jct JOURNAL OF COATINGS TECHNOLOGY

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FEBRUARY 1979

# Reversibility of Yellowing Phenomenon in Linseed-Based Paints


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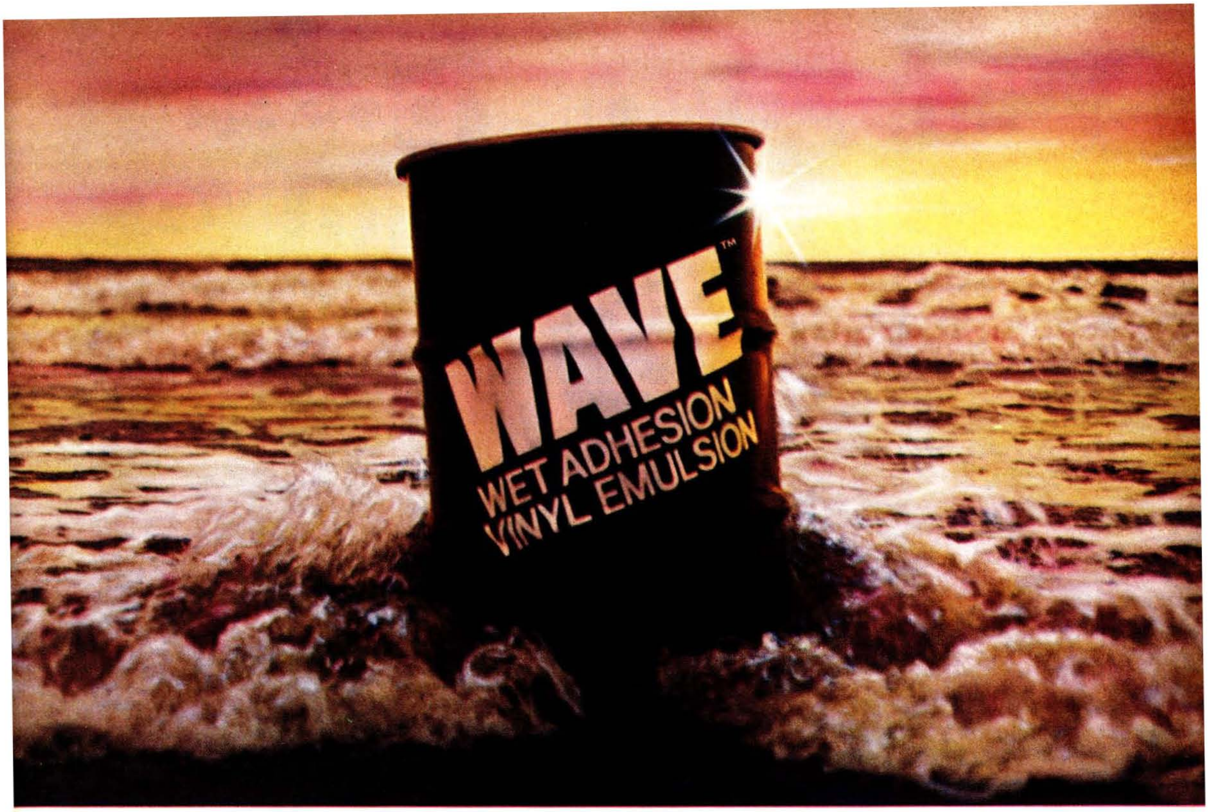
SEMI-GLOSS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FEBRUARY 1979

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Volume 51 Number 649

## Features

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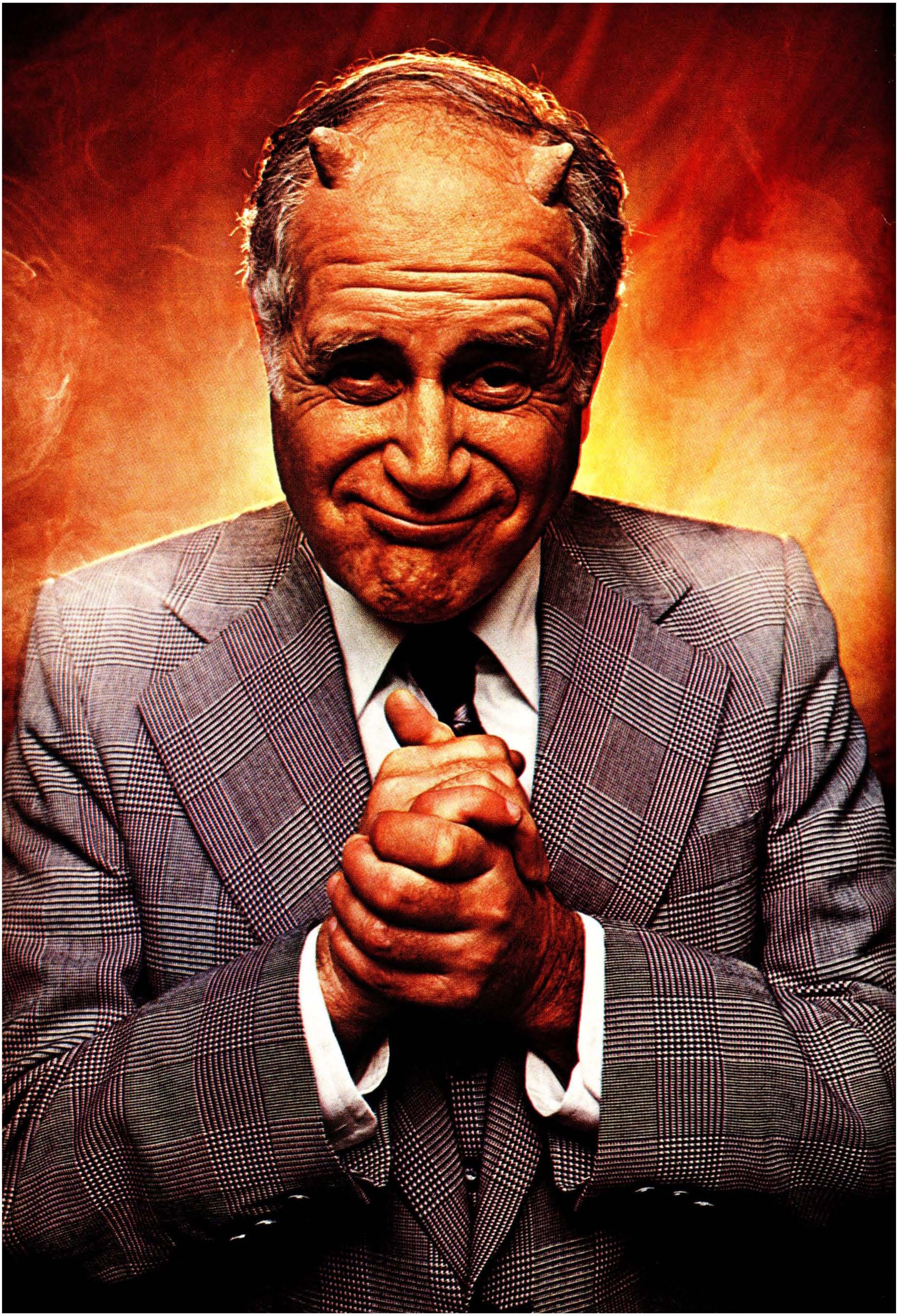

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# Comment

## Facing the Industry Through JPICC

A recent meeting notice from the Florida Paint and Coatings Association brings to mind an industry organization in which the Federation is privileged to be a member: the JPICC.

First, the Florida meeting. Called "Face the Paint Industry," it was a panel discussion with an *owner, production superintendent, technical director,* and sales director of a paint manufacturing company on one side. Facing them were a *painting contractor,* an architect, a *paint store dealer,* and an industrial paint user. The questions and answers, plus the resultant dialogue between manufacturer and purchaser must have been spirited and informative.

The *italics* tie into JPICC - the Joint Paint Industry Coordinating Committee - composed of the officers and top staff of the: National Decorating Products Association, St. Louis (paint store dealers); National Paint and Coatings Association, Washington, D.C. (paint company management); Painting and Decorating Contractors of America, Falls Church, Va. (painting contractors); and the Federation (production/technical).

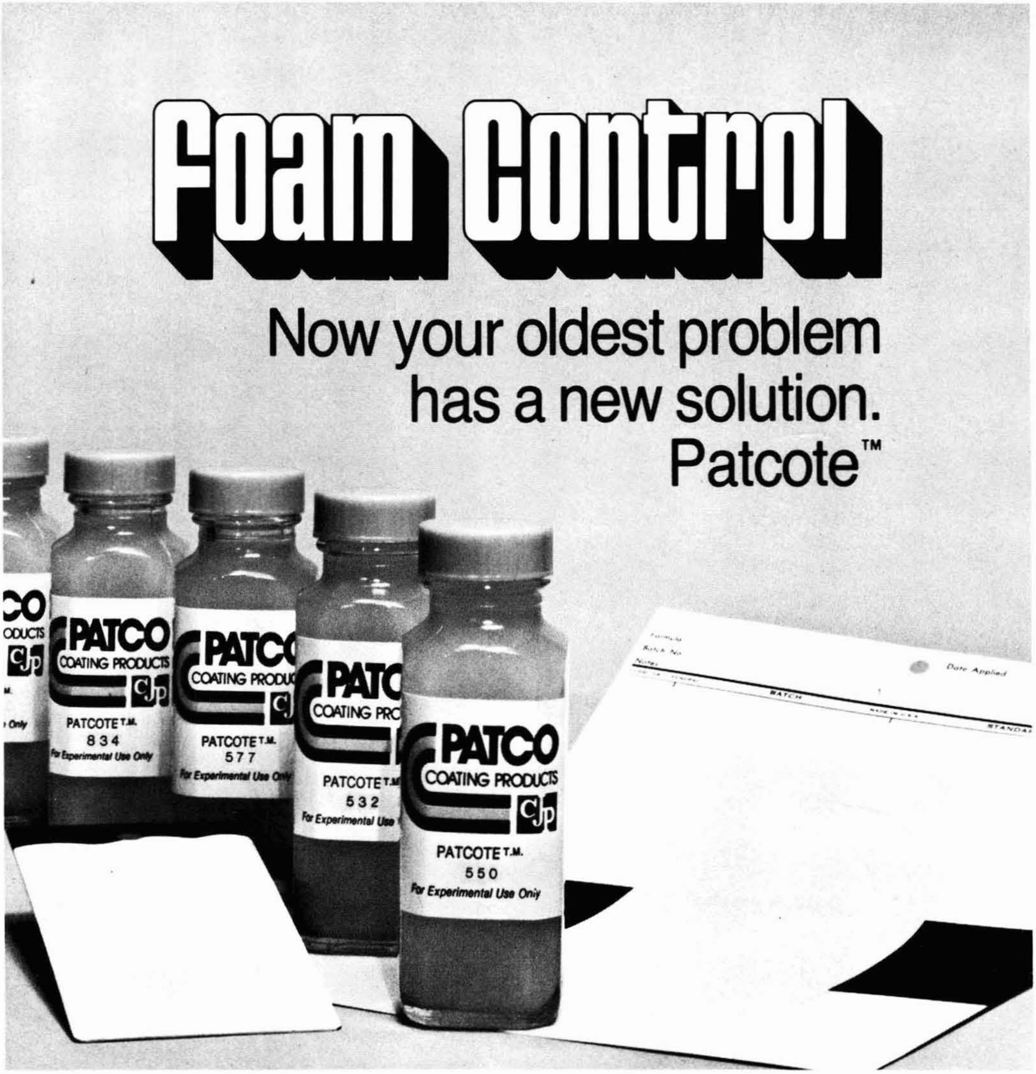
The JPICC meets twice yearly and each group uses the occasion to present an update on its activities since the last meeting. These reports from your counterparts in the coatings industry are always interesting and enlightening.

Getting together with representatives from other segments of the industry creates a better understanding and appreciation of each other's role, problems, and needs. The national JPICC, in which there is great rapport, is a good example.

It follows then that it should also be beneficial to organize JPICC-type discussion groups or informal meetings at the local level. Leading the way in the Federation is the St. Louis Society which schedules a "JPICC Night" once a year.

There should be more.

After all, we're all in the same paint pot together.—FJB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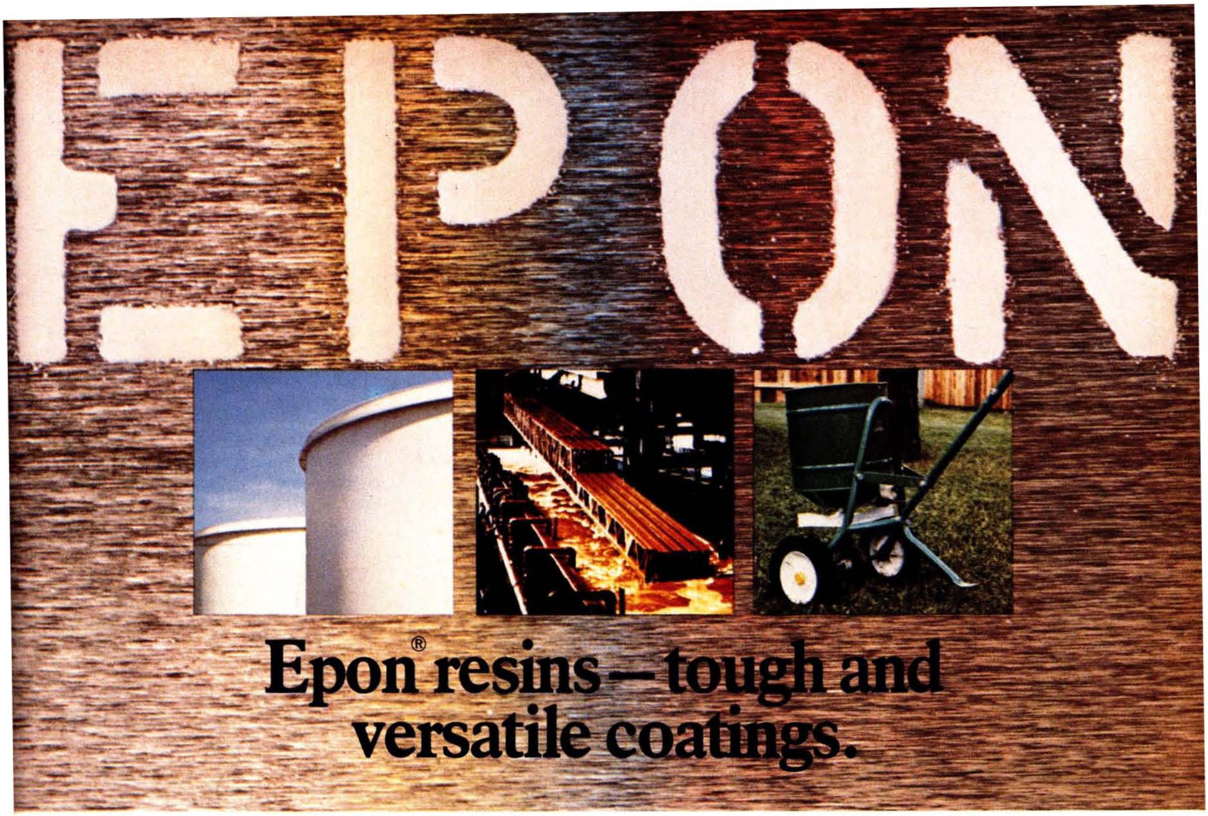
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## REVERSIBILITY OF YELLOWING PHENOMENON IN LINSEED-BASED PAINTS—H. Rakoff, F.L. Thomas, and L.E. Gast

Journal of Coatings Technology, 51, No. 649, 25 (Feb. 1979)

A commercial linseed oil paint, a commercial linseed alkyd paint, and three experimental oil paints (prepared with alkali-refined linseed oil, decolorized linseed oil, or pure trilinolenin as vehicle) were exposed to light, darkness, or to cycles of light and darkness to ascertain if yellowing is a reversible process. It was determined that linseed-based paints can be taken through several cycles of yellowing and bleaching. The extent and rate of yellowing in successive cycles depend on the nature of the paint. Linseed alkyd paints yellow at about the same rate and to about the same extent through each cycle; linseed oil paints yellow less and more slowly through successive cycles.

## PROCESSING INTERMEDIATES FOR HIGH-BUILD PAINTS—I. Kikic, R. Lapasin, G. Torriano, and A. Papo

Journal of Coatings Technology, 51, No. 649, 29 (Feb. 1979)

The rheological properties of a castor oil derivative/chlorinated rubber processing intermediate employed in the manufacture of chlorinated rubber high-build paints are investigated. Transient behavior and steady values are studied in steady flow using coaxial cylinder viscometers. Transient pattern depends on sample history and shear sequence. Steady values are not a unique function of the shear rate; oscillatory phenomena often superimpose themselves on initial transients at low shear rate. The complex rheological behavior is discussed and a structural interpretation is suggested. A master curve is obtained for a series of related intermediates of different castor oil content.

## SURVEY OF THE APPLICATIONS, PROPERTIES, AND TECHNOLOGY OF CROSSLINKING EMULSIONS. PART VI—J.R. Grawe and B.G. Bufkin

Journal of Coatings Technology, 51, No. 649, 34 (Feb. 1979)

This review summarizes most of the principal concepts which have been employed over the last ten years to develop thermosetting or crosslinking emulsions. The conceptual elements which have formed the foundation for this new generation of coatings are discussed in terms of the science, technology, properties, and applications of the emulsion systems which have been reported in the literature. This survey presents the advantages and limitations afforded by each of the functional monomers or polymer classes which have been investigated for the development of crosslinking emulsions. This paper concludes the topic of post-emulsified systems with a discussion on epoxy, epoxy ester, polyurethane, silicone, and phenolic-based emulsions and also briefly discusses such topics as precrosslinked emulsions, thermoset water-in-oil emulsions, nonaqueous dispersions, and other methods of application and curing.

## SELECTION OF STRAINING (NOMINAL FILTRATION) EQUIPMENT FOR COATINGS—A. Tankha

Journal of Coatings Technology, 51, No. 649, 68 (Feb. 1979)

Straining is the last unit operation in a chemical coatings facility before the coating is filled into containers for shipment. This article discusses filtration equipment and filter mesh size which optimize the filtration rate.

## VOLUMETRIC COMPUTER COLOR MATCHING—B.H. Todd

Journal of Coatings Technology, 51, No. 649, 71 (Feb. 1979)

The paper explains how conventional computer color matching (CCM) programs have been modified to yield data in volumetric units for greatly expanded flexibility and cost savings. Volumetric handling and dispensing of coloring materials are discussed in their application to a variety of laboratory and manufacturing situations common to the coatings industry, including the new innovation of totally automated factory tinting. The advantages and limitations of this approach to CCM are frankly discussed.

## DEVELOPMENT OF COMPUTER COLOR FORMULATION AND CONTROL—H.R. Davidson

Journal of Coatings Technology, 51, No. 649, 78 (Feb. 1979)

Eloquent testimony to the effectiveness of instrumental color matching has been given by the Symposium on Color and Appearance Instrumentation. Many suppliers, competing in the field, and many producers of colored materials came to evaluate the new equipment and methods. But we cannot claim complete success in all color formulation and control problems, so it is well to consider both the strong points and the weak points of our instrumentations.

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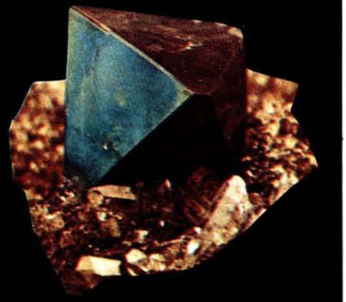
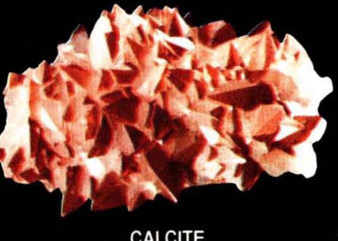


MAGNETITE
$Fe_3O_4$

CHROMITE
$FeCr_2O_4$



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# MMA Awards for Notable Achievements Won by Los Angeles and Baltimore Societies

The MMA Awards for notable achievements during 1978 were won by the Los Angeles and Baltimore Societies. Presentation was made at the Federation Annual Meeting in Chicago.

The Awards, established in 1975 by Materials Marketing Associates, a national marketing group of manufacturers' representatives, recognize notable achievements by Constituent Societies, other than Society papers presented at the Federation Annual Meeting.

There are three categories of awards, based on Society membership size, but it is not mandatory that an award be presented each year to any or all categories. Each category winner receives $350 in cash, plus a handsome, appropriately engraved plaque.

### Liaison Efforts On Behalf of Coatings Industry At Hearings of Regulatory Agencies

The Los Angeles Society was cited for its liaison efforts on behalf of the coatings industry at hearings of various regulatory agencies in California.

When the first draft of Rule 66 was published in January 1965 by the Los Angeles County Air Pollution Control District, its restrictions were completely impractical: it specified solvents that could be used — all were pure chemicals and most were not feasible because of cost and total potential supply. As a result, an inter-industry committee under the banner of the California Manufacturers Association was formed to negotiate a more workable rule, and a number of Los Angeles Society members were among the original committee participants who conducted experiments and were involved in subsequent negotiations that led to a rule that specified what could *not* be used — or, at least, what could not be emitted to the atmosphere, a key change. It was not only workable, it was more effective than the original proposal in reducing photochemically reactive emissions.

The Rule 66 Committee activities also established a precedent for cooperative development of air pollution legislation by government and industry. Rule 66 has undergone many revisions and is now known as Rule 442. At each step, industry has been consulted, and Los Angeles Society members have continued to be involved in these activities.

Most recent efforts have been directed toward helping shape realistic air pollution laws.

**Materials Marketing Associates Awards for notable achievements other than Society papers, were presented to Baltimore Society, Alex Chasan accepting (right), and Los Angeles Society, Bill Ellis accepting, by Awards Committee Chairman Don Fritz**

### Correlative Testing Program

The Baltimore Society won an award for the activities of its Correlative Testing Subcommittee.

This program was initiated to help with testing problems by correlating round-robin testing with cooperating laboratories. Members were solicited to determine testing instruments and equipment available, and as requests for testing were received, samples were distributed to the appropriate participants. Results were distributed to all who took part.

All results, tests, and sampling were on a "blind" basis so as to maintain complete confidentiality of requester and tester.

Among the services offered were: interpretation of acid value calibrations; confirming reflectance values; and checking of viscometers, weight-per-gallon devices, film applicators, weatherometers, abrasion testing equipment, and impact testers.

### Principles Governing Awards

The MMA Awards recognize notable achievements in the field of education, manufacturing and training procedures, technology, public service, and other achievements deemed proper and desirable by the Awards Committee.

Not eligible are Society papers offered for presentation at the Federation Annual Meeting. Although the Awards are to be presented at the Annual Meeting, it is not mandatory that they be presented to any or all categories each year.

The President of any Society wishing to enter the competition must send a letter of intent, no later than March 31, to the MMA Awards Committee Chairman, William T. Cochran, Bruning Paint Co., Inc., 601 S. Haven St., Baltimore, Md. 21224. A complete description of the Society activity to be considered in the competition must be submitted by the Society President to Chairman Cochran by August 31.

Materials Marketing Associates is composed of the following: Apco Industries Co., Ltd., Toronto, Ont.; George C. Brandt, Inc., Kansas City, Kan. and St. Paul, Minn.; The Cary Co., Chicago, Ill.; Dar-Tech, Inc., Cleveland, Ohio; E. T. Horn Co., Los Angeles, Calif.; Lukens Chemical Co., Boston, Mass.; Matteson-Ridolfi, Inc., Detroit, Mich.; McCullough & Benton, Inc., Atlanta, Ga.; Ribelin Distributors, Inc., Dallas, Texas; Shanahan's Limited, Vancouver, B.C.; Wm. B. Tabler Co., Inc., Louisville, Ky.; Van Horn, Metz & Co., Inc., Philadelphia, Pa.; Walsh & Associates, Inc., St. Louis, Mo.; and C. Withington Co., Pelham Manor, N.Y. and Rio Piedras, Puerto Rico.

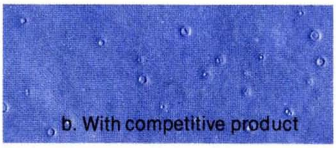
# How to Select the Right Antifoam for Water-Based Paints

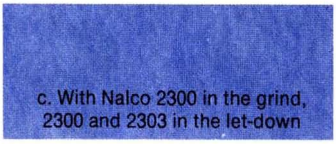
## Results are What Count

Below is just one example of the results you get when Nalco tackles a foam problem. Take a good look. Results are what count and what Nalco is committed to.

**"Vinyl Acrylic Terpolymer in an Interior Semi-gloss Paint"**


a. Control-no antifoam


b. With competitive product


c. With Nalco 2300 in the grind, 2300 and 2303 in the let-down

Nalco has a unique approach to the selection of antifoams. And the results we achieve are proof of its effectiveness.

## The Starting Point

Nalco starts from these basic premises.

What an Antifoam Should Do

1. It should prevent and destroy foam.
2. It should be persistent.
3. It should not adversely affect color acceptance, gloss, water sensitivity, or intercoat adhesion.
4. It should not cause surface defects such as crawling, pinholes, or fisheyes.

Nalco is not satisfied with an antifoam selection until it meets all four requirements and achieves the results you are looking for.

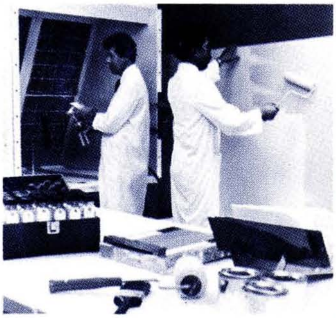
## A Systematic Approach

Nalco uses a systematic screening procedure to determine antifoam performance. And before Nalco recommends an antifoam for your coating, we thoroughly analyze its effect in your total system. We:

**Analyze Your Formulation** The surfactants, co-solvents, flow control additives, pH, pigments, and resin type in your formula are studied since they affect foam stability and antifoam performance.

**Analyze Your Manufacturing Procedures** High sheer dispersion phases, transfer, and filling operations cause foam and may require several antifoams for effectiveness.

**Analyze Your Application Procedures** Techniques involving recirculation and high shear, such as spray and flow, can cause persistency problems and are considered when solving the foam problem.

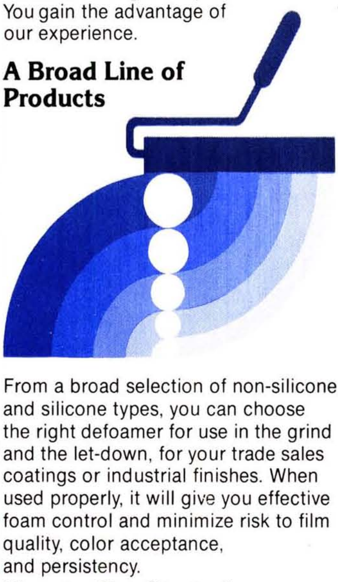


Nalco's tests are designed to be as similar to actual usage as possible. Our recommendations may include products for the grind and others for the let-down. And to make sure the recommendation is right, Nalco checks results with the actual lab manufacture of your paint whenever possible.

## A Bank of Knowledge

Nalco works directly with new resin technology and may have already screened the resin you are using. And there probably is not a foam problem in manufacturing or application we have not seen before. We are old hands at licking foam problems. You gain the advantage of our experience.

## A Broad Line of Products



From a broad selection of non-silicone and silicone types, you can choose the right defoamer for use in the grind and the let-down, for your trade sales coatings or industrial finishes. When used properly, it will give you effective foam control and minimize risk to film quality, color acceptance, and persistency.

## How to Get Started

If you are reformulating or developing a new water-based paint, give Nalco a call. Our paint experts will work with you from the start to develop the best foam control program possible. Just use our "hotline" number (312) 887-7500 ext. 1214. Or consult our literature. Write for bulletins K-8 and K-10.

**Nalco Chemical Company**
2901 Butterfield Road Oak Brook, IL 60521
(312) 887-7500

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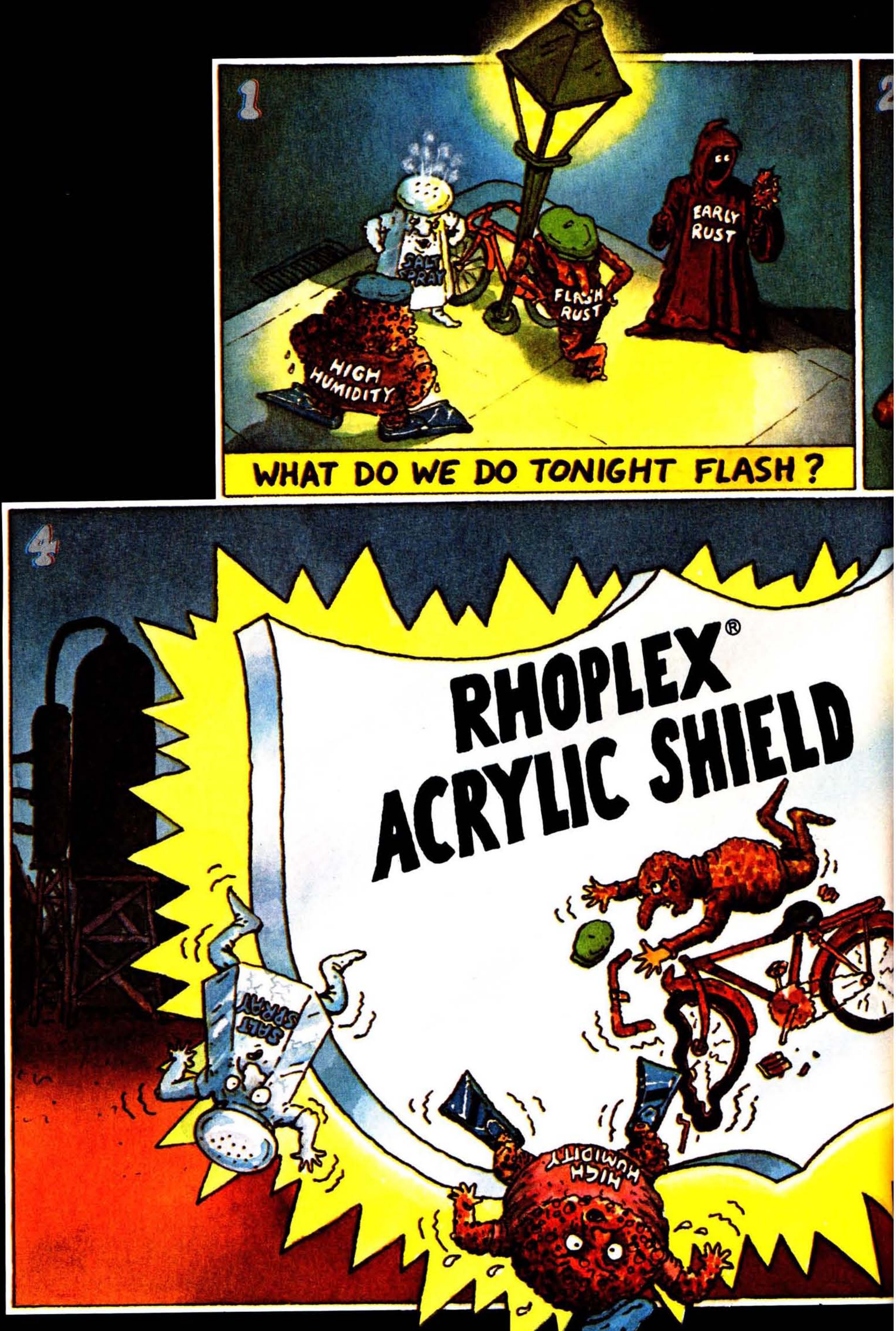
## RHOPLEX® MV-23 zaps salt spray, flash rust, early rust, high humidity, stains.

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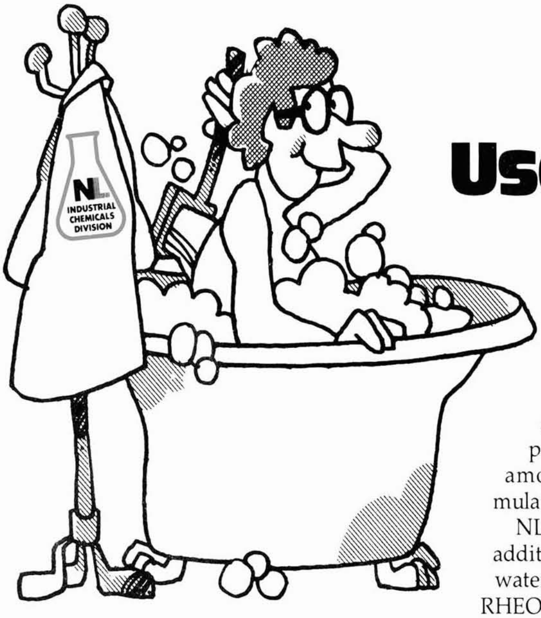
3
ATTACK!
EARLY RUST
HIGH
EARLY RUS
R. Brown



# Use water for bathing.

You can't produce a quality acrylic latex paint using excessive amounts of water. Yet, the incorporation of traditional latex paint thickeners ties up substantial amounts of water. Using predispersed slurries further restricts the amount of free water in your formulation.

NL's new RHEOLATE™ 1 rheological additive eliminates the need for excessive water in your paint formulation because RHEOLATE 1 requires no prehydration. With RHEOLATE 1 there are no powders to dissolve in water, no sticky gums to dilute. RHEOLATE 1 is ready to use just as we ship it to you. Simply pour it directly into your paint formula for efficient, reproducible thickening, batch after batch. And while RHEOLATE 1 is saving you time and money on production, it's also helping you make a better paint by giving you other tangible benefits.

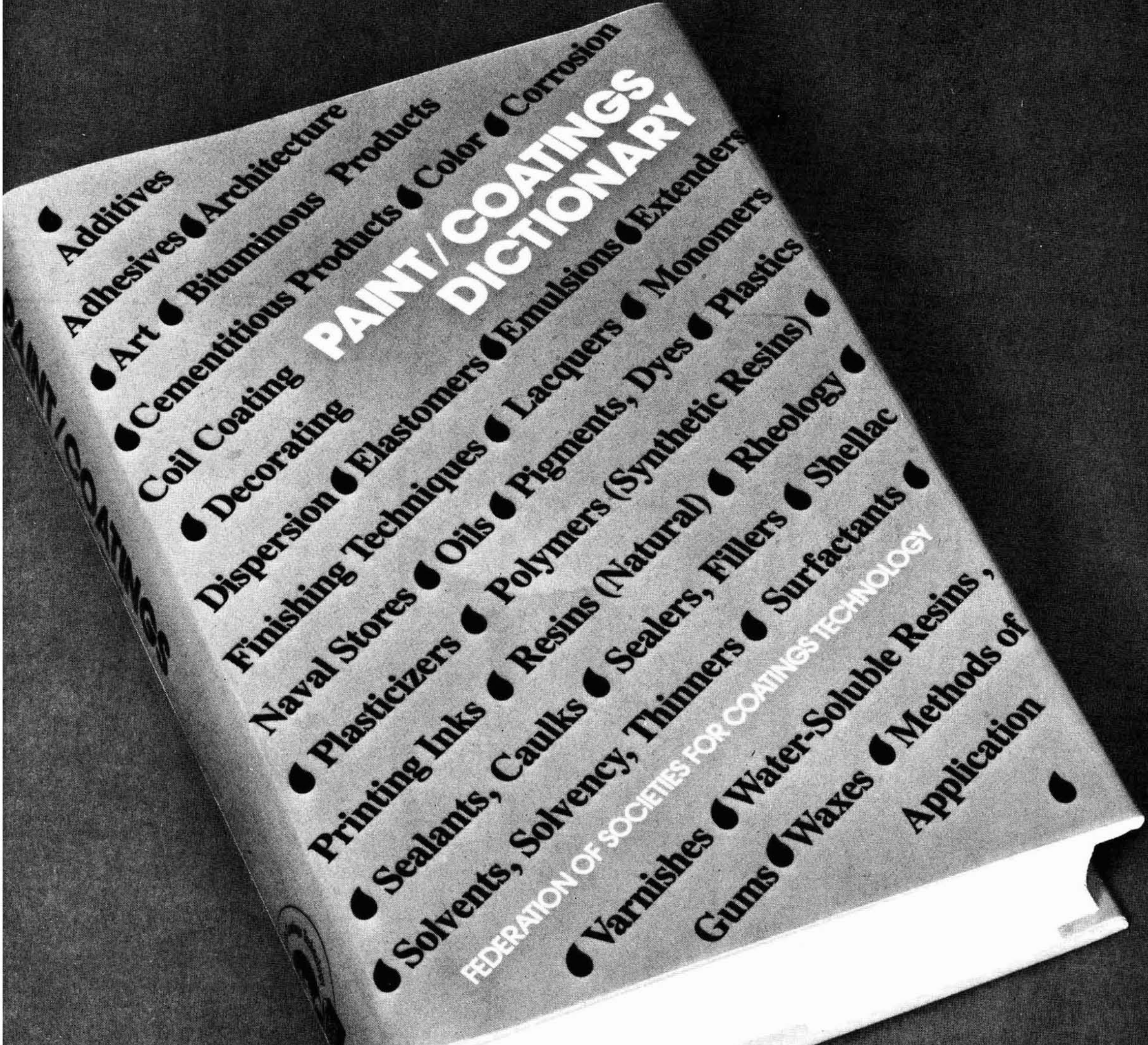
RHEOLATE 1 additive gives your paint good shelf stability. Because RHEOLATE 1 is enzyme resistant, there is no viscosity loss due to enzymatic degradation of the thickener. You can also salvage enzyme-spoiled paint by rethickening with RHEOLATE 1. Plus, RHEOLATE 1 further improves shelf life by reducing settling and syneresis, especially in latex wood stains.

RHEOLATE 1 additive gives you many other excellent paint film and application properties such as reduced roller spattering and improved color acceptance. All at a competitively attractive price. So, before you tie up more water with conventional thickening additives, get the dollars-and-cents facts on RHEOLATE 1. Contact Industrial Chemicals Division, NL Industries, Inc.,
P.O. Box 700, Hightstown, NJ 08520. (609) 443-2547.

# Not for thickening acrylic latex paint.

**NL Industrial Chemicals**

RHEOLATE 1 is a trademark of NL Industries.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## MCA Survey Predicts Record Sales, Increased Growth for 1979

In spite of economic uncertainty for the nation's business in 1979, a survey by the Manufacturing Chemists Association of member companies of the U.S. chemical industry predicts record sales of $140 billion for the year. Members also predict almost normal growth for the industry in all regards.

The expected median sales growth of 11% is very close to that of the last 10 years and that of 1978 over 1977. Net income is likely to increase by the same 11%, an increase above the 6% improvement of 1978 over the previous year.

The single greatest regulatory problem, said MCA, is compliance with the Toxic Substances Control Act, the same as in 1978. Other problems noted were compliance with the Occupational Safety and Health Act, air and water pollution abatement requirements, and the cost of regulations overall. Aside from specific regulatory issues, the most often mentioned concern of member companies polled was inflation. The cost-price squeeze, overcapacity, and the country's economic condition were also referred to frequently.

General optimism about 1979 was expressed in capital additions planning. The total spent in the United States for 1979 was expected to be 7% over 1978. About one-half of this spending will be for new facilities, the balance for modernization of existing plants. Expenditures abroad should be up 11% in 1979 compared to 13% in 1978. Capital outlays for environmental controls for health and pollution abatement are expected to be 15% of total capital expenditures.

The survey predicts costs to rise sharply for the year. The median raw material cost should increase 7% but fuel and electric energy costs will be up 10%. Hourly wage rates will likely go up 8%, which is very close to the President's wage guideline. Construction labor and materials cost in capital expenditures will increase by 8%.

Employment will go up 3%, a larger increase than for last year or for the average of the last 10 years. The growth of research and development efforts at 8% will be below previous growth rates.

Exports by the respondent companies are expected to continue strong with a median 10% increase, close to the historical average increase. This likely would result in another, more than $5 billion trade surplus for the chemical industry. The median capacity utilization will be at 80%, the same as for 1978 but not yet an optimum, best performance level.

The MCA survey respondents report a variation in their expected 1979 performance on the above factors. However, the median responses indicate a respectable year considering the state of the world economies with unusually slow growth rates in the developed countries. Still unknown is the effect on the economy of the Carter Administration's anti-inflation program. The survey results showed no appreciable anticipation of a slowdown in the industry from any decline expected in U.S. business activity.

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## Estimated 1979 R&D Funding Set at Over $52 Billion

Expenditures during 1979 for research and development in the U.S. are expected to reach $52.567 billion, according to the annual R&D forecast of Battelle's Columbus Laboratories. This represents an increase of $5.272 billion (11.1%) over the National Science Foundation estimate of $47.295 billion that was actually spent in 1978. While most of the increase will be absorbed by continued inflation, Battelle forecasts a 3% increase in real R&D expenditures, bringing them to the highest level in 10 years.

Both industrial funding and performance are expected to increase faster over 1978-79 than any other sector (14.6 and 12.6%, respectively). The Battelle report notes, regretfully, that breakdowns of R&D funding by industry groups had to be terminated due to the inadequate data caused by the nonresponse to the NSF R&D surveys by industry. (1978 R&D funding for the chemical and allied industries was estimated to increase 13%, or $2 billion.)

The report notes that 1979 is expected to be characterized by continued inflation—an estimated 7.9% from 1978 to 1979. For statistical purposes, the forecast assumes success for the Carter Administration's current anti-inflation programs; however, many critics feel that this comes too late to have more than limited success.

Despite this, there are many intangibles that favor greater than usual increases in support, particularly the reawakened feeling on the parts of many decision-makers that R&D is essential to long-term economic health.

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## Battelle Labs to Evaluate Tests for Toxic Chemicals

Battelle's Columbus Laboratories has been awarded a three-year, $2.8 million contract from the U.S. Environmental Protection Agency (EPA) to evaluate environmental testing methods for potentially hazardous chemicals.

The EPA will use the study results to assist it in selecting tests related to the chemical fate and ecological effects of toxic chemicals. Under the 1976 Toxic Substances Control Act industry may be required to perform such tests.

According to Dr. Charles W. Townley, who heads the new study, researchers initially will identify classes of tests and available methods for predicting the chemical fate of substances in the environment and for predicting ecological effects of chemicals on aquatic and terrestrial plants and animals.

Dr. Townley said after available tests are identified and generically classed, they then will be tentatively ranked as to which tests would be best to use. Rankings will be based on a test's significance to the risk assessment process, applicability to a broad spectrum of chemicals and life forms, reliability of results, and acceptability to the scientific community. Also to be considered are factors such as the complexity of equipment and facilities required for testing and the length of time needed to obtain results.

The final portion of the study will be devoted to validating the rankings. This will involve conducting laboratory experiments on specific chemicals so that final recommendations can be made. The EPA then will consider these results in selecting the tests industry may be required to conduct.

## Report Shows Chemical Industry Exceeded Energy Conservation Target

The Manufacturing Chemists Association, in a report presented to the U.S. Department of Energy, stated that a major portion of the U.S. chemical industry has again exceeded the 1980 energy conservation target set by the U.S. government.

The 105 companies that took part in the latest survey saved a total of 630 trillion Btu in fuel for the 12-month period which ended June 30, 1978.

MCA pointed out that this was equal to the gasoline it would take to travel 70.9 billion miles at 14 miles a gallon—or to the moon and back 15,000 times. It is enough saved energy to heat 4.5 million households, each using 1,000 gallons of heating oil.

It was also noted that 630 trillion Btu represents 108 million barrels of oil not imported—a saving of more than $1.5 billion—at a time when the dollar was declining and the trade deficit growing.

The 105 companies cut their fuel use by 16% for each unit of output compared with 1972. This compares to a 14% savings recorded just six months ago. After making allowances for energy needed to comply with workplace and environmental regulations not in effect in 1972, the actual energy savings is 17.4%. This figure surpasses the 1980 target of a 17% reduction established by the Department of Energy.

Some of the methods used to accomplish the conservation were detailed in a booklet that accompanied the report. They ranged from simple good housekeeping—turning out lights, insulating, improving maintenance—to projects as complicated as the construction of new plants.

One company now burns 40 million pounds of hydrocarbon waste residue in its boilers where before it would have used 120,000 barrels of oil per year. The waste residue couldn't be used economically until 1975. Since then its inherent fuel value and the desire to reduce the use of natural gas have made its recovery and use economical.

Because the chemical industry is one of the most energy-consuming industries per unit of output, ways to conserve energy were sought long before the 1973 oil embargo. But the energy conservation reporting system formalizes this concern while providing a company with the means to compare its performance against the entire industry.

## PRI Programs Supported by 87 Contributors in 1978

Additional funds for the 1978 research projects of the Paint Research Institute, received during the fourth quarter of 1978, increased to 87 the total number of contributors to PRI for the year — two more than the 1977 total. These funds supplement those committed by the Federation in support of PRI research efforts.

### 1978 Contributors to PRI

#### Societies

Southern; New York; Baltimore; Rocky Mountain; Pacific Northwest; Golden Gate; C-D-I-C; Montreal; Toronto; Cleveland; Houston; Kansas City; Piedmont; Pittsburgh; and Philadelphia.

#### Corporations

Rohm and Haas Co.; Harrison & Crosfield (Canada) Ltd.; Ohio Solvents & Chemicals Co.; Mameco International; Union Carbide Corp.; Midland Div., Dexter Corp.; Benjamin Moore & Co.; David Litter Labs.; Drew Chemical Corp.; Reed Decorative Products Ltd.; DeSoto, Inc.; Dow Chemical, U.S.A.; Nacan Products Ltd.; L.V. Lomas Chemical Co.; Benjamin Moore & Co. Ltd.; Dumar Paints & Chemicals Ltd.; Reichhold Chemicals Ltd.; Bate Chemical Co.; Ltd.; Mobil Chemical Canada Ltd.; Canadian Industries Ltd.; Shell Chemical Co.; Tioxide of Canada Ltd.; Inmont Canada Ltd.; Dar-Tech, Inc.; Donald McKay Smith, Inc.; Jamestown Paint & Varnish Co.; Body Bros., Inc.; The M.F. Cachat Co.; Gulf & Western Chem.-Div. Titanium; Kalcor Coatings Co.; Internat'l Minerals & Chemical Corp.; Ferro Corp.; Monsanto Co.; Palmer Supplies Co. of Cleveland; Q-Panel Co.; Seegott, Inc.; Davies Can Co.; Pfizer, Inc.; Mobay Chemical Corp; Canada Colors & Chemicals Ltd.; Gulf Oil Canada Ltd.; Celanese Canada, Inc.; Selectone Paints Ltd.; Canadian General Electric Co.; PPG Industries, Inc.; Troy Chemical Corp.; Buckman Laboratories, Inc.; Cosan Chemical Corp.; Daniel Products Co.; Tenneco Chemicals, Inc.; Cook Paint & Varnish Co.; Polyvinyl Chemical Industries; and SCM Corporation.

