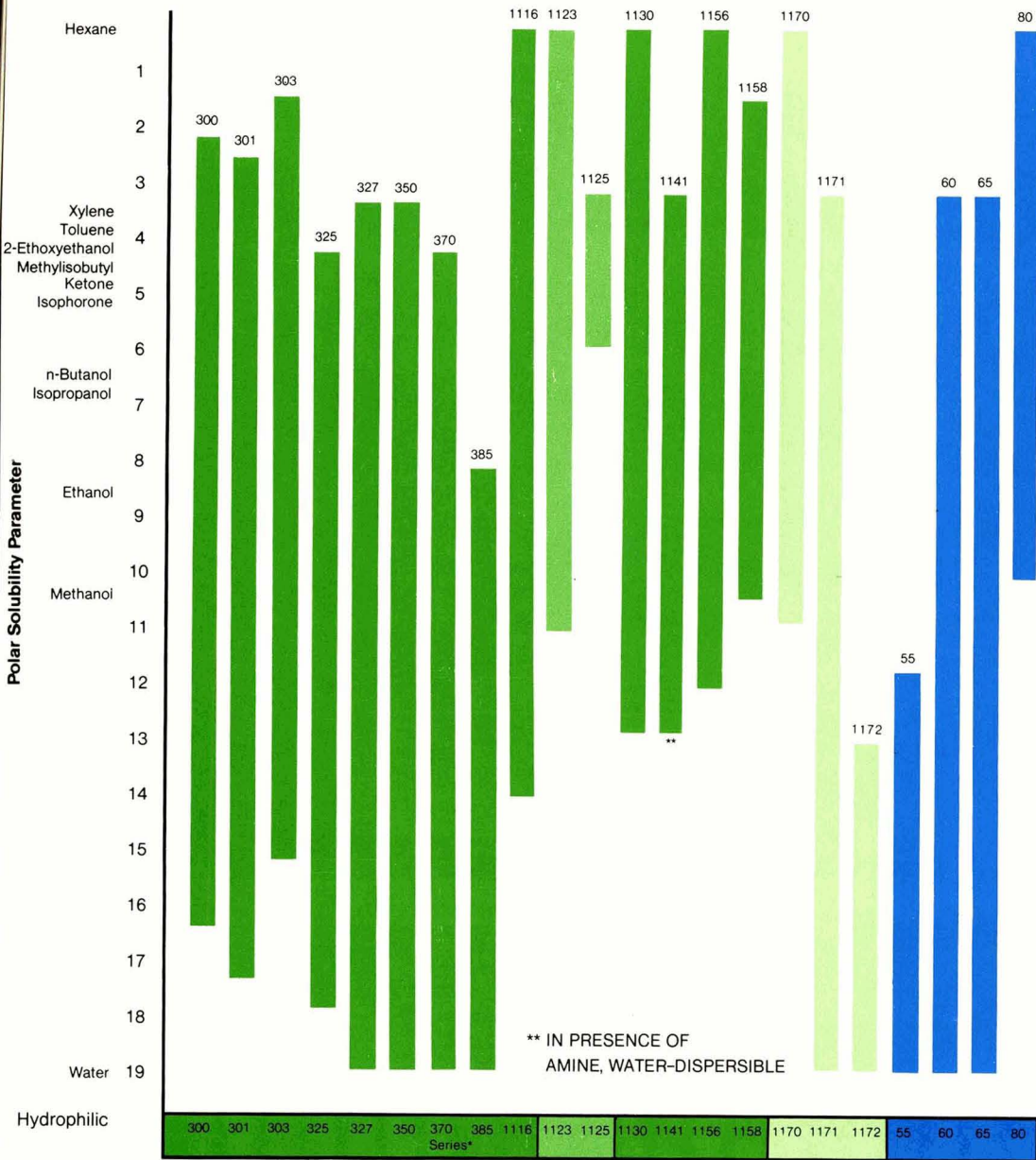
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• • • <sup>3</sup>	*		• •	*							• •
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									★	• •	• •

Footnotes:

1. Equivalent weight; grams of cross-linking agent required to react with one gram mole of carboxyl, hydroxyl or amide groups.
2. Equivalent weight not determinable; cross-linking agent has high tendency to self-condense.
3. Cationic electrocoating.
4. Recommended for aqueous neutral or acidic, hydroxyl or amide functional polymers.

# Solubility in Solvents and Water

Hydrophobic



## CYMEL

cross-linking agents

\*The 370 series includes CYMEL 370, 373, and 380 resins.

## BEETLE

cross-linking agents

The solubilities of the crosslinking agents in various solvents are compared according to polar solubility parameters, which are numerical constants for these solvents and solvent combinations.

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 Industrial Products Division  
 Resin Products Department  
 One Cyanamid Plaza  
 Wavne, New Jersey 07470

## Report of the Research Director

Raymond R. Myers\*

Research Director, Paint Research Institute

### Introduction

The annual summary of research conducted under PRI auspices is given. Consortium activity is responsible for the gaining of considerable momentum in studying mildew defacement, where emphasis has shifted from finding an anchored fungicide to the broader objective of building programmed release into a coating.

Various ways of achieving programmed release are included in the list of potential research subjects. Field tests are starting on one species of anchored fungicide and a search is under way to find the most qualified person to extend the program into encapsulation and other means of controlling the release of fungicide after the coating is applied.

Concerted effort has begun in the two areas of compliance coatings: aqueous and high solids. Both program managers have submitted proposals that have their stamp on the program but still need better coordination with the original prospectuses for the two programs.

Corrosion control research has been reactivated without resorting to a consortium. One grant proposal is being used as the main device to secure outside funding and a second proposal is expected as a result of our critical survey on the mechanism of inhibition.

The PRI symposium on stability and stabilization of coatings was a success. Over 50 people attended and the effort netted \$3000. Research ideas generated by the symposium were transmitted to the program managers.

### Grant in Progress in 1981

#### MILDEW CONSORTIUM

Last year marked the completion of a grant at the New York State College of Forestry and Environmental Science at Syracuse. It resulted in several papers in JCT

on nutrient and humidity requirements of the prevalent defacing organism. More significantly, it resulted in a bioassay procedure that is used by consortium members and that was judged important enough to be included in the ongoing program of making and evaluating anchored fungicides. Dr. Zabel published the method in the April 1981 JCT.

The mildew consortium recognized the need for an independent laboratory devoted to the performance of rapid assays and engaged Dr. George Hollis, of Memphis State University, to take over this portion of the mildew program. His assays will be an integral part of the study of fungicides and systems formulated according to the broader-based program on programmed release of fungicide.

Also published last year was the successful preparation of viable protoplasts of fungus whose cell wall was regenerated in the laboratory in the presence of inhibitors. This study was reported by Dr. Donald Siehr, of the University of Missouri at Rolla, in the October 1981 JCT.

Dr. Richard Crang, of the University of Illinois, joined the consortium as a grant holder. He now directs the electron microscopy laboratory for the entire institution and has a technician working solely on the morphological and membrane changes that accompany the treatment of fungus with sublethal levels of three separate fungicides. Secondary emission techniques are employed to reveal the location of heavy atoms.

Although this grant has not yet reported formally, an indication of the kind of information one can expect is given in Dr. Crang's earlier paper in the August 1979 JCT.

The heart of the consortium activity continues to be the development of anchored fungicides, with an eventual metamorphosis into studying systems with programmed release capability. The present goal is to control hydrolysis rates so that esters or other hydrolyzable types remain intact in the can during storage but

\*Kent State University, Dept. of Chemistry, Kent, OH 44240.

release fungicide at a rate which will keep the surface protected after the coating is exposed to the elements. In this connection a paper from Dr. Charles Pittman, of the University of Alabama, has been submitted for publication in the JCT. After review by the MCSC it will be sent to the Federation review board. [This paper is currently undergoing Federation review—Ed.]

Society technical committee activity has begun in connection with the anchored fungicide effort. A quantity of pentachlorophenyl acrylate was made available to Glidden personnel for conversion into acrylate copolymer which, in turn, will be distributed to the Chicago and Southern Society Technical Committees. Colin Penny, of the Federation's Technical Committee, was admitted to the consortium in order to coordinate this effort.

We offer a word of caution with regard to expectations. Having proved that anchored fungicides retain their activity in paint squares inoculated in Petri dishes, we have not yet found the optimum degree of hydrolysis needed for the fungicide to do the job and yet remain in the film long enough to extend its life appreciably. Research is needed on the nature of the linkage, and only by fortunate accident would the first graft be successful in test fences. It is expected that the various personnel performing the field experiments will recognize this fact before mapping a program and in assessing the data later on.

Program Manager Yeager has performed superbly. He has kept abreast of the ongoing program while launching four new grants that have his stamp on them. His activities in fund raising have resulted in the enrollment of one of his clients in the consortium and the possibility of two more joining the fold. He holds his ground in the quarterly meetings of the committee, yet he conducts the meetings democratically and diplomatically. His trips to Washington are not made at PRI expense.

Three new grants were voted into existence by the consortium.

**Paul Klens, of Lock Haven State College,  
"Effect of *Alternaria* spp. on Paint"**

This proposal was passed unanimously by the committee and the level of funding was not to be negotiated. Starting date was set at January 1, 1982. The committee wants an independent investigator of Dr. Klens' caliber to take a fresh look at typical mildew colonies.

**Robert Langer, of the Massachusetts Institute of  
Technology, "Understanding Diffusion in Controlled  
Release Systems"**

The committee desires that this grant be started on or before July 1, 1982, in the realization that the model currently being developed at MIT is not relevant to coatings. The model pictures a tortuous path with no allowance for mildewicide to diffuse through the walls of the maze. There is little chance that the second stage will start in 1982 in which fungicide solubility in the walls of the maze will be factored into the equation. This delay

in considering the relevant model should be accepted by the Trustees before the work begins.

Dr. Langer has a convincing reason for proving the present model before computer-simulating the more advanced model. He has a controlled-release drug system which provides experimental data that match the simplified model. In this kind of research, having an empirical approach to mesh with the theoretical study makes the difference between a routine study and a first-class investigation. Instead of curve fitting one can compare a calculated trend with actual data and thereby judge how to refine the model.

In the specific case considered here the combination approach may reveal some startling facts about how a fungicide is released. It may enable one to incorporate a fungicide of low solubility but high volatility into a capsule where it resists hydrolysis, yet escapes at a slow controlled rate by the tortuous path modeled by Dr. Langer.

**Warren P. Iverson, of the National Bureau of Standards,  
"A Study of the Biodeterioration of Exterior Paint Films  
by Actinomycetes, Bacteria, and Algae"**

This grant is not classed in the urgent category, as are the two above. Individuals on the committee want some work to be done on non-fungal species, such as actinomycetes, bacteria, and algae. Their acceptance of Iverson's proposal was based on his reputation and ongoing program.

This grant will start during the second half of 1982.

## AQUEOUS COATINGS

The consortium on aqueous coatings did not develop on schedule and the program was abandoned by the Trustees.

## HIGH SOLIDS COATINGS

Dr. Philip Weiss, of Oakland University, was engaged during the year as program manager for the high solids program. He studied the prospectus which appeared in the April 1981 JCT as a result of an encounter session and produced a program.

The prospectus, the proposal, and an invitation to bid will be sent to academic departments as soon as the following actions have been taken:

(1) It will be necessary to hire a fund raiser. PRI's new directions call for my successor to be largely a fund raiser, and there is a possibility that this action can be taken in 1982.

(2) Constituent Society Technical Committee input should be sought. A questionnaire covering high solids is being prepared.

(3) It is highly advisable that a subcommittee of the Trustees be formed to guide these early efforts. The subcommittee could be disbanded when the consortium takes root, but I recommend that subcommittees be formed for all of our consortia. Interestingly, we have a subcommittee for corrosion, which does not operate via consortium.



## CORROSION CONTROL

Henry Leidheiser's commissioned paper on "Mechanism of Corrosion Inhibition With Special Attention to Inhibitors in Inorganic Coatings" appeared in the July 1981 JCT. This paper was prefaced by me in an attempt to secure bids from interested parties to do some of the research outlined in the critical survey, with a suggestion that the bidder refer to our original prospectus published in the September 1973 JCT.

Henry Leidheiser has submitted a bid. Trustee Philip Heiberger has taken the responsibility of securing funding for this rather large bid, in pursuit of which we engaged Herbert Lowell to write a bankers' version of the original proposal.

This approach is an alternative to a consortium. We may emerge with a consortium of supporters and the formal committee structure to oversee the research, but a program manager is not envisioned at this time. Eventually, if other contractors enter the program, other vestiges of a consortium may develop.

Our critical survey on corrosion inhibitors brought a proposal from one of the best corrosion scientists in the country, Jerome Kruger.

His letter proposal was considered by the Trustees, who decided that its evaluation would be one of the first orders of business for the steering committee to be formed from supporters of the corrosion program.

Kruger has developed ellipsometry to the stage at which it can show changes in microscopically thin surface

coatings (such as oxides, conversion coatings, and precipitated inhibitors). In 1980 he reported in *Surface Science*, 96, 364 that changes in the phase retardation of elliptically polarized light were due to thickness variations in the substrate oxide film, using collodion as the coating under which the oxide was growing.

Chromate particles positioned around the ellipsometer observation region beneath the clear coating dissolve slowly and modify the environment under the coating. A microprobe also monitors pH changes. Whereas oxide film formation takes place in three stages without inhibitor, the chromate particles inhibit oxide film growth in the second stage and prevent dissolution of the metal in the third stage.

The proposed research would extend these findings by comparing changes occurring on bare steel panels with those occurring beneath coatings.

## ADIEU

This will be my last published report as Research Director of the PRI. Effective January 1, 1983 a Director will be chosen from industry to implement the changes recommended by the ad hoc committee on PRI.

I have enjoyed the association with many good people in the coatings industry. It will have lasted for nineteen years by the time I retire from this position. It is hoped that some of the friendships will last for an additional two decades.

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# Amino Resins in High Solids Coatings

Werner J. Blank  
American Cyanamid Company\*

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The decrease in molecular weight and functionality required to increase the application solids of coating systems has a profound effect on the network formed during the crosslinking process with amino resins.

Because of reduced functionality the gel point of high solids coating systems takes place at increased conversion of functional sites; furthermore, higher conversion of functional groups is necessary to achieve the required properties. The amount of functional groups available and the functionality of an amino resin will depend on the structure of the resin, its degree of alkylation, methylation, degree of polymerization, and the type of catalysis used. Amino resins catalyzed by a weak acid catalyst have potentially far fewer reactive sites available than fully alkylated or strong acid catalyzed resins. Additionally, general acid catalyzed amino resins have a tendency to self-condense. This competing reaction further reduces the amount of functional groups available for crosslinks. Therefore, for most high solids coatings only fully alkylated amino resins can give the combination of high application solids with excellent performance. Partially alkylated amino resins responding to general acid catalysis are predominately suitable for medium solid content and when performance does not require a highly crosslinked network.

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## INTRODUCTION

Significant amounts of organic solvent are being emitted during the application and curing process of industrial coatings. These solvents are necessary to adjust the application viscosity and rheology of a paint system to that required by the application process and equipment.

Increasing concern about the effect of these solvents

on ozone formation in the atmosphere has resulted in air pollution regulations in the United States which restrict the emission of solvents during the application and curing process. Furthermore, increased cost of hydrocarbon feedstocks has made it less and less economically viable to reduce industrial coatings with solvents to a conveniently low application solids. Also, the end user of industrial paints has become more and more aware that the true cost of an industrial coating is not reflected in \$/gal he pays for the liquid coating, but in his applied cost of  $\epsilon/\text{ft}^2/\text{mil}$ .

The approaches to reduce organic solvent emission in industrial coatings are the design of either low molecular weight polymers (also called oligomers), the use of reactive diluents, the use of 100% solid coatings, the replacement of organic solvent with water, or the treatment of the organic solvent air stream by absorption or incineration. Although, absorption and incineration are easy solutions to the solvent emission problems and are often used, they do not, in many instances, in the long run provide the most desirable solution to the problem. But often, at the present state of coating technology, solvent absorption or incineration are the only solutions to reduced solvent emission.

Crosslinking of low molecular weight oligomeric polymers with amino resins presents a special problem in the network formation of crosslinked polymers. To achieve acceptable properties, the conversion of functional groups on the polymer backbone has to be driven higher compared to higher molecular weight polymers.

## POLYMER BACKBONES FOR HIGH SOLIDS COATINGS

To achieve the desired reduction in solvent content of solvent borne coatings, either increased application viscosity, higher application temperature, or a change in the polymer backbone used is necessary. Although all three methods are commercially used, I will limit myself to the discussion involving a chemical change in the polymer backbone to achieve higher application

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Presented at the 59th Annual Meeting of the Federation of Societies for Coatings Technology, in Detroit, MI, October 30, 1981.

\*Chemical Research Div., 1937 W. Main St., Stamford, CT 06904.



## 1981 MATTIELLO LECTURER Werner Blank

Mr. Blank received his B.S. degree in chemistry from Technical University in his native city of Vienna, Austria. He began his professional career at the Applied Research Laboratory of Farbwerke Hoechst AG, in Frankfurt, where he studied the effect of additives on application characteristics and film properties of latex paint.

Mr. Blank gained additional experience in water-borne coatings and resin structure at Peter Stoll Lackfabrik and Vianova Graz, in Austria. This work led to a proprietary antifoam system for dip and flow coatings, which was commercially used in many automotive dip and flow-coat primers.

After leaving Europe in 1965, Mr. Blank joined the American Cyanamid Co. at its Research Laboratory in Stamford, CT, where his research efforts concentrated on the development of amino resins for electrocoating applications. Later, he devoted his efforts to the synthesis of electrocoating acrylic and epoxy primer resins. His work addressed the fractionation of

polymers during the electrodeposition process and the effect of this fractionation on turnover stability. This work led to the design of polymers for other water-borne applications and to the development of nonionic polyether polyols for high solids water-borne applications.

Mr. Blank's current position is Manager of the Resin Products Department in the Chemical Research Division of American Cyanamid. He is responsible for research and technical service in crosslinking agents and specialty polymers. His present research is concentrated on low temperature cure and the physical limitations in obtaining high crosslinking agents.

Mr. Blank is author of several publications and U.S. and foreign patents on the subjects of electrocoating, water reducible coatings, high solid systems, and crosslinking agents.

He has been a speaker for the Gordon Research Conferences, American Chemical Society meetings, and Federation meetings.

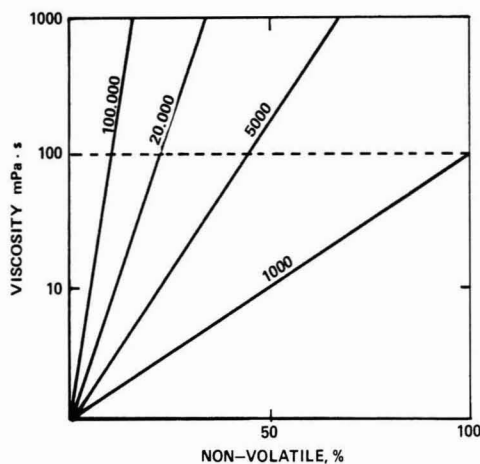


Figure 1—Viscosity - molecular weight relationship of polymers with similar composition

solids. Most existing application equipment requires a paint application viscosity in the range of 60–200 mPa · s.

An approximate relationship between average molecular weight (MW) of polymers, dynamic viscosity ( $\eta$ ), and nonvolatile content (NV) can be expressed in the following equation:

$$\log \eta = C \cdot \overline{MW}^{1/2} \cdot NV \quad (1)$$

C represents a constant for a specific polymer

Figure 1 shows the theoretical viscosity nonvolatile relationship of polymers with similar composition. The molecular weights chosen are typical for those found in industrial coatings. The assumption of similar composition is necessary for comparison. Practical, useful polymers with similar end use properties would be different in functionality and also monomer composition, as the molecular weight is changed.

The highest molecular number ( $\overline{MW}$  100,000) shown in this figure is for a typical thermoplastic acrylic resin. The next resin, with molecular weight of 20,000, would be characteristic of a thermoset acrylic similar to those used in automotive finishing.

A decrease in average molecular weight to 5,000 or less is required for high solids application.

For the very sensitive application in automotive topcoats, a decrease in average molecular weight below 5,000 is not possible because of performance and application problems. For most other coating applications, high solids polymers with an average molecular weight

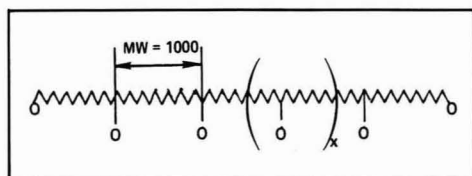


Figure 2—Structure of linear polymer with functional endgroups and uniform spacing of functional sites along the main chain

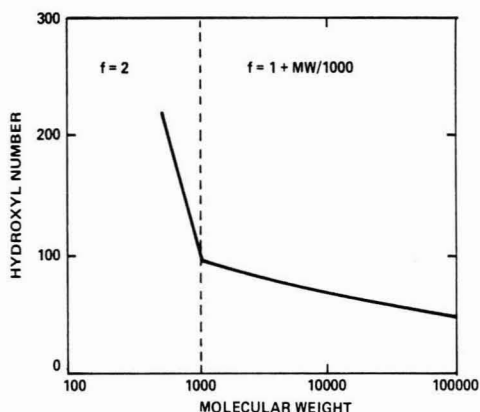


Figure 3—Change in hydroxyl number of polymers as function of molecular weight

as low as 1,000 are acceptable. Acrylic technology is normally not suited to produce these extremely low molecular weight polymers. Therefore, polyesters are the prime contender for this extreme high solids market.

As shown in Figure 1, the  $\overline{MW}$  100,000 polymer has an application solids of about 10% at 100 mPa · s viscosity. A thermoplastic acrylic coating for automotive applications that might utilize such a high molecular weight resin can have an actual weight solid at application viscosity of about 15–20% because of pigmentation and other additives.

Thermoset automotive acrylic coatings utilize lower molecular weight resins with an average molecular weight of 20,000 or higher. Compared to the higher molecular weight thermoplastic resins, the thermoset resins have a higher functionality. Application solids for a paint using such a resin is higher than that shown in Figure 1. Because of the addition of pigments and low molecular weight crosslinking agents, an application of 30–35% solids dependent on pigmentation is common for thermoset acrylic automotive coatings.

Reduction of the molecular weight of a polymer to 5,000 brings us into the range of presently tested high solids coatings for automotive topcoat applications. With the use of monomeric melamine crosslinking agents and pigments, the application solids can be between 50–65 weight percent.

Theoretically, a 1,000  $\overline{MW}$  polymer should give the

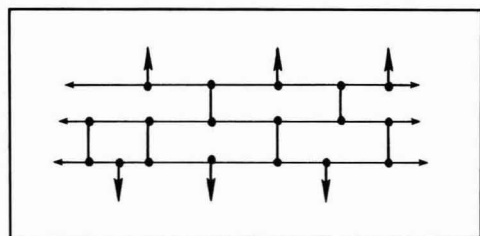


Figure 4—Ideal network structure of high molecular weight polymer and difunctional crosslinker agent

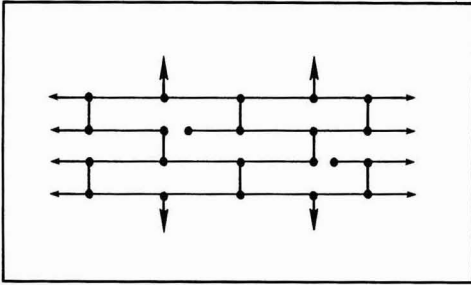


Figure 5—Ideal network of lower molecular weight polymer and difunctional crosslinking agent

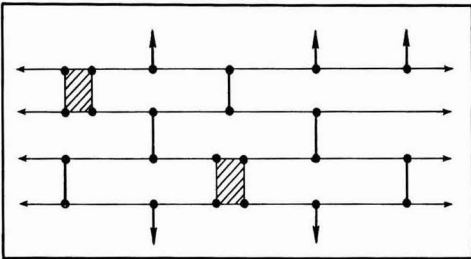


Figure 6—Ideal network of low molecular weight polymers and di and tetra functional crosslinker

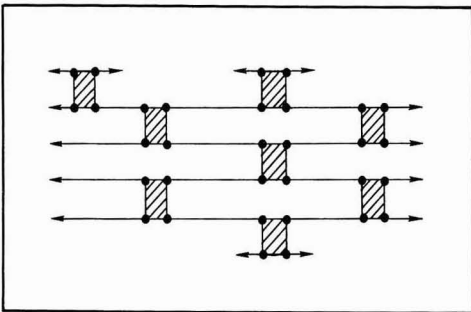
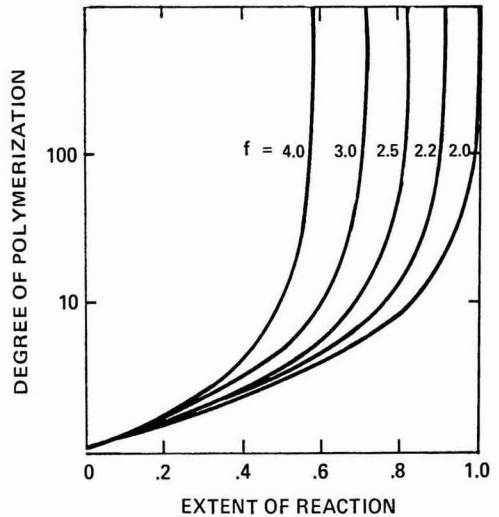


Figure 7—Ideal network of difunctional polymer and tetra functional crosslinker

“ideal” 100% nonvolatile coating. Unfortunately, the content of functional groups in such a polymer has to be significantly higher than that of previous discussed polymers to achieve an acceptable crosslinking density during cure. This higher functionality raises the viscosity significantly and also increases the weight loss during cure with amino resins. Other problems encountered are wetting of surfaces and poor pigment dispersions. These have to be overcome by addition of higher molecular weight polymers. Not much is to be gained by further reducing the molecular weight because these problems are further enhanced. Therefore, the practical limit for high solids coatings cured by amino resins is normally around 80 weight percent.

SYSTEM  $A_f + B_2$



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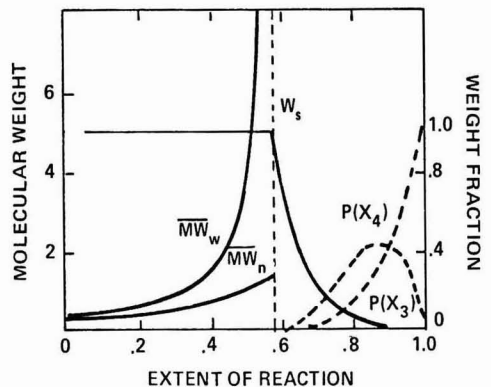
Figure 8—Degree of polymerization of polyfunctional system vs extent of reaction

### FUNCTIONALITY OF POLYMER BACKBONES

As described earlier, the content of potential crosslinking sites on a polymer is related to the molecular weight of the polymer. Also important are the desired end use properties of the coating and the nature of the crosslinking agent used.

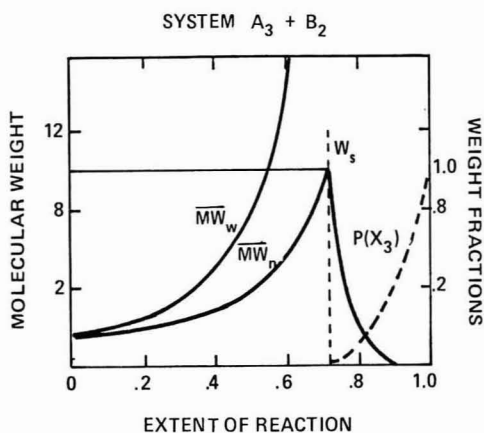
A thermoplastic resin, of course, would contain no crosslinking sites, only some small amount of functional sites to enhance adhesion. Therefore, this type of resin will not be discussed.

SYSTEM  $A_4 + B_2$



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Figure 9—Molecular weight and soluble weight fractions vs extent of reaction of a polyfunctional



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**Figure 10**—Molecular weight and soluble weight fractions vs extent of reaction of a polyfunctional system

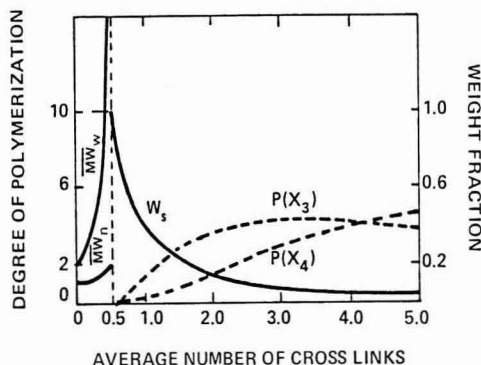
It is very difficult to predict theoretically the requirements for crosslinkable sites on polymers with different molecular weight to achieve similar properties; however, we can make some reasonable assumptions.

Let's assume we are interested in building polymers of different molecular weight which result, when cross-linked, in a similar crosslinked network, so that we have an average distance between crosslinks of, say, 1,000 MW. We can construct such a polymer, as shown in *Figure 2*, by having functional endgroups and regular spacing of functional sites, with an average distance corresponding to MW of 1,000 between functional sites. We choose initially to design the polymer with very high molecular weight. We can express the total functionality of a polymer chain as

$$f = 1 + n \quad (2)$$

$$n = \overline{MW}/1000 \quad (3)$$

$n$  represents the repeating unit in the polymer containing one functional group.



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**Figure 11**—Degree of polymerization of multifunctional polymer chain vs average number of crosslinks per chain

$$f = 1 + \overline{MW}/1000 \quad (4)$$

If the molecular weight is high, the total functionality is high and the concentration of functional groups is virtually independent of the molecular weight. As we decrease the molecular weight, the functional endgroups become a large portion of the total functional group content. The concentration of functional sites also increases, as shown in *Figure 3*.

For a molecular weight below 1,000, we assumed a minimum functionality of two. Using these assumptions, there is a steady increase in functional group content from 1000,000 to 1,000. From there, the concentration of functional groups rises rapidly.

## CROSSLINKED NETWORK FORMATION

Crosslinking in a polymer is a random process following statistical laws, resulting in a network that is far from the ideal structure one can visualize.

*Figure 4* shows such an ideal structure formed from a high molecular weight polymer with regular spacing of functional groups and a low concentration of endgroups. The crosslinking agent is difunctional and complete reaction is achieved. Please note, that every crosslinking agent shown is difunctional but the junction formed by this crosslinker connects to four polymer chains.

If we decrease the molecular weight of the polymer so that the concentration of endgroups compared to the functional sites in the chain is high, a difunctional crosslinker would result in a film with lower crosslinking density (*Figure 5*).

To achieve equal crosslinking density we would need a tetrafunctional crosslinker for the endgroups (*Figure 6*).

The extreme case of a crosslinked network using a polymer with a molecular weight of 1,000 would require solely a tetrafunctional crosslinker (*Figure 7*) to achieve the crosslinking density of a high molecular weight polymer utilizing a difunctional crosslinker.

In the normal process of network formation of sites with equal reactivity, we encounter a random process of interaction of functional groups. The network obtained is therefore defective. Not only is complete conversion of functional groups impossible, but some chains will have only a few crosslinks and others many. The reaction of polyfunctional compounds with functional sites of equal and different reactivity has been extensively described in the practical and theoretical literature and the resulting network formation has been discussed.<sup>1</sup>

Of practical concern, in a coating system, is the gel point of a polyfunctional system and the network formation past the gel point. Macosco<sup>2</sup> shows the relationship weight average DP as a function of the extent of the reaction of a two component system consisting of a first reactant with functional groups A and a second reactant with functional groups B. The functionality of compounds with functionality group A was changed from 4, 3, 2.5, 2.2, to 2.0. The functionality of compound B was kept constant at 2.0. Equal molar ratios of A to B groups were used. As shown in *Figure 8*, the DP of the molecule rises rapidly for the system with  $A_4$  functionality at about 0.57 conversion of the groups and, for the



lower functionality of A, 3.0, 2.5, 2.2, and 2.0, the extent of the reaction has to be about 0.71, 0.82, 0.92, and about 1.0, respectively.

This change in average degree of polymerization with the same extent of reaction is important in understanding the different behavior of a crosslinking agent in high functional, high molecular weight polymers and in lower functional, oligomeric systems. We can assume that for a practical coating system to have acceptable properties, we have to obtain an extent of reaction of the functional groups of Polymer A and B at least to the gel point and probably beyond. Therefore, for the lower molecular weight, lower functional polymer, higher conversions are required to achieve useful properties if the same crosslinker is used as for the higher functional, high molecular weight polymer. To obtain similar properties at equal extent of reaction, it would require an increase in functionality for the crosslinker used with the lower functional polymer.

The point of gel structure formation also has significant implication during film formation of a coating. Past the gel point, diffusion, not convection, becomes the main means of transport in a film. In the case of a crosslinking reaction involving a condensation mechanism, the volatile reaction products can lead to blister formation. The tendency of a system to show blistering at a given film thickness is higher with systems having a gel point at low conversion.

At the gel point of a polyfunctional system, only few polymer chains have reached infinite molecular weight. The majority of the chains are still soluble and reacted to a different extent.

They represent the sol fraction of the gel ( $W_s$ ). The amount of sol fraction rapidly diminishes as shown by Miller, et al.,<sup>3</sup> Figures 9 and 10. The systems shown have a tetra/difunctional and tri/difunctional functionality. Figure 11 (Miller<sup>3</sup>) gives similar information regarding the gel point and sol fraction of a linear polymer chain. Not unexpected for a high polymeric chain, only 0.5 crosslinks per chain are sufficient to achieve the gel point.

Most crosslinking reactions cannot lead to complete conversion of functional sites. If we, indeed, have random statistical reactions of functional groups on the polymer and the crosslinking agent, we would expect that a considerable amount of sites on the polymer and the crosslinker remain trapped in the polymer matrix.

Flory has pointed out that, depending on the functionality of the polymer chains, the crosslinking agent, and the conversion of functional groups, any real polymer network contains different amounts of terminal chains bound only at one end to the crosslinked network; the remainder of the chain is free and unattached and such chains contribute nothing to the properties of the network.

Scanlan and Case<sup>4,5</sup> have properly defined the concept of elastically active network chains (EANC). The Scanlan and Case EANC concept of network formation is more correct and simpler than other theories based on  $M_c$  (mean chain length between crosslinks). Allowance is made in the EANC concept for the presence of polymer chains either attached only at one end to the network or not at all. This is an important consideration in high solids coatings where the probability of partially

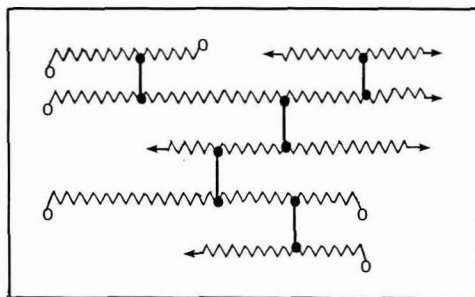


Figure 12—Defective network with elastic ineffective crosslinked chains

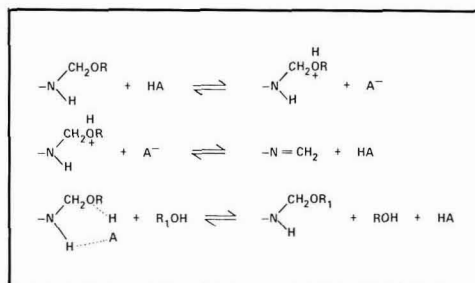


Figure 13—Potential reaction mechanism of general acid catalyzed amino resins

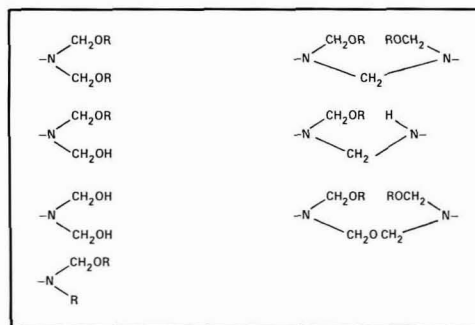


Figure 14—Functional groups on an amino resin responsive to specific acid catalysis

attached polymer chains is increased because of lower functionality of certain polymer backbone resins.

For crosslink junctions with a functionality, four fractions of junctions with various numbers of nonterminated chains are given according to Case<sup>5</sup> by the following relationship.

$$\begin{array}{l}
 \text{Junctions with 4 nonterminated chains} = (1-r)^4 \\
 \text{Junctions with 3 nonterminated chains} = 4r(1-r)^3 \\
 \text{Junctions with 2 nonterminated chains} = 6r^2(1-r)^2 \\
 \text{Junctions with 1 nonterminated chains} = 4r^3(1-r) \\
 \text{Junctions with 0 nonterminated chains} = r^4
 \end{array} \quad (5)$$

The term  $r$  represents the fractions of chains in a polymer with geometrical distribution of chain lengths.

The junctions with two nonterminated chains are

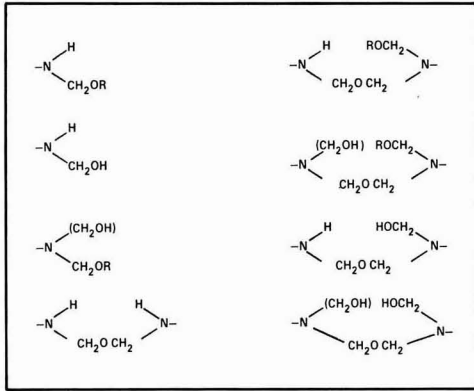


Figure 15—Functional groups on an amino resin responsive to general acid catalysis

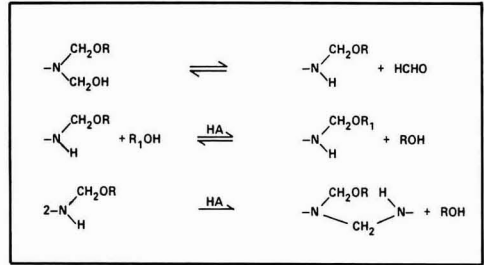


Figure 18—Reaction sequence of general acid catalyzed amino resin

located within the network but are not multifunctional network junctions. All of those junctions with more than two nonterminated chains are multifunctional network junction points.

Junctions with one nonterminated chain are attached to the network and those with 0 nonterminated chains are in the sol fraction.

For a crosslinked coating system with  $\nu$  junction points, the number  $\tau$  of chains actively linking multifunctional junctions is given by the expression:

$$\tau = \nu/2 [4(1-r)^4 + (3)4r(1-r)^3] \quad (6)$$

$$= 2\nu(1-r)^3(1+2r) \quad (7)$$

An example of crosslinked polymer chains with network defects and different junction points is shown in Figure 12.

In order to obey the requirements of Case and Scanlan for an elastic active network (EANC), high solids coatings using low molecular weight and low functional polymers require a higher conversion of functional groups or crosslinking agents with increased functionality.

The formation of junction points in tetra, tri, and highly functional systems was calculated by Macosco and Miller. The probability (P) that a junction point is trifunctional ( $P(X_3)$ ) increases with the extent of reaction, as shown in Figures 9, 10, and 11. For systems containing a tetrafunctional reactant (Figure 10), initially junction points with trifunctionality are formed  $P(X)_3$  and only at very high conversion is tetrafunctionality achieved  $P(X)_4$ .

### CHOICE OF AMINO RESINS

As explained in the previous discussions on network formation, amino resins suitable for certain low molecular, low functional polymer backbone resins have to be higher in functionality than crosslinking agents used in lower solids, lower functional systems.

Also, Figure 3 illustrates that for lower molecular weight polymers, even with a minimum functional group content, a significant increase in functional group concentration is required to achieve a theoretical crosslinking density equal to that of higher molecular weight polymers.

We will see to what extent existing melamine crosslinking agents can satisfy the above stated requirements.

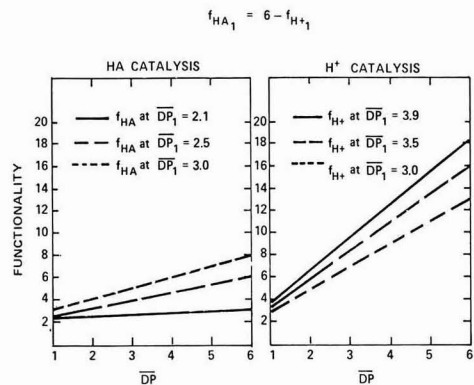


Figure 16—Functionality of fully alkylated melamine resins of different degree of polymerization as a function of structure and mode of catalysis

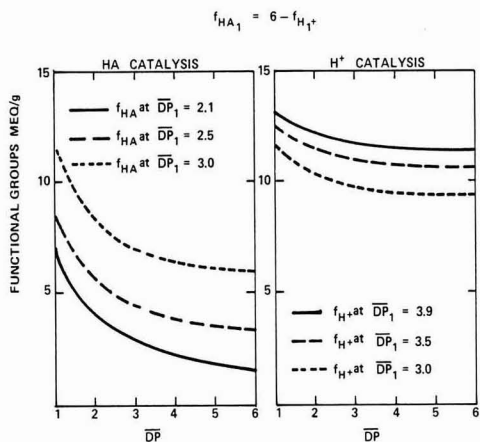
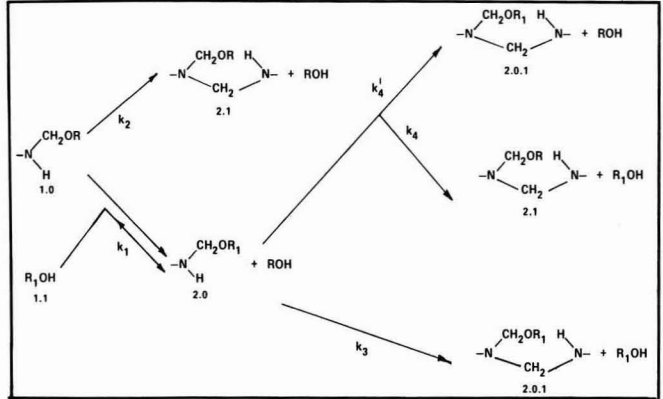


Figure 17—Functional group content of a fully alkylated melamine resin of different degree of polymerization as a function of structure and mode of catalysis

Figure 19—Reaction pathway of general acid catalyzed amino resin with hydroxy functional polymer



CROSS-LINKING GENERAL ACID CATALYSIS A1.0/A1.1=1.0  
 K1=1.0, K2=1.0, K3=1.0, K4=1.0, K4P=1.0

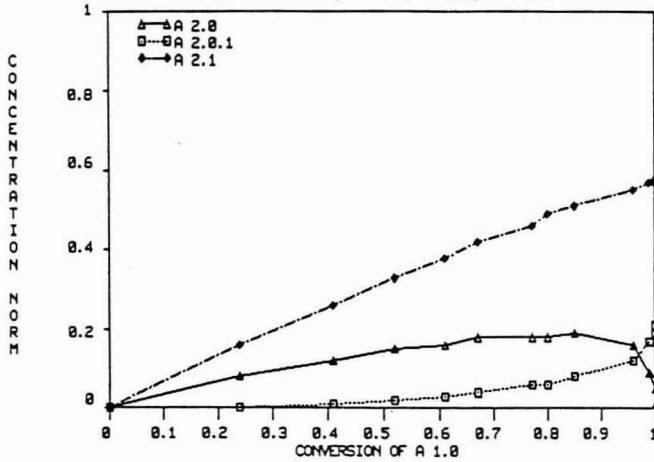
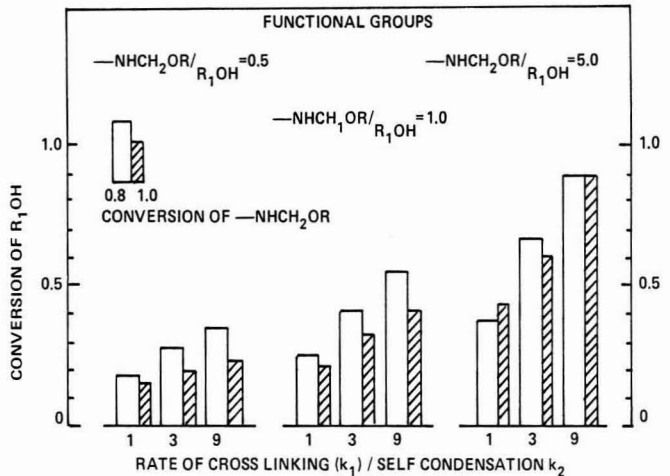


Figure 20—Formation of crosslinking and self-condensation structures in general acid catalyzed amino resin as a function of conversion of the amino resin

Figure 21—Conversion of functional groups on the polymer with general acid catalyzed amino resin with different relative rates of crosslinking and self-condensation



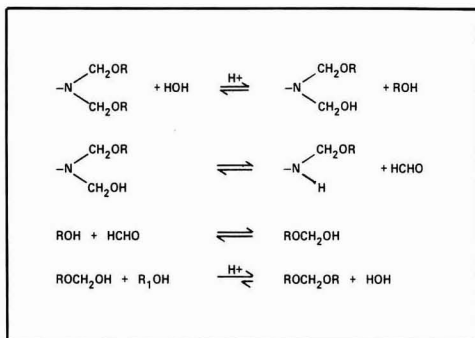


Figure 22—Hydrolysis of fully alkylated melamine resin during the curing process

As discussed by Berge,<sup>6,7</sup> alkylated melamine formaldehyde resins hydrolyze by either a specific acid or general acid catalysis mechanism, depending upon their structure. In subsequent work, Blank<sup>8</sup> has shown that specific acid and/or general acid catalysis is the reaction mechanism of different melamine resins during cure of a coating film.

The difference between specific acid catalysis and general acid catalysis for amino resins requires further clarification and explanation. Based on the definition of specific acid catalysis, the proton is the active specie in specific acid catalyzed reactions. Therefore, the rate of reaction will increase in specific acid catalyzed reactions with an increasing pKa value of the acid. In an organic system, depending on the solvent system, we have in all probability not a dissociated acid present, but an undissociated acid. The ability of the acid to protonate is, therefore, crucial, but not the presence of a proton. In going from an aqueous solution to a nonaqueous system, the acid strength of an acid actually increases.<sup>9</sup>

Not only can water compete with the amino resins for protons, so can additives, amines, and pigment surfaces. The rate of reaction of specific acid catalyzed systems will, therefore, often depend upon all of the ingredients in the formulation.

In general acid catalyzed amino systems we find no relationship between pKa value of the acid catalyst and catalytic effect. The catalytic specie is the undissociated acid and not the proton. Amine salts and larger levels of weak acid will be active catalysts for such an amino resin.

The definition of general acid catalysis does not exclude that specific acid catalysis can be faster or that both mechanisms are operative at the same time. Blank also showed that partially alkylated melamine resins first undergo a demethylation reaction which leads to an intermediate ( $-\text{NHCH}_2\text{OR}$ ) responsive to general acid catalysis (Figure 13).

Other functional sites on the melamine resin ( $-\text{N}(\text{CH}_2\text{OR})_2$ ) respond to specific acid catalysis (Figure 14). Considerable discussions have occurred in the European literature<sup>10</sup> on the mechanism of the specific acid catalyzed reaction. Berge<sup>11</sup> and also Saxon<sup>12,13</sup> interpreted experimental results to be evidence for a  $\text{SN}_1$  mechanism. Holmberg<sup>10</sup> and also Santer<sup>14</sup>

Table 1—Composition of Amino Resins

MOLAR RATIO per TRIAZINE	RESINS					
	HMMM	MBMM	HBMM	MMM-NH	MMM	PBMM
-CH <sub>2</sub> -	5.8	5.8	5.8	4.0	5.5	5.2
-OR	5.0 Me	3.6 Me, 1.2 Bu	4.1 Bu	3.0 Me	3.1 Me	2.7 Bu
-OH	0.3	0.15	0.5	0.4	1.5	≅1.0
>NH	<0.1	<0.1	<0.1	1.5	0.1	≅0.2

concluded from cure studies on coating films and gel point determinations that the reaction rate is dependent on the concentration of the functional groups on the crosslinker and backbone polymer and is, therefore, a second order reaction or  $\text{SN}_2$  reaction.

To draw conclusions about reaction mechanisms of a system which continuously changes viscosity as the reaction progresses is quite dubious. As shown by Rys,<sup>15</sup> selectivity of a reaction and reaction rates can be influenced significantly by the mixing of the reaction components. And any chemical event in a solution which occurs in a time shorter than 0.01–1.0 second might be hindered by mixing processes.

In our high solids polymer film, we might have in the early stages of the crosslinking processes conditions which approach solution kinetics, but as soon as the viscosity increases and we approach the gel point, diffusion and the collision of functional sites will determine the reaction rate. Solution kinetics is only applicable in a highly viscous or solid film if the functional groups form a complex prior to reaction and this complex is formed in the initial stages of the reaction. In any reaction which depends on the collision of functional groups which are not associated, diffusion has a major effect on reaction rates. A higher concentration of functional sites will increase the frequency of collision because of lower mean path length and, therefore, increase reaction rates. Regardless of the mechanism of the reaction,  $\text{SN}_1$  or  $\text{SN}_2$ , the rate of crosslinking of high solids polymers will be favored by a higher concentration of functional sites.

## GENERAL ACID CATALYZED AMINO RESINS

In previous papers, we have shown that<sup>8</sup> the sites responsive to general acid catalysis or a weak undissociated acid on an amino resin are predominately the  $-\text{NHCH}_2\text{OR}$  groups or the  $-\text{NCH}_2\text{OH}(\text{CH}_2\text{OR})$  functionality (Figure 15) which in a melamine resin can demethylolate and form the  $-\text{NHCH}_2\text{OR}$  group insitu. The self-condensation reaction of amino resins during the manufacturing process produces either methylene or methylene ether bridges between the amino compound. In melamine resins, the methylene group is predominately and thermodynamically favored if a lower formaldehyde charge is used in processing.

General acid catalyzed melamine resins are processed with a formaldehyde charge  $< 6$  and, therefore, methylene bridges are dominated. The alkoxyethyl groups next to a methylene group again respond to specific acid

Table 2—Characteristics of Backbone Polymers

	ACRYLIC	POLYESTER
MW <sub>n</sub>	5500	400
FUNCTIONALITY (OH)	8.8	2
HYDROXYL MEQ/g	1.61	5.0
CARBOXYL MEQ/g	0.54	0.05
NON-VOLATILE, %	75	96
SOLVENT	MAK	—
VISCOSITY mPa · s	7000	4500

catalysis and not to a weak acid catalyst. The maximum possible general acid catalyzed functionality on a melamine resin is, therefore:

$$f_{\text{HACAT(MAX)}} = \frac{(3\overline{DP}_n) - (\overline{DP}_n - 1)2}{\overline{DP}_n + 2} \quad (8)$$

and the maximum content of specific acid catalyzed sites

$$f_{\text{H}^+ \text{CAT(MAX)}} = \frac{(6\overline{DP}_n) - (\overline{DP}_n - 1)2}{4\overline{DP}_n + 2} \quad (9)$$

The maximum functionality available in a general acid catalyzed amino resin is significantly less than that available in a specific acid catalyzed resin. This reduced functionality of general catalyzed amino resins, compared to specific catalyzed resins could limit the utility of these resins in high solids coatings.

A simple calculation can show the theoretically available specific and general acid catalyzed sites of melamine resin with different structures.

Let's assume we start with a monomeric general acid catalyzed melamine resin with a structure [(triazine) f<sub>HA</sub>, f<sub>H<sup>+</sup></sub>] wherein f<sub>HA</sub> represents functional sites responsive to general acid catalysis and f<sub>H<sup>+</sup></sub> sites responsive to specific acid catalysis. We can express the functionality f<sub>HA<sub>n</sub></sub>, f<sub>H<sup>+</sup><sub>n</sub></sub> at a  $\overline{DP}_n$  as follows:

$$f_{\text{HA}_n} = f_{\text{HA}_1} \cdot (\overline{DP}_n) - (\overline{DP}_n - 1)2 \quad (10)$$

and

$$f_{\text{H}^+_n} = f_{\text{H}^+_1} (\overline{DP}_n) - (\overline{DP}_n - 1) \quad (11)$$

$$f_{\text{HA}_1} + f_{\text{H}^+_1} = 6 \quad (12)$$

As shown in Figure 18, two general acid catalyzed groups are lost in the formation of a self-condensed resin and only one group if the resin is specific acid catalyzed.

Using the above equations, we can calculate the theoretical functionality, equivalent weight or MEQ/g of functional groups of different melamine resins at specific or general acid catalyzed conditions.

Figure 16 gives the theoretical functionality (f<sub>HA<sub>n</sub></sub>, f<sub>H<sup>+</sup><sub>n</sub></sub>) of melamine resins with functionality responsive to general and specific acid catalysis as a function of  $\overline{DP}$  as calculated in equations (10) and (11).

Most practical commercial general acid catalyzed melamine resins have an average  $\overline{DP}$  from 2 to about 8. Commercial methylated melamine formaldehyde resins have a  $\overline{DP}$  from about 1.5 to 3 and butylated resin > 4.

The calculations show that the functionality of general acid catalyzed melamine resins rises rapidly with  $\overline{DP}$  if the functionality f<sub>HA<sub>1</sub></sub> is close to 3. For a functionality f<sub>HA<sub>1</sub></sub> of 2, no raise in functionality with higher  $\overline{DP}$  is expected. Specific acid catalyzed functionality on the

Table 3—Functional Groups of Amino Resin

	HMMM	MBMM	HBMM	MMM-NH	MMM	PBMM
$\overline{DP}$	1.7	2.2	2.9	2.1	2.5	6.2
FUNCTIONAL GROUPS PER MOLECULE						
CATALYST HA	—	—	—	4.0	3.8	7.4
CATALYST H <sup>+</sup>	9.0	10.9	13.3	7.1	11.5	22.9
FUNCTIONAL GROUPS MEQ/g						
CATALYST HA	—	—	—	6.8	4.7	3.0
CATALYST H <sup>+</sup>	14.7	12.4	8.9	12.0	14.4	9.2
MOLECULAR WEIGHT	610	880	1480	590	800	2490

other hand increases significantly with  $\overline{DP}$  regardless of f<sub>H<sup>+</sup><sub>1</sub></sub> in the starting molecular.

This calculation can explain why rather significant changes in performance with general acid catalyzed melamine resins can be seen with only slight changes in  $\overline{DP}$  or molar ratio of reactants.

Continuing this calculation, we can express the average molecular weight of the monomeric methylated melamine unit (MW<sub>DP</sub>) and the molecular weight of the polymerized molecule (MW<sub>DP</sub>) as:

$$\overline{MW}_{\text{DP}} = 126 + 44(6 - f_{\text{HA}_1}) \quad (13)$$

$$\overline{MW}_{\text{DP}_n} = (\overline{MW}_{\text{DP}_1}) \overline{DP}_n - (\overline{DP}_n - 1)32 \quad (14)$$

The results of these calculations are shown in Figure 17. The concentration of functional groups responsive to general acid catalysis drops rapidly with increasing  $\overline{DP}$ . The concentration of sites responsive to specific acid catalysis is only slightly influenced by  $\overline{DP}$ .

Let's assume we use a melamine resin with a functionality f<sub>HA</sub> at  $\overline{DP}_1 = 2.5$ . We find on Figure 17 that such a resin at a  $\overline{DP}$  of 2 has a theoretical MEQ/g of 5.7, functional sites under general acid catalyzed conditions, and a theoretical MEQ/g of 11.3 at specific acid catalyzed conditions. If we use 30 weight percent of amino resin in a formulation, this amino resin could theoretically, assuming complete reaction with the polymer and no self-condensation, react completely with a polymer with hydroxyl numbers of 136 and 271 under general acid or under specific acid catalyzed conditions, respectively.

The literature reports that general acid catalyzed amino resins readily undergo self-condensation reaction.<sup>16</sup> This competing reaction can reduce the amount of functional groups available for crosslinking (Figure 18). Because of the reactivity of the -NHCH<sub>2</sub>OR<sub>1</sub> group formed during the crosslinking process, we can describe the total reaction pathway as follows (Figure 19). Computer simulations<sup>17</sup> of the total reaction sequence have shown that general acid catalyzed amino resins have limited ability to crosslink because of the competing self-condensation reaction (Figure 20). Higher rates of crosslinking vs self-condensation reaction can increase conversion of functional sites but complete reaction of functional sites on the polymer is unlikely.

Figure 21 gives the degree of crosslinking and self-condensation for different ratios of functional groups on the polymer backbone to crosslinking agent and different relative rates of self-condensation and crosslinking.

From these computer simulations, it can be concluded that high solids coatings using general acid catalyzed

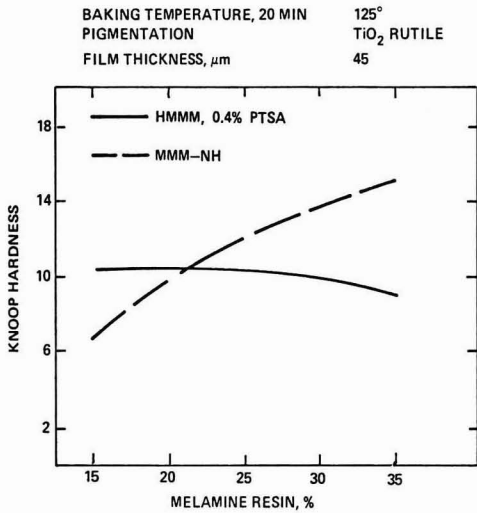


Figure 23—Film hardness development of acrylic-melamine resin systems at different levels of amino resin

amino resins require highly functional polymer backbones to assure sufficient reaction between polymer and crosslinking agents. Furthermore, difunctional reactive diluents will remain partially unreacted in such a coating.

**SPECIFIC ACID CATALYZED AMINO RESINS**

The requirement for a strong acid catalyst for cure of an amino resin is usually related to the degree of

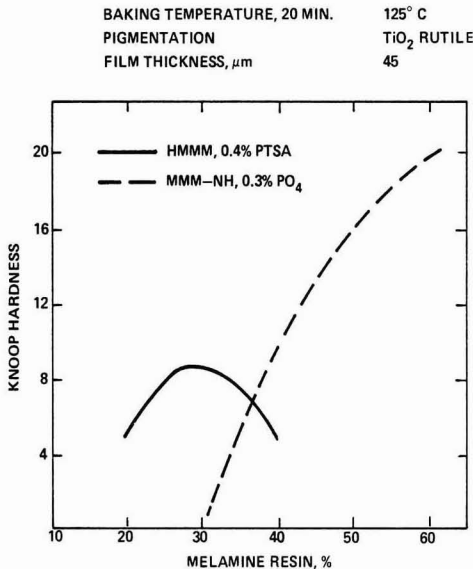


Figure 24—Film hardness development of polyester melamine resin systems at different levels of amino resin

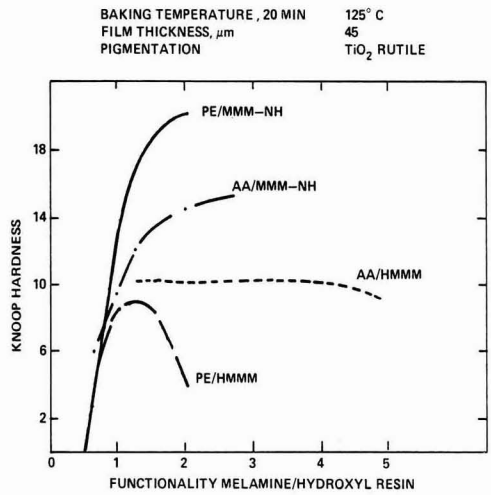


Figure 25—Film hardness development of a polyether and acrylic resin crosslinked with melamine resins as a function of the melamine functionality to hydroxyl ratio

alkylation. Fully alkylated amino resins require a strong acid catalyst; partially alkylated resins require a weak acid catalyst. This observation is only partially correct for melamine-formaldehyde resins and incorrect for many other resins. The difference in the type of acid catalyst required is related to the presence or absence of a hydrogen on the nitrogen next to the alkoxyethyl or methylol group (Figure 15).

Specific acid catalyzed amino resins show a high tendency for crosslinking and only a low tendency to self-condense.<sup>18</sup> The formation of methylol as the reaction product of the self-condensation reaction of hexa(methoxymethyl) melamine has not been found to be the major pathway for the formation of self-condensation products in thin films,<sup>18</sup> but migration of moisture in the air combined with hydrolysis of the alkoxy groups and subsequent demethylation has (Figure 22).

The stability of the nitrogen carbon linkage has a large effect on self-condensation and formaldehyde emission as shown by Parekh.<sup>19</sup> In glycoluril-formaldehyde resins, he demonstrated a significant reduction in formaldehyde emission and less self-condensation reaction compared to melamine resins. In high solids coatings, high functionality and high concentration of functional groups on the crosslinker, combined with low rates of self-condensation, is important for effective network formation.

Comparisons of a commercial hexa(methoxymethyl) melamine resin (HMMM) and a highly methylated partially methylolated melamine resin (MMM-NH) in a high solids difunctional polyester and a high solids multi-functional acrylic resin show the influence of different ability to crosslink on network formation and on film properties.

Table 1 gives the average DP, functionality, and concentration of functional groups under different conditions of catalysis of HMMM and MMM-NH resin. The

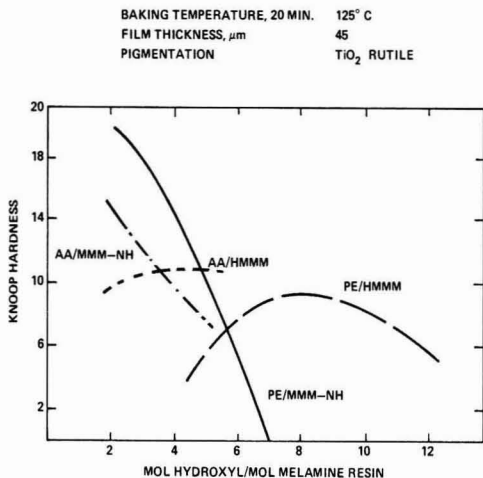


Figure 26—Film hardness for development of a polyester and acrylic resin crosslinked with melamine resins as a function of mol hydroxyl to mol melamine resin ratio

other amino resins listed will be described later. Table 2 lists the characteristics of the acrylic and polyester resin used.

In the highly functional acrylic resin ( $f = 8.8$ ), acceptable hardness is obtained over a wide range of melamine resin levels from 15 to 35 weight percent (Figure 23). The MMM-NH resin gives harder films with increased levels. For HMMM, higher levels actually decrease film hardness. If we repeat the experiment with the low functional polyester resin (Figure 24) we see a major change in behavior. Because of the low functionality of the polyester resin, we find gel formation and cure are obtained over a much narrower range of HMMM level. For MMM-NH resin, very large levels of melamine resin are required to achieve any cure or crosslinking. Weight percent of melamine resins, although the conventional method of comparing amino resins in a coating formulation, does not give a good explanation as to why these amino resins behave the way they do. We can get more information from our tests when we compare functional groups on melamine resin to hydroxyl groups vs hardness (Figure 25) or mol hydroxyl to mol of melamine resin (Figure 26). The hardness in the acrylic and the polyester system crosslinked with MMM-NH falls off rapidly when the ratio of functional groups on melamine to functional groups on acrylic is  $< 1$ . This point is about at the 80/20 acrylic/MMM-NH level and 60/40 polyester/MMM-NH level. This does not indicate that we can indeed react all groups on the polyester or acrylic resin with the MMM-NH melamine resin, but shows it to be the minimum required to achieve a network.

For the acrylic/HMMM and polyester/HMMM system we obtain optimum crosslinking between 1-1.5 functional groups on the HMMM to functional groups on the polymer. Plots of mol of hydroxyl groups per mol of melamine resins are also quite interesting (Figure 26). With the MMM-NH resin for both backbone polymers, if the ratio of mol OH to mol melamine resin is in excess of about 6, no crosslinking is achieved.

	HMMM	MBMM	HBMM	MMM-NH
ACRYLIC/MELAMINE	80/20	80/80	80/20	70/30
FUNCT. GR. MELAM/ACRYLIC	2.28	1.92	1.38	1.81
MOL OH/MOL MELAMINE RES.	3.91	5.63	9.46	2.20

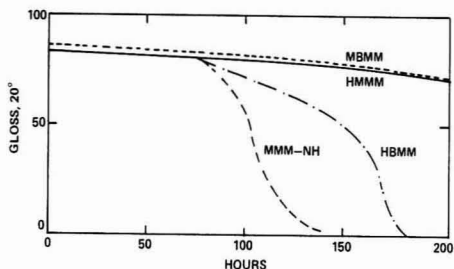


Figure 27—Detergent resistance of acrylic resin crosslinked with different melamine resins

For the HMMM crosslinked coating, we see differences for the polyester and the acrylic resin. Optimum hardness is achieved at a ratio of hydroxyl to HMMM melamine resin of 8 for the polyester and 3 to 5 for the acrylic. Please note that the HMMM and MMM-NH melamine resins had a  $\overline{DP}$  of 1.7 and 2.1, respectively; therefore, nine functional groups are theoretically available for

**ACRYLIC/CLA 70/30**

CURE TEMPERATURE °C	125
FILM THICKNESS $\mu\text{m}$	38
CATALYST, PTSA, %	0.4

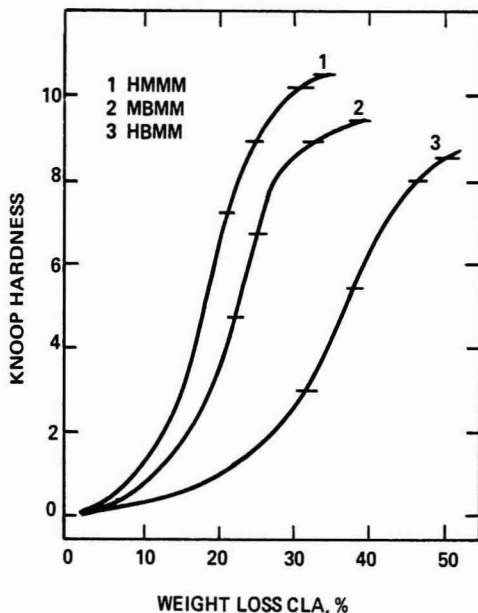
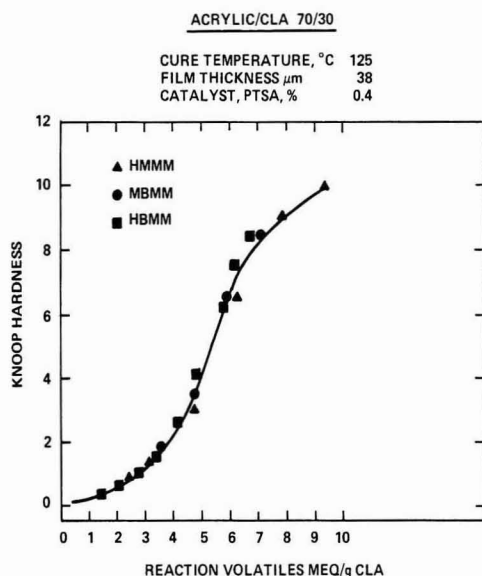


Figure 28—Film hardness development vs weight loss of an acrylic resin crosslinked with different fully alkylated melamine resins



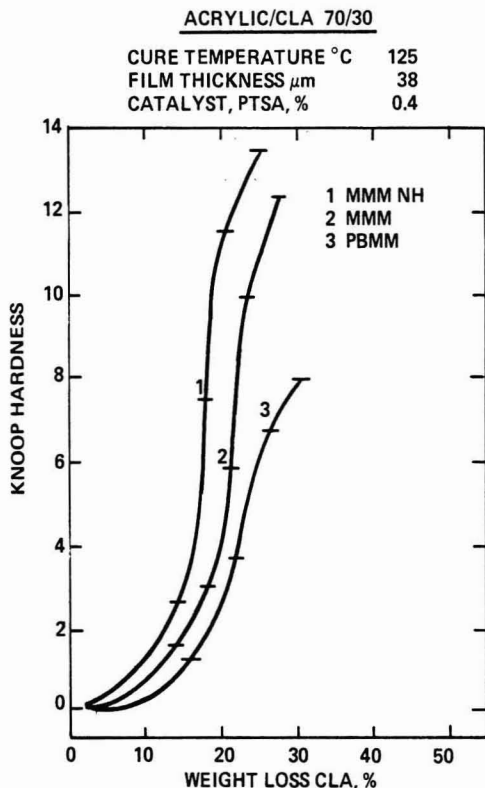
**Figure 29—Film hardness development vs molar reaction volatile loss of acrylic resin crosslinked with different fully alkylated melamine resins**

HMMM and four for MMM-NH (Table 2). The polyester/MMM-NH system was catalyzed with a dimethyl acid pyrophosphate catalyst at the cure temperature of 125°C. This acid is in the form of salt in this basic melamine resin. We can assume catalysis to be by general acid catalysis in such a buffered system. This assumption is based on cure studies with HMMM catalyzed with a strong acid catalyst in the presence of small levels of MMM-NH. The basicity of MMM-NH is sufficient to form a salt with a sulfonic acid inhibiting specific acid catalysis of HMMM and also cure at 125°C. Increased basicity was also observed by Berge and Dixon<sup>6,20</sup> for MMM-NH type crosslinking agents compared to HMMM.

Hardness measurements alone do not confirm our assumptions about incomplete conversion of functional groups with a MMM-NH type crosslinker. Based on our computer simulation, the reaction of functional groups on the acrylic resin should have been < 50% even when large levels (30–35%) of MMM-NH crosslinker were used. One indication of a deficient network can be formed by comparative resistance studies of fully and partially alkylated melamine resin crosslinked films (Figure 27).

Detergent resistance tests were performed on an acrylic resin crosslinked with HMMM, MMM-NH, a mixed methyl n-butyl methyl melamine resin (MBMM), and a fully butylated methylol melamine resin (HBMM). Detailed descriptions of these resins are in Tables 1 and 3. Although higher levels of MMM-NH resin are used (70/30), the detergent resistance of the crosslinked film is superior with the HMMM, MBMM, and HBMM crosslinked system at the 80/20 crosslinking agent level. For comparison, to gain better insight into the crosslinking process, we calculated mol functional groups on

the melamine resins to hydroxyl groups on the acrylic and mol hydroxyl per mol of melamine molecule. The molar ratio of functional sites on the melamine resin to hydroxyl groups was 2.28, 1.92, 1.38, and 1.81 for HMMM, MBMM, HBMM, and MMM-NH, respectively. If the sites on these amino resins have equal ability to crosslink, one would assume the poorest resistance properties for HBMM and about equal resistance for MMM-NH and MBMM. Molar ratios of hydroxyl groups to mol of melamine resin were 3.91, 5.63, 9.63, and 2.2 for HMMM, MBMM, HBMM, and MMM-NH. Higher conversion of functional groups on the acrylic polymer should be possible if the average functionality of the melamine resin is lower. For the average MMM-NH molecule, an average functionality of 2.2 is necessary to react all hydroxyl groups on the acrylic resin. This amino resin gives the poorest resistance properties, again indicating that the actual crosslink density obtained with this resin is lower than that of the fully alkylated amino resin. For the more polymeric HBMM resin, an average theoretical molecular functionality of 9.5 is required to react all functional groups. There is a very low probability that such a high functionality of a crosslinker can be achieved. The loss of resistance properties for the HBMM crosslinked film might be explained by the inability of a more polymeric crosslinker to achieve the high functionality necessary



**Figure 30—Film hardness development vs weight loss of an acrylic resin crosslinked with partially alkylated melamine resins**



to give complete conversion of sites on the polymer backbone.

In the automotive industry, there is considerable interest in crosslinking high solids acrylic resins with monomeric melamine resins. We were interested in the extent that the choice of alkylation alcohol influences the development of film hardness and how hardness relates to weight loss during the curing process.

HMMM, MBMM, and HBMM were used as crosslinkers for an acrylic resin (Table 2). The crosslinkers were used at an equal weight level of 30%. The system was cured for 5, 10, 20, and 40 minutes at 125°C and catalyzed with 0.4% p-toluene sulfonic acid. The results are shown in Figure 28.

The HBMM crosslinker loses considerable more weight than MBMM and this crosslinker loses more weight on cure than HMMM. One would expect a larger weight loss of a coating if n-butanol is lost as the reaction product compared to methanol or a mixture of n-butanol/methanol. Because the melamine resins were used at an equal weight and not molar basis in the formulation, one could interpret the different hardness obtained as a result of residual alkoxy group plasticizing

ACRYLIC/CLA 70/30

CURE TEMPERATURE °C	125
FILM THICKNESS μm	38
CATALYST, PTSA, %	0.4

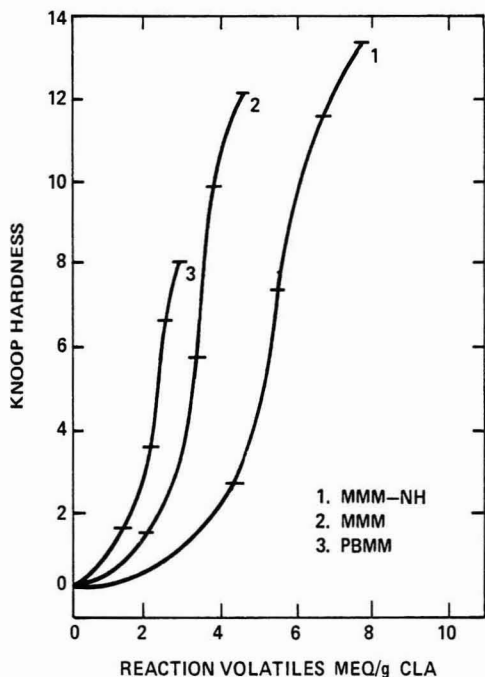


Figure 31—Film hardness development vs molar reaction volatile loss of an acrylic resin crosslinked with partially alkylated melamine resins

BAKING TEMPERATURE, 20 MIN. 125° C  
FILM THICKNESS, μm 40

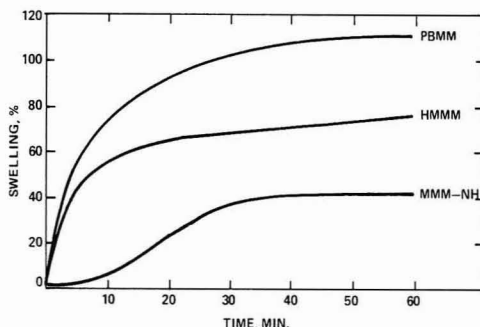


Figure 32—Solvent swelling of acrylic resins crosslinked with different melamine resins

the film or the triazine resin contributing to the hardness of a coating. Plots of mol reaction volatile lost vs hardness (Figure 29) dispell this notion.