#### Foundations

The N L Industries Foundation; Henry Reichhold Foundation; and Sherwin-Williams Foundation.

#### Associations

Baltimore Coatings Association; Ontario Paint Association; Canadian Paint & Finishing Magazine; and the National Paint & Coatings Association.

#### Individuals

Mr. & Mrs. Flemming Kress; Mr. & Mrs. James Broggini; Ross Galati; Alan R. Hatter; Kenneth C. Waldo, Jr.; Helen M. Skowronska; Thomas D. Tuckerman; Charles A. Kumins; Fred Schwab; Raymond R. Myers; and Anonymous.

# Reversibility of Yellowing Phenomenon In Linseed-Based Paints

Henry Rakoff, Freddie L. Thomas, and Lyle E. Gast
United States Department of Agriculture*

A commercial linseed oil paint, a commercial linseed alkyd paint, and three experimental oil paints (prepared with alkali-refined linseed oil, decolorized linseed oil, or pure trilinolenin as vehicle) were exposed to light, darkness, or to cycles of light and darkness to ascertain if yellowing is a reversible process. It was determined that linseed-based paints can be taken through several cycles of yellowing and bleaching. The extent and rate of yellowing in successive cycles depend on the nature of the paint. Linseed alkyd paints yellow at about the same rate and to about the same extent through each cycle; linseed oil paints yellow less and more slowly through successive cycles.

## INTRODUCTION

Over the years, many people have studied the yellowing of linseed-based paints, but the materials responsible for the yellow color are still unknown. It is known[1] that yellowing is associated with increasing unsaturation in the vehicle, temperature, humidity, nature of the drier, and the wavelength of light to which the film is exposed. Levison[2] has described a small degree of reversal in the yellowing of linseed and safflower oil paints after storage successively under indoor illumination, dark, and indoor illumination again. However, he did not repeat the light dark cycles nor did he bleach his samples in sunlight. It is also known that yellowed films bleach on exposure to sunlight,[3,4] but no quantitative measurements have been reported on the extent to which bleaching occurs, whether the bleached film will yellow again, and, if so, to what extent. We, therefore, set out to try to answer the following questions: (1) Can yellowing and bleaching be carried out over several cycles? (2) Will the extent of yellowing and bleaching be different in each cycle? (3) Will the rate of yellowing and bleaching be different in each cycle? (4) Is direct sunlight a necessity for bleaching? Experimentally, would panels exposed to the north or to the south show different results?

## MATERIALS AND METHODS

### Paints

The two commercial paints, a linseed oil paint and a linseed alkyd paint, were supplied by Spencer-Kellogg Div., Textron, Inc. Their compositions are listed in *Tables* 1 and 2. The experimental paints differed only in the vehicle used and were prepared in the laboratory as described previously.[5] Their composition is listed in *Table* 3. The vehicle used was either alkali-refined linseed oil (ARLSO), a decolorized linseed oil (DLSO), or pure trilinolenin ($Ln_3$).

### Film Preparation

Masonite® squares (2 × 2 × ⅛ in.; 50 × 50 × 3 mm) were primed on both faces and all edges with Sherwin Williams' "Exterior Wood Undercoater." The test paints were applied in two coats several days apart to the smooth faces of the squares. Paints were exposed on the south or north side of the paint test fence at the Northern Regional Research Center in Peoria, Ill. and in the dark in a cupboard in a room controlled to 73°F (23°C) and 50% relative humidity.

### Color Measurement

Color was determined as previously described[5] by a weighted ordinate method after taking 17 readings 20 nm apart from 700 to 380 nm on a Beckman Model DB spectrophotometer with a reflectance accessory. From these data a computer program[6] calculated the three CIE tristimulus values, X, Y, and Z, and from these a yellowness index, YI, where

$$YI = \frac{100Y - 84.674Z}{Y}$$

---

*Northern Regional Research Center, Federal Research, Science and Education Administration, Peoria, Ill. 61604.

This paper was presented before the American Oil Chemists' Society in St. Louis, Mo., May 14-18, 1978, and is reprinted here with the permission of the AOCS, the copyright owner.

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Masonite is a registered trademark of the Masonite Corp.

**Table 1—Composition of Linseed Oil Paint**

| Components | Pounds |
|---|---|
| Rutile $TiO_2$ (R-900®) | 100 |
| Anatase $TiO_2$ (Ti-Pure®FF) | 100 |
| Zinc oxide (XX-601®) | 300 |
| Talc (Nytal® 300) | 350 |
| K.V.O. Linseed varnish oil | 304 |
| T1215 Z2 Heat-bodied linseed oil | 132 |
| Mineral spirits | 116 |
| Fungicide (Metasol® TK-100) | 1 |
| 6% Cobalt naphthenate (0.05%) | 3.5 |
| 24% Lead naphthenate (0.5%) | 9 |
| Total | 1415.5 |

R-900 and Ti-Pure are registered trademarks of E.I. du Pont de Nemours and Co., Inc.
XX-601 is a registered trademark of New Jersey Zinc Co.
Nytal is a registered trademark of R.T. Vanderbilt Co., Inc.
Metasol is a registered trademark of Merck and Co., Inc.

**Table 2—Composition of Linseed Alkyd Enamel**

| Components | Pounds |
|---|---|
| Rutile $TiO_2$ (R-900) | 300 |
| Medium oil linseed alkyd[a] | 687 |
| Kelecin F® | 6 |
| Mineral spirits | 65 |
| High-boiling solvent[b] | 65 |
| Fungicide (Metasol TK-100) | 0.5 |
| 6% Cobalt naphthenate (0.05%) | 3 |
| 24% Lead naphthenate (0.5%) | 7.7 |
| Total | 1134.2 |

Kelecin F, a registered trademark of Spencer Kellogg, Div. of Textron, Inc., is a soybean lecithin used as an emulsifying agent.

(a) Cook's Medium Oil Linseed Alkyd L50-N6 (55% NV/MS/7.78 lb per gal/Y-Z viscosity).

(b) No. 460 Solvent (S and C Group).

**Table 3—Composition of Experimental Paints**

| Ingredients | g |
|---|---|
| Rutile $TiO_2$ (R-900) | 10 |
| Anatase $TiO_2$ (Ti Pure FF) | 10 |
| Zinc oxide (XX-601) | 30 |
| Talc (Nytal 300) | 35 |
| Vehicle | 43.6 |
| Mineral spirits | 11.6 |
| 6% Cobalt naphthenate (0.05%) | 0.35 |
| 24% Lead naphthenate (0.5%) | 0.9 |
| Total | 141.45 |

**Table 4—Comparison of North and South Exposures of Linseed Oil and Linseed Alkyd Paints**

| | | Yellowness Index | | |
|---|---|---|---|---|
| Paint | Exposure | Initial[a] | 27 weeks[a] | 103 weeks[b] |
| Linseed oil | South | 8.5 | 6.7 | 3.7 |
| Linseed oil | North | 8.7 | 7.2 | 4.6 |
| Linseed alkyd | South | 5.6 | 5.3 | 6.3 |
| Linseed alkyd | North | 6.2 | 4.5 | 6.5 |

(a) Average of two values.
(b) One value.

Color was measured initially and at 33 other times over a period of 103 weeks for the commercial paints, and initially and at 28 other times over a period of 99 weeks for the experimental paints. Measurements were made more frequently during bleaching cycles and less frequently during yellowing cycles. This study was conducted between November 1974 and November 1976.

## RESULTS

### Linseed Oil Paint

Two panels of the linseed oil paint were kept in the dark for 103 weeks. They went from an initial average YI of 8.8 to an average YI of 25.8. Most of the yellowing (to an average YI of 19.0) occurred in the first 20 weeks.

Two panels were exposed to the north and two to the south on the test fence for 27 weeks (*Table* 4), and there was no significant difference between the YI's of the north and south panels. One panel of each pair was kept on the test fence for 103 weeks. More bleaching occurred but there was still no significant difference between the north and south facing panels.

Two panels of linseed oil paint were exposed alternately—first to darkness and then to the south, through two cycles. The second yellowing was much less extensive and much slower than the first yellowing as shown in *Figure* 1.

During the same time period, two panels were exposed to three successive cycles of darkness and southern exposure (*Figure* 2). In this case, also, consecutive yellowings were less extensive and much slower with each successive sojourn in the dark. The second and third yellowings of the triple cycle achieved a larger YI than the second yellowing of the double cycle. Perhaps this is due to the fact that the bleaching times in the triple cycle were much shorter than the bleaching times in the double cycle.

### Linseed Alkyd Paint

Two panels of the linseed alkyd paint were kept in the dark for 103 weeks. They went from an average initial YI of 5.3 to one of 17.5. Most of the yellowing (to an average YI of 13.7) occurred in the first 40 weeks.

Two panels were exposed to the south and two to the north for 27 weeks, with no significant differences in their YI's (*Table* 4). One panel from each of these pairs was kept on the test fence for 103 weeks with still no significant difference in YI between the north and south facing panels. It is interesting to note that, although initially the linseed alkyd had a lower YI than the linseed oil paint, at 103 weeks this relationship was reversed.

One set of panels of the linseed alkyd paint was moved alternately from the dark to the south through two cycles (*Figure* 3). The second yellowing was slower but almost as extensive as the first yellowing.

During this same time period, another pair of linseed alkyd panels was moved alternately to the dark and to the light through three cycles (*Figure* 4). These panels yellowed about the same amount each time in the dark and bleached to about the same value each time in the

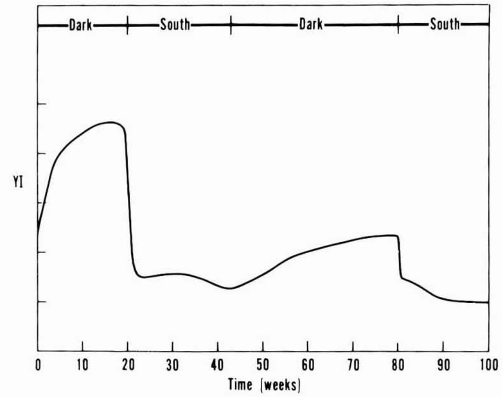

Figure 1—Yellowness index vs. time for linseed oil paint exposed to darkness and to the south through two cycles

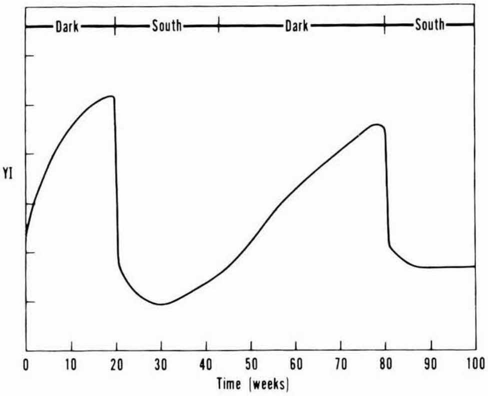

Figure 3—Yellowness index vs. time for linseed alkyd paint exposed to darkness and to the south through two cycles

light. Here, as in *Figure* 3, the extent of yellowing does not seem to correlate with bleaching time for the alkyd paints.

The rate of yellowing, as indicated by the slopes of the peaks in *Figure* 4, is about the same each time. This is in marked contrast to the results obtained with the linseed oil paint in which successive yellowings were slower and less extensive (*Figure* 2).

The first bleaching illustrated in *Figure* 4 resulted in a YI lower than the initial color of the paint. This is not surprising since all paints tended to bleach initially when placed on the fence. The bleached YI reaches the same value during each successive bleaching.

### Experimental Paints

The experimental paints were carried through three cycles of exposure to darkness and to the south on the paint test fence. The alkali-refined linseed oil (ARLSO) and the decolorized linseed oil (DLSO) paints behaved in a similar manner through the three cycles (*Figure* 5). During each cycle, both yellowed rapidly in the dark to about the same extent and bleached rapidly and to about the same extent in the light. In both cases, the first yellowing was substantially more extensive than the second or third yellowings, which were about equal. In both cases, the first yellowing was most rapid, the third yellowing least rapid, and the second yellowing intermediate in rate.

With the trilinolenin ($Ln_3$) paint, the first yellowing was very rapid and very extensive (*Figure* 6). The second and third yellowings developed slowly and to a much smaller extent than the first yellowing. It is interesting to note here that a slower rate of yellowing (third cycle) follows a longer bleaching time.

The results obtained with the experimental paints are similar to the results obtained with the linseed oil paint but are in marked contrast to the results obtained with the linseed alkyd paint, in which the rate and extent of yellowing were about the same in each successive cycle. In all cases, after the first yellowing, each paint, on bleaching, reached a lower YI than its initial value.

## DISCUSSION

The linseed oil paints and the linseed alkyd paint differ substantially in formulation, especially in the pig-

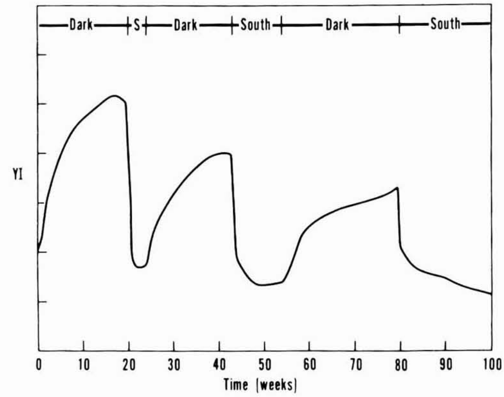

Figure 2—Yellowness index vs. time for linseed oil paint exposed to darkness and to the south through three cycles

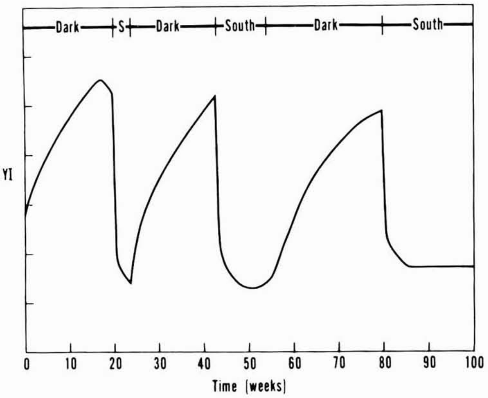

Figure 4—Yellowness index vs. time for linseed alkyd paint exposed to darkness and to the south through three cycles

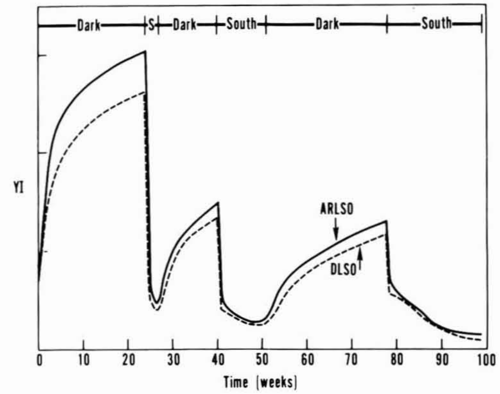

**Figure 5—Yellowness index vs. time for alkali refined linseed oil (ARLSO) paint and decolorized linseed oil (DLSO) paint exposed to darkness and to the south through three cycles**

ments and the vehicle in each. It is likely that both the vehicle and the pigment contribute to the results obtained.

The linseed oil paint yellowed more (from 8.8 to 25.8 YI) than the linseed alkyd paint (from 5.3 to 17.5 YI) in the dark. This confirms the observation of Johnston and Fitzgerald.[7]

As shown clearly in the triple cycle experiments, the linseed alkyd paint (*Figure* 4) yellowed at about the same rate and to the same extent during each cycle. The commercial linseed oil paint (*Figure* 2) and the experimental oil paints [which were made with linseed oil (*Figure* 5) or with trilinolenin (*Figure* 6)] yellowed more slowly and less extensively after each sojourn in the dark. The longer the bleaching time, the slower and less extensive was the yellowing for the oil paints. This would suggest that there are materials involved in the yellowing process—present in the linseed oil paint formulation but not in the linseed alkyd paint formulation—which change during the bleaching cycle, thereby resulting in less yellowing in later cycles for the linseed oil paints.

There is no significant difference in YI between linseed alkyd paint panels exposed to the north and to the south or between linseed oil paint panels similarly exposed.

While yellowing is a slow process, bleaching is extremely rapid. A decrease in YI of as much as six units was noted after one day's residence of a yellowed sample on the test fence.

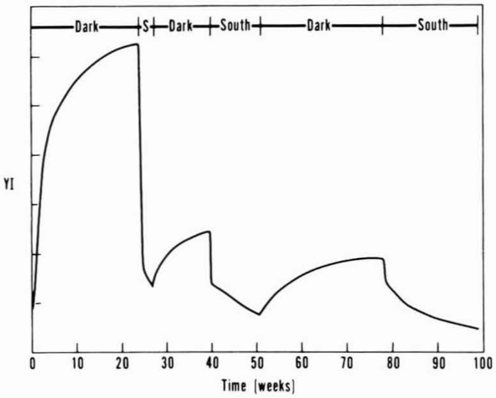

**Figure 6—Yellowness index vs. time for trilinolenin ($Ln_3$) paint exposed to darkness and to the south through three cycles**

Thus, linseed-based paints can be taken through several cycles of yellowing and bleaching. The extent and rate of yellowing in successive cycles depends on the nature of the paint. Linseed alkyd paints yellow at about the same rate and to about the same extent through each cycle; linseed oil paints yellow less and more slowly through successive cycles.

## ACKNOWLEDGMENT

The authors thank the National Flaxseed Processors Association for providing a fellowship under which this work was done.

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# Processing Intermediates For High-Build Paints

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The rheological properties of a castor oil derivative/chlorinated rubber processing intermediate employed in the manufacture of chlorinated rubber high-build paints are investigated. Transient behavior and steady values are studied in steady flow using coaxial cylinder viscometers. Transient pattern depends on sample history and shear sequence. Steady values are not a unique function of the shear rate; oscillatory phenomena often superimpose themselves on initial transients at low shear rate. The complex rheological behavior is discussed and a structural interpretation is suggested. A master curve is obtained for a series of related intermediates of different castor oil content.

## INTRODUCTION

At present, high-build paints are widely employed in the modern coatings industry; they are usually put into work by means of airless equipment. Products of low viscosity at the high shear rates set up in airless spraying device are required for an easy application. At the same time, high viscosity at the very low shear rates involved in film formation is necessary to avoid sagging on vertical surfaces.

Main compositive features of high-build paints are: binder of low viscosity; medium-high PVC; and presence of rheological agents.

To obtain the required rheological performance from the rheological agents it is often convenient to work it into the paint during the manufacturing processes in the form of a previously prepared paint (processing intermediate). The use of processing intermediates is a common practice with Bentones.[1] Castor oil derivatives[2] also are advantageously added during manufacturing in the form of a paint. To develop their beneficial action, in fact, they need to be brought under strong shearing into a well defined range of temperatures. These conditions are difficult to obtain and control during milling; the addition of the castor oil derivative to the mill-base is performed before letting down in the form of an intermediate previously prepared in the optimal working conditions.

Binary systems (castor oil derivative + solvent) display some tendency to separate solvent during storage and change into two-phase material. Ternary systems, i.e., castor oil derivative + solvent + binder, are reasonably stable for lengthy intervals. The presence of a polymer deprives the intermediate of the distinctive feature of being a product of general application. Like binary systems, ternary systems are usually formulated which fulfills the requirements of up-to-date working technology.

Castor oil processing intermediates create problems as far as agitating, mixing, pumping, transportation, and storage are concerned. Moreover, the complex rheological behavior of high-build paints largely depends on quality, quantity, and the mode of addition of the rheological agent. It follows that a comprehensive knowledge of the rheological properties of these processing intermediates is of paramount importance: (1) for the best way to deal with these materials, and (2) for a thorough understanding of the rheological behavior of the ready-mixed paint to which they are added.

Objectives of the present work are the study and characterization of the rheological properties of a castor oil processing intermediate employed in the formulation of high-build chlorinated rubber paints.[3,4]

## EXPERIMENTAL

### Materials and Apparatus

Castor oil derivative processing intermediates are advantageously formulated at a castor oil derivative concentration ranging between 6 and 10%. The processing intermediate studied here was a 6% paste[**] whose vehicle was composed of a 20% chlorinated rubber[††] solution in aromatics. For the sake of complete-

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[**] Thixatrol® ST - The Baker Castor Oil Co., Bayonne, N.J.

[††] Alloprene® R 10 - I.C.I. Ltd, England.

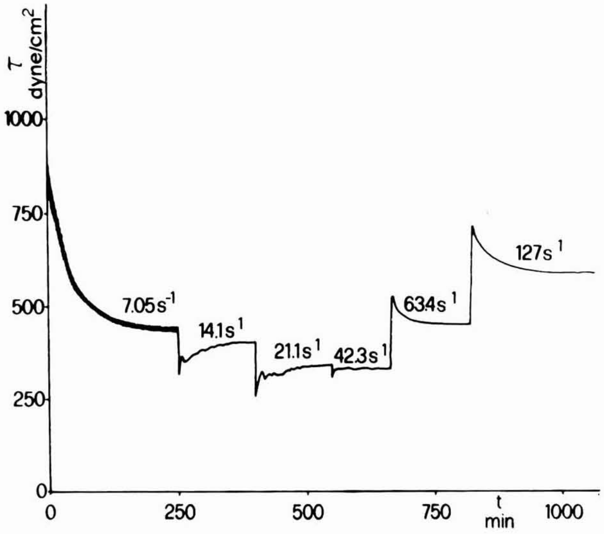

**Figure 1—Time dependence of shear stress of 6% castor oil derivative/chlorinated rubber paste in aromatics at increasing shear rates**

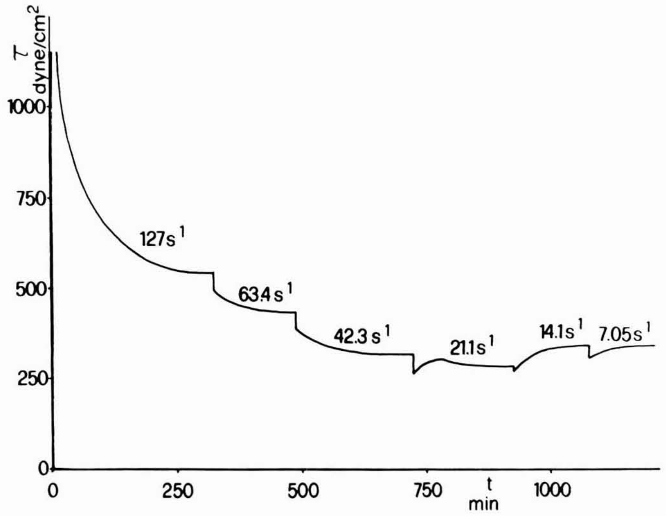

**Figure 2—Time dependence of shear stress of 6% castor oil derivative/chlorinated rubber paste in aromatics at decreasing shear rates**

ness, the investigation was extended to pastes with different castor oil derivative contents.

A Rotovisko-11, coaxial cylinder viscometer, was employed. Tests are carried out at 25°C.

### Rheological Tests

Initially, to study the flow properties of the processing intermediate, two different samples were submitted to a series of $\dot{\gamma}$ increasing and $\dot{\gamma}$ decreasing sequences, respectively; $\dot{\gamma}$ application was continued until $\tau$ steady values were attained. As examples, two sequences are reported in *Figures* 1 and 2. It appears that the rheological behavior of the tested material is very complex. It involves:

(1) oscillatory phenomena;

(2) transients not always monotonic and which correspond to different rheological patterns; and

(3) $\tau$ steady values nonunique function of $\dot{\gamma}$.

To understand better the phenomena of (3), procedures reported in *Table* 1 were applied.

In detail:

PROCEDURE A - A series of shear rates, two at a time, are applied, each pair with a first high shear rate followed by the same low (reference) shear rate. For both shear rates, $\tau$ steady values were attained and for each sequence new samples were employed.

PROCEDURE B - A modification of Procedure A, where a decreasing sequence is introduced between the higher and the lower (reference) shear rate.

Results are reported in *Tables* 2 and 3.

**Table 1—Testing Procedures and Shear Rate Sequences**

| Procedure | Shear Rate ($s^{-1}$) Sequence |
|---|---|
| A – A1 | 14.1 - 7.05 |
| A2 | 42.3 - 7.05 |
| A3 | 127 - 7.05 |
| A4 | 381 - 7.05 |
| A5 | 1140 - 7.05 |
| B – B1 | 14.1 - 7.05 |
| B2 | 42.3 - 21.1 - 14.1 - 7.05 |
| B3 | 127 - 63.4 - 42.3 - 21.1 - 14.1 - 7.05 |
| B4 | 381 - 190 - 127 - 63.4 - 42.3 - 21.1 - 14.1 - 7.05 |
| B5 | 1140 - 571 - 381 - 190 - 127 - 63.4 - 42.3 - 21.1 - 14.1 - 7.05 |

**Table 2—Shear Stress Steady Values Obtained By Means Of Procedure A for 6% Castor Oil Derrivative-Chlorinated Rubber Paste in Aromatics**

| Procedure | $\dot{\gamma}$ ($s^{-1}$) | $\tau$ (dyne/cm$^2$) |
|---|---|---|
| A1 | 14.1 | 651 |
| | 7.05 | 577 |
| A2 | 42.3 | 459 |
| | 7.05 | 372 |
| A3 | 127 | 525 |
| | 7.05 | 385 |
| A4 | 381 | 612 |
| | 7.05 | 273 |
| A5 | 1140 | 1010 |
| | 7.05 | 237 |

## DISCUSSION

### Oscillatory Phenomena

From an examination of the sequences of increasing $\dot{\gamma}$ started from low initial $\dot{\gamma}$ values (like that reported in *Figure* 1), it follows that the initial transient is always characterized by a shear stress decrease on which an oscillatory phenomenon is superimposed. Oscillations last longer at lower shear rates and are generally damped. Oscillatory phenomena had already been experimentally noticed in some polymeric systems[5] and were the subject of theoretical treatments on the basis of corotational rheological models.[6]

### Transients

Transients are not always monotonic. Moreover, as to pattern, they belong to different classes of rheological behavior. It can be suggested that a structural network is present in the material in rest conditions, and, when a shear rate is applied, it undergoes a breakdown (which involves the shear stress decrease). In the course of this process, structural units are released which have a high "elasticity" and, accordingly, give rise to the oscillatory phenomenon already noticed in viscoelastic systems.

The transients which follow the initial one can be either monotonic or nonmonotonic. The former type resemble both thixotropic and anti-thixotropic* behavior patterns, depending on whether the shear stress decreases or increases with time as a consequence of the application of a shear rate higher than that previously experienced by the material until attainment of a steady value. Thixotropic and anti-thixotropic behaviors can be encountered in the course of the same transient, at constant shear rate; this situation results in a nonmonotonic behavior.

### Steady Values

Steady values are not a unique function of the shear rate. This phenomenon was previously noticed by the authors for more complex materials;[7] it is explicitly presented by Cheng as a manifestation or proof of the fact that the fluid is multi-structured.[8] The presence of more than one structure in a material can also explain the nonmonotonic transient.[9] The hypothesis of the existence of a large spectrum of structural elements of different size (multi-component system) is assumed as the basis of a theory on thixotropic fluids.[10] Accordingly, thixotropic phenomena are explained by the evolution in time of the structure size distribution.

From an examination of the results obtained by the application of Procedures A and B (see *Tables* 2 and 3), it appears that the steady values at 7.05 $s^{-1}$ in Procedure A are decreasing when increasing the shear rates previously experienced by the material.

Procedure B confirms the dependence of 7.05 $s^{-1}$ steady values on the previously applied shear rate (7.05 $s^{-1}$ steady values decrease when increasing the highest shear rate previously applied). A comparison of steady values at 7.05 $s^{-1}$, obtained after application of Procedures A and B having the same highest shear rate value, displays the role of the sequence of decreasing shear rates applied between the high shear rate and the reference of 7.05 $s^{-1}$ in B sequences.

The curves of the various B sequences in a $\tau$ versus $\dot{\gamma}$ plot show a pseudoplastic behavior with yield value. The single curves locate regularly with the increasing of the higher shear rate proper to each sequence, as does the corresponding $\tau_0$.

**Table 3—Shear Stress Steady Values Obtained By Means Of Procedure B for 6% Castor Oil Derivative-Chlorinated Rubber Paste in Aromatics**

| Procedure<br>$\dot{\gamma}$ ($s^{-1}$) | B1 | B2 | B3<br>$\tau$ (dyne/cm$^2$) | B4 | B5 |
|---|---|---|---|---|---|
| 1140 | — | — | — | — | 1100 |
| 571 | — | — | — | — | 722 |
| 381 | — | — | — | 558 | 590 |
| 190 | — | — | — | 405 | 453 |
| 127 | — | — | 512 | 301 | 394 |
| 63.4 | — | — | 415 | 262 | 328 |
| 42.3 | — | 410 | 306 | 221 | 273 |
| 21.1 | — | 415 | 279 | 204 | 230 |
| 14.1 | 651 | 362 | 319 | 244 | 230 |
| 7.05 | 577 | 372 | 306 | 225 | 200 |

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*The words "thixotropic" and "anti-thixotropic" correspond to a convenience distinction. The word "anti-thixotropic" is employed with the aim to distinguish a behavior opposite to "thixotropic", without involving on account of this any connection with rheopexy.

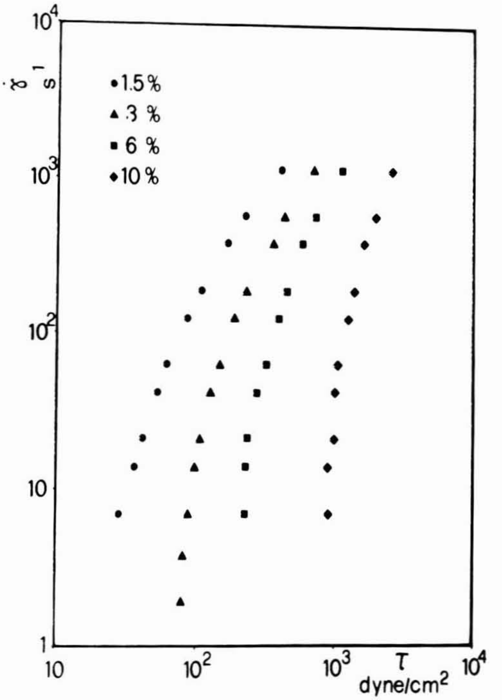

**Figure 3—Shear rate versus shear stress for 1.5 - 3 - 6 - 10% castor oil derivative/chlorinated rubber paste in aromatics**

A B5 procedure was also applied to corresponding 1.5,3, and 10% castor oil derivative processing intermediates; $\tau$ steady values are reported in *Figure* 3.*

Flow curves of the materials with different castor oil derivative content show a marked pseudoplastic behavior in the range of the shear stress considered. Experimentally determined steady values correlate with Casson equation, so that a $\tau_0$ value is made available.

In subsequent investigations, at very low shear rate ($10^{-1}s^{-1} \div 1\ s^{-1}$) the flow behavior tends towards a second Newtonian plateau. This fact calls to mind analogies with the flow behavior in the same shear rate range described[11] for disperse systems of spherical particles in polystyrene solutions, and once more presents problems concerning the existence of an actual yield value.

A master curve could be drawn by shifting the curves of various castor oil derivative content on the 1.5% castor oil derivative curve which was assumed as the reference curve. A master curve is given in *Figure* 4, in a $\dot{\gamma}$ reduced versus $\tau$ reduced plot. Shift factors $a_{\dot{\gamma}}$, $a_\tau$ and ratio $C_R = C/C_r$ are given.

## GENERAL COMMENT

A comparison of the results obtained in the application of sequences A and B with the same high initial shear rate shows that:

(1) Transients at 7.05 $s^{-1}$ reference shear rate are of thixotropic type if the initial shear rate is very high (high $\Delta\dot{\gamma}$), of anti-thixotropic type if the initial shear rate is low (low $\Delta\dot{\gamma}$).

(2) Steady values at 7.05 $s^{-1}$ reference shear rate are different when Procedure A or B with the same high initial shear rate is applied; different steady values at 7.05 $s^{-1}$ reference shear rate are obtained by applying Procedure A or B; and values obtained with the latter are generally smaller.

According to Cheng,[8] the fact that steady values are not a unique function of shear rate is typical of multistructured thixotropic systems, in which, for equal shear disturbances, different structures set in, depending on different rheological histories. It is suggested that in materials of this type the application of any shear rate after a lengthy rest causes a breakdown of the initial structure into units of smaller dimensions which are aligned, or, in any case, oriented in the flow direction. Single flow units are deformed and accumulate elastic energy. The subsequent application of a much lower shear rate (reference shear rate 7.05 $s^{-1}$—Procedure A5, A4), allows the maximum development of the tendency to structural rebuilding by virtue of the high $\Delta\dot{\gamma}$. A very fast structural build-up takes place so that not all the structural units may be able to release the elastic energy previously accumulated and a thixotropic response at 7.05 $s^{-1}$ follows. On the other hand, the appli-

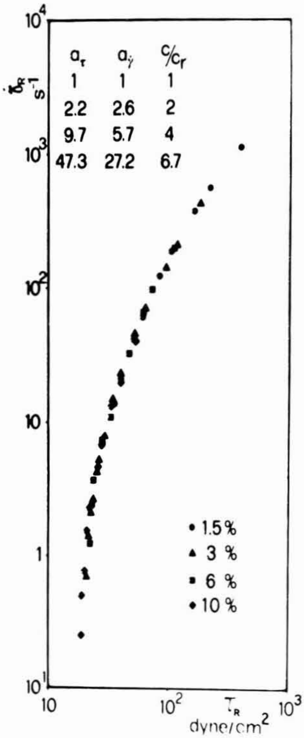

**Figure 4—General master curve of reduced shear rate versus reduced shear stress for 1.5 - 3 - 6 - 10% castor oil derivative/chlorinated rubber paste in aromatics**

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*B5 procedure was selected as it gives the widest information within the family of B sequences; B type procedure was preferred to A procedure since it allows the same material to be used for the determination of all the steady values considered.

cation of moderate lower shear rates after any higher shear rate, as in Procedure B, results in conditions of slower and quantitatively smaller rebuilding. A better relaxation of the accumulated elastic tension may take place and the response is no longer necessarily of thixotropic type; however, it can also be of essentially viscoelastic type. So, at the end of a B procedure involving only small steps in shear rate, the material structure in steady conditions develops a stress lower than that shown by the material brought straight to 7.05 $s^{-1}$ from the highest shear rate.

Summarizing, transient behavior is governed by both $\Delta\dot{\gamma}$ and previous history; for low $\Delta\dot{\gamma}$ the effect of history may play a determining role and, accordingly, must be taken into account. Steady values are strongly affected also by rheological history. Steady values at the low shear rate for the application of pairs of high-low shear rates depend on $\Delta\dot{\gamma}$ value and on how the lower shear rate is applied. Elastic relaxation can be present so that steady values obtained are different in correspondance to different structural situations.

### Comparison of the Flow Properties Of the Processing Intermediate Studied with Related Products

A better understanding of the flow behavior of the processing intermediate ternary system, castor oil derivative + polymer + solvent, can be achieved through a comparative examination of the flow properties of the binary systems, polymer + solvent and castor oil derivative + solvent, and, eventually, of the ready-mixed paint, i.e., of the complex system containing polymer + solvent + pigment + additives + castor oil derivative, of which the ternary system only forms part.

POLYMER SOLUTION—The polymer considered is a material of very low molecular weight. In the ranges of shear rate and concentration considered, its solutions in aromatics exhibit a slightly pseudoplastic behavior. The viscosity is close to the zero shear rate viscosity.

BINARY SYSTEM CASTOR OIL DERIVATIVE + AROMATICS—In general, the flow pattern is of the pseudoplastic type for low castor oil derivative concentrations (1.5%–3%–4.5%). For higher concentrations, pseudoplasticity and presence of a yield value were noticed. Steady values are not a unique function of the shear rate. The phenomenon, however, is much less marked than for ternary systems. Similarly, transients are of thixotropic and anti-thixotropic types, but with a far less marked differentiation in shape.

The complex system, which in its turn contains the ternary system, exhibits some of the particular rheological features of the latter. In fact, at a given shear rate, high-build paints exhibit steady values which are not a unique function of the shear rate, but which depend to a greater or lesser extent on the previous rheological history. Transients are of both thixotropic and anti-thixotropic type (with a clear predominance of the former type) according to the different shear sequences applied. In addition, high-build paints usually exhibit a yield value.

Hence, a comparison of the flow behavior of the ternary system, castor oil derivative + polymer + solvent, with that of the binary system, castor oil derivative + solvent, shows that the presence of the polymer in the ternary system enhances the particular features of the flow behavior of the binary system (larger differences in steady values at constant shear rate after different rheological histories, different transient patterns). This suggests that the polymer takes active part in the structure developed by the rheological agent. On the other hand, the presence of the suspended solid phase in more complex systems, like ready-mixed high-build paints, only partially reduces flow peculiarities of the ternary system.

## SUMMARY

The reported investigations allow the following considerations to be made:

- The processing intermediates studied exhibit a complex rheological behavior, which displays both a thixotropic and an anti-thixotropic (viscoelastic) nature, depending on the shear rate and the shear sequence applied, and the previous rheological history.
- Rheological history is not cancelled by the application of strong shearing and is cancelled only by resting for a time exceeding several weeks.

These peculiar features must be kept in mind for any correct manipulation of the material.

The application of Procedure B5 is suggested for production control.

A comparison between the rheological properties of the intermediate and those of a couple of high-build paints to which the intermediate was added shows that, in the ready-mixed paint, the main features of the intermediate are found.

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# Survey of the Applications, Properties, and Technology Of Crosslinking Emulsions

## Part VI

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This review summarizes most of the principal concepts which have been employed over the last ten years to develop thermosetting or crosslinking emulsions. The conceptual elements which have formed the foundation for this new generation of coatings are discussed in terms of the science, technology, properties, and applications of the emulsion systems which have been reported in the literature. This survey presents the advantages and limitations afforded by each of the functional monomers or polymer classes which have been investigated for the development of crosslinking emulsions. Parts I through IV of this review presented a state of the art introduction into crosslinking emulsions and discussed the development of conventional emulsions which produce either homogeneously or interfacially crosslinked coatings. Part V discussed the topic of conventional emulsions which produce predominantly interstitially crosslinked coatings and introduced the topic of post-emulsified systems with a discussion of drying oil and alkyd emulsions. This paper concludes the topic of post-emulsified systems with a discussion on epoxy, epoxy ester, polyurethane, silicone, and phenolic-based emulsions and also briefly discusses such topics as precrosslinked emulsions, thermoset water-in-oil emulsions, nonaqueous dispersions, and other methods of application and curing. This six-part review serves as an introduction to a series of publications from these laboratories describing the research and development of crosslinking emulsions which are new and novel or which have not been adequately described in the present literature.

### POST-EMULSIFIED SYSTEMS

Because many classes of polymers are not mechanistically or kinetically tractable to emulsion polymerization methods and principles, several post-emulsification techniques have been developed to disperse preformed, step-growth, or condensation-type polymers in an aqueous medium.

Common emulsification procedures involve reducing the viscosity of the resin through solvent or reactive diluent addition or by the application of heat; addition of up to 7% of an emulsifier with a suitable HLB value to either the resin or aqueous phases; and subsequent emulsification of the resin-surfactant-water mixture by means of mechanical dispersion, homogenization, or ultrasonic irradiation. Typically, the products which result possess a broad distribution of particle sizes with maximum particle sizes exceeding 20 microns for mechanically dispersed systems and 4-8 microns for homogenized or ultrasonically processed systems. As a result of the large particle sizes, these emulsions rarely display storage stabilities exceeding six to nine months; and because of the presence of contaminating surfactants, the overall resistance and durability properties of the coatings are usually less than those of identical systems applied from solvents.

While recent results indicate that post-emulsified systems may be classified topographically according to such modes as homogeneous crosslinking (alkyd resin emulsions) or interfacial crosslinking (catalyzed epoxy emulsions), such classifications are difficult to apply to post-emulsified systems because of the greater degree of disorder in the particle packing morphology of emulsion systems possessing a broad distribution of particle sizes (20 to 150 microns). Therefore, the following post-emulsified systems will not be presented in terms of topographical classifications.

### Catalyzed Epoxy Emulsions

Because of the escalating costs of conforming aromatic and oxygen-containing solvents (esters and ketones) together with the projected enactment of environmental legislation requiring the utilization of practically solvent-free or emissionless coating systems, the marketing advantage possessed by certain high-

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performance, solvent-borne systems has eroded to such an extent that many of these higher integrity systems are being replaced by such lower performance systems as water-reducible alkyd resins or even conventional, acrylic emulsions. Nevertheless, because many marine and industrial applications demand products with superior performance capabilities, industry has placed considerable emphasis on the development of two-component epoxy emulsions which would ideally possess the high-performance characteristics displayed by conventional, solvent-borne epoxy systems.

While some of the earlier attempts yielded epoxy emulsions which were useful as block fillers, maintenance primers, and seamless floor coatings, these incipient developments usually yielded products displaying short pot lives, low solids contents, poor flow and leveling, and reduced gloss and impact resistance,[1] in addition to a general diminution in overall performance characteristics. Fortunately, many of the product deficiencies displayed by these initial attempts have been somewhat ameliorated through the establishment and subsequent utilization of more efficacious technology. The improved systems are reported to approximate the solvent, alkali, and acid resistance; adhesion to steel, concrete, and inorganic zinc coatings; and abrasion resistance of their solvent-borne counterparts. However, the improved systems are still not recommended for applications requiring continuous immersion in water.

In general, epoxy coatings cast from emulsion form tend to cure slightly faster than coatings cast from solution, but through-dry time for the emulsion coatings is somewhat slower. For example, films of a catalyzed epoxy emulsion coated at 3 to 4 mils dry film thickness (DFT) and dried at 21°C displayed a set-to-touch dry time of one to two hours, a tack-free time of two to three hours, a dry-hard time of four to five hours, and a light-traffic-hard time of 16 hr.

While epoxy coatings cast from emulsion form give better gloss retention upon weathering than coatings derived from solution, the initial gloss of epoxy coatings cast from emulsion form is somewhat lower (70% and up) than solvent-borne systems. However, gloss characteristics may be improved somewhat through the addition of coalescing aids and wet edge extenders. Also, high-build coatings of these emulsions can be obtained over alkyd coatings without lifting of the substrate coating by use of airless spray.[2]

A typical formulation which employs a conventional, single-stage dispersion technique and which yields a glossy, white brushing enamel is illustrated in *Table* 1. The manufacturing procedure for this emulsion involved combining the epoxy resin and surfactant in a suitable container, agitating the mixture with a high-speed disperser to form a uniform blend, adding an equal quantity (by weight) of tap water, and dispersing the mixture at high speed to form an oil-in-water emulsion. The pigment was added after emulsification, and the resulting paint was let-down with the remaining quantity of water. The polyamide curing agent, which was in solution form, was added to the epoxy component just prior to use.[3]

**Table 1—Conventional Polyamide-Cured Epoxy Emulsion**

| Ingredient | Pounds Used | Gallons Used |
|---|---|---|
| Vehicle Component | | |
| Bisphenol A Epoxy Resin (Mol.Wt. of 380) | 96.5 | 9.95 |
| Ep-Aqua Emulsifier | 31.1 | 3.83 |
| $TiO_2$ | 255.2 | 7.48 |
| Water | 381.7 | 45.82 |
| | 764.5 | 67.08 |
| Curing-Agent Component | | |
| Van-Amid 315 | 190.9 | 23.57 |
| Mineral Spirits | 55.0 | 7.74 |
| Cellosolve Acetate | 27.0 | 1.61 |
| | 272.9 | 32.92 |
| Total Formulation | 1037.4 | 100.00 |

As in other post-emulsified systems (see the sections describing drying oil and alkyd emulsions in Part V of this review, JCT, December 1978, pp 65-96), various procedural techniques have been employed with epoxy resins to generate emulsions with improved colloidal, physical, and rheological characteristics. For example, epoxy emulsions which yielded coatings with properties comparable to those of solvent-borne epoxy systems were prepared using a two-step inversion process in which the resin component was first emulsified in a suitable jacketed mixer (Daymax or Ross Double Planetary) to generate an emulsion with an average particle size of 1 to 3 microns. In the second step, the crude emulsion was passed through a Gaulin homogenizer at 5000 psi to yield an emulsion with a particle size of 1.0 microns or less. The following procedure was used to prepare pilot quantities of a 93/7-Epon® 1007/Resimene® X-745 emulsion which was useful in applications amenable to thermal curing:

(1) A mixture consisting of Epon 1007 (75% solids) and Resimene X-745 was charged into a Daymax mixer, and the blend was shear processed until the temperature increased to 66°C (150°F).

(2) Upon attaining the required temperature, Alipal® EP-110, the ammonium salt of a sulfated alkylphenoxypoly(ethyleneoxy) ethanol, was added to the resin blend, and the resulting mixture was agitated for 5 min.

(3) A first addition of distilled water was added over a 6 min period, and this mixture was agitated for an additional 7 min.

(4) A blend of Natrosol® solution, ammonium hydroxide, and the final addition of water were then added, and the dispersion was subsequently mixed for an additional 2 min.

(5) The resulting crude emulsion, which was still at 66°C, was processed through a Gaulin laboratory homogenizer, Model 15M, at 5000 psi to yield the reduced particle size product. The completed system possessed a pH of 8.0 to 9.0 and contained 7% Resimene X-745 as a crosslinking agent.

The Epon 1007/Resimene X-745 emulsion formulation was found to adequately wet tin-free steel and

**Table 2—Performance Characteristics Of a 95/5-Araldite 6097/Cymel 303 Dispersion**

| | |
|---|---|
| Cured Film Appearance: | |
| Color | Clear |
| Tack | Dry |
| Flow | Some Small Craters |
| Cross Cut Adhesion | Excellent |
| MEK Resistance (single rubs) | >500 |
| Fabrication of 303 can end[a] (inside coated) | Pass |
| 303 Can end immersed 8 min in $CuSO_4$ solution[b] ($pH=1.8$) | Pass |
| Beer pasteurization, 30 min at 82°C (in open container) | |
| Appearance | No Change |
| Adhesion | Excellent |
| Water pasteurization, 30 min at 82°C (in open container) | |
| Appearance | No Change |
| Adhesion | Excellent |
| Steam processing, 90 min at 121°C | |
| Vapor Phase | |
| Appearance | No Change |
| Adhesion | Excellent |
| Water Phase | |
| Appearance | No Change |
| Adhesion | Excellent |

(a) The stretch involved in fabricating a 303 can end is approximately equal to a 2T-3T bend.

(b) Acid resistance was determined by immersing a fabricated 303 aluminum food can end in a 10% acidic (HC1) $CuSO_4$ solution. A darkening of the can end indicates permeation of the coating by the $CuSO_4$ solution.

produced coatings with sufficient flexibility to pass a 1-T bend test. After curing for 10 min at 204°C (400°F), coatings of this emulsion (0.2 to 0.3 mil) passed between 80 and 100 methyl ethyl ketone (MEK) double rubs. When applied to a treated aluminum substrate (A1 401-45) and subjected to a beer pasteurization test (30 min at 66°C), the product demonstrated only very slight blushing and passed a cellophane tape adhesion test although after a steam processing test, identical samples displayed from slight to moderate blushing.

If the flash time prior to baking was kept to a minimum (about 30 sec or less), such properties as the gloss, uniformity of appearance, and chemical resistance of the coatings could be improved. However, as was the case with other post-emulsified compositions, the epoxy resin/melamine curing agent blend was found to possess such typical deficiencies as a lack of freeze-thaw stability together with poor rheological characteristics for roller coating applications.

In an effort to upgrade the qualities of the Epon 1007/Resimene X-745 system, other emulsifying agents, in addition to Alipal EP-110, were evaluated for their effectiveness in the emulsion formulation. In general, Alipal CO-436, which is compositionally similar to Alipal EP-110, did not provide the emulsification efficiency of Alipal EP-110 but did yield coatings with essentially equal physical properties. Gafac® RE-960, an anionic surfactant based upon complex organic phosphate esters of aromatic compounds, presumably afforded good emulsification qualities but was less effective than sulfate-containing surfactants in catalyzing the crosslinking of the melamine resin.

Besides investigating the capabilities of melamine-cured coatings, both urea and phenolic resin-containing epoxy emulsions were also evaluated, but neither compositional blend provided the coating qualities of the melamine-containing epoxy emulsion. Coatings of the epoxy resin/urea crosslinking agent blend demonstrated moderate to severe blushing during steam processing and beer pasteurization while the epoxy resin/phenolic system was intermediate between the melamine and urea modifications in these evaluations. Both the urea and phenolic modified systems showed greater solvent resistance (>100 MEK double rubs) than the melamine system, presumably due to the use of higher concentrations of both crosslinking resins.[4]

As suggested by these results, epoxy emulsions containing either phenolic or melamine resin curing agents are not completely free of certain product deficiencies; yet in comparison with systems containing other types of curing agents, phenolic or melamine resin/epoxy resin combinations have been claimed to provide a reasonable balance of properties for such applications as coil, drum, and various interior can coatings. When properly formulated, melamine/epoxy emulsions can provide coatings whose chemical resistance is similar to that of coatings derived from conventional, solvent-borne epoxy systems. For example, fine particle size, epoxy resin dispersions with improved rheological characteristics for roller coating applications were formulated from 86.4% epoxy resin dispersion (55% solids), 2.5% Cymel® 303 curing agent, 0.2% morpholine salt of p-toluenesulfonic acid, 0.13% BYK 301 flow agent, and 10.8% water. The epoxy resin dispersion was prepared either by dispersing a melted, high molecular weight epoxy resin (Araldite® 6097) in water containing a nonionic surfactant or by adding the finely ground, solid resin to an aqueous surfactant solution and shearing the resulting mixture to produce a thick slurry. Once formed, the slurry was ground in a ball mill to reduce the particle size. The 95/5-Araldite 6097/Cymel 303 dispersion was applied to Alodine® treated aluminum at a film thickness of 0.2 mils and cured 10 min at 205°C to yield a product whose properties were similar to those of coatings derived from solvent-borne, Araldite 6097 systems. *Table* 2 presents the performance characteristics of the 95/5-Araldite 6097/Cymel 303 formulation described above.

These results suggest that the 95/5-Araldite 6097/Cymel 303 dispersion yielded coatings which displayed excellent adhesion to aluminum and which provided good resistance to acid, water and steam, and solvents. While this formulation was successfully applied by means of roller coating, some difficulty was encountered during spray applications.

A single-stage emulsification technique similar to the one employed in the preparation of the 95/5-Araldite

6097/Cymel 303 dispersion was also used in the preparation of a 70/30-Araldite 6097/Phenolic HZ 949U emulsion except that the formulation contained up to 20%, by volume of the total volatiles, of an organic solvent. While the coating properties of this emulsion were very similar to those of the epoxy/melamine dispersion (*Table* 2), the epoxy/phenolic emulsion provided greater ease of preparation and superior wetting and flow and was repeatedly roller coated with good success.[5]

## Examples of Epoxy Emulsions

Some of the other compositional variations, performance characteristics, properties, and applications of post-emulsified epoxy systems are presented in the following examples.

Finishes for fabrics which provided improved resistance to shrinkage were formulated from 50 parts of a bisphenol A-epichlorohydrin resin having a viscosity of 150 poise at 25°C, a molecular weight of about 350, and an epoxide equivalent weight of 175 which was combined with 10 parts of a polyethylene glycol ether of sorbitan monopalmitate surfactant (Tween® 40) at 100°C. The molten epoxy resin/surfactant mixture was mixed with 50 parts of a 5% aqueous poly(vinyl alcohol) solution and subsequently inverted by the addition of 500 parts of warm water to form an epoxy resin emulsion with good stability. The resulting product (100 parts) was catalyzed with 0.75 parts of zinc fluoroborate, applied to a rayon cloth, dried at 100°C, and cured for 5.5 min at 160°C to yield a product with improved shrink and crease resistance, wet strength, and permanent stiffness. The emulsion could be stored for several months without deterioration.[6,7]

Finishing agents for textiles which improved the printing and dye receptivity of fabrics were prepared from a solution of an epoxy resin which was emulsified in water containing such emulsifying agents as condensates of triethanolamine with oleic acid or adducts of poly(ethylene oxide) with fatty acids. The epoxy resin consisted of a diglycidyl ester of a dianhydride with an average molecular weight of 450 and containing 20% by weight of epoxide groups. The epoxy resin emulsion was combined with either low molecular weight amine curing agents (ethylenediamine, diethylenetriamine, or other polyethylenepolyamines) or a second emulsion consisting of a polyamide of a dimerized fatty acid or a precondensate of an epoxy resin with ethylenediamine, diethylenetriamine, or m-phenylenediamine to yield the desired product. Such properties as the stability and pot life of the latex and chemical resistance of the coatings were not disclosed.[8]

Epoxy/polyamide emulsions which cured at low temperatures and which were useful as heat-sealing adhesives were formulated from 16.7 g of a bisphenol A epoxy resin solution which was mixed with 16.7 of a polyamide resin possessing an amine value of 80 to 90 and prepared from a polymeric fatty acid and diethylenetriamine. The epoxy resin/polyamide mixture was emulsified in 66.5 ml of water without the aid of emulsifying agents and was catalyzed with 0.4 equivalents of formic acid to yield an emulsion which when applied to a paper substrate and dried yielded an impregnate with excellent appearance and properties. The resulting product was useful as wet-sealing and heat-sealing adhesives which were stable for greater than 15 weeks.[9]

Epoxy/polyamide emulsions useful as adhesives for joining ceramic tiles to wood, concrete, and asbestos were formulated from 18.5 parts of Epikote® 828 which contained 1.9 parts of Armac® cationic emulsifying agent. The epoxy resin/surfactant mixture was heated to 80°C and mechanically dispersed in 6.0 parts water. The resulting emulsion was combined with 2.1 parts rutile $TiO_2$ and 71.5 parts of common extender pigments to yield the base of reactive component of the adhesive. The curing agent component was prepared by dispersing 12 parts of Versamid® 140 in 23 parts of water and combining the resulting emulsion with 65 parts of a blend of extender pigments. The epoxy base component (3 parts) was combined with 2 parts of the curing agent dispersion to produce a white masonry pointing compound.[10]

Amine-cured epoxy emulsions which displayed good roller coating, spray, and brushing characteristics on concrete surfaces were derived from 100 parts of an epoxy resin emulsion (50% solids) which was combined with 10 parts of a conventional polyamide curing agent and 40 parts of an epoxy-modified aliphatic amine solution (50% solids) consisting of a 1/1 adduct of xylylenediamine and monoglycidyl ether. The epoxy resin/amine-modified epoxy combination possessed a working pot life of 1.5 hr and yielded coatings with good performance and durability characteristics for masonry surfaces.[11]

Coating compositions useful for application over inorganic building slates were derived from a formulation consisting of 5 parts Epikote 1001, 45 parts Epikote 828, 2 parts Emulgen® 905 nonionic surfactant, 0.5 parts carboxymethyl cellulose, 102.5 parts iron oxide, 102.5 parts talc, 2.5 parts propylene glycol, and 50 parts water. The epoxy resin base component (3 parts) was combined with 1 part of a modified aromatic polyamine curing agent, and the mixture was spray coated onto slate board. After curing the system for 30 min at 80°C, the emulsion coating was overcoated with a formulation consisting of 100 parts acrylic emulsion, 0.2 parts defoaming agent, and 0.3 parts antiseptic agent, and the two coatings were bonded together by post-curing the system for 30 min at 80°C. The resulting coating combination displayed a pencil hardness of 3H, a crosscut peel strength of 100/100, resisted discoloration, and provided excellent water and weather resistance.[12]

Weather-resistant compositions for use in architectural applications were formulated from 500 g of a bisphenol A-epichlorohydrin epoxy resin dissolved in 75 g of toluene which was dispersed at 70°C in 475 g of water containing 200 g of a 5% solution of poly(vinyl alcohol). The resulting 40% solids emulsion demonstrated a shelf stability of approximately six months, and when combined with sand and an amine crosslinking agent ($Me_2N(CH_2)_3NH_2$) yielded a product two to three times stronger than Portland cement mortar. The water resistance of the product could be increased by adding up to 60% coal or pine tar to the formulation.[13]

Coal tar/epoxy emulsions with improved corrosion-resistance properties for metal substrates were formulated from 65 parts Epikote 828 which was emulsified in 35 parts of water containing 3 parts of a nonionic surfactant consisting of a 60/40-block copolymer of polyethylene glycol/polypropylene glycol with a molecular weight of 2900 together with 0.2 parts of a similar Pluronic®-type nonionic emulsifier with a molecular weight of 3-4 million. The curing agent component was derived from the reaction product of 39 parts coal tar with 26 parts of a condensate of aminophenol and m-cresol, and the resulting coal tar/amine mixture was emulsified in water using 0.46% calcium dodecylbenzenesulfonate, 4.1% of the Pluronic-type nonionic emulsifier with a block copolymer molecular weight of 2900, and 0.75% of the Pluronic-type nonionic emulsifier with a block copolymer molecular weight of 3-4 million. The epoxy emulsion was combined with an equal amount of the coal tar/amine curing agent emulsion, and the mixture was coated onto steel sheets. After three hours of aging at room temperature, the coating developed a Sward hardness of 9.[14]

Epoxy coatings displaying good abrasion and chemical resistance were derived from a formulation consisting of 100 parts of an epoxy resin, from 2 to 8 parts of a Pluronic-type nonionic surfactant, 50 parts $TiO_2$, 80 parts barytes, 60 parts polyamide curing agent, and 300 parts water. Since the formulation did not contain organic solvents or coalescing agents, the epoxy/polyamide emulsion was recommended for applications requiring emission-free topcoats with high performance capabilities.[15]

Room temperature curable coatings with good flexibility and resistance properties were prepared from an epoxy emulsion consisting of 30 parts Epon 828, 0.7 parts of a Triton® nonionic surfactant, 3.5 parts of partially hydrolyzed poly(vinyl alcohol), and 66.5 parts water. The curing agent component was prepared by reacting 50 g of 4,4′-diaminodiphenylmethane with 62 g of glycidyl versatate for 30 min at 130°C and 60 min at 150°C. The resulting epoxy/amine adduct was mixed with 2 g of Pluronic F-108 emulsifier and dispersed in 50 g of water. The epoxy emulsion component (60 parts) was blended with 40 parts of the curing agent emulsion to yield a product which provided good coatings properties after drying for two days at ambient conditions.[16]

Rapid-curing epoxy coatings with good water resistance were prepared by emulsifying a mixture consisting of 100 parts of a poly(bisphenol A diglycidyl ether) resin, 40 parts of a phenol-formaldehyde/m-xylylenediamine copolymer, 10 parts of a polyamide curing agent, and 10 parts of an N-(2-hydroxyethyl)ethylenediamineoleic acid condensate. The resulting emulsion was applied to metal plates and yielded dry-to-touch coatings in 5 to 6 hr at room temperature. After aging for seven days, the coatings were found to display an Erichsen penetration of 8 mm and passed a 24 hr immersion in water. A control coating which substituted 10 parts of triethylenetetramine for the diamine/oleic acid condensate became tacky and whitened upon immersion in water.[17]

High-performance epoxy coatings with possible application as pipe linings were prepared by inverting 100 g of Epon 828 resin in a mixture consisting of 3 g Pluronic F-108 dissolved in 12 g of water. The paste-like mixture was subsequently reduced with an additional 39 g of water to yield an oil-in-water emulsion containing 65% solids and displaying a viscosity of 1500 cps. The Epon 828 emulsion (154 g) was combined with 1 g Nopcosant® L anionic surfactant, 63 g $TiO_2$, 43 g ASP-100 silica, 156 g $BaSO_4$, and 122 g water. The resulting paint was combined with a stoichiometric amount of a curing agent consisting of the reaction product of 94.5 g of p-chlorophenol and 94.5 g tetraethylenepentamine or the reaction product of 3 moles of diethylenetriamine with 1 mole of phenol and 3 moles of formaldehyde. The amine-cured coatings displayed good hardness together with improved water and solvent resistance.[18]

Amine-cured epoxy coatings with rapid-drying characteristics were formulated from a mixture of 100 parts Sumiepoxy® ELA 128 and 30 parts Sumicure® MT 1 which was combined with 5 parts of Emulgen A 90 emulsifying agent. The resin/surfactant mixture was inverted using 20 parts water, and the resulting emulsion was combined with 30 parts Sumiflex® 400 plasticizer. The modifier/epoxy blend (100 parts) was subsequently combined with 3 parts of an aminoethylpiperazine curing agent. The emulsion containing Sumicure MT 1 yielded coatings with a dry-to-touch time of 90 min while a control emulsion which omitted the Sumicure MT 1 curing agent yielded coatings with a dry-to-touch time of 150 min.[19]

Single or two-component epoxy emulsions with improved storage stability were prepared from 100 parts bisphenol A epoxy resin, up to 8 parts of an alkoxylated alkylphenol nonionic emulsifier, 50 parts $TiO_2$, 80 parts barytes, and 30 parts water. The resulting emulsion paint was combined with up to 60 parts of an aromatic amine or polyamide curing agent to yield products which were useful as coatings, adhesives, or foamed wood substitutes.[20]

Epoxy resin emulsions with improved low-temperature storage stability were formulated from 50 parts of a bisphenol A-epichlorohydrin resin containing 1.0% residual chlorohydrin units. The epoxy resin was dispersed at 60°C in 50 parts of water containing 2 parts of a nonylphenyl polyethylene glycol ether nonionic surfactant and 2 parts of a poly(vinyl alcohol) protective colloid. The emulsion prepared from the epoxy resin containing chlorohydrin units was found to display improved storage stability in comparison with a control emulsion prepared from an epoxy resin which did not contain chlorohydrin units.[21]

Storage-stable epoxy emulsions useful in applications such as coil coatings were prepared from 204 g of a bisphenol A-epichlorohydrin resin solution (85% in acetone) which was heated to 80°C and subsequently added over a 30 min period to a solution of 7 g of poly(vinyl alcohol) in 400 ml of water. The resulting emulsion was cooled and freed of acetone to yield a product which contained 30% vehicle solids, a particle size of <5 microns, and a viscosity of 12 cps at 25°C and which displayed <1% volume settling after standing at room temperature for five days. The nonionic, epoxy emulsion (33.3 g) was combined with 2 g of a methylolated melamine resin, applied to untreated aluminum sheets, and baked 30 min at 175°C to yield coatings with a mandrel flexibility of 38.1 mm, a reverse impact strength of 40 in.-lbs, and which displayed good water resistance.[22]

Semi-gloss enamel coatings demonstrating good performance characteristics in various industrial applications were prepared by combining an emulsion consisting of 56.8 parts Vanoxy® RD 707 (aliphatic glycidyl ether), 18.9 parts 2-[(nonylphenoxy)pentakis (ethyleneoxy)] ethanol, 18.9 parts 2-[(nonylphenoxy)nonadecakis(ethyleneoxy)] ethanol and 5.4% GE Silicone SR-82 with an emulsion prepared from 50.6 parts Vanoxy 128, 16.3 parts of a 1/1 ratio of the previous nonionic surfactants, 97.4 parts $TiO_2$, 45.2 parts magnesium silicate, and 182.1 parts water. The resulting blend of epoxy emulsions was found to provide protective coatings useful for application over paper, glass, metal, or wood substrates.[23]

Epoxy emulsions with improved storage stability at low temperatures and which yielded coatings with good resistance to corrosion and chemicals were derived from 19.6 parts Epikote 828, 0.4 parts Epiclon 800 (a bisphenol A-methylepichlorohydrin derivative), 10 parts iron oxide, 35 parts talc, 2 parts poly(ethylene glycol) monolaurate surfactant, and 34 parts water. The resulting emulsion displayed no agglomeration after six months of storage at 0 to 5°C and, when combined with a curing agent consisting of a dimer acid of 3,3′-oxybis(propylamine), yielded coatings which provided good chemical and water resistance properties.[24]

Epoxy emulsions with improved room temperature curing ability and which displayed an 11 to 12 hr pot life were prepared from 200 parts of a bisphenol A epoxy resin (epoxide equivalent weight of 186 to 192) which was dispersed in 195 parts of water containing a surfactant mixture consisting of 4.5 parts of an alkylphenol condensed with 40 moles of ethylene oxide and 1.2 parts of an alkylphenol condensed with 4 moles of ethylene oxide. The resulting emulsion (200 parts) was combined with 65 parts of a curing agent derived from 17.7% 4-(aminomethyl)octamethylenediamine, 68.1% bodied tall oil fatty acids, and 14.2% triethylenetetramine. The epoxy resin/amine curing agent combination was applied to steel sheets and yielded coatings which dried to touch in 6 hr and which displayed a pencil hardness of H, Erichsen penetration of >7 mm, and a falling dart impact strength (500 g, 1.27 cm) of >50 cm after curing for seven days at 25°C.[25]

Epoxy resin emulsions with good storage stability and improved thixotropic characteristics were formulated from 49 parts Epikote 828, 21 parts DER® 732 epoxy resin, 0.2 parts fungicide, 18.2 parts water, and an emulsifier combination consisting of 10.5 parts carboxymethyl cellulose (2% in water), 0.7 parts of a Pluronic-type ethylene oxide-propylene oxide block copolymer (HLB range of 16 to 20), and 0.4 parts of an alkylphenol poly(ethylene glycol) ethanol blend with an HLB range of 4 to 15 (0.2 parts Noigen® EA 137 and 0.2 parts Noigen EA 50). The combination of nonionic surfactants was found to improve the colloidal stability of the system and yielded an emulsion which was stable for >70 days. In addition to conventional amine-based curing agents, the emulsions could be optionally extended with a coumarone-indene resin.[26]

**Table 3—HLB Values of Surfactants Used to Prepare BPADG Emulsions**

| % Surfactant | HLB Range | Characteristics | Ref. |
|---|---|---|---|
| 10% poly(oxyethylene) benzylated phenyl ether | 16.6 | Water and salt-water resistant coatings | (27) |
| 10% poly(oxyethylene) benzylated phenyl ether<br>5% poly(ethylene glycol) nonylphenyl ether | 18.2<br>16.0 | Coating properties comparable to solvent-borne epoxy system | (28) |
| polyethylene-polypropylene block copolymer (HLB 16-20) poly(ethylene glycol) monoaryl ether (HLB 4-15) | 13-18 for the blend | Highly stable emulsion yielding high gloss coatings | (29) |
| 12.5% poly(ethylene glycol) nonylphenyl ether<br>5% poly(ethylene glycol) benzylphenyl ether | 12<br>18 | Water and salt-water resistant coatings | (30) |
| 1.2% poly(ethylene glycol) benzylphenyl ether | 18 | Chemically resistant coatings with good hardness | (31) |

## USE OF NONIONIC SURFACTANTS

Besides illustrating the methods of preparation, the compositional and formulation variables, physical characteristics, and applications of epoxy emulsions, the previous examples also demonstrate the nearly exclusive use of nonionic surfactants for the emulsification of bisphenol A diglycidyl ether epoxy resins (BPADG). Apparently, since the oxirane ring of BPADG systems is more reactive to nucleophilic addition than is the oxirane ring of, for example, glycidyl methacrylate systems,* the use of anionic surfactants for the emulsification of BPADG resins could ostensibly result in an even greater amount of oxirane loss than the 25% loss experienced in glycidyl methacrylate-based emulsions prepared with anionic surfactants.† Thus, for epoxy resins containing fairly reactive oxirane groups, the undesirable loss of oxirane content and/or premature gelation may be minimized by employing nonionic surfactants or nonionic protective colloids as the emulsifying agents for BPADG resins.

While relatively stable epoxy emulsions have been prepared by using a variety of compositionally different nonionic surfactants,[6,7,21,26] such factors as the chemical nature of the epoxy resin, the rheological and viscosity characteristics of the resin, the preparation method and temperature, shearing time and rate, surfactant concentration, and the hydrophilic lipophilic

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*See reference 131 in Part IV (JCT, October 1978, p 98).

†See reference 129 in Part IV (JCT, October 1978, p 98).

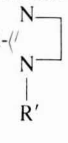
balance (HLB) of the surfactant all influence the storage stability of these systems. *Table* 3 lists the HLB values and the type of surfactants commonly used in the preparation of conventional BPADG emulsions.

The examples presented in *Table* 3 illustrate in part that BPADG resins can be emulsified using conventional nonionic surfactants with an HLB range of from 15 to 18. Surprisingly, and with certain misgivings, these examples also suggest that coatings with properties comparable to solvent-borne epoxy systems or coatings with good water and salt-water resistance can be derived from epoxy emulsions containing inordinately high concentrations [15%[28] and 17.5%[30]] of surfactants.

## USE OF CATIONIC SURFACTANTS

As the quantity of surfactant required to achieve adequate colloidal stability is controlled in part by the efficiency of the emulsifier, other types of surfactants have been investigated in order to prepare stable epoxy emulsions which yield coatings possessing reduced levels of contaminating emulsifier. One approach to this end has been the use of cationic surfactants such as N-tallow-N,N,N′,N′-pentamethyltrimethylenediammonium dichloride which is capable of reacting with the epoxy resin and yields coatings with the surfactant as an integral part of the crosslinked matrix.[32] For example, cationic epoxy emulsions with improved colloidal stability were prepared from 65 parts of a bisphenol A epoxy resin which was dispersed at 66°C in water containing 2.5%, based on total formulation, of an N-[9-(10-phenyl)octadecyl]-N,N,N′,N′-pentamethyltrimethylenediammonium dichloride cationic surfactant ($Me(CH_2)_7C_2H_3Ph(CH_2)_8\overset{+}{N}Me_2(CH_2)_3\overset{+}{N}Me_3\,2Cl^-$). The resulting epoxy emulsion (70% by volume) was combined with 30% by volume of a polyamide emulsion consisting of a dimer fatty acid/diethylenetriamine condensation product which was emulsified in water containing 2% of the above cationic surfactant. The epoxy resin/polyamide emulsion combination was found to be stable for over a six month period and when cured for 30 min at 149°C yielded coatings suitable for protecting metal surfaces.[33]

Cationic epoxy emulsions useful as a finishing treatment for glass fibers which were formulated into laminates were obtained from an Epon 828 epoxy resin, 0.3 to 2% of a coupling agent [$H_2N(CH_2)_3Si(OEt)_3$], 0.1 to 2% of a glass lubricant, and up to 15%, based on total resin, of an emulsifier combination consisting of a Pluronic-type nonionic surfactant, an amide surfactant, and a cationic surfactant. The amide surfactant contained tertiary amine sites, $C_8H_{17}CH=CH(CH_2)_7CONHC_2H_4NEt_2$, while the cationic surfactants were based upon imidazoline derivatives with the preferred composition of R-



where R=8-heptadecen-l-yl and R′=Et or where R=$C_{16}H_{33}$ and R′=$CH_2CH_2OH$. The resulting emulsions possessed a viscosity of <100 cps at 20°C and were claimed to be stable. However, such characteristics as the degree of sedimentation, freeze-thaw, and shear stability, or the effects of aging were not described.[34]

**Table 4—Typical Storage Stability Times For BPADG Emulsions**

| Surfactant Type | Storage Stability (days) | Applications or Characteristics | Ref. |
|---|---|---|---|
| 3% poly(vinyl alcohol), 89% saponified<br>3% conventional nonionic surfactant | 60 | After storage, particle size increased to 500 μm | (36) |
| barium dodecylbenzenesulfonate<br>nonylphenol-ethylene oxide adduct | 90 | — | (37) |
| 4% polyethylene glycol monoabietate<br>1.5% nonylphenol poly(ethylene oxide) ethanol | 84 | Solvent resistant coatings | (38) |
| 0.7% nonylphenol poly(ethylene oxide) ethanol<br>0.43% sorbitan monolaurate poly(oxyethylene) ether<br>0.29% lauryl poly(oxyethylene) ether<br>0.43% poly(ethylene glycol) distearate | 60 | Chemically resistant coatings | (39) |
| 2% sodium alginate<br>0.5% nonionic surfactant | 90 | High shear strength adhesives | (40) |
| 30% poly(ethylene glycol) esters with OH value of 20-500, acid value of 10-200 mg KOH/g resin, and mol. wt. of 3000-10,000. | 30 | Flexible coatings | (41) |

### Typical Stability Characteristics

Even though BPADG emulsions are usually depicted as possessing good or improved colloidal stabilities,[35] these post-emulsified systems rarely possess the stability characteristics displayed by latexes prepared by means of emulsion polymerization methods. *Table* 4 illustrates typical storage stability times for BPADG emulsions prepared with various types of conventional surfactants.

Besides the baneful effects which the contaminating surfactants have on the physical characteristics of these systems, the relatively short-term storage stability, as illustrated in the above examples, is an additional factor which has restricted the commercial growth and application of BPADG emulsions.

### Other Types of Epoxy Resins

In addition to the post-emulsification of BPADG resins, other epoxy-containing compounds have been investigated by the industry as possible candidates for the development of stable epoxy emulsions which yield high-performance coatings. For example, 100 parts of an epoxidized, formaldehyde cresol novalak resin were condensed at 55°C with 10 parts of 2-($\beta$-dimethylaminoethoxy)-4-methyl-1,3,2-dioxaborinane producing an epoxy resin containing an internal surfactant constituent. The resulting epoxy resin-boric acid ester adduct was spontaneously emulsified upon the addition of 165 parts of water generating an emulsion with a particle size of less than 0.9$\mu$m. The characteristics of the emulsion together with the chemical resistance properties of its coatings are presented in *Table* 5.

The boric acid ester-modified epoxy emulsion required no additional curing agent as the tertiary amine moiety of the adduct promoted self-condensation of the oxirane groups at elevated temperatures. The chemical resistance properties of an epoxidized cresol-novolak resin (Kopox® 357) containing 16 and 50 weight-percent boric acid ester adduct are presented in *Table* 6. The emulsion was coated onto steel panels at 75 $\mu$m wet film thickness, cured a total of two hours at 110°C, one hour at 160°C, two hours at 190°C, and five hours at 250°C, and did not contain an external curing agent.

The epoxy resin-boric acid ester adduct emulsions could also be combined with such conventional curing agents as tetramethylethylenediamine or tetraethylenepentamine to yield coatings which dried within 24 to 48 hr to water and solvent resistant products.[42]

A silicone-epoxy emulsion useful for improving the tensile, compressive, and flexural strengths; trowelability; and surface laitence of hydraulic cement compositions was prepared by.condensing 257 g Dow Corning silicone resin 6188 (approximate molecular weight of 750) with 625 g of a bisphenol A diglycidyl ether having a molecular weight of approximately 500 (Resypox 1634) over a three hour period at 50 torr vacuum and 180°C. After the initial reaction period, the resulting product was cooled to 100°C, combined with 300 g of butanediol diglycidyl ether, and further condensed by heating to 180°C and at 50 torr vacuum for two hours. The final product possessed an epoxy value of 3.9 equivalents per kilogram and was emulsified in water using up to a stoichiometric amount of a poly(ethylene glycol)/amine curing agent. The emulsifier/curing agent combination was prepared by reacting 250 g diethylenetriamine with 722 g of a diglycidyl ether of poly(propylene glycol)-425 over a four hour period at 65°C. Similar epoxy resin/curing agent blends displayed a pot life of two hours and when combined at the 5% level with portland cement yielded products with a 200% increase in compressive strength and 50% increase in tensile strength as compared to the corresponding unmodified concrete. Other resins which formed stable emulsions with the emulsifier/curing agent resin included tetrabromo bisphenol A epoxy resins and epichlorohydrin-novolac resin condensates.[43]

**Table 5—Properties of an Epoxidized, Formaldehyde/Cresol/ Novalak Resin Modified with a Boric Acid Ester**

| Characteristic | Appearance or Property |
|---|---|
| Room temperature stability after seven weeks | Some precipitate formation |
| Storage stability after three months of aging at 4°C | No precipitate |
| Viscosity | |
| Initial | 58,000 cps |
| After 32 days | 7,800 cps |
| Epoxide Content (wt.%) | |
| Initial | 2.94 |
| Aged 4 days | 2.91 |
| 11 days | 2.86 |
| 32 days | 2.67 |
| Chemical resistance of coatings after 500 hr immersion in:[a] | |
| toluene | No effect |
| methyl isobutyl ketone | No effect |
| 5% aq. NaOH | Hard and intact |
| 5% aq. HC1 | Development of pinholes |

(a) Coatings were applied to steel panels at 75 $\mu$m wet film thickness, dried one hour at ambient, and cured for one hour at 150°C.

**Table 6—Chemical Resistance Properties Of an Epoxidized Cresol/Novolak Resin Modified With a Boric Acid Ester[a]**

| | Epoxidized cresol/novolak resin containing | | | |
|---|---|---|---|---|
| | 16 wt. % Boric Acid Ester | | 50 wt. % Boric Acid Ester | |
| | % wt. increases after | | % wt. increases after | |
| Type of Immersant | 7 days | 30 days | 7 days | 30 days |
| Acetone | 1.72 | disintegrated | 0.85 | 3.8 |
| Ethylene Dichloride | 1.70 | — | 0.60 | 3.8 |
| Distilled Water | 0.71 | 1.54 | 0 71 | 1.54 |
| 10% NaOH | 0.56 | 1.20 | 0.59 | 1.26 |
| 30% NaOH | 0.19 | 0.46 | 0.20 | 0.54 |
| 10% HCl | 0.54 | 1.47 | 0.54 | 1.20 |
| 30% HCl | 3.23 | 6.5 | 0.82 | 2.3 |
| 10% $H_2SO_4$ | 0.63 | 1.34 | 0.65 | 1.37 |
| 30% $H_2SO_4$ | 0.42 | 0.91 | 0.43 | 0.91 |

(a) 2-($\beta$-dimethylaminoethoxy)-4-methyl-1.3.2-dioxaborinane.

Polyether epoxy/bisphenol A epoxy emulsion blends useful in applications demanding highly flexible coatings were formulated from 90 parts of a polyether epoxy emulsion consisting of 50 parts EP-4000 epoxy resin, 1.5 parts poly(vinyl alcohol), 1.5 parts surfactant, and 50 parts water which was combined with 10 parts of a bisphenol A epoxy emulsion consisting of 50 parts Epikote 828, 1.5 parts poly(vinyl alcohol), 1.5 parts surfactant, and 50 parts water. The resulting epoxy emulsion blend (100 parts) was catalyzed with 16 parts of a polyamide curing agent, and the epoxy resin/polyamide mixture was molded into a 0.5 mm crosslinked film which displayed improved folding endurance as com-

Table 7—Amine-Based Curing Agents for BPADG Emulsions

| Curing Agent Composition | Surfactants used for emulsification | Characteristics | Reference |
|---|---|---|---|
| 2/1/2-adduct of xylylenediamine/Epikote 828/phenyl glycidyl ether | Pluronic nonionics, poly(oxyethylene) sorbitan monooleate, poly(vinyl alcohol) | Extended pot life, enhanced flexibility and water resistance | (48) |
| Butanediol diglycidyl ether condensate with polyamines | none | Reactive emulsifiers for epoxy resins | (49) |
| Condensate of trimethylol propane glycidyl ether/diethylene triamine/soya ester-styrene reaction product | none | Coatings with good hardness, strength, and drying characteristics | (50) |
| Condensate of phenyl glycidyl ether/triethylene tetramine/ polymd. tall oil fatty acids | — | Long term emulsion stability | (51) |
| Aromatic polyamine | Emulgen 81 nonionic surfactant | Flexible, chemically resistant coatings | (52) |
| Polyamidoamine emulsions | Polyacrylamide or poly(vinyl pyrrolidinone) | Several months storage stability | (53) |

pared to a control film derived predominantly from a bisphenol A epoxy emulsion.[44]

Polyether epoxy emulsions which provided coatings with good hardness and water resistance qualities were formulated from 92 parts EP-4000 polyether epoxy resin, 3.5 parts poly(ethylene glycol) nonylphenyl ether surfactant (Emulgen 950), 1.5 parts Emulgen 905, 195 parts water, and 108 parts of an amine-type epoxy curing agent (EH-230). The resulting emulsion was combined with an equal portion of an epoxy emulsion consisting of 200 parts Epon 828, 3.5 parts Emulgen 950, 1.5 parts Emulgen 905, and water to yield a blend which provided coatings that displayed a pencil hardness of 3H and a water absorption of 0.6% after seven days of immersion in water. At 20°C and 60% relative humidity, the coatings cured in approximately eight hours.[45]

Cycloaliphatic epoxy emulsions providing increased wash and wear properties and decreased yellowing in textile applications were formulated by dispersing 15% by weight vinyl cyclohexane dioxide epoxy resin in water containing 2% zinc fluoroborate curing agent and 0.2% ZnO stabilizer. A cotton fabric impregnated with 7.8% of the emulsion and cured for 2.5 hr at 77°C demonstrated a wash and wear rating of 5, a crease resistance of 69% (ASTM D-1295-53T), and a 57% increase in strength as compared to the untreated fabric. The wash and wear characteristics of the coating could be improved by including a complementary curing agent such as dimethylolethyleneurea in the epoxy emulsion formulation. Other epoxy resins which were also useful for improving the wash and wear characteristics of fabrics included diethylene glycol mono(2,3-epoxy-2-methylpropyl)ether and diethylene glycol bis(2,3-epoxy-2-methylpropyl)ether while other suitable epoxy curing agents included $NaBF_4$, $BF_3OEt_2$, and $AlCl_3$.[46]

Cycloaliphatic epoxy emulsions providing heat-curable coatings for nontextile applications were formulated from 600 parts of an epoxide resin which was dispersed at 50°C in 216 parts of water containing 24 parts of poly(vinyl alcohol) and 10.4 parts poly(ethylene oxide) cetyl alcohol nonionic surfactant. The resulting emulsion (8.4 parts) was combined with 90 parts of an aminoplast emulsion consisting of 320 parts of a dibutyl methylolmelamine solution (75% in butanol) dispersed in 112 parts water using a poly(oxyethylene)/ hexamethylene diisocyanate/hydroabietyl alcohol adduct (48 parts) as an emulsifying agent. The epoxy resin/melamine resin emulsion blend was adjusted to a pH of 7 with triethanolamine, combined with 0.5 parts $Zn(BF_4)_2$, applied to aluminum sheets at a thickness of 50 to 100 $\mu$m, and dried for 10 min at 150°C to yield coatings with good to very good resistance to white spirits and displaying an Erichsen value of 3.1 to 3.4. Other epoxy compounds that were suitable for the application included 8,9-epoxy-3-(3,4-epoxycyclohexyl)-2,4-dioxaspiro [5.5] undecane, (3,4-epoxy-5-methylcyclohexyl)methyl-3,4-epoxy-5-methylcyclohexanecarboxylate, and an epoxidized butadienestyrene copolymer.[47]

## TYPES OF CURING AGENTS

Besides the various curing agents discussed in the previous examples, a myriad of other amine-containing compounds have been investigated as crosslinking agents for epoxy compounds. While many of these curing agents have been classified as being less effective or desirable than fatty acid-amine adducts or polyalkylenepolyamines, which serve as standards of comparison, the usefulness of each epoxy resin/amine curing agent combination must be established through empirical methods since the principles and technologies

Table 8—Performance Characteristics Of a Single-Component Epoxy Emulsion in Comparison With a Solvent-Borne Epoxy/Polyamide System[a]

| Coating Resistance to | Epoxy resin/curing agent-emulsifier combination[b] | Solvent-borne Epoxy/polyamide combination[b] |
|---|---|---|
| Water at 25°C | 8 | 8 |
| Water at 66°C | 7 | 7 |
| 10% NaOH | 10 | 10 |
| 10% HCl | 10 | 8 |
| 10% HOAc | 9 | 6 |
| Xylene | 10 | 10 |
| Methyl ethyl ketone | 6 | 8 |
| Impact resistance | | |
| Forward (in-lbs) | 20 | 30 |
| Reverse (in-lbs) | 15 | 10 |
| 60° gloss | 100% | 100% |

(a) Coatings were applied to aluminum; however mode of application, coating thickness, flash-off and cure times were not specified.
(b) Coatings were rated 10 for no effect and 0 for failure.

established with similar solvent-based epoxy/amine combinations may not directly transfer to mixtures of the same components which are in colloidal form. *Table* 7 illustrates typical amine-based curing agents along with the advantages associated with each combination.

While successful products have been obtained by combining epoxy resin emulsions with amine curing agents which were developed primarily for conventional, solvent-borne systems, additional performance and marketing advantages may be achieved by designing the composition of the curing agent in accord with the requirements set by the environmental and two-phase nature of the colloidal system. For example, protective coatings with improved resistance properties were obtained from a single component, 84.5/15.5-DER 331/DER 732 epoxy emulsion which utilized a water-soluble, amine-functional emulsifying agent which also served as the curing agent upon coalescence of the particles.

The curing agent/emulsifier combination was prepared from a carboxyl-functional, acrylic solution copolymer which was condensed with a hydroxyl-functional polyamine and subsequently neutralized with HCl to yield a water soluble compound possessing cationic, amine hydrochloride sites. Presumably, the hydrophobic component of the acrylic backbone becomes interfaced with the epoxy resin while the cation groups become oriented in the aqueous phase. Thus, the amine sites are relatively isolated from the epoxy resin, and the epoxy resin/curing agent-emulsifier combination undergoes very little premature gelation in the emulsion form. However, as the particles coalesce upon drying, the cationic sites become comingled with the epoxy resin, and crosslinking occurs in the normal fashion. *Table* 8 illustrates the performance characteristics of pigmented coatings derived from an epoxy paint consisting of 93 parts DER 331 epoxy resin, 17 parts DER 732 epoxy resin, 300 parts $TiO_2$, 3 parts defoamer, 40 parts solvent, 20 parts Dowanol® DPM glycol ether, and 250 parts of the cationic amine curing agent-emulsifier (49% solids).

The chemical resistance properties of the epoxy resin/curing agent-emulsifier combination could be improved by replacing all or part of the DER 732 epoxy resin with DER 331 epoxy resin. However, such substitutions usually resulted in a loss of impact resistance, and the utilization of such epoxy resins as DER 337 or DER 671 was recommended as a somewhat more auspicious approach to formulating chemically resistant, flexible, epoxy emulsion coatings. While epoxy emulsions prepared with the curing agent-emulsifier component were claimed to possess long pot lives, such properties as the loss of oxirane content with time, the formation of gel with time, the mechanical, freeze-thaw, and storage stabilities of the system were not described.[54]

## Formulation Variables

Obviously, such factors as freeze-thaw stability during transport and cold-weather storage, shear stability during paint manufacturing and application, storage stability prior to application, as well as achieving the performance characteristics associated with solvent-borne epoxy systems or other high-performance, ecologically-compatible systems were major determinants governing the commercial acceptance and success of epoxy emulsion systems. Recently, several of the variables influencing the colloidal and performance characteristics of epoxy emulsions were disclosed in a study which investigated the usefulness of epoxy emulsions for military aircraft applications. The primary emphasis of the investigation was to assess the feasibility of developing epoxy emulsions whose physical and resistance properties were comparable to those of their solvent-borne counterparts. However, since films cast from colloidal dispersions frequently retain microstructures reminiscent of their equilibrium packing orders while solvent-cast films usually achieve an intimate comingling of epoxy resin and curing agent, the use of emulsions with fine particle sizes was recognized

Table 9—Critical Particle Size for Settling Upon Standing[a]

| Particle Density, g/cm³ | Viscosity of medium, cps | Particle Diameter, μm |
|---|---|---|
| 1.10 | 0.895 (water at 25°C) | 0.44 |
| 1.10 | 1.0 | 0.46 |
| 1.10 | 2.0 | 0.65 |
| 1.10 | 5.0 | 1.03 |
| 1.10 | 10.0 | 1.46 |
| 1.15 | 0.895 | 0.36 |
| 1.15 | 1.0 | 0.38 |
| 1.15 | 2.0 | 0.53 |
| 1.15 | 5.0 | 0.84 |
| 1.15 | 10.0 | 1.19 |

(a) Calculations were based upon settling of particles in a medium with a density of 1.00 g/cm³.

Table 10—Effect of Several Emulsification Variables on the Particle Size of Epoxy Emulsions[a]

| Epoxy/Diluent Ratio[b] | Emulsification Temperature (°C) | Emulsification Method | Particle Size Description[c] | |
|---|---|---|---|---|
| | | | Relative Distribution | Max. Size Observable ($\mu$m) |
| 50/50 828/PGE | 63 | Simple stirring | Very broad | 75 |
| 50/50 828/PGE | 63 | Ultrasonic irradiation | Moderately narrow | 3.8 |
| 68/32 828/PGE | 63 | Simple stirring | Very broad | 140 |
| 68/32 828/PGE | ambient | Ultrasonic irradiation | Moderately narrow to intermediate | 5.2 |
| 75/25 828/PGE | 63 | Simple stirring | Very broad | 48 |
| 75/25 828/PGE | 63 | Ultrasonic irradiation | Moderately narrow | 3 |
| 75/25 1001/PGE | 63 | Simple stirring | Very broad | 60 |
| 75/25 1001/PGE | 63 | Ultrasonic irradiation | Moderately narrow to intermediate | 4.5 |

(a) Emulsions were prepared using the 1/2-sodium lauryl sulfate/cetyl alcohol combination.
(b) 828=Epon 828, 1001=Epon 1001, PGE=phenyl glycidyl ether.
(c) Particle sizes were estimated from electron photomicrographs.

as being an essential element for overcoming the property differences arising from the compositional heterogenities inherent in two-component emulsion systems.

In addition to yielding films of a more uniform composition, a second advantage for using emulsions with fine particle sizes emanated from the need to attain improved shelf stability. According to Stokes' Law, the rate of sedimentation varies directly with the square of the particle diameter; thus, at a critical particle size, the rate of sedimentation exceeds the limit which is acceptable for adequate shelf stability. This point is illustrated in *Table* 9 which describes particle sizes as a function of the viscosity of the medium whereupon settling occurs.

Since most epoxy resins have densities between 1.10 and 1.15 g/cm$^3$, the above data suggest that settling due to sedimentary factors will be minimized by achieving particle sizes below 0.36 $\mu$m to 0.44 $\mu$m unless the viscosity of the medium is increased by a false-bodying agent such a poly(vinyl alcohol) or hydroxyethyl cellulose.

A third advantage for using emulsions with fine particle sizes emanated from a consideration that a direct relationship exists between the physical properties of the developing film and the degree of coalescence. This relationship can also be discussed in terms of the shear modulus of the particle; for if the shear modulus of the polymer is too high for the emulsion particle size, particle coalescence may be incomplete and, as a result, the ultimate film properties may never develop. The greater the shear modulus of the polymer, the smaller the particle size must be in order for the particles to coalesce upon drying. Particle diameters of 0.1 $\mu$m, 1.0 $\mu$m, and 10 $\mu$m require shear moduli below a maximum of 1600 psi, 160 psi, and 16 psi, respectively, if particle deformation and film coalescence are to occur. Thus, for harder particles a decrease in particle size may result in an increase in coalescing ability and, consequently, provide films of greater integrity.

In light of these considerations, preliminary evaluations were devoted to achieving fine particle size emulsions through the optimization of the emulsifier composition and the emulsification technique. For the initial survey, a surfactant combination of 1/2 ratio of sodium lauryl sulfate/cetyl alcohol was selected for evaluation since previous work with this surfactant combination produced styrene monomer emulsion droplets of about 0.2 $\mu$m in diameter. The epoxy resins (Epon 828 and Epon 1001) were dissolved in phenyl glycidyl ether (PGE) in order to produce solutions with low enough viscosity for efficient emulsification. Various epoxy resin/reactive diluent combinations were emulsified by using simple stirring or ultrasonic irradiation, and the results of this study are presented in *Table* 10.

Even though variations in the viscosity of the resin phase or changes in the emulsification temperature did not uncover guideline relationships, the previous data clearly indicate the advantages offered by using ultrasonic irradiation as a means of emulsification. For the 68/32-Epon 828/PGE combination, ultrasonic irradiation effected a 27-fold decrease in particle size in comparison with simple stirring.