The hardness developed in the coating depends solely on the mol of reaction volatiles lost or crosslinks formed and not on the structure of the crosslink or molar levels of crosslinkers used. Surprisingly, even the difference in DP between HMMM and HBMM has no influence on these results.<sup>21</sup>

The fact that neither the amount of triazine in the coating nor the residual alkoxy groups on the crosslinker have any effect on hardness development indicates that any mobility of polymer chains, within a certain distance from the crosslink, is severely restricted so that any compositional variations within this region do not contribute to mechanical properties.

Similar tests made with a partially alkylated butylated melamine resin, PBMM, a high NH methylated resin, MMM-NH, and a partially methylated melamine resin, MMM (See Tables 1 and 3 for composition), gave different results. Although, the weight loss increase (Figure 30) is in the expected order, MMM-NH, MMM, PBMM, the loss of molar reactants or hardness does not fall on the same curve for these three amino resins. For MMM-NH, reaction volatile is predominately methanol; for MMM it's formaldehyde and methanol; and for PBMM it's formaldehyde and n-butanol.

A potential explanation for the differences in hardness development for fully methylated melamine resin and partially methylated melamine resins is that the network formed in the fully alkylated resin is homogenous wherein crosslinking density is directly related to hardness development. In the partially methylated or high NH melamine resin, the network formed can be heterogenous.

We have shown in our computer simulation that self-condensation is a major reaction in high NH melamine resins. Therefore, incompatibility between the polymer backbone and crosslinking agent could develop as fractions of the crosslinker increase in molecular weight without grafting on the polymer backbone. With the

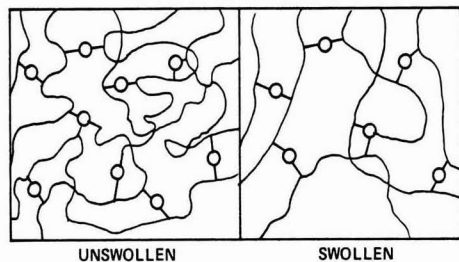


Figure 33—Network structure of acrylic polymer crosslinked with HMMM and swollen with solvent

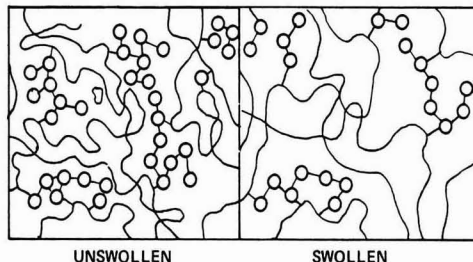


Figure 34—Network structure of acrylic polymer crosslinked with MMM-NH and swollen with solvent

right composition of the crosslinker these higher molecular weight fractions of the crosslinker could become incompatible with the matrix, precipitate, and form a second phase.

The crosslinking process could cause phase separation and formation of a heterogeneous network. The methylated high NH melamine resin, being compatible only at a low molecular weight, will partially precipitate after a certain increase in  $\overline{DP}$  due to incompatibility with the matrix. This portion of the amino resin which has separated from the matrix is still grafted to the rest of the polymer, but its contribution to hardness development is less.

Swelling experiments<sup>22</sup> of acrylic films crosslinked with MMM-NH, HMMM, and PBMM also seem to indicate a heterogeneously crosslinked structure with the MMM-NH resin (Figure 32).

Swelling in a solvent (xylene), which is a poor solvent for the methylated melamine (MMM-NH) resin rich phase, results in films which do not swell as much as either films crosslinked by a partially butylated melamine resin (PBMM) or hexa(methoxymethyl)melamine (HMMM). The crosslinking density of the HMMM crosslinked film, as measured by volatiles weight loss, was actually higher than that of the MMM-NH crosslinked coating (Figures 33 and 34).

Swelling experiments in a good solvent for self-condensed MMM-NH (DMF) give a significant increase in swelling which indicates that regions of the coating which are less swellable in the hydrocarbon are swollen under these conditions.

The butylated melamine resin, although prone to self-condensation, gives a harder film based on the molar loss of reaction volatiles. This is partially due to an initially higher  $\overline{DP}$  and also due to the high compatibility of the polymeric self-condensed crosslinking agent with the polymer backbone. Another factor which should contribute to the more uniform crosslinked network is the significantly higher molecular weight and viscosity of the butylated melamine resin, which might make it impossible for larger agglomeration to take place during the initial stages of the crosslinking process.

The formation of a visible second phase in melamine crosslinked coatings has been observed<sup>23</sup> in water reducible coatings. In these systems, precipitation of a high NH melamine formaldehyde resin occurred during evaporation of the water.

## CONCLUSIONS

The lower molecular weight and functionality of high solids polymer backbone resins require either an increase in conversion of functional groups during the crosslinking process or higher functional crosslinkers to achieve performance characteristics of regular coating systems.

The potential amount of functional reactive groups on an amino resin depends on the nature of the catalyst.

Under general acid or weak acid catalysis conditions, not all functional sites are reactive. Methylol or alkoxy-methyl groups on a nitrogen next to hydrogen are the reactive sites. Therefore, the maximum potential amount of functional groups on an amino resin, responsive to a weak acid catalyst, can only be half of that responsive to a specific acid catalyst.

Furthermore, computer simulation of the crosslinking process with amino resins reacted under general acid catalysis conditions has shown that the prime reaction with general acid catalyzed amino resins is the self-condensation reaction and in all probability less than 50% of the functional groups on the polymer backbone can be reacted with the crosslinking agent. The crosslinked network thus formed is quite imperfect and high functionality of the polymer backbone is essential for the formation of an elastic effective network.

Amino resins catalyzed with specific acid or a strong acid catalyst do not have the above limitation. It is possible to design these resins with the necessary high concentration of functional groups which predominately crosslink the functional groups on the backbone polymers and only to a lesser extent self-condense.

Polymers with low functionality and high concentration of functional groups are, therefore, effectively crosslinked with these amino resins and the network formed has fewer defects and also has a higher content of effective elastic network chains.

Mechanical properties and chemical resistance suffers especially if the crosslinked network is imperfect.

During the crosslinking process with amino resins, reaction volatiles are lost. We found that with fully alkylated melamine resins, loss of molar levels of reactants are directly related to hardness development. The composition of the melamine resin, the nature of the alkylation alcohol, and amounts of triazine in the amino resin had no effect on hardness development.

With partially alkylated, methylated, and butylated resins and high NH-methylated melamine resin, no relationship between hardness development and molar loss of reaction volatiles was observed. I postulate that the rather lower contribution to hardness development per molar level of reaction volatiles of the high NH methylated resin compared to the butylated melamine resin is a result of partial precipitation and formation of a second phase during the earlier stages of the crosslinking and self-condensation reactions. This leads to a network of self-condensed melamine formaldehyde resin which is interpenetrated by a partially crosslinked and attached resin phase. Evidence for this proposed network structure can also be formed in swelling experiments. Although, the crosslink and self-condensation reaction of the high NH methylated melamine resin based on the loss of molar reactants was about equal to that of the hexa(methoxymethyl)melamine crosslinked system, the swelling in toluene was significantly less. In the butylated melamine resin crosslinked system, we experience significantly larger swelling than with either the HMMM or MMM-NH crosslinked films.

The network formed is also, based on the loss of molar amounts of reaction volatiles, less densely crosslinked. Further work will be required to establish the nature of the crosslinked network obtained with different amino resins and to what extent the network structure can influence the mechanical and chemical resistance properties of a film.

#### ACKNOWLEDGMENT

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# Kinetics of Thermal Dissociation Of Blocked Isocyanate Crosslinkers

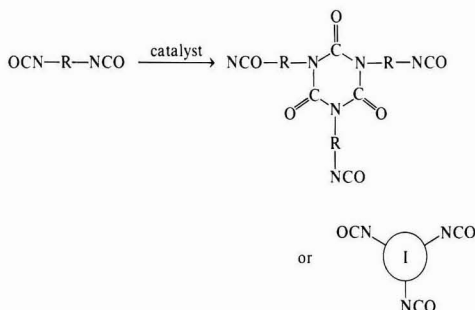
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
Novel isocyanurate crosslinkers containing blocked isocyanate groups were synthesized and the kinetics of thermal dissociation were studied by isothermal thermogravimetric analysis in air and nitrogen. Isocyanurate crosslinkers containing aromatic and aliphatic isocyanates were blocked with methyl-ethyl ketoxime and 2-ethylhexanol. The activation energies and frequency factors were measured and a method of calculating dissociation temperatures is proposed.

## INTRODUCTION

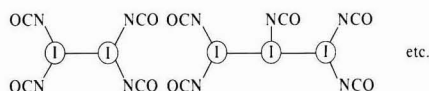
Recently, new isocyanates of higher functionality, containing thermally stable isocyanurate rings, have been developed. The main advantage of these isocyanates is that they can be used as higher temperature resistant crosslinkers in coating systems.

The isocyanurate containing isocyanates are prepared by cyclotrimerization of difunctional isocyanates according to the following equation:<sup>1,2</sup>

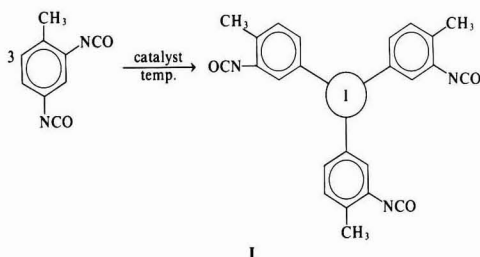


Where  is an isocyanurate ring.

Usually, the reaction does not stop in this stage and continues through the formation of polyfunctional oligomers.



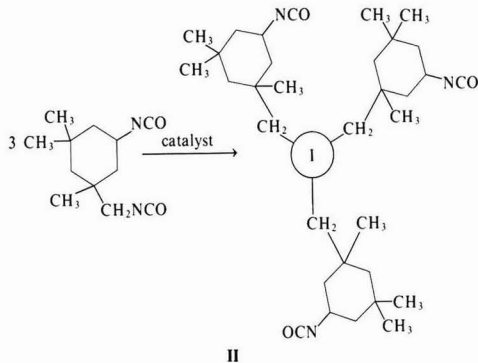
Therefore, the reaction has to be interrupted at specific conversion points and the catalyst deactivated in order to obtain a trifunctional product. In addition to the difficulty of obtaining a pure product during the trimerization of isocyanates, cyclotrimerization of 2,4 tolylenediisocyanate or isophorone diisocyanate produces isocyanates with lower reactivity. This is a result of the cyclotrimerization reaction which consumes the more reactive NCO— groups.



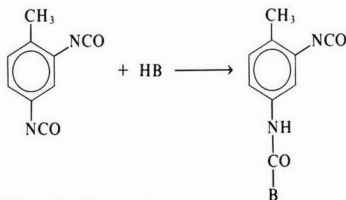
Presented by Andrew Dervan at the 59th Annual Meeting of the Federation of Societies for Coatings Technology, in Detroit, MI on October 29, 1981.

**Table 1—Elemental Analysis**

Model Crosslinkers	Actual %			Theoretical %		
	C	H	N	C	H	N
I .....	66.75	7.26	10.44	67.08	7.95	9.20
II .....	59.96	5.12	15.99	59.76	5.79	16.84
III .....	68.52	10.36	8.21	68.15	10.29	7.95
IV .....	63.17	8.69	13.50	62.11	8.80	13.58

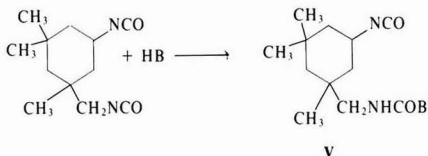
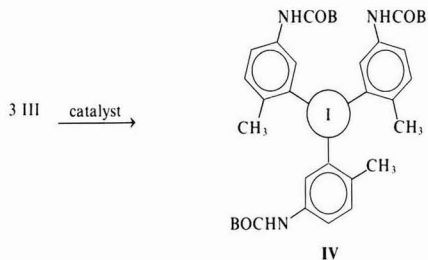


In this study, it was found that by utilizing a blocking reaction and a subsequent cyclotrimerization reaction, it is possible to prepare isocyanurate crosslinkers, containing blocked isocyanate groups of higher reactivity.

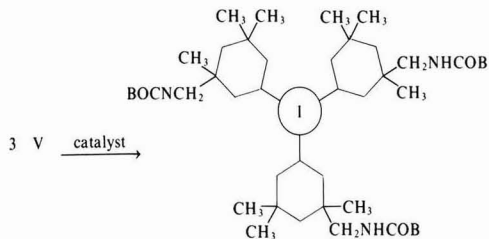


Where HB is a blocking agent

III



Where HB is a blocking agent.



By employing this synthetic method it is possible to prepare blocked isocyanurate crosslinkers that are pure trifunctional products.

A considerable amount of information exists in the chemical literature on blocked isocyanates. However, very few kinetic studies have been done on the unblocking process and the parameters that effect it.<sup>3-10</sup>

In this paper, the preparation of the blocked crosslinkers containing an isocyanurate and the kinetics of unblocking of these products will be described in detail.

## EXPERIMENTAL

### Preparation of Model Crosslinkers

The preparation of model crosslinkers was carried out in a two-liter round bottom resin flask equipped with a stirrer, reflux condenser, thermometer, and heating mantle. Throughout the reaction the flask was flushed with dry nitrogen to maintain an inert atmosphere. All solvents and blocking agents were dried with molecular sieves and filtered before use. The progress of the reaction was followed by measuring the NCO concentration by titration with dibutylamine solution or disappearance of the NCO peak in infrared spectra.

The actual synthesis was carried out in two stages. In the first stage, the blocking agents were reacted selectively with the more reactive NCO group at low temperature (below 50°C). In the second stage, the half-blocked isocyanate was cyclotrimerized at 85°-125°C using potassium octoate as a catalyst to form the isocyanurate crosslinker.

**MODEL CROSSLINKER I: TDI-Isocyanurate-2-Ethylhexanol Adduct**—2,4 Tolylenediisocyanate (3.0 moles) was weighed into the flask and 2-ethylhexanol was added dropwise over 40 minutes, the reaction was held below 50°C using a cold water bath. After two hours the conversion was 49.7%. Five grams of potassium octoate (0.55% on solids) was added to the flask. An exothermic reaction took place and 500 g Cellosolve acetate were added to stop the exothermic reaction at 125°C. The temperature dropped and held at 105°C. After two hours the trimer conversion was 98% and the reaction was terminated. The crosslinker was purified by repeated recrystallization using Cellosolve acetate and heptane.

**MODEL CROSSLINKER II: TDI-Isocyanurate-Methyl-Ethyl Ketoxime Adduct**—2,4 Tolylenediisocyanate (2.0 moles) was weighed into the flask and methyl-ethyl ketoxime (2.0 moles) was added, dropwise over 30

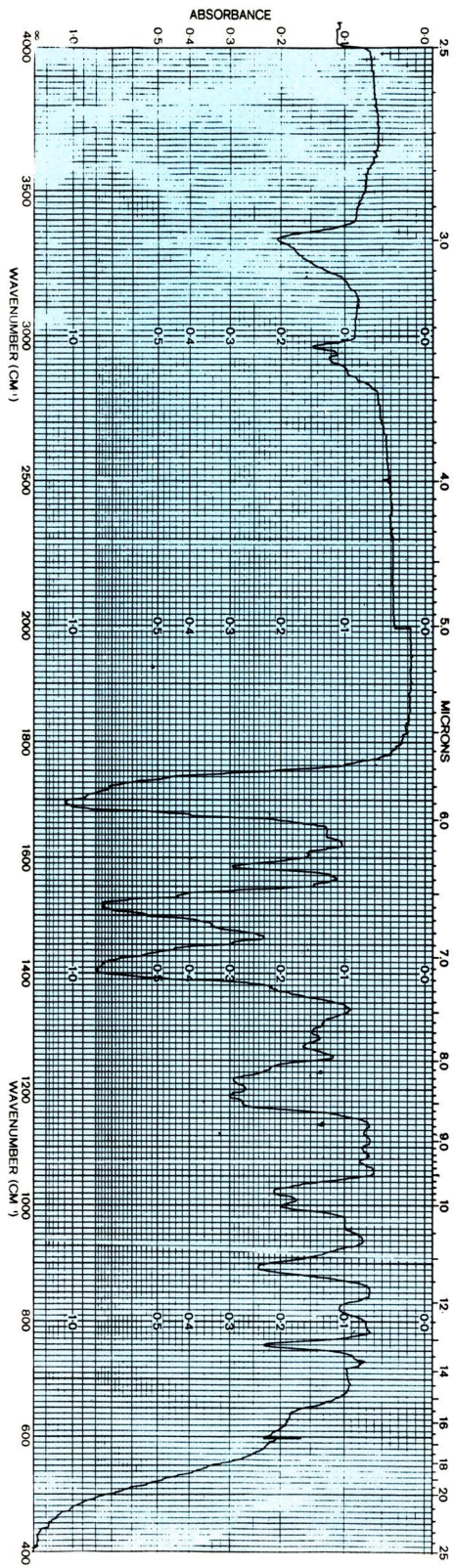


Figure 2—Infrared spectrum of TDI-isocyanurate - methyl-ethyl ketoxime adduct

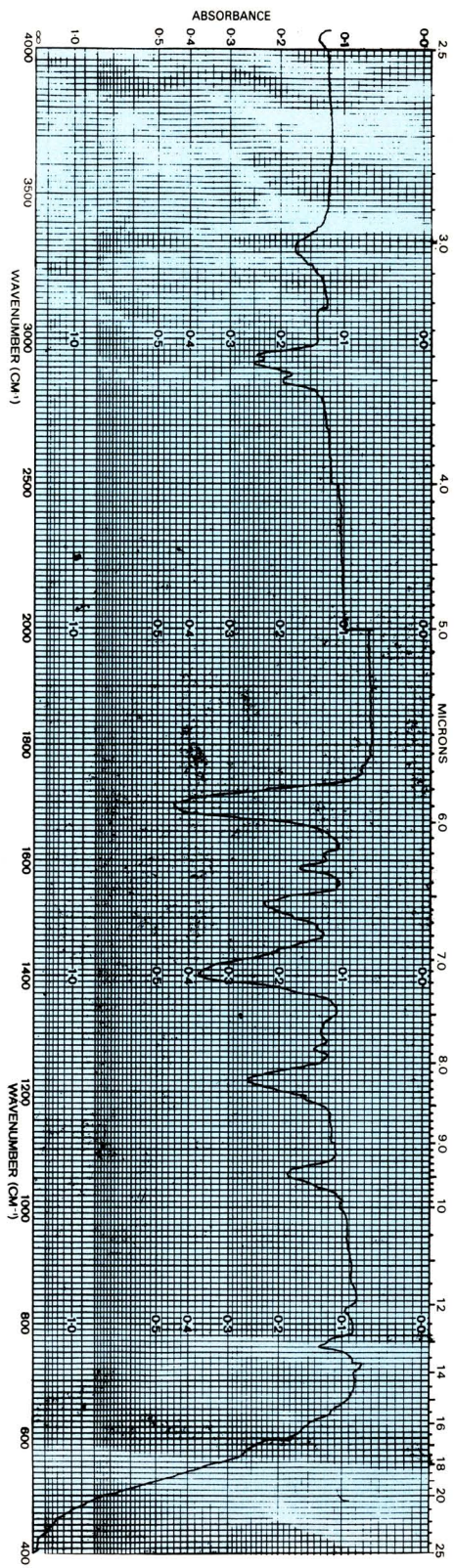


Figure 1—Infrared spectrum of TDI-isocyanurate - 2-ethylhexanol adduct

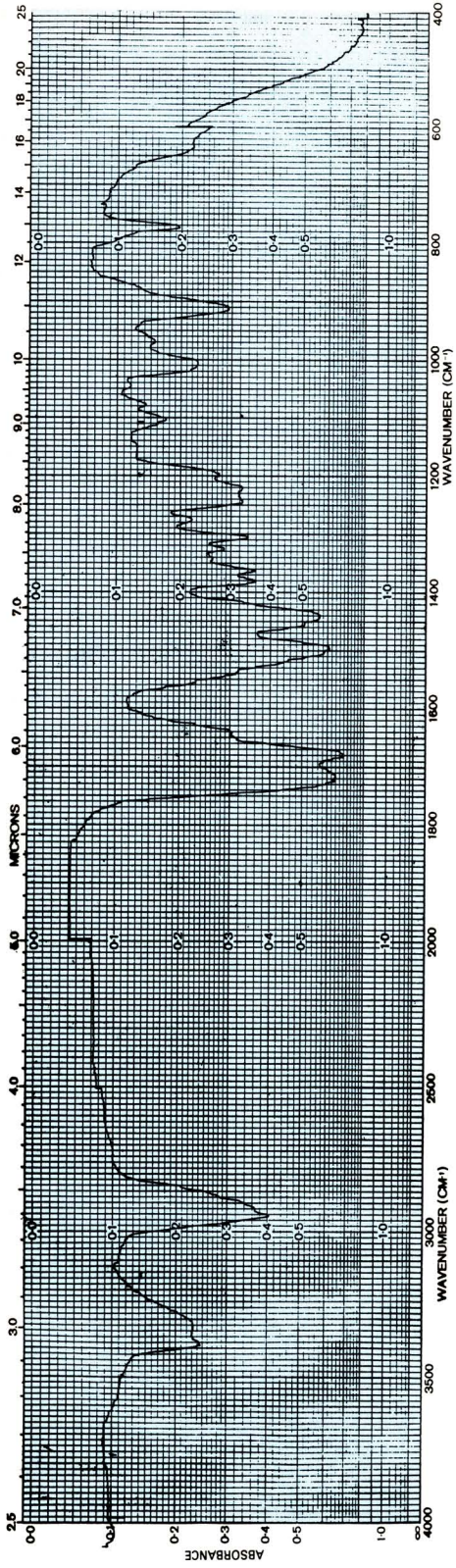


Figure 3—Infrared spectrum of IPDI-isocyanurate - 2-ethylhexanol adduct

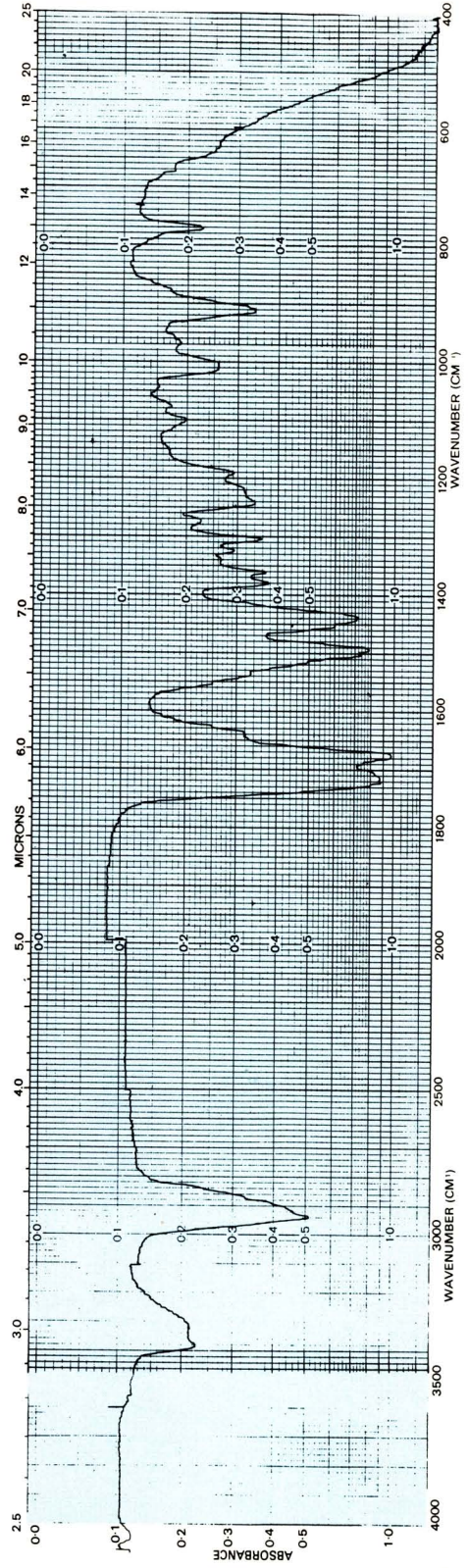
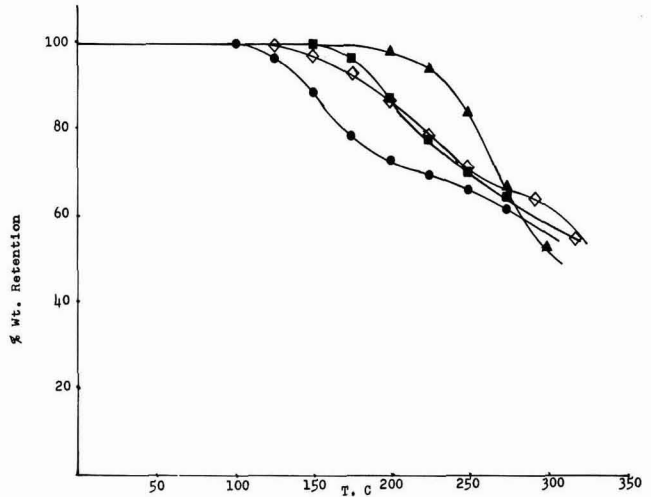


Figure 4—Infrared spectrum of IPDI-isocyanurate - methyl-ethyl ketoxime adduct



Figure 5—Dynamic TGA of TDI-isocyanurate blocked with (■) 2-ethylhexanol, (●) methyl-ethyl ketoxime; and IPDI-isocyanurate blocked with (▲) 2-ethylhexanol and (◇) methyl-ethyl ketoxime



minutes; the temperature was held below 45°C. After one hour the NCO conversion was 49.5%. The mixture was heated to 65°C, 0.8 g of potassium octoate (0.15% on solids) was dissolved in 50 g xylene and added dropwise to the reaction mixture. After 30 minutes the temperature rose to 85°C and an additional 80 g of xylene were added. After one hour the NCO conversion was 91.3%. 0.3 g of potassium octoate (0.06%) was dissolved in 100 g of xylene and added to the reaction mixture. After 45 minutes 100% conversion to trimer was achieved. The crosslinker was purified by repeated recrystallization using methyl-ethyl ketone and heptane.

**MODEL CROSSLINKER III: Isophorone-Isocyanurate-2-Ethylhexanol Adduct**—Isophorone diisocyanate (3.0 moles) was weighed into the flask and seven drops of dibutyl tin dilaurate catalyst were added. 2-ethylhexanol (3.0 moles) was added, dropwise over 45 minutes; the temperature was held below 50°C. After one hour, 2.0 g potassium octoate (0.18% on solids) was added and the reaction temperature rose to 80°C in 30 minutes. An additional 2.4 g of potassium octoate was added. After one hour at 130°C an infrared spectra showed no NCO peak and the reaction was terminated. The crosslinker was recrystallized from heptane.

**MODEL CROSSLINKER IV: IPDI-Isocyanurate-Methyl-Ethyl Ketoxime Adduct**—Isophorone diisocyanate (3.0 moles) was weighed into the flask and eight drops of dibutyl tin dilaurate were added. Methyl-ethyl ketoxime (3.0 moles) was added dropwise over one hour and the temperature was held below 50°C. After 30 minutes the NCO conversion was 49.8%. Potassium octoate (1.2 g, 0.13%) was added and the temperature rose to 80°C over 30 minutes. Three grams of potassium octoate was added and the reaction temperature began to rise. One hundred grams of Cellosolve acetate was added in order to control the temperature at 120°C. After eight hours, the trimer conversion reached 88.75% and would proceed no further. Fifty grams of cellosolve and 28 g of methyl-

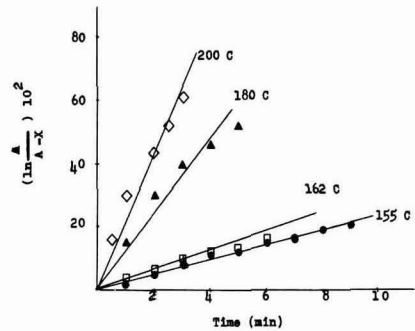


Figure 6—Kinetics of thermal dissociation of TDI-isocyanurate-2-ethylhexanol adduct in air

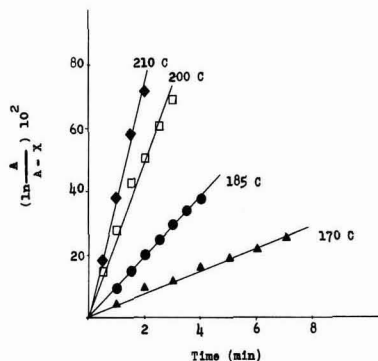


Figure 7—Kinetics of thermal dissociation of TDI-isocyanurate-2-ethylhexanol adduct in nitrogen

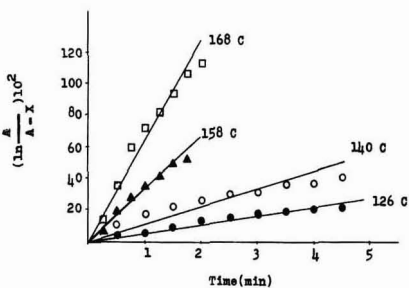


Figure 8—Kinetics of thermal dissociation of TDI-isocyanurate-methyl-ethyl ketoxime adduct in air

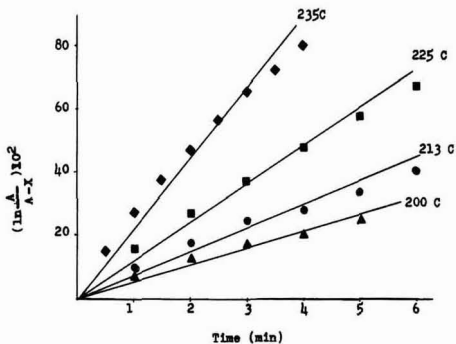


Figure 11—Kinetics of thermal dissociation of IPDI-isocyanurate-2-ethylhexanol adduct in nitrogen

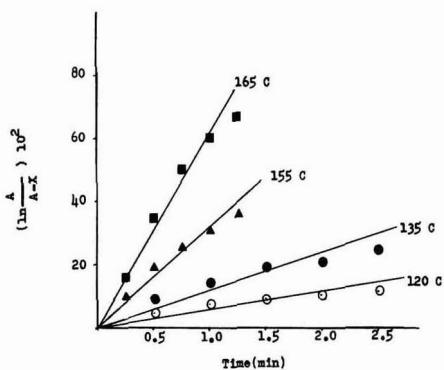


Figure 9—Kinetics of thermal dissociation of TDI-isocyanurate-methyl-ethyl ketoxime adduct in nitrogen

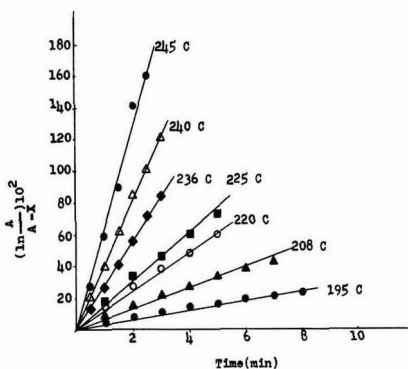


Figure 10—Kinetics of thermal dissociation of IPDI-isocyanurate-2-ethylhexanol adduct in air

ethyl ketoxime were added and then the reaction was terminated. The crosslinker was purified by repeated recrystallization from toluene and heptane.

### Measurement of the Thermal Dissociation of Model Crosslinkers

The thermal dissociation of novel crosslinkers was studied by means of Thermogravimetric Analysis (TGA). The thermogravimetric studies were conducted using the DuPont 951 Thermobalance. The work carried out with this thermobalance was of two distinct types: dynamic and isothermal. All measurements were carried out under nitrogen and compressed air. The furnace tube was flushed with the gas for 15 minutes before the start of the thermolysis. A flow rate of 50cc/min was maintained during thermolysis run.

The dynamic TGA curves were obtained at the linear heating rate of 5°C min<sup>-1</sup>. The accuracy of the temperature reading was ±1°C. The sample weight was 100-120 mg. All samples were dried at 60°C overnight and pulverized before being run.

The isothermal TGA measurements were obtained at different temperatures on a time-axis scale of two minutes per inch. The furnace tube containing an empty platinum pan was allowed to equilibrate at the desired temperature. A sample was placed quickly on the pan and suppression adjusted to 100%, interrupting the equilibrated temperature for only a few seconds before starting the measurements. Blank runs without samples at temperatures up to 230°C indicated that recovery time to reach the equilibrated temperature was 15-20 seconds.

### Analytical Methods

The infrared spectra were recorded on a Perkin-Elmer Spectrophotometer 457. The spectra were obtained by dissolving crosslinker on NaCl disc using methyl-ethyl ketone and flashed off solvent using an electric heat gun.

The crosslinkers were pyrolyzed at 220°C and the dissociation products were analyzed on a Hewlett-Packard 7620A gas chromatograph equipped with a flame ionization detector.

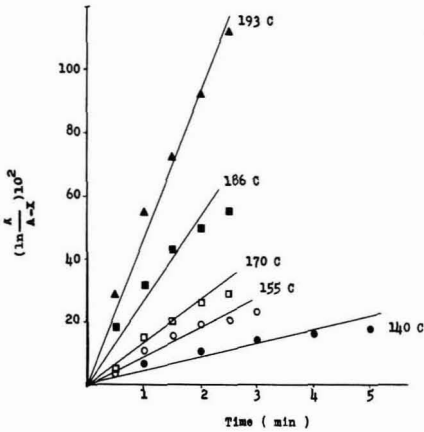


Figure 12—Kinetics of thermal dissociation of IPDI-isocyanurate - methyl-ethyl ketoxime adduct in air

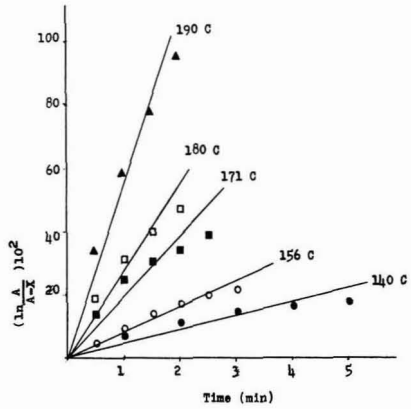


Figure 13—Kinetics of thermal dissociation of IPDI-isocyanurate - methyl-ethyl ketoxime adduct in nitrogen

**Elemental Analysis**

The elemental analysis C,H,N was determined by Midwest Microlab, Ltd., Indianapolis, IN. All samples were recrystallized repeatedly and dried before they were sent. The results of the analysis are displayed in Table 1.

**RESULTS AND DISCUSSION**

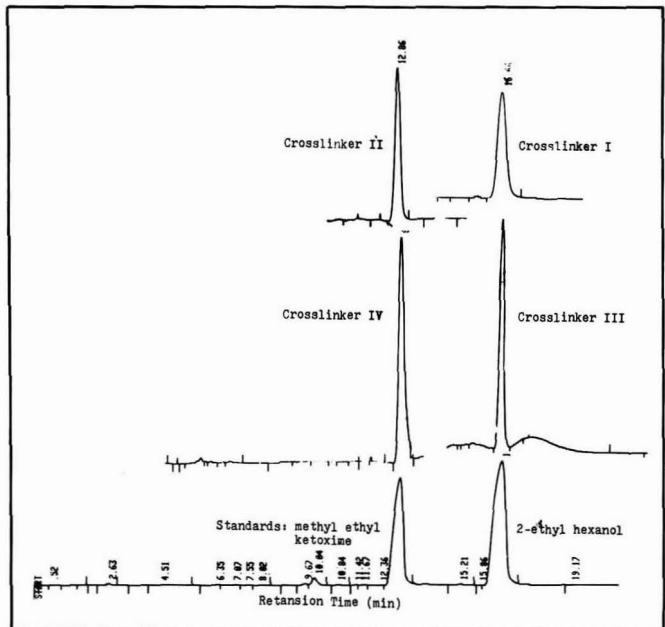
The blocked isocyanate crosslinkers, based on 2,4 tolylenediisocyanate and isophorone diisocyanate, were prepared by the procedure described in the experimental

section. Methyl-ethyl ketoxime and 2-ethylhexanol were used as blocking agents. The infrared spectra of the model crosslinkers are summarized in Figures 1-4.