Table 11—Effect of Several Emulsification Variables on the Particle Size and Stability of Epoxy Emulsions

| Polyamide/Solvent Ratio[a] | Surfactant Combination[b] | Emulsification Method | Stability and Particle Size |
|---|---|---|---|
| 70/30<br>V-115/Xylene | Sodium lauryl sulfate/cetyl alcohol | Simple stirring | Formed pastes |
| 70/30<br>V-115/Xylene | HDTMAB/CA | Simple stirring | 1 day stability;<br>size = 0.5 to 50 μm |
| 70/30<br>V-115/Xylene | HDTMAB/CA | Ultrasonic irradiation at 60°C | Slightly greater than 1 day stability;<br>size = 0.5 to 20 μm |
| 18.7/32.5/20/20/8.8<br>V-115/toluene/IPA/BA/Xylene | HDTMAB/CA | Homogenization | 60 day stability;<br>after 30 days aging, size increased up to 10 μm |
| 18.7/32.5/20/20/8.8<br>V-115/toluene/IPA/BA/Xylene | HDTMAB/CA | Ultrasonic irradiation | Good stability after 5 days; sizes up to 10 μm |
| 80/8/5/5/2<br>V-115/toluene/IPA/BA/Xylene | 2%-HDTMAB/<br>4%-CA | Homogenization | Stable up to 7 days;<br>sizes up to 13 μm |
| 80/8/5/5/2<br>V-115/toluene/IPA/BA/Xylene | 2.7%-HDTMAB/<br>5.4%-CA | Homogenization | Stable up to 3 weeks;<br>sizes up to 8 μm |

(a) V-115 = Versamid 115, IPA = isopropanol, BA = n-butanol.
(b) HDTMAB = hexadecyltrimethylammonium bromide, CA = cetyl alcohol.

In order to generate suitable curing agent emulsions, several attempts were made to emulsify a Versamid 115 polyamide curing agent using the 1/2-sodium lauryl sulfate/cetyl alcohol emulsifier mixture; however, due to an interaction between the amine and the surfactant, only pastes were formed. This impasse was overcome by substituting hexadecyltrimethylammonium bromide for sodium lauryl sulfate in the surfactant mixture. Since the resulting cationic emulsion of Versamide 115 displayed good stability, the same hexadecyltrimethylammonium bromide/cetyl alcohol combination was used for a subsequent study which evaluated the parameters associated with achieving fine particle size, cationic epoxy emulsions.

Because the viscosity of the Epon 1001/PGE combination was inordinately high for efficient emulsification, a 50/50-toluene/methyl isobutyl ketone (MIBK) mixture was substituted for PGE in order to reduce the viscosity of the resin and yet prepare higher solids solutions. The Epon 1001/toluene/MIBK solutions were employed in a ladder study which evaluated the effects which varying ratios of hexadecyltrimethylammonium bromide to cetyl alcohol and varying concentrations of total surfactant to epoxy resin had on emulsion particle size and stability. The emulsions were prepared using simple stirring followed by ultrasonic irradiation of 25 parts of a 50/25/25-Epon 1001/toluene/MIBK mixture in 75 parts of water at 60°C. The surfactant combinations which yielded the most stable epoxy emulsions (12% solids) consisted of 3.2/12.8, 3.2/9.6, 3.2/6.4, 2.6/5.4, 4/4, 5.4/2.6, and 6.2/2 weight percent ratios (based on epoxy solids) of hexadecatrimethylammonium chloride to cetyl alcohol. Emulsions prepared from 1.4/2.6, 2/6, or 1.6/6.4 weight percent ratios of these emulsifiers were not stable. These results combined with an optical microscopic evaluation of the emulsion droplet sizes at various aging times indicated that emulsions of the 50/25/25-Epon 1001/toluene/MIBK mixture required at least 2.6% hexadecyltrimethylammonium bromide and 5.4% cetyl alcohol in order to retain acceptable stability.

The 50/25/25-Epon 1001/toluene/MIBK mixture was then emulsified in a Manton-Gaulin homogenizer using the hexadecyltrimethylammonium bromide/cetyl alcohol emulsifier combination to generate an emulsion with a droplet size of 1 μm or smaller after one pass and an even further reduction in droplet size after three passes through the homogenizer. However, little difference was observed between the particle size produced by homogenization and that produced by ultrasonic irradiation. These emulsions could be concentrated to contain 25% epoxy resin solids by vacuum steam distillation of the solvents without significant alteration in particle sizes.

Electron microscopic evaluation of a solvent-stripped, Epon 1001 emulsion which had been hardened in colloidal form with ethylenediamine revealed the existence of a broad distribution of particle sizes ranging from 0.03 μm to 0.2 μm. Similar techniques were employed to elucidate the parameters involved in generating stable emulsions of Versamid 115 curing agent, and the major results of this survey are presented in *Table* 11.

These results suggest that under a given set of conditions Versamid 115 emulsions produced by means of homogenization tend to display greater stability (up to 60 days) than emulsions produced by means of ultrasonic irradiation (only greater than five days). The most stable emulsions contained low concentrations of the polyamide resin or higher concentrations of surfactant. Since each emulsion possessed relatively large particle sizes (8 μm or greater), the maximum shelf stability of these emulsions was limited to approximately 60 days.

In order to produce higher solids, Versamid 115 emulsions, an attempt was made to vacuum steam distill the solvents used to carry the polyamide resin. However, upon concentration, the emulsion became viscous and turned yellowish and translucent, indicating a spontaneous breakdown of the emulsion droplets to an even smaller size. These products could still be combined with the cationic, epoxy emulsion without complications to yield a catalyzed blend.

In order to evaluate the performance differences between coatings derived from emulsified systems and their solvent-borne counterparts, some of the more propitious epoxy resin and polyamide emulsions were combined in varying proportions, applied to aluminum panels, and cured either at room temperature or at temperatures up to 55°C. The combinations which were evaluated included cationic emulsion blends of Epon 1001/Versamid 115, Epon 1001/Genamid® 250, Epon 828/Versamid 115 together with solvent-borne mixtures of Epon 828/Versamid 115 and Epon 1001/Versamid 115. The coatings were rated for appearance and compatibility, and curing ability as inferred from hardness and adhesion. Unfortunately, such coatings properties as impact resistance, flexibility, gloss, tensile strength, elongation, and chemical resistance were not described. The main conclusions drawn from this survey were as follows:

(1) The optimum blend of epoxy resin to curing agent was found to be 2/1 for cationic emulsions of Epon 1001/Versamid 115 and Epon 1001/Genamid 250 and 1/2 for cationic emulsions of Epon 828/Versamid 115.

(2) The film properties of the Epon 1001/Versamid 115 emulsions were judged to be slightly better than those of the Epon 828/Versamid 115 emulsions and equivalent to those of the Epon 1001/Genamid 250 emulsions.

(3) The film properties of the solvent-borne Epon 1001/Versamid 115 system were judged to be slightly better than those of the Epon 828/Versamid 115 system.

(4) Emulsion cast films cured at higher temperatures generally displayed better film properties than those cured at room temperature. However, several room temperature cured films cast from solvent-stripped emulsions which employed the optimum ratio of epoxy resin to curing agent displayed good coatings properties while those cured at room temperature for longer times (36 days) were almost equivalent to or at times slightly superior to those cured at the higher temperatures.

(5) Mixtures of epoxy resin emulsions and polyamide emulsions displayed good coalescing ability producing transparent films at room temperature.

(6) The overall properties of the best emulsion-cast films were rated as being nearly equivalent to those of the best solvent-cast films.

Moduli versus temperature measurements conducted on emulsion-cast films derived from 5/2, 5/1, and 2/1 blends of Epon 1001/Versamid 115 together with solvent-cast films derived from a 1/1 blend of Epon 828/Versamid 140 also indicated some similarity in the properties of emulsion and solution cast systems.[55]

The difficulty of developing epoxy emulsions with the performance characteristics of solvent-borne epoxy systems was shown to arise, in part, from the morphological differences in microstructure which exist between films cast from emulsion and solution forms. In this investigation, the morphological changes which occur during film coalescence were monitored by means of scanning electron microscopy for coatings derived from an Epon 1001 epoxy emulsion which was combined in stoichiometric proportions with three curing agent emulsions prepared from Versamid 115 polyamide resin, a mixture of Versamid 115 and Dion® DPM-3-800 mercaptan-terminated polymer, and a 96/4-Epon 1001/Bisphenol A mixture.

The emulsification procedure was similar to the method employed in the previous study[55] in that the resin/solvent mixture was dispersed in an aqueous solution containing a surfactant mixture of hexadecyltrimethylammonium bromide and co-emulsifier (presumably cetyl alcohol), and the resulting crude emulsion was further homogenized in a Manton-Gaulin Micron Disperser. The homogenized emulsions were concentrated by vacuum steam distillation of the carrier solvents to yield Epon 1001 and 96/4-Epon 1001/Bisphenol A emulsions with about 40% solids and Versamid 115 and Versamid 115/Dion DPM-3-800 emulsions with about 15% solids. The concentrated emulsions were storage stable for more than two years and demonstrated no signs of aggregation or phase separation.

Particle size determinations using scanning electron photomicrographs of the Epon 1001 emulsion whose particles were hardened in colloidal form with ethylenediamine showed discrete spheres of from 0.03 to 0.2 μm in diameter while the Versamid 115 emulsion particles appeared as discrete spheres of from 0.02 to 0.03 μm in diameter connected with strands of amorphous polymer or coalesced particles of even smaller sizes. The amorphous polymer phase was postulated to arise from a solubilization of the polyamide resin during or after dispersion.

When stoichiometric quantities of the Epon 1001 emulsion and Versamid 115 emulsion were combined, the viscosity of the mixture was observed to decrease with time. Examination of the blend using transmission electron microscopy and a cold-stage technique revealed that after 30 min of aging at room temperature the Versamid 115 particles clustered around the Epon 1001 particles forming aggregates of several Epon 1001 particles held together with Versamid 115 particles. The aggregation of the two systems effectuated a particle size increase which reduced the viscosity of the latex mixture.

The film forming mechanism of two-component emulsion systems was postulated to take place in accord with a three-stage model whereby (1) physical contact occurs between Epon 1001 and Versamid 115 particles by coalescence during film formation, (2) reaction takes place at the interface between Epon 1001 and Versamid 115 particles forming a crosslinked polymer barrier, and (3) diffusion of the Versamid 115 phase through the crosslinked interfacial layer into the core of the Epon 1001 particles takes place causing further curing of the system.

In accord with this model gradual coalescence observations, which reflected the comingling of the polyamide curing agent phase with the epoxy resin phase, were conducted on coatings of the Epon 1001/Versamid 115 latex blend after aging for 15 hr, 7.5 days, and 15 days, respectively, at room temperature. The particle contours in the film were observed to disappear gradually, and after 15 days of aging the film surface was relatively smooth. However, if the Epon 1001/Versamid 115 emulsion mixture was allowed to age for one week before application, the coatings which resulted were observed to possess remnants of the original surface packing order up through the 33 days of examination.

The formation of a crosslinked interfacial layer which hindered further gradual coalescence of the dried film was also observed for the Epon 1001-bisphenol A emulsion blended with the Versamid 115 emulsion. Films of this mixture retained the particle contours up to 11.5 days with only slight change. In this case, the presence of the phenol groups of bisphenol A accelerated the reaction between the epoxide and amine groups causing a rapid development of the crosslinked interfacial layer.

The internal structure of the Epon 1001/Versamid 115 emulsion coatings was investigated from microtomed sections which had been stained with osmium tetroxide vapors. Osmium tetroxide would preferably add to the residual double bonds of Versamid 115 thus labeling areas of the sample which contained the curing agent. Areas containing Epon 1001 were supposedly unaffected by the osmium tetroxide. Photomicrographs of microtomed sections of the film indicated large areas of Epon 1001-rich material containing a few streaks of Versamid 115-rich material as well as the spherical Epon 1001-rich particles dispersed in a Versamid 115-rich matrix.

Microtomed sections of the 96/4-Epon 1001/bisphenol A-Versamid 115 emulsion combination showed more areas of Epon 1001-rich material dispersed in a Versamid 115-rich matrix indicating significant inhomogenity of the film. Thus, Epon 1001 particles coalesced to a greater extent giving uniform regions of coalesced Epon 1001 particles containing interspersed Versamid 115 while the Epon 1001 particles containing bisphenol A somewhat maintained their initial colloidal characteristics.

This lack of compositional homogenity was also manifested by the effects which solvent extraction had on the surface characteristics of crosslinked films. Coatings of the Epon 1001/Versamid 115 combination showed a slightly greater surface porosity after solvent extraction, but the difference in surface craters or depressions before and after extraction was not great. Films of the Epon 1001/bisphenol A/Versamid 115 emulsion combination displayed more accentuated and deepened surface depressions upon solvent extraction, indicating that a portion of the uncured polymer had been removed. Solvent extraction of films of the Epon 1001/Versamid 115/Dion DPM-3-800 combination removed large spherical areas and produced many more surface depressions and craters. The extractable portion was suspected to be uncured epoxy resin.[56] It is obvious from the preceding section that epoxy emulsion systems require the optimization of all the methodological, compositional, and topographical parameters if these systems are to approach the high-performance capabilities of solvent-borne epoxy systems. Several of these variables have been surveyed elsewhere to various degrees.[57,58]

## EPOXY ESTER EMULSIONS

The versatility and good performance characteristics of single-component, epoxy ester emulsions have been major factors for the expansion of these products in water-reducible architectural, commercial maintenance, and industrial coatings markets. In contrast with the difficulties encountered in the development and production of two-component, bisphenol A-based epoxy emulsions, epoxy ester emulsions are lauded as providing simplicity in formulation, relative ease of manufacturing, reasonable storage stability, and overall performance properties comparable to those of solvent-borne epoxy ester systems.

While epoxy ester systems do not provide the chemical resistance of the two-component, bisphenol A-based epoxy system, they are similar in this respect to short-oil phenolic varnishes and superior to the more flexible, long-oil type. Epoxy ester emulsions thus provide an intermediate balance of properties between those of the catalyzed epoxy emulsions and alkyd emulsions. The oil-modified epoxy resin provides good adhesion, toughness, abrasion resistance, flexibility, high gloss, and durability in combination with intermediate chemical resistance. The alkali resistance of epoxy ester coatings is usually superior to that of alkyd coatings.

Epoxy ester emulsions can be prepared in conventional paint manufacturing equipment, illustrating the ease of production of these systems. A typical formulation which employs the use of a high-speed disperser is presented in *Table* 12.

After the let-down stage of the manufacturing procedure, the paint-base was slowly combined with water using good agitation until the water-in-oil emulsion inverted and became less viscous. The remaining amount

**Table 12—Formulation for a Medium-Oil Length, Soya Oil Epoxy Ester Emulsion Primer**

| Ingredient | Pounds Used | Gallons Used |
|---|---|---|
| Epoxy ester resin[a] | 308 | 38.71 |
| GAF Emulphor EL-719 Surfactant | 42 | 4.77 |
| Iron Oxide | 142 | 3.32 |
| China Clay, ASP 400 | 142 | 6.63 |
| Barytes | 284 | 7.76 |
| Grind above to 5 and let down with | | |
| Epoxy ester resin | 308 | 38.71 |
| Total Formulation | 1227 | 100.00 |

(a) The epoxy ester resin was composed of 57.6 wt. % of bisphenol A-epoxy resin with a molecular weight of 2900 and 42.4 wt. % soya fatty acids; 50% NVS in xylene.

of water could then be added rapidly. The total reduction of paint base with water amounted to 75% by volume in order to achieve a sprayable viscosity. The resulting emulsion was useful in automotive primer applications and displayed similar performance characteristics to those of conventional solvent-borne epoxy ester primers.[59]

Some of the compositional variations, methods of production, applications, and performance characteristics of epoxy ester emulsions are briefly summarized in the following examples.

Varnishes of epoxy ester emulsions displaying relatively long-term storage stabilities were prepared from epoxy ester resins consisting of 1 equivalent of bisphenol A epoxy resin esterified with 0.4 equivalents of dehydrated castor oil fatty acids. The resulting esterified epoxy resin (300 g of a 50% solution in toluene) was treated with 12.2 g poly(ethylene glycol) morpholinium maleate for 15 min at 120°C, and the final mixture was inverted with 300 g water to yield a varnish emulsion which was stable for eight months at room temperature.[60]

Epoxy ester emulsions, using vehicles similar in composition to those above,[60] were produced by treating the epoxy ester resin solution with up to 25% poly(ethylene glycol) acid maleate or the half maleate ester of $NaSO_3CH_2CH_2OH$ at 80°C to 180°C, neutralizing the free acid groups with sodium carbonate, amines, or phosphines, and emulsifying the resulting resin/surfactant mixture in water. The final products displayed good shelf stability for over six months.[61]

Epoxy ester emulsions useful in industrial applications requiring light to moderate durability characteristics were derived from the esterification products of dehydrated castor oil fatty acids and a bisphenol A epoxy resin having a molecular weight of 1400. The epoxy ester resin was combined with a surfactant prepared from a condensate of ethylene oxide and tributylphenol, and the resulting mixture was inverted in an aqueous solution containing methyl cellulose and cobalt or zinc siccative driers. The epoxy ester emulsion was subsequently let down into a mill-paste consisting of $TiO_2$, $CaCO_3$, 2-ethylhexanol, sodium hexametaphosphate, sodium polyacrylate, ammonium hydroxide, and methyl cellulose. The resulting paint was suitable for topcoat applications and displayed good shelf stability.[62]

Epoxy ester emulsions yielding coatings with improved exterior durability were formulated from an epoxy ester resin which was dispersed in water containing a trialkylphenol poly(ethylene oxide) emulsifier, carboxymethyl cellulose thickener, an antifoam agent, and cobalt, calcium, and zirconium siccative driers. The epoxy ester resin was prepared from bisphenol A epoxy resins with molecular weights ranging between 600 to 1200 and dehydrated castor oil fatty acids or mixtures of soybean, linseed, marine, and isomerized linseed oil fatty acids.[63]

Textile printing compositions providing good adhesion and durability were formulated from a mixture consisting of 42.2 parts of an epoxy ester resin prepared from 58.9% of a bisphenol A epoxy resin and 41.1% dehydrated castor oil fatty acids, 0.31 parts mixed naphthenate driers, and 8.2 parts sodium dodecyl sulfate solution (10% solids) which was inverted through the incremental addition of a solution consisting of 4.07 parts sodium dodecyl sulfate solution (10% solids), 20.65 parts casein solution (20% solids) containing $NH_4OH$ and preservatives, and 8.65 parts water. The crude emulsion was post-stabilized with 5.48 parts of a 1/1-$NH_4OH$/water mixture and 10.54 parts of a sodium carboxymethyl cellulose solution (20% solids). The entire mixture was then homogenized to yield a stable emulsion which was suitable as a vehicle for pigmented textile paints.[64,65]

Primers for metal automotive parts which could be applied by conventional spray or electrophoretic methods were derived from an Epon 1001/tall oil fatty acid epoxy ester resin which was further condensed with a maleinized linseed oil resin for five hours at 125°C to 135°C to yield a product with self-emulsification abilities. Upon neutralization, the resulting modified-epoxy ester resin was dispersed in water, combined with carbon black pigment and a formaldehyde-benzoguanamine crosslinking agent, applied electrophoretically to phosphatized steel sheets, and cured to yield primer coatings with excellent adhesion, hardness, corrosion and alkali resistance. The epoxy ester primer could be topcoated with conventional acrylic lacquers without lifting or loss of adhesion.[66]

In addition to various industrial applications, epoxy ester emulsions have also performed well when applied over plaster, concrete, wood, and cardboard substrates.[67] Usually in such applications, the epoxy ester emulsion serves as a modifier for commercial poly(vinyl acetate), acrylic, or butadiene-styrene emulsions to improve chalk adhesion, water resistance, and coalescence ability[68] although, at times, resin incompatibility may detract from certain characteristics of the resulting blend.*

It is obvious from the brief discussion above that

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*See section describing reference 99 in Part V of this review (JCT, December 1978, p. 78).

much of the technology which was developed for alkyd resin emulsions, i.e., more efficient means of emulsification and the use of internal surfactant techniques, could easily be transferred to epoxy ester resin emulsions to generate systems with subsequent product improvements.

## POLYURETHANE EMULSIONS

Polyurethane resins are among the few polymer classes which can be tailored to meet the needs required by such broad product categories as adhesives, coatings, fibers, rubbers, foams, and plastics. While this capacity to be designed in accord with the property requirements of the end application has been a significant factor towards the expanding use of polyurethane resins, the increased utilization and acceptance of these systems also emanates from the extraordinary integrity which the urethane group provides in high-performance applications. For example, two-component, aliphatic polyurethane coatings are being used in industrial and marine applications which require the physical and chemical integrity of a highly crosslinked epoxy resin together with the gloss and gloss retention, weatherability, and appearance of an acrylic system. The product versatility afforded by this capacity to vary the polymer structure together with the increased physical and chemical integrity provided by the polar urethane linkages have been contributory factors promoting the expansion of urethane technology into the areas of ecologically-compatible and water-borne polyurethane systems.*

Much of the present technology that was used for the development of thermosetting polyurethane emulsions was patterned after the methods and techniques used for the development of thermoplastic polyurethane emulsions. Consequently, as many of the variables associated with the development of thermosetting polyurethane emulsions are of a proprietary nature and have not yet been disclosed, some of the emulsification techniques, surfactant combinations, compositional variations, and general conceptual approaches used in the preparation of thermoplastic polyurethane emulsions are briefly surveyed in the following examples. Most of these techniques and variables are readily applicable to the design of thermosetting polyurethane emulsions.

## USE OF NONIONIC SURFACTANTS

Emulsions useful as binders for nonwoven fabrics or as adhesives for construction materials were prepared from a tolylene diisocyanate/poly(oxypropylene) glycol/1,2-bis(2-cyanoethylamino)ethane polyurethane resin solution which was directly emulsified in water containing a propylene glycol/ethylene glycol copolymer. nonionic surfactant. The resulting emulsion displayed a viscosity of 125 poise, a pH of 6.4, a solids content of 35%, a particle size of about 1 μm and yielded coatings with a tensile strength of 216 Kg/cm$^2$ and 1200% elongation after drying for 10 min at 80°C.[69]

Thermoplastic polyurethane emulsions demonstrating improved shelf stability were derived from a 4,4′-diphenylmethane diisocyanate/1,4-butanediol/poly(tetramethylene)adipate polyurethane resin solution which was emulsified at 55°C to 60°C in an aqueous solution containing poly(vinyl alcohol) and condensates of cetyl alcohol and ethylene oxide as emulsifiers. The resulting emulsion possessed a solids content of 17.5%, a viscosity of 7000 cps, and a shelf stability of three months.[70]

Protective coatings with good tensile strength and elongation were formulated from a 78.8/21.2-poly(propylene glycol)/tolylene diisocyanate resin which was emulsified by means of a colloid mill in an aqueous solution containing 3% of a nonionic surfactant prepared by condensing nonylphenol with 35 moles of ethylene oxide. The resulting emulsion was post-stabilized with $Et_3N$ and yielded films with a tensile strength of 246 Kg/cm$^2$ and an elongation of 650%.[71]

Other types of nonionic surfactants which have been used to prepare relatively stable, thermoplastic polyurethane emulsions include poly(oxyalkylene) sorbitan monooleate condensates,[72] silicone derivatives such as Ucar® Super Wetter FP,[73] block copolymers of poly(ethylene glycol) and poly(propylene glycol),[74] and propylene oxide-ethylene oxide adducts of ethylene diamine (HLB value of 28.0). This latter surfactant (Tetronic® 908) produced homogeneous emulsions with a shelf stability of 15 days.[75]

## USE OF ANIONIC SURFACTANTS

Polyurethane emulsions stabilized with conventional anionic surfactants were prepared from a polyurethane prepolymer (molecular weight of 1500 and containing 5.7% free NCO groups) which was mechanically mixed in water at 20°C containing a sodium dodecylbenzenesulfonate emulsifier. The resulting crude emulsion was further homogenized by shear reduction through 15 μm slits at a rate of 64 m/sec. The final product possessed a particle size of 0.5 μm and showed no signs of phase separation after three months of storage.[76]

Polyurethane latexes with improved degradation resistance to heat or acids were formulated from a tolylene diisocyanate/glycerol/propylene oxide-bisphenol A adduct/2-methylpiperazine copolymer which utilized an anionic surfactant prepared from the reaction product of 1 mole octylphenol with 5 moles ethylene oxide which was subsequently end-terminated with $ClCH_2CO_2H$ and neutralized with morpholine. Films of the emulsion demonstrated a tensile strength of 1807 psi.[77]

Compositions which could be used as adhesives or coatings were prepared from a 4,4′-diphenylmethane diisocyanate/poly(ethylene glycol) (molecular weight of 1000)/polycaprolactone glycol (molecular weight of 1250) copolymer which was emulsified in water containing either a polyol-sultone adduct or a sulfosuccinic acid compound as the dispersing agent. Thus, a sur-

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*See references 126-128 in Part III (JCT, September 1978, p. 109); references 58-60 in Part IV (JCT, October 1978, p. 97); and references 100-111 in Part V (JCT, December 1978, p. 95).

factant prepared from the alkali-treated reaction product of polycaprolactone glycol (molecular weight of 1200) with propane sultone yielded an emulsion which showed no change over a six month storage period at room temperature and whose films demonstrated a tensile strength of 418 Kg/cm$^2$ and an elongation of 620%.[78]

## USE OF INTERNAL, ANIONIC SURFACTANTS

Thermoplastic polyurethane emulsions stabilized with an internal, anionic surfactant were formulated from an isocyanate-terminated 4,4′-diphenylmethane diisocyanate/poly(ethylene glycol) (molecular weight of 1000)/polycaprolactone glycol (molecular weight of 1250) copolymer which was chain extended with an oligomeric polyol-diamine-propane sultone condensate. Thus, 132.5 g of the NCO-terminated prepolymer was dispersed in 200 g of water containing the reaction product of 96 g polycaprolactone glycol, 19.5 g propane sultone, and 5.22 g hexamethylenediamine together with 4.0 g of a sulfosuccinic acid emulsifier to yield a thermoplastic polyurethane emulsion containing internal, anionic surfactant groups and which displayed a shelf stability of over six months.[79]

Thermoplastic polyurethane emulsions stabilized with an internal, anionic surfactant were prepared from an isocyanate-terminated prepolymer consisting of 1 mole of poly(propylene glycol) condensed with 2 moles of tolylene diisocyanate which was emulsified and chain-extended upon addition to an aqueous solution containing the sodium salt of triaminosulfonic acid. The internal surfactant compound was prepared by reacting 0.1 mole of propane sultone with 0.1 mole of diethylenetriamine in benzene and neutralizing the resulting adduct with sodium hydroxide. The polyurethane emulsion yielded films with a tensile strength of 70.2 Kg/cm$^2$, a 100% modulus of 27.2 Kg/cm$^2$, and an elongation of 205%.[80]

Finishes for textiles which yielded leather-like materials were formulated from a 1,6-hexamethylene diisocyanate/poly(hexamethylene phthalate) prepolymer which was end-terminated with a condensate of ethylenediamine and 1,3-propane sultone. The resulting anionic-functional copolymer was dispersed in water and combined with hemp fibers and urea to yield coatings containing 55.6% fibers, 31.7% polyurethane resin, and 12.7% urea and which demonstrated a tensile strength of 28.2 Kg/cm$^2$.[81]

Adhesives and binders for textiles with improved water and oil resistance were derived from an adipic acid/ethylene glycol polyester diol which was end-capped with 1,6-hexamethylene diisocyanate and subsequently chain-extended with an anionic-functional compound consisting of $H_2NCH_2CH_2[H_2N(CH_2)_3]CHSO_3Na$. The resulting anionic, polyurethane resin was dispersed in water to yield a 40.5% solids emulsion whose coatings demonstrated good clarity, elasticity, and tensile strength.[82]

Protective coatings with excellent water resistance were formulated from a 79.1/26.9-poly(hexamethylene adipate)/1,6-hexamethylene diisocyanate prepolymer which was end-capped with a mixture consisting of 0.01182 moles of the potassium salt of a 1/1-ethylenediamine/propane sultone adduct in combination with 0.0201 moles of potassium lysinate. The resulting dual-functional anionic polyurethane resin was dispersed in water and yielded a 44% solids latex which could be sheared at 1400 rpm for >10 min without coagulation.[83]

Fireproof polyurethane coatings containing up to 50% by weight of bromine were prepared from an adipic acid/2,3-dibromo-2-butene-1,4-diol/ethylene glycol polyester diol which was end-capped with 1,6-hexamethylene diisocyanate and subsequently chain-extended upon addition to an aqueous solution containing the reaction product of 3.8 parts ethylenediamine, 7.6 parts propane sultone, and 3.5 parts sodium hydroxide. The resulting emulsion (45% solids) was applied to a plastic substrate and dried to yield films with good tensile strength, elasticity, and flame resistance.[84]

Protective coatings displaying good light resistance were derived from a 1,6-hexamethylene diisocyanate/poly(ethylene adipate prepolymer which was chain-extended in an aqueous solution containing sodium 2-[β-[(ω-aminoethyl)amino]propionamido]-2,2-dimethylethanesulfonate. The resulting anionic polyurethane emulsion yielded clear, colorless coatings with good elasticity and tensile strength.[85]

Finishes for polyester tire cords providing increased adhesion to vulcanized rubber stock were prepared from a 4,4′-diphenylmethane diisocyanate/ethylene glycol/poly(ethylene terephthalate)/sodium 3,5-bis(2-hydroxyethoxycarbonyl)benzenesulfonate copolymer which self-dispersed in water upon homogenization to yield emulsions with good stability. A poly(ethylene terephthalate) cord impregnated with the anionic polyurethane emulsion at a weight-percent gain of 0.89% and further treated with a formaldehyde/resorcinol latex displayed an H-test adhesion of 14.7 Kg to vulcanized rubber after curing three minutes at 210°C.[86]

## USE OF INTERNAL, CATIONIC SURFACTANTS

In addition to the use of conventional nonionic and anionic surfactants or anionic-functional compounds with groups capable of chain-extending the urethane prepolymer, polyurethane resins may also be conveniently prepared having amine groups which upon acid neutralization yield cationic sites with good emulsification abilities. For example, cationic-functional polyurethane emulsions with good wet and dry adhesion to such diverse substrates as glass, metal, wood, paper, masonry, and fabrics were prepared by end-terminating a polyester or polyether diol with tolylene diisocyanate and chain-extending the resulting NCO-terminated prepolymer with an alkyldiethanolamine. The amine sites of the resulting polyurethane were then neutralized with acetic acid to yield a cationic resin which was easily dispersed in water using a high-speed mixer. The resulting cationic polyurethane emulsion was further stabilized by the addition of a polyvinylpyrrolidone protective colloid.

Because of the volatilization of acetic acid during the drying process, the cationic sites were fugitively converted to the less hydrophilic amine groups which subsequently increased the water resistant properties of the system. Because of the polarity of the functional groups and the strong intermolecular associative forces, these systems demonstrated a marked improvement in physical properties as compared to those of conventional, vinyl-type emulsions. *Table* 13 illustrates the coatings properties of a cationic emulsion prepared from a tolylene diisocyanate-terminated poly(oxytetramethylene) prepolymer (Adiprene® L-167) which was chain-extended with 4.4 phr methyldiethanolamine and 1.3 phr triethanolamine and subsequently neutralized with acetic acid.

The low solubility of these films in tetrahydrofuran (11% by weight) was attributed to the use of triethanolamine which served as a source of crosslinking during chain-extension. However, when 1,4-butanediol was substituted for the alkylethanolamine, the resulting resin could not be emulsified in aqueous acetic acid. Thus, both the tertiary amine groups in the urethane backbone and the acetic acid in the water phase were found to be essential components in the system. The cationic repulsive charge on the particles in one case amounted to 86 mv.[87]

Cationic polyurethane emulsions have also been prepared from a 4,4′diphenylmethane diisocyanate/polyester diol prepolymer which was chain-extended with N-butyldiethanolamine and subsequently neutralized with acid,[88] by the neutralization of polyurethane resins containing amine units distributed throughout the backbone structure,[89,90] and through the neutralization of terminal-amine groups in combination with nonionic emulsifiers.[91] Other surfactant combinations and methods of emulsification have been discussed in other surveys.[92]

## THERMOSETTING POLYURETHANE EMULSIONS

The procedural and compositional elements employed in the design of thermosetting polyurethane emulsions utilize many of the previous formulation techniques and emulsification methods in combination with such modes of crosslinking as chain-extension, thermal unblocking, autooxidation, and the utilization of functional group other than isocyanates to effect crosslinking.

### Chain Extension Methods

For example, thermosetting polyurethane emulsions which yielded crosslinked products upon chain extension were produced from a polyurethane resin, derived from an adduct of poly(oxytetramethylene) glycol and possessing 4.2% free NCO groups, which was emulsified in water using a nonionic silicone surfactant in combination with a liquid paraffin. After dispersion, the polyurethane emulsion was combined with 4,4′-methylenedianiline crosslinking agent, applied to a glass substrate, and cured one hour at 100°C to yield tough, flexible films with a water-vapor transmission rate of 0.94 g per 30 $cm^2$ for a 24 hr period and through 20 mils of film thickness.[93]

**Table 13—Physical Properties of Coatings Derived From a Cationic Polyurethane Latex[a]**

| | |
|---|---|
| Latex particle size | 1-2 μm |
| Tensile strength, psi | 2229 |
| Modulus, psi | |
| 100% | 200 |
| 300% | 300 |
| Elongation, % | 676 |
| Hardness, Shore A-2 | 63 |
| Taber Abrasion (CS-17 wheel), mg/1000 cycles | 3 |
| THF[b] soluble, wt-%[c] | 11 |
| THF swell, wt-%[c] | 270 |
| Water soluble, wt-%[d] | 2.4 |
| Water swell, wt-%[d] | 3.4 |

(a) 10-15 mil films dried overnight at ambient and at 75°C for 24 hr.
(b) THF = tetrahydrofuran
(c) 10 mil films immersed for three days at 24°C.
(d) 10 mil films immersed for seven days at 24°C.

Coatings and adhesives with improved water resistance were formulated from a 4,4′-diphenylmethane diisocyanate/1,4-butenediol/poly(oxytetramethylene) glycol/maleic acid/styrene/m-cresol copolymer which was emulsified in aqueous sodium hydroxide using a poly(oxyethylene) lauryl ether surfactant. The resulting emulsion was combined with a 1,6-hexamethylenediamine crosslinking agent and heated to 60°C to yield coatings with a tensile strength of 160 Kg/$cm^2$ and an elongation of 650%.[94]

Crosslinkable polyurethane latexes have also been formulated by emulsifying an isocyanate-terminated prepolymer in an aqueous surfactant mixture. Presumably, the free NCO groups of the polyurethane resin are converted to amine functionality which subsequently chain-extend the prepolymer through the formation of urea linkages. These products are usually applied as soon as the prepolymers are emulsified and have been found useful as binders for nonwoven fabrics,[95] or as substitutes for leather.[96]

Self-emulsifiable polyurethane resins which crosslink upon chain-extension in water were prepared from a mixture of 47 parts ethylene glycol, 264 parts poly(oxydiethylene adipate) diol (molecular weight of 2000), 740 parts of a diethylene glycol/sodium dimethyl sulfoisophthalate/adipic acid polyester diol, and 278.4 parts tolylene diisocyanate which was condensed at 80°C to a product with a free NCO content of 2.1%. The resulting anionic-functional urethane prepolymer was emulsified by simple stirring in water to yield a storage-stable polyurethane latex whose coatings displayed a tensile strength of 290 Kg/$cm^2$, a modulus at 100% of 14.3 Kg/$cm^2$, an elongation of 1280%, and swelling percentages in water, trichloroethane, and methyl ethyl ketone of 11.2, 118, and 66.4%, respectively.[97]

## USE OF BLOCKED ISOCYANATES

As the chain-extension reaction in water may yield products of varying performance consistency and as the presence of urea linkages usually enhance the water sensitivity of the product, a more practical and auspicious approach for obtaining high-performance polyurethane emulsion coatings has been the use of blocked isocyanate groups capable of crosslinking upon thermal treatment. For example, thermosetting polyurethane emulsions containing blocked isocyanate groups and yielding high-performance coatings were obtained by reacting 255 parts Desmodur® N-75 isocyanate with 119 parts ε-caprolactam at 100°C for three hours. The resulting blocked isocyanate was combined with 213 parts Desmophen® 651, 213 parts ethyl acetate, and 0.26 parts dibutyltin laurate. This mixture (100 parts) was emulsified in 150 parts water containing 1 part of a 3/1 blend of a polyoxyethylene alkylaryl ether (Emulgen 935)/polyoxyethylene nonylphenol ether (Nonipol® 100). The resulting emulsion was applied to galvanized steel and cured for 10 min at 180°C to yield transparent, glossy coatings with good water and chemical resistance.[98]

Thermosetting polyurethane emulsions containing blocked isocyanate groups which reacted upon heating at 150°C were prepared from 187 g of an 80/20 blend of 2,4-tolylene diisocyanate and 2,6-tolylene diisocyanate which was reacted with 56.5 g ε-caprolactam in 77.5 g acetone at 60°C for three hours. The blocked diisocyanate was combined with a tolylene diisocyanate-terminated poly(propylene glycol) intermediate which had been chain-extended with glycerol and subsequently end-capped with maleic anhydride. The resulting carboxylic acid-functional polyether/blocked diisocyanate mixture was emulsified in $NH_4OH$ to yield an anionic, polyurethane resin emulsion (pH of 8.7) which when applied to glass, dried, and cured for 30 min at 150°C yielded coatings with good water and chemical resistance.[99]

Protective coatings with good flexibility and chemical resistance were formulated from a tolylene diisocyanate-terminated poly(ethylene adipate) diol which was subsequently end-capped and chain-extended with diethylenetriamine. The amine-functional polyurethane prepolymer was combined with a monooxime blocked adduct prepared from 174 parts tolylene diisocyanate and 17.4 parts 2-butanone oxime, and the resulting mixture was emulsified in water containing an acid to yield a cationic, thermosetting polyurethane emulsion which when applied to metal and cured for 20 min at 120°C provided coatings with a modulus of 21 Kg/cm$^2$, a tensile strength of 207 Kg/cm$^2$, and an elongation of 570%.[100]

Protective coatings with good mechanical properties and chemical resistance were formulated from a tolylene diisocyanate-terminated poly(ethylene adipate)/polyethylene glycol mixture which was chain-extended and end-capped with diethylenetriamine. The resulting polyurethane/polyurea/polyamine mixture was combined with a blocked isocyanate prepared from 87 parts methyl ethyl ketoxime and 174 parts tolylene diisocyanate, and the blend was dispersed in water containing acid to yield a 10% polyurethane emulsion which when applied to glass and cured for 20 min at 120°C provided coatings with a 100% modulus of 13 Kg/cm$^2$, a tensile strength of 159 Kg/cm$^2$, an elongation of 780%, and swelling upon immersion in water for one hour of 35%.[101]

High-performance coatings with good adhesion to steel and improved exterior durability were formulated from a blocked-urethane monomer containing vinyl functionality which was prepared by condensing 1.1 moles of 2,4-tolylene diisocyanate with 1.2 moles of cresol and subsequently reacting the partially blocked product with 1.0 moles of 2-hydroxyethyl methacrylate. The resulting blocked-urethane, vinyl-type monomer (30 parts) was free radically polymerized with 17 parts isobutyl methacrylate, 10 parts styrene, 30 parts methyl methacrylate, 10 parts hydroxyethyl methacrylate, and 3 parts maleic anhydride in 100 parts tetrahydrofuran. The hydroxyl, carboxyl, blocked isocyanate-functional polymer was emulsified in 150 parts water containing 2.0 parts NaOH and 3.0 parts poly(ethylene glycol) lauryl ether surfactant. After emulsification, the tetrahydrofuran carrier solvent was vacuum stripped to give a 40% solids emulsion. The resulting latex was applied to steel and cured 30 min at 150°C to yield glossy coatings with good bending resistance.[102]

Polyurethane emulsions containing blocked isocyanate constituents have also been recommended for such applications as high-temperature, adhesion-promoting finishes for tire cords[103] and as high-tensile strength, porous coatings useful as leather substitutes.[104] In the latter application, low density films were produced from the liberation of carbon dioxide which was formed when the freed NCO groups combine with the carboxyl functionality of the resin component.

## AUTOOXIDIZABLE POLYURETHANE EMULSIONS

Because systems containing blocked isocyanate functionality usually require curing temperatures of approximately 150°C in order to ensure development of optimal properties,[105] other means of crosslinking such as autooxidation or the use of less water sensitive functional groups (methylol or epoxide) have been employed to develop polyurethane emulsions with lower thermal curing requirements. For example, air-drying polyurethane emulsions with good exterior durability characteristics were prepared from a polyester intermediate consisting of 500 g linseed oil fatty acids, 98 g poly(ethylene glycol) (molecular weight of 3000), 184 g pentaerythritol, 122 g hydrogenated bisphenol A, and 98 g phthalic anhydride which was chain-extended with a mixture consisting of 164 g tolylene diisocyanate, 64 g pentaerythritol triallyl ether, and 0.04 g dibutyltin dilaurate in 122 g methyl ethyl ketone. The resulting uralkyd-type resin was combined with $TiO_2$ and cobalt driers and emulsified in water to yield a paint which demonstrated good resistance to ultraviolet light and yellowing.[106]

Air-drying coatings with good hardness and solvent resistance were formulated from a hydroxyl, carboxyl-functional alkyd resin intermediate prepared from 0.094 moles phthalic anhydride condensed with 0.3 moles of a 2/1-N,N-bis(2-hydroxyethyl)linseed amide/linseed monoglyceride mixture. The free carboxyl groups of the resulting half-ester product were neutralized with a stoichiometric amount of triethylamine, and the remaining free hydroxyl groups were condensed at 50°C with 0.253 moles of tolylene diisocyanate. The uralkyd resin (85 parts) was combined with 15 parts of 2-butoxyethanol and dispersed in water to yield a 30% solids emulsion with a pH of 8.0 to 8.5. Coatings of the emulsion dried tack-free in 0.33 hr, displayed a Sward hardness of 21, 45, 49, 56, and 53 after aging 1, 4, 7, 14, and 21 days, respectively, and were resistant to immersion in xylene for >240 hr.[107]

Protective coatings which air-dried rapidly and displayed good mechanical properties were derived from an alkyd intermediate consisting of 48 parts phthalic anhydride condensed with 182 parts of a 1/2 molar adduct of bisphenol A/propylene oxide, 64 parts bisphenol A epoxy resin (epoxide eq. wt. of 1650), 94 parts trimethylolpropane, 38 parts polyethylene glycol (molecular weight of 3000), and 392 parts castor oil fatty acids. The mixture was esterified to an acid number of 2, combined with 364 parts styrene and 64 parts methacrylic acid, and free radically polymerized using 8 parts tert-butyl peroxide over a four hour reaction period. The resulting vinyl-modified alkyd intermediate was combined with 108 parts pentaerythritol triallyl ether and 84 parts tolylene diisocyanate, and the mixture was allowed to react over an eight hour period at 75°C. The uralkyd resin was neutralized with triethylamine and inverted in 1240 parts water containing cobalt driers to yield a polyurethane emulsion whose coatings demonstrated improved physical properties and displayed a dry time of 1.5 hr.[108]

Air-drying polyurethane emulsions with good flow properties and aging characteristics were derived from an autooxidizable substrate consisting of 512 g linoleic acid, 134 g trimethylolpropane, and 344 g of a 1/2 molar adduct of bisphenol A/propylene oxide which was esterified to an acid number of 6, combined with 98 g of a bisphenol A-epichlorohydrin epoxy resin, and further esterified with 148 g of trimellitic anhydride to an acid number of 46. The resulting alkyd-epoxy resin intermediate was chain-extended with 120 g of tolylene diisocyanate, neutralized with triethylamine, and inverted with a mixture of butyl cellosolve and water to yield a 48% solids emulsion which provided coatings with good drying properties, gloss, and corrosion resistance.[109]

## POLYURETHANE EMULSIONS CONTAINING VARIOUS CROSSLINKABLE FUNCTIONALITY

In addition to such methods of crosslinking as chain-extension reaction, thermal unblocking of masked isocyanate groups, and autooxidation, crosslinkable polyurethane emulsions have also been prepared with functional constituents other than isocyanate groups to effect crosslinking. For example, a hydroxymethylated polyurethane emulsion capable of self-condensation was derived from an adipic acid/diethylene glycol polyester intermediate which was end-capped with 1,6-hexamethylene diisocyanate and subsequently chain-extended with sodium-2-[$\beta$-(cyclohexylamino)-propionamido]-2-methylpropanesulfonate. The resulting anionic polyurethane resin was emulsified in water and subsequently hydroxymethylated through the addition of 30% formaldehyde. The modified polyurethane latex was catalyzed by reducing the pH to 3-4 and yielded elastic films having improved strength.[110]

Aziridine-functional polyurethane emulsions which were useful for improving the shrinkproofing and flatsetting of wool fabrics were derived from a tolylene diisocyanate-terminated poly(tetramethylene ether) glycol intermediate which was end-capped with ethyleneimine. The resulting aziridine-terminated polyurethane resin was emulsified with an alkylphenoxy poly(oxyethylene) ethanol to yield an emulsion which imparted durable-crease properties to the fabric. A woolen textile was immersed in a 4% emulsion of the aziridine-modified polyurethane resin together with 1% $NaHSO_3$, squeezed to a wet pickup of 80 to 100%, and cured at 149°C to yield a product displaying a 0.5% shrink and excellent crease retention after three washes (75 min) at 41°C.[111]

Crosslinkable polyurethane emulsions containing an aziridine curing agent have also been evaluated for applications requiring high gloss, water, and hydraulic fluid resistance. However, because of their toxicity under spray application conditions, the use of such systems was not recommended.[112]

A siloxane-functional polyurethane emulsion with enhanced shear stability and yielding water and chemically resistant coatings was derived from a mixture of 875 parts poly(oxypropylene) diol (molecular weight of 700) and 68.5 parts poly(oxypropylene) triol (molecular weight of 410) which was chain-terminated with 522 parts of an 80/20 mixture of 2,4 and 2,6-tolylene diisocyanate. The resulting isocyanate-terminated prepolymer was subsequently reacted with 73.3 parts $[CH_3C(CH_2OH)_2CO_2]^-$ $[HN(C_2H_5)_3]^+$ to chain-extend and provide anionic functionality together with 29.3 parts of $H_2N(CH_2)_3Si(OC_2H_5)_3$ to provide self-condensibility. The resulting anionic, siloxane-functional polyurethane resin was self-emulsified in 3000 parts of water and yielded films which upon curing at 105°C demonstrated a tensile strength of 320 $Kg/cm^2$, a tensile modulus at 100% of 87 $Kg/cm^2$, a tear strength of 45 Kg/cm, and an elongation at break of 420%. A clay-treated paper board coated at 0.0005 $g/cm^2$ of polymer solids with the siloxane-functional polyurethane emulsion displayed a weight loss of 0.02 g after 100 revolutions in a Taber Abraser while a commercial acid-catalyzed, melamine-cured alkyd resin showed a weight loss of 0.031 g under identical conditions.[113]

Epoxide-functional polyurethane emulsions which were useful as binders and coatings with good tear strength and elasticity were derived from 202 parts of a poly(tetramethylene ether) glycol intermediate which was end-capped with 34.8 parts of tolylene diisocyanate. The resulting polyurethane prepolymer was subsequently chain-extended with 3.83 parts of diethylenetriamine which provided internal reactive sites for the condensation of 4.8 parts of epichlorohydrin and 5.1 parts of maleic anhydride. The epoxidized, maleated polyurethane resin was emulsified in 200 parts water containing 2.1 parts sodium hydroxide. The epoxidized, anionic polyurethane emulsion yielded self-curing films which after baking for 20 min at 120°C demonstrated a tear strength of 248 Kg/cm$^2$, a modulus at 100% of 20.5 Kg/cm$^2$, and an elongation of 710% and were insoluble but swelled in dimethylformamide.[114]

Epoxide-functional polyurethane emulsions which were self-curing and useful as binders and coatings were formulated from 1020 parts of a poly(tetramethylene ether) glycol intermediate which was end-capped with 348 parts tolylene diisocyanate and subsequently chain-extended with 26.5 parts of diethylenetriamine. The resulting amine-functional polyurethane resin (357 parts) was further condensed with 6.1 parts epichlorohydrin and 12.6 parts 1,3-propane sultone and subsequently emulsified in water containing base to yield an anionic, epoxide-functional polyurethane emulsion whose coatings demonstrated a tear strength of 194 Kg/cm$^2$, a 300% modulus of 35.7 Kg/cm$^2$, and an elongation of 850% after curing for 20 min at 120°C.[115]

Cationic, self-curing polyurethane emulsions useful as coatings or binders were prepared from 327 parts of a poly(oxytetramethylene) glycol intermediate which was end-capped with 55.7 parts of tolylene diisocyanate. The isocyanate-terminated prepolymer was subsequently chain-extended with an amine-epoxide adduct prepared by reacting 61.8 parts diethylenetriamine with 18.5 parts epichlorohydrin. The resulting amine and epoxide-functional polyurethane resin was emulsified upon addition to a water-acid mixture and produced a cationic, epoxy-functional polyurethane resin emulsion whose self-curing coatings demonstrated a tensile strength of 320 Kg/cm$^2$, an elongation of 640%, and good flexibility after curing for 20 min at 120°C.[116]

Melamine-cured, anionic polyurethane emulsions useful as finishes for leather or as a leather substitute were prepared from 212 g of an adipic acid/1,6-hexanediol/neopentyl glycol intermediate which was end-capped with 38 g 1,6-hexamethylene diisocyanate and subsequently chain-extended with an anionic-amine compound prepared from 3.75 g ethylenediamine, 7.6 g 1,3-propane sultone and 3.5 g KOH. The resulting anionic, polyurethane resin (300 g) was emulsified in 350 g water containing 5 g $NH_4OH$, combined with 58.5 g trimethylolmelamine trimethyl ether crosslinking agent, applied to a polyurethane leather substitute, and cured for 15 min at 90°C to yield smooth, glossy, microporous finishes with good tear strength and crack resistance.[117]

Melamine-cured, polyurethane emulsions useful as vehicles for stoving lacquers were derived from an alkyd-type intermediate prepared from 120 parts ricinoleic acid, 160 parts pelargonic acid, 84 parts pentaerythritol, 294 parts trimethylolpropane, 104 parts poly(ethylene glycol) (molecular weight of 3000) and 242 parts phthalic anhydride. The alkyd-type intermediate (acid number of 15) was combined with 66 parts of pentaerythritol triallyl ether, and the resulting mixture was chain-extended with 172 parts tolylene diisocyanate. The acid-functional (acid number of 12) polyurethane resin was neutralized with triethylamine, dispersed in water, combined with a water-dilutable 25/75-formaldehyde/melamine curing agent, applied to metal or glass, and cured for one hour at 80°C to yield coatings with good gloss, elasticity, and resistance to water.[118]

### Formulation Variables

While the previous examples illustrated some of the compositional variations and procedural techniques used in the development of crosslinkable polyurethane emulsions, much of the science and technology of these systems is still being held confidential as experimental polyurethane emulsions progress toward commercialization. However, a recent study has been conducted which illustrated some of the difficulties associated with the development of crosslinkable polyurethane emulsions which possess properties comparable to those of commercial, two-component, solvent-borne polyurethane systems. The polyurethane resin which served as the control for the investigation consisted of 71 parts Desmodur N-100, a high molecular weight biuret of 1,6-hexamethylene diisocyanate, 29 parts Multron® E-380, a short-oil alkyd resin adduct, and 0.072 parts dibutyltin dilaurate. The various approaches used to develop crosslinkable polyurethane emulsions with the capabilities and integrity of the solvent-borne control are summarized as follows.