The kinetics of deblocking of the model crosslinkers was determined by means of isothermal TGA. The dynamic TGA curves (Figure 5) showed that the deblocking of the model crosslinkers occurs in the temperature range of 110°-180°C increasing in the following order: TDI-Ketoxime crosslinker < IPDI-Ketoxime crosslinker < TDI-2-ethylhexanol crosslinker < IPDI-2-ethylhexanol crosslinker.

The macrokinetic measurements of deblocking deter-

Figure 14—GC analysis of decomposition products of model isocyanurate crosslinkers



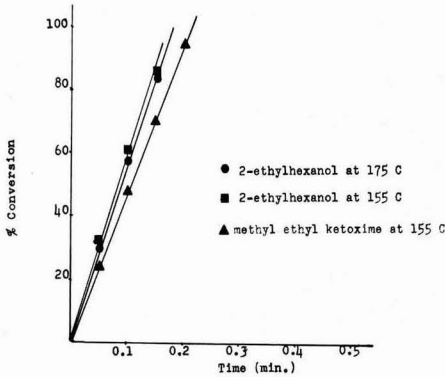


Figure 15—Evaporation rates for 2-ethylhexanol and methyl-ethyl ketoxime

mined by the isothermal TGA are shown in Figures 6-13. These experimental results showed that the crosslinker deblocking reaction followed first reaction order. The degradation products were analyzed (Figure 14) and it was found that the volatile decomposition products were 2-ethylhexanol and methyl-ethyl ketoxime, respectively. It was determined that the rate of evaporation of degradation products ( $k = 14.05 \text{ min}^{-1}$ , 2-ethylhexanol, 155°C) is significantly higher than the rate of decomposition and therefore, the dissociation reaction is the rate determining reaction (Figure 15).

The temperature effort on the thermal dissociation of the model crosslinkers was investigated and the results are summarized in Tables 2 and 3 and Figure 16. The isothermal TGA measurements were performed under air or nitrogen atmosphere. It was observed that the

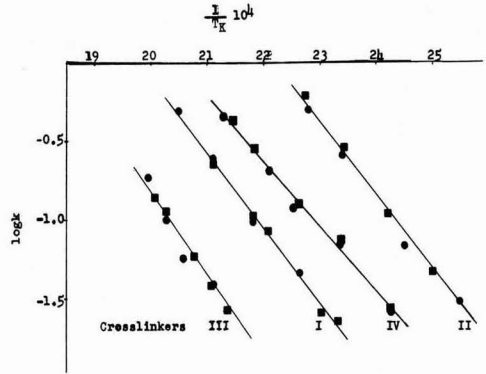


Figure 16—Arrhenius plots of model isocyanurate crosslinkers: (■) in air; (●) in nitrogen

kinetic data obtained in air or nitrogen were very close and that under those experimental conditions (time, temperature) apparently no thermal oxidation took place. The rate constants activation energies and frequency factors were calculated by using the following equations:

$$k = \frac{\ln \frac{100}{100-x}}{t}$$

$$k = A \exp \left( -\frac{E_a}{RT} \right)$$

Where  $E_a$  is the activation energy and  $A$  is the frequency factor. The calculated values of  $k$ ,  $E_a$ , and  $A$  are summarized in Tables 2 and 3. As can be seen from Table 2, the activation energies for dissociation of blocked

Table 2—Kinetics of Thermal Dissociation of Model Crosslinkers in Air

Model Crosslinkers	Temp. °C	$\frac{1}{T_k} \cdot 10^4$	$K \cdot 10^2 \text{ min}^{-1}$	$E_a$	$A_{\text{min.}^{-1}}$	$T_d \text{ } ^\circ\text{C}$
I	155	23.3	2.19	22.05	$3.3 \times 10^9$	178
	162	23.0	2.48			
	180	22.07	8.47			
	185	21.8	10.43			
	200	21.1	22.02			
II	126	25.0	4.63	21.86	$3.9 \times 10^{10}$	133
	140	24.2	10.84			
	158	23.2	28.59			
	168	22.7	62.78			
III	195	21.36	2.71	24.99	$1.3 \times 10^{10}$	212
	200	21.14	3.78			
	208	20.79	5.88			
	220	20.28	9.43			
	225	20.08	13.65			
IV	140	24.21	2.49	19.46	$0.5 \times 10^9$	157
	155	23.36	7.39			
	170	22.57	11.88			
	186	21.78	29.09			
	193	21.45	40.90			

Table 3—Kinetics of the Thermal Dissociation of Model Crosslinkers in Nitrogen

Model Crosslinkers	Temp. °C	$\frac{1}{T_k} \cdot 10^4$	$K \cdot 10^2 \text{ min}^{-1}$	$E_a$	$A_{\text{min.}^{-1}}$
I	168	22.6	3.67	22.52	$5.3 \times 10^9$
	185	21.8	9.60		
	200	21.1	24.36		
	215	20.49	40.88		
	120	25.44	3.02		
II	135	24.50	6.72	21.38	$2.0 \times 10^{10}$
	155	23.36	25.11		
	165	22.83	49.19		
	200	21.14	3.83		
III	213	20.58	5.64	22.05	$5.4 \times 10^9$
	220	20.28	9.78		
	235	19.69	18.30		
	140	24.21	2.49		
IV	156	23.31	6.77	23.90	$1.15 \times 10^{10}$
	171	22.52	11.39		
	180	22.07	19.82		
	197	21.27	21.27		
	140	24.21	2.49		

crosslinkers based on TDI and IPDI were in the 19-25 kcal range. In order to quantify the thermal stability of model crosslinkers the decomposition temperature,  $T_d$  was calculated for the same rate constant. The rate constant  $k = 0.0693 \text{ min}^{-1}$  was chosen arbitrarily and represents 50% dissociation of the model crosslinker if the thermal exposure was 10 minutes. The calculated value of  $T_d$  in the model crosslinkers are summarized in *Table 2*. As can be seen, the  $T_d$  values increase in the same order as was determined by the dynamic TGA.

## CONCLUSION

The model blocked isocyanate crosslinkers containing isocyanurate rings were prepared by cyclotrimerization of partially blocked isocyanates (TDI, IPDI). 2-ethylhexanol and methyl-ethyl ketoxime were used as blocking agents. The dissociation of the model blocked crosslinkers (first reaction order rate constants,  $E_a$ ,  $A$ , and  $T_d$ ) were calculated from the isothermal TGA data. It was established that the evaporation rate of volatile degradation products is very fast and does not influence kinetic data of the thermal dissociation of model crosslinkers.

It was determined that the model crosslinkers based on the aromatic isocyanate dissociated at lower temperatures than the aliphatic one. Similarly, the isocyanate adducts with methyl-ethyl ketoxime dissociated at lower temperatures than adducts with 2-ethylhexanol.

## ACKNOWLEDGMENTS

The committee wishes to thank Mr. F. Lewis for his valuable contributions to the analytical techniques used in this study and the Ford Motor Co., PP&V Div., Paint R&D, for permission to use their laboratories and TGA equipment.

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# Time-Lapse Infrared Spectroscopic Investigation of Alkyd and Linseed Oil Cure

Jack H. Hartshorn

E.I. du Pont de Nemours & Company, Incorporated\*

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The air-drying mechanism of a conventional soya alkyd and of a linseed oil has been investigated by a new technique, Time-Lapse Infrared Spectroscopy. This technique is capable of assessing not only the reactions taking place during polymer cure but when and in which order they occur. Each of these materials was found to follow a similar though different oxidation route. Both are initiated by oxygen via hydroperoxide formation; however, their generation/decomposition rates and their oxidation and crosslinking paths differed markedly. Several competing reactions are identified. Some of these predominate in the alkyd case while others prevail in the oil. These may be divided into three phases: an inhibition period, a relatively fast initiation, and crosslinking. The observed reactions are compared with some previously reported drying mechanisms.

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## INTRODUCTION

The chemical reactions by which vegetable oils and their successors, the oil modified alkyds, are converted from viscous liquids to functional solid films have fascinated chemists for decades. A great many theoretical mechanisms have been postulated to explain this phenomenon,<sup>1</sup> but the analytical proof of these proposed reactions has been limited by the experimental techniques available. The analysis techniques formerly employed required either destruction of the film or determination of some secondary physical property which may not necessarily be related to the reactions taking place.

However, investigators would prefer to examine the chemical changes taking place within the film while they are occurring and without disturbing the process. Infrared spectroscopy admirably fulfills this desire since cast films are among the favorite methods of sample preparation, and the absorption bands are a measure of the functional groups within the molecular structure. Infrared has thus been used for many years in this laboratory<sup>2</sup> and others<sup>3</sup> to examine resin curing.

Unfortunately, infrared is a rather insensitive technique and the subtle changes associated with cure are difficult to detect. For that reason, many studies have been limited to the later stages of drying and to the yellowing tendencies of alkyds and oils.<sup>4-6</sup> This insensitivity results from the fact that an infrared spectrum is an integrated representation of all the functional groups within the area of the film examined. The spectral changes which take place during cure are not very great since only a few of the many molecular bonds are involved and these may not be strong infrared absorbers. Carbon-carbon double bonds are notably poor absorbers in the infrared. In addition, the most significant bands for a particular study seem always to fall beneath or alongside the strongest feature in the spectrum, thus completely masking any minor change in intensity.

With the advent of computer supported infrared instruments, the sensitivity problem can be largely overcome. The infrared spectrum may now be converted into digital form where the computer can perform a point by point subtraction of one spectrum from another. This subtraction will effectively cancel unwanted bands and by doing so enhance the bands of interest. Features associated with structures not involved in the curing mechanism are eliminated so that attention is focused on those spectral shifts resulting from the reactions taking place. Particularly useful are the Fourier transform infrared systems with their greater speed, sensitivity, computer size, and data storage capacity. In addition, the arduous task of making frequent repetitive scans of

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\* Marshall R&D Laboratory, P.O. Box 3886, Philadelphia, PA 19146.

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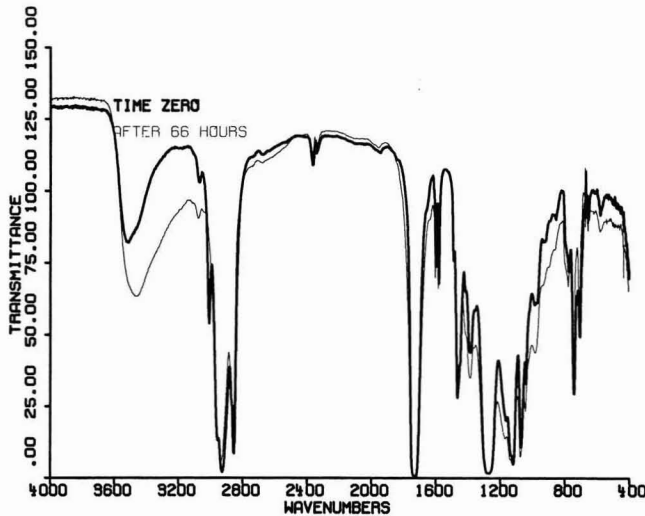


Figure 1—Soya alkyd before and after cure

the film as it cures can be delegated to the computer, thus the minor changes with time and the sequence of reactions can be assessed. A technique based on such an approach has recently been developed, called Time-Lapse Infrared Spectroscopy.<sup>7</sup> Using this technique, the air drying of a soya-alkyd and a bodied linseed oil has been examined and the results compared with some previously reported mechanisms.

**EXPERIMENTAL**

**Instrumental**

A dry-air purged Nicolet-7199 Fourier Transform System equipped with a standard TGS detector was instructed to co-add 100-2 cm<sup>-1</sup> resolution scans. These measurements were repeated at approximately two-hour

intervals using the interferometer oscillations as a clock. The collected interferograms were then mathematically converted to spectrograms by well-known Fourier transform computer algorithms<sup>8</sup> and stored in disk memory for further data processing.

**Soya Alkyd**

A conventional 60% soya oil pentaerythritol orthophthalic alkyd (Acid # = 5, GH Visc. = Z), to which cobalt naphthenate was added equivalent to 0.05% cobalt metal, was cast on a KBr plate to give full scale absorbance (~1% T) at the ester carbonyl band (near 1730 cm<sup>-1</sup>). The film was vacuum (~1 mm Hg) dried one hour at room temperature to remove the carrier solvent and then examined at room temperature in the dry-air instrument purge. The nominal drying time of this alkyd is 24-48 hours at room temperature.

Figure 2—Differential spectrum before and after cure

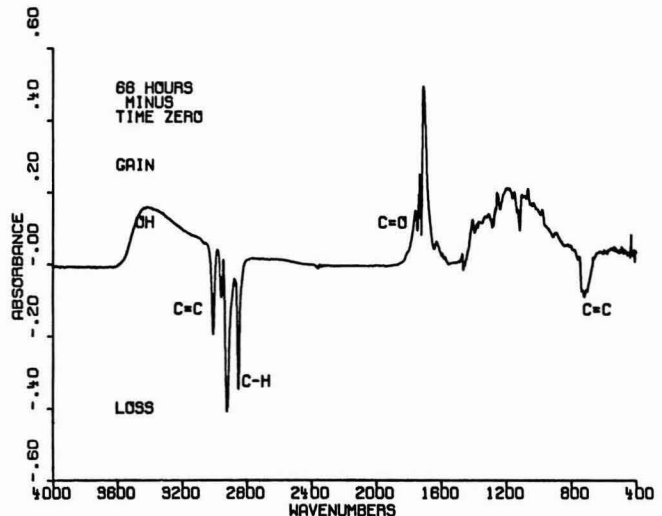
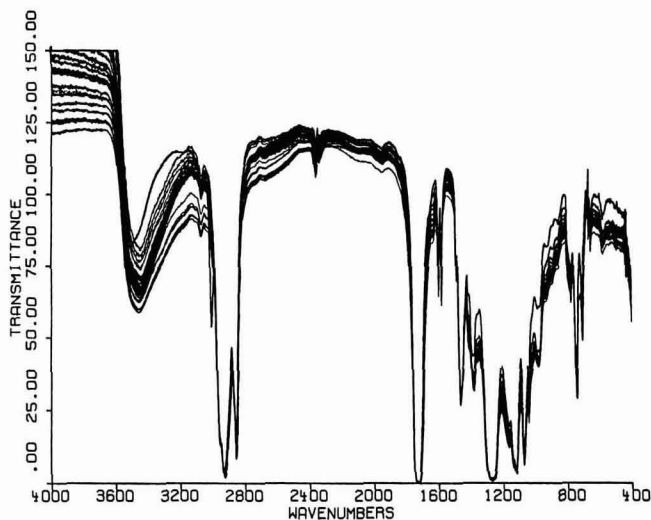




Figure 3—Twenty-nine time-lapse spectra



**Linseed Oil**

Linseed oil was heated (350–375°F) under an inert atmosphere until the GH viscosity increased to R. The cooled oil was catalyzed with 0.05% cobalt drier. Films were then prepared and examined in the same manner as the alkyd above.

**RESULTS AND DISCUSSION**

**Time-Lapse Infrared Technique**

This technique was developed to evaluate relatively slow, complex reactions such as the air drying of coatings. To illustrate this application, the soya-alkyd cure will be examined. Figure 1 shows the infrared spectrum of that alkyd. The heavy line represents the film immediately after carrier solvent removal under vacuum. The lighter line is a spectrum of the same film taken after air-

drying 66 hours, when the cure is presumably complete. A few changes in band intensity are apparent, such as the hydroxyl band increase around 3400 cm<sup>-1</sup>. Other spectral variations might easily be mistaken for a slight change in film thickness. As a precaution against that possibility, the film was allowed to cure undisturbed in the spectrometer beam. Thus, the same molecular array was examined throughout the experiment and any spectral variations can reasonably be assumed to have resulted from the curing reactions.

Since these spectra are now in digital form within the computer's memory, recently developed spectral subtraction techniques<sup>9</sup> can be applied. Figure 2 shows the result of subtracting the original spectrum from the final spectrum. In this format, those bands which are being generated point upward, while those being consumed point downward. The overall curing reaction shows an increase in the hydroxyl (near 3400 cm<sup>-1</sup>) content and a broadening of the carbonyl band (around 1750 cm<sup>-1</sup>)

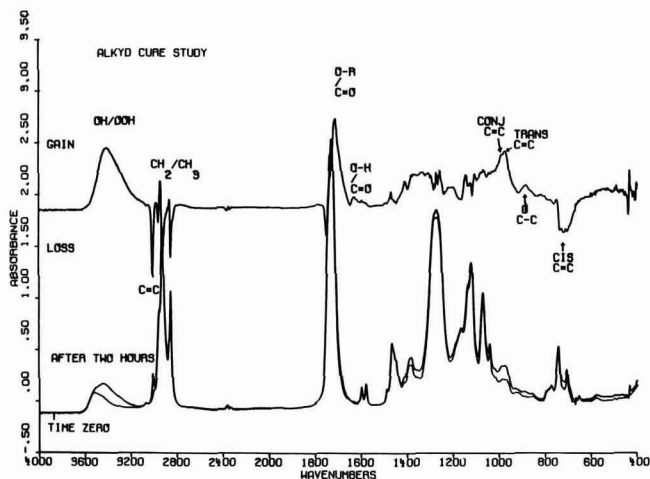


Figure 4—Initial and two hour alkyd absorbance spectra

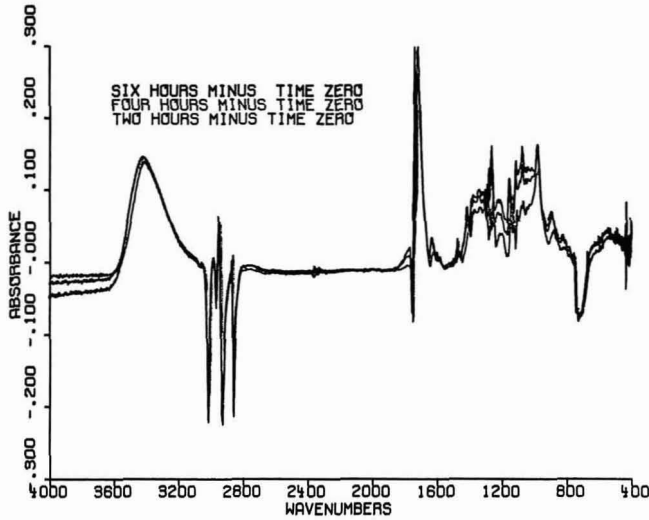


Figure 5—Differential alkyd spectra to six hours

indicating formation of several new oxidized forms. There has also been a loss of unsaturation ( $3010\text{ cm}^{-1}$ ) which can now be identified as the *cis* form by the band at  $720\text{ cm}^{-1}$ . Previously this band was completely masked by the orthophthalate bending modes at  $740$  and  $710\text{ cm}^{-1}$ . The loss of hydrocarbon, which was imperceptible before because of near spectral saturation in the C-H stretch region, is now evidenced by the reduced intensity of the  $\text{CH}_2$  bands at  $2930$  and  $2860\text{ cm}^{-1}$ . From this, it might be concluded that the *cis*-unsaturation has been oxidized to several carbonyls and that part of the aliphatic groups have disappeared. Interesting as this information may be, it tells little of what reactions took place and in what order.

Once an instrument has been committed to such a lengthy experiment, the computer would normally be idle waiting for the reactions to take place. However, it

is a relatively easy task to program the computer to take spectra at regular intervals and to store them in its memory. Most computers have or can be equipped with accessory disk or magnetic tape memories so that the entire cure process can be stored in a kind of time-lapse sequence. Figure 3 shows some 29 spectra taken at roughly two-hour increments during the 66-hour alkyd cure study. Since the total drying process is now stored as digital spectra, various subtractions can be performed and displayed in order to examine different facets of the curing mechanism.

### Soya Alkyd Cure

The progress of a reaction is difficult to assess when displayed in transmittance form. It is far easier to evaluate quantitative spectral shifts in absorbance presentation where the band intensities are proportional to

Figure 6—Incremental alkyd spectra at two hour intervals

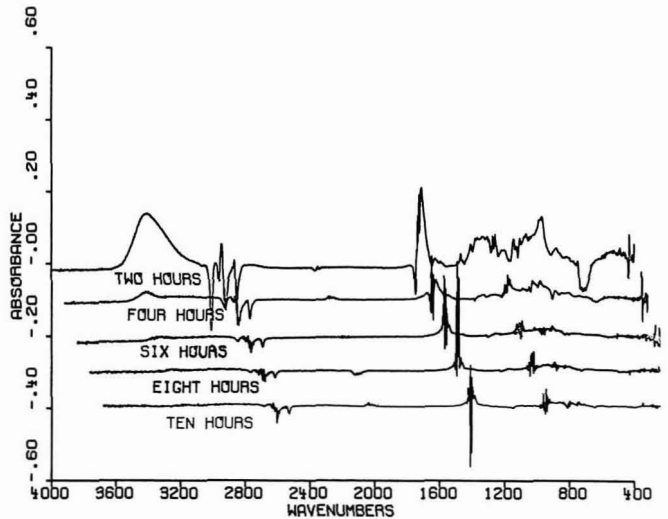
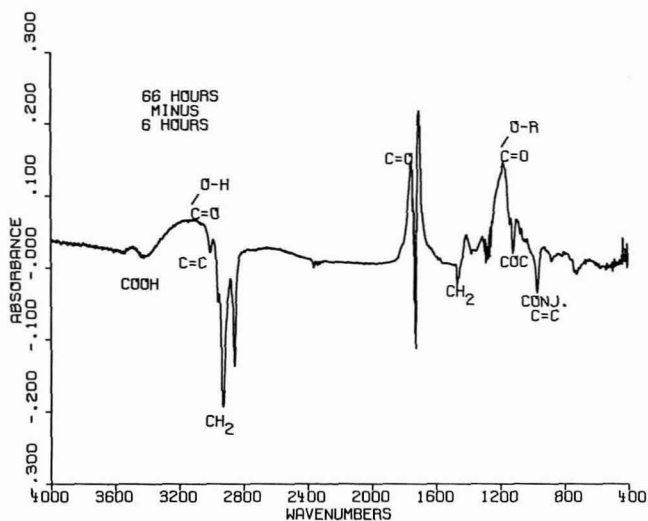


Figure 7—Differential alkyd spectrum between six and 66 hours



concentration. Figure 4 shows the original alkyd spectrum in absorbance and the stored spectrum taken roughly two hours later. Some small but significant changes have taken place within the film during this time as evidenced by the shifts in the spectra. These shifts can be magnified by a one to one spectral subtraction. If the film thickness had changed due to slumping of the film or some other physical change, a reduced intensity spectrum would have resulted with all the bands either above or below the baseline. Here, shifts can be found in both directions. During this time hydroxyl and/or hydroperoxide ( $3450\text{ cm}^{-1}$ )—more likely the latter—has been formed. At the same time an unsaturation band ( $3010\text{ cm}^{-1}$ ) has been consumed. This band can be identified as a *cis*-unsaturation since it is associated with the band at  $720\text{ cm}^{-1}$  which has also shrunk. The *cis*-unsaturation appears to have been isomerized, since bands normally assigned to *trans*-double bonds ( $970\text{ cm}^{-1}$ )

and conjugate-unsaturation ( $990\text{ cm}^{-1}$ ) are being produced. This is consistent with the formation of hydroperoxide on the alpha carbon to a *cis* double bond which is then isomerized into *trans* and conjugate forms as first proposed by Swern, et al.<sup>10</sup> Some ester and acid carbonyls (near  $1700\text{ cm}^{-1}$ ) appear to be formed during this period. It is difficult to envision this process unless a peracid-like structure is formed at some of the double bonds as proposed by Rieche.<sup>11</sup> That could result in a cleavage of the fatty acid chain and the ultimate loss of ketones, aldehydes, and acidic oxidation products.<sup>12</sup> These short chain oxidation products evaporate and give oil-based coatings their traditional "painty odor." They appear here as a loss of  $\text{CH}_2$  at  $2930$  and  $2860\text{ cm}^{-1}$ . A small band has also appeared at  $880\text{ cm}^{-1}$  which might be attributable to epoxide formation as the first stage of the oxidation. It should be pointed out that all of these reactions occurred within the first two hours of drying.

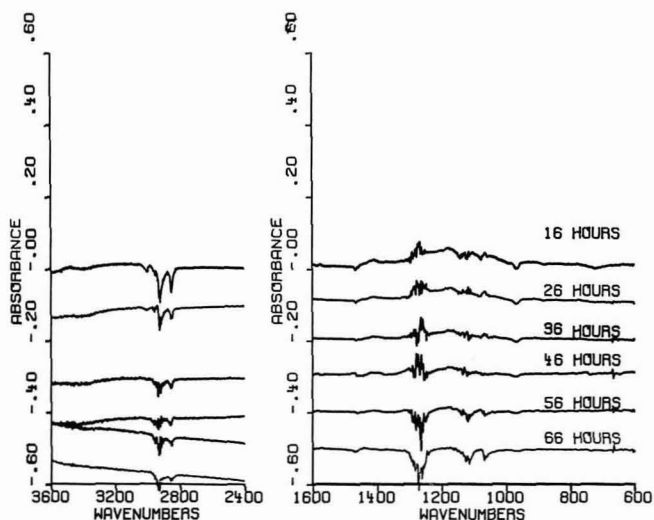


Figure 8—Incremental alkyd spectra between six and 66 hours at 10 hour intervals

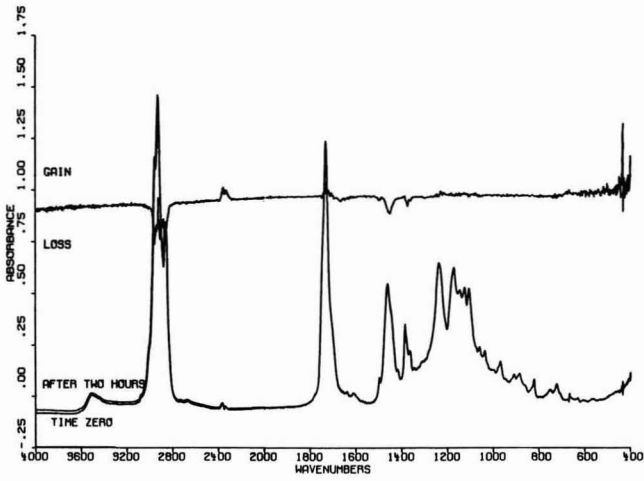


Figure 9—Initial and two hour linseed oil absorbance spectra

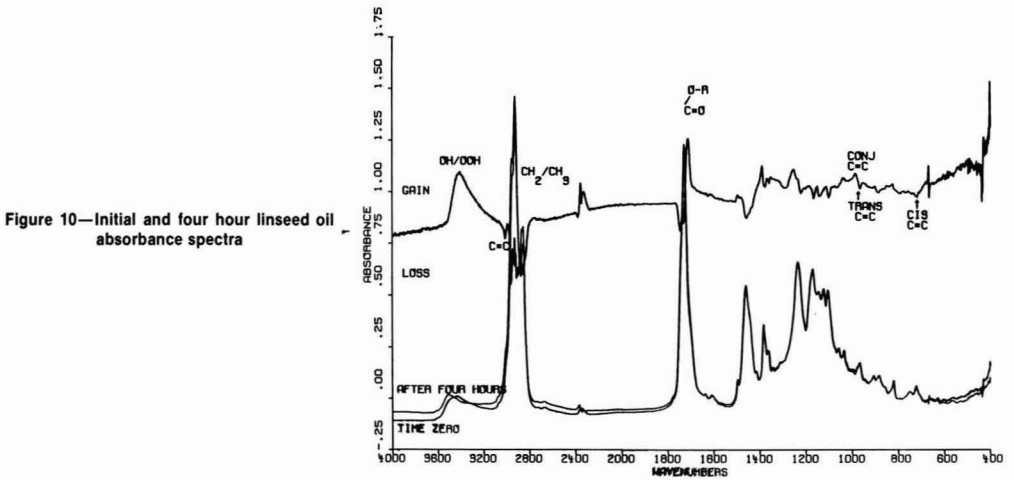


Figure 10—Initial and four hour linseed oil absorbance spectra

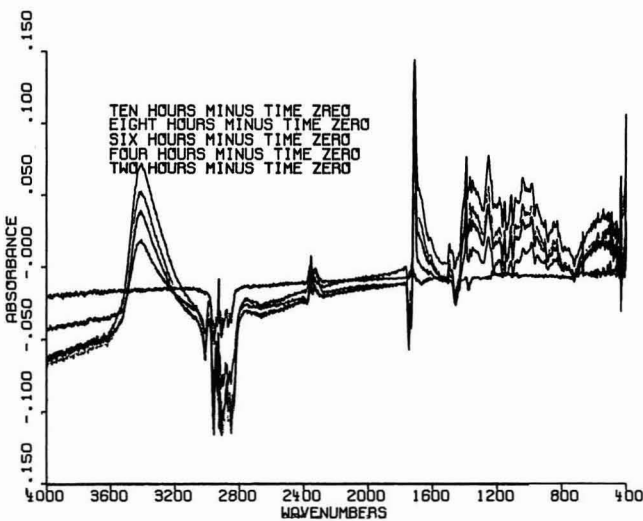
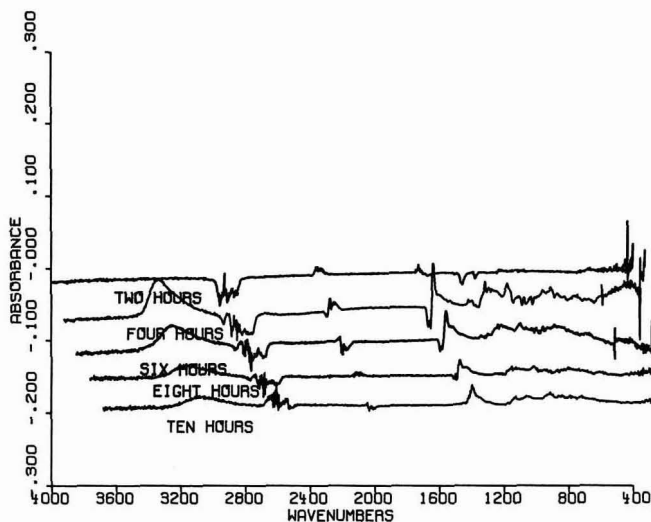


Figure 11—Differential linseed oil spectra to 10 hours

Figure 12—Incremental linseed oil spectra at two hour intervals



If the original spectrum is then subtracted from succeeding time increments, as in Figure 5, some bands appear to increase while others remain relatively constant. The hydroperoxide formation and *cis* isomerization are changing very little; thus, they either are complete or have reached a steady state. The carbonyl band, on the other hand, continues to increase as do bands in the "finger-print" region from 1500–1000  $\text{cm}^{-1}$ . The loss of alkyl groups is also continuing. This can be more graphically demonstrated by subtracting each spectrum from the one taken two hours later to show the incremental changes with time (Figure 6). The axes here have been displaced to minimize band overlap. It is apparent from this presentation that most of the hydroperoxide formation and *cis*-isomerization takes place within the first two hours and that the reaction is essentially completed in the first six hours, since no further change is observed for these bands in the six to eight hour increment. The hydrocarbon loss and the carbonyl and "fingerprint" increase are continuing although the rate is decreasing with time. Interestingly, the conjugate band (990  $\text{cm}^{-1}$ ), which was formed during the first two hours, is thereafter consumed and continues to disappear. Initiation seems to be confined to the first six hours.

Now, if the six-hour spectrum—after initiation is over—is subtracted from the last spectrum, the final stages of cure can be examined, (Figure 7). No more hydroperoxide is generated. In fact, there may be some loss of the band at 3450  $\text{cm}^{-1}$ . The conjugated unsaturation (990  $\text{cm}^{-1}$ ) band continues to disappear presumably by intermolecular crosslinking.<sup>13</sup> The loss of alkyl oxidation products and the increase in their residual fragments, which show up in the "fingerprint" region and in the carbonyl band broadening, appear to be an ongoing process. There also seems to be a further oxidation from ethers to ester and acid groups. An increase in the carboxylic acid type hydroxyl is evident as well. If this period is examined incrementally at 10-hour intervals, the rate of reaction can be observed to slow with time

(Figure 8). After about 46 hours the rate of band change has degenerated beyond the level which can be detected by this technique. That is consistent with what is known about the drying characteristics of this alkyd. Beyond that time the band shifts are so small within a single increment that they are masked by the statistical noise of the infrared measurement, especially in the regions where there are very intense bands in the original spectra.

### Linseed Oil Cure

Linseed oil cure was also examined by the same technique. In order to prevent the film from flowing off the infrared plate, a highly heat bodied oil was used. When the two-hour spectrum is compared with its original a nearly straight baseline is produced (Figure 9). Only a slight disappearance of alkyl bands at 1460 and 1380  $\text{cm}^{-1}$  is observed. This is probably due to the loss of traces of retained solvent or evaporation of very low molecular weight oil components. Inhibition of vegetable oils caused by their impurities has been recognized for many years.<sup>14</sup> After two more hours, reaction has begun (Figure 10). Hydroperoxide is now being formed and the *cis*-isomerization is occurring; however, *trans*-unsaturation also appears to be converted to the conjugate form. The carbonyl shift as noted with the alkyd is also present. When the differential from time zero is examined all bands seem to be growing with time (Figure 11). This is even more evident with the incremental presentation (Figure 12). Once the reaction starts, the rate decreases much more slowly and extends much longer than with the previous alkyd study, otherwise the initiation reactions appear quite similar. If the 10-hour spectrum is arbitrarily subtracted from the sixtieth hour, the later stages are quite different (Figure 13). A hydroxyl/hydroperoxide band continues to be generated; however, it is more likely to be hydroxyl than hydroperoxide since the maximum is closer to 3500  $\text{cm}^{-1}$  and another hydroxyl band near 1100  $\text{cm}^{-1}$  is also growing. Some

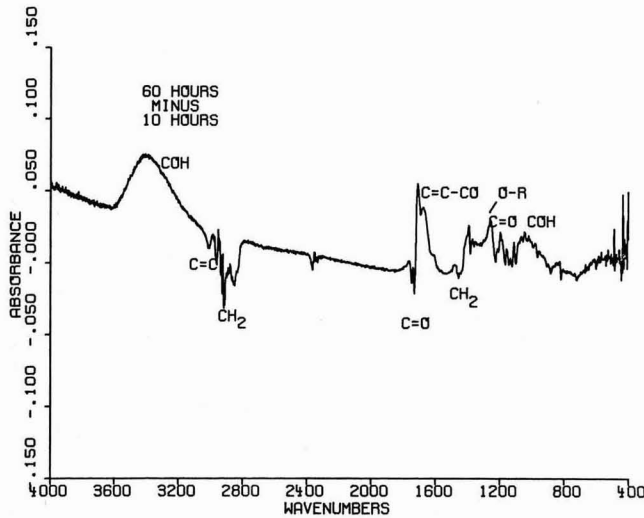


Figure 13—Differential linseed oil spectrum between 10 and 60 hours

unsaturation is disappearing, but it is not possible to determine its structure since its longer wavelength bands are very small and are masked by the measurement noise. As might be expected there is a loss of  $\text{CH}_2$ . Carboxylic acid is being formed as evidenced by the carbonyl band at  $1710\text{ cm}^{-1}$  and the high frequency baseline drift. An interesting new band has appeared at  $1680\text{ cm}^{-1}$ . This is believed to be an alpha-unsaturated carbonyl as predicted by Fritsch and Deatherage.<sup>15</sup> That seems to be a likely site for intermolecular crosslinkage. Incremental examination of this crosslinking period shows that these reactions continue at a decreasing rate until about the fiftieth hour when they become lost in the statistical noise (Figure 14).

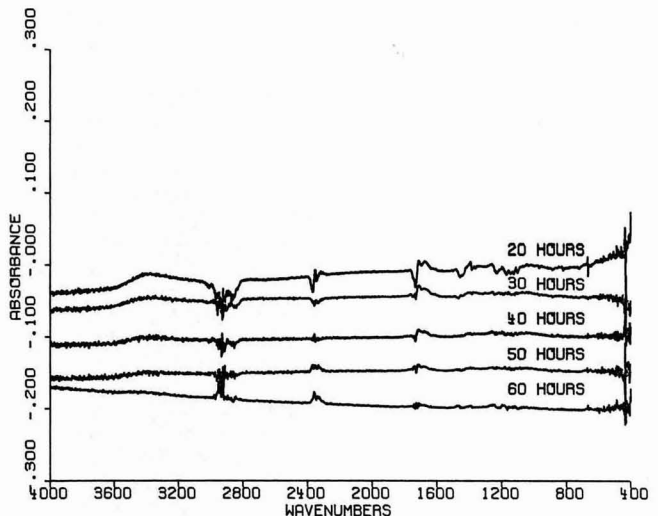
It should be noted (as pointed out in Reference 7) that extreme caution must be exercised when interpreting differential spectra. Highly absorbing peaks in the original spectrum (such as the carbonyl, the CH stretch,

and to some degree all bands) have large measurement errors which are proportional to the peak intensity. These errors are frequently compounded by the subtraction process and they can appear to be absorption bands. Great care must be taken not to consider these false peaks to be a result of some reaction.

**SUMMARY AND CONCLUSIONS**

A new computerized infrared technique, called Time-Lapse Infrared Spectroscopy, has been used to examine the air-drying mechanism of a soya alkyd and a bodied linseed oil. The technique is capable not only of determining what reactions are taking place but in what order they occur. This investigation has shown that the curing of air-dry materials is a very complex process where many reactions are taking place in three identifiable phases: Inhibition, Initiation, and Crosslinking. These reactions may be summarized as follows:

Figure 14—Incremental linseed oil spectra between 10 and 60 hours at 10-hour intervals



### Soya Alkyd

**INHIBITION:** Cure begins almost immediately (well within the first two-hour increment)—possibly as soon as the carrier solvent has evaporated.

**INITIATION:** Hydroperoxide is formed very rapidly, probably on the alpha carbon next to a *cis*-unsaturation. This causes an isomerization of the *cis* double bonds to produce the conjugate and *trans*- forms. This process is essentially complete within six hours.

**CROSSLINKING:** The conjugate unsaturation probably polymerizes with other unsaturated groups in nearby fatty acid chains. Some double bonds (possibly the *trans* configuration) are oxidized to epoxide which in turn may either crosslink or be further oxidized. This further oxidation leads to cleavage of some unsaturated fatty acid chains and the evaporation of their products—aldehydes, ketones, etc. Acidic and hydroxyl end groups are left behind. These may react or exchange with previously formed esters or be left as hydrophilic sites. Curing slows as the film sets up until by the forty-sixth hour the rate is too low to observe under the conditions of this study.

### Bodied Linseed Oil

**INHIBITION:** The onset of curing is delayed for nearly four hours while traces of solvent and impurities disappear.

**INITIATION:** Hydroperoxide and hydroxyl are both formed. They produce a different isomerization where both *cis* and *trans* double bonds are involved to generate the conjugate form. The reaction continues for more than 10 hours and perhaps extends throughout the cure period.

**CROSSLINKING:** The peroxide seems to break down to produce a carbonyl adjacent to the double bond, which then becomes a site of crosslinking. The cure reactions degenerate, below detection by this study, after about 50 hours.

These observations have been compared to previously suggested mechanisms and were found to support many of them. It appears that several reactions are competing simultaneously and that a change in the type of oil acid employed (i.e., the unsaturated fatty acid distribution) or a variation in the dryer formulation could cause different reactions to predominate. That would drastically modify the course and rate of cure and, thus, affect the final properties of film obtained. Time-Lapsed Infrared appears to be a useful tool for investigating new combinations of oils and dryers which will lead to improved air-dry vehicles.

### ACKNOWLEDGMENTS

The author would like to acknowledge cooperation and encouragement of the Marshall R&D Laboratory Staff.

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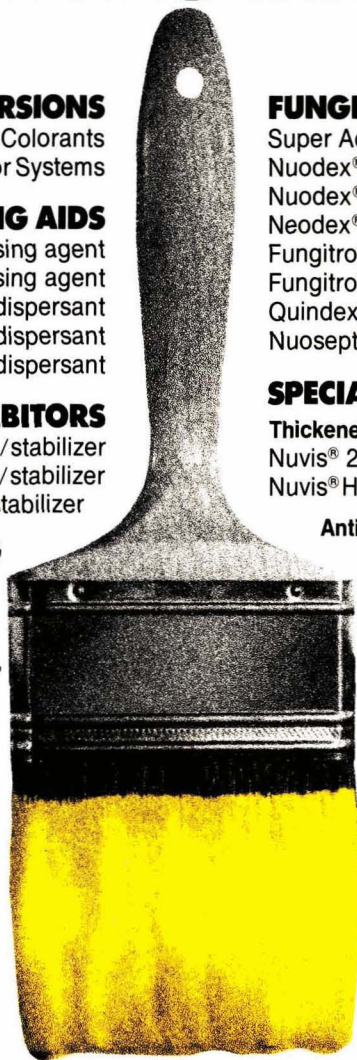
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# Determination of Lead in Paint By Energy Dispersive X-Ray Fluorescence Spectrometry

Gordon S. Kuntz  
Sherwin-Williams Company\*

and

Robert L.R. Towns  
Cleveland State University†

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Available methods for the determination of lead in paint require lengthy sample preparation and may neither provide positive identification of the element nor address the matrix effects without a significant number of additional measurements. An X-ray fluorescence spectrometric method of quantitation using the fundamental parameters "pure element behind a thin sample" technique is described. This approach accounts for both calibration and matrix correction while requiring relatively simple sample preparation. Minimum detection limits ( $3\sigma$ ) are 0.2 and 0.4 ppm at the Lead  $L\alpha$  and  $L\beta$  lines, respectively, with a relative standard deviation of approximately four percent.

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## INTRODUCTION

The toxicity of lead has been known since the Middle Ages. Government regulation also dates from this time period.<sup>1</sup> It is only fitting that this much-studied element is the subject of regulation by various government agencies such as Occupational Safety and Health Administration (OSHA),<sup>2</sup> Environmental Protection Agency (EPA),<sup>3</sup> and Consumer Products Safety Commission (CPSC).<sup>4</sup> Because of regulatory limits, the subject of lead in paint has been widely studied.

Several analytical methods have been implemented, featuring such techniques as atomic absorption spectroscopy (AAS), electrochemical methods, and simple X-ray fluorescence analysis.<sup>5-16</sup> These methods generally require lengthy and costly sample preparation. Those having simplified sample preparation tend to lack sensitivity or specificity and may require substantial additional measurement to address the need for matrix correction.

The currently accepted reference method for the determination of lead in paint (ASTM D3335-74) requires a lengthy sample preparation, making it difficult to use in production situations. Experience with the method has produced some difficulties which are probably the result of the wide variety of components used in the formulation of various types of paints. These difficulties are most likely attributable to three factors: (1) the skill of the operator; (2) the familiarity of the operator with paint systems; and (3) the use of a single extraction procedure for all types of paints. As a result of these problems, research was undertaken to develop an alternative method for the analysis of lead in paint. In developing this method the following guidelines were used:

- (1) Simplest possible sample preparation;
- (2) Reasonable analysis time;
- (3) Straightforward standardization;
- (4) Capability for automation;
- (5) Matrix correction; and
- (6) Provision for determining interferences.

The approach used in this technique takes advantage of the film-forming properties of paints. Previous research has shown that preparation of thin paint films is feasible for use in X-ray fluorescence analysis.<sup>17,18</sup> In the method described herein, thin paint films are pre-

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\*Corporate Headquarters, P.O. Box 6027, Cleveland, OH 44101.  
†Department of Chemistry, Cleveland, OH 44115.

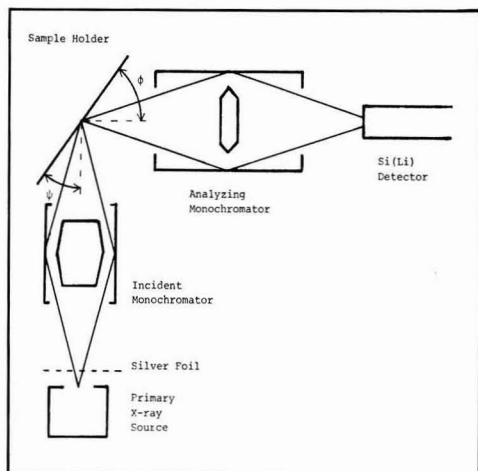


Figure 1—Schematic representation of the x-ray fluorescence analysis system

pared by accepted drawdown techniques; the resulting films are then analyzed using "the pure element behind the thin sample" fundamental parameters technique of X-ray fluorescence spectrometry.<sup>19</sup>

## EXPERIMENTAL

### Apparatus

The X-ray fluorescence system used for analysis consists of a tube-excited X-ray spectrometer equipped with cylindrical monochromators in the incident and characteristic beams.\* The excitation source was a 1000-watt (peak) Dunlee molybdenum-target X-ray diffraction tube supported by a Picker Ultra-stable Model 6238 X-ray generator. Tube current is monitored by reading the base voltage on the current stabilizer with a Simpson Model 461 digital multimeter. The X-ray spectrometer itself consists of an incident beam monochromator, sample holder, and linear translational device for proper positioning of the analyzing beam monochromator. A schematic representation of the system is shown in Figure 1. The general principles of the spectrometer system are discussed in a previous paper.<sup>20</sup> The monochromators themselves are hollow, cylindrical aluminum housings inlaid with flat, compression-annealed UCAR® Class A pyrolytic graphite crystals. The incident and analyzing beams were configured at right angles to one another with an incident angle of  $37.5^\circ$  and a take-off angle of  $52.5^\circ$ , both relative to the sample plane. The effective band pass of the analyzing monochromator is approximately 1.8 keV at 8.0 keV. The data collection system consists of a Nuclear Data 4410 data system with an Ortec Si(Li) detector and Ortec 459A Amplifier. The detector has an active diameter of 10 mm and an ex-

perimentally measured resolution of 200 eV FWHM at Mn K $\alpha$ .

Comparative analyses by AAS were conducted utilizing two spectrometers: a Varian Model 175 AA single beam instrument with a Bauch & Lomb hollow cathode lamp for the analyses of selected commercial products; and a Perkin-Elmer Model 703 AAS dual beam with an electrodeless discharge lamp (EDL) for the repeatability studies.

### Preparation of Standards

Standard solutions for use in the AAS technique were prepared in accordance with ASTM D3335-74. To prevent contamination, all glassware, test tubes, and crucibles were acid-washed for 24 hours in a solution of equal parts by volume of concentrated nitric and hydrochloric acids (Fisher Brand, ACS certified) and water. Standard lead solutions for calibration were prepared by pipetting appropriate amounts of a 1000-ppm lead stock solution prepared from lead nitrate (Mallinckrodt Analytical Reagent) using a calibrated Clay-Adams Selectapette pipette system. The water used in solution preparations was distilled-deionized with at least 15 megohms resistance.

Standard samples of lead in dextran were prepared for XRF analysis by weighing one gram samples of 250,000 weight average molecular weight dextran (Pharmacia, Sweden) into 15-mL polypropylene test tubes. Appropriate amounts of the 1000-ppm lead stock solution were pipetted into the test tubes using Eppendorf pipettes to achieve the desired levels of 25 to 800 ppm. The dextran-lead mixture was then dissolved in approximately 10 mL of distilled-deionized water and mixed with a vortex mixer to ensure sample homogeneity. Solution preparation was completed within 60 minutes. When the dextran-lead mixture was completely dissolved, the sample was immediately frozen in liquid nitrogen and then lyophilized for 48 hours. Half-inch pellets were prepared for X-ray analysis by grinding the lyophilized dextran-lead powder in a Wig-L-Bug® mortar, followed by pressing of approximately 35 mg of the material in a KBr die at 13,000 PSI for two minutes. Teflon® discs were used to prevent direct contact with the die faces.

Samples of NBS Orchard Leaves (SRM 1571) were prepared by drying in accordance with the NBS instructions and pressing pellets by the procedure used for the dextran-lead samples.

Standard lead-containing paint samples required special procedures to provide for homogeneous incorporation of lead into the paint samples. Lead naphthenate drier, obtained from Sherwin-Williams Chemical Coatings Div., was used to prepare standard paints. The lead content of the drier was determined by EDTA back titration (ASTM D2374). For the purpose of sample preparation, a small, high speed disperser was fabricated using a drill press. The paint standards were prepared by weighing 250 to 350 g of premixed paint into 1200-mL tallform Teflon-linked stainless steel beakers. High speed dispersion was achieved using a two-inch impeller blade at 2500 rpm. The lead drier was added by syringe

\* The spectrometer and monochromators were manufactured by Molecular Data Corp., 2869 Scarborough Rd., Cleveland, OH 44118.

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to the paint sample under agitation. The sample was then subject to high shear for five minutes followed by approximately five minutes of slow speed agitation (300–500 rpm). Samples were stored in acid-washed brown glass bottles with polyethylene cap liners.

**Preparation of Samples**

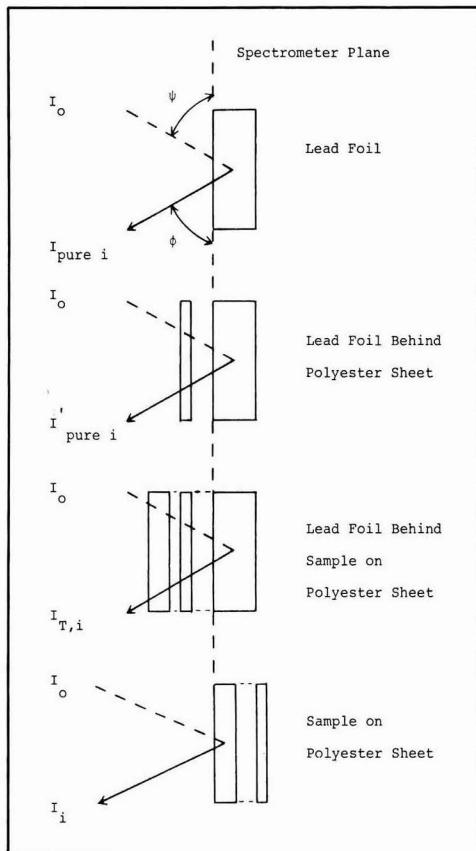
Sample preparation for AAS analysis was performed according to the ASTM D3337-74 procedure. The non-volatile content (of all paints) was determined by ASTM D2369-73. The toluene addition was omitted for determination on water-based paints. A 250-watt infrared lamp was used in conjunction with the nitric acid extraction procedure to reduce the incidence of bumping. The extracts were then filtered with Whatman No. 22 filter paper.

Samples for X-ray analysis were prepared using standard paint drawdown techniques. The film applicator used was #38 wirewound drawdown bar (RDS Co., Webster, NY). Acetone, toluene, xylenes (ASTM D95-46), and 10% sodium hydroxide were used as wash solvents. Polyester sheets (15 × 25 cm) of 2.5-mil Scotchpar® type GAB were used as the substrates. Paint samples were thoroughly stirred and inspected for uniformity. Paint films were made by placing approximately two grams of paint on the polyester sheet and drawing a paint film with the wirewound drawdown bar. A large glass plate was used for the drawdown surface. Sample films containing water as a solvent were given a flash off time of five to 10 minutes before proceeding. Completed drawdowns were placed on a aluminum sheet and dried for 20 minutes in a 110°C forced draft oven. After films were dry, two-inch square samples were cut from the drawdown panel using a machined 2 × 2-inch acrylic template and a razor blade. An adjacent unused section of the substrate sheet was also cut in the same manner for use as a blank. The sample and blank were weighed to 0.1 mg and the areal density determined.

**Analytical Procedure**

The atomic absorption data were collected using the procedure specified by the ASTM method. Each data point used was the average of three absorption readings. The standard curve was determined by least-squares fit of the absorbance versus concentration data in the 1–10 ppm range.

The X-ray spectrometer used in this research provides the essentially monochromatic source that is a necessary condition for application of the fundamental parameters quantitation technique. This method requires that three intensity measurements be made for the calculation: (1) the pure lead, to obtain a relative intensity for a sample whose weight fraction of lead equals 1.0; (2) pure lead behind the sample, to obtain a measure of the attenuation of the incoming and exiting beams due to absorption of these X-rays by the sample (matrix effect); and (3) the sample only, to obtain a relative intensity due to the lead in the sample. In addition, a fourth intensity measurement was made of the pure element behind the polyester sheet to correct for absorption and positioning of the substrate.



**Figure 2—Illustration of intensity measurements**

The intensity measurements are illustrated in Figure 2 and the equations used for the calculations are as follows:<sup>19</sup>

$$C_i = \frac{I_i Q_i (\sin \psi) \ln(I_{T,i} / I'_{pure i})}{\rho_s T [(I_{T,i} / I'_{pure i}) - 1]} \quad (1)$$

- $C_i$  = concentration of analyte (g/g of sample)
- $I_i$  = fluorescent intensity of analyte (counts/s.)
- $Q_i$  = response factor for pure element foil (g/cm<sup>2</sup>/counts/sec.)
- $\psi$  = angle of incident radiation
- $\phi$  = angle of characteristic radiation
- $I_{T,i}$  = intensity for pure element behind thin sample (counts/s.)
- $I'_{pure i}$  = intensity of pure element (counts/s.)
- $I'_{pure i}$  = intensity of pure element behind the polyester sheet (counts/s.)
- $\rho_s T$  = areal density of the sample (g/cm<sup>2</sup>)

The response value “ $Q_i$ ” is determined as follows:<sup>16</sup>

$$Q_i = \frac{1}{I_{pure i} \left( \mu(E_o) + \mu(E_i) \frac{\sin \psi}{\sin \phi} \right)} \quad (2)$$

- $\mu(E_o)$  = the mass absorption coefficient of the element for the excitation energy
- $\mu(E_i)$  = the mass absorption coefficient of the element for the fluorescence energy.

The response values for the Pb L $\alpha$  and L $\beta$  lines were

**Table 1—Determination of Lead in Dextran Standards by X-ray Fluorescence**

Added Pb (ppm)	Measured Pb (ppm)	
	L $\alpha$	L $\beta$
24.5	24.4	22.2
94.4	97.5	95.5
400.0	389.0	381.0
603.0	604.0	582.0
793.0	836.0	818.0

calculated using equation (2). To increase the dynamic range of the instrument, the incident X-ray beam was attenuated with a 0.0130-cm silver foil, and the current was adjusted to maintain optimum performance of the analyzer. The observed pure element intensity was corrected for attenuation caused by absorption of the silver foil and lower tube current setting. Values for the mass absorption coefficients and foil densities were those of McMasters.<sup>21</sup> Concentrations of lead in paint films were calculated using equation (1) with the intensity from the pure element corrected for the absorption of the substrate.

The instrument operating conditions were a tube voltage of 38 kV and a current range of 10 to 16 mA, the current being adjusted to limit the analyzer input to approximately 2000 counts per second or less. The spectra collected were smoothed using a five point smoothing routine, and peak area was determined using a Gaussian fit.<sup>22</sup> Intensity data for the pure element were collected for 50 seconds (live time) using a silver foil for incident beam attenuation. Sample spectra collection times were 50 seconds for lead levels greater than 100 ppm and 200 seconds for all others. Peak integrals were determined for a range of 0.9 and 0.6 keV for the L $\alpha$  and L $\beta$  lines, respectively.

## RESULTS AND DISCUSSION

Initial evaluation of the procedure was made by analyzing lead in dextran-prepared standards. Table 1 shows the results of this evaluation. Although the corre-

**Table 2—Analysis of Commercially-Available Paints By X-ray Fluorescence and Atomic Absorption Techniques**

Paint Type	Measured Lead (ppm) in Dried Film		
	XRF L $\alpha$	XRF L $\beta$	AAS
Latex flat, white	<sup>a</sup>	55.8	67.9
Alkyd flat, white	21.0	16.1	17.0
Floor enamel, red	86.8	75.7	83.4
Latex flat, yellow	<sup>a</sup>	41.1	48.5
Alkyd enamel, yellow	83.7	70.1	80.1
Brushing industrial enamel, red	7030	6760	8880
Acrylic latex, white	<sup>a</sup>	34.4	33.1
Alkyd gloss, white	29.7	24.9	34.5
Automotive enamel, white	164	151	184
PVC metal coating, green	13600	13200	13600
	(1.36%)	(1.32%)	(1.36%)

(a) Arsenic interference confirmed (As K $\beta$ ).

**Table 3—Statistical Comparison of XRF and AAS Analyses Of Typical Paints with Added Lead**

	XRF <sup>a</sup>		AAS <sup>b</sup>
	L $\alpha$	L $\beta$	283.3 nm
<b>Alkyd Baking Enamel</b>			
$\bar{X}$	857	839	839
s	28.2	31.2	95.9
% Std. Dev.	3.29	3.71	10.24
% Recovery	98.6	97.1	96.8
<b>Latex White Flat</b>			
$\bar{X}$	534	524	542
s	12.4	12.6	14.5
% Std. Dev.	2.09	2.40	2.68
% Recovery	100.0	99.1	99.4
<b>Alkyd Gloss White</b>			
$\bar{X}$	511	498	481
s	12.4	15.1	52.1
% Std. Dev.	2.43	3.04	10.83
% Recovery	99.4	97.4	93.6

(a) XRF data results of 5 determinations on separate days.  
(b) AAS results of 7 determinations on 3 days.

lation with lead content is excellent, some samples have a larger error than is desirable. A significant fraction of the error is attributable more to the pellet preparation process than to any other part of the analysis. Detection limits were calculated on the 24 ppm lead/dextran sample by collecting spectra for 1000 seconds.

The 3 $\sigma$  detection limits were 0.2 and 0.4 ppm for the L $\alpha$  and L $\beta$  lines, respectively.

For a standard reference material, the NBS SRM 1571 Orchard Leaves was selected. The results from four samples each ranging in size from 30 to 45 mg, gave an average value of  $44.9 \pm 7.4$  ppm (2 $\sigma$ ) compared to the NBS certified values of  $45.0 \pm 3.0$  ppm. The evaluation was made by using the L $\beta$  line to avoid a significant arsenic interference at the Pb L $\alpha$  line. The results of dextran and orchard leaves studies, together with literature reports of thin coating sample preparation techniques,<sup>17,18</sup> indicated that a thin film approach to lead in paint should be feasible.

A comparison of the results of the AAS and XRF methods for the analysis of 10 commercially available paint samples is presented in Table 2. These paint samples were chosen for analysis on the basis of composition in an effort to provide maximum differing matrix effects. Relatively good agreement is demonstrated between the X-ray lines and the atomic absorption method.

Although the sample preparation was designed to yield a 25 to 50  $\mu\text{m}$  (1–2 mils) film, the film thickness on these samples ranged from 25 to 115  $\mu\text{m}$  (1–4.5 mils) as measured using a micrometer. Film thickness control, in terms of the range used, did not present any problems for the XRF analysis. It is most important, however, that the film be of uniform thickness.

A more pronounced effect was observed with the change in matrix of each paint. For the 10 samples, matrix correction ranged from 9 to 40% for the L $\alpha$  line

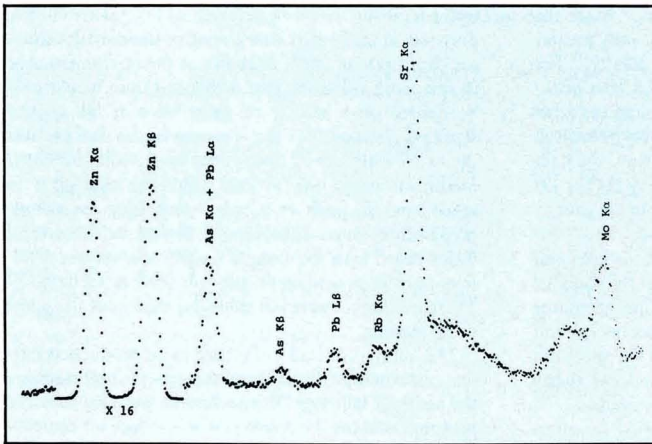


Figure 3—XRF composite spectrum of yellow latex house paint. (collection time: 200 sec; counts full scale: 2047)

and 5 to 34% for the  $L\beta$ . Using the atomic absorption as the reference method, the X-ray method performed satisfactorily over a broad range of lead concentrations with no systemically observable matrix correction problems.

To assess the method for reliability, three evaluations were used. The same sample was run on 10 different days. The standard deviations for these measurements at both the  $L\alpha$  and  $L\beta$  lines were 10 ppm (2.5% relative standard deviation) at the 400 ppm concentration level. To evaluate sample preparation uniformity, the four outer quadrants of the  $2 \times 2$ -inch sample and the center were measured on the same day. The standard deviation for this set of measurements was 14 ppm at the 400 ppm level (3.6%).

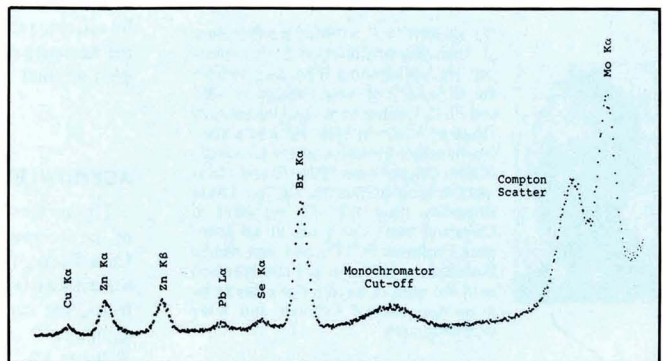
For the purpose of evaluating the method under realistic field conditions, three samples of paint were prepared with known lead content. A water-based latex flat, alkyd gloss, and a baking enamel were selected to provide a range of matrix and sample preparation variables. Table 3 gives the results of the study conducted on these three paints. The atomic absorption data were collected on three different days because of instrument availability. The recovery is calculated by correcting for the premixed paint as a blank. An examination of the data illustrates that the standard deviation for the analysis

of the two alkyd materials is significantly less for the X-ray method compared to the ASTM method. The recovery is also better for the X-ray method. The slightly higher values for the  $L\alpha$  measurements are attributed to the minor interferences from arsenic  $K\alpha$  in some of the samples. Generally, the lead content as analyzed by this method is in good agreement with the ASTM reference method.

The drying techniques used in the sample preparation were modeled after ASTM D-2832. The 20 minute drying time in this method produced stable films suitable for analysis. The change in drying time to 60 minutes<sup>23</sup> proposed by the Environmental Protection Agency should not greatly affect the results of the measurement of nonvolatile content for this method.<sup>24</sup>

A comparison of results between use of the Pb L lines shows that values for the  $L\alpha$  line appear to be somewhat higher than the  $L\beta$ . These differences in results can be attributed to a number of factors. The most prominent is the interference from arsenic found in the paint samples examined in this work. Where the arsenic interference is of sufficient quantity to be identified by the appearance of the As  $K\beta$ , use of the Pb  $L\alpha$  is inappropriate, as indicated in Table 2, unless correction can be made for interference. The error of the Pb  $L\alpha$  line may also be greater because the absorption correction is logarithmic

Figure 4—XRF spectrum of whole blood. (collection time: 100 sec; counts full scale: 4096)



and increases with increasing matrix effect.<sup>19</sup> Since the matrix correction for the  $L\alpha$  line is significantly greater than that for the  $L\beta$ , the error will also be greater.<sup>19</sup> The  $L\beta$  line, however, is in a spectral area of higher background generated by the Compton Effect, and the error associated with the background would be influenced accordingly. One final possible consideration, the data available for calculation of the cross-section for the  $L\beta$  energy for lead may be more uncertain due to the paucity of experimental data compared to the  $L\alpha$ .<sup>22</sup>

Figure 3 shows a composite spectrum of the latex flat (yellow) paint in Table 2. The composite is the result of data collections with the analyzing monochromator aligned for the  $L\alpha$  and  $L\beta$  lines, respectively, and collected for 200 seconds at each position. The spectrum illustrates the sensitivity of the method and the ability to make an assessment of possible interferences.

Other X-ray methods were briefly studied for comparison. A dedicated X-ray analyzer using a <sup>109</sup>Cd source may be used on thin paint films, but count times are long and the analysis must be performed by standard additions. Implementation of other commercially available tube-excited X-ray systems is possible. One particular energy dispersive instrument, produced a count rate of four counts per second utilizing a 400 ppm paint sample compared to 135 counts per second using the instrumentation described herein. Geometry considerations of some commercial systems may preclude use of this fundamental parameters technique.

Data collection at the 600 ppm maximum as specified

GORDON S. KUNTZ is a chemist and administrator of Regulation and Toxic Data in the Regulatory Information Service Department for Sherwin Williams Company. He has earned a B.S. Degree from Pennsylvania State University and a M.S. in Chemistry from Cleveland State University. He is currently pursuing his Ph.D. Degree in Analytical Chemistry from Cleveland State University. His work experience includes nine years as an Analytical Chemist in various Sherwin-Williams coatings groups and five years experience in industrial hygiene and product safety.



DR. ROBERT L.R. TOWNS is a Professor of Chemistry at Cleveland State University. He has earned a B.Sc. Degree from the University of New Orleans in 1965 and Ph.D. Degree from The University of Texas at Austin in 1969. He was a Visiting Assistant Professor at the University of New Orleans from 1968-70 and Assistant Professor of Chemistry at Texas A&M University from 1970-73. He went to Cleveland State University as an Associate Professor in 1973 and was named Professor in 1977. His primary research is in the area of trace metal analysis by X-ray fluorescence methods and X-ray crystallography.



by Federal law<sup>1</sup> could be determined for quality control purposes in 20 seconds with a relative standard deviation for count rate of 2.2%. Stability of the instrument used in this work indicated that standardization would only be needed on a weekly or daily basis as the relative standard deviation for the response factor was 1% over the two-month period that testing was conducted. Other modifications of the method could be developed to speed analysis such as a smear technique for sample preparation, microwave sample drying in the case of water-based systems, sample weight, and matrix determinations from incoherent and coherent scatter data.<sup>25,26</sup> These techniques were not studied in detail but all appear to be feasible.

The analysis of lead in finished paint products is only one of several analytical requirements of importance in the coatings industry. This technique was also found to be applicable for the analysis of workplace air samples, aerosol spray paints, paint chips, waste paint, and whole blood. Figure 4, for example, shows a spectrum of lead at the 25  $\mu\text{g}/\text{dL}$  level in a pressed pellet of lyophilized whole blood.

The use of XRF methodology for the determination of lead in paints provides a feasible alternative to AAS methods. The XRF technique yields high sensitivity with minimal sample preparation and matrix correction and is readily adaptable to production analysis.

## SUMMARY

A new X-ray fluorescence technique has been developed for the analysis of lead in paint. This method utilizes the film forming properties of paint samples to provide for quick and simple sample preparation from accepted paint film casting techniques. The fundamental parameters technique "pure element behind a thin sample" incorporated the use of the thin paint films for matrix correction and provides standardization from pure metal foils. The XRF method was evaluated for a number of varied paint compositions and samples. A comparison of the method to the ASTM AAS method gave excellent agreement. The minimum detection limits ( $3\sigma$ ) of 0.2 and 0.4 ppm for the  $L\alpha$  and  $L\beta$  lines, respectively, provide good sensitivity. The combination of good sensitivity and the matrix correction feature of the technique make the method ideal for quality control by offering relatively rapid analysis time and eliminating the necessity of establishing a standardization curve for each product.

## ACKNOWLEDGMENTS

The authors gratefully acknowledge the contributions of the following: Dr. Thomas C. Furnas, of Molecular Data Corp., for providing the X-ray instrumentation; Steven Kosztya, of the National Spectrographic Laboratories, for consultation and use of atomic absorption equipment; and Arthur Andrews, of the Sherwin-Williams Co., for providing several of the paint samples.

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# Society Meetings

## Baltimore Virginia Section

Dec. 9

Alex Elbrechter, of Degussa Corp., discussed "CARBON BLACKS IN TODAY'S COATINGS INDUSTRY."

The presentation featured slides and a discussion of the various processes used to manufacture carbon blacks. Mr. Elbrechter pointed out differences in the more traditional American manufacturing procedures and the more modern and efficient processes used by Degussa Corp., Germany.

Also discussed were the property differences between channel blacks, lamp blacks, and other carbon blacks. Mr. Elbrechter related the properties to particle size, method of manufacture, and surface treatment. He concluded the presentation by citing specific uses which optimized the advantages of the various types of black.

CARL B. MINCHEW, *Secretary*

## C-D-I-C

Dec. 14

"ACID CATALYSTS IN HIGH SOLIDS COATINGS" was presented by Dr. Leonard J. Calbo, of King Industries, Inc.

Dr. Calbo explained that catalysts are tools and not cure-alls on low energy curing systems. He described dinonylnaphthalene disulfonic acid (DNNSDA) as a strong acid of exceptional purity, for example, having low levels of sulfuric acid. This small residual amount of sulfuric acid is especially important in electrostatic spraying since the DNNSDA

exhibits only about half of the conductivity of p-TSA, explained Dr. Calbo.

A series of slides demonstrated that DNNSDA provides better detergent resistance in alkyd-melamine systems than does p-TSA. The major reason for this, according to Dr. Calbo, is that the DNNSDA is hydrophobic. He also noted that in high solids acrylic systems where viscosity stability is a common problem, the DNNSDA offers a considerable advantage over p-TSA. Slides illustrated improved salt spray resistance of a DNNSDA catalyzed acrylic system on zinc phosphated steel compound, in comparison to a p-TSA catalyzed system.

Slides depicted a study comparing five sulfonic acids in a polyester/melamine system and the properties each of the acids yields. Dr. Calbo presented a brief review of a proposed reaction scheme for a polyester/melamine system, in the presence of an acid catalyst; the polyester having primary hydroxyl functionality and the melamine being hexamethoxymethyl melamine (HMM). He noted that the crosslinking reaction was observed to occur as low as 90°C and that the selfcondensation reaction needed temperatures of 102°C or higher.

Dr. Calbo presented two possible reaction mechanisms for HMM crosslinking. The rate of  $S_N1$  mechanism is determined by the concentration of the HMM resin and the strength of the acid catalyst only. The rate of the  $S_N2$  mechanism is determined by the melamine, the acid catalyst, and the binder resin (acrylic or polyester)

all coming together. It has been observed, said Dr. Calbo, that in polyester melamine reactions, a primary hydroxyl will cure faster than a secondary hydroxyl, which in turn is faster than a carboxyl. These observations cannot be explained by the  $S_N1$  mechanism, but they can be explained by the  $S_N2$  mechanism, he said.

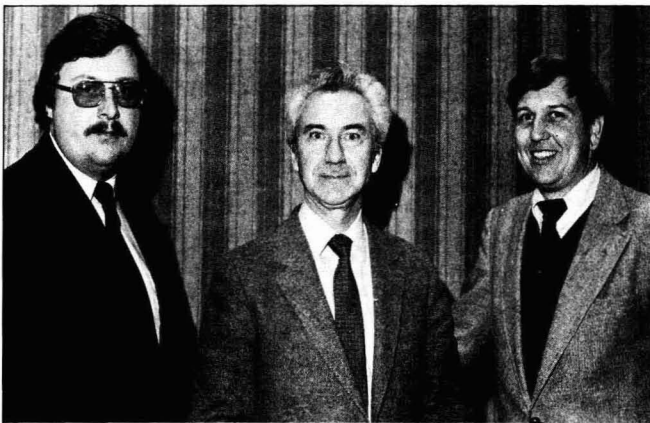
The possible reasons why the DNNSDA will provide better film properties than will p-TSA were discussed. DNNSDA is hydrophobic; it has low solubility in water compared to p-TSA. It will not lead to degradation and will not leave the film as sensitive to water. The structure of the catalyst perhaps has some effect, although it has not been researched completely at this time, said Dr. Calbo. DNNSDA exerts some control over the competing reactions of crosslinking vs. self-condensation through steric effects—possibly differences in ionic strength or phase mixing, which means that the DNNSDA will preferentially associate with nonpolar phase (the polyester) rather than in the melamine phase lessening the chances for self-condensation. Also, DNNSDA does not accumulate at the film/substrate interface as does p-TSA, which will lessen the potential for degradation, explained Dr. Calbo.

Some of the problems associated with high solids systems were discussed. When you increase solids and lower solvent level, very likely the formulation will run into stability problems which generally indicates the use of blocked catalysts, specifically amine blocked catalysts, he said. Some of the factors which must be considered are the volatility of the blocking amine. In water-borne systems will the amine form an azeotrope with water, and how strong of a base is the amine? Will the amine be able to get out of the film before the gel stage in the bake cycle, particularly if carboxyl groups are present? There are also a number of secondary reactions which can become very important. Will the amine decompose to a less volatile compound? Will it form an amide or an ester and will it react with formaldehyde? There is also some evidence that an amine could react with melamine, according to Dr. Calbo.

Dr. Calbo then offered possible solutions to the potential problems in catalyzed high solids enamels.

Dr. Calbo noted five factors affecting the choice of catalyst: pigments; backbone resin and cross linking agent; substrate; film properties; and post curing.

ROBERT A. BURTZLAFF, *Secretary*



Baltimore Society—Virginia Section Officers for 1981–82. (Left to Right): Vice-Chairman, C. Herbert Pund III; Chairman—Colin Penny; and Secretary-Treasurer—Carl Minchew



## Golden Gate

Jan. 18

A moment of silence was observed in memory of Chuck Cary, of A.J. Lynch & Co., and Robert Roder, retired, who died recently.

Technical Committee Chairman Tim Specht reported that the Society will work with the Paint Research Institute in reviewing the polymer developed by the Mildew Consortium. This review may involve other West Coast Societies. In addition, the Golden Gate Society is attempting to correlate the results of the salt fog cabinet and local exterior exposure tests.