Desmodur N-100 and Multron E-380 were allowed to react for two hours, and the resulting adduct was emulsified with a surfactant mixture of 1.6%/3.2%-sodium lauryl sulfate/cetyl alcohol using either ultrasonic irradiation or homogenization. Since the polyurethane adduct contained some free isocyanate groups, chain-extension presumably occurred upon mixing the adduct in water. The emulsions prepared by this method produced coatings comprised partially of a friable, white powder and partially of a transparent film. However, suitable products could not be generated by using the adduct/water-extension method.

In order to eliminate the hydrolysis reaction between water and the free isocyanate groups, the urethane prepolymer was partially blocked with various monohydric alcohols. Thus, 10 ml portions of methanol, ethanol, and isopropanol were added, respectively, to mixtures of 8.1 g Desmodur N-100, 19.6 g Multron E-380, and 0.02 g dibutyltin dilaurate. After the alcohol-urethane mixture reacted, 2 g of trimethylolpropane trimethacrylate was added to each of the ungelled, alcohol-capped urethane prepolymer samples, and the resulting mixture was emulsified in water using the sodium lauryl sulfate/cetyl alcohol surfactant mixture and ultrasonic irradiation. The resulting emulsion was heated to 60°C and combined with a persulfate initiator in order to effect crosslinking. Presumably, the sulfate ion radicals would abstract a hydrogen atom from both the blocked isocyanate and hydroxyl-terminated polyester prepolymers, and the resulting radicals would then combine with the trimethylolpropane trimethacrylate compound, thereby crosslinking the constituents of the system. The polymerization attempts produced lumps of coagulum which were softer than would have been expected had extensive crosslinking occurred, and no evidence was found which indicated radical induced crosslinking.

Therefore, in order to enhance the free radical crosslinking reaction with the urethane moiety, an adduct of Desmodur N-100 and hydroxyethyl methacrylate was prepared using dibutyltin dilaurate as a catalyst. The acrylate-containing urethane adduct was subsequently dissolved in a vinyl monomer, and the mixture was emulsified using the sodium lauryl sulfate/cetyl alcohol surfactant combination and ultrasonic irradiation. The resulting pre-emulsion, containing the vinyl-type monomer and the bifunctional acrylate-urethane adduct, was converted to a precrosslinked latex by heating the mixture over a three hour period in the presence of a persulfate free radical initiator. Theoretically, the acrylate-urethane adduct served as a multifunctional crosslinking agent which when polymerized with conventional vinyl-type monomers yielded fully-cured polyurethane emulsions. The concentration of the hydroxyalkyl methacrylate-Desmodur N-100 adduct in the blend was varied between 40.8% and 48.3% by weight, and the comonomers which acted as a reactive diluent for the acrylate-urethane adduct included methyl methacrylate, isobutyl methacrylate, butyl acrylate, and acrylonitrile.

In general, the film properties of these precrosslinked emulsions depended upon the composition of the copolymer, the degree of crosslinking, and the degree of compatibility achieved between the vinyl polymer and the acrylate-urethane adduct. Incompatibility between the constituents produced opaque, cheesy films, while excessive crosslinking produced brittle, nonadherent films. The best overall coatings properties were obtained from a 44.4/27.8/27.8-hydroxyethyl acrylate-Desmodur N-100 adduct/n-butyl acrylate/isobutyl methacrylate copolymer, and the modulus-temperature response of this combination was typical for that of a flexible coating. However, most blends were excessively crosslinked and produced opaque and brittle films with poor coalescence.

In order to reduce the degree of crosslinking and subsequently to improve the flexibility and coalescing ability, the crosslinking functionality of the Desmodur N-100 adduct was decreased by replacing 75% of the hydroxyalkyl methacrylate constituent with methyl ricinoleate. The modification resulted in a polyurethane adduct containing 47.4% Desmodur N-100, 8.2% hydroxyethyl methacrylate, and 44.4% methyl ricinoleate. A fully-cured latex prepared from 74% of the Desmodur N-100/hydroxyethyl methacrylate/methyl ricinoleate adduct and 26% isobutyl methacrylate produced clear, soft, semi-gloss coatings with good adhesion after baking for 16 hr at 60°C. However, the coatings were rated too soft for topcoat applications.

In an effort to improve the film properties of this system, the methyl ricinoleate constituent was replaced with Paraplex® P-444A, an unsaturated polyester prepolymer of lower functionality which was claimed to provide good weathering properties and resistance to discoloration by ultraviolet light. A crosslinkable polyurethane adduct was prepared from a mixture consisting of 28.8% Desmodur N-100, 17.8% hydroxyethyl methacrylate, and 53.4% Paraplex P-444A by allowing the constitutents to react for 24 hr at room temperature. The resulting unsaturated urethane adduct was crosslinked at 60°C for two hours using 0.5% azobisisobutyronitrile and yielded coatings with good gloss, toughness, and hardness. However, because the viscosity of the adduct was too high for efficient emulsification, further work with this approach was discontinued.

Consequently, several epoxide-functional urethane adducts were prepared from Desmodur N-100 and 2,3-epoxy-1-propanol (glycidol) in order to generate an intermediate which could be crosslinked by means of conventional epoxy curing agents. An epoxy-urethane crosslinking agent consisting of 42.5% glycidol and 57.5% Desmodur N-100 was found to gel within three to four hours after the addition of ethylenediamine; however, when the same adduct was combined with the diglycidyl ether of 1,4-butanediol, the glycidol-urethane adduct/diglycidyl ether mixture could not be cured with ethylenediamine, and, therefore, this approach was also abandoned.

As a result of the difficulties encountered with adducts of Desmodur N-100, several attempts were made to prepare crosslinking intermediates from adducts of 1,6-hexamethylene diisocyanate (HMDI).

The crosslinking intermediates prepared from the condensation of hydroxyethyl methacrylate with HMDI were difficult to solubilize in the flexibilizing, longer side-chain acrylate monomers; and copolymerization with the shorter-chain acrylate monomers yielded hard, brittle coatings. Attempts to produce a more flexible intermediate by substituting part of the hydroxyethyl methacrylate with Polypropylene Glycol-425 were unsuccessful since the modification resulted in the formation of a waxy solid which was insoluble in the desired acrylate monomers. Also, attempts to prepare a crosslinkable intermediate from adducts of HMDI and glycidol were unsuccessful since the desired product could not be isolated from the reaction mixture.

As a result of the limited success provided by the approaches used to develop fully-cured, polyurethane emulsions, the project emphasis was shifted to the investigation of polyurethane emulsions which possessed air-drying capabilities. In light of this objective, an air-drying intermediate was prepared consisting of 36.3% Desmodur N-100, 9.9% allyl alcohol, and 53.8% Paraplex P-444A unsaturated polyester prepolymer. The resulting autooxidizable intermediate was blended with 26.9% (based on weight of the intermediate) methyl methacrylate, combined with 0.05% cobalt drier, and air dried for 24 hr at ambient to yield hard, semi-brittle films. Similar results were obtained from mixtures of the Desmodur N-100/allyl alcohol/Paraplex P-444A intermediate with such reactive diluents as butyl acrylate, isobutyl methacrylate, and a blend of n-butyl acrylate with styrene.

In order to overcome the brittleness displayed by these coatings, the autooxidizable intermediate was modified by substituting Paraplex P-444A with Multron R-16, a hydroxyl-terminated polyester prepolymer. A mixture composed of 68.1% of a Desmodur N-100/allyl alcohol/hydroxyethyl methacrylate/Multron R-16 intermediate dissolved in 31.9% isobutyl methacrylate and catalyzed with 0.05% cobalt drier yielded glossy, flexible coatings which dried slowly in air. The drying rate could be improved by a partial replacement of Multron R-16 with Paraplex P-444A unsaturated polyester. One blend containing 63% Paraplex P-444A and 37% Multron R-16 demonstrated improved drying properties while retaining its flexibility and toughness.

As a further means of improving the drying characteristics of these systems, the use of a methyl ethyl ketone peroxide/cobalt naphthenate initiator combination was investigated since this type of initiator system effectively cures such analogous systems as unsaturated polyester/styrene monomer blends. The experimental design of the evaluation utilized a fairly large compositional ladder study which included various concentrations and types of reactive diluents in combination with several polyurethane adducts. Typically, Desmodur N-100 was condensed with varying amounts of hydroxyethyl methacrylate, hydroxypropyl methacrylate, allyl alcohol, and Multron R-16 polyester. After a standardized reaction period, the vinyl-functional-urethane adduct was combined with a mixture of Paraplex P-444A unsaturated polyester dissolved in either butyl acrylate, isobutyl methacrylate, or some combination of the two monomers (reactive diluents). The resulting mixture was emulsified in water using the sodium lauryl sulfate/cetyl alcohol surfactant combination together with ultrasonic irradiation. Methyl ethyl ketone peroxide (1%) was added to the emulsified mixture together with varying amounts of 6% cobalt naphthenate drier. In general, most of the resulting systems cured within 16 hr to form tack-free films which were brittle; none of the combinations provided toughness; and some coatings were friable. These systems, on the average, yielded coatings displaying less integrity than those of the 34.1/29.4/25.2/6.3/5.0-Desmodur N-100/Multron R-16/isobutyl methacrylate/hydroxyethyl methacrylate/allyl alcohol combination which was air-dried using cobalt naphthenate as the sole catalyst and which yielded tough, transparent, flexible films.[55,119]

While some of the techniques and conceptual approaches employed in this last study were similar to those discussed in previous examples of this section, the impasses illustrated in this final study bring to light many of the difficulties which must be resolved if crosslinkable polyurethane emulsions are to become a viable, marketing reality to the industry.

## SILICONE-CONTAINING EMULSIONS

The utilization of silicone-based modifiers in conventional, vinyl-type emulsions[120-124] and the emulsification of polyorganosiloxane resins[125-128] are two of the more common approaches used to upgrade the performance characteristics of emulsion systems. Typically, coatings derived from silicone-containing emulsions have been claimed to provide such property improvements as greater adhesion;[129-131] elasticity,[132] flexibility,[133] and abrasion resistance;[134,135] improved tear[136,137] and tensile strength;[138,139] increased anti-blocking[140,141] and lubricity[142] together with greater stain,[143] water,[144] weather,[145] and heat resistance.[146,147]

In light of their numerous advantages, silicone-containing emulsions have been recommended as being useful in such applications as finishes,[148] coatings,[149] and binders[150] for textiles; foam backings for carpets;[151] finishes for paper[152,153] and leather;[154] components in vulcanized rubber;[155,156] polishes[157] and protective coatings;[158-161] and anti-foam agents[162,163] and surfactants[164,165] for emulsion systems.

The emulsifying agents used in the preparation of silicone-containing latexes include nonionic surfactants such as poly(vinyl alcohol),[166,167] oleyl alcohol condensates containing 20 moles of ethylene oxide,[168] lauryl alcohol condensates containing 10 moles of ethylene oxide,[169] alkylphenol condensates containing 10 moles of ethylene oxide,[170] and block copolymers of nonhydrolyzable polysiloxanes and oxyalkylenes;[171] such anionic surfactants as sodium lauryl polyethylenoxysulfate (Trex® K5),[172] sodium bis(2-ethylhexyl)-sulfosuccinate,[173] sodium undecylsulfate;[174] and such cationic surfactants as cetylbenzyldimethylammonium chloride,[175] dodecylbenzyldimethylammonium chloride,[176] and pyridinium salts such as $C_{18}H_{37}OCH_2$  $\overset{+}{N}C_5H_5\ Cl^-$.[177]

Through an optimization of the HLB requirements of the system[178] and through the use of ultrasonic dispersion[179] or homogenization,[180] silicon oil emulsions have been prepared with particle sizes ranging from 0.03 μm to 0.3 μm.[181] However, as was the case with other post-emulsified systems, the storage stability of silicon oil emulsions usually varies from several weeks[182] or months[183] up to six months.[184]

Because silicone resins such as poly(dimethylsiloxane) lack the pendent, crosslinking sites possessed by other polymer systems, the hydroxyl units of the chain ends serve as the main function group for crosslinking silicone oil emulsions.[185] Typically, hydroxyl-functional polysiloxane emulsions have been crosslinked with such curing agents as aluminum salts of $C_{6-8}$-chain fatty acids,[186,187] tin-containing fatty acids,[188] chromium stearyl chloride,[189] Chromium (III) complexes of $C_{12-18}$ mono- or diesters of $H_3PO_4$,[190] $Zn(NO_3)_2$ or $Zr(OAc)_4$,[191] or urea-formaldehyde, epoxy resin, or polyamine-epichlorohydrin reaction products in combination with lead, cobalt, zinc, tin, titanium, or cerium salts.[192]

Several of the compositional variations, physical properties, crosslinking variables, and applications of crosslinkable silicone resin emulsions are briefly discussed in the following examples.

Crosslinkable silicone emulsions with improved storage stability were prepared by dispersing 1200 g of a hydroxyl-terminated dimethylpolysiloxane resin/ethyl acetate/toluene/xylene solution (33% resin solids) in 825 ml of water using 75 g of a hexadecylamine acetate condensate of ethylene oxide as the emulsifying agent. The crude mixture was homogenized for 30 min at 250 atmospheres pressure and solvent stripped at 12 to 15 mm of vacuum and 50°C to yield a 38% solids polysiloxane emulsion containing 5% organic solvents and demonstrating a stability of greater than six months at room temperature or eight days at 40°C. Presumably, crosslinking of the resin occurred as the free hydroxyl groups combined during thermal curing.[193]

Finishes for textiles which provided enhanced water-repellency characteristics were formulated from 220 parts of a trimethylsiloxy-blocked poly(methylhydrogensiloxane) resin, 180 parts of a trimethylsiloxy-blocked poly(dimethylsiloxane) resin, 25 parts poly(vinyl alcohol), 175 parts water, and 400 g (9% solids) of a curing agent consisting of the condensation product of adipic acid, diethylenetriamine, and epichlorohydrin. The mixture was emulsified, applied to cotton cloth, and dried at temperatures up to 150°C to yield a product which demonstrated less water absorption than a fabric treated with a polysiloxane resin emulsion which had been cured with a mixture consisting of zinc 2-ethylhexanoate and dibutyltin bis(2-ethylhexanoate).[194]

Coatings for fabrics with improved water-repellency and crease resistance were formulated from 15 parts of a poly(methylsiloxane) resin (molecular weight of 2500), 7.5 parts isopropyl alcohol, 74 parts water, and 1 part of a crosslinkable surfactant prepared from 75 g of a formaldehyde solution (30%), 42 g dicyandiamide, 6.75 g stearylamide, 12.5 ml 2N HCl, and 30 g isopropyl alcohol. The resulting silicone resin emulsion (80 ml) was diluted with 100 ml of water, combined with 720 ml of water containing 0.8 g $SnCl_2 \cdot 2H_2O$ curing agent, applied to a cotton poplin fabric at 80% wet pickup, and cured for 5 min at 150°C to yield a water-repellent coating with good resistance to washing and scrubbing. Presumably, the water sensitivity of the coatings decreased as the emulsifier combined with the tin curing agent upon being incorporated into the crosslinked matrix.[195]

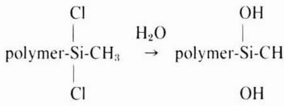
Finishes for fabrics which provided increased water-repellency were derived from 30 g of a poly(methylhydrogensiloxane) resin (molecular weight of 3000) which was dissolved in 7 g o-dichlorobenzene and 2 g perchloroethylene and subsequently combined with 61 g of an aqueous solution containing 2 g zirconium ion as zirconium acetate-formate (1.8 acetyl groups and 0.2 formate group/Zr ion). The catalyzed mixture (480 g) was emulsified with 300 g of a solution containing the titanic acid ester of triethanolamine, applied to a fabric, squeezed to a conventional degree of pickup, and cured at 105°C to yield a fabric with good hydrophobic properties.[196]

Compositions for imparting water repellency and permanent press to textiles were formulated from an emulsion (50 parts) containing 20% of a ∝,ω-bis(trimethylsilyl)poly(dimethylsiloxane) resin and 20% of a ∝,ω-bis(trimethylsilyl)poly(methylhydrogensiloxane) resin, 7 parts $Zn(NO_3)_2$ curing agent, and 843 parts water. The siloxane emulsion was combined with 70 parts dihydroxydimethylolethyleneurea and 30 parts of a bisphenol A epoxy resin (25 parts)/diethylenetriamine (10 parts) adduct dispersed in 25 parts methanol, 25 parts acetic acid, and 130 parts water. A polyester-cotton blend fabric was impregnated with the emulsion mixture, dried, pleated, and cured for 14 min at 170°C. The pleating remained intact after eight washes, and the fabric displayed good hydrophobicity both before and after washing.[197] A cotton poplin fabric impregnated with a similar urea-formaldehyde/epoxy resin-amine adduct/$Zn(NO_3)_2$/poly(alkylsiloxane) emulsion blend demonstrated a water absorption and penetration of 11% and 0 ml, respectively, before washing and 27% and 3 ml after five machine washings at 60°C.[198]

## OTHER TYPES OF SILICONE EMULSIONS

In addition to such techniques as the post-emulsification of preformed polysiloxane resins which crosslink primarily through the free hydroxyl groups located at the ends of the polymer chain, thereby limiting the crosslink density attainable by such systems, crosslinkable, silicone-containing emulsions have also been prepared containing self-condensable silanol groups which have been selectively placed throughout the polymer structure by means of the hydrosilylation of residual unsaturation sites.[199]

The hydrosilylation was performed on an unsaturated polyester substrate derived from the condensation of 306 g phthalic anhydride, 299 g pentaerythritol, 962 g tall oil fatty acids, and 167 g alpha-eleostearic acid. The residual hydroxyl and carboxyl groups of the unsaturated polyester were blocked with trimethylsiloxane units prior to the hydrosilylation step in order to eliminate any undesired side-reactions. The addition reaction was effected by condensing 9.46 g methyldichlorosilane with 360 g of the blocked polyester resin, which contained 10% triene unsaturation, and the hydrosilylation step was complete after a reaction period of 30 min at 60°C when chloroplatinic acid was used as the catalyst. The methyldichlorosilylated polyester resin was then treated with water and agitated to form the silanediol moiety by the hydrolysis of Si-Cl bond, i.e.,



$$\text{polymer-}\overset{\overset{\displaystyle\text{Cl}}{|}}{\underset{\underset{\displaystyle\text{Cl}}{|}}{\text{Si}}}\text{-CH}_3 \xrightarrow{H_2O} \text{polymer-}\overset{\overset{\displaystyle\text{OH}}{|}}{\underset{\underset{\displaystyle\text{OH}}{|}}{\text{Si}}}\text{-CH}_3$$

The resulting silanediol-containing polyester was spontaneously emulsified in water using an inversion process and without the aid of external surfactants. Thus, the silanediol moiety served as a fugitive, internal surfactant affording colloidal stabilization qualities for the system in the emulsion form and providing hydrophobic, crosslinked sites upon silanol combination during curing. The increased integrity provided by crosslinked films of the silanediol-containing polyester emulsion in comparison with the solvent-borne, parent polyester resin is presented in *Table* 14.

**Table 14—Effect of Silanol Condensation on the Film Properties Of a Polyester Resin Containing Different Alkylsilanediol Moieties**

| Hydrosilylating Group Employed | Curing Conditions | Electrical Resistance (volts/mil) | Sward Hardness | Tensile Strength (Kg/cm$^2$) | Elongation (%) | Impact Resistance at −80°C (in-lbs) | Salt Fog Exposure (Days to Failure) | |
|---|---|---|---|---|---|---|---|---|
| | | | | | | | Initial | Complete |
| None ................ | Air dried | 537 | 4 | 26 | 67 | — | 14 | 38 |
| Methyldichlorosilane ................ | Air dried | 2475 | 8 | 43 | 55 | — | 18 | 40 |
| Phenyldichlorosilane ................ | Air dried | 2778 | 10 | 28 | 53 | — | 5 | 40 |
| None ................ | 30 min @ 120°C | 1442 | 10 | 76 | 28 | 20 | 5 | 21 |
| Methyldichlorosilane ................ | 30 min @ 120°C | 3000 | 64 | 109 | 49 | 80 | 28 | 62 |
| Phenyldichlorosilane ................ | 30 min @ 120°C | 3000 | 38 | 132 | 36 | 80 | 23 | 62 |

These results indicate that the hydrosilylation of an unsaturated resin and the subsequent condensation of the silanediol groups upon curing provided emulsion-cast films with improved impact, electrical, and corrosion resistance together with greater tensile strength and hardness. Moreover, the gloss retention of these products was characteristic of that associated with other silicone-modified systems.[200]

## PHENOL-FORMALDEHYDE RESIN EMULSIONS

Besides serving as a crosslinking agent for hydroxyl[*] and carboxyl[†] functional, thermosetting emulsions, phenol-formaldehyde resole resins have also been formulated into emulsion products which are suitable for such applications as binders for paper,[201-203] protective coatings,[204,205] and adhesives for wood.[206] When used in applications requiring lower cost, higher performance products, phenol-formaldehyde resin emulsions have been claimed to yield coatings with excellent water[207] and chemical resistance together with excellent adhesion to metal and wood substrates.

Thermosetting phenol-formaldehyde resin emulsions are formulated using conventional dispersion techniques in combination with such emulsifying agents as poly(vinyl alcohol),[204,206] sodium caseinate,[207,208] or conventional nonylphenol condensates of ethylene oxide.[201-203,205] The particle sizes of the resulting emulsions may range as low as 0.02-0.8 $\mu$m,[209] and representative products usually display reasonable storage stabilities of up to six months.[205,206]

Typical methods of preparation, formulation variables, physical characteristics, performance properties, and applications of thermosetting, phenol-formaldehyde resole emulsions are briefly summarized in the following examples.

Compositions suitable as binders for thermal insulation or as impregnates for cellulosic sheet members were prepared using a two-stage, novolac-resole process which reduced the free concentration of phenol and formaldehyde in the product to less than 1% by weight.

In the first stage (novolac) of the synthesis, 0.28 moles of aqueous formaldehyde (50% solution) was heated at reflux with 1 mole of phenol and 0.004 moles of oxalic acid catalyst until approximately 98% of the formaldehyde was consumed. The resulting novolac resin was then used in the second stage (resole) of synthesis to form a product which crosslinked upon thermal curing.

Thus, the novolac resin prepared in the first stage was neutralized with 0.2 moles of NaOH, combined with 2.26 moles of aqueous formaldehyde (50% solution) and 0.15 moles of calcium hydroxide catalyst, and heated at 65°C until the free formaldehyde content dropped to 2.3%. Upon achieving the desired degree of conversion, the reaction mixture was cooled to 40°C, combined with 0.075 moles of aqueous ammonia (29% solution), and sparged with carbon dioxide to form a calcium carbonate precipitate which was removed by filtration. The resulting resole resin mixture (100 parts) was combined with 5 parts of a casein emulsifier solution (20% solids) and mixed with a conventional propeller, blade, or turbine agitator to yield an emulsion which displayed a pH of 7.7, a solids content of 51.1%, a Brookfield viscosity of 38 cps, a free formaldehyde content of 0.8%, a free phenol content of 0.68%, a degree of polymerization of 1.8, and a stability at 0 to 5°C of greater than one month. The resole emulsion was used as an impregnate for paper sheets which were useful as automotive oil, air, and fuel filters.[209]

Phenol-formaldehyde resole emulsions useful as adhesives for wood products were obtained by condensing 941 g phenol with 1461 g of formalin over a 110 min period at 85°C using NaOH and $Et_3N$ as catalysts. The resulting product was subsequently emulsified at 85°C using 78.7 g of poly(vinyl alcohol) which was 89.1% hydrolyzed and possessed a degree of polymerization of 1700. The pH of the emulsion was adjusted to 5.5 with p-toluenesulfonic acid to yield a product with a storage stability of 32 days, a curing time of 2.5 min at 115°C, and demonstrating an adhesive strength to plywood of 12.5 Kg/cm$^2$ after a 72 hr immersion in boiling water.[210]

Compositions useful as surface coatings or as impregnates for cellulosic sheets were prepared by reacting 100 parts phenol, 5 parts melamine, and 85 parts formalin (50% solution) over a 155 min period at 95°C

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[*] See references 109 and 122 in Part I (JCT, June 1978, p. 55); and reference 55 in Part IV (JCT, October 1978, p. 97).

[†] See reference in Part III (JCT, september 1978, p. 109).

while in the presence of 3 parts triethylamine catalyst. The resulting modified resole resin was cooled to 45°C and combined with 0.11 parts NaOH (50% aqueous) and an emulsifier solution consisting of 15 parts water, 6.5 parts urea, 6.5 parts casein, and 0.9 parts $NH_4OH$ (30% solution). The modified resole resin/emulsifier blend (222 parts) was then slowly combined with water (182 parts) whereupon, after approximately 75% of the total water addition, phase inversion occurred forming an oil-in-water emulsion. The resulting resole emulsion displayed a solids content of 55%, a Brookfield viscosity of 1000 cps using an LVF viscometer at 30 rpm with spindle number 3, and a particle size range of approximately 0.02 $\mu$m to 0.8 $\mu$m. After curing for 10 min at 150°C, films of the emulsion demonstrated good resistance to water. Other modifiers which could be used in place of the melamine resin included guanamine or benzoguanamine compounds.[211]

## PRECROSSLINKED EMULSIONS

While multifunctional monomers such as divinylbenzene,[212] diallyl phthalate,[213] N,N′-methylenebis(methacrylamide),[214,215] 1,1′-divinylferrocene,[216] ethylene glycol dimethacrylate,[217] diallylidenepentaerythritol,[218,219] polyethylene glycol bis(n-vinylcarbamate),[220] 1,2-bis(p-vinylphenyl)-1,2-ethanediol,[221] triallyl cyanurate,[222,223] trimethylolpropane trimethacrylate[224] and 1,1,2,2,-tetrakis(allyloxy)ethane[225] are normally used as crosslinking agents in high-solids, resin/monomer formulations that are post-cured by means of peroxide compounds and heat[226] or irradiation;[227] recently, such multifunctional monomers have received increased attention as a means of developing higher-integrity, precrosslinked emulsion systems.[228-232] Coatings and laminates crosslinked by means of multifunctional monomers usually display increased hardness,[233] scratch resistance,[234] modulus,[235] glass transition temperature,[236] impact resistance,[237] cohesive[238] and tensile strengths.[239]

The use of multifunctional monomers in vinyl acetate-based emulsions has been found to be an effective means of increasing the molecular weight of the copolymer and to correspondingly upgrade the performance characteristics of architectural coatings based upon such products. For example, when 2% trimethylolpropane triacrylate, based on weight of total monomer, was included in a typical butyl acrylate/vinyl acetate monomer mixture, the resulting latex was found to be crosslinked and yielded coatings with a significant quantity of the composition being insoluble in an 80% acetic acid solution. Upon filtration of the insoluble portion, the terpolymer was found to possess an inherent viscosity of 1.15 dl/g compared to an inherent viscosity of 0.70 dl/g for a similar composition which omitted the multifunctional monomer. In qualitative terms, if these copolymers had been homopolymers of vinyl acetate, a difference in inherent viscosity of this magnitude would have reflected an increase in average molecular weight of from 130,000 to approximately 240,000, or an increase of 85% or more.

**Table 15—Effect of Precrosslinked Emulsion Vehicles On the Scrub Resistance of Latex Films[a]**

| Ratio of VA/BA[b] | % TMPTA[c] | MFFT[d] (°C) | Scrubs to failure[e] 60% PVC | Scrubs to failure[e] 65% PVC |
|---|---|---|---|---|
| 81.5/18.5 ......... | None | 11 | 212 | 105 |
| 83.5/16.5 ......... | None | 13 | 206 | 105 |
| 80/20 ............ | 2% | 13 | 274 | 123 |
| Commercial All-acrylic emulsion ......... | None | — | 169 | — |

(a) Paint films were aged one week prior to scrub test.
(b) VA = vinyl acetate; BA = butyl acrylate.
(c) TMPTA = trimethylolpropane triacrylate.
(d) MFFT = minimum film forming temperature.
(e) Scrub test was a modification of ASTM-D2486-69 whereby the paint was applied to a Leneta chart, dried, placed atop a base plate to which a 10 mil raised shim had been attached, and scrubbed with a weighted nylon brush. Failure usually occurs quickly in the coating directly over the raised shim.

The increased molecular weight and the network structure which resulted upon crosslinking were also responsible for a subsequent improvement in the tensile strength properties of an 85/15-vinyl acetate/butyl acrylate copolymer which was modified with varying concentrations of trimethylolpropane triacrylate. Films of the vinyl acetate/butyl acrylate copolymer emulsion containing 2%, 4%, and 6% trimethylolpropane triacrylate demonstrated tensile strengths of 1710, 1870, and 2020 psi, respectively, in comparison to a tensile strength of 1540 psi for a control composition which omitted the multifunctional crosslinking agent. Thus, as a result of crosslinking, the tensile strength of the films containing 2%, 4%, and 6% trimethylolpropane triacrylate improved 11%, 21%, and 31%, respectively, in comparison with the uncrosslinked control.

The increased integrity provided by the multifunctional comonomer was also demonstrated by the superior scrub resistance of a paint prepared from the vinyl acetate/butyl acrylate/trimethylolpropane triacrylate terpolymer emulsion as compared to other paints prepared from uncrosslinked emulsion vehicles. *Table* 15 illustrates the scrub resistance of various emulsion coatings which were formulated at 60% and 65% pigment volume concentrations and which were aged at ambient conditions for one week.

These data indicate that the paint which was formulated from the precrosslinked emulsion provided 33% better scrub resistance at 60% pigment volume concentration than the average of two controls which were not crosslinked. Even at the higher pigment volume concentration of 65%, the scrub resistance of the paint prepared with the crosslinked vehicle was almost 20% greater than that of the controls.

Precrosslinking the 85/15-vinyl acetate/butyl acrylate copolymer emulsion with 4% trimethylolpropane triacrylate was also found to increase the wet adhesion of the film since the resulting network structure presumably served to reduce the rate at which water vapor was transmitted through the film.[240]

Other applications, compositional variations, physical properties, and performance characteristics of precrosslinked emulsions are briefly discussed in the following examples.

Weather-resistant coatings useful in architectural applications were formulated from a 55/27.4/15/2.6-styrene/dibutyl itaconate/ethylene glycol dimethacrylate/acrylic acid copolymer emulsion which utilized 5% sodium dodecylbenzenesulfonate as the polymerization emulsifier. The resulting precrosslinked emulsion (100 parts), which possessed a solids content of 49.5%, a pH of 7.9, and a viscosity of 1550 cps, was combined with 50 parts $TiO_2$, applied to steel panels, and dried for one week at 25°C to yield glossy coatings with improved water, alkali, and acid resistance and which demonstrated a pencil hardness of 3H and Ericksen hardness of 6.7 mm.[241]

Compositions useful as packaging films with low gas permeability were derived from a 63.6/27.3/9.1-acrylonitrile/methyl acrylate/ethylene glycol dimethacrylate copolymer seed emulsion (50% solids) which was overcoated with a composition consisting of 70/30-acrylonitrile/methyl acrylate. The resulting emulsion (10 parts), which possessed a crosslinked core and uncrosslinked shell, was blended with 90 parts of a 70/30-acrylonitrile/methyl acrylate copolymer emulsion to yield a blend which formed films with 400% elongation at break.[242]

Precrosslinked emulsions which were used as modifiers for thermoplastic emulsion compositions were prepared from a 92/8-butyl acrylate/acrylonitrile copolymer emulsion which was precrosslinked with small amounts of allyl methacrylate. The resulting crosslinked emulsion (5 to 40 parts) was combined with from 95 to 60 parts of a thermoplastic emulsion consisting of a 75/25-styrene/acrylonitrile copolymer to yield a blend whose coatings demonstrated improved workability, heat stability, and impact strength.[243]

Compositions with a rubbery, crosslinked core and a rigid, thermoplastic shell were prepared from a 92.5/5/2.5-butyl acrylate/ethylene glycol dimethacrylate/allyl methacrylate copolymer seed emulsion which used 0.5% sodium lauryl sulfate as the polymerization emulsifier. The crosslinked seed latex was overcoated or grafted with methyl methacrylate at 65°C utilizing benzoyl peroxide as the initiator to yield an emulsion with a thermoplastic shell to crosslinked core ratio of 33 to 67 which was found to be useful as protective coatings with improved weatherability.[244]

Elastomeric emulsion compositions possessing an uncrosslinked core and a crosslinked, intermediate shell were prepared from a seed latex consisting of 28.5 parts butyl acrylate and 1.5 parts methyl methacrylate which was overcoated or grafted with a mixture consisting of 63 parts butyl acrylate, 4.2 parts methyl methacrylate, 2.8 parts ethylene glycol dimethacrylate, and 0.7 parts triallyl cyanurate. The resulting emulsion (100 parts) was subsequently overcoated at 70°C with 30 parts of a 60/20/20-styrene/methyl methacrylate/acrylonitrile monomer feed using 1.5 parts benzoyl peroxide as the initiator to yield a moldable product which demonstrated a Dynstat impact strength of 81 $Kg\text{-}cm/cm^2$ and a weather resistance (time required for the impact strength to decrease by 50% of its initial value in a Weather-Ometer®) of 950 hr.[245]

Multilayered emulsion compositions possessing a crosslinked core with a glass transition temperature (Tg) of $\geq$ 10°C, crosslinked interlayers with Tg of $\leq$ 0°C, and a crosslinked shell with Tg $\geq$ 50°C were prepared from a seed latex consisting of 10 parts methyl methacrylate and 0.05 parts allyl methacrylate which utilized 1 part sodium dialkyl sulfosuccinate as the polymerization surfactant and which was overcoated five consecutive times with varying amounts of methyl methacrylate, allyl methacrylate, and butyl acrylate. The final shell layer contained 15 parts methyl methacrylate and 0.015 parts allyl methacrylate, and the total composition consisted of 55 parts methyl methacrylate, 45 parts butyl acrylate, and 0.425 parts allyl methacrylate. The resulting crosslinked emulsion yielded moldable products with very good transparency and stress-clouding resistance and improved resistance to impact and weathering.[246]

## THERMOSET, WATER-IN-OIL EMULSIONS

In contrast with thermoplastic, inverse emulsions (water-in-oil) which have served mainly as model systems in kinetic and mechanism studies,[247] thermoset water-in-oil emulsions have been developed which show promise in such applications as protective coatings,[248] adhesives,[249] sealants,[250] molding compounds,[251] foams,[252] and insulating materials,[253] wood substitutes,[254] and leather substitutes.[255]

Typically, these systems are prepared by dissolving unsaturated polyester resins such as 1/1.5/1.8-maleic anhydride/phthalic anhydride/propylene glycol copolymers,[256-258] 1/1/2.2-isophthalic acid/maleic acid/propylene glycol copolymers,[259,260] or a 37.7/34.9/23.1/4.3-propylene glycol/phthalic anhydride/maleic anhydride/tolylene diisocyanate copolymer[261] in such monomers as styrene[262] or methyl methacrylate[263] and emulsifying water into the resulting resin/monomer mixture with the aid of conventional, nonionic surfactants.[264] The water-in-oil emulsion is then combined with such free radical initiators as methyl ethyl ketone peroxide and dicumyl peroxide[265] or methylcyclohexanone peroxide (2%)[266] along with a promoter such as diethanolphenylamine combined with cobalt undecanoate[267] in order to produce a crosslinked dispersion. The gel times of the catalyzed mixture usually vary in accord with the nature of the peroxide and promoter, but typically a 1% $H_2O_2$, 1% cobalt neodecanate, diethanolphenylamine (co-amine coordination ratio of 1/6) mixture provided a gel time of 0.8 to 1.1 min, a gel to peak exotherm time of 8.0 to 8.6 min, and a peak exotherm of 102 to 104°C[268] while 0.75% $H_2O_2$, 0.625% of a 1/1 blend of dimethylaniline and diethanolphenylamine, and 1% cobalt neodecanate (co-amine coordination ratio of 1/4) provided a gel time of one minute, a gel to peak exotherm time of 10 min, and a peak exotherm of 100°C.[269] The water-in-oil polyester emulsions are usually stable only for short periods (one day).[270]

The following two examples illustrate the procedures and components used to produce crosslinked, water-in-oil polyester emulsions.

Water-in-oil polyester emulsions yielding flame-resistant compositions with improved mechanical properties were obtained from 60 parts of a 2.2/1/1-isopropylene glycol/maleic acid/isophthalic acid polyester which was dissolved in 40 parts styrene, combined with 9 parts of a Surfonic®-type nonionic surfactant (ethylenediamine/propylene oxide/ethylene oxide adduct) and 1 part of a calcium stearate emulsifier, and subsequently mixed with 50 parts water. The resulting water-in-oil emulsion was treated with 0.5% cobalt naphthenate and 1% methyl ethyl ketone peroxide and cured 20 hr at room temperature and two hours at 80°C to yield a self-extinguishing product with a specific gravity of 1.07, Shore D hardness of 70, bending strength of 2.30 Kg/mm$^2$, impact strength of 2.36 Kg-cm/cm$^2$, and volatile content of 7.7%.[271]

Water-in-oil polyester emulsions useful as coatings which could be applied by means of spray applicators were formulated from 48.5 parts of a liquid polyester resin (specific gravity of 1.05 and viscosity of 160 cps) which was emulsified with 48.5 parts water using ultrasonic irradiation at a frequency of 90 kHz and power of 40 kW/gal. The resulting water-in-oil emulsion was catalyzed with 1% methyl ethyl ketone peroxide, 0.5 parts N,N-dimethyl aniline promoter, and 1.5 parts cobalt octanoate (12% cobalt) to yield a crosslinked product with a Shore A hardness of 100, a flexural strength of 4050 psi, a flexural modulus of 180,000 psi, a tensile strength of 2500 psi, and a heat distortion temperature of 77°C at 270 psi.[272]

Besides forming water-in-oil emulsions, polyester resins have also been used to form conventional oil-in-water crosslinkable emulsions for use as coatings[273,274] and binders.[275]

## NONAQUEOUS DISPERSIONS

Nonaqueous dispersions (NAD) were designed as an intermediate generation system which complied with prevalent photochemical emissions standards and which also afforded a more suitable environment for water reactive systems such as polyurethane resins[276-278] or aziridine-functional resins than that provided by an aqueous emulsion. Also, nonaqueous dispersions usually wet unprimed surfaces better than emulsion systems which tend to crawl and fisheye, and the water-free dispersions also eliminate the flash rusting problems associated with coating untreated steel. Both thermoplastic[279-281] and thermosetting nonaqueous dispersions are available commercially, and various aspects of their science and technology have been reviewed elsewhere.[282,283] The following examples briefly illustrate the general approaches used in the vehicle design and formulation of nonaqueous dispersions.

Urethane/acrylic nonaqueous dispersions containing unsaturated functionality for post-irradiation curing were derived from 480 parts of a 73/18.7/8.3-methyl methacrylate/2-hydroxyethyl acrylate/butyl acrylate copolymer which was dispersed in 200 parts of an aliphatic hydrocarbon. The dispersion was heated to 80°C, and the hydroxyl functionality of the copolymer were subsequently condensed with 300 parts of a 1/1 molar adduct of tolylene diisocyanate and 2-hydroxyethyl acrylate which was dispersed in 200 parts of an aliphatic hydrocarbon. The resulting vinyl-functional, urethane/acrylic copolymer dispersion was solvent stripped at 140°C to remove 250 parts of aliphatic hydrocarbon which was replaced with 250 parts of 2-ethylhexyl acrylate reactive diluent. The vinyl-functional copolymer/2-ethylhexyl acrylate dispersion (200 parts) was formulated with 50 parts of a 39/39/22-styrene/butyl acrylate/glycidyl methacrylate copolymer dispersion (containing both methyl methacrylate and 2-ethylhexyl acrylate reactive diluents) and 500 parts $TiO_2$. The white enamel dispersion was applied to a galvanized steel panel coated with a nonaqueous primer and cured with electron beams (3 Mrad) to yield crosslinked coatings with good hardness and high bonding strength.[284]

Epoxy/phenolic resin nonaqueous dispersions useful as corrosion-resistant topcoats for metal were prepared from 20 parts of Epikote 1007 epoxy resin (powder) dispersed in 40 parts aliphatic hydrocarbon (Shellsol® No. 140, boiling point of 186.7-203.3°C, kauri-butanol value of 31), 5 parts of ethyl cellosolve acetate, and 5 parts isobutyl alcohol. The epoxy resin nonaqueous dispersion was combined with 8.5 parts of a methylol-functional phenolic resin crosslinking agent, 5.0 parts iron oxide, 0.1 parts dispersing agent, and additional aliphatic hydrocarbon (70 parts) to yield a sprayable coating composition. The epoxy/phenolic resin nonaqueous dispersion was applied to steel sheets, dried one minute at 20°C, and cured for 10 min at 300°C to yield pinhole-free, impact resistant coatings with good bonding strength.[285]

Polyester/melamine resin nonaqueous dispersions providing impact resistant coatings for metal were derived from an oil-free polyester resin (acid value 7.1) which was obtained by condensing 194 parts dimethyl terephthalate, 174.6 parts dimethyl isophthalate, 116.2 parts isophthalic acid, 43.8 parts adipic acid, 27.6 parts glycerol, 126 parts 1,3-butylene glycol, and 146 parts neopentyl glycol while in the presence of 1.0 part lead oleate catalyst. The hydroxyl, carboxyl-functional polyester resin (80 parts) was dispersed in 170 parts aliphatic hydrocarbon (Shellsol No. 140), 20 parts butyl cellosolve acetate, milled with 100 parts $TiO_2$, and blended with 40 parts of a commercially available, butylated melamine resin curing agent (many systems require specifically-designed, butylated melamine-formaldehyde curing agents with high white spirit tolerance, i.e., 400 to 1500 as determined by ASTM D1198-55)[286] to yield a nonaqueous dispersion whose coatings provided good impact strength and corrosion resistance after curing for 10 min at 250°C.[287]

Acrylic/melamine resin nonaqueous dispersions useful as a topcoat for coil coating applications were prepared from 100 parts of an 88/10/2-acrylonitrile/2-hydroxyethyl methacrylate/methacrylic acid copolymer which was dispersed in 100 parts of isopropyl alcohol using 43 parts of an 80/15/5-hydroxyethyl acrylate/styrene/acrylic acid copolymer as a dispersing agent. The hydroxyl, carboxyl-functional dispersion (140 parts) was combined with 43 parts of a commercial melamine-formaldehyde curing agent (Smimar M-53), 7 parts aluminum pigment, 50 parts isopropyl alcohol, 20 parts cellosolve, and 30 parts butyl cellosolve to yield a composition whose coating demonstrated good chemical resistance and high gloss after curing for 30 min at 140°C.[288]

## OTHER METHODS OF APPLICATION AND CURING

In addition to the conventional application techniques and curing methods discussed throughout this survey, crosslinkable emulsion systems have also been developed which may be applied by means of electrodeposition[289-296] and cured by means of ultraviolet,[297-299] $\gamma$-rays,[300-302] microwave,[303] infrared,[304] and electron beam irradiation.[305-307] An example of each of the more commonly investigated methods is given below.