Terry Bowerman, of Degussa Corp., discussed the topic of "FUMED SILICA."

Fumed silicas were defined by Mr. Bowerman as being formed by reacting silicon tetrachloride with oxygen and hydrogen at approximately 1000° C. The particle size of the material formed in this method is between 7-16 nanometers in diameter. These particles have available surface area ranging from 130-300 m<sup>2</sup>/g. Fumed silicas are hydrophilic because of the available hydroxyls, explained Mr. Bowerman.

Hydrophobic fumed silicas are formed, according to Mr. Bowerman, by reacting the surface hydroxyls with silane materials terminated with methyl groups. Materials used for modifying the fumed silicas included hexamethyldisilane and dimethyldichorosilane. Replacement of the surface hydroxyls is done in a particle way, explained Mr. Bowerman. As the degree of substitution of the surface hydroxyls increases, the hydrophobic nature of the product increases.

Mr. Bowerman discussed the potential applications for hydrophobic fumed silica. An example of the enveloping process was presented which mixed zinc with hydrophobic silica. A slide demonstrated the appearance of the zinc particles before and after the enveloping process, which minimized the gassing of zinc rich coatings. Mr. Bowerman discussed the application of a suspending agent which also employed a zinc primer and 2% hydrophobic silica. A slide illustrated the superior suspension gained from this application. Other coatings application discussed included epoxies, water-reducible alkyd system, and high solids.

*Q. Could excellent dispersion, required in high solids systems, be achieved with a high speed disperser?*

A. A high speed disperser would be adequate, provided a good sheer viscosity was achieved in the dispersing blend.

*Q. How do you achieve the envelopment of the particles?*

A. The materials could be put into grind phase on an individual basis, and actually because of the fine particle size of the hydrophobic silica, they would serve to envelop the larger pigment particles.

K.E. TRAUTWEIN, *Secretary*

## Houston

Dec. 9

John Bax, of Pacific Scott Bader, presented "USES OF VINYLIDINE CHLORIDE COPOLYMERS FOR THE PAINT INDUSTRY."

According to Mr. Bax, vinylidene chloride copolymers have the following advantages: fire retardancy; good oil resistance; and low molecular volume

transmission rate. However, they have the disadvantages of low pH; poor color stability in higher pH systems and over some metals; and a tendency toward crystallinity. The progress made to overcome these disadvantages was discussed.

Major application for these polymers, according to Mr. Bax, is in high build, fire retardant, and barrier coatings and combinations of these.

GEORGE SCHWARTZ, *Secretary*

## Kansas City

Jan. 14

"ORGANIC PIGMENTS: PAST, PRESENT, AND FUTURE" was presented by Hugh M. Smith, of Sun Chemical Corp. Mr. Smith reviewed the organic pig-

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ments business in the United States with emphasis on its history, changing technology, and raw material supply. Current trends within the industry were highlighted and predictions were made for future innovations.

MEL BOYER, *Secretary*

## Los Angeles

Jan. 13

Honored guests included Federation President Howard Jerome, Past-President William Ellis, and Field Services Director Thomas Kocis.

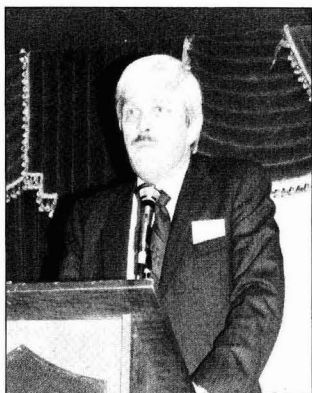
President Jerome discussed Federation and Society activities, the restructuring of PRI, and the development of a correspondence course.

Jim Elliott, Environmental Committee Co-Chairman, introduced Charles J. Bruck, Compliance Officer for the Los Angeles Area of the U.S. Consumer Product Safety Commission.

Mr. Bruck thanked the members of the paint industry for cooperation in interpretation, understanding, and compliance with the consumer protection laws and regulations. He referred specifically to the laws pertaining to lead-in-paint, packaging, and labeling. Mr. Bruck enlisted the continued cooperation and goodwill of the paint industry.

Organizational Committee Chairman Bert Osen reported that 251 of the 585 L.A. Society membership surveys recently sent have been returned and are currently being tabulated.

Bob McNeill, Society Representative, discussed on the recent restructuring of the Paint Research Institute and recommended reinstatement of Society support. As approved by the Board of Directors, therefore, Mr. McNeill presented a check for \$500 to President



**Bob Athey, Los Angeles Society Educational Committee Chairman, awards certificates to members completing polymer course**



**Attending the Los Angeles Society meeting are (left to right): Field Services Director—Thomas Kocis; Federation President—Howard Jerome; and Society President—Jan Van Zelm**

Howard Jerome for PRI. President Jerome graciously accepted the support of the Society.

Bob Athey, Educational Committee Chairman, awarded Certificates of Completion to those students completing the California State University at Fullerton course in High Polymer Technology. Mr. Athey stated that the course was designed to combat technological obsolescence in plastics and coatings resins, and he described the course as long and comprehensive. Nine certificates were awarded for the course completed in December, as follows: Douglas Cavanaugh, John Kapadia, Marie Lui, Hermangildo Mislano, Lan Ngo, Alpheus San Doke, Frederick Saremi, Patricia Shaw, and Mike Stuart.

Mr. Athey also awarded a \$100 Savings Bond for Best Student to Douglas Cavanaugh.

Terry Bowerman, of Degussa Corp., presented "HYDROPHOBIC FUMED SILICAS AND THEIR USE IN THE COATINGS INDUSTRY."

Fumed silicas are manufactured by the flame hydrolysis of silicon tetrachloride, explained Mr. Bowerman. After cooling, deacidification removes the hydrochloric acid by-product. Fumed silicas are distinguished by large surface areas and extremely fine particle sizes.

Hydrophobic fumed silicas are formed by treating the silica surface with organic silanes to replace many of the hydroxyl groups with methyl groups (CH<sub>3</sub> termination). It is these methyl groups that render the surface of the silica particle hydrophobic, hence water repellent, according to Mr. Bowerman. He described both batch and continuous processes to produce different types of products by varying the surface area of the base fumed silica. He demonstrated that increasing the surface area diminishes the percentage of reacted hydroxyl groups; but leaves a hydrophobic behaving product with more free hydroxyl groups for structuring ability (thickening efficiency). The higher surface areas are more difficult to disperse, or require more shear

than the lower surface area types, explained Mr. Bowerman. They give higher structure, but lower levels of hydrophobicity. Mr. Bowerman summarized: for maximum hydrophobicity use low surface area; for good structuring and acceptable hydrophobicity use higher surface area. Hydrophobic silicas also retain low moisture pickup at high humidity, said Mr. Bowerman.

*Q. How do you determine the proper concentration of hydrophobic fumed silica in a zinc rich primer to get the best properties?*

A. Add various concentrations to your coating to determine optimum anti-settling behavior and cost. Conventional zinc primers require about 2% concentration based on the zinc for perfect suspension. This is expensive, so a compromise downward may be necessary.

*Q. What mechanism do you propose for the activity of hydrophobic silica as an anti-settling agent in water-based systems?*

A. Conventional dispersion equipment. In the functional sense, hydrogen-bonding provides structure. Hydrophilic type will also perform in this manner, but will not add corrosion protection.

*Q. Would hydrophobic silica be effective to prevent seed formation with reactive zinc oxide?*

A. I don't know. However, if the seeding is caused by a reaction, coating of the particles should help.

*Q. Carbon black has a tendency to float when used for tinting. Can silica be used to reduce this tendency?*

A. I suppose; but a larger sized carbon black, like a lampblack or a furnace black would be better for the job.

*Q. Should hydrophobic fumed silica be dispersed into a water dispersible alkyd before or after neutralization?*

A. I suggest the silica should be dispersed as early as possible; thus, before neutralization.

*Q. What effect does the incorporation*

*of hydrophobic silica have on recoatability and, specifically, intercoat adhesion?*

A. I believe this would be related to the absence of effect of the hydrophobic silica on gloss of a film. Since the particles would not come to the surface, there should be no problem.

*Q. What does fuming refer to in fumed silica?*

A. It is the burning process of silicon tetrachloride, air, and hydrogen ignited together. Former reference was pyrogenic silica, but that inferred explosives, so fumed is now used.

*Q. In a water thinnable alkyd high gloss, does the hydrophobic silica contribute to anti-sag properties as well as anti-settling.*

A. Generally, yes. However, a lot depends on the formulation. If well dispersed before water is added, there should be a beneficial anti-sagging effect.

*Q. What type of bonding do you have by the hydrophobic fumed silica particles to the zinc particles? Does this impart milkiness to the film at certain concentrations?*

A. Physical absorption of the silica particles on the zinc. The fumed silica does not generally impart milkiness to the film; although, the characteristic may be related to the refractive index of the bonding medium.

*Q. Do treated silicas have an effect on drying of some coating systems?*

A. No effect whatever. The percentage of addition is too low.

EARL B. SMITH, *Secretary*

## **Montreal**

**Jan. 6**

B. Melsbach, of SICO, Inc., presented a talk on "WOOD FINISHING SYSTEMS."

Mr. Melsbach commented on the various types of wood finishing systems used in North America. He emphasized the differences in demands and restrictions between Canadian and United States markets.

"INDUSTRIAL WOOD COATINGS" was discussed by R. Wint, Hercules Corp.

Presently, the wood furniture industry in the United States is experiencing a sharp reduction in demand, higher costs, regulations, and requirement for improvement in quality, explained Mr. Wint. However, the industry should have a positive outlook due to economic improvement, better understanding by regulators, moderation of labor demands, and the fact that a product quality advantage commands a premium, according to Mr. Wint.

Trends in the "case good furniture" segment of the market were outlined. Mr. Wint stressed the need for finishing

systems under development to be more versatile since no two manufacturers produce the same piece of furniture in the same manner. In addition, said Mr. Wint, the substrate (wood) is extremely variable and cutting alone has a major influence on the finishing capabilities. Graphs representing concurrent 37-year studies illustrate type of wood used, governed by availability and economics, as well as styling.

According to Mr. Wint, the market potential for furniture finishes, O.E. coatings in 1979, represented 27% of the total coatings market. Mr. Wint outlined the development of furniture finishing systems' components including the six categories of nitrocellulose lacquers, which were developed as innovations between the years 1935-1975 as the need arose to solve many problems in finishing. Water-borne types which were sufficiently flexible to formulate products appeared on the scene, but were not more successful. The reasons were difficulty in handling and the high cost of application, i.e. in terms of heat required to evaporate the water. At best the cost ratio was 5:1 said Mr. Wint. He also claimed that in an actual study of WB industrial finishing the control of relative humidity between 30-60% was absolutely essential to maintain finish quality. Additional data presented suggested that the WB system studied was not adequate in drying time, film hardness or pounds of solids deposited per pound of solvent vs. the conventional lacquer control. However, Mr. Wint stated that emulsified nitrocellulose WB coatings should be explored.

Mr. Wint presented an impressive case for "Compliance Lacquers" using chlorinated solvents such as 1, 1, 1 - trichloroethane or methylene chloride, the former being exempt in almost all states and the latter in at least 50%. Comparison of TLV's showed the chlorinated solvents to be less hazardous than conventional lacquer solvents. When the solubility parameters of nitrocellulose were studied it was found that the chlorinated solvents were not solvents for nitrocellulose but rather diluents, explained Mr. Wint.

Mr. Wint cited the following example:

### *Conventional Lacquer*

5.8 lb Volatile

2.2 lb Nonvolatile

### *Compliance Lacquer*

3.2 lb Volatile

2.6 lb Chlorinated diluent

2.2 lb Nonvolatile

which resulted in VOC being reduced by approximately 46%.

Mr. Wint stated that in some studies compliance lacquers based on chlorinated solvents have proved feasible whereas water-borne have not.

ERIC J. TEMPLETON, *Secretary*

## **Montreal**

**Feb. 3**

The meeting was held with the Montreal chapter of the National Association of Corrosion Engineers.

R. Hartley, of International Paints, spoke on "THE PAINT BUSINESS—A CHALLENGE OR A DEAD END."

Mr. Hartley reviewed "Hartley's Laws of the Paint Industry" which presented a satirical view of all facets of a typical paint organization.

Graphical interpretations of the number of companies involved in the industry, the fluctuation of the number of employees, and salary comparisons over the past 15 years were featured. Mr. Hartley stated that payroll plus raw materials account for approximately 71% of the cost of a unit of paint.

A review of the various aspects of the U.S. paint industry, which compared sizes and numbers of companies to their share of the market, was given. Mr. Hartley noted that trade sales as a group exhibited less growth than the gross national product of the U.S. Industrial coatings were likewise showing less growth than durable goods, said Mr. Hartley, possibly suggesting the substitution of other forms of product finishes.

Mr. Hartley discussed percent return on net worth and reinforced his view with a graphical representation that profitability of the paint industry is steadily dropping below that of all other industry combined. A probable cause for this, stated Mr. Hartley, could be the highly fragmented state of the industry, with no one company directing the market.

Technical aspects of the industry from the 1920's into the 1970's were reviewed, covering the development of synthetic film formers, polymer chemistry, sophisticated resins including epoxies, vinyls, polyurethanes, PVA's, acrylics, and specialized application methods.

Trends in consumption of the various types of resins were explained, and methods of manufacturing from the late 1800's to the present were discussed. Mr. Hartley commented on automation and the potential use of robots. Various aspects of products were briefly considered.

Mr. Hartley stated that the paint industry is easy to enter due to low capital investment required, readily available information, and many easily manufactured products. However, at the same time profitability is poor, markets are declining, worker loyalty is low, and the industry tends to be non-innovative. Also, advances in the form of better products have evolved and an overall better understanding of basic processes is a part of the industry, explained Mr. Hartley.

The advantage of being involved in the paint industry, according to Mr. Hartley, was being part of a relatively

small, interesting, non-scientific industry in which one came in contact with many people.

*Q. Are declining paint sales the result of improved products?*

A. This is probably partly true but not the only factor.

ERIC J. TEMPLETON, *Secretary*

## New England

Jan. 21

James Joudrey, of Columbian Chemicals, presented a talk entitled "CARBON BLACK IN AQUEOUS COATINGS APPLICATIONS."

The different types of carbon blacks were discussed, and Mr. Joudrey noted that furnace blacks were the most used and the most important category for coatings.

A review of the manufacturing process was presented and a slide presentation illustrated a typical industrial process. Mr. Joudrey discussed, with the aid of slides, the paracrystalline nature of carbon black and the domain concept to explain the particles of carbon black.

The important characteristics of carbon black, according to Mr. Joudrey, are particle size, surface area, structure, surface chemistry, and physical form.

Particle size is between 12–70 micrometers and affects the masstone, or jetness, of the furnace black. The jetness and tint strength increase as particle size decreases, explained Mr. Joudrey.

Surface area of furnace blacks is in the range of 20–1000 m<sup>2</sup>/g. By increasing the surface area, color, tint strength, drier absorption, system viscosity, and dispersion, time increases. Nitrogen and iodine absorption tests are used to measure carbon black surface area, said Mr. Joudrey.

According to Mr. Joudrey, low structure furnace blacks provide low vehicle demand, low viscosity, and poor conductivity. High structure blacks exhibit vehicle demand, high viscosity, good conductivity, low pigment loadings, and improved dispersibility.

Mr. Joudrey noted that some of the furnace blacks are surface treated by chemical oxidation with oxidation levels varying from 0.3%–10% and a corresponding pH range of 9–2.

There are three general industrial grades of carbon blacks for coatings, according to Mr. Joudrey: low color characterized by large particle size (62–70 micrometers) and low surface area (24–38 m<sup>2</sup>/g); medium color of medium particle size (23–31 micrometers) and surface area (58–124 m<sup>2</sup>/g); and high color characterized by small particle size (13–18 micrometers) and high surface area (187–935 m<sup>2</sup>/g). Low

color blacks are used in low cost drum enamels and chassis paint, medium color blacks are the general purpose black pigments and the high color blacks are used in specialty coatings, automotive, and appliance coatings, explained Mr. Joudrey.

The major factors to be considered in the selection of carbon black include jetness, form (beads or powder), vehicle compatibility, tint, opacity requirements, rheology, and dispersion.

Mr. Joudrey explained the properties affecting dispersion. Large particle size carbon blacks are generally easier to disperse than carbon blacks of small particle size, explained Mr. Joudrey. Likewise, the higher structured blacks are more easily dispersed. The surface treatment of furnace blacks can act as built-in wetting agent and dispersion aid, said Mr. Joudrey.

The steps in the dispersion process are: wetting—displacing the air and moisture on the particle surface with vehicle; dispersion—deagglomerating the particles and coating surrounding them with vehicle; and stabilization—completing the dispersion to make it complete and irreversible.

In aqueous systems, the pigment can be dispersed in vehicle containing unsolubilized resin or it can be dispersed in water with a dispersing aid, explained Mr. Joudrey. One such formula for the latter would be 35% carbon black, 62% water (pH 9.5), and 3% dispersing aid, said Mr. Joudrey.

The most effective dispersing aids for carbon blacks were discussed. Mr. Joudrey emphasized that the correct amount of surfactant must be used and that it varies in direct proportion to the surface area: 8% dispersant (based on carbon black) for low color blacks; 12% dispersant for medium color blacks; and 14% for high color carbon blacks.

According to Mr. Joudrey, sand and bead mills are the best for dispersion of carbon blacks in aqueous systems, and the roller mill is also acceptable. High speed mixers should be used for premix only, stressed Mr. Joudrey. Pebble and ball mills are excellent but one must be aware of potential foaming problems.

Mr. Joudrey concluded by giving some formulating tips for carbon blacks in water-borne coatings: (1) color/tint properties should be similar to solvent systems; (2) the surface chemistry of the pigment will greatly effect the gloss and stability of the coating; (3) trial and error methods are needed for evaluation; and (4) highly acidic carbon blacks could cause problems in some systems. A slide presentation illustrated some actual formulations.

*Q. Can you measure dispersion by conductivity studies?*

A. Yes and no. With a low structure black you would start very low in conductivity and end very low, therefore this method could not be used. It may be possible with high structure blacks, but it would not be a very good measure of dispersion. The best method involves using an electron microscope, otherwise start with the Hegman gauge.

*Q. What dictates the structure?*

A. The structure is determined in the furnace itself; sometimes with additives.

*Q. As you break down the structure how do you change the conductivity?*

A. This occurs with high structure black only. At low dispersion you have fair conductivity. At high dispersion the conductivity drops off dramatically.

*Q. What are the optimum conditions for grinding carbon black?*

A. Optimum conditions depend on the vehicle and can vary considerably.

*Q. Are gloss measurements a good way to measure dispersion?*

A. Yes, but you have to know what the optimum is before you go into the plant with the method.

*Q. What is the effect on viscosity with dispersion?*

A. The viscosity will go up with better dispersion.

*Q. Could the Pfund cryptometer be used to measure dispersion?*

A. Possibly, but I have no experience with this. It sounds like a good project for someone.

N. BRADFORD BRAKKE, *Secretary*

## Pacific Northwest

Dec. 17

Frank Henning, Environmental Consultant, presented a talk entitled, "RESPONSIBLE, HAZARDOUS WASTE MANAGEMENT IN CANADA."

According to Mr. Henning, more stringent waste regulations are expected in the near future.

A review of the types of wastes and their generators was presented. Mr. Henning discussed the various disposal options such as simple dump, high temperature incineration at sea, and sophisticated treatment plants.

A recent federal report was summarized which recommended physical chemical treatment along with secure land fills in each of the four Western Provinces, with high temperature incineration only in Alberta. Mr. Henning summarized the recommendation from the British Columbia Waste Management Committee as follows: (1) do not generate waste if an alternate is available; (2) recycle waste if possible; (3) treat

waste on site; and (4) treat waste elsewhere. Problems that arise with this recommendation, according to Mr. Henning include: (1) the need for a waste specification system; and (2) the need for a manifest system to keep track of waste. In 1982, legislation to deal with waste disposal will be enacted, said Mr. Henning.

OTTO SCHMIDT, *Secretary*

## Piedmont

Jan. 20

Dr. Ronald Bower, of the Shell Development Co., presented "ENVIRONMENTALLY ACCEPTABLE AND ENERGY EFFICIENT EPOXY RESIN COATINGS."

With the increasing number of environmental regulations concerning coatings application, many industries are forced to modify current practices, according to Dr. Bower. Most industries require a 60-65% volume solids level to meet EPA requirements in reducing emissions. It is estimated that by 1985 the market share of coatings systems utilized will be: 33% low solids; 25% high solids systems; 30% water-borne; 6% powdered coatings; and 6% UV, EB, heat cure systems, explained Dr. Bower.

According to Dr. Bower, epoxy usage will increase, enabling formulators to meet emission restrictions and reduce energy costs. Current research is developing epoxy resins that are compatible with high solids, low cure temperature coatings systems, that retain excellent gloss, flex, adhesion, and weathering characteristics. By careful selection of epoxy resins and cross-linkers, good viscosities and stability can be achieved for application, said Dr. Bower. Continuing research will lead to more epoxy formulation in aqueous and UV heat cure systems.

J.E. HUSTED, *Secretary*

## Pittsburgh

Jan. 4

Dr. George W. Gerhardt, of Mobil Chemical Co., spoke on "WETTING AND FLOW-OUT OF ORGANIC COATING."

Dr. Gerhardt discussed the formulation of the coating. Important in the formulation, according to Dr. Gerhardt, are percent solids and viscosity; evaporation rate of the solvent; and cure rate and final film properties.

Dr. Gerhardt pointed out that the EPA is now setting limits for VOC emissions and has challenged the formulation of coatings. Most solvents have low surface tension rates and wetting the substrate is not a problem, explained Dr. Gerhardt. Water has a high surface tension rate. A ratio of 80% water and 20% cellulose has a rate of 40 dynes per cm. Even this rate is too high for some surfaces and a prime

coat is needed to wet the surface, said Dr. Gerhardt.

A demonstration of good and poor wetting of solvents on metal and plastic was featured. Dr. Gerhardt explained the curve of good and poor wetting solvents for high and low surface tensions versus high and low viscosity. According to Dr. Gerhardt, the film must start curing before part of the fast evaporating solvent leaves the film in order to keep good surface wetting. If you start to lose surface wetting, you might have to change to a slower solvent. A too high boiling solvent might be left in a film and if this were a can coating, it could damage the product in the can, said Dr. Gerhardt.

Dr. Gerhardt explained the importance of having enough solvent in the water reducible coating in order to get coalescence of the film before all of the solvent evaporates. How fast the surface tension increases as the solvent evaporates is quite important, stressed Dr. Gerhardt.

MICHAEL GILLEN, *Secretary*

## Pittsburgh

Feb. 1

"HOW SCIENCE CAN HELP PIGMENT DISPERSION" was discussed by Dr. Godfrey D. Parfitt, of Carnegie-Mellon University.

According to Dr. Parfitt, titanium dioxide is relatively expensive, and in order to get its full value, good dispersion is needed. Poor dispersion, explained Dr. Parfitt, causes loss of gloss, hiding, durability, and UV resistance. Uncoated TiO<sub>2</sub> has very high surface tension and is very difficult to deagglomerate. Water on the surface of the dry TiO<sub>2</sub> also hurts dispersion, said Dr. Parfitt.

Dry powders were discussed by Dr. Parfitt and their sizes, shapes, and surface chemistry were explained. Dr. Parfitt stressed the importance of surface chemistry.

Graphs illustrated the free energy curve and several equations.

Dr. Parfitt explained the changes in dispersion stability which can be caused by pH, temperature, and solvents.

MICHAEL GILLEN, *Secretary*

## Rocky Mountain

Jan. 11

Honored guests were Federation President Howard Jerome and Field Services Director Thomas Kocis.

President Jerome discussed the Federation and its membership, PRI, and the progress of the coatings correspondence course. He urged members to get involved in Society and Federation projects.

Mr. Kocis presented a slide presentation which discussed Federation in-

come and allocations. Also, a presentation produced by the Manufacturing Committee of the New York Society was shown.

DON M. SHILLINGBURG, *Secretary*

## St. Louis

Jan. 19

A moment of silence was observed in memory of Society Past President Herbert Rosenblatt, of Steelcote Mfg. Co., who died recently.

Dr. Herman Lanson, Education Chairman, explained to the 26 high school teachers attending Education night, a "take-with" experiment to demonstrate thermoplastic and thermosetting polymers to their students. Also part of the Education Night program was Dr. Jim Stoffer, Professor of Polymer Chemistry at the University of Missouri-Rolla, who discussed the opportunities available for students entering the coatings field.

Dr. John C. Graham, Professor of Polymer and Coating Technology at Eastern Michigan University, discussed "POLYMERS AND RESINS IN THE COATINGS INDUSTRY."

Dr. Graham described the opportunities available at the university and discussed a program leading to a B.S. degree in Polymer Science. Numerous slides showed a coating laboratory and its typical testing equipment.

A second slide presentation discussed polymers and resins in the industry.

ROBERT J. GIERY, *Secretary*

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# Future Society Meetings

## Birmingham

(May 6)—"COMPARISON OF EXPOSURE TEST METHODS"—Speaker from Hoechst UK Ltd.

## Cleveland

(Apr. 20)—PLANT TOUR.  
(May 18)—60th ANNIVERSARY OF FEDERATION MEMBERSHIP. PAST PRESIDENTS AND SPOUSES' NIGHT.

## Dallas

(May 13)—"ANATOMY OF AN EMULSION POLYMER"—Benjamin Kine, Rohm and Haas Co.

## Detroit

(Apr. 27)—"FUMED SILICA FOR RHEOLOGY CONTROL OF HIGH SOLIDS COATINGS"—Speaker from Cabot Corp.  
(May 4)—FOCUS—"NEW FRONTIERS IN APPLICATION TECHNIQUES"—

Michigan State University, Education Center.

(May 25)—JOINT MEETING with Detroit Paint & Coatings Association.  
(Sept. 28)—Presentation by Cargill Corp.

(Oct. 19)—EDUCATION COMMITTEE'S PRESENTATION/PAST PRESIDENTS' NIGHT

(Dec. 14)—"HIGH SOLIDS COATINGS"—Dick Hong, Spencer Kellogg Div., Textron, Inc.

## Golden Gate

(Apr. 19)—"HAZARDOUS WASTE DISPOSAL"—Michael L. Markowitz, Rollins Environmental Services of Texas, Inc.

(May 17)—"NEW DEVELOPMENTS IN SANDMILL DISPERSIONS TECHNOLOGY"—Roland F. Swett, Moorehouse Industries, Inc.

(June 14)—"COMPLIANCE SOLVENTS FOR INDUSTRIAL COATINGS"—Violette Stevens, Dow Chemical Corp.

## Houston

(May 12)—"ANATOMY OF AN EMULSION POLYMER"—Benjamin Kine, Rohm and Haas Co.

## Los Angeles

(May 12)—"NEW DEVELOPMENTS IN SANDMILL DISPERSION TECHNOLOGY"—Ronald E. Swett, Moorehouse Industries.

(June 9)—"COMPLIANCE SOLVENTS FOR INDUSTRIAL COATINGS"—Violette Stevens, Dow Chemical Corp.

## Montreal

(May 5)—"THE PURCHASING, SALES INTERFACE"—John Humphreys, Sherwin-Williams Co., and Paul Rheume, NL Chemicals, Canada, Inc.

## Northwestern

(May 5)—MANUFACTURING SEMINAR.

## Piedmont

(Apr.)—JOINT MEETING with Virginia Section.

(May 20)—"COMPUTER SELECTION OF SOLVENT BLENDS"—Dr. Albert Rocklin, Shell Development Co.

(June 16)—"ECONOMIC RECOVERY OF SOLVENT VAPORS"—Larry J. Durr, DCI Corp.

## Pittsburgh

(May 3)—"DESIGN CONSIDERATION FOR HIGH SOLIDS REACTIVE COATINGS"—Dr. Loren W. Hill, Monsanto Plastics and Resins Co.

## Rocky Mountain

(May 10)—"NEW DEVELOPMENTS IN SANDMILL DISPERSIONS TECHNOLOGY"—Ronald E. Swett, Morehouse Industries.

(June 7)—"COMPLIANCE SOLVENTS FOR INDUSTRIAL COATINGS"—Violette Stevens, Dow Chemical Co.

## St. Louis

(May 18)—"THE WHYS AND WHEREFORES OF CARTRIDGE FILTRATION IN THE COATINGS INDUSTRY"—D. S. Onnen, AMF Cuno Div. "EFFECTIVE PAINT WASTE TREATMENT"—E. M. Antonucci, Drew Chemical Corp. "MODERN PAINT DISPERSIONS"—H. Purcell, Morehouse, Ind.

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 BUTLER, JAMES M.—Sherwin-William Co., Chicago, IL.  
 COLEMAN, KENNETH W.—Elpaco Chemical Ctg., Elkhart, IN.  
 ERICSSON, ROBERT—Rust-Oleum Corp., Evanston, IL.  
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 JUNG, PETER A.—DeSoto, Inc., Des Plaines.  
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 MILLER, ROBERT G.—Whittaker Corp., Batavia.  
 MURPHY, EUGENE M.—Whittaker Corp., Batavia.  
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 SANTOS, EDUARDO S.—Rust-Oleum Corp., Evanston.  
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 STEWART, JOHN, JR.—Hempel's Marine Paints, Wallington, NJ.  
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 Fred Daniel, of Daniel Products Co.

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 Lenny Freund, of L. Freund Associates, Inc.  
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 Bill Stewart, of Tenneco Chemicals, Inc.  
 John Toscano, of F.O. Pierce Co.  
 Len Treinkman, of Orelite Chemical Coatings Inc.  
 Steve West, of United Laboratories Co., Inc.

## Constituent Society Meetings and Secretaries

**BALTIMORE** (Third Thursday—Eudowood Gardens, Towson). JOSEPH D. GUISTO; Lenmar, Inc., 150 S. Calverton Rd., Baltimore, MD 21223.

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

**CHICAGO** (First Monday—meeting sites in various suburban locations). JOHN R. INGRAM, DeSoto, Inc., 1700 S. Mt. Prospect Rd., Des Plaines, IL 60018.

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

**CLEVELAND** (Third Tuesday—meeting sites vary). DONALD C. DENISON, JR., Hilton Davis Chemical Co., 5254 Berkshire Dr., N. Olmsted, OH 44070.

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

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

**GOLDEN GATE** (Monday before third Wednesday—Alternate between Sabella's Restaurant on Fisherman's Wharf and the Sea Wolf at Jack London Square, San Francisco). KEN E. TRAUTWEIN, Sherwin-Williams Co., P.O. Box 23505, Oakland, CA 94623.

**HOUSTON** (Second Wednesday—Sonny Look's, South Main). GEORGE SCHWARTZ, Cook Paint & Varnish Co., P.O. Box 3088, Houston, TX 77001.

**KANSAS CITY** (Second Thursday—Cascone's Restaurant). MEL BOYER, Patco Coatings Products, 3947 Broadway, Kansas City, MO 64111.

**LOS ANGELES** (Second Wednesday—Steven's Steak House). EARL SMITH, Spencer Kellogg Div., Textron, Inc., P.O. Box 7205, Long Beach, CA 90807.

**LOUISVILLE** (Third Wednesday—Hasenour's Restaurant). E. D. THOMAS-SON, Louisville Varnish Co., 1400 Maple St., Louisville, KY 40207.

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

**MONTREAL** (First Wednesday—Bill Wong's Restaurant). ERIC TEMPLETON, NL Chemicals Can., Inc., 2140 Sun Life Bldg., Montreal, Que., Can., H3B 2X8.

**NEW ENGLAND** (Third Thursday—Fantasia Restaurant, Cambridge). N. BRADFORD BRAKKE, Lilly Chemical Products, P.O. Box 188, Templeton, MA 01458.

**NEW YORK** (Second Tuesday—Landmark II, East Rutherford, NJ). H. ELLIS, D. H. Litter Co., Inc., 116 E. 16th St., New York, NY 10003.

**NORTHWESTERN** (Tuesday after first Monday—Edgewater East Restaurant). HERBERT DAVIDSON, Spencer-Kellogg Div., Textron, Inc., 525-25th Ave. S.E., Minneapolis, MN 55414.

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

**PHILADELPHIA** (Second Thursday—Valle's Steak House). RALPH MYERS, Del Vac Ink & Color Co., 1301 Taylors Ln., Riverton, NJ 08077.

**PIEDMONT** (Third Wednesday—Howard Johnson's Coliseum, Greensboro, NC). JAMES E. HUSTED, Mobil Chemical Co., P.O. Box 2438, High Point, NC 27261.

**PITTSBURGH** (First Monday—Skibo Hall, Carnegie Mellon Univ.). MICHAEL GILLEN, Van Horn, Metz & Co., Inc., 400 Keystone Dr., Carnegie, PA 15106.

**ROCKY MOUNTAIN** (Monday following first Wednesday—Gustaus Ridgeview, Lakewood, CO). DONALD SHILLINGBURG, Union Chemical Div. of Union Oil, 1535 W. 13th Ave., Denver, CO 80204.

**ST. LOUIS** (Third Tuesday—Salad Bowl Restaurant). ROBERT J. GIERY, Spatz Paint Industries, Inc., 1601 N. Broadway, St. Louis, MO 63102.

**SOUTHERN** (Gulf Coast Section—Various Dates; Central Florida Section—Third Thursday after first Monday; Atlanta Section—Third Thursday; Memphis Section—Second Tuesday; Miami Section—Tuesday prior to Central Florida Section). J.E. GEIGER, Sun Coatings, Inc., 12295 75th St., N., Largo, FL 33540.

**TORONTO** (Second Monday—Mayfair Restaurant). R. KUHNEN, Tioxide Canada, Inc., 1 Eva Rd., Etobicoke, Ontario, Canada M9C 4Z5.

**WESTERN NEW YORK** (Third Tuesday—Lord Amherst Restaurant, Williamsville, NY). MICHAEL C. KAUFMAN, Bisonite Co., Inc., P.O. Box 84, Kenmore St., Buffalo, NY 14217.

### Associate

BARUFALDI, DANIEL J.—Ciba-Geigy Corp., Ardsley, NY.  
BOGATIN, ROBERT S., JR.—Peltz Rowley Chemical Co., Philadelphia, PA.  
CUBA, RUDY N.—Alcan Ingot & Powders, Elizabeth, NJ.  
DAVIS, EDWARD A.—Henkel Corp., Maywood, NJ.  
EVANS, MARK D.—Charter Chemical Co., Wayne, PA.  
JACKSON, SUZANNE—Spencer Kellogg Div., Textron, Inc., Rutherford, NJ.  
JORDAN JAMES J.—BASF Wyandotte Corp., Parsippany, NJ.  
OJENIYI, HENRY B.—Arco Chemical Co., Philadelphia, PA.  
PRZYBYLOWSKI, ROBERT—Whittaker, Clark, & Daniels, Inc., South Plainfield, NJ.  
SMERAK, LANCE P.—Mobay Chemical Corp., Hawthorne, NJ.

## NORTHWESTERN

### Active

FARMER, RICHARD—Conklin Co., Inc., Shakopee, MN.  
HERMANSON, RAY—Conklin Co., Inc., Shakopee.  
LUNSFORD, DUANE—Conklin Co., Inc., Shakopee.

## PHILADELPHIA

### Active

WOOLER, NEIL A.—Lawrence-McFadden Co., Philadelphia, PA.

### Associate

REITANO, PHILIP A.—Kay-Fries Inc., Montvale, NJ.

## ST. LOUIS

### Active

MURPHY, PATRICK N.—Crown Zellerbach, Shrewsbury, MO.  
STORM, RICHARD W.—Sinnott Lacquer Co., St. Louis, MO.

### Associate

MEUSER, RALPH—Transchemical Inc., St. Louis, MO.  
PHILLIPS, W.T.—Pfizer Inc., MPM Div., E. St. Louis, IL.

### Educator and Student

BECHMANN, MICHAEL ROY—University of Missouri-Rolla, Rolla, MO.  
FAHIM, MAHER—University of Missouri-Rolla, Rolla.  
KOHLE, KAREN—University of Missouri-Rolla, Rolla.  
ROTH, STEVEN P.—University of Missouri-Rolla, Rolla.  
STUDEBAKER, B. GARTH—University of Missouri-Rolla, Rolla.



# Meetings/Education

## 46th Annual Meeting Held By Southern Society; Officers Elected

The Southern Society for Coatings Technology hosted its 46th Annual Meeting entitled, "Coatings Technology—Strategies for the Eighties," on March 10-12 at the Hyatt Regency—Savannah, Savannah, GA. According to General Chairman John R. Lawson, of Kerr McGee, the theme emphasized strategies that will be used by paint manufacturers to survive and profit in the upcoming decade.