### Application by Electrodeposition

A cationic, vinyl-functional copolymer was prepared by reacting 114 g of glycidyl methacrylate with the secondary amine groups of a terpolymer consisting of 500 g of ethyl acrylate, 185 g (tert-butylamino)ethyl methacrylate, and 157 g (dimethylamino) ethyl methacrylate. The resulting vinyl-functional copolymer was combined with a monomer mixture consisting of 11% ethylene glycol dimethacrylate and 4% lauryl methacrylate, and the internal amine groups of the copolymer were neutralized with acetic acid to yield a 10% solids emulsion. A steel sheet, which served as the cathode, was electrocoated for one minute at 50 volts while immersed in the emulsion, dried five seconds at 150°C, and cured under nitrogen with electron beams (5 Mrad) to yield impact resistant coatings with a pencil hardness of H and a crosscut adhesion of 100/100.[308]

### Curing by Ultraviolet Light

UV-curable coatings for wood applications were derived from an unsaturated polyester, consisting of 1.0 moles maleic anhydride, 0.4 moles triethylene glycol, 0.5 moles propylene glycol, 0.35 moles trimethylolpropane diallyl ether, and 0.035 moles linseed oil fatty acids, which was emulsified in water using 3% nonionic surfactant as the dispersing aid. The resulting emulsion was combined with 5 parts benzoin isopropyl ether UV sensitizer, coated on decorative plywood, and exposed 20 min to an infrared lamp and 30 sec to a UV source to yield solvent, water, and weather resistant coatings with a pencil hardness of H.[309]

### Curing by $\gamma$-Radiation

$\gamma$-radiation-curable coatings useful in textile, leather, and paper applications were formulated from a 33% solids poly(ethyl acrylate) emulsion which was modified with 1% diallyl maleate crosslinking monomer. Films of the latex/crosslinking monomer mixture demonstrated 67% insolubility and a swelling index of 100 after exposure to 1 Mrad irradiation, 92% insolubility and a swelling index of 41 after exposure to 2 Mrad irradiation, and 93% insolubility and a swelling index of 33 after exposure to 5 Mrad irradiation. Other monomers which could effect crosslinking included dichloroethylene, vinylidene chloride, and N-phenylmaleimide.[310]

### Curing by Electron Beam

Vinyl-functional emulsion coatings which could be cured by electron beam irradiation were prepared by reacting 142 parts glycidyl methacrylate with a solution, hydroxyl-, carboxyl-functional copolymer consisting of 498 parts butyl acrylate, 100 parts methyl methacrylate, 100 parts styrene, 172 parts methacrylic acid, and 130 parts 2-hydroxyethyl acrylate. The epoxy addition reaction was conducted at 80°C until the acid number of the mixture reached 50. The resulting vinyl-functional copolymer was vacuum stripped of the solvent (ethanol), neutralized with an equivalent amount of ammonium hydroxide, and subsequently dispersed in 2000 g of water. The emulsion was applied to a sanded, mild steel plate and irradiated with electron beams (10 Mrads) to yield a crosslinked coating having a pencil hardness of 2H, an impact resistance of > 50 cm (500 g, 1.27 cm diameter dart), and a water resistance of > 20 days at 40°C.[311]

## OVERVIEW

It is apparent from the preceding discussion that a number of approaches have been investigated over the past 20 years to develop crosslinkable emulsions which are suitable for application in a reasonably broad spectrum of industrial areas. Although this effort has resulted in the commercial development of several products with good shelf stability, adequate application characteristics, and with more than competitive performance capabilities, crosslinkable emulsions as a class have achieved only limited commercial success in spite of the manifold nature of the coatings market. While certain interest groups attribute the restricted growth of these systems to elements such as consumer resistance and the preference of competitive solvent-borne products, perhaps the most ostensible factor preventing the continual expansion of the present-generation of crosslinkable emulsions into areas such as heavy-industrial or marine applications stems from the method of crosslinking which binds these systems to thermal curing processes such as those used in present coil or wood finishing applications. With the increasing need for high-performance, water-borne systems which are suitable for general application and which cure at or near ambient conditions, the coatings industry is presently attempting to develop industrial-quality emulsions which crosslink through autooxidation since the restrictions imposed upon products crosslinked by means of methylol-functional groups [aminoplast resins, N-methylolacrylamide and N-(isobutoxymethyl)-

acrylamide] have become increasingly more obvious. However, the limitations associated with autooxidizable systems are also apparent since even the better drying oil-based, solvent-borne systems have never achieved the properties provided by such systems as solvent-borne vinyl, chlorinated rubber, two-component epoxy, and polyurethane resins. Consequently, a considerable effort is presently underway to develop the technology required to produce two-component epoxy emulsions with adequate colloidal stability and with properties that are equivalent to those of their solvent-borne counterparts.

If, however, emulsion-based coatings are to progress beyond the growth-rate projected over the next 10 years for ecologically-based systems, then other high-performance vehicles in addition to autooxidizable and improved two-component epoxy emulsions must be developed which offer advantages commensurate with those provided by high-solids (over 70%), conforming solvent-borne systems; 100% solids, two-component systems; powder coatings; and other water-reducible or water-borne systems.[312] Such future-generation crosslinkable emulsions will utilize, out of necessity, other irreversible, low activation energy addition reactions which are tractable with the emulsion environment and which provide coatings with the properties required in high-performance applications.

## Trademark References

| | |
|---|---|
| Adiprene | E.I. du Pont de Nemours & Co., Inc. |
| Alipal, Gafac | GAF Corp. |
| Alodine | Amchem Products, Inc. |
| Araldite | CIBA-GEIGY Corp. |
| Armac | Armac Co. |
| Cymel | American Cyanamid Co. |
| D.E.R., Dowanol | Dow Chemical Co. |
| Desmodur, Desmophen | Naftone, Inc. |
| Dion, Nopcosant | Diamond Shamrock Chemical Co. |
| Emulgen | Kao-Atlas Co. Ltd. |
| Epikote, Epon, Shellsol | Shell Chemical Co. |
| Genamid, Versamid | General Mills Chemical Corp. |
| Kopox | Koppers Co., Inc. |
| Multron | Mobay Chemical Co. |
| Natrosol, Tween | Hercules Incorporated |
| Noigen | Dai-ichi Kogyo Seiyako Co. Ltd. |
| Nonipol | Sanyo Chemical Industries Ltd. |
| Paraplex, Triton | Rohm and Haas Co. |
| Pluronic, Tetronic | Wyandotte Paint Products Co. |
| Resimene | Monsanto Co. |
| Sumicure, Sumiflex, Sumipoxy | Sumitomo Chemical Co. Ltd. |
| Surfonic | Jefferson Chemical Co. |
| Trex | Scott Paper Co. |
| Ucar | Union Carbide Corp. |
| Vanoxy | R.T. Vanderbilt Co., Inc. |
| Weather-Ometer | Atlas Electric Devices Co. |

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# Selection of Straining (Nominal Filtration) Equipment for Coatings

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Straining is the last unit operation in a chemical coatings facility before the coating is filled into containers for shipment. This article discusses filtration equipment and filter mesh size which optimize the filtration rate.

## INTRODUCTION

The straining operation in the coatings industry is generally the last step in the manufacturing process before coatings, like paints and resins, are filled into containers. Most products are strained through 1 to 125 micron filters or screens. Straining in this range is called "nominal filtration." The filtering operation is used to clean paint when the cause of dirt is a "seedy" vehicle, or a lower-than-specified dispersion grind due to poor quality raw material or incomplete dispersion. The main object of filtration is to remove material foreign to the coating formula that would affect the customer's application equipment or quality of their coated products.

To increase filtration throughout and decrease filter cost, the following have to be selected: (1) Type of filtration equipment; (2) Filter size, or mesh opening.

Evaluated first are the advantages and limitations of filtration equipment followed by a discussion of the criteria for selection of filter size or screen mesh.

## FILTRATION EQUIPMENT

### Vibrating Screen

Manufactured by Russell Finex, Inc., Vorti-Siv Co., and others. The screens are made of synthetic materials, like nylon, and are available over a wide size range—from 80 to 400 mesh. In general, vibrating screens are suitable for water-borne products, low gloss oil products (5½ grind or less on the Hegman scale), and as a prefilter for any dirty products. The viscosity of the product going through a screen should be 80 Krebs Units or less, and not more than 100 Krebs Units as higher viscosities decrease filtration rate sharply. This filter is not recommended for dilatant products because the intense screen vibrations increases the product viscosity and filtration rate falls off. A disadvantage of this equipment is that during filtration the coating is open to the atmosphere and is, therefore, subject to aeration, drying, and contamination. Unlike cartridge filters, the screen can be washed and used again.

### Cartridge Filters

Manufactured by AMF Cuno Div., Filterite Co., Carborundum Co., Johns-Manville Co., and others.

The cartridges are either of a continuous filament yarn or of a graded density type. Cartridges are recommended for filtering coatings with a grind above 5 on the Hegman scale, which generally comprise intermediate and high gloss conventional solvent systems. Because pressure can be exerted on the product during filtration, a good filtration rate can be achieved with high viscosity (up to 140 Krebs Units) and dilatant products. Cartridges are available in sizes ranging from 1 to 200 microns.

The collapse pressure (the pressure above which the pores of a cartridge collapse) of a typical cartridge is about 70 lb/in.$^2$ differential. Filtration should be carried out below this pressure. Also, the average velocity of the coating through the cartridge should be kept below 10 ft/sec. Cartridge filters are not suitable for products containing long or gelatinous particles, such as occur in latex. Long particles break under pressure, and gelatinous particles larger than the cartridge micron size ex-

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**Table 1—Comparative Fineness Data**

| Hegman Scale | Production Club Scale | Depth of Walls or Groove | | | U.S. Mesh Size |
|---|---|---|---|---|---|
| | | NPIRI[a] | Mils | Microns | |
| 0 | 0.00 | 40 | 4.0 | 100.0 | 140 |
| 1 | 1.25 | 35 | 3.50 | 87.50 | 170 |
| 2 | 2.50 | 30 | 3.00 | 75.00 | 200 |
| 3 | 3.75 | 25 | 2.50 | 62.50 | 230 |
| 4 | 5.00 | 20 | 2.00 | 50.00 | 325 |
| 5 | 6.25 | 15 | 1.50 | 37.50 | — |
| 6 | 7.50 | 10 | 1.00 | 25.00 | 400 |
| 7 | 8.75 | 5 | 0.50 | 12.50 | — |
| 7.5 | — | 2.5 | 0.25 | 6.25 | — |
| 8 | 10.00 | 0 | 0.00 | 0.00 | — |

(a) National Printing Ink Research Institute.

trude through the cartridge, unless the filtration is carried out at low pressure, below about 20 lb/in.$^2$.

### Filter Bags

Manufactured by Textile Industries Inc., FSI Company, and others, the filter bags are made of various synthetic materials, with mesh size openings of 1 - 200 microns. The bags are of two types: (a) bags which are enclosed in pressure vessels, the filtration being carried out under pressure; (b) tie-on bags: the end of the bag is tied to the discharge nozzle of the tank containing the liquid to be filtered. The maximum pressure which tie-on bags should be subjected to is 6 psi, which is approximately the pressure exerted on a bag tied to the end of a nozzle with a 12 ft liquid head above it. When used as a prestrainer or for filtering coarse products, a filter bag gives a throughput equivalent to 12 cartridges, assuming both are operated at the same low pressure (max. 6 psi). This is because the standard 9.75 in. cartridge has about 0.5 ft$^2$ of surface area, and the standard size filter bags (1 x 2 ft, 1.5 x 2 ft) provide 7 and 10 times the surface area of a cartridge filter. Thus, six cartridges are required to provide the same filtering area as one bag. Add to this the fact that for the same area, bags (surface filtration) provide a higher filtration rate than cartridges (depth filtration). The net result is that under low pressure (max. 6 psi), a filter bag gives about the same throughput as 12 cartridges.

**Table 2—Typical Thickness of Industrial Coatings**

| Type | Thickness of film applied, mil. | |
|---|---|---|
| | Normal | Range |
| Coil coatings: | | |
| Primer | 0.2 | 0.2 ± 0.05 |
| Topcoat | 0.7 | 0.7 ± 0.1 |
| Electrocoats | 0.75 | 0.1 – 1.2 |
| Can coatings | 0.001 | 0.001 ± 0.002 |
| Gen. industrials | 1.0 | 0.8 – 1.2 |
| Appliance | 1.0 | 1.0 – 1.2 |
| Paper coatings | 0.1 | 0.1 – .2 |
| Wood coatings | 0.8 | 0.1 – 2.0 |
| Gel - coats | 18 | 16 – 20 |

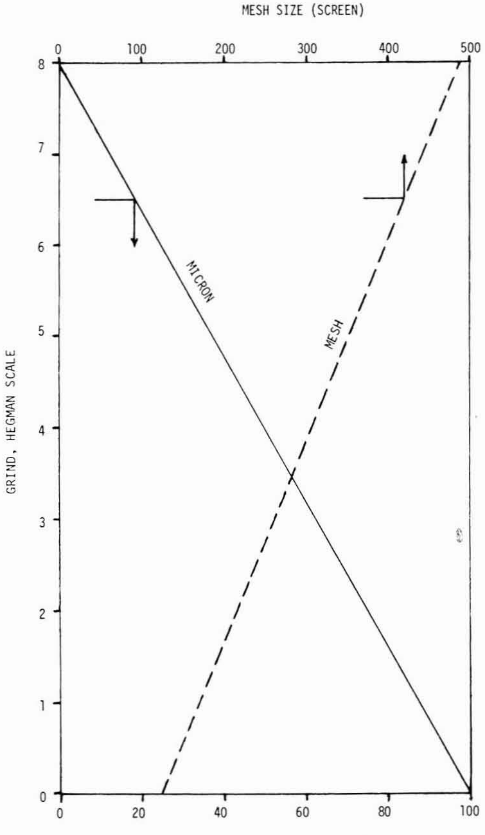

Figure 1—Mesh or micron filter size which should be used to filter a coating with a specific grind

### Continuous Filtration

Manufactured by Jenag Equipment Co., this is a totally enclosed self-cleaning strainer with a much higher throughput than a vibrating screen with a comparable sieve area because of the fact that suction/pressure can be applied on the material being filtered. The back-flushing system reduces cleaning downtime, and gives an almost constant throughput as the sieve is periodically back washed.

This unit is suitable for filtering large batches (over 2000 gal) and, basically, it finds an application on the same products suitable for vibrating screens, as discussed earlier.

## COMPARATIVE FILTRATION RATES

In general, the amount of product filtered/unit time/unit area increases as one goes from a tie-on bag filter, to a vibrating screen, to a cartridge filter and, finally, to a pressure bag filter. Although the operating cost of a vibrating screen is negligible compared to a cartridge filter, because the filtering medium of the vibrating screen is reusable, the time saved by using a cartridge filter instead of a vibrating screen may justify its use.

The set-up and clean-up time of all these filters is comparable, about 30 min, except the tie-on bag filter, which is much less.

### Filter Size or Mesh Opening

Below are a few criteria which are used to determine the cartridge size or screen mesh of the filter media. Sometimes more than one of the criteria may be applicable to a coating, and, if they conflict, one of them will have to be discarded.

### Thickness of Coating Applied

Final dry film thickness of the coating should be one of the major considerations for the selection of micron size of the cartridge or screen mesh opening. For the coating to appear smooth, the particle size of the dispersed pigment should be less than the coating film thickness. If it is larger, the pigment particle will be visible above the film, resulting in reduced gloss and a rough texture. Therefore, the mesh or micron size of the filter media selected to filter the coating, or the degree of fineness of the premix (as measured on a Hegman grind gauge), should be equal to or less than the thickness of the coating applied.

*Tables* 1 and 2 show comparative fineness, and film thickness of typical industrial coatings. For example, *Table* 2 shows that a coil coating top coat would have to be dispersed to a 6.5 grind on the Hegman scale and then be filtered through a 10 micron cartridge, the next smaller size cartridge commercially available after 25 microns. The same result can be obtained from *Figure* 1 which gives the mesh or micron filter size which should be used to filter a coating with a specific grind.

### Gloss

As the desired gloss of the coating increases, the particle size of the dispersed pigment particle must decrease.

| | Desired Glossmeter Reading | Required Hegman Grind |
|---|---|---|
| High gloss | 90+ | 7+ |
| Semi-Gloss | 60 – 80 | 5 – 7 |
| Flat | 0 – 15 | 0 – 7 |

A coating can have a 7 grind on the Hegman scale, and still be given a low gloss by the addition of selected flating pigments.

### Color Development of Pigment

Certain pigments have to be dispersed to a specified fineness in order to achieve properties such as color or transparency. For example, a mixture of phthalo blue and titanium dioxide with a $7^+$ Hegman grind will be a darker blue than the same mixture with a 5 Hegman grind; quinacridone pigments require a $7^+$ Hegman grind to become transparent.

## SUMMARY

The straining operation in a chemical coating facility is a major factor in determining throughput. Simply increasing the filtering media would increase throughput, but not necessarily improve cleanliness and certainly increase cost. To optimize the straining operation, first select the straining equipment that will give the maximum filtration rate and then the coarsest filter that will do the required job.

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# Volumetric Computer Color Matching

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The paper explains how conventional computer color matching (CCM) programs have been modified to yield data in volumetric units for greatly expanded flexibility and cost savings. Volumetric handling and dispensing of coloring materials are discussed in their application to a variety of laboratory and manufacturing situations common to the coatings industry, including the new innovation of totally automated factory tinting. The advantages and limitations of this approach to CCM are frankly discussed.

## INTRODUCTION

The intent of this paper is to present some simple concepts which are being used to expand the versatility of computer color matching (CCM), to illustrate some money saving ideas achievable by improving color matching efficiency and productivity, and to present some ideas for helping to work more effectively with some special color matching situations often encountered in the coatings industry.

It is not our intent to provide programming information or present programming examples, but simply to illustrate a few cost saving or problem solving ideas.

## DISCUSSION

### Concept

The concept behind the volumetric approach to CCM is simple: Volumetric expression of computer calculated colorant formulations or batch adjustments has been added to the output format of existing CCM programs without altering current pigment calibrations. This allows a variety of volumetric dispensing devices to be efficiently and effectively utilized in the color matching process without compromising match quality.

Utilization of volumetric dispensing devices will in turn allow many tedious and often unnecessary weighing steps to be eliminated. This can result in greatly improved efficiency and productivity in many color matching situations with realization of significant cost savings.

### Advantages and Limitations

None of the well known advantages of CCM are compromised by making the match volumetrically. However, the money saving advantages of decreased matching time or of increased productivity can be measured in real terms.

Of course, for every added advantage we may have to accept a limitation. A major limitation of volumetric CCM is that all of the coloring ingredients must be in predispersed form in order to be dispensible. Obviously, a laboratory or manufacturing operation using dry pigments entirely cannot take advantage of volumetric methods. However, this is an exception to the general rule. Most color matching situations within the coatings industry, particularly with the utilization of computer techniques to obtain a final color adjustment, use liquid or semi-liquid dispersions of color pigments.

Another limitation that should be considered is the loss of confidence which may be associated with volumetric techniques. However, considering weighing of ingredients to be an absolute certainty is dubious. What is needed with volumetric techniques is development of a high confidence level based upon results. However, knowledge of the accuracy of dispensing devices is a must, as are periodic calibration and preventive maintenance if appropriate.

### Volumetric CCM Programs

Now that the concept, advantages, and limitations of volumetric CCM are understood, the next thing needed will be a CCM program which will yield output data in

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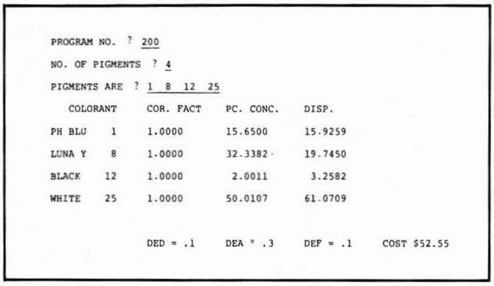


```
PROGRAM NO.  ? 200

NO. OF PIGMENTS  ? 4

PIGMENTS ARE  ? 1  8  12  25

   COLORANT       COR. FACT     PC. CONC.     DISP.

PH BLU     1      1.0000        15.6500       15.9259

LUNA Y     8      1.0000        32.3382       19.7450

BLACK     12      1.0000         2.0011        3.2582

WHITE     25      1.0000        50.0107       61.0709


                  DED = .1      DEA = .3      DEF = .1      COST $52.55
```

**Figure 1—Automatic formulation, conventional**

volumetric units. Although volumetric outputs are standard or custom options in certain commercial CCM programs, this type of output format will need to be programmed into other commercial programs, older versions of commercial programs, and captive programs.

To illustrate volumetric computer outputs, let us refer to a program, available commercially from Davidson Colleagues, called COLOR2. A variety of volumetric options can be added to COLOR2 as a custom service; volumetric expression is standard on certain other commercial programs. *Figure* 1 illustrates the conventional output format of an automatic formulation program. After the operator enters the spectral reflectance data of the reference color and chooses suitable colorants, the program calculates the best match expressed as percentage concentrations of dry pigment and pigment dispersions (shown in the last two columns).

As shown in the very abbreviated flowchart in *Figure* 2, the program calculates the percent by weight of dry pigment to make the match by using stored calibration data. Then, using stored pigment percentages of dispersions, the parts per 100 parts of each dispersion are calculated. Finally, using stored price data, the cost of the match per 100 pounds of ingredients is calculated.

Altering the program to yield volumetric output data is illustrated by the abbreviated flowchart in *Figure* 3. The percentages and parts per 100 parts of colorants are calculated as before. The operator specifies the exact volume of paint to be tinted. Then, the volumetric units are calculated using a simple arithmetical process. Finally, the actual cost of materials used can be calculated. Note that none of the stored calibrations, percent pigment, or price data needed to be changed.

The automatic formulation from *Figure* 1 is illustrated in *Figure* 4 with the volumetric output expressed in fluid ounces and 1/48ths ounces. Note the extra interactive step where the program asks for the paint volume to be tinted. Although the previous illustration used fluid ounces, a wide variety of volumetric outputs can be programmed to be consistent with user requirements.

*Figure* 5 illustrates a conventional format for batch adjustment using the COLOR2 program. The actual weights of dispersions contained in the batch are specified by the operator. Their percentages, correction factors, i.e. relative error, the new percentages necessary to correct the batch, and weights of dispersions to be added to correct the batch are calculated by the program.

Using the volumetric approach, illustrated in *Figure* 6, the operator inputs paint and dispersion volumes instead of weights and the program calculates the volumes to be added to correct the batch.

## Major Uses

Now that the reader has been provided with concept, advantages and limitations, and program examples, we shall turn to a discussion of the major potential uses of volumetric CCM. At the same time, various dispensing devices especially suitable for particular applications will be discussed.

The major areas where volumetric CCM can, and does, provide utility are: color systems, both trade sales and industrial; as a paint formulating tool; in manufacturing; and as a marketing tool.

COLOR SYSTEMS: First, let's evaluate the utility of volumetric CCM in working with color systems, both for trade sales and industrial finishes such as consumer coatings, general industrial, and automotive refinishes.

For purposes of definition, a color system can be thought of as a series of tintable paints, usually called tint bases, consisting of a series of hiding power levels (including unpigmented, colored, or aluminum tint bases for certain applications) and often representing a variety of product lines. These are tinted at the retail store, factory outlet, or application site with a series of liquid colorant dispersions, typically 10 to 16 in number. The colorants are volumetrically introduced

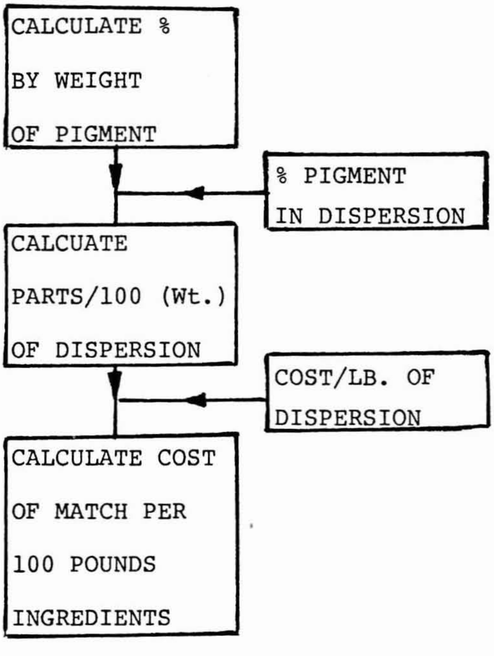


**Figure 2—Automatic formulation flowchart**

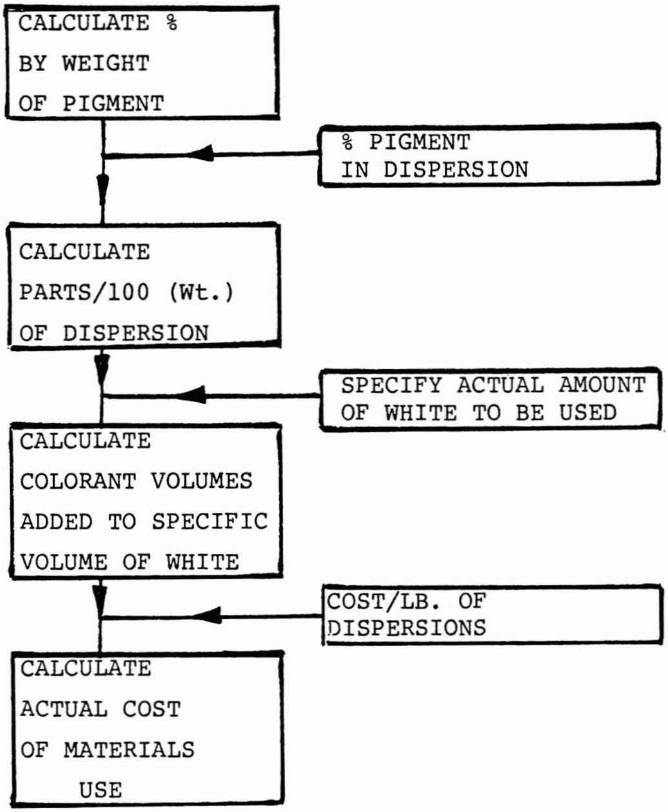


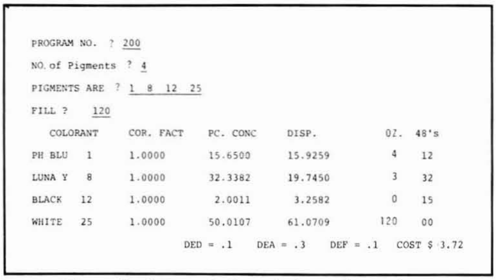
**Figure 3—Volumetric automatic formulation flowchart**

using a series of dispensing cannisters with self-contained storage reservoirs. Dispensing can be done manually or fully automatically. Using published colorant addition formulas, a wide variety of colors can be produced.

With all color systems, special emphasis is placed on the decorative qualities of the coating. This is especially true with trade sales systems which are merchandised using attractive color display racks, decorating guides, and color cards. The intention, of course, is to market color, not just paint.

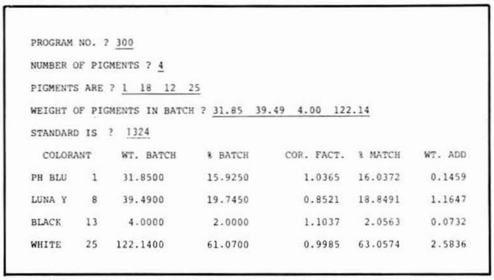
Volumetric CCM can be used with particular effectiveness in color systems by producing new color arrays (not by just "punching out" a huge number of colorant permutations, but by quickly computer matching a creatively designed new color array), in producing new color formulas in the quickest possible manner, in troubleshooting problems, and may even save enough time and expense to make a custom color matching service an economic feasibility.

*Calibration Techniques* — Since most color systems not only represent a variety of product lines, but also a ladder series of white hiding pigment levels within each product line, it may be worthwhile mentioning that colorants can be calibrated in the conventional manner



```
PROGRAM NO.  ? 200

NO. of Pigments  ? 4

PIGMENTS ARE  ? 1  8  12  25

FILL ?    120

   COLORANT      COR. FACT     PC. CONC      DISP.            OZ.  48's

PH BLU    1      1.0000        15.6500       15.9259           4    12

LUNA Y    8      1.0000        32.3382       19.7450           3    32

BLACK    12      1.0000         2.0011        3.2582           0    15

WHITE    25      1.0000        50.0107       61.0709         120    00

                          DED = .1    DEA = .3    DEF = .1   COST $ 3.72
```

**Figure 4—Automatic formulation, volumetric**



```
PROGRAM NO. ? 300

NUMBER OF PIGMENTS ? 4

PIGMENTS ARE ? 1  18  12  25

WEIGHT OF PIGMENTS IN BATCH ? 31.85  39.49  4.00  122.14

STANDARD IS  ?  1324

  COLORANT     WT. BATCH      % BATCH        COR. FACT.  % MATCH     WT. ADD

PH BLU     1    31.8500       15.9250           1.0365   16.0372      0.1459

LUNA Y     8    39.4900       19.7450           0.8521   18.8491      1.1647

BLACK     13     4.0000        2.0000           1.1037    2.0563      0.0732

WHITE     25   122.1400       61.0700           0.9985   63.0574      2.5836
```

**Figure 5—Automatic batch adjustment, conventional**

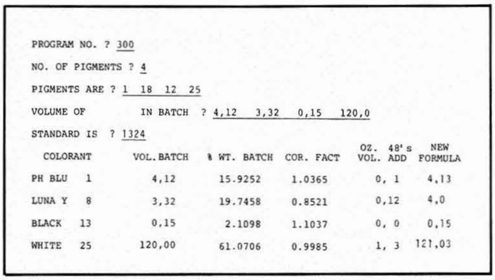
by adding each colorant to just one white base to obtain its absorption and scattering coefficients.

However, since color systems contain a limited number of colorants, a user may consider separate calibrations in tint bases representing each titanium dioxide level or in each product line represented. This will minimize the effects of such idiosyncrasies as selective pigment flooding, floating, flocculation, color contribution of extenders, vehicles, additives, and critical PVC effects. This should yield improved accuracy of the initial trial match and make fewer corrections necessary.

Many color systems also use colored tint bases containing substantial amounts of both white and colored pigments. This is done to improve the hiding of bright, organic red, yellow and orange colors and to minimize the amount of pigment added in dispersed form. Because no white base is used in making matches, some users may be confused or intimidated by colored tint bases.

However, calibrations and color matching using colored tint bases in place of white bases can be easily accomplished in one of three ways, depending upon the versatility of any particular color matching program. First, one can treat the colored base as if it contained a white pigment, with its colored pigment calibrated separately. When making matches, however, the program must be versatile enough to specify a constant color pigment to white pigment ratio. Second, one can calibrate each colorant in the colored tint base and simply ignore the constant ratio of colored pigment present. Third, the colored tint base can be calibrated in a compatible white base to obtain its relative K and S values. These can be used in place of the relative K and constant S = 1.0 used for more conventional "white" pigments if the particular program allows.

*Dispensing Devices for Color Systems* — The most commonly used dispensing devices for both laboratory and field use of color systems are the colorant dispensing machines which are familiar objects in paint stores and paint sections of department stores. The best types have precision machined parts and storage resevoirs fitted with slow speed stirring mechanisms. Most are well engineered, durable, require infrequent maintenance, but often suffer from inaccuracies at small dispensing volumes on the order of 1/10 fluid ounce or less. For general purpose use in volumetric CCM they are usually adequate.



```
PROGRAM NO. ? 300

NO. OF PIGMENTS ? 4

PIGMENTS ARE ? 1  18  12  25

VOLUME OF            IN BATCH  ? 4,12   3,32   0,15   120,0

STANDARD IS  ? 1324
                                                          OZ.  48's   NEW
  COLORANT         VOL.BATCH   % WT. BATCH  COR. FACT   VOL. ADD  FORMULA

PH BLU    1          4,12       15.9252      1.0365       0, 1     4,13

LUNA Y    8          3,32       19.7458      0.8521       0,12     4,0

BLACK    13          0,15        2.1098      1.1037       0, 0     0,15

WHITE    25        120,00       61.0706      0.9985       1, 3   121,03
```

Figure 6—Automatic batch adjustment, volumetric

BRUCE H. TODD is Technical Service Manager, Coatings and Plastics, for the Pigments Div., Chemetron Corp. Prior to joining Chemetron in early 1977, he was Group Leader, Color Systems and Standards, and Project Manager for the Dutch Boy Paints Div., NL Industries, Inc. He has also held coatings technical service positions with Cyprus Mines Corp. and Rohm & Haas Co. Mr. Todd earned his B.S. Degree in Chemistry from Philadelphia College of Textiles and Science and his M.B.A. degree from Seton Hall University.

Automatic dispensers have been available for a limited number of years. The newest development in automatic dispensing is the Graco Series 2000 Automatic Color Processor which combines quality dispensers with a solid state microprocessor. When used for volumetric CCM, the operator merely inputs the desired colorants and amounts via keyboard for automatic dispensing into quarts, gallons, or 5-gal containers.

The ultimate in state of the art technology, however, is an innovation recently introduced by Applied Color Systems. Its key feature is that the colorant dispensing module is directly coupled to either the ACS-500 or 600 Computer Color Control System for total integration of the spectrophotometer, computer, and colorant dispensers.

Called the Spectra-Tinter, the system can measure a color sample, compute the match formula or correction from a field of 12 or 16 colorants, dispense into an operator selected batch size, and store the data in memory. Storage, retrieval, preparation, and correction of standard recipes is also done automatically. Spectra-Tinter is ideal for use with color systems, but, of course, can be effectively used in a variety of other situations.

The heart of the Spectra-Tinter is a unique dispensing system containing two pumps per colorant. The large pump dispenses 1/10 fluid ounce per shot; the smaller pump dispenses in miniscule increments of 1/100 fluid ounce per second. Under computer control and at two shots per second, the system can dispense colorants with a previously unattainable combination of speed and accuracy.

FORMULATING TOOL: Computer color matching is undoubtedly an effective laboratory tool for the formulator. Volumetric techniques add utility and speed to the process.

Standard amounts of tinting colors or shading bases can be quickly calculated for use in formula costing or for production guidelines.

No one likes to use off-standard raw materials, but when the inevitability arises, accurate usage recommendations or formula adjustments can be quickly obtained for marginal, but usable colorants.

Money saving consolidation of factory tinting colors can be accomplished much quicker than by color matching by weight.

Color related troubleshooting can be done in more expedient manner.

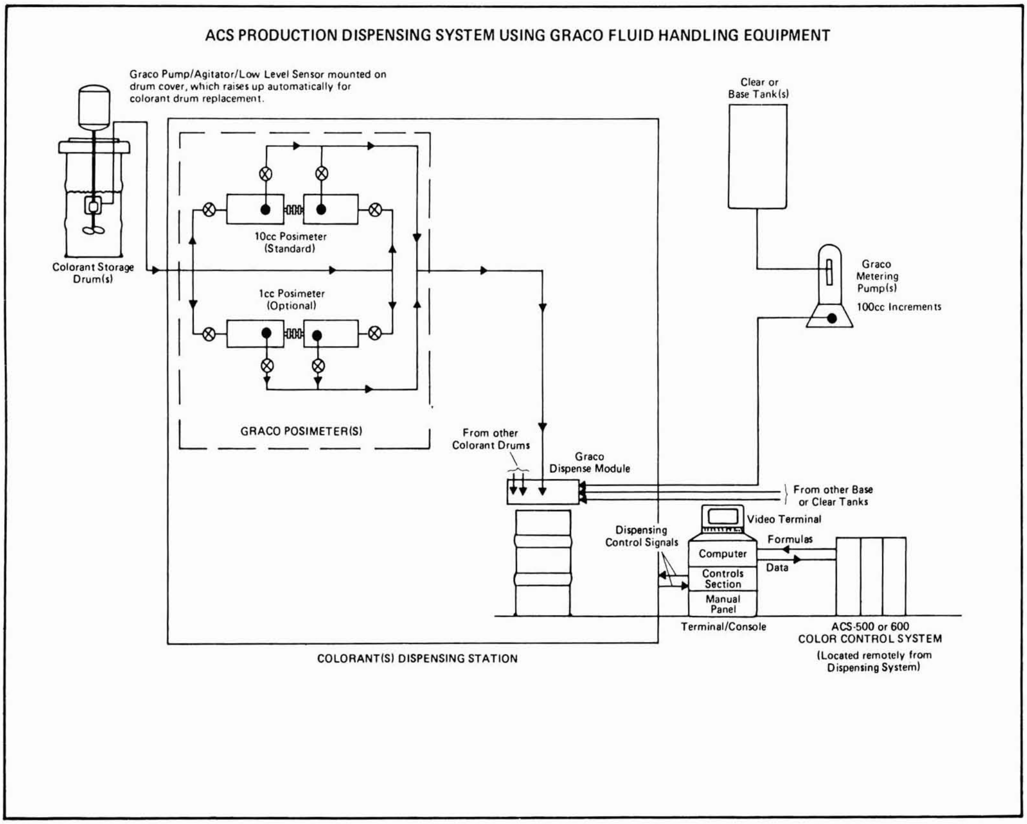


**Figure 7—ACS production dispensing system using Graco fluid handling equipment**

It is also expected that volumetric tinting will allow quicker preparation of cost-optimized paint formulas when integrated into a pigment loading or hiding power formulating program.

*Dispensing Devices for Formulating* — All of the dispensing devices mentioned previously are applicable to laboratory formulating, provided accuracy and reproducibility are acceptable. The Spectra-Tinter would be especially suitable if the field of colorants is not large.

For very small colorant volumes the Pantone® or IGT* ink pipets are quite suitable because of their accurate micrometer feature. Their limitations are lack of colorant reservoirs and need for clean-up.

Another device which is very useful for small colorant volumes and has good accuracy is the common plastic throwaway hypodermic syringe.

"Plunkerator" devices, simple calibrated sleeves fitted with pistons, are less accurate, and must be cleaned; but they may find usefulness in less discriminating situations.

Marketing Tool: The added speed and efficiency gained when volumetric techniques are added to CCM systems may have enough of a cost impact to make use of custom color matching or custom color production as a marketing tool. Many good marketing ideas associated with color are, regrettably, unfeasible because of expense versus expected benefit to be derived from color matching. When the lower cost per match and added productivity of volumetric CCM are introduced, however, many otherwise excellent ideas may be brought into the realm of economic feasibility, particularly if ingredients are standardized, the work is systematized, and as much automation as possible is introduced.

Manufacturing: It should be obvious at this point in the discussion that volumetric CCM can take an important role in paint manufacturing. Since the volumetric approach requires pigment dispersions, it is consistent with modern manufacturing practices which utilize pigment slurries, volumetrically meter major liquid ingredients, prefer the use of easy mixing tinters, and utilize simple mixers, high speed dispersers, or sand mills in preference to pebble mills, attritors, and three-roll mills.

We have already touched upon the use of volumetric CCM in certain laboratory situations which have close analogies in manufacturing: elimination of weighing

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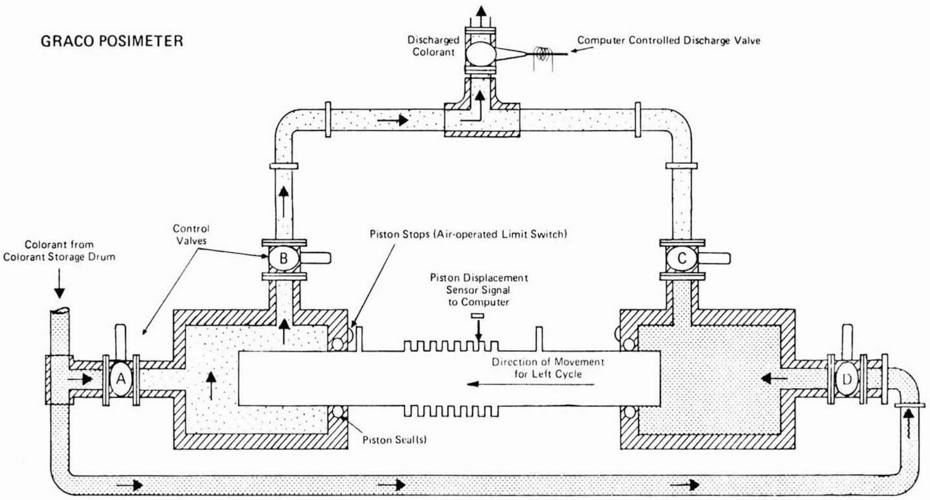
Trade name of Pantone, Inc.
*Trade name of the Research Institute for the Printing and Allied Industries.



**LEFT CYCLE**

Inlet Valve "A" Closed
Inlet Valve "D" Open
Discharge Valve "B" Open
Discharge Valve "C" Closed

Valves switched automatically for right cycle

**OPERATION**

As piston moves to left (inlet colorant at valve "D" under pump pressure as a result of pumping system at colorant drum), it displaces colorant in left cycle chamber, forcing fluid out through discharge valve "B" and to mixed colorant drum.

Right cycle begins when piston engages left cycle stop limit switch and valves "B" and "D" automatically close and valves "A" and "C" automatically open. Inlet colorant (under pressure) now enters left chamber through valve "A", and piston moves to right forcing colorant through valve "C".

**Figure 8—Graco Posimeter**

steps, expeditious problem solving and troubleshooting, colorant consolidation programs, economical production of custom colors, and automatic tinting systems.

The last point, automatic factory tinting, deserves further discussion because it is a recent development which is a major innovation for the coatings industry. The ACS Colorant Dispensing System can be used for automatic shading of full size factory batches. The system, jointly developed by Applied Color Systems and Graco, uses ACS computer and color matching expertise combined with Graco pumping and metering technology.

*Figure* 7 is a schematic diagram illustrating an automatic production dispensing system. The system works in the following manner*:

- Base data is stored in the computer. Color formulas, updated colorant inventory, and application programs are retained by a remotely located ACS model 500 or 600 color control system. The same system can also service your laboratory color control needs.
- Formulations are transmitted from the color control system to a Dispenser Controller. The Controller, a second microprocesser, becomes the command station for the dispensing operation. Up to 100 job formulations are transferred for production use at any one time by a key operator. Adjustments can be made to the formulations at this time, prior to dispensing.
- When ready for production dispensing the production operator enters formula identification on a remote terminal. The Controller automatically retrieves the proper formulations and awaits a batching command. The operator enters the batch quantity and dispensing begins.
- Dispensing streams are controlled simultaneously for all items in the batch. The base or extender is pumped initially to coat the container, then all colorants are dispensed in a simultaneous staged sequence to assure that the color is interspersed through the batch.
- Throughout the operation, the Controller (microprocessor) monitors the colorant level in the supply tanks, controls sequence of adds, collects material usage data, and, if the sequence is halted for any reason, can provide information as to the materials batched to that point.
- The key to precision fluid handling is the patented Graco Posimeters which are precision machined dual displacement reciprocating metering cylinders. They are fed and powered by the supply fluid under pres-

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*Reprinted with permission from ACS literature.

sure from the supply pump modules. The unique design of the Posimeter allows direct control of the dispensing sequence by the Dispenser Controller. A computer controlled slowdown feature assures accuracy during the critical last steps in the batching process.

The standard posimeter dispenses in 10 cc increments and, for even greater accuracy, 1 cc Posimeters are also available. See *Figure* 8 for operating details of the Posimeter.

A typical color match is said to be accomplished three to five times faster than when using manual weighing.

### Helpful Hints for Volumetric CCM

Anyone trying the volumetric approach to CCM is advised to keep the following suggestions in mind:

(1) Well calibrated tints are essential to the success of any computer color matching process, volumetric included.

(2) If colorants are actually mixtures of two or more colored pigments, they can be calibrated and utilized as if they were a single pigment, for simplicity.

(3) Don't try to treat pigment dispersions as if they were 100% solids dry pigments. Results will be unsatisfactory.

(4) To help alleviate the uncertainties which may accompany volumetric techniques, pay close attention to program "correction factors" which are the first indication of error.

(5) Keep dispensing devices in good condition. Do not allow them to clog or become dirty. Use a preventive maintenance program for bushings and parts subject to wear.

(6) Use periodic accuracy checks to assure confidence.

(7) Avoid trying to produce color matches using "micro" quantities of colorants: i.e., quantities of colorants which are too small for accurate dispensing. Error may be magnified and results prove unsatisfactory.

(8) Pay close attention to colorant stability. Even the best dispensing devices can have their accuracy compromised by settling, vehicle separation, unusually high viscosity, or unusual rheological properties.

## CONCLUSION

In conclusion, it is appropriate to list those who should be considering the volumetric approach to computer color matching:

Every laboratory working with a color system or which can take a systems approach to color matching.

Every laboratory producing color matches using liquid colorants.

Every manufacturing plant using computer color matching with shading dispersions.

Manufacturers of custom colors.

Every company which can use color matching as a marketing tool.

Every company interested in automated paint production.

Any firm doing a considerable amount of color matching that wishes to save time . . . save money.

## ACKNOWLEDGMENT

The author thanks Mr. Hugh R. Davidson, of Davidson Colleagues, for permission to use his COLOR2 program format in my examples and Mr. James T. DeGroff, of Applied Color Systems, for supplying schematics and literature on automatic tinting systems and for permission to refer to the ACS/Graco equipment.

# Development Of Computer Color Formulation And Control

Hugh R. Davidson
Davidson Colleagues*

Eloquent testimony to the effectiveness of instrumental color matching has been given by the Symposium on Color and Appearance Instrumentation. Many suppliers, competing in the field, and many producers of colored materials came to evaluate the new equipment and methods. But we cannot claim complete success in all color formulation and control problems, so it is well to consider both the strong points and the weak points of our instrumentations.

## Introduction

In the comparatively short time since instrumental color matching was introduced in the late '50's, the technique has made considerable progress. This is well exemplified by the comments and questions raised by potential users. In the early days, the most common comment was "It won't work." A long string of reasons could be given ranging from the supposed mystical nature of color to lack of control of manufacturing conditions which, of course, is still a valid comment today. A few years later the comments had changed to "It may work for some companies, but not for mine." Again, a long string of reasons could be given, but they were considerably more rational—success had eliminated some of those based primarily on superstitions of one sort or another. Today the comments are usually something like this: "Yes, it will probably work for me, but is it worthwhile and, if so, what is the best system for me?"

Now that we are in the era of rational discussion, the promoters of instrumental color matching can afford to be less dogmatic. We can admit that there are some areas of poor performance, and even some cases where the cost is simply not justified by the results.

During these twenty years or so we have gone from simple, one-constant, manual-input analog computer to automatic-input, high-speed, digital computers employing two-constant programs. Our systems have become much faster, much easier to operate, and much more flexible in that they can be applied to a wider range of color problems. It is interesting to note, however, that in those plants where the best practices have been followed since the first systems were used, the average accuracy, as measured by the number of hits required to adjust a batch for example, has not improved dramatically. Computations can be obtained much faster today, of course, but it is apparent that even rather primitive instrumentation, when used intelligently, can produce results comparable to those obtained with our most sophisticated systems.

Oddly enough, during this period of growth in our hardware, our basic theoretical approach has not changed at all. We still rely on the simple Kubelka-Munk two-constant approach. Some of our mathematical techniques have become more sophisticated, but we still deal primarily with a constant of scatter and a constant of absorption. Other theoretical approaches have been proposed, but so far they have not led to any overall improvement in accuracy. This may be so simply because the two-constant approach, with empirical corrections where called for, is sufficiently accurate for most problems. As an industry, we have not as yet taken full advantage of what we have.

## Success in Areas of Use

Color control instrumentation has been most successful in establishing initial formulations where metamerism is a problem, and in reducing the number of corrections required in production. There is no doubt that in most cases we can make better nonmetameric matches, much more quickly, with instrumentation than we can without instrumentation.

In the case of batch corrections, the improvement is due partly to a direct and partly to an indirect effect of instrumentation. When instrumentation is used, there is a general tendency to pay more attention to production variables and errors, a general cleaning up of production methods, in other words. This in itself produces better control and, hence, fewer batch corrections. In any case, there is usually a marked decrease in the number of corrections when instrumentation is introduced.

We often claim great success for instrumentation in reducing the total cost of pigments used in production. Un-

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Presented by Mr. Davidson at the Symposium on Color and Appearance Instrumentation, March 15, 1978, Cleveland, Ohio.

*P.O. Box 157, Tatamy, Pa. 18085.

doubtedly this is true in some cases, but our claims are probably a bit too strong for the average case. If a manufacturer has paid reasonable attention to pigment cost before introduction of instrumentation, the chances are that he will show only a modest cost decrease by use of instruments.

### High Chroma and Metallic Colors

There are two types of paint colors in which our success has been less than desired. The first of these is the area of very deep or high chroma colors, that is, on colors which contain little or no white pigment. Our measurements seem adequate on these colors. The major difficulties encountered with these colors involve (1) the accuracy and repeatability with which the paint mixtures can be prepared and the panels made and (2) the method used for characterizing the absorption and scattering of the individual pigments. A difference in dispersion of a pigment may be of no importance when it is mixed with a quantity of white, but can be of major importance when little or no white is present; the scattering characteristics of a pigment must be known accurately to predict dark and high chroma colors; a difference in surface reflectance so small that it cannot be detected with gloss measuring equipment can cause a visible color difference on dark colors. In short, the effects of all areas of poor control and inadequate pigment characterization are magnified many fold on dark and high-chroma colors, contributing significantly to poorer results with computer colorant formulations.

The second type of paint color on which we have had relatively little success is metallics. Our present programs can be helpful, but real success still eludes us. Part of the problem is measurement — instruments utilizing variable angles of illumination and viewing are not commercially available here; the other part of the problem is the lack of a workable mathematical model to use with the angular measurements, if we had them. Full success will not be realized unless more appropriate measurement instrumentation and further research is brought to the effort.

### Types of Instrumentation

Now, assuming that instrumentation for color formulation and control is desirable, what is available? Details of various instruments and computing systems cannot be discussed here, but a few general descriptions and costs should be helpful to those considering a purchase.

Measuring instruments can be divided into two general categories, colorimeters and spectrophotometers. Colorimeters are far less expensive than spectrophotometers, but can be recommended only for production control. If any formulation, pigment strength determination, or general color trouble-shooting is to be done, a spectrophotometer is necessary. Colorimeters range in price from about $4,000 to $10,000; spectrophotometers range from about $12,000 to $20,000 and even much higher.

There is also a wide range of choice for the computational systems that are needed for formulation and batch adjustment. The simplest is commercial time-share. In this case, the operator uses a terminal to communicate by telephone with a large computer in some remote location. The terminal costs range from about $1,500 to $3,000, and aside from the measuring instrument this is all the hardware needed. An initial fee of about $500 is paid to subscribe to the system and from then on the charges are for use only. The costs range around $2 for a batch correction and $2 to $8 for an initial formulation. If large numbers of batch corrections or formulations must be made, this method becomes expensive, but it still may be justified as a first step in setting up an instrumental system.

Use of a computer presently owned by the company is another possible step. In this case a computer program may be purchased and placed in the company computer. Program costs range from around $10,000 to $15,000 depending on the computer and complexity of the program. The computer used may be either a time-share system or one used primarily for color work.

Several companies are now offering complete systems including the measuring instrument, computer, printer, and a suitable enclosure. The least expensive systems presently available use colorimeters and are recommended only for batch corrections. The computing system costs about $23,000 and the colorimeter about $5,000, making a total package cost of about $28,000. For full formulation capability a spectrophotometer must be used, thus raising the minimum cost to about $40,000. From here, the prices range up to about $80,000 for a complete system. Increased computer size and complexity, time-sharing capability, speed of operation, and various options on peripheral equipment are some of the factors leading to this wide price range.

The best advice that can be given to anyone considering the use of instrumentation for color control is to shop around. All systems or methods offered have their advantages and disadvantages. No one is best suited to all applications or to all particular circumstances in all companies. Only by comparing all systems can an optimum selection be made.

### Conclusion

Please note that no method or system will be successful unless the operator wants it to be. All system manufacturers can point to some of their own systems which are standing idle simply because the operators had no real interest in making them work. No magic buttons are available which, when pressed, solve all color problems, but all systems now on the market will be successful in most applications if intelligently and diligently used.

# Constituent Societies Report on Education Programs

This report is published as part of the Federation's ongoing efforts to keep members informed of educational activities, both underway and planned, and to stimulate interest in formulating new programs at both the local and national levels.

### Baltimore

Jointly sponsoring, with local PCA, a scholarship award for children of members . . . Continuing sponsorship of annual seminar.

### Birmingham

Completed slide-tape program on "Setaflash Tester" for Federation training series; work continuing on three additional programs for the series.

### Chicago

Efforts are jointly sponsored with local PCA . . . Two seminars are scheduled each year: one technical, and one management; 1978 management development seminar dealt with understanding human behavior, particularly between age groups, and technical seminar (SYMCO '78) was devoted to technology of polymers involved in the coatings industry . . . 1979 management seminar will be held April 26; SYMCO '79 will be held March 20 & 21, and will focus on pigments and color . . . Coatings technology course has been started at Elmhurst College, the first such course in the Chicago area in five years—approximately 100 students registered for the 31 sessions; current plans are to present the course every two years . . . No coatings curriculum is offered at any area colleges, and efforts are underway to orient faculty at Elmhurst in this direction.

### C-D-I-C

Educational presentation precedes each Society monthly meeting; topics covered in recent programs included toxicity testing, acrylic urethanes, and lasers.

### Cleveland

Presented program on "Science and Technology in the Coatings Industry" for high school science teachers at Cleveland Regional Council Science Teachers Workshop; teachers attending were later invited to Society monthly meetings to hear presentation on "Chemistry and Art" . . . Continuing to assist in publicizing annual nine-session Saturday morning credit course on organic coatings, held September through November at Kent State University . . . Sponsored two-day symposium in March on "Advances in Coatings Technology" — program addressed three major topics: "Characterization and Behavior of Coatings;" "Testing and Coatings Consensus Standards;" and "Environmental and Toxicity Influences on the Coatings Industry" . . . Assisted in judging for Greater Cleveland Science Fair (also awarded, on behalf of Society, a savings bond for best project with a coatings theme), and will repeat effort in 1979 . . . Completing production of slide/tape presentation on "Tinting Strength of Paint by Instrumental Means" . . . Issued call for papers for 1979 symposium, to be held March 20-21 . . . Continuing contributions to Cleveland Public Library, Kent State University, and Case-Western Reserve University for purchase of coatings textbooks.

### Dallas

Continuing support of scholarship program at University of Southern Mississippi . . . Hosted local high school chemistry teachers at regular Society monthly meeting, at which Dr. Herman Lanson presented lab experiments on Hansa Yellow and Toluidine Red; teachers attending were given written outlines — program will be repeated in current meeting year . . . Planning one-day educational seminar in February; also planning technician's course at junior college, in conjunction with local PCA.

### Detroit

In cooperation with local PCA, set up and equipped coatings lab at University of Detroit's Polymer Institute . . . Jointly sponsors, with PCA and Polymer Institute, two courses: 14-week presentation on "Surface Coatings Technology;" and 12-week course on "Modern Resin Technology;" also helps publicize University of Detroit's credit courses on "Chemistry of Protective Coatings" and "Synthesis of Organic Coating Polymers;" as well as annual Polymer Conference Series . . . Sponsored annual Symposium on Future of Coatings Under Study (FOCUS) in April — topic was "Coatings for Automotive Plastics;" 1979 FOCUS, to be held May 3, will have as its theme, "Recent Advances in Automotive Coatings" . . . Purchased complete package of Federation A/V programs, and make them available on a loan basis to area paint companies (also include projector, if needed).

### Golden Gate

Actively participates in California Air Resources Board meetings . . . Coatings course for high school students is being continued; however, due to impact of Proposition 13, classes are held at company facilities rather than at high schools or colleges — this offers spin-off benefit of making local firms more aware of Society activities, as well as giving them opportunity to observe students taking courses, which should be helpful in job placement considerations . . . Study grant program has proved successful and is being continued — Society pays expenses of selected member to attend coatings seminar (there were three seminars selected for the program last year) and make report to membership . . . Two slide-tape presentations were completed for the Federation training series: "Wet Film Thickness Gages" and "A Salt Spray (Fog) Testing Cabinet." . . . New coatings section has been established at Redwood City Public Library which is sponsored and maintained by the Society.

### Houston

Monitoring developments of Texas Air Control Regulations . . . Continuing sponsorship of annual seminar.

### Kansas City

Invited area high school teachers to Society "Education Night" and presented kits on basic paint composition, making of rigid polyurethane foam, and preparation of Hansa Yellow and Toluidine Red . . . Participates in Greater Kansas City Science Fair, by assisting in judging of coatings-related projects and in providing three prizes — each winner receives $50, with matching sum awarded to prizewinner's school science department for purchase of lab equipment; students awarded prizes at "Education Night" meeting, to which their parents and high school teachers are also invited . . . Planning to expand experiments for the teachers' kits . . . Contributes to scholarship fund at University of Missouri — Rolla.

### Los Angeles

Continues active participation at hearings of California Air Resources Board and other regulatory agencies . . . Restructuring basic coatings course taught at Los Angeles Trade Tech School to encourage greater enrollment — curtailed funds as result of passage of Proposition 13 mandates increased attendance if course is to continue . . . Providing advisory assistance for polymer science courses offered at University of Southern California . . . Continuing scholarship funding for benefit of members — scholarships are awarded directly to students, basically children of members, who can attend college of their choice . . . Also continuing support of scholarship program at North Dakota State University, and maintenance of coatings library at City of Commerce.

### Louisville

Continuing to sponsor coatings courses at University of Louisville; last fall presented course which focused primarily on resins, followed last spring with a presentation on formulations. Currently underway is course on quality control; spring semester will offer course on instrumental analysis . . . Society, local PCA, and City of Louisville are jointly sponsoring new scholarship at University of Louisville to study "Industrial Waste Disposal."

### Mexico

Assisted in planning a graduate credit course on organic coatings presented at the University of Mexico; first group of students has completed course, and another more advanced course is contemplated . . . Has volunteered to translate Color-matching Aptitude Test Set instructional folder into Spanish.

### Montreal

Continuing sponsorship of annual 15-week evening course on Introductory Coatings Technology . . . Co-sponsored with Quebec Paint Industries Association a course for chemical coatings sales personnel which featured introduction to basic coatings technology, and wood and metal coatings . . . Planning to publish coatings technology information in French language, and to organize basis coatings course in French language . . . Working on plans for special short seminar on "What's New in Vehicle Polymers?"

### New England

Cooperated with Lowell University in the presentation of two courses: "Adhesive Chemistry" and "Polymer Chemistry;" these were offered as part of university's Master of Science program, but were available to undergraduates on an elective basis. Courses being offered this school year are "Coatings Science and Technology" and "Chemistry of Plastics and Foam" . . . Society schedules presentations of Federation A/V training programs prior to monthly meetings.

### New York

Continued sponsorship of two coatings courses: (1) "Fundamentals of Coatings Technology," two-semester (15 weeks, 2 hours per week) course for new chemists and advanced technicians; and (2) "Laboratory Course for Coatings Technicians" — 10-week (2 hours per week) "hands-on" lab course on the techniques of fundamental test methods, held at laboratories of various coatings and raw materials manufacturers . . . Also sponsored course on "Principles of Color Technology," a 10-week, 2-hr per week lecture presentation on color and color matching . . . Funded two scholarships at New York Institute of Technology . . . Applied to New York State for Vocational Education Grant to fund basic course to train laboratory technicians for the coatings industry.

### Northwestern

Continuing program of offering a pre-monthly meeting seminar based on a booklet from Federation Series on Coatings Technology . . . Have purchased Federation A/V programs, and make them available to members on loan basis . . . Assisted local vocational school in application for state aid to set up coatings materials lab technician program . . . Continuing contributions to North Dakota State University for purchase of coatings equipment.

### Pacific Northwest

Portland Section continuing sponsorship of two seminars and course on basic paint technology in Salem, Ore. . . . British Columbia Section also continuing to support coatings course at B. C. Vocational Institute . . . Seattle Section is sponsoring new coatings course; planning seminar for 1979.

### Philadelphia

Offering repeat of ACS course on coatings technology, conducted by Seymore Hochberg; eight 2-hour classes will be held prior to Society and/or Technical Committee meetings . . . Planning five-lesson course on color matching, with emphasis on colorimetry and computer color formulation . . . Also offering, in conjunction with Temple School of Pharmacy, two free courses: one on lab procedure useful in health and safety hygiene, the other on general health and safety matters . . . Completed production of slide-tape presentation on "A Simple Method to Determine Microbiological Activity" for the Federation training series.

### Piedmont

Contributed to Chemistry Dept. scholarship fund at Appalachian State University . . . Continuing efforts to initiate coatings courses at Appalachian State . . . Planning full-day workshop and/or seminar.

### Pittsburgh

Planning to participate in Spring Science Fair by awarding prizes and assisting in judging; will help stimulate interest in coatings projects by visiting high schools and presenting A/V programs on paint test procedures to chemistry and science classes . . . Will assist in preparation of Spring symposium on "Corrosion."

### Rocky Mountain

Monitors proposed regulations by Colorado Air Pollution Control Commission . . . Planning seminars on paint formulation, application, and troubleshooting, to be aimed at lower echelon lab personnel, and sales and paint store people . . . Annual June meeting featured presentation on Colorado State University's free service of providing a complete industrial hygiene inspection of a store or manufacturing facility.

### St. Louis

Continuing to devote one monthly meeting for educational purposes; area high school teachers are invited and given demonstration kits for classroom use . . . Developing member speakers' bureau for presentations at local high school on coatings industry career opportunities . . . Contributes funds to scholarship program at University of Missouri—Rolla.

*(Continued on page 90)*

# Society Meetings

## Birmingham Nov. 2

Ken Downes, of CIBA-GEIGY Ltd., spoke on "USE OF ORGANIC PIGMENTS IN PREPARATION OF OPAQUE PAINT FILMS."

Mr. Downes said that in 1973, samples of commercial decorative paints to match BS4800 08E51 and 10E53 had been found to be based either on lead chromes or on organics such as C.I. Pigment Yellows 3 and 1, which were poor matches with low opacity. A similar exercise in 1978 found all the paints to be based on organic yellows with titanium dioxide as an opacifier. He mentioned that C.I. Pigment Yellow 74 appeared to give the best results, two grades being available. They differed in strength and durability but both showed good opacity and flow properties.

Bright reds, such as BS4800 04E53 Poppy Red, could be obtained by various blends, but poor opacity was still a problem. This was an area where there was still a requirement for a new high opacity organic red, continued Mr. Downes.

Industrial paints, he explained, present further problems of resistance to strong solvents, heat, and acid catalysts. C.I. Pigment Yellow 74 can be considered for industrial systems. It does show a tendency to bleed on over-lacquering, but does not bloom out of the film. It also has excellent weather fastness, marginal color fade, and shows no sign of blooming on exposure. Mr. Downes then presented tables of paint cost per square yard for various pigment combinations that could be used to match Ford Tractor Yellow and International Harvester Red.

At equal weather fastness, the organics are 2-4 times the price. New shades will have to be adopted based on organics. Mr. Downes then presented color charts showing the feasibility of organic/oxide/white combinations to give acceptable opacity at 3 mil film thickness. There are still gaps in the range of organic pigments suitable to give opaque paint films, concluded Mr. Downes.

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*Q. Have you a quick technique for detecting the two grades of Pigment Yellow 74?*

A. Accelerated weathering in an Atlas Weather-Ometer® for 500-1000 hr.

BRIAN F. GILLIAM, *Publicity Officer*

Toronto Society for Coatings Technology Executive Committee for 1978-79. Front row (left to right): Secretary — A.G. Morris; Federation Council Representative — A.C. Boyce; President — S. Palleschi; President-Elect — H. Zapfe; and Treasurer — J.A. Grodzinski. Back row (left to right): Society Committee Chairmen — S.R. Balmer; L.T. Ham; G.E. Abboud; G. Major; R. Blondin; E.L. Humberger; and M. Hanzen. (Committee chairmen not shown: F.N. Reckless; F.W. Laroche; L. Harrison; T. Bird; and A. Jones.)

## C-D-I-C Dec. 11

A moment of silence was observed in memory of William Matteson, retired, and Frank Splinter, of PPG Industries, Inc., who died recently.

Steven E. Cantor, of Uniroyal Inc., spoke on "ROLE OF POLYELECTROLYTE AS A DISPERSANT FOR $TiO_2$ SLURRIES AND LATEX PAINTS."

Dr. Cantor said that good dispersion is helpful for hiding, flow or leveling, package stability, gloss and reflection, and high solids content. The mechanism for dispersing $TiO_2$ pigment is wetting, mechanical disruption, and stabilization. The physical forms associated with these steps, continued Dr. Cantor, are (1) aggregators—large, relatively solid masses of particles with little ability to disperse light; (2) flocculated—smaller masses of particles which still do not maximize the ability to disperse light; and (3) deflocculated—separation into individual particles with almost no agglomeration into masses with more than one particle. This maximizes ability to disperse light and hide the substrate.

Polyelectrolyte dispersants which have been used to disperse pigments include sodium or potassium polyphosphates, sodium polyacrylates (short oligomeric chaines per charge), and bisulfite regulated oligomers. The dispersant molecule should not be too large or it may absorb onto two or more pigment particles, resulting in flocculation.

A dispersant will decrease the viscosity of a slurry of $TiO_2$ in water (commercial grades are about 64% solids for flat and about 76% solids for glossy) to a certain minimum which is referred to as the demand point. Excess dispersant will cause the viscosity to go back up. An ideal dispersant will cause slight or no viscosity increase as excess is added.

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*Q. Is the dispersant demand for a mixed pigment system additive or a function of the system?*

A. Observation and literature indicate it is additive.

*Q. Why does the viscosity increase with excess dispersant?*

A. According to Helmholtz double layer effect, the "bumper" effect of the like charges becomes compressed and over-ridden, resulting in some attractions between particles and flocculation.

W.J. FROST, *Secretary*

## Cleveland Nov. 9

Benjamin Luberoff, of Chemtech Magazine, spoke on "MANAGEMENT AND BEING MANAGED."

Dr. Luberoff covered many facets of management. He mentioned that a well run company with a problem in even one department can have difficulties. Another point stressed by Dr. Luberoff was that we need to become effective listeners.

J.S. MALAGA, *Secretary*

## Dallas Dec. 14

Dr. B. George Bufkin, of the University of Southern Mississippi, presented a talk and slide program detailing the coatings and polymer science work done at the University during EDUCATION NIGHT. He discussed the University's source of finances as well as available scholarships and grants, which was of special interest to the students and teachers from area schools who were attending this education emphasis program.

RICHARD D. WILLIAMSON, *Secretary*

## Montreal Dec. 6

R. Rauch, of Tioxide of Canada, gave a history of the Technical Committee. He also described the function and aim of the committee. He then introduced the Chairmen of the Freeze Thaw and Adhesion of Latex Paints subcommittees who outlined their programs for the coming year.

Claude Dessureault, of Canadian Industries Ltd., spoke on "UNRESTFUL SITUATION OF MIDDLE MANAGEMENT."

Mr. Dessureault's presentation encompassed the problems and grievances encountered by middle management people in large and small, public and private companies.

CUTHBERT A. MCWADE, *Secretary*

## New York Nov. 14

Robert W. Lenz, of the University of Massachusetts, spoke on "UNUSUAL RESINS FOR COATING APPLICATIONS."

Dr. Lenz dealt with a number of recent developments in polymer science which could be applicable to the curing of surface coatings. Some of the developments covered included spontaneous polymerization reactions which require no catalysts or initiators, and which occur readily at low temperatures; polycondensation reactions which can be run in water or alcohol solvents; solid-state polymerization reactions; reversible crosslinking reactions; and polymerization systems in which volume shrinkage is minimized or eliminated.

Some of the possible applications to surface coatings include low energy curing reactions, reversible or re-usable crosslinked coatings, water-based systems having reactive functional groups for crosslinking, and the improvement

# Constituent Society Meetings and Secretaries

BALTIMORE (Third Thursday—Eudowood Gardens, Towson). GORDON ALLISON, McCormick Paint Works, 2355 Lewis Ave., Rockville, Md. 20851.

BIRMINGHAM (First Thursday—Calthorpe Suite, Edgbaston). BRIAN J. ADDENBROOKE, Croda Paints Ltd., Brodesley Green Rd., Birmingham B9 4TE, England.

CHICAGO (First Monday—meeting sites in various suburban locations). RICHARD M. HILLE, United Coatings, Inc., 3050 N. Rockwell, Chicago, Ill. 60618.

C-D-I-C (Second Monday—Sept., Nov., Jan., Mar., May in Columbus; Oct., Dec., Feb., Apr. in Cincinnati, Kings Island Inn). WILLIAM J. FROST, Ashland Chemical Co., P.O. Box 2219, Columbus, Ohio 43216.

CLEVELAND (Second Tuesday—meeting sites vary). JACK S. MALAGA, Body Brothers, Inc., 214 Northfield Rd., Bedford, Ohio 44146.

DALLAS (Thursday following second Tuesday—Vic's Gallery Restaurant). RICHARD WILLIAMSON, Trinity Coatings Co., P.O. Box 721, Fort Worth, Tex. 76101.

DETROIT (Fourth Tuesday—Rackham Memorial Bldg.). GARY VAN DE STREEK, Wyandotte Paint Products, Inc., 1430 Sycamore, Wyandotte, Mich. 48192.

GOLDEN GATE (Monday before third Wednesday—Sabella's Restaurant, San Francisco). SHARON VADNAIS, E.T. Horn Co., 7700 Edgewater Dr., Oakland, Calif. 94621.

HOUSTON (Second Wednesday—Marriott Hotel, Astrodome). SAMUEL L. LOE, JR. Samuel Loe Co., Inc., 4715 S. Main St., Houston, Tex. 77002.

KANSAS CITY (Second Thursday—Washington Street Station). WILLIAM SMITH IV, Conchemco, Inc., P.O. Box 37, Kansas City, Mo. 64141.

LOS ANGELES (Second Wednesday—Steven's Steak House). JAN P. VAN ZELM, 5412 Calle de Arboles, Torrance, Calif. 90505.

LOUISVILLE (Third Wednesday—Essex House). P.W. HARBAUGH, Reliance Universal, Inc., 4730 Crittenden Dr., Louisville, Ky. 40221.

MEXICO (Fourth Thursday—meeting sites vary). MAURICIO ESQUIVEL, Pinturas Azteca.

MONTREAL (First Wednesday—Bill Wong's Restaurant). B. MCWADE, Stahl Finish (Canada) Ltd., 380 Des Lauriers St., Montreal, Que., P.Q.

NEW ENGLAND (Third Thursday—Fantasia Restaurant, Cambridge). ROBERT G. MODRAK, Benjamin Moore & Co., P.O. Box 416, Milford, Mass. 01151.

NEW YORK (Second Tuesday—Landmark II, East Rutherford, N.J.). MARVIN J. SCHNALL, Troy Chemical Corp., One Ave. L, Newark, N.J. 07105.

NORTHWESTERN (Tuesday after first Monday—Jax Cafe). ROGER ANDERSON, 3M Company, 3M Center, St. Paul, Minn. 55101.

PACIFIC NORTHWEST (Portland Section—Tuesday following second Wednesday; Seattle Section—the day after Portland; British Columbia Section—the day after Seattle). CURTIS BAILEY, Parker Paint Co., 3302 S. Junett St., Tacoma, Wash. 98409.

PHILADELPHIA (Second Thursday—Valle's Steak House). BARRY OPPENHEIM, McCloskey Varnish Co., 7600 State Rd., Philadelphia, Pa. 19136.

PIEDMONT (Third Wednesday—Howard Johnson's Coliseum, Greensboro, N.C.). WILLIAM J. CUNANE, Sherwin-Williams Co., P.O. Box 4528, High Point, N.C. 27263.

PITTSBURGH (First Monday—Skibo Hall, Carnegie-Mellon University Campus). RAYMOND C. UHLIG, Technical Coatings Co., 1085 Allegheny Ave., Oakmont, Pa. 15138.

ROCKY MOUNTAIN (Monday prior to second Wednesday). F.H. MEYERS, Kwal Paints, Inc., P.O. Box 5231 T.A., Denver, Colo 80217.

ST. LOUIS (Third Tuesday—Salad Bowl Restaurant). FLOYD THOMAS, JR., Thomas & English, Inc., 108 Mullanphy St., St. Louis, Mo. 63102.

SOUTHERN (Gulf Coast Section—Second Tuesday; Central Florida Section—Thursday after third Monday; Atlanta Section—Third Thursday). FRANKLIN D. RECTOR, Indurall Coatings, Inc., 3333 Tenth Ave., N., Birmingham, Ala. 35234.

TORONTO (Second Monday—Town and Country Restaurant). A.G. MORRIS, L.V. Lomas Chemical Co., 6365 Northwest Dr., Mississauga, Ont., Canada.

WESTERN NEW YORK (Second Tuesday—Holiday Inn, Cheektowaga, N.Y.). GEORGE C. REID, Spencer Kellogg Div., P.O. Box 210, Buffalo, N.Y. 14225.

# Society Meetings (Continued)

of adhesion by eliminating shrinkage, he said.

Dr. Lenz concluded by giving details of chemical structure and the reactions involved as well as a slide presentation of the new developments.

MARVIN J. SCHNALL, *Secretary*

### Philadelphia Dec. 14

A moment of silence was observed in memory of John C. Moore, George Woodward, and William Uzelmeier who died recently.

Among the honored guests present were Frank J. Borrelle, Federation Executive Vice-President, and Thomas Kocis, Federation Director of Communications. Mr. Borrelle accepted a check of $500 for the Paint Research Institute, presented by President Sander.

Members of the Definitions Committee were presented with tokens of appreciation in the form of personalized copies of the *Paint/Coatings Dictionary* on which they worked so hard. Stanley LeSota, Committee Chairman, also received a large wall plaque.

William J. Stewart, of Tenneco Chemicals, Inc., spoke on "HEAVY METAL ADDITIVES FOR COATINGS—A PROSPECTUS."

The heavy metals have been important in the coatings industry, in many functions, for years, and they are going to continue to be important. Phenyl mercury compounds are used as preservatives and as fungicides. Since mercury is a toxic metal, however, it is a good idea to plan for the future possibility of not being allowed to use mercurials, said Mr. Stewart.

In the oil-based paint field, the most widely used products are substituted thio phthalimide and ethyl sulfone. There are, mentioned Mr. Stewart, a number of nonmercurial fungicides for exterior latex coatings, but none have performed as well as mercurials. The problem in the first year is early chalk resistance, but they do last a long time. The organics are hydrolized at high pH and are not effective in latexes for very long when compared to the performance of mercurials, said Mr. Stewart.

In water-based finishes there is a tendency to plug in conventional drier systems which may not work well; the polymers are different, he said. The solubilizing agents (amines) cause problems with cobalt, however, they don't affect manganese. Because of calcium's water sensitivity, zirconium should be used, continued Mr. Stewart. For the drier to work, it must be soluble in the organic portion of the system. Cerium, zirconium, and the rare earths are acceptable, concluded Mr. Stewart.

BARRY OPPENHEIM, *Secretary*

### Piedmont Nov. 28

David Watson, of Rohm and Haas Co., spoke on "DISPERSION PROCESS."

Mr. Watson, aided by a slide presentation, explained in detail such areas of interest as (1) reducing pigment to its primary particle size, (2) complete wetting of exposed pigment surfaces, and (3) stabilizing the resultant dispersion.

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*Q. Is there an effective way to control foaming?*

A. Select a defoamer and a wetting agent which does not contribute to the foaming.

W.J. CUNANE, *Secretary*

### Pittsburgh Dec. 4

Jim Dulaney announced that the Society will sponsor a symposium on Corrosion Protection, on May 22, at Duquesne University.

Peter Fletcher, of PPG Industries, Inc., spoke on "NUCLEAR MAGNETIC RESONANCE."

Mr. Fletcher gave a brief history of the development of NMR and also covered the basic theory of NMR. Both proton NMR and carbon 13 NMR techniques were discussed. Mr. Fletcher showed several NMR spectra and explained chemical shifts and peak splitting. Carbon 13 was recommended for polymer analysis because of the wider range of chemical shifts which give a sharp line for each carbon, and, thus, better identification. The problem of getting accurate peak areas was discussed. Mr. Fletcher concluded by explaining more accurate techniques to get good quantitative analyses.

RAY UHLIG, *Secretary*

### Western New York Dec. 12

Roy Flynn, of Rohm and Haas Co., spoke on "ADVANCES IN ACRYLIC LATEX MAINTENANCE PAINTS."

GEORGE C. REID, *Secretary*

# Elections

## MEXICO

*Active*

Alavez H., Angel — Simon S.A., Mexico D.F., Mexico.

Cabezas, Ruben — Mobil Atlas S.A. De C.V., Mexico D.F.

Colin C., Pedro — Colin Y Lozano S R L, Mexico D.F.

Esquivel, Mauricio — Pinturas Azteca S.A., Mexico D.F.

Garcia M., Aldegunda — Pinturas Alfagama S.A., Naucalpan, Edo, Mexico.

Guevara Z., Antonio — Sinclair & Valentine, Mexico D.F.

Irazabal, Juan Pedro — Grafex De Mexico S.A., Mexico D.F.

Molina, Miguel — Prod. Quim. Y Pint. S.A., Mexico D.F.

Morales C., Aureliano — Mobil Atlas S.A. De C.V., Mexico D.F.

Ontiveros E., Luchar — Pinturas Aztlan S.A., Mexico D.F.

Perez, Mario E. — Mobil Atlas S.A. De C.V., Mexico D.F.

Rosas S., Marcelino — Pints. Cuauhtemoc S.A., Puebla, Mexico.

Sanchez, Jose De J. — Colorquim S.A. De C.V., Mexico D.F.

Santillan, Emilio R. — Ind. Tec. De Pinturas, Puente De Vigas, Edo.

*Associate*

Angon T., Enrique — Comsolmex S.A., Mexico D.F., Mexico

Del C., Javier Martin — Distribuidora Jamaca, Guadalajara, Jalisco, Mexico

Denis A., Rene — Ferroenvases Conicos, Naucalpan, Edo De Mexico

Fojo, Carlos Sierra — Synres, Mexico D.F.

Garduno C., Leonardo — Nopco Industrial S.A., Mexico D.F.

Maass B., Elizabeth — C.T.J. Maass, Mexico D.F.

Martinez Z., Jose Luis — Synres, Mexico D.F.

Montejano, J. Armando — Pigm. Prod. Quimicos, Mexico D.F.

Quiroz B., Victor M. — Pigm. Y Prod. Quimicos, Mexico D.F.

Romero, Pedro — BASF Mexicana S.A., Mexico D.F.

Torres GT Z., Jose Luis — Pigm. Y Prod. Quimicos, Mexico D.F.

Vidrio P., Praxedis — BASF Mexicana S.A., Mexico D.F.

## PACIFIC NORTHWEST

*Active*

Ritchie, Norman — Reliance Universal, Inc., Langley, B.C.

Tapper, A. William — Tonecraft Paints Ltd., Vancouver, B.C.

*Associate*

Zappitelli, Phil — Shell Canada Ltd., Vancouver, B.C.

## ROCKY MOUNTAIN

*Active*

Trousil, Edward G. — Storage Technology Co., Louisville, Colorado.

Valis, K.S. — Colorado Paint Co., Denver, Colorado.

*Associate*

Masilotti, Hugo — Packaging West, Inc., Denver, Colo.

O'Neil, Wayne G. — Diamond Shamrock Corp., Downey, Calif.

Rupprecht, H.J. — N L Industries, Titanium Pigments Div., Los Angeles, Calif.

## AFFILIATE

Fink-Jensen, Paul H. — Sadolin & Holmblad Ltd., Central Research Departments, Copenhagen, Denmark.

# Future Society Meetings

### Birmingham

(Mar. 1) — "COMMENCING PRODUCTION IN IRAN AND ALASKA" — J.R. Taylor, of B.P. Ltd.

(Apr. 5) — "WASTE DISPOSAL" — H.G. Pullen, of Redland Purle Ltd.

(May 3) — "CATHODIC ELECTRODEPOSITION—CURRENT STATUS" — E. Millington, of International Paints.

### Chicago

(Mar. 5) — "AMINE SOLUBILIZERS FOR WATER-SOLUBLE INDUSTRIAL COATINGS" — Dr. Zeno W. Wicks, Jr., of North Dakota State University; and "MARKETING - RESEARCH INTERACTIONS" — Dr. Robert Bumb, of Dow Chemical Co.

(Apr. 2) — "ACCELERATED WEATHERING" — George Grossman, of Q-Panel Co.; and "YOU CAN'T AFFORD TO BE WITHOUT SAFETY" — Gilbert Cain, of Hercules Incorporated.

### C-D-I-C

(Mar. 12) — FSCT SLIDE PRESENTATION — Federation Officers.

(May 14) — "A NEW, IMPROVED THICKENER SYSTEM" — R.J. Duncan, of Kelco Div., Merck & Co., Inc.

### Cleveland

(Mar. 13)—"FSCT SLIDE PRESENTATION"—Federation Officers; Panel Discussion on "NEW CONCEPTS IN PAINTS/COATINGS APPLICATION TECHNOLOGY."

(Apr. 12) — Meeting to be held at Ohio Edison Nuclear Power Plant, Perry, Ohio.

(May 17) — "ART OPENS WAY FOR SCIENCE" — Dr. Jon B. Eklund, of Smithsonian Institution.

### Golden Gate

(Mar. 19) — "NEW DEVELOPMENTS IN THE FIELD OF GLOSS EMULSION PAINTS" — John Bax, of Scott-Bader.

(Apr. 16) — "ACCELERATED WEATHERING AND FADING" — R. Metzinger, of Atlas Electric Devices Co.

(May 14) — "USE OF ORGANO TITANATES IN COATINGS" — Salvatore J. Monte, of Kenrich Petrochemicals, Inc.

### Los Angeles

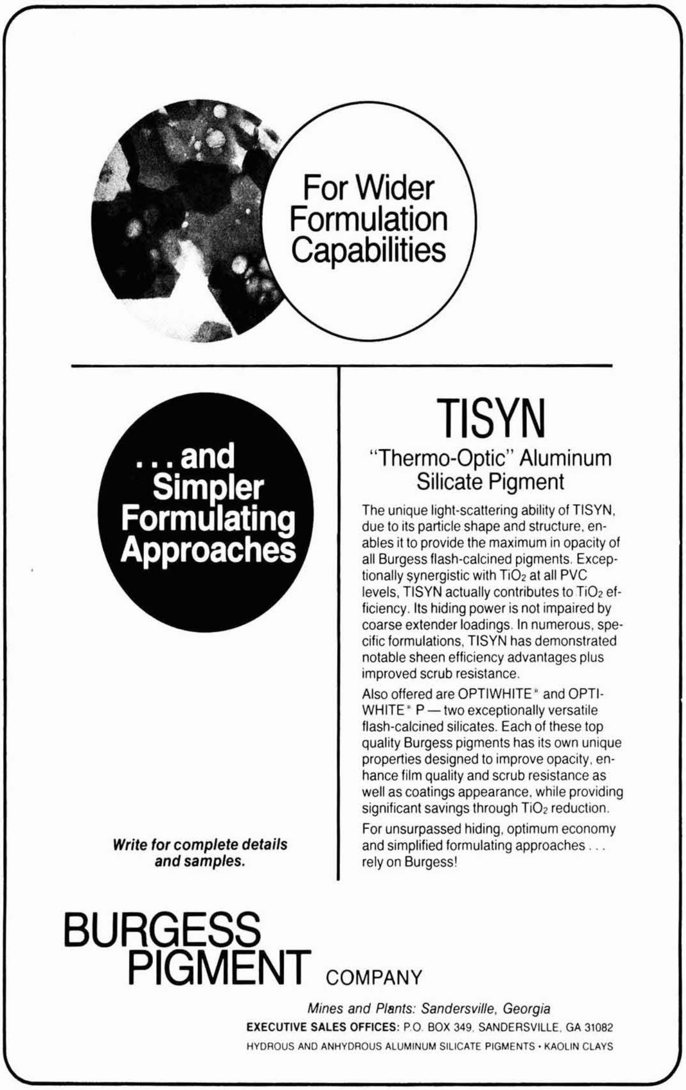
(Mar. 14) — "NEW DEVELOPMENTS IN THE FIELD OF GLOSS EMULSION PAINTS" — John Bax, of Scott-Bader.

(Apr. 11) — "ACCELERATED WEATHERING AND FADING" — R. Metzinger, of Atlas Electric Devices Co.

(May 9) — "USE OF ORGANO TITANATES IN COATINGS" — Salvatore J. Monte, of Kenrich Petrochemicals, Inc.

### Montreal

(Mar. 7) — Annual joint meeting with Quebec Paint Industry Association. "MARKETING OF CONSUMER PAINT PRODUCTS" — B. Wayne, of International Paints (Canada) Ltd.; and "THE PROFESSIONAL CHEMIST IN THE COATINGS INDUSTRY" — F. Bonnier, of Carter White Lead.



# Technical Articles in Other Publications

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

### Industrial & Engineering Chemistry Product Research & Development

Published by American Chemistry Society, 1155 - 16th Street
N.W., Washington, D.C. 20036

Vol. 17 No. 4 December 1978

Seiner, J.A. - "Microvoids as Pigments. A Review;" 302-317.

Pierce, P.E., Kovac, Z., and Higginbotham, C. - "Kinetics and Mechanism of Film Growth During the Electrodeposition Process;" 317-322.

### Journal of the Oil and Colour Chemists' Association

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

Vol. 61 No. 10 October 1978

Ghanem, N.A., Naser, A.M., Ismail, M.F., and Ghafar, M.A. - "Synthesis of Modified Phthalocyanine Compounds Suitable as Pigments;" 369-371.

Naser, A.M., Naoum, M.M., Salman, A.A., and Taha, A. - "Recent Aspects of Some Coloured Urea/Dibasic Acid Fusion Adducts;" 372-374.

Zahoor, M.A., Chandra, S., and Vaskshtha, A.K. - "Cellulose-Based Resins for Surface Coatings;" 375-382.

Rascio, V., Giudice, C.A., Benitez, J.C., and Presta, M. - "Ship's Trials of Oleoresinous Antifouling Paints. Part I: Formulations with High and Medium Toxicant Contents;" 383-389.

Vol. 61 No. 11 November 1978

Carr, W. - "The Theory and Practice of Pigment Dispersion;" 397-410.

Callaghan, B.G. - "The Corrosion and Protection of Metals in the Building and Construction Industries;" 411-418.

Vetere, V., Rozados, E., and Carbonari, R. - "Measurement of Conductivity, Capacity, Electrical Resistance and Permeability of Paint Films in an Aqueous Solution;" 419-426.

Guruswamy, S. - "Electrical Resistance of Electrodeposited Rubber Per Unit Coulomb;" 427-430.

### Deutsche Farben-Zeitschrift (Defazet)
(in German)

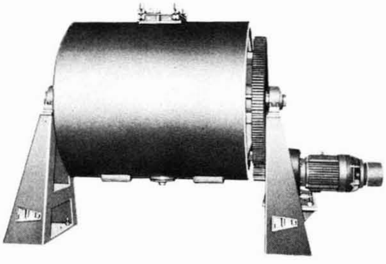
Published by Elvira Moeller GmbH, Karl-Benz-Strasse 11,
Postfach 11 68, 7024 Filderstadt 1, Germany

Vol. 32 No. 9 September 1978

Parfitt, G.D. - "Pigment Dispersion - in Theory and Practice;" 322-331.

Rechmann, H. - "Titanium Dioxide Pigments - Manufactured by the Chloride Process;" 331-338.

Gläser, F. - "Optical Behavior of Aligned Pigments;" 338-342.



## Cleveland Society to Host 22nd Conference On "Advances in Coatings Technology" in March

The Cleveland Society for Coatings Technology's Education Committee will present their 22nd technical conference, "Advances in Coatings Technology," on March 20-21 at Baldwin-Wallace College in the Cleveland suburb of Berea, Ohio.

Committee Chairman George R. Pilcher, of Sherwin-Williams Co., has announced that this conference will be divided into four, ½-day symposia: "Crosslinking Chemistry;" "Environmentally Acceptable Coatings;" "Surface Characterization;" and "Recent Technological Advances."

During the first day, the morning session, "Crosslinking Chemistry," will be chaired by F. Louis Floyd, of Glidden Div., SCM Corp.; the afternoon symposium, "Environmentally Acceptable Coatings," will be chaired by Dr. John C. Weaver, of Sherwin-Williams Co.

Two symposia will be held the second day. Chairing the morning session, "Surface Characterization," will be Dr. Theodore Provder, of Glidden Div., SCM Corp.; and Charles C. Beck, of Premier Industrial Corp., will chair the afternoon symposium, "Recent Technological Advances."

The following papers will be presented:

### MARCH 20

*Symposium on Crosslinking Chemistry*

"Reaction Mechanisms of Melamine Resins" — W.J. Blank, of American Cyanamid Co.

"Recent Trends in Aminoplast Resins" — Dr. P.J. Schirmann, of American Cyanamid Co.

"Amino Crosslinkers: Relationship of Structure to Coating Properties/Performance" — F.S. O'Connell, of Monsanto Plastics and Resins Co.

*Symposium on Environmentally Acceptable Coatings*

"High Solids are Here — And They Work — But Not for Everyone, Everywhere" — J.D. Pontius, of Sherwin-Williams Co.

"The Evolving Role of Thermoplast Emulsions in Industrial Finishing" — Dr. E.L. Leasure, of Rohm and Haas Co.

"Requirements of a Polymeric Binder for Thin Film Thermosetting Powder Coatings" — G.E. Gorrow, of Glidden Div., SCM Corp.

"UV Coatings: A Possible Answer" — H. Ragin, of Sun Chemical Corp.

Banquet Lecture — "Technical Investigation of Painting Forgeries" — R.M. Merrill, Chief Conservator, Cleveland Museum of Art.

### MARCH 21

*Symposium on Surface Characterization*

"Electron and Ion Bombardment Spectroscopy: Principles and Potential Applications in Coatings Technology" — Dr. G.D. Mateescu, of Case Western Reserve University.

"Application of Photogoniometry for the Characterization of Coatings Systems" — T.H. Grentzer, R.A. Zander, M.E. Koehler, T. Provder, and R.M. Holsworth, of Glidden Div., SCM Corp.