Program for the meeting was as follows:

### Wednesday, March 10

Opening the program was the Past President's Paint Seminar, "Exterior Clear Coatings." Featured topics included:

"Introduction to Clear Coatings: A Broad Overview."

### New England Society To Host 'Coatings Tech Expo '82'

The New England Society for Coatings Technology will sponsor the biennial "Coatings Tech Expo '82" at the Sheraton Inn & Conference Center in Boxborough, MA, May 19-20.

Following the theme, "PAT: Productivity, Availability, and Technology in the 80's," the exposition will open Wednesday, May 19 at 12:00 noon with 13,000 square feet of exhibit. Concurrently scheduled are seminars planned for the manufacturers and managers from 12:00 noon-5:00 p.m. The exhibits will open on Thursday, May 20 at 10:00 a.m. and continue to 4:00 p.m., with technical seminars being held concurrently.

The program will conclude with the Annual Dinner featuring Dick Flavin, "The Art Buchwald of Television," as keynote speaker.

Chairman for the exposition is Robert Marderosian, of Cypress Color & Chemical, Inc. Other chairmen include: *Program*—John Fitzwater, Polyvinyl Chemical Industries; *Technical Program*—N. Bradford Brakke, Lilly Chemical Products and Maureen Lein, Polyvinyl Chemical Industries; *Manufacturers/Managers Program*—Tom Manning, Samuel Cabot, Inc. and Fran Koebert, Kyanize Paints, Inc.; *Publicity/Advertising*—Robert Modrak, Benjamin Moore & Co.; *Exhibit Coordinator*—Michael Paisner, M.S. Paisner, Inc.; and *Social Functions*—Daniel Toombs, Lukens Chemical Co.

For additional information, contact Dame Associates, Inc., 51 Church St., Boston, MA 02116.

"Trade Sales Clears."

"Industrial Non-Product Finishes: Maintenance, Commercial, Institutional, Specialties."

"Industrial Product Finishes: Brass, Copper, Aluminum."

### Thursday, March 11

The Keynote Address entitled, "How's Our Visibility," was presented by William A. Bours, III, retired Vice-President, Fabrics and Finishes, E.I. du Pont de Nemours & Co. Mr. Bours examined the low profile of the paint industry in the public's eye and outlined the benefits of better public understanding of the industry's contributions to society.

Presentations for Thursday's session included:

"Strategies for the 80's—Learning from the Past to Plan for the Future"—V.L. Stevens, Dow Chemical U.S.A.

"Your Product, Your Plant., Your People: They Build Your Public Image"—F. Schulenberg, American Paint & Coatings Journal.

Panel: "Production Design for Profit"—Moderated by Terrence J. Walsh, Kinsmen Corp.

A. "Necessities of a Paint Plant—Systems, Procedures, Controls"—Kenneth Erdman, DeSoto, Inc.

B. "A Modern Trade Sales Manufacturing Plant"—Howard Horton, Glidden Coatings and Resins, SCM Corp.

C. "Upgrading Existing Facilities"—Scott McKenzie, Southern Coatings.

D. "Optimizing Your Human Resources"—John Covington, Gilman Co.

"Historic Preservation in Savannah"—Frederick Spitzmiller.

### Friday, March 12

Panel: "Paint Application—Present and Future Trends"—Moderated by Ron Nelson, Kelco Co.

A. "Application by Brush"—David Howard, Purdy Brush Co.

B. "Application by Spray"—Robert H. Klass, Graco, Inc.

C. "Application by Roller"—Jack Kane, Wooster Brush Co.

D. "Rheology and Application"—Phil Winston, Kelco Co.

"Coatings Opportunities in the Eighties—They're Disguised as Problems"—John C. Dean, Chemical Marketing Services.

### Bob English Honored

After the morning session's presentations, the Society's luncheon was held at which time awards were presented. J. Robert "Bob" English, retired partner of R.T. Hopkins Co., was honored as the Senior Statesman for 1982 [See People p. 84—Ed.]. The Society's Annual Meeting and Board of Director Meeting concluded the program.

### Officers Elected

Southern Society officers for 1982-83 were elected as follows: *President*—Dan M. Dixon, Freeport Kaolin; *President-Elect*—Bill Early, Piedmont Paint; *Vice-President*—Jim Geiger, Sun Coatings; and *Secretary-Treasurer*—Sal Sanfilippo, Reichhold Chemicals. Bobby Moore, of Interstate Paint, continues as Society Representative.

### FSCT Scholarships Available

To meet the demand for more technically trained people in the coatings industry, the Federation is continuing its scholarship program, with funds made available to various universities in the U.S.

These funds are to be used as grants-in-aid for students in the coatings technology program at each institution, with preference given to qualified scholarship applicants who are children of members of the Federation.

Members who have children wishing to make application for the 1982-83 academic year should contact Federation headquarters. Write Scholarship Fund, Federation of Societies for Coatings Technology, 1315 Walnut St., Suite 830, Philadelphia, PA 19107.

## 25th Annual Conference Sponsored by Cleveland Society

The Education Committee of the Cleveland Society for Coatings Technology presented its 25th Annual Technical Conference entitled, "Advances in Coatings Technology," March 23-24, at Baldwin-Wallace College, Berea, OH.

Under the direction of Chairman John C. Weaver, the Conference was divided into the following two, day-long symposia:

### *Coatings Application Technology*

Presiding were Doug Wolf, of Sherwin Williams Co., and Carl J. Knauss, of Kent State University.

"Mechanics of Electrostatic Atomization, Transport, and Deposition of Coatings"—G.C. Bell, Jr. and J. Hochberg, of E.I. duPont de Nemours Co.

"Equipment Requirements for Conversion to Environmental Coatings"—A.C. Walberg, of Arvid C. Walberg Co.

"Estimation of Vapor Composition Above Solvent-Resin Solutions"—R. Huddleston, Nordson Corp.

"Brookfield Viscometers for Determination of Low-Shear Viscosity and Leveling Behavior"—R.E. Smith, of PPG Industries, Inc.

"Basic Principles of Electrostatics in Powder Coating Application"—G.E. Gorrow, Glidden Coatings & Resins Div., SCM Corp.

"Automation and Computerization in the Coatings Analytical Laboratory"—G.P. Cunningham and J.J. Shutak, of PPG Industries, Inc.

### *Advances in Surface Analysis*

Presiding were Theodore Provder, of Glidden Coatings & Resins Div., SCM Corp., and Alexander Jamieson, of Case Western Reserve University.

"Alphabet Soup of Surface Spectroscopy for the Characterization of Solids"—M. Mehicic, J.R. Mooney, and J.G. Grasselli, The Standard Oil Co. (Ohio).

"SEM Studies of Latex Paint Film Morphology as a Function of the PVC/CPVC Ratio"—D.T. Smith, Sherwin William Co.

"A Study of Defects in Container Coating/Substrate Composites"—R.M.

Holsworth, Glidden Coatings & Resins Div., SCM Corp.

"Surface Analysis by Epitaxial Adsorptions and Polymerizations"—S.E. Rickert, Case Western Reserve University.

"The Nanotensilemeter—A Tensile Test Instrument with Applications to Coatings and Interfaces"—R.W. Hoffman, Case Western Reserve University.

"Steel Surface Cleanliness: Preparation, Characterization, and Paint Performance"—R.P. Wenz, 3M Co.

"Spectroscopic Analysis of Polymers"—J.A. Young, P.A. Budinger, and J.R. Mooney, The Standard Oil Co. (Ohio).

## WPTG To Sponsor 22nd Annual Symposium in D.C.

"Technology Trends" will be the theme of the Washington Paint Technical Group's 22nd Annual Symposium to be held at the Marriott Twin Bridges Motel, Washington, D.C., April 19-20.

Milton A. Glaser, Consultant and Vice-President, R&D, of Midland Div., Dexter Corp., will be the keynote speaker. His speech, designated as the Maurice S. Moen Address, is entitled "Innovative Concepts in R&D Management."

The program will also feature the following topics and speakers:

"Opaque Polymers"—Dr. Kayson Nyl, Rohm and Haas Co.

"Performance of Silicone Modified Organic Resins in Maintenance Applications"—William A. Finzel, Dow Corning Corp.

"Trends in Appliance Coatings"—Dr. Thomas J. Miranda, Whirlpool Corp.

"Biodeterioration of Water-Based Coatings"—Dr. Robert Opperman, Cosan Chemical Corp.

"A Look at Painting Robots of the Eighties"—Norman N. Fender, Graco Robotics, Inc.

"New Developments in Epoxy Curing Agents"—John J. Sinclair, Pacific Anchor Chemical Corp.

"Occupational Exposures and Environmental Control Measures for Polyurethane Coatings"—C. Emory Tate, B.D.M. Corp.

"Current Federal Highway Research in Traffic Lane Marking Materials"—Dr. Brian H. Chollar, Federal Highway Administration.

"Development of New Coatings Technology"—Fred Lafferan, U.S. Army Mobility Equipment, R&D Command, Fort Belvoir.

"Navy Shipboard Corrosion Control Program for an Expanding Navy"—Stephen D. Rogers, Naval Sea Systems Command.

"The Impact of Government Regulations on Chemical Analyses in the Coatings Industry"—Dr. Darlene Brezinski, DeSoto, Inc.

For further information, contact Washington Paint Technical Group, P.O. Box 12025, Washington, D.C. 20005, or Bernard Appleman at (703) 285-2004 or Mildred Post at (301) 530-1664.

## Philadelphia Society Announces Dispersion Seminar

The Technical Committee of the Philadelphia Society for Coatings Technology will sponsor a one-day seminar entitled, "Modern Dispersion Technology: Advances in Dispersions with Related Phenomena as Applied to Coatings, Inks, and Plastics," on May 3 at the Stadium Hilton Inn, Philadelphia, PA.

The seminar is designed as a comprehensive course for theoretical and applications minded individuals in the coatings, ink, plastic, and other allied industries who are concerned with problems associated with pigment dispersion. Emphasis of the session will be directed toward raising the level of understanding of the basic phenomena, as well as discussing specific problems related to today's polymer systems.

Presentations to be featured include:

"Pigment Dispersion—In Principle and Practice"—Dr. Geoffrey Parfitt, Carnegie-Mellon University.

"Selection and Application of High

Speed Dispenser"—William Gerlach, Hockmeyer Equipment Co.

"Dispersants and Dispersing Resins"—Dr. Louis Freimiller, Rohm and Haas Co.

"Dispersion of Titanium Dioxide in Fluid and Solid Media"—Richard Emsinger, NI. Chemicals Co.

"Surface Treatment of Filler and Pigments in Plastics"—Donald Cope, Byk-Mallinckrodt Inc.,

"Practical Aspects of Dispersion of Particles in Polymer"—Daniel Mielcarek, Werner and Pfeleiderer Co.

"Dispersion for New Technologies"—Michael Frantz, Daniel Products Co.

Additional information may be obtained from Phil Reitano, Kay-Fries, Inc., 10 Link Dr., Rockleigh, NJ 07647 or Robert Sonntag, Superior Varnish and Drier Co., P.O. Box 1310, Merchantville, NJ 08109.

## Rensselaer Offers 18th Annual Summer Program in Color Technology

Dr. Fred W. Billmeyer, Jr., Professor of Analytical Chemistry at Rensselaer Polytechnic Institute, Troy, NY, and Director of the Rensselaer Color Measurement Laboratory, has announced the courses for the 18th Annual Summer Program in Color Technology. Short courses scheduled include: "Colorimetry and the Eye" (May 25-27); "Color Technology for Management" (June 2-3); "Principles of Color Technology" (June 7-11 or June 14-18); and "Advances in Color Technology" (June 21-25).

### COLORIMETRY AND THE EYE

This course will cover the visual processes of color perception, color matching, the standard observer, color differences, and color appearance. This one-time opportunity is designed for all concerned with color science and technology in all its many branches to attain a better understanding of the first principles behind the use on instrumentation and computers in color technology. The lecturer, Professor W. David Wright, retired, Imperial College, London, England, pioneered the study of most of the course topics over a 50-year career. Fee for the session is \$575.

### COLOR TECHNOLOGY FOR MANAGEMENT

The principles of color technology as they influence management decisions will be discussed. Information on physical and perceptual aspects of color, color measurement, color differences and tolerances, and color matching will be provided. Typical problems in the production and sale of colored products will be covered, with solutions presented.

The course is designed solely for executive and management personnel responsible for programs of production and sales of colored products. Cost for the course is \$550.

### PRINCIPLES OF COLOR TECHNOLOGY

This course provides information on color description, color-order systems, measurement principles, color-difference calculations and tolerances, computer color matching, and colorant properties. Laboratory periods provide hands-on experience in measurement, computation, and problem solving using the latest commercial equipment.

Individuals interested in color science are invited to attend. Fee is \$550.

### ADVANCES IN COLOR TECHNOLOGY

Latest information on the developments and techniques of color science and technology at an advanced level will be provided. Selected advanced laboratory workshops are also scheduled.

The course is designed for those having two or more years of direct personal experience in instrumental color measurement. Course cost is \$600.

For further information, contact The Office of Continuing Studies, Rensselaer Polytechnic Institute, Troy, NY 12181.

## Chemical Institute of Canada To Hold Annual Symposium

The Protective Coatings Division of the Chemical Institute of Canada will hold its annual symposium entitled, "The Color Science of Coatings," on April 28, at the Airport Hilton, Montreal, Quebec, and on April 29, at the Constellation Hotel, Toronto, Ontario.

The following presentations will be featured at the two-day session:

### Detroit Society To Hold FOCUS Conference, May 4

The 7th Annual FOCUS Conference, entitled "New Frontiers in Coatings Applications," will be sponsored by the Detroit Society for Coatings Technology on May 4 at the Management Education Center, Troy, MI.

The conference will FOCUS on the latest coating application technologies. Topics to be featured include: equipment advances; transfer efficiency measurements; color matching considerations; new formulations and solvent requirements; health and safety factors; and government regulations.

For further information, contact Pete Burnett, Detroit Society for Coatings Technology, 765 Dellwood Dr., Ann Arbor, MI 48103.

"Application of Color Science to the Coatings Industry"—Dr. Fred W. Billmeyer, Jr., Rensselaer Polytechnic Institute, Troy, NY.

"Color Difference Evaluation"—A.R. Robertson, National Research Council, Ottawa.

"Computerized Color Control in the Coatings Industry"—Wes A. Coppock, Applied Color Systems, Princeton, NJ.

"The Pigments Are Not Colored"—Professor Peter K. Kaiser, York University, Toronto.

"Coating Pigmentation for Long-Term Color Retention on Precoated Architectural Aluminum"—John W. Wright, Alcan Canada Prod., Ltd., Kingston.

"High Solids Automotive Coatings and Pigmentation"—Dr. George R. Patrick, E.I. du Pont de Nemours & Co., Wilmington, DE.

Advance registration for CIC members is \$40, for nonmembers, \$45. At the door, the fee is \$50 for all participants.

Further information can be obtained by contacting, (for Montreal) Art Hagopian, CIL Paints, Inc., 6930 Clanranald Ave., Montreal, Quebec H3X 2V1; (for Toronto) Dieter Merk, Bayer (Canada) Inc., 1355 Aerowood Dr., Mississauga, Ontario L4W 1C2.

## CALL FOR PAPERS

Electrocoat/82

Cincinnati, OH  
November 9-10

Electrocoat/82, a major technical conference featuring the electrodeposition of paint, will be held November 9-10, at the Westin Hotel, Cincinnati, OH. Sponsored by *Products Finishing Magazine*, the conference will present the latest information of interest to current and prospective users of electrocoating.

Electrocoating has gained wide use as an energy-efficient, environmentally sound, cost-effective method for priming or painting metal parts in a range of industries, notably automotive, appliance, general metal products, and others. Electrocoat/82 will provide a forum for disseminating the latest information on the process.

A call for papers addressing current state of the art in equipment and materials for electrocoating has been issued. Those wishing to submit papers are invited to send an abstract to *Products Finishing Magazine*, 600 Main St., Cincinnati, OH 45202. Abstracts must be received by May 1.

For additional information and a complete program, contact *Products Finishing Magazine*.

## Western Societies' 16th Symposium Scheduled for 1983

Symposium Chairman, Ted Favata, of Triangle Coatings Co., has announced that the 16th Biennial Western Coatings Societies' Symposium and Show will be held February 23-25, 1983, at the Hyatt Regency Hotel—Embarcadero Center, San Francisco, CA.

The tradition of technical excellence will be featured in the symposium, entitled "Spectrum 83—Tomorrow's Technology: Today." The paint equipment show, the nation's second largest, will host over 100 exhibitors of raw materials, and application and production equipment.

Symposium committee chairmen include: *Co-Chairman*—Rob Holt, Sher-

win-Williams Co.; *Technical*—Gordon Rock, The O'Brien Corp.; *Registration and Housing*—Tom Dowd, E.T. Horn Co.; *Entertainment*—Ernest "Bud" Harmon, Borden Chemical Co.; *Exhibits*—John Beater, The O'Brien Corp.; *Ladies' Program*—Rhoda Harmon, Harmon Associates; *Treasurer*—Barry Adler,

Royell, Inc.; and *Publicity*—Dave Waldron, E.I. du Pont.

For exhibit information, contact John Beater, The O'Brien Corp., 450 E. Grand Ave., S. San Francisco, CA 94080. For additional information, contact Chairman Ted Favata, Triangle Coatings Co., 2222 Third St., Berkeley, CA 94710.

### "Textile Country" is Theme for ISCC Annual Meeting

The Inter-Society Color Council will hold its 51st Annual Meeting, "Welcome to Textile Country," April 18-20, at the Sheraton Center Hotel, Charlotte, NC.

Sunday, April 18 opens the annual meeting with a wine-and-cheese reception, dinner, and meetings of the Delegation Chairman, Project Committee Chairman, and New Projects Planning.

A symposium with the theme topic, "Welcome to Textile Country," will be held Monday, April 19. Featured will be the keynote speaker, William R. Martin, Jr., Executive Director of the American Association of Textile Chemists and Colorists. Presentations on "Design Concepts" by Ann Dillon and Jim Brown, and on "Translation to Production" by Vernon Smith, J.R. Aspland, and Tom McCullough, will follow. The symposium will conclude with presentations on "The Garment" by Scott Bailey, and on "Marketing" by Cecil Bessellieu.

An Awards Luncheon is scheduled for noon, at which time the 1982 ISCC Macbeth Award will be presented to Henry W. Levison.

Problems Committee Day is scheduled for Tuesday, April 20, with open sessions of ISCC Project Committees. Tuesday's luncheon will incorporate a brief annual business meeting and the installation of the 1982-84 Officers and 1982-85 Directors.

For additional information, contact Fred Billmeyer, Jr., Rensselaer Polytechnic Institute, Troy, NY 12181.

### NPCA Schedules Additional Production Seminar in Oct.

Registration for the "Production Planning and Inventory Management" seminar sponsored by the National Paint and Coatings Association March 3-5 in Atlanta, GA reached capacity. As a result, another seminar has been scheduled for October 6-8 at the Colony Square Hotel, Atlanta. Attendance will be limited, with registrations accepted on a first-come, first-served basis.

The cost of the seminar is \$350.

For additional information and to register, contact NPCA's Meetings and Conventions Div., 1500 Rhode Island Ave., N.W., Washington, D.C. 20005.

### NCCA Annual Meeting To Be Held May 2-5

Precoated steel for the appliance industry and advanced coating techniques are the key items to be discussed during the National Coil Coaters Association's Annual Meeting, May 2-5, at the Camelback Inn, Scottsdale, AZ.

Representing the largest convention in the coil coating industry, the annual meeting will convene at the General Session scheduled for Monday morning, May 3. Presentations will focus on topics related to advanced coating techniques.

Monday's session will feature the presentation of the annual NCCA Design Competition Awards for innovative, cost-saving applications of coated coil.

Open meetings of the committees comprising NCCA's Technical Section are scheduled for Monday afternoon.

Association business will be conducted during a Tuesday, May 4 NCCA membership meeting, with reports from the Technical and Marketing Sections.

Dean W. Coston, of Coston & Associates, Washington, D.C., will conclude Tuesday's agenda by updating the government regulatory outlook. Mr. Coston is NCCA's Washington representative.

Results of the election of NCCA Officers and Directors will be announced at the General Session, on Wednesday.

For additional information, contact Don White, NCCA, 1900 Arch St., Philadelphia, PA 19103.

### PRI Announces Fourth Annual Howard L. Gerhart Fellowship

A Memorial Fellowship was established in 1978 by PPG Industries, Inc. in memory of Howard L. Gerhart, who published extensively during his career as Vice-President of Coatings R&D. The fellowship acknowledges the scholastic and professional achievements of Dr. Gerhart, who was a Trustee of the Paint Research Institute.

The 1979-80 recipient was Mitchel M.-J. Lin, of Carnegie-Mellon University; the 1980-81 recipient was Thomas Shiah, of Polytechnic Institute of New York; the 1981-82 recipient was Marc Aronhime, of Princeton University.

Chemists or chemical engineers who are doing coatings-related research at accredited universities in the U.S. are eligible for the fellowship. The selected student will receive an award of \$5000 for the applicable academic year.

To apply for the 1982-83 fellowship a letter must be written describing the research problem and its bearing on coatings science. The letter must be signed by the student, endorsed by the professor, and sent before June 5 to:

Dr. Raymond R. Myers, Research Director, Paint Research Institute, Kent State University, Kent, Ohio 44242, (216) 672-2034.

The winner will be notified on July 15, 1982.

# People

**Frank C. Petty** has been appointed Vice-President of Sales and Marketing for Red Devil Paints and Chemicals, Mount Vernon, NY. **Jack Cameron** has joined the firm as Sales Manager based in Chicago, IL.

**Robert A. Van Brederode** has been named Technical Director of Reichhold Chemicals' Thermoplastic and Elastomers Division, headquartered in Hackettstown, NJ.

**Royal A. Brown** has retired from his position as Vice-President, Technical of the National Paint and Coatings Association.

After a career in technical and production positions with member firms of the NPCA, Mr. Brown was appointed Technical Director of the Association in 1966 and was elected Vice-President in 1978.



In addition to representing the Association as its technical spokesman, Mr. Brown was Staff Director of several key committees which included: the Industry Suppliers' Committee; the Scientific Committee; and the Manufacturing Management Committee. Mr. Brown was also Chairman of the Joint Coatings—Forest Products Industry Steering Committee, a model for inter-industry cooperation.

Mr. Brown began his career with Gilman Paint and Varnish Co., Chattanooga, TN, where he was employed as Chief Chemist. He was appointed Vice-President, Research and Production, for Southern Varnish Corp., Roanoke, VA, and was later named Technical Director of Egyptian Lacquer Manufacturing Co., South Kearny, NJ.

Throughout his career, Mr. Brown has been active in various Societies of the Federation of Societies for Coatings Technology and is presently a member of the Baltimore Society. He is a member of the Executive Committee of ASTM Committee D-1, is a Trustee of the Paint Research Institute, and is a member of the American Chemical Society and the American Association for the Advancement of Science.

Mr. Brown plans to be available for consultative work. [See *FSCT Newsletter* in this issue.—Ed.]



F.C. Petty



R.A. van Brederode



J.L. Keenan



R.E. Gulick

**James L. Keenan** has been appointed Vice-President and General Manager of the U.S. Paint Division, Grow Group, Inc.

Also announced by the firm were the appointments of **Laurence Nye Miller III** and **Thomas E. Whitehurst** to the positions of Southern Field Representatives.

International Paint Co., Inc., Union, NJ, has announced the appointments of **Thomas M. Reinhardt** to Chairman of the Board and **John P. Merrill, Jr.** to President.

Union Chemical Division, Petrochemical Group, Union Oil Co. of California, has announced the promotions of **Austin D. Byers** and **James D. Clover** to the newly-created positions of Market Managers for the Chemicals and Solvents divisions, respectively.

**Donald J. Theeuwes** has joined Mooney Chemicals, Inc., Cleveland, OH, as Industry Manager for the Coatings, Lubricants, Fungicides, and Fuel Oil Additives industries.

**William A. Adams** has been appointed General Sales Manager for Morehouse Industries, Inc., Fullerton, CA.

**Robert Dey** has been appointed to the position of Laboratory Supervisor for NL Chemicals, Coatings and Specialty Chemicals. Based in Hightstown, NJ, Mr. Dey is a member of the Philadelphia Society.

**Russell D. Robison, Jr.** has been promoted to Southeastern Region General Manager for The O'Brien Corp. in Brunswick, GA. In his previous position, Mr. Robison was Plant Manager for the Brunswick operation. He is a Chicago Society member.

**Rodwin E. Gulick** has been appointed Senior Marketing Executive in the Coatings Materials Division of Union Carbide Corp., Danbury, CT.

The Valspar Corp., Industrial Coatings Group, has appointed **Don Emch** as Technical Director, Product Finishing. Currently, Mr. Emch is Vice-President of the Northwestern Society. Also announced by the Group was the appointment of **Dr. Larry Brandenburger** as Director, Resin Research and Development. Dr. Brandenburger is a member of the Northwestern Society.

**Kenneth R. Lawson**, Manager of Market Development at DeSoto, Inc., Des Plaines, IL, was elected the 1982-83 President of the Association for Finishing Processes of the Society of Manufacturing Engineers. Mr. Lawson will be installed at SME's 1982 International Tool and Manufacturing Engineering Conference and Exposition in Philadelphia, PA, May 17-20. He succeeds **Carl P. Isso**, Senior Engineer with Westinghouse Electric Corp., R&D, Pittsburgh, PA. Mr. Lawson has been a member of AFP/SME since 1976 and has served as Chairman of the Radiation Curing Division prior to his election to the Board of Directors in 1980.

The Spencer Kellogg Division of Tectron Inc. has announced the appointment of **Charles A. Dougherty** to the position of Market Development Manager for Encapsulant and other Specialty Resins.

The appointment of **Irving J. Gerlach** and **Gary C. Warehime** as Sales Representatives was also announced by Spencer Kellogg. Mr. Gerlach will be based in the Cleveland, OH area and Mr. Warehime will relocate to the Cincinnati, OH headquarters.

# Future Shock!

age

65

years worked

40

retirement benefits

0

Many of the 50 million Americans who are covered by private pension plans think they'll automatically qualify for benefits when they reach retirement age.

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Every plan has requirements that must be met under the Employee Retirement Income Security Act. And the time to find out about those requirements is now—even if retirement is 30 years down the road.

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Pueblo, Colorado 81009

U.S. Department of Labor



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**J. Robert "Bob" English**, retired partner of R. T. Hopkins Co. in Atlanta, was honored as the Senior Statesman for 1982 by the Southern Society "for the many years of steadfast support he has given to the Society and the coatings industry." Presentation of the award, and Honorary Membership in the Society, were made at the annual Southern Society meeting on March 12, in Savannah, GA.



J. R. English

Mr. English joined the forerunner organization of the Southern Society in 1938. In 1949 he started "through the chairs" of what was then known as the Southern Paint and Varnish Production Club, serving as President in 1952. He then left the ranks of Active membership to become a supplier to the industry, but quickly became one of the Society's most loyal Associate Members. For years he was a fixture as Publicity Chairman for the Atlanta Section. Most recently he has served as Society Historian—a fitting office for a man who has been personally acquainted with every President of the Society since its inception.

Numerous as have been Mr. English's contributions in official capacities, few who know him would disagree that his greatest service has been as unofficial friend and supporter of the paint and coatings industry and its people. During his 28 years of calling throughout the South, while associated with R. T. Hopkins Co., there was none better. Many an individual and company turned to him and found help when it was needed.

Mr. English attended Georgia Tech from 1931 to 1936, and entered the paint industry while still in school, as a co-op student in chemical engineering. He continued to work in formulation and production of paints until World War II, including involvement in development of the original reflectorized traffic paints. During the war he switched to explosives manufacturing for a few years, but returned to coatings in 1945 as Executive Vice President of Leland Moore Paint & Oil Co., of Charleston, S.C. There he also served on the War Production Board, Civilian Production Administration, and Office of Price Administration. During his "second career" as a supplier, he was associated with the universally recognized "Dean" of manufacturers' representatives in the industry, R. T. Hopkins, ably representing several prominent raw material manufacturers.

Hercules Incorporated, Wilmington, DE, has announced three assignments in its Southern Region. **Dale L. KenKnight** joins the Atlanta, GA office as a Technical Representative for Water Soluble Polymers and Coatings. Promoted to Senior Technical Sales Representative for Polypropylene Films in the Dallas, TX area is **Robert A. Adler**. **Grier C. "Bo" Bovard** was named Technical Sales Representative for Paper Chemicals at the Shreveport, LA office.

## Obituary

**Dr. Vincent C. Vesce**, Research Director for Harmon Colors Corp., died on January 27. He delivered the Joseph J. Mattiello Memorial Lecture in 1959 at the Federation of Societies for Coatings Technology's 37th Annual Meeting in Atlantic City, NJ. His paper was entitled "Exposure Studies of Organic Pigments in Paint Systems."

Dr. Vesce had been responsible for an impressive number of achievements in organic pigment chemistry and technology. Among these are the commercial production of durable maroon and red organic pigments for the automotive industry and the first preparation of transparent colloidal iron pigments which make possible iridescent metallic finishes. The commercialization of thioindigo pigments in the United States had been an exclusive development of the Harmon laboratories under Dr. Vesce's direction. He developed the first commercial nonfloculating phthalocyanine blue and pioneered in the production of soft textured, easily dispersed pigments.

In recent years, Dr. Vesce's work was directed toward the preparation of new light fast pigments suitable for pastel coatings and in the preparation of special dispersions for specific industries. His outstanding paper on "Vivid Light Fast Organic Pigments" was published as Part 2 of the June 1956 *Official Digest*.

Dr. Vesce was Chairman of Sub-Group VII (Red Pigments), Sub-Committee XV (Pigments) of ASTM, Committee D-1 on Paint and Related Coatings and Materials; a Fellow and charter member of the American Institute of Chemists; a Fellow of the New York Microscopical Society; and a member of the American Chemical Society, the Inter-Society Color Council, and the Chemists' Club.

## Polyester Coating Resin

A revised brochure featuring polyester coating resins is now available. The literature lists the physical characteristics of 16 resins including a series of high molecular weight, high solids, and powder resins available in solid and/or liquid form. Recommended usage in coil, can, container, automotive, and general industrial coatings is discussed, along with formulations and outstanding properties of these polyester coating resins. For a copy of the brochure, contact Kay-Fries, Inc., 200 Summit Ave., Montvale, NJ 07645.

## Color Measurement

A 46-page booklet entitled, "The Science and Technology of Appearance Measurement," provides the novice with an introduction to the basic concepts of appearance measurement science and technology and clarifies the need for appearance measurement information in business and industry. The publication offers a brief definition of appearance measurement and a description of its impact on the manufacturing process and the goods that are a part of our daily lives. *Included in the booklet are sections on:* the interaction of objects and materials with light; the geometric and chromatic attributes of appearance; an explanation of color measurement scales; instrument classifications; specimen selection, preparation, and presentation; and the application of appearance science. Featured is a glossary of words and terms useful in the field of appearance measurement. For additional information, contact D. Miner, Hunter Associates Laboratory, Inc., 11495 Sunset Hills Rd., Reston, VA 22090.

## Dimer Acids

A detailed monograph providing virtually all the information of industry interest on dimer acids, polymerized acids used in many different chemical applications, is now available. Offered as a convenient hardbound reference booklet, the 112-page text contains chapters on the general characteristics of dimer acids, their structure and properties, chemical reactions, and commercial applications of these chemicals and their derivative. Each chapter is documented with extensive references. Free copies of the monograph may be obtained from the Humko Chemical Div., Witco Chemical Corp., P.O. Box 125, Memphis, TN 38101.

## Acrylic Resins

Literature has been published which features two new hydroxyl functional acrylic resins for curing with polyisocyanates. Uses and advantages are described. Also, a new thermosetting acrylic copolymer solution for combination with melamine resins is described in the literature. For more information, contact Kunststoffsabrik Synthese B.V., Ringersweg 5, 4612 PR Bergen OP Zoom, Holland.

## Infrared Spectrophotometer

The totally new microprocessor controlled Infrared Spectrophotometer, Model 983, which offers the best in terms of overall performance, versatility, and low noise operation, is the subject of recent literature. *Featured applications include in organic chemistry research; infrared microsampling; biological and forensic chemistry fields; the analysis of individual components in pharmaceutical mixtures; accurate difference spectroscopy; measurement of difficult samples that transmit low energy; and measurements on traditionally difficult samples such as dilute aqueous solutions.* Data list instrumental features and performance characteristics. For further information, contact the Perkin-Elmer Corp., Main Ave., Mail Station 12, Norwalk, CT 06856.

## Surfactant

*Product information is available on a new surfactant that co-polymerizes with monomers in emulsion polymerization.* Called Trem LF-40, it offers improved water resistance and low foam characteristics for emulsion polymers commonly used in coatings production. For information, contact Frank B. Hawkinson, Process Chemicals Div., Diamond Shamrock Corp., 350 Mt. Kemble Ave., Morristown, NJ 07960.

## Abstract Newsletter

A weekly abstract newsletter is available which covers such areas as adhesives and sealants; coatings, colorants, and finishes; corrosion and corrosion inhibition; elastomers; materials degradation and fouling; and plastics. For more information, contact the U.S. Department of Commerce, National Technical Information Service, 5285 Port Royal Rd., Springfield, VA 22161.

## Laboratory Supplies

Interex Corp. has announced the availability of its 1982 Laboratory Supplies Catalog. Consisting of 100 pages, the catalog has been extensively revised but retains its easy-to-find format. To obtain a copy, write Interex Corp., 3 Strathmore Rd., Natick, MA 01760.

## Coating Resins

*The Industrial and Fine Chemicals Division of Degussa AG, Frankfurt am Main, has published a reprint of its booklet "Degadur®—Methacrylate Resins for Jointless Floor and Wall Coatings."* This 16-page illustrated booklet describes the cold-hardening Degadur coating resins and their potential applications, along with the necessary processing equipment and ancillary materials needed. It also quotes the durability values for fully-hardened coatings when exposed to more than 100 possible contact substances. The booklet is available in English, German, or French. For a copy, contact Degussa AG, Public Relations Dept., Postfach 11 05 33, D-600 Frankfurt 11.

## CIE Publication on Daylight Simulators

A report entitled, "A Method for Assessing the Quality of Daylight Simulators for Colorimetry," has been published by the Commission Internationale de l'Eclairage. The publication is the result of active cooperation between different countries and has been produced by the members of CIE Technical Committee 1.3 (Colorimetry). Featured is a method for evaluating the suitability of a test source as a simulator of CIE Standard Illuminant D<sub>55</sub>, D<sub>65</sub>, or D<sub>75</sub>. For each of these three standard illuminants, spectral-radiance-factor data are supplied for five pairs of nonfluorescent samples that are metameric matches. The colorimetric differences of the five pairs are computed for the test illuminant; the average of these differences is taken as the Visible Range Metamerism Index, and this is used as a measure of the quality of the test illuminant as a simulator for nonfluorescent samples. Information is also provided for fluorescent samples. *Copies of the document, CIE Publication #51, may be obtained post-paid at \$24 from Dr. Klaus D. Mielenx, Secretary, U.S. National Committee, CIE, c/o National Bureau of Standards, Washington, D.C. 20234.*

## Pressure Capsule for DSC

Literature features a new high pressure capsule for samples to be analyzed by differential scanning calorimetry. Described advantages of the new capsule include: capacity of withstanding internal pressures up to 2,175 psi; availability in either stainless steel or gold plated stainless steel varieties; and applications where high temperatures and/or higher pressures are required. For further information, contact the Perkin-Elmer Corp., Main Ave., Mail Station 12, Norwalk, CT 06856.

## Metric Balances

Information is available which discusses Port-O-Gram™ metric balances that are ideal for users who require weighing in grams only, feature fast readout, and are compact, lightweight, and battery-operated. Various uses and model features are described. Contact Ohaus Scale Corp., 29 Hanover Rd., Florham Park, NJ 07932.

## UV Stabilizer

A new liquid hindered amine ultraviolet light stabilizer, Tinuvin 292, for general purpose applications in automotive, industrial, trade sales, and radiation cured coatings is discussed in a recently published brochure. The stabilizer reportedly provides excellent protection against UV degradation and can be used to prevent gloss loss, discoloration, loss of film integrity, chalking, cracking, and checking. The product brochure is available from Mark McCusker, Additives Dept., Ciba-Geigy Corp., Ardsley, NY 10502.

## 'PIP' Slide Show

A new, 15-minute slide show which documents the initial year of "Picture It Painted" (PIP) and previews 1982 projects is now available from the National Paint and Coatings Association (NPCA). The 80-slide presentation is designed to provide paint industry groups with creative, innovative ways in which they can utilize the PIP campaign in 1982. The show brings viewers up-to-date on the variety of industry-wide support the program has and continues to receive. Topics covered include the increasing role of the National Decorating Products Association (NDPA) and the Painting and Decorating Contractors of America (PDCA); the local association involvement in the program; and the use of the media and promotional projects. The slide show package is available for \$155 which includes an 80-slide carousel tray and cassette type. Contact Kathy Haley, Consumer Products Div., NPCA, 1500 Rhode Island Ave., N.W., Washington, D.C. 20005.