"Coatings Analysis and Characterization" — R.M. Holsworth, of Glidden Div., SCM Corp.

*Symposium on Recent Technological Advances*

"Surface Modification of Inorganic Pigments and Extender Pigments" — J.E. Moreland, of Malvern Minerals Co.

"Optimization of Cure Conditions During Processing of Acrylic Latex Coatings" — Dr. C. Huang and E.J. Leeson, of B.F. Goodrich Co.

"A Newly Available Aliphatic Isocyanate for Light-Stable Urethane Coatings" — Dr. G.L. Driscoll, of Sun Petroleum Products Co.; and K.C. Frisch, J.C. Kresta, and T.B. Lin, of University of Detroit.

Additional information on the conference can be obtained from the Education Committee Chairman, George R. Pilcher, The Sherwin-Williams Co., 601 Canal Rd., Cleveland, Ohio 44113.

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## "Pigments and Color" to be Examined During Chicago Society's SYMCO '79

The Educational Committees of the Chicago Society for Coatings Technology and Chicago Paint and Coatings Association will jointly sponsor the ninth annual Chicago Coatings Symposium, SYMCO '79, March 20-21, at the Fountain Blue Restaurant in Des Plaines, Ill.

According to Program Chairman Daryl Luxmore, the symposium will focus on the theme, "Pigments and Color," and will be highlighted by an exchange on extenders and vesiculated polymeric beads (*see JCT, October 1978, pp. 112-113 – Ed.*).

Scheduled for presentation are the following:

### MARCH 20

"Pigment/Polymer Grafting"—Ted Muzycko, of Elmhurst College.

"Pigments for Water-Borne Coatings"—Ted Kolski, of Du Pont Co.

"Flatting Agents"—Peter Marston, of Davidson Chemical Div., W.R. Grace Co.

"Theories and Principles of Color"—Donald Parker, of Sherwin-Williams Co.

### MARCH 21

"Extenders as $TiO_2$ Spacers"—Fred B. Stieg, of Pigmentech Consulting.

"Vesiculated Polymeric Beads in Flat Latex Paints"—Robert Johnson, of Dulux Australia Ltd.

"Extender Pigments"—William Grimm, of Plews Stouffer.

"Psychological Effects of Color"—Joyce Davenport, of DeSoto, Inc.

"Color Mapping"—John McMillan, of ICI United States.

For complete information, or to register, contact Program Chairman Daryl Luxmore, 9 S. 684 Highland Ave., Hinsdale, Ill. 60521.

## 1979 PRI Symposium to Focus On Analytical Methods For Regulated Materials

A Symposium on Analytical Methods Used to Monitor Product Compliance with Regulations has been scheduled for May 1 and 2 at Battelle Columbus Laboratories, Columbus, Ohio, by the Paint Research Institute.

The symposium will inform suppliers, formulators, and users of methods available for the analysis of materials that are regulated or are likely to be regulated, will assess the validity of mandated limits and sampling practices, and will enable the coatings chemist to select the most appropriate method for a given compliance problem, with consideration of the acceptability of the method to a referee.

The tentative program schedules four sessions dealing with Metal Analysis; Volatile Organic Components (VOC); Flammability; and Trace Organic Materials.

Program Committee Chairman Raymond R. Myers, PRI Research Director and Chairman of the Chemistry Dept. of Kent State University, will be assisted by William Golton, of du Pont Marshall Laboratories; Richard Holsworth, of Glidden Div., SCM Corp.; Percy Pierce, of PPG Industries; John Vandeberg, of DeSoto, Inc.; and John Weaver, Consultant.

Members of the Host Committee include: Chairman William Mirick, of Battelle Columbus Laboratories; Herbert Fenburr, Consultant; and Carl J. Knauss, of Kent State University.

## Federation to Host Society Technical Committee Chairmen At March 30 Meeting in Atlanta

Technical Committee Chairmen of the Constituent Societies of the Federation will be hosted by the Federation at a meeting to be held in Atlanta, Ga., on March 30.

The full-day conference will be devoted to reports on projects currently underway, as well as discussions on suggested undertakings for Society Technical Committees.

The meeting has been scheduled as an outgrowth of recent discussions by the Federation's Technical Advisory Committee on development of projects that would generate practical work on topics of current concern to the industry.

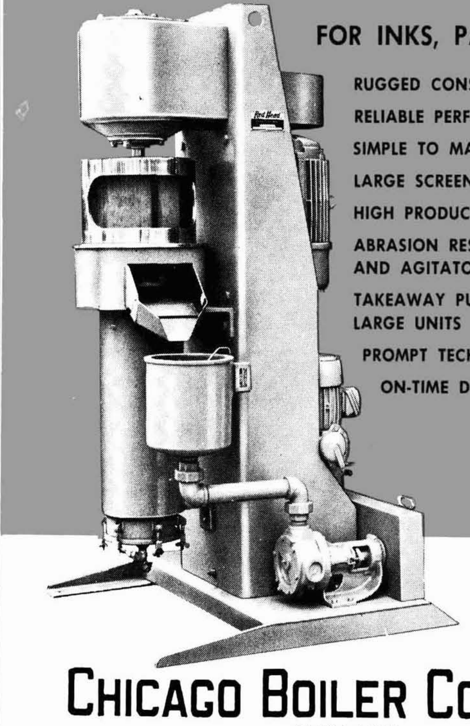
## Philadelphia Society Sponsoring Courses On Color Matching and Coatings Technology

The Joint Educational Committee of the Philadelphia Society for Coatings Technology and the Philadelphia Paint and Coatings Association has announced sponsorship of two evening courses for coatings personnel.

The first is a repeat of the ACS Course in Coatings Technology offered in 1977, which is for persons with a college chemistry background. Dr. Seymore Hochberg, of E.I. du Pont de Nemours & Co., Inc., will again be the instructor in a series of eight two-hr sessions which will be presented prior to the regular monthly Society and the monthly Technical Committee meeting. The course will offer a thorough discussion of paint formulation, evaluation, and raw materials usage. Fee, including texts, is $150.

Meanwhile, a course on Color Matching — Theory and Applications will be presented in a series of five two-hr sessions by P.J. Sotorrio, of DeSoto, Inc. An updating of color matching practices will be presented. A comprehensive evaluation of old vs. new will be discussed, along with computer color matching. Fee is $50.

The Philadelphia Society is also sponsoring two presentations at Temple University's School of Pharmacy. One is on lab procedure useful in health and safety hygiene, and the other is a health and safety seminar. Both are free.



# 1980 FATIPEC Congress to Focus on '3-E' Activities of Coatings Industry

The XVth Congress of FATIPEC (Federation of the Associations of Technicians in the Paint, Varnish, Lacquer and Printing Ink Industries of Continental Europe) will be held from June 8-13, 1980, at the Rai Congress Centre in Amsterdam, Netherlands.

Dr. M.F. Kooistra, of N.V.V.T. and the President of FATIPEC, announced that the Congress theme will be "Activities of the Coatings Industry in the Framework of Ecology, Energy, and Economy Problems."

Society's developments and their resulting consequences in controlling and regulating environmental pollution, in saving energy and resources, and in reducing labor and production costs, will continue to have a heavy impact on coatings industry's technology, and on its relevant research and development for the years to come.

It is envisaged to have presented at the Congress an actual picture of the state of the art, composed of the contributions which hopefully will be offered from different and international sectors of the coatings industry.

### Call for Papers

Discussion papers for the FATIPEC-Congress are invited.

It is emphasized that submitted papers should deal effectively with the theme and that the Technical-Scientific Committee, as part of the Dutch organizing structure, has been instructed to apply their acceptance criteria for Congress contributions accordingly.

The discussion papers will be divided over section meetings. It is the intention of the Technical-Scientific Committee to group appropriate discussion papers, so as to be able to organize more effective discussion sections or panels.

As usual, registration of papers from speakers from FATIPEC member countries should be made to the respective national FATIPEC association. Speakers from other countries are requested to register their paper directly to the Congress Secretariat: C. Korf, Oostenrijklaan 43, Haarlem, Netherlands.

Authors must send their names and titles of papers to Mr. Korf by June 1; the abstract by September 1; and the full text of paper by January 1, 1980. Both plenary lectures and discussion papers will be presented in one of the three official FATIPEC languages: English, French, or German.

### Exhibition

Running concurrently with the program sessions will be the FATIPEC exhibition in the Rai exhibition halls.

Dr. Kooistra (Kerkhoflaan 8, 6718 ZJ Ede Gld., Netherlands) said that Congress Circulars #1and #2 will be issued later this year. Copies will be available upon request.

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## *Society Educational Programs* *(Continued)*

### Southern

Co-sponsors with University of Southern Mississippi annual symposium on water-borne and higher solids coatings . . . Continuing support of scholarship grants at USM . . . USM students invited to present papers at Society Annual Meeting.

### Toronto

Coatings courses at George Brown College continuing under Society sponsorship — currently offering courses on basic and advanced coatings technology, as well as one for technicians; also presented one-day program in January on color technology and instrumentation . . . Working on production of slide-tape presentation on "Training of a Resin House Operator."

### Western New York

In attempt to improve liaison with area colleges to make them aware of coatings industry needs and activities, polymer instructors from these institutions have been invited to attend regular Society monthly meetings; students will be invited to tour industry facilities, and Society members solicited to present lectures on careers in coatings at local schools . . . Efforts being pursued with Erie Community College to establish polymers course . . . Working on production of slide-tape presentation on "Impact Resistance."

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## PHILADELPHIA SOCIETY MEMBERS CITED FOR DICTIONARY WORK

**Members of the Philadelphia Society who worked on the Paint/Coatings Dictionary were cited recently by the Federation for their outstanding contribution. Shown left to right are: Dave Engler, of Finnaren and Haley, Inc.; J. Richard Kiefer, Jr., of McCloskey Varnish Co.; Lothar Sander, of Amchem Products Co.; Bruce Schirm, of Thibaut & Walker, Inc.; Chairman Stan LeSota, of Rohm and Haas Co.; Michael McGroarty, of DeSoto, Inc.; and Francis Willard, retired from Finnaren and Haley, Inc. Not shown, but also cited for their efforts on the dictionary, were Seymour Mark, of New Jersey Zinc Co. and John Landis, of Reichard-Coulston, Inc., along with Ruth Johnston-Feller, Consultant, of the Pittsburgh Society.**

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## Larry L. Thomas, of NPCA, To be Keynote Speaker At WCS Symposium, March 1

Larry L. Thomas, Executive Director of the National Paint and Coatings Association, will be featured as Keynote Speaker at the 14th Biennial Western Coatings Societies Symposium and Show to be held at the Fairmont Hotel, San Francisco, Calif., March 1-3. He will speak during the luncheon on March 1.

The previously announced keynote speaker, Congressman Leo J. Ryan (D-Calif.), was slain last December in Jonestown, Guyana.

Mr. Thomas, who recently succeeded Robert Roland as Executive Director of NPCA, joined the Association's Legal and Government Relations Div. in 1971. Prior to his promotion he served as NPCA General Counsel.

## Decorating Products Show Attracts Industry Leaders

**Shown are a number of industry leaders who were on hand for the 31st Annual National Decorating Products Show in St. Louis. From left to right (seated) are Raymond D. Stevens, of Pratt & Lambert, Inc., Immediate Past Chairman of the National Paint and Coatings Association; James A. McCormick, of Inland-Leidy Corp., President of the Federation of Societies for Coatings Technology; William Fuller, President of the National Decorating Products Association; Roger Plauche, President of the Wallcoverings Manufacturers Association; and Joe Gallagher, Jr., President of the Wallcoverings Wholesalers Association. Standing (left to right) are Larry Thomas, Executive Director of NPCA; Dollard Carey, Chief Operating Officer of the WWA; Glen Craven, Executive Vice-President of the Painting and Decorating Contractors of America; Frank Borrelle, FSCT Executive Vice-President; and Robert Petit, NDPA Executive Vice-President**

## OCCA to Host Biennial Conference June 20-23

The Oil and Colour Chemists' Association will hold its Biennial Conference at the Stratford Hilton Hotel, Stratford-upon-Avon, June 20-23, 1979.

Papers reflecting the theme of the conference, "The Challenge to Coatings in a Changing World," will be presented at four technical sessions by lecturers from the United Kingdom, Denmark, Germany, and the United States. The following papers are scheduled:

"The Challenge to Coatings in a Changing World — Predestination or Prophecy?" — J.B. Harrison, Consultant.

"Surface Coatings for the Nuclear Industry" — P. Walker and A. Haighton, of the Ministry of Defence.

"Original and Maintenance Painting Systems for North Sea Oil and Gas Platforms" — D. Atherton, of Sigma Coatings Ltd.

"Solvent Loss from Paint Films" — J. Ramsbotham, of Shell Research BV, Netherlands.

"Water-Borne Industrial Thermosetting Systems" — H.J. Luthardt and W. Burckhardt, of Deutsche Azko GmbH.

"Influence of Light Intensity on Photoactivity in $TiO_2$ Pigmented Systems" — T.A. Egerton and C.J. King, of Tioxide International Ltd.

"Engineering for Corrosion Protection" — Speaker to be named. (Presented on behalf of the Federation of Societies for Coatings Technology.)

"The Technologist in Chains" — A.R.H. Tawn, of Coates Group of Companies.

"The Changing Role of Polymer Chemistry in Organic Coatings Technology" — M.F. Kooistra, of Azko Research, Netherlands. (Presented on behalf of FATIPEC.)

"Characterisation of the Interactions between Pigments and Binder Solutions Using Rheological Measurements" — A. Saarnak, of the Scandinavian Paint and Printing Ink Research Institute. (Presented on behalf of the SLF.)

"Aqueous Coil Coatings" — E.J. Percy and F. Nouwens, of Synres International BV, Netherlands.

"Aluminum Compounds as Major Components of Surface Coatings" — J.H.W. Turner, Consultant, and W.K.H. Lakin, of Manchem Ltd.

"Dispersion of Pigments: What is the Ultimate?" — D.M. Varley and H.H. Bower, of CIBA-GEIGY Plastics & Additives Co.

"Dry on Wet — A New Concept in Low Pollution Coatings Utilising Cellulose Acetate Butyrate" — B.P. Windibank and R.W. Mahar, of Kodak Ltd.

Registration fees for the conference are £60 for members; £80 for nonmembers; and £20 for wives. A daily registration fee of £35 has been set for members only.

For additional information, contact the Director and Secretary of OCCA, Priory House, 967 Harrow Rd., Wembley, Middlesex, England HA0 2SF.

## NACE to Feature 40 Symposia At CORROSION/79 in Atlanta

The National Association of Corrosion Engineers will present 260 technical papers at 40 symposia during its annual conference, CORROSION/79, in Atlanta, Ga., March 12-16. Headquarters for the event will be the Hilton Hotel.

On Wednesday, March 14, Dr. Mars G. Fontana, Executive Director of the Materials Technology Institute of Chemical Processes Industries, Inc., will deliver the Plenary Lecture, "Corrosion Science, Corrosion Engineering, and Then What?", which will discuss the importance and value of synergistic relationships between the corrosion scientist and corrosion engineer.

Dr. Fontana, early in his career, worked for the Du Pont Co., where he pioneered industrial uses of nylon and Teflon. Since 1945, he has been a consultant to both industry and government. He has been an active member of NACE for over 32 years, and served as its President in 1952. He is also a member of the National Academy of Engineering, an Honorary Member of the American Society for Metals, and is a Fellow in the Metallurgical Society of AIME and the American Institute of Chemical Engineers.

In addition to the technical symposia, 35 research-in-progress reports will be given during the '79 Corrosion Research Conference. The '79 Corrosion Show, to be held in conjunction with the Conference, will feature over 150 exhibits of products and services used in controlling corrosion.

Conference registration information, hotel reservation forms, and a complete listing of presentations are available from NACE, P.O. Box 986, Katy, Tex. 77450.

## Southern Society to Hold Annual Meeting, March 14-16

The 43rd Annual Meeting of the Southern Society will be held March 14-17 at the Dutch Inn Resort Hotel, Lake Buena Vista, Fla.

Featured presentations on the program will focus on technical, manufacturing, and economic subjects.

Two changes from the traditional format have been announced. The Business Meeting will be held at the concluding session instead of at the first. Also, the banquet will be replaced this year with a reception.

Complete information on the program and registration is available from Al Hendry, A. L. Hendry & Co., P. O. Box 17099, Tampa, Fla. 33612.

# People

**Richard L. Vaughan** has been appointed Western Regional Manager, Resins and Adhesives, for the Industrial Chemicals Div., of the American Cyanamid Co. He will be located in Chicago. Mr. Vaughan is a member of the Philadelphia Society for Coatings Technology.

**Fredric M. Guist** has been promoted to Director of New Business Development for Engelhard Minerals & Chemicals Corp., Minerals & Chemicals Div. In his new position, he will take charge of the selection, evaluation, and implementation of new business opportunities developed by the division outside its normal lines of business.

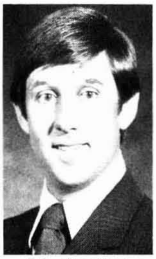
The Enterprise Companies, an Insilco Co., has named **James Moe** to the position of Vice-President/National Sales Manager of the BPS Paint Div., and **James M. Thompson** to the position of National Accounts Manager of the Magicolor Paint Div.

**Neil C. Hawkins** has been promoted to Plant Manager of the San Leandro, Calif., plant of Standard T Chemical Co., Inc. Mr. Hawkins has been with the company for over 17 years, and served most recently as Laboratory Manager. He is a member of the Golden Gate Society for Coatings Technology.

**Motowo Takayanagi** was presented with the Society of Plastics Engineers' Distinguished Educator Award at a special ceremony in Tokyo during the Japanese Plastics and Rubber Fair.

Dr. Takayanagi is Professor of Applied Chemistry, Faculty of Engineering, at the University of Fukuoka, Japan. Among his many original scientific contributions are the invention of the Rheovibron, a widely used powerful tool for investigating structure-property relationships in polymers, and the development of the Takayanagi model to describe the visco-elastic behavior of two-phase polymeric systems.

In a series of promotions, J.T. Baker Chemical Co. has named **Thomas P. Merrick** to the position of Manager of Marketing and New Products Development, **John J. Elmo** to the posotion of Product Manager—Laboratory Products, and **Judith A. Decker** to the position of Assistant Product Manager—Laboratory Products. Ms. Decker is a member of the American Chemical Society.



R.L. Vaughan

F.M. Guist

A. Starkey

R.R. Missar

**Allen Starkey**, a Technical Representative for the Polymers, Resins, and Monomers business team of the Rohm and Haas Co., has been named one of the ten winners in *Purchasing* magazine's 1978 Top Salesmen Contest. Mr. Starkey is a member of the Northwestern Society for Coatings Technology.

**Dr. Kamil Klier,** Professor of Chemistry at Lehigh University, has been named Associate Director—Surfaces in the Center for Surface and Coatings Research at the university. **Dr. John W. Vanderhoff** continues as Associate Director—Coatings. Simultaneous with Dr. Klier's appointment, the reorganization of the Center into the following laboratories was announced: Color Laboratory, directed by **Dr. Eugene M. Allen;** Catalysis Laboratory, directed by **Dr. Klier;** Corrosion Laboratory, directed by **Dr. Henry Leidheiser, Jr.;** Environmentally Sensitive Fracture Laboratory, directed by **Dr. Robert P. Wei;** Surface Analysis Laboratory, directed by **Dr. Gary W. Simmons;** and Colloid Laboratory, directed by **Dr. Fortunato J. Micale.**

**Robert J. Mitchell** has been promoted to Business Manager of the Pigments Div., of the Degussa Corp., Teterboro, N.J. He served previously as National Sales Manager.

**M. Joyce Specht** has joined the Porter Coatings Div., Porter Paint Co., as Manager of the Testing, Analytical and Compliance Laboratory. Miss Specht is a member of the Louisville Society for Coatings Technology, NACE, ASTM, ACS, Society of Instrumental Analysis, Coblentz Society, and the Society of Applied Spectroscopy.

**Arthur C. Wrotnowski** has been appointed Vice-President of Engineering and Development for Filter Specialists, Inc., Michigan City, Ind.

**Richard R. Missar** has been elected President and Chief Executive Officer of DeSoto, Inc., Des Plaines, Ill. He had been Executive Vice-President and Chief Operating Officer. **George A. Nichols,** currently the company's Chairman of the Board and Chief Executive Officer, will relinquish the office of Chief Executive, but will remain as Chairman until his retirement on May 31, 1979.

Air Products and Chemicals, Inc. has appointed **Clifford A. Bridges** Marketing Manager for its Plastics Dept. He will be responsible for establishing a marketing data base and developing business plans and strategies for the polyvinyl chloride business area.

Three key appointments in the Chemistry Dept., of Battelle's Columbus Laboratories, have been announced. They are: **George E. Cremeans,** Manager of the Organic and Polymer Chemistry Section; **Joseph A. Wray,** Manager of the Polymer Science and Technology Section; and **Michael M. Epstein,** Program Manager for Service Life of Polymeric Materials Research.

Meanwhile, **Dr. Howard C. Woodruff** has joined Battelle's Houston Operations as a Research Leader. He will coordinate and assist in research and project marketing directed toward coatings and polymer applications for the petroleum, gas, and allied industries. Dr. Woodruff is a member of the Houston Society for Coatings Technology.

**George W. Ingle** has been named to the new position of Director of Association Liaison for the Manufacturing Chemists Association. He will continue as Assistant Technical Director for Toxic Substances Control and Secretary to the Chemical Regulations Advisory Committee.

## John C. Moore, Federation Past-President, Dies at 79

**J.C. Moore**

John C. Moore, Coatings Consultant, Past-President of the Federation of Societies for Coatings Technology, and former Technical Director of the National Paint and Coatings Association, died in Southampton, Pa. on December 10, 1978 following a brief illness. He was 79-years-old and still active as a chemical engineering consultant and legal witness.

Mr. Moore, President of the Federation in 1946-47, also served as President of the Philadelphia Society and was an Honorary Member of both the Baltimore Society and the Federation.

In addition to his Federation involvement, Mr. Moore served as Technical Director of the NPVLA, now the National Paint & Coatings Association, from 1947 until 1956, when he organized his own firm, Moore Research Laboratories, Inc., at Bethesda, Md., which operated as a research and investigatory arm of local, state, and Federal government for 20 years.

Mr. Moore served on Committee D-1 of the American Society for Testing and Materials from 1934 until his death, and was Chairman of that committee from 1963 to 1972. From 1955 to 1958, he was an Officer and National Director of ASTM. He was elected an ASTM Fellow and received the Society's Award of Merit in 1970.

Born in Matador, Tex, Mr. Moore was graduated from Texas A&M University in 1922 with a B.A. Degree in Chemical Engineering. He served as a Chemical Engineer with the Texas Co. from 1922 to 1927 and was Assistant Superintendent with the Texas Pacific Coal & Oil Co. from 1927 to 1930. In 1930, he joined Sinclair Refining Co. as a Chemical Engineer and was Superintendent of the firm's paint plant from 1937 to 1947.

In addition to ASTM, Mr. Moore was a member of the American Chemical Society and the American Institute of Chemists. He was active in the Boy Scouts of America from 1935 to 1950, on the local and regional level. From 1935 to 1938, he was Scoutmaster of Troop 2, Swarthmore, Pa., sponsored by the Swarthmore Presbyterian Church in which he was an elder. He was appointed Commissioner to Valley Forge District of BSA and participated in rewriting the Scout Manual. From 1946 to 1948, he joined in the development of a series of learning aids, along with three completely new manuals, for Cub Scouts of America. For these and similar activities, he received, in 1948, the Silver Beaver, the highest award of the Boy Scouts of America.

Mr. Moore is survived by two sons, Charles, 51, and William, 49; three younger sisters, 10 grandchildren, and one great-grandchild.

Services and burial were at Warminster, Pa.

Beatrice Foods Co., Wilmington, Mass., has announced the election of **Harry C. Wechsler,** President of the Chemical Div., to the position of Corporate Vice-President. He will continue to supervise operations of all domestic and international units of the Chemical Div.

**Dr. Ronald K. Eby,** Chief of the Polymer Science and Standards Div., National Bureau of Standards, Gaithersburg, Md., was elected Chairman of Committee D-20 on Plastics of the American Society for Testing and Materials.

Union Oil Co. of California, Union Chemicals Div., Petrochemical Group, has named **Nicholas E. Lynam** Manager, Manufacturing. He will direct the activities of the division's six polymer and adhesive plants. Mr. Lynam will be located in Schaumburg, Ill.

Spencer Kellogg Div. of Textron, Inc. has acquired the coating resins business of Ashland Oil, Inc., effective December 11, 1978 and has appointed **G. William Harrison,** Div. President. Under the terms of the agreement, Spencer Kellogg assumes control of three manufacturing facilities in Newark, N.J.; Valley Park, Mo.; and Pensacola, Fla. These facilities will complement the company's other plants in Baltimore, Md.; Bellevue, Ohio; Buffalo, N.Y.; Edgewater, N.J.; Minneapolis, Minn.; and San Carlos, Calif. In addition, about 50 administrative and technical personnel will be hired or transferred to the company's headquarters and research facility in the Buffalo area.

Witco Chemical Corp. has named **John T. Foley** Vice-President/Director of Marketing and General Sales Manager of the Organics Div. He was formerly Vice-President for Surfactant Product Management in Houston, Tex. Mr. Foley will be relocating to division headquarters in New York City.

Meanwhile, **Alfred P. Marone** has been elected a Corporate Vice-President and General Manager of the division. He had been Assistant General Manager and Vice-President—Director of Marketing for the past five years.

## Obituary

**George P. Woodward,** 75, a Past-President and Honorary Member of the Philadelphia Society, and long-time Paint industry worker, died December 10, 1978, in Roslyn, Pa. Mr. Woodward, during his career, worked for McCloskey Varnish Co., Archer-Daniels-Midland Co., and Ashland Chemical Co. He retired in 1966. Mr. Woodward also served on numerous local and national paint committees. He is survived by his wife, two children, and four grandchildren.

# Literature

## Industry Trend Study

Non-residential coatings are entering a new era of change and opportunity according to a just-completed 200 page study by the Ozimek Data Corporation (ODC), Rye, New York, entitled *Maintenance and New Construction Coatings*.

Currently the market for non-residential coatings is pegged at about the $670 million level and is forecast to grow about $180 million in five years time. The profit opportunities for individual companies—raw material and paint manufacturers—are better than the sales figures suggest. The products serving the market are undergoing wide-scale reformulation.

The pressure for reformulation comes from the U.S. Government. The Government is concerned with four problem areas: air pollution, water pollution, safety and health, toxic substances.

Although the decline in solvent usage is certain, the ODC study points out, there are several unanswered questions: Will the switch be to coatings using water or to coatings using little or no solvent? Water as a coatings vehicle presents technical problems. It is difficult, to say the least, to produce coatings with water as the vehicle that can hold up under heavy-duty conditions.

The ODC study underscores the major trends, concepts to be aware of, for the engineering/design construction firms, the end-users of coatings, as well as the raw material suppliers and coatings producers.

*Maintenance and New Construction Coatings* can be ordered from the Ozimek Data Corp., 92 Mendota Ave., Rye, N.Y. 10580. Price $800 + New York State tax where applicable.

## Code Provisions for Urethane Foam

A 12-page bulletin has been published which provides a summary of various building codes, tests to attain code compliances, fire testing methods, toxicity factors, and a detailed compendium of source materials. The brochure notes factors that make urethane foam an efficient insulating material and discusses possible fire hazards if it is not given a protective coating. To obtain a copy of SPI Bulletin U-104, contact Isocyanate Products Div., Dept. 1574-A, Witco Chemical Corp., 900 Wilmington Rd., New Castle, Del. 19720.

## New French Journal, "Couleurs," Makes Debut

The first issue of a new version of "Couleurs," French journal dealing with color, has been published by Etude et Realisations de la Couleur, 68 Rue Jean-Jaures, 92800 Puteaux, France.

P. Chassaigne, Vice-President of EREC, stated that the editorial staff includes scientists, professors, architects, and others who deal with the fine arts. "Couleurs" will be published four times a year, at irregular intervals.

## Paint Specifications

The 20th edition of NPCA's "Guide to U.S. Government Paint Specifications" is now available. This edition, which supersedes all previously issued sets, can be obtained by members for $12.50 per copy, prepaid, and by non-members for $25.00 per copy, prepaid. Prices include periodical supplements for a period of 4-5 years. To order, contact National Paint & Coatings Association, 1500 Rhode Island Ave., N.W. Washington, D.C. 20005.

## Epoxy Resins

Availability of development quantities of new weatherable epoxy resins is announced in a recent brochure. Available in three grades, the resins feature an unusually low viscosity of 20 to 25 poise at 25°C. For additional information on Eponex resins, write Shell Chemical Co., Manager, Chemical Communications, One Shell Plaza, Houston, Tex. 77002.

## Polyurethane Coatings

A new eight-page bulletin describing a polyurethane coating system which offers resistance to chipping, abrasion, chemicals, and corrosion is now available. The bulletin includes a full technical description of the product along with information on recommended uses, surface preparation, application, clean-up, storage, and mixing. In addition, the brochure contains illustrations showing corrosion, impact, and abrasion resistance, and gloss retention as compared to other polyurethanes, as well as epoxy and alkyd coating systems. Color chip samples of nine standard and more than 22 custom coating colors are also included. For additional information, contact Rust-Oleum Corp., 11 Hawthorn Parkway, Vernon Hills, Ill. 60061.

## Diisocyanate Neutralizer

A granular neutralizer said to inhibit evaporation and control toxicity problems stemming from urethane spills is described in literature now available. This product is designed for urethane processors who use diisocyanate, either MDI or TDI, as a raw material component. For further information, write Pigment Dispersions Inc., 54 Kellogg Court, P.O. Box 412, Edison, N.J. 08817.

## Pollution Control

Publication of a 444-page guidebook, "Handbook of Pollution Control Management," edited by H.F. Lund, has been announced. Among the topics covered are preparation of environmental impact statements, forecasting costs, equipment maintenance and performance guarantees, re-cycling waste products (and other cost recovery opportunities), government financial assistance, and the assignment of pollution control responsibilities. The book is available at a cost of $39.95 from Prentice-Hall, Inc., Englewood Cliffs, N.J. 07632.

## Crosslinking Agents

Technical bulletins which provide information on two thiochemicals identified as pentaerythritol tetrakis (3-mercaptopropionate) and trimethylolpropane tris (3-mercaptopropionate) are now available. The thiochemicals are suggested for use as crosslinkers in the photopolymerization of unsaturated carbon-carbon double bonds, carboxyls, halides, epoxides and other chemicals, and as stabilizers for radiation-cured plastics. Copies of the bulletins on Halby mercaptopropionate esters may be obtained from Argus Chemical Corporation, Dept. 1544, 633 Court St., Brooklyn, N.Y. 11231.

## Slip Additive

A unique product has been developed which improves the resistance of enamel coatings to metal marking, marring, and blocking without impairing gloss. Particularly effective in coil coatings where it minimizes paint defects which can occur during post-forming operations, Slip-Ayd® SL-133 is a dispersion of a polymerized wax in melamine and a high KB naphtha. For further information, or samples, write Daniel Products Co., 400 Claremont Ave., Jersey City, N.J. 07304.

# Literature (Continued)

### Ultrasonic Flowmeter

A four-page data sheet describes a new ultrasonic flowmeter utilizing the Doppler principle, and operating on 24 V d-c or 120 V a-c, for non-intrusive, obstructionless measurement of liquid flow in any size pipe. To obtain a copy of Data Sheet C4.3111-DS, contact Leeds & Northrup Co., MD210, North Wales, Pa. 19454.

### Urethane

A brochure about a new alkyd-modified urethane describes a product designed for interior uses such as floor finishes, wood and paneling finishes, aerosol coatings, and household finishes. To learn more about Spenkel F32-50MS, write Spencer Kellogg, Div. of Textron, Inc., Box 807, Buffalo, N.Y. 14240.

### ASTM Standards

New and revised standards are combined in the 1978 *Annual Book of ASTM Standards* Parts 27-30, 41, and 42. Parts 27, 28, and 29 contain over 300 standards test methods, specifications, definitions, practices, and classifications for paint and related products. There are eight new standards in Part 30, three in Part 41, and seven in Part 42. For further information contact M. Holst, Sales Service Dept., 1916 Race St., Phila., Pa. 19103.

### Product Finishes Study

An economic study of U.S. markets for product finishes has been done, and is divided into 12 sections including Passenger Car Finishes, Marine, Factory Finished Wood, and Metal Decorating. It also covers such areas a Electrocoating, Raw Materials, and Marketing Product Finishes. The markets are analyzed by types of coatings and vehicles used, coating volume, geographic breakdown of demand, application methods, and a 1982 market forecast. The study is available at the post-publication price of $7,500. For further information, contact Fred Smith, Smith, Stanley & Co., Inc., 72 Old Kings Highway North, Darien, Conn. 06820.

### Chemical Intermediate

Data sheets are now available which describe a large variety of end uses for this hydroxyl functional liquid which consists of various glycerols in an aqueous solution. Copies of the Polyglycerine W-80 data sheets may be obtained by contacting Dow Chemical U.S.A., Plastic Dept., 2040 Dow Center, Midland, Mich. 48640.

### Thermogravimetric System

An eight-page color brochure describing the TGS-2 thermogravimetric system is now available. A description of the instrument is given, and basic principles of operation are discussed. There are three pages of performance data and nine examples of sample runs showing: thermal separation characterizations, oxidation analysis, moisture analysis, thermomagnetometry, and simultaneous analysis. To obtain a copy of brochure L-508, write Perkin-Elmer Corp., Instrument Div., Main Ave., Mail Station 12, Norwalk, Conn. 06856.

### Vinyl Emulsions

Two new brochures describing wet adhesion vinyl emulsions are available. The first emulsion is a hard polymer designed for use in premium interior semi-gloss and eggshell enamels. The second emulsion is a more flexible polymer designed for use as a multipurpose vehicle in both exterior and interior paints and enamels. The brochures detail typical properties, as well as recommended uses, suggested formulations, availability, and technical support services. To obtain copies of the WAVE 345 and WAVE 375 brochures, contact Air Products and Chemicals, Inc., Polymer Chemicals Dept., Box 538, Allentown, Pa. 18105.

### Signal Processor

A new automatic signal processor which features four internationally used color scales and memory storage for up to ten product standards is described in recently issued literature. For more information on the D25-9, write Hunter Associates Laboratory, Inc., 9529 Lee Highway, Fairfax, Va. 22031.

### Color Analyzer

A spectrophotometric sensor that can help users analyze color samples or determine the thickness of transparent thin films is the subject of a new technical bulletin. Advanced techniques and features of the IBM 7409 Scanning Color Sensor allow for the rapid handling of measurement applications and reproducible results under a wide range of environmental conditions. For more information write IBM Instrument Systems, 1000 Westchester Ave., White Plains, N.Y. 10604.

### Industrial Waste Treatment

A new, full-color brochure describes the regional disposal of difficult industrial wastes in accordance with governmental regulations. The advantages of utilizing an outside waste management contractor, the concept of regional facilities, and the procedures involved in collecting, analyzing, and properly disposing of such wastes are also discussed. Copies of the booklet are available from Rollins Environmental Services, Inc., One Rollins Plaza, Wilmington, Del. 19899.

### Viscosity Determination

A six-page brochure discussing a system for high temperature viscosity determination is now available. The two-color pamphlet describes how the system solves the difficult problem of hot melt and liquid viscosity measurements by providing a stable, precisely controlled sample environment. To obtain a copy, write Dept. NR 28, Brookfield Engineering Laboratories, Inc., 240 Cushing St., Stoughton, Mass. 02072.

### Surfactants

Two new surfactants for powder formulators are discussed in a newly-published bulletin. The products, Surfynol® 104S and 82S, are reported to offer good powder wetting, hard water stability, compatibility with a wide variety of ingredients, and anti-foaming properties. Both surfactants can also be used as post-additives to reduce static charge build-up after powder grinding. For further information contact Air Products and Chemicals, Inc., Acetylenic Chemicals Div., Box 538, Allentown, Pa. 18105.

## Book Review

### HANDBOOK OF ELECTROPAINTING TECHNOLOGY

Written by
Willibald Machu
Translated by
Dr. Peter Neufeld
Electrochemical
Publications Ltd.
Ayr, Scotland
$67.00

Reviewed by
George E.F. Brewer
Coating Consultant
Brighton, Mich.

This book appeared in the German language, in 1974, under the title "Electrotauchlackierung," Verlag Chemie GmbH. The present, somewhat enlarged, English Edition is well translated and well printed.

Prof. Machu anticipates many questions the reader may pose, which makes the book valuable. The text is subdivided into 25 chapters, making a total of 307 pages, illustrated with 133 figures, 34 tables, and over 900 references, which provide access to a substantial section of essentially European literature. At the time of writing, cathodic electrodeposition had ". . . not yet gone beyond the trial stage," as Prof. Machu states on page 136.

The first six chapters (75 pages) deal with history, anodic processes, volt/ampere relation, throwing power, etc. The next 45 pages are devoted to metal preparation. A 122-page section elaborates on resin composition, design of installations, bath stability, ultrafiltration, power supplies, and cost studies.

The author has surveyed the patent situation and lists 176 Process Patents, 43 Equipment Patents, and 254 Composition Patents.

Prof. Machu's book contains much material of interest to Coatings Technologists.

# Coming Events

## FEDERATION MEETINGS

(May 17-19)—Federation Spring Meetings. Third-ranking Society Officers—17th; Board of Directors—18th; Executive Committee—19th. Hilton Hotel, New Orleans, La. (FSCT, Suite 832, 1315 Walnut St., Philadelphia, Pa. 19107).

(Oct. 3-5)—57th Annual Meeting and 44th Paint Industries' Show. St. Louis Convention Center, St. Louis, Mo. (FSCT, Suite 830, 1315 Walnut St., Philadelphia, Pa. 19107).

## PAINT RESEARCH INSTITUTE MEETING

(May 1-2)—Paint Research Institute Symposium on Analytical Methods Used to Monitor Product Compliance With Regulations. Battelle Institute, Columbus, Ohio. (Dr. Raymond R. Myers, Chemistry Dept., Kent State University, Kent, Ohio 44242).

## SPECIAL SOCIETY MEETINGS

(Feb. 28-Mar. 2)—Western Coatings Societies' Symposium and Show, Spectrum '79. Fairmont Hotel, San Francisco, Calif. (Ed Kevin, The O'Brien Corp., 450 E. Grand Ave., South San Francisco, Calif. 94080).

(Mar. 14-17)—Southern Society Annual Meeting. Dutch Inn, Buene Vista, Fla.

(Mar. 16)—Baltimore Society's Coatings Show, Hilton Inn, Baltimore, Md. (Richard C. Chodnicki, The A.B. Kohl Sales Co., 8765 Mylander Lane, Towson, Md. 21204).

(Mar. 20-21)—22nd Symposium of the Cleveland Society, "Advances in Coatings Technology." Baldwin-Wallace College, Berea, Ohio. (Sid Lauren, Coatings Research Group, Inc. 2340 Hamilton Ave., Cleveland, Ohio 44114).

(Mar. 20-21)—Chicago Society SYMCO '79,"Pigments and Color." Fountain Blue, Des Plaines, Ill. (Daryl Luxmore, Drew Chemical Corp., 9 S 684 Highland Ave., Hinsdale, Ill. 60521).

(Apr. 5-7)—Dallas and Houston Societies. Southwestern Paint Convention. Shamrock Hilton Hotel, Houston, Tex. (Don Webb, Jones-Blair Co., P.O. Box 35286, Dallas, Tex. 75235).

(May 3)—Detroit Society FOCUS Seminar, "Recent Advances in Automotive Coatings." Michigan Inn, Detroit, Mich.

(May 3-5)—Pacific Northwest Society. Annual Symposium. Vancouver, B.C., Canada.

(May 22)—Pittsburgh Society. Symposium on "Corrosion Protection." Duquesne University.

(June)—Golden Gate Society Manufacturing Seminar, "Mixing Time '79."

## OTHER ORGANIZATIONS

(Feb. 26-Apr. 27)—Paint Short Courses at University of Missouri—Rolla. For Painting Contractors and Maintenance Engineers—Feb. 26-Mar. 2; Composition of Paints and Coatings—Mar. 5-9; For Paint Inspectors and Quality Controllers—Mar. 26-30; Tinting, Shading, and Matching of Colored Paints—Apr. 2-6; Advanced Coatings Workshop—Apr. 23-27. (Normal Fleming, Extension Div., University of Missouri—Rolla, 501 W. 11th St., Rolla, Mo. 65401).

(Mar. 5-9)—30th Pittsburgh Conference on "Analytical Chemistry and Applied Spectroscopy," Pittsburgh Section, Analytical Group, et al., Convention Center, Cleveland, Ohio, (PCAC & AS, P.O. Box 2128, Lower Barrell, Pa. 15068).

(Mar. 12-13)—Conference on "Quality Assurance in Air Pollution Measurement," sponsored by the Air Pollution Control Association. Grand Hotel, New Orleans, La. (Gus Von Bodungen, Air Quality Section, Louisiana Air Control Commission, P.O. Box 60630, New Orleans, La. 70160).

(Mar. 12-16)—National Association of Corrosion Engineers Annual Conference and Materials Performance and Corrosion Show, CORROSION/79. Atlanta Hilton, Atlanta, Ga. (NACE, P.O. Box 986, Katy, Tex. 77450).

(Mar. 20)—"Toxic Considerations in Fats and Oils Products and Derivatives," sponsored by Northeast Section of American Oil Chemists' Society. Sheraton Inn-Newark Airport, Elizabeth, N.J. (George Willhite, American Oil Chemists' Society, 508 S. Sixth St., Champaign, Il. 61820).

(Mar. 21-22)—Seventh Annual Air Pollution Control Association Seminar, "The Clean Air Act—What's Happening?" Mayflower Hotel, Washington, D.C. (Public Relations Dept., Air Pollution Control Association, P.O. Box 2861, Pittsburgh, Pa. 15230).

(Mar. 22)—"Evaluation Update of America's Voluntary Standards System," sponsored by the American National Standards Institute, Inc. International Inn, Washington, D.C. (Claude H. Burns, Deputy Managing Director, American National Standards Institute, 1430 Broadway, N.Y., N.Y. 10018.

(Mar. 22-23)—Coatings - 79: International Symposium on Coatings. Carillon Hotel, Miami Beach, Fla. (V.M. Bhatnagar, Alena Enterprises of Canada, P.O. Box 1779, Cornwall, Ont., K6H 5V7, Canada).

(Mar. 28)—Annual Symposium of North Central Section of American Oil Chemists' Society. North Shore Hilton, Skokie, Ill. (George Willhite, American Oil Chemists' Society, 508 S. Sixth St., Champaign, Il. 61820).

(Mar. 27-29)—1979 Industrial Pollution Conference. Philadelphia, Pa. (Alan Krigman, ICON Inc., 211 S. 45th St., Philadelphia, Pa. 19104).

Mar. 28-29)—NPCA Symposium on "Benefits and Risks of Computer Use for the Small Paint Manufacturer." Admiral Benbow Inn, Tampa, Fla. (George Savickas, National Paint and Coatings Association, 1500 Rhode Island Ave., N.W., Washington, D.C. 20005).

(Mar. 29-30)—International Conference on Spectroscopy. Konover Hotel, Miami Beach, Fla. (V.M. Bhatnagar, Alena Enterprises of Canada, P.O. Box 1779, Cornwall, Ont. K6H 5V7, Canada).

(Mar. 29-30)—"Control of Volatile Organic Compound Emissions," sponsored by the U.S. Environmental Protection Agency, National Paint and Coatings Association, Association of Finishing Processors of the Society of Manufacturing Engineers, and Air Pollution Control Association. Stouffer's Valley Forge Hotel, Valley Forge, Pa. (Mr. Michael R. Taylor, JACA Corp., 550 Pinetown Rd., Fort Washington, Pa. 19034).

(Apr. 1-6)—Pacific Chemical Conference: 1979. Honolulu, Hawaii. (A.T. Winstead, ACS, 1155 - 16th St., N.W., Washington, D.C. 20036).

(Apr. 2-6)—Div. of Organic Coatings and Plastics Chemistry Symposiums and Spring American Chemical Society Meeting. Hyatt Regency-Waikiki Hotel, Hawaii. (American Chemical Society, 1155 16th St., N.W. Washington, D.C. 20036).

(Apr. 3-6)—OCCA-31. Oil and Colour Chemists' Association 31st Annual Technical Exhibition. Alexandra Palace, London, England. (The Director & Secretary, Oil and Colour Chemists' Association, Priory House, 967 Harrow Rd., Wembley, Middlesex, HAO 2SF, England).

(Apr. 4-5)—NPCA Marine Coatings Conference. Omni International Hotel, Norfolk, Va. (Georgene Savickas, National Paint and Coatings Association, 1500 Rhode Island Ave., N.W., Washington.

(Apr. 19-20)—Second Canadian Chromatography Conference. Hampton Court Hotel, Toronto, Canada. (V.M. Bhatnagar, Alena Enterprises of Canada, P.O. Box 1779, Cornwall, Ont. K6H 5V7, Canada).

(Apr. 9-10)—Washington Paint Technical Group 19th Annual Symposium, "Uncle Sam Wants Your Paint—$100 Million Opportunity. Marriott Twin Bridges Motel, Washington, D.C.

(Apr. 23-24)—Inter-Society Color Council. Annual meeting. Roosevelt Hotel, New York, N.Y.

(Apr. 25-26)—"Control of Volatile Organic Compound Emissions," sponsored by the U.S. Environmental Protection Agency, National Paint and Coatings Association, Association of Finishing Processors of the Society of Manufacturing Engineers, and Air Pollution Control Association. Holiday Inn Chicago City Centre, Chicago, Ill. (Mr. Michael R. Taylor, JACA Corp., 550 Pinetown Rd., Fort Washington, Pa. 19034).

(Apr. 25-May 3)—70th Annual Meeting of the American Oil Chemists' Society. Fairmont Hotel, San Francisco, Calif.

(Apr. 29-May 2)—National Coil Coaters Association Annual Meeting. Marco Beach Hotel and Villas, Marco Island, Fla. (Don White, National Coil Coaters Association, 1900 Arch St., Philadelphia, Pa. 19103).

(Apr. 29-May 3)—70th Annual Meeting, American