## High Solids Coatings

A new 16-page brochure, entitled, "High Solids Resins," is now available which contains technical information on the 5000-series of high solids resins, including descriptions of seven distinct resins for air and force-dry coatings and six resins for baking-type coatings. Comprehensive data for each of the 5000-series resins includes descriptions and features, specifications, film properties, suggested uses, and methods of application. Each data category is presented in easy-to-read chart format containing all pertinent information to assist the coatings formulator in comparing the various resins. The brochure also contains a comprehensive formulating guide for each of the air- and force-dry resins and baking-type resins and recommends the area of application for which each resin is best suited. For a free copy of "High Solids Resins", write Marketing Dept., Cargill Inc., P.O. Box 5631, Minneapolis, MN 55440.

## Lubricant Spray

Literature is available which describes Molykote® 321R bonded lubricant spray, which solves lubrication stability problems in paint areas as well as reduces trolley body run-in-wear. Application procedures, advantages, and recommended uses are highlighted. For information, contact Dow Corning Corp., Midland, MI 48640.

## Atlas of Science

A new research and teaching aid that identifies and describes more than 100 highly active life sciences research areas primarily derived from biochemistry and molecular biology is now available from the Institute for Scientific Information. Entitled "The ISI Atlas of Science: Biochemistry and Molecular Biology, 1978/80," it provides a quick and easy introduction to the important subfields, or research fronts which comprise the cutting edge of biochemical research. Each of the 102 research areas covered in the atlas is introduced by a one-page "minireview" essay that traces its development. Each essay is followed by bibliography of core journal articles comprising the research area. A list of core or milestone papers and current papers published in 1980 are included. Also, the atlas contains a multi-colored, fold-out map which provides an over-all view of how the 102 research fronts are related. Expanded issues of the atlas are prepared annually and reflect newly identified research. The atlas is available at a cost of \$45 to individuals and \$90 to institutions from Marketing Services, ISI, 3501 Market St., University City Science Center, Philadelphia, PA 19104.

## Aluminum Stearates

Application information and property data for aluminum stearates, available in various compositional forms for coating formulations and other industrial uses, are provided in a recently published brochure. Details are given on their use in the manufacture of solvent-based protective and decorative coatings, where they can act to wet and suspend pigments, modify viscosity and rheology, prevent sag, impart flattening properties, control film penetration, and improve brushing characteristics. Other applications described include dry lubrication in metal processing, thickening of ink solvents, gelling of plasticizers, formation of greases, and water repellency in concrete mixes. Copies of Bulletin, "Witco Aluminum Stearates," #113, may be obtained from Witco Chemical Corp., Organics Div., 400 N. Michigan Ave., Chicago, IL 60611.

## CIE Proceedings

The Proceedings of the 19th Session of the International Commission on Illumination (CIE), held in Kyoto, Japan, August 21-28, 1979, have been published as CIE Publication No. 50. The publication includes the minutes of the Opening and Closing Plenary Meetings; an invited lecture; six general papers; the minutes of the 25 CIE Technical Committee meetings and three study groups; the minutes of four joint meetings; and 76 contributed papers dealing with research in the lighting field in different countries. Published every four years, the CIE Session Proceedings represent a valuable update of knowledge of light and lighting. Copies of the CIE Publication No. 50 may be obtained, postpaid at \$115, from the U.S. National Committee, CIE, c/o National Bureau of Standards, Washington, D.C. 20234.

## Electrocurtain

Literature is available which details an electrocurtain electron processing unit, used after drying and calendaring to provide a positive cure in the processing of magnetic tape and disks. By using the electrocurtain in the production line, the manufacturer experiences significant advantages such as: immediate and complete cure of the magnetic coating following calendaring, eliminating the need for long thermal storage to achieve cure; harder, more abrasion-resistant coating surface; elimination of pot life problems; reduced blocking of coating surface to back of tape particularly on large reels; improved yields and reduced cost; and increased line speeds. For further information, contact Bertram S. Quintal, Marketing Manager, Energy Sciences Inc., 8 Gill St, Woburn, MA 01801.



## Organic Pigment Identification Package

The Rensselaer Color Measurement Laboratory has developed the method of solution spectrophotometry for the identification of organic pigments. It is applicable to the analysis of paints, plastics, printing inks, works of art, or any other material colored with organic pigments. The method is simple and rapid, requires only small samples (down to 20-30 micrograms for semimicro analysis), and uses only readily available equipment. Mixtures of pigments can be analyzed, and the presence of resins, binders, or other uncolored additives generally does not interfere with the analysis.

To facilitate the use of the solution-spectrophotometry method, the Rensselaer Color Measurement Laboratory offers an *Organic Pigment Identification Package* consisting of (1) complete instructions, including analytical schemes and a curve-shape-index method of searching the reference library, and (2) the reference library, consisting of 416 log-absorbance plots of 166 organic pigments in the solvents recommended. In Package No. 1, the log-absorbance plots are computer plotted to be identical in size and scale (to facilitate identification by overlaying experimental plots) to those produced on General Electric-Hardy, Diano Hardy, Diano Match-Scan, Applied Color Systems Spectro Sensor, and other spectrophotometers utilizing the same scales. The cost of Package No. 1 is \$250.

Other packages will be developed on request, with library curves plotted to match the size and scales of other log-absorbance plots submitted with the request. Prices will vary upward from \$250 depending on demand, and will be quoted on request. Arrangements can also be made for users to access Rensselaer's computer data files for the library.

For a more complete description of the solution-spectrophotometry method for the identification of organic pigments, request a reprint of a recent publication from Dr. Fred W. Billmeyer, Jr., Department of Chemistry, Rensselaer Polytechnic Institute, Troy, NY 12181.

## Product Guide

A 43-page guide to the complete line of raw materials manufactured for industry by Goodyear's Chemical Div. is now available. The full-color brochure contains general product application descriptions as well as technical charts. Special sections are included on technical services, field offices, and assistance contacts. The booklet may be obtained from Goodyear Chemicals Data Center, P.O. Box 9115, Akron, OH 44305.

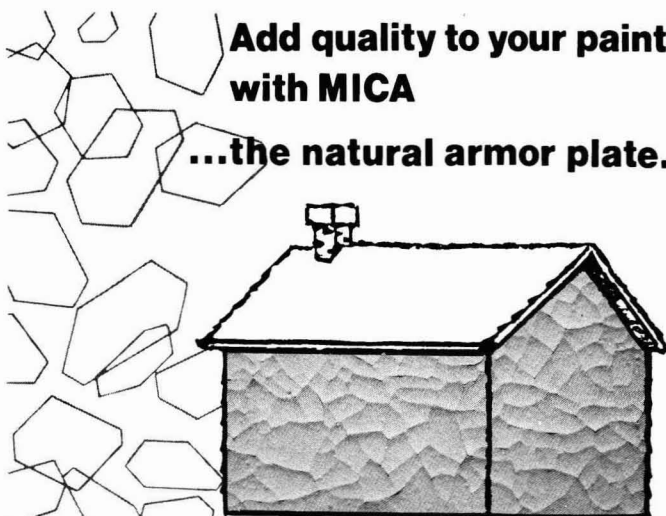
## Hi/Lo Mixing Systems

A new line of hoist-mounted hi/lo mixing systems, designed for special medium-viscosity applications, is discussed in recent literature. Features discussed include: dual offset shafts with one high speed dispersion blade and one low speed mixing blade; wide range of sizes and horsepower; and a variety of models. Uses in the coatings, sealants, adhesives, plastics, and chemical industries are noted. For additional information, contact Hockmeyer Equipment Corp., P.O. Box 113, 610 Worthington Ave., Harrison, NJ 07029.

## Functional Fillers

A 30-page brochure covering the properties and applications of Celite® diatomite and Micro-Cel® synthetic hydrous calcium silicate is available. The brochure is intended as a guide to better understanding and application of the materials and for that reason emphasizes the material's "functional" properties rather than its "filler" aspect. For a free copy of "Functional Fillers for Industrial Applications" (FF-396), contact Manville Corp., Service Center West, 1601 23rd St., Denver, CO 80216.

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## Forensic Analysis

"Forensic Examination and Analysis of Paints," newly published by the Scandinavian Paint and Printing Ink Research Institute and authored by Hans K. Raaschou Nielsen, deals with the use of paint as evidence in criminal investigations. The preface states:

"The present publication is mainly based on the practical experiences gained in the Scandinavian Paint and Printing Ink Research Institute through more than 15 years of close co-operation with the Technical Department of the Danish Police in the examination and analysis of paint samples discovered in connection with bank robberies and other types of break-in, murders, rape, insurance frauds and traffic accidents (including collisions at sea). The knowledge gained from practical cases has been supplemented with discussions on analysis in forensic laboratories in Europe, the United States of America, and Canada, and a study of more than 200 articles and reports on forensic analysis received since 1965."

"The purpose of this publication is to serve as a reference book in forensic laboratories and, in addition, to be used in the training of personnel in charge of criminal investigations to instruct them on the possibilities inherent in the new instrumental methods which make the analysis of minute paint particles possible."

To acquire this interesting and useful monograph, write to the Scandinavian Paint and Printing Ink Research Institute, Agern Alle 3, 2970 Hørsholm, Denmark.

## Rotary Vibrators

Six new rotary electric vibrators are discussed in recently published literature. Described features of the new line, which replaces 16 obsoleted units, are detailed and include: new dual phase 50/60 Hz. electric motor; new malleable or ductile iron castings with an improved food design; new motor service factor rating of 1.15 on 60 Hz.; new eccentric weight design; and a new, larger internal air circulatory system for cooler operation. For information, contact Rhino Sales Corp., 620 Andrews Ave., Kewanee, IL 61443.

## Powder Cleaners

A newly formulated series of economical alkaline powder cleaners developed for medium and heavy duty cleaning of ferrous and nonferrous metals is featured in recent literature. Recommended uses and advantages of the cleaners are highlighted. For full technical data, contact S.J. Damato, Hexcel Chemical Products, 205 Main St., P.O. Box 41400, Lodi, NJ 07644.

## Acrylic Copolymer

Product information is available which describes a new, fast dry, water-borne acrylic copolymer for use in formulating low cost, one-coat finishes. The new polymer, NeoCryl A-625, can be formulated into economical coatings that exhibit high gloss and resist oils and anti-freeze without blistering or loss of adhesion. For information write to Michael Pezzuto, Polyvinyl Chemical Industries, 730 Main St., Wilmington, MA 01887.

## Pigment Dispersant

An anionic, acrylic-based dispersant that works with a variety of pigments in water-based coatings and slurry formulations has been introduced in recent literature. The uses of Nalco 2324 are described such as in the dispersion of titanium dioxide, extenders, zinc oxide, and other pigments; and in a wide range of formulas such as flat, semigloss, gloss, interior, exterior, acrylic, modified acrylic, and vinyl acetate polymers. Literature can be obtained from Nalco Chemical Co., Specialty Chemicals Div., 2901 Butterfield Rd., Oak Brook, IL 60521.

## Cavity Pump

Literature is available which describes a new, two-stage, open throat, auger feed, size 40 progressive cavity pump, which is capable of moving up to 19 tons of animal protein, with 16-18% solids content per day. The pump develops 140 PSI and a discharge volume of between 5-8 gallons per minute. For information, contact Rick Strait, Netzsch, Inc., 119 Pickering Way, P.O. Box 453, Lionville, PA 19353.

## Black Colorant

A recently developed "1805 Black" colorant which improves maturation stability and color uniformity in low-profile SMC/BMC/TMC systems is featured in literature. Advantages, uses, and properties of 1805 Black are featured. Further information is available from Pigment Dispersions, Inc., 54 Kellogg Court, Edison, NJ 08818.

## Thickness Gauge

Literature is available which introduces a new analog coating thickness gauge. Described are the features of the three types of Model 707 which include: "F"—for measurement of nonferrous coatings applied to ferrous; "N"—nonconductive coatings applied to nonferrous substrates; and "F/N"—a combination of both types. For more information, contact Frank Rueter, Vice-President, Marketing, Zor-elco Ltd., P.O. Box 25468, Dept. J.26, Cleveland, OH 44125.

## Light Monitor

A recently introduced integrating radiometer designed for continuous measurement of solar radiation in the ultraviolet and/or visible regions of the spectrum, is the subject of new literature. Uses, features, and advantages of Model LM-3 Light Monitor are discussed. For bulletin #1405, write Atlas Electric Devices Co., Chicago, IL 60613.

## PRA Projects

In two recent Paint Research Association projects, instrumental methods were developed which are useful for optimizing the formulation of powder coatings and ensuring quality control of their manufacture. The first method is a new instrument devised by the PRA and the second is an application of an existing technique. The PRA tribometric (sliding plate) viscosimeter was devised to measure the viscosity of powder coatings through the liquid melt state to the onset of the curing reaction. Differential thermal analyzers (DTA) have been available and measure the progress of reactions which absorb or release heat. The PRA work showed that DTA can readily establish the percentage cure obtained after different times at various stoving temperatures. Information concerning these two projects is available from the PRA, Waldegrave Rd., Teddington, Middlesex TW11 8LD, England.

## Metering Pumps

Literature has been published which features FL cam-and-spring drive metering pumps that achieve accurate metering at very low flow rates whether used with plunger, diaphragm or bellows heads. Descriptions of the pumps' operational abilities and features are provided. For information, contact Wolfgang Gellrich, American Lewa, Inc., 11 Mercer Rd., Natick, MA 01760.

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# Coming Events

## FEDERATION MEETINGS

(Apr. 29–30)—Spring Meetings. Society Officers on 29th; Board of Directors on 30th. Lenox Hotel, Boston, MA. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, PA 19107).

(Nov. 3–5)—60th Annual Meeting and 47th Paint Industries' Show. Sheraton Washington Hotel, Washington, D.C. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, PA 19107).

1983

(Oct. 12–14)—61st Annual Meeting and 48th Paint Industries' Show. Queen Elizabeth Hotel, Montreal, Quebec, Canada. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, PA 19107).

## SPECIAL SOCIETY MEETINGS

(Apr. 20–21)—Chicago Society, SYMCO '82. "Back to Basics." Fountain Blue Restaurant, Des Plaines, IL. (Ray Cziczko, Glidden Coatings & Resins, 1855 N. LeClaire Ave., Chicago, IL 60639).

(Apr. 21–23)—Southwestern Paint Convention. Shamrock Hilton Hotel, Houston, TX. (Pete Schaff, Houston Solvents Co., 1010 W. Loop North, Houston, TX 77055).

(May 3)—"Modern Dispersion Technology" Seminar sponsored by the Technical Committee of the Philadelphia Society for Coatings Technology. Stadium Hilton Inn, Philadelphia, PA. (Phil Reitano, Kay-Fries, Inc., 10 Link Dr., Rockleigh, NJ 07647 or Robert Sonntag, Superior Varnish & Drier Co., P.O. Box 1310, Merchantville, NJ 08109).

(May 4)—Detroit Society for Coatings Technology 7th Annual FOCUS Conference, "New Frontiers in Coatings Applications." Management Education Center, Troy, MI. (Pete Burnett, Detroit Society, 765 Dellwood Dr., Ann Arbor, MI 48103).

(May 6–8)—Pacific Northwest Society. Annual Symposium. Bayshore Inn, Vancouver, B.C. (Barry Lamb, Harrisons & Crossfield, Ltd., 810 Derwent Way, New Westminster, B.C. V3M 5R1).

(May 19–20)—New England Society Coatings Tech Expo '82. Sheraton Inn, Boxborough, MA. (Robert Marderosian, Chairman, c/o Dame Assoc., 51 Church St., Boston, MA 02116).

(June 11–12)—Joint meeting of Kansas City and St. Louis Societies on "Instrumentation for Quality Coatings in the Eighties." Hilton Plaza Hotel, Kansas City, MO.

(Oct. 5)—Cleveland Society for Coatings Technology Manufacturing Committee Symposium on "The Use of Computers in the Manufacture of Coatings." Cleveland Engineering and Scientific Societies Bldg., Cleveland, OH. (Chairman Charles K. Beck, Premier Industrial Corp., 4415 Evelid Ave., Cleveland, OH 44103).

1983

(Feb. 23–25)—16th Biennial Western Coatings Societies' Symposium and Show. Hyatt Regency, San Francisco, CA. (Ted Favata, Chairman, Triangle Coatings Co., 2222 Third St., Berkeley, CA 94710).

## OTHER ORGANIZATIONS

(Apr. 19–20)—Inter-Society Color Council. Annual meeting. Sheraton Center Hotel, Charlotte, N.C. (Fred Billmeyer, Jr., Rensselaer Polytechnic Institute, Troy, NY 12181).

(Apr. 19–20)—22nd Annual Symposium of the Washington Paint Technical Group. Marriott Twin Bridges Motel, Washington, D.C. (John Montgomery, Secretary, Washington Paint Technical Group, P.O. Box 12025, Washington, D.C. 20005).

(Apr. 19–23)—"Printing Inks and Printing Processes" Short Course. Lehigh University, Bethlehem, PA. (Dr. Mohamed S. El-Aasser, Dept. of Chemical Engineering, Sinclair Lab #7, Lehigh University, Bethlehem, PA 18015).

(Apr. 21–28)—Hanover Fair '82 for Surface Treatment and Coatings Technology Equipment and Materials. Hanover, West Germany. (The Hanover Fairs Information Center, P.O. Box 338, Whitehouse, NJ 08888).

(Apr. 27–29)—Oil & Colour Chemists' Association's 34th Annual Exhibition, "International Forum for the Surface Coatings Industries." Cunard International Hotel, Hammersmith, London, England. (R.H. Hamblin, OCCA, Priory House, 967 Harrow Rd., Wembley, Middlesex, HA0 2SF).

(Apr. 28)—Symposium on Color in Protective Coatings sponsored by the Chemical Institute of Canada, Protective Coatings Division. Montreal and Toronto. (D.S. Wiersma, Celanese Canada, Inc., 2 Robert Speck Pkwy., Suite 900, Mississauga, Ont., L4Z 1H8 Canada).

(May 2–5)—National Coil Coaters Association's Annual Meeting. Marriott's Camelback Inn, Scottsdale, AZ. (Don White, NCCA, 1900 Arch St., Philadelphia, PA 19103).

(May 2–6)—American Oil Chemists' Society 73rd Annual Meeting. Sheraton Centre, Toronto, Canada. (James Lyon, Executive Director, AOCs, 508 S. Sixth St., Champaign, IL 61820).

(May 3–5)—"Composite Materials—Interface, Structure and Performances" Course. State University of New York, New Paltz, NY. (Dr. Angelos V. Patsis, SUNY-New Paltz, Chemistry Dept., New Paltz, NY 12561).

(May 4–6)—"Industrial Market Research for the Chemical Industry" Short Course. Raphael Hotel, Chicago, IL. (Norma Fleming, Senior Coordinator, Arts & Sciences Continuing Education, University of Missouri-Rolla, Rolla, MO 65401).

(May 6–7)—NPCA Conference for Small Paint Manufacturers, "Problem Sharing/Problem Solving." Crown Center Hotel, Kansas City, KS. (Karen Welch, National Paint and Coatings Association, 1500 Rhode Island Ave., N.W., Washington, D.C. 20005).

(May 9–14)—XVth Congress of FATIPEC, Palais des Congres, Liege, Belgium. (Destree-Congres, 10 rue du Coggege St. Michael, 1150 Brussels, Belgium).

(May 11–13)—Powder & Bulk Solids Conference/Exhibition. O'Hare Exposition Center, Rosemont IL. (Cahners Exposition Group, 22 W. Adams St., Chicago, IL 60606).

(May 17–21)—"Dispersion of Pigments and Resins in Fluid Media" Short Course. Kent State University, Kent, OH. (Carl J. Knauss, Chemistry Dept., Kent State University, Kent, OH 44242).

(May 18)—"Industrial Paints and Painting Methods" Workshop. Philadelphia Civic Center, Philadelphia, PA. (Susan Buhr, Technical Activities Dept., Society of Manufacturing Engineers, One SME Dr., P.O. Box 930, Dearborn, MI 48128).

(May 18–20)—"Estimating Workshop for Painting Contractors." Villa Hotel, San Mateo, CA. (Norma R. Fleming, UMR, Rolla, MO 65401).

(May 24–27)—"Basic Microcomputer Programming for Coatings Manufacturers" Short Course. University of Missouri-Rolla, Rolla, MO. (Norma R. Fleming, UMR, Rolla, MO 65401).

(May 25)—"The Influence of Associative Thickeners and Rheology on Coatings Performance" Symposium. Fountain Blue Restaurant, Des Plaines, IL. (Professor J.E. Glass, North Dakota State University, Polymers & Coatings, Dunbar Hall, Fargo, ND 58105).

(May 25-27)—"Colorimetry and the Eye" Short Course. Rensselaer Polytechnic Institute, Troy, NY. (Office of Continuing Studies, Rensselaer Polytechnic Institute, Troy, NY 12181).

(June 7-11)—"Adhesion Principles and Practice for Coatings and Polymer Scientists" Short Course. Kent State University, Kent, OH. (Carl J. Knauss, Chemistry Dept., Kent State University, Kent, OH 44242).

(June 7-11)—"Advances in Emulsion Polymerization and Latex Technology" Short Course. Lehigh University, Bethlehem, PA. (Dr. Mohamed S. El-Aasser, Dept. of Chemical Engineering, Sinclair Lab #7, Lehigh University, Bethlehem, PA 18015).

(June 7-11)—"Surface Modification, Cleaning, and Adhesion" Short Course. Soderkopings Brunn, Sweden. (Course Registration Office, Anita Hall, Gustav Adolfsgatan 9, S-582 20 Linköping, Sweden).

(June 7-18)—"Coatings Science" Short Course. North Dakota State University, Fargo, ND. (Dr. Zeno Wicks, Jr., NDSU, Polymers & Coatings, Fargo, ND 58105).

(June 13-16)—Dry Color Manufacturers' Association Annual Meeting. The Greenbrier, White Sulphur Springs, WV. (P.J. Lehr, DCMA, Suite 100, 1117 N. 19th St., Arlington, VA 22209).

(June 20-23)—American Society for Testing and Materials Committee D-1 Meeting on Paint and Related Coatings and Materials. Sheraton Center, Toronto, Canada. (Jane M. Turner, ASTM, 1916 Race St., Philadelphia, PA 19103).

(June 20-25)—75th Air Pollution Control Association's Annual Meeting and Exhibition. The Rivergate Convention Center, New Orleans, LA. (APCA, P.O. Box 2861, Pittsburgh, PA 15230).

(June 21-25)—"Applied Rheology for Industrial Chemists" Short Course. Kent State University, Kent, OH. (Carl J. Knauss, Chemistry Dept., Kent State University, Kent, OH 44242).

(June 21-25)—"High Solids Coatings" Short Course. North

Dakota State University, Fargo, ND. (Dr. Zeno Wicks, Jr., NDSU, Polymers & Coatings, Fargo, ND 58105).

(June 28-30)—"Organic Coatings Technology" Short Course. Center for Professional Advancement's Academic Center, East Brunswick, NJ. (Center for Professional Advancement, Dept. NR., P.O. Box H, East Brunswick, NJ 08816).

(June 28-July 8)—"Polymer Colloids" Short Course sponsored by NATO Advanced Study Institute. University of Bristol, United Kingdom. (Dr. Gary W. Poehlein, School of Chemical Engineering, Georgia Institute of Technology, Atlanta, GA 30332).

(June 29-July 7)—20th Session of the International Commission on Illumination. National Philharmony Bldg., Warsaw, Poland. (U.S. National Committee, CIE, c/o National Bureau of Standards, Washington, D.C. 20034).

(July 12-16)—8th International Conference on Organic Coatings, Science and Technology. Athens, Greece. (Angelos V. Patsis, Coykeydall Science Bldg., State University of New York, New Paltz, NY 12561).

(July 12-16)—International Union of Pure and Applied Chemists' Macromolecular Symposium. Amherst, MA. (James C.W. Chien, Dept. of Polymer Science & Engineering, University of Massachusetts, Amherst, MA 01003).

(Aug. 2-20)—Battelle's International Program in Productive R&D Management. Columbus, OH. (Dr. William D. Hitt, Battelle's Columbus Laboratories, 505 King Ave., Columbus, OH 43201).

(Aug. 9-13)—"Introductory Short Course on Composition of Coatings." University of Missouri-Rolla, Rolla, MO. (Norma Fleming, Senior Coordinator, Arts & Sciences Continuing Education, UMR, Rolla, MO 65401).

(Aug. 16-20)—Fifth Annual Short Course on "Advances in Emulsion Polymerization and Latex Technology." Schatzalp Berghotel, Davos, Switzerland. (Dr. Gary W. Poehlein, School of Chemical Engineering, Georgia Institute of Technology, Atlanta, GA 30332).

(Aug. 16-20)—"Physical Testing of Paints and Coatings" Short Course. University of Missouri-Rolla, Rolla, MO. (Norma R. Fleming, UMR, Rolla, MO 65401).

(Aug. 30-Sept. 3)—"Films and Coatings for Technology" Short Course. Davos Congress Center, Davos, Switzerland. (Course Registration Office, Anita Hall, Gustav Adolfsgatan 9, S-582 20 Linköping, Sweden).

(Sept. 12-17)—American Chemical Society's 184th National Meeting. Kansas City, MO. (A.T. Winstead, ACS, 1155 Sixteenth St., N.W. Washington, D.C. 20036).

(Sept. 13-17)—"Introduction to Paint Formulation" Short Course. University of Missouri-Rolla, Rolla, MO. (Norma Fleming, Senior Coordinator, Arts & Sciences Continuing Education, UMR, Rolla, MO 65401).

(Sept. 18-21)—Canadian Paint and Coatings Association. 70th Annual Convention. Four Seasons Hotel, Toronto, Ont. (R. Murry, CPCA, 515 St. Catherine St. W., Montreal, Que., Canada H3B 1B4).

(Sept. 20-24)—"Advanced Paint Formulation—Industrial" Short Course. University of Missouri-Rolla, Rolla, MO. (Norma Fleming, Senior Coordinator, Arts & Sciences Continuing Education, UMR, Rolla, MO 65401).

(Sept. 21-23)—"Radiation Curing VI" Conference and Exhibition sponsored by the Association for Finishing Processes of the Society of Manufacturing Engineers. Ramada O'Hare Inn, Des Plaines, IL. (Susan Buhr, Technical Activities Dept., SME, One SME Dr., P.O. Box 930, Dearborn, MI 48128).

(Sept. 29-Oct. 1)—"Surfair '82"—Conference on surfaces' treatments in aeronautical and aerospace industries sponsored by *Surfaces* magazine. Cannes (French Riviera), France. (Thierry Delmotte, *Surfaces*, 46, Rue Ampere, 75017 Paris).

(Oct. 6-8)—"Production Planning and Inventory Management" Seminar. Colony Square Hotel, Atlanta, GA. (National Paint & Coatings Association's Meetings & Conventions Div., 1500 Rhode Island Ave., N.W., Washington, D.C. 20005).

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(Oct. 11-13)—10th Congress of the Federation of Scandinavian Paint and Varnish Technologists. Copenhagen, Denmark. (G. Christensen, Sadolin & Holmblad Ltd., Holmbladsgade 70, DK-2300, Copenhagen S, Denmark).

(Oct. 13-15)—"Fundamentals of Adhesion: Theory, Practice and Applications" Course. State University of New York, New Paltz, NY. (Dr. Angelos V. Patsis, SUNY-New Paltz, Chemistry Dept., New Paltz, NY 12561).

(Oct. 18)—"Fire Resistant Coatings: The Need for Standards" Symposium. Philadelphia, PA. (Symposium Chairman Dr. Morris Lieff, County College of Morris, Dover, NJ 07801).

(Oct. 26-27)—"New Coatings Systems for Bridges" World Congress. Holiday Inn LaGuardia, New York, NY. (Norma Fleming, Senior Coordinator, Arts & Sciences Continuing Education, University of Missouri-Rolla, Rolla, MO 65401).

(Oct. 31-Nov. 2)—"Women in Coatings—Meeting the Challenges" Seminar. Washington, D.C. (Norma Fleming, Senior Coordinator, Arts & Sciences Continuing Education, University of Missouri-Rolla, Rolla, MO 65401).

(Nov. 1-3)—National Paint and Coatings Association. 95th Annual Meeting. Washington Hilton Hotel, D.C. (Karen Bradley-Welch, NPCA, 1500 Rhode Island Ave. N.W., Washington, D.C. 20005).

(Nov. 9-10)—Electrocoat'82". Westin Hotel, Cincinnati, OH. (*Products Finishing Magazine*, 600 Main St., Cincinnati, OH 45202).

(Nov. 9-11)—"Refresher for Painting Contractors, Maintenance Engineers and Inspectors" Short Course. Sheraton St. Louis Hotel, St. Louis, MO. (Norma Fleming, Senior Coordinator, Arts & Sciences Continuing Education, University of Missouri-Rolla, Rolla, MO 65401).

(Nov. 12-14)—National Decorating Products Association 35th Annual Show. Superdome, New Orleans, LA. (Lillian Smysor, NDPA, 9334 Dielman Industrial Dr., St. Louis, MO 63132).

(Nov. 16-18)—"Estimating Workshop for Painting Contractors" Short Course. Sheraton St. Louis Hotel, St. Louis, MO. (Norma Fleming, Senior Coordinator, Arts & Sciences Continuing Education, University of Missouri-Rolla, Rolla, MO 65401).

(Nov. 29-Dec. 3)—"Principles of Industrial Coatings" Short Course. Sheraton St. Louis Hotel, St. Louis, MO. (Norma Fleming, Senior Coordinator, Arts & Sciences Continuing Education, University of Missouri-Rolla, Rolla, MO 65401).

(Dec. 7-9)—Fifth Annual Western Plastics Exposition. Long Beach Convention Center, Long Beach, CA. (Western Plastics Exposition, 1625 17th St., Unit 2, Santa Monica, CA 90404).

1983

(Jan. 23-27)—Semi-Annual Meeting of the American Society for Testing and Materials Committee D-1 on Paint and Related Coatings and Materials. Dutch Inn, Lake Buena Vista, FL. (ASTM, 1916 Race St., Philadelphia, PA 19103).

(Jan. 26)—"New Concepts for Coating Protection of Steel Structures" Symposium sponsored by ASTM Committee D-1 on Paint and Related Coatings and Materials and the Steel Structures Painting Council. Lake Buena Vista, FL. (Cochairman R.F. Wint, Hercules Incorporated, 910 Market St., Wilmington, DE 19899).

(June 15-18)—Oil & Colour Chemists' Association Biennial Conference on "The Efficient Use of Surface Coatings." Viking Hotel, York, England. (R.H. Hamblin, Director and Secretary, OCCA, Priory House, 967 Harrow Rd., Wembley, Middlesex, HAO 2SF, England).

(Sept. 28-Oct. 1)—Oil & Colour Chemists' Association's Silver Jubilee Convention and Exhibition. Southern Cross Hotel, Melbourne, Australia. (O.C.C.A.A., 1983 Pacific Coatings Convention, C/- Tioxide Australis Pty. Ltd., Private Bag 13, Ascot Vale, Victoria, 3032, Australia).

(Oct. 11-13)—"Finishing '83" sponsored by the Association for Finishing Processes of the Society of Manufacturing Engineers. Cincinnati Convention Center, Cincinnati, OH. (Susan Buhr, AFP/SME Administrator, One SME Dr., P.O. Box 930, Dearborn, MI 48128).

# Advertisers Index

AMERICAN CYANAMID CO. ....	20E-H
BURGESS PIGMENT CO. ....	71
ECLIPSE SYSTEMS, INC. ....	90
ENGLISH MICA CO. ....	87
GLIDDEN PIGMENTS DIV. OF SCM CORP. ....	Cover 3
HENKEL CORP. ....	15
HANS HEUBACH GMBH & CO. ....	17-20
INDUSMIN LTD. ....	25
S.C. JOHNSON & SONS, INC. ....	16
NL CHEMICALS/NL INDUSTRIES, INC. ....	9
PFIZER, INC., MPM DIV. ....	13
ROHM AND HAAS CO. ....	4-5, 10-11
SPENCER KELLOGG DIV., TEXTRON, INC. ....	Cover 4
TENNECO CHEMICALS, INC. ....	62
UNION CARBIDE CORP. ....	Cover 2, 1, 52
UNION PROCESS, INC. ....	76
R.T. VANDERBILT CO., INC. ....	2

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# 'Humbug' from Hillman

## Diary of a First Time Conventioneer

### Final Day

I awoke early today, fully rested, and so, proceeded to the Paint Show for a real educational experience amid the glamour of short-skirted, lovely girls. After studying the directory, I planned an agenda of booths to visit to provide me with some helpful information and to arrange for samples.

As I entered, the first thing I saw, though, was that darn golf game. I knew I couldn't waltz by without winning a flashlight or something, so, again, I stood up to the tee and went after a hole in one. No good! By this time, the girls recognized me and sneaked me back for another try. I was lucky this time—somehow the dark-haired girl's foot struck the ball, and lo and behold, the ball dropped into the hole. With the next attempt the same miracle happened, and I was awarded a prize. Of course, I didn't admonish the girl for getting in my way. She was too nice and had a great smile. Some fellow nearby had the nerve to say, "Why don't you date her? You're half way there already!" I wonder what he was getting at?

By now, I had to get down to some serious business. I headed for the booth, which I had spotted yesterday, that contained some very interesting information. After wandering down the wrong aisle three times, I finally found it only to be crowded with people, apparently interested in the same products. To my surprise I discovered the crowd to be participating in some sort of guessing contest—making sure their guesses were in before closing. When one of the reps at the booth heard that I was interested in their additives, he very graciously sat down with me and offered his help. After a long talk, in which he tried very hard to answer my questions, it turned out that he was not the resident expert on the product I needed. He proceeded to take my name and address and then I went on my way somewhat frustrated but a little flattered with all the attention I received.

In the remaining hour and a half, I did manage to get some new product information that I hope to put to use during the year. I left my name and address in so many booths, that my boss is sure to be impressed with all the mail I will be receiving.

As I was heading for the exit, one of my suppliers stopped me and asked if I would like a ticket for the Federation luncheon. I said "sure" and was happy I did. First I had a few drinks, being careful not to overdo because of my recent experiences. I sat down at a table with a bunch of really nice fellows. The food was O.K. and we had quite a lot of fun, telling jokes and stuff, but then, the program began.

Several individuals made short speeches, which made things not too bad, and then awards were presented. It was funny—there was an award for heckling because a nice little guy kept talking about the heckle award. He was playing some kind of game—a guessing game—like guessing who the winner is. The mystery recipient's long list of accomplishments was itemized—I knew it couldn't be me and so I lost interest.

Finally, the winner was revealed and called to the head table to receive his reward. As the fellow headed toward the table, he appeared pleasantly surprised. Maybe he didn't recognize himself. He was very nice though and didn't make a long speech.

The award presentation was followed by a very amusing and good speaker. We all enjoyed him, and all in all, it was a pleasant experience.

With the luncheon over, I headed back to the hotel, checked out, and bused back to the airport for the long trip home. As I look back, my first convention was quite wonderful. I expect that next year, it will be even better—now that I know the score.

•••

I was again pleased to have a note from our old friend who had a building named after him. How many of our readers could say that? The only thing I've had named after me is my mail box—and I have to share that with my wife. You know, of course, it is Past-President Milt Glaser, and here is a sampling of his collection.

The president of a coatings company asked his chemist a simple question—

"How much is 2 and 2?"

"Why, 4 of course," was the answer.

The president then posed the same question to one of his bright young engineers who replied, "Why, 4,000, of course."

When the president asked the same question of his chief accountant, the answer came back, "Well sir, how much do you want it to be?"

And from Sid Lauren, who, according to our Convention Diarist, got this year's award for heckling and is such a nice fellow, we received.

### Shape Up, Kid!

There was a mother who was having a hard time getting her son to go to school one morning.

"Nobody likes me at school," said the son, "The teachers don't and the kids don't. The superintendent wants to transfer me, the bus drivers hate me, the school board wants me to drop out, and the custodians have it in for me."

"You've got to go," insisted the mother, "You're healthy. You have a lot to learn. You've got something to offer others. You are a leader. Besides, you are forty-nine years old. You're the principal and you've got to go to school."

(From the *Nacogdoches Daily Sentinel*.)

John Bax has added to our long list of definitions of the much maligned consultants who apparently defy definitions.

"A man who knows 187 ways to make love, but knows no women."

"Someone who borrows your watch, tells you the time, and keeps the watch."

—Herb Hillman

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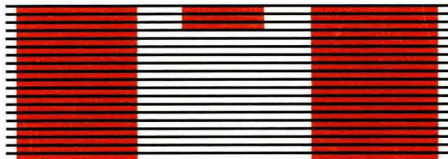
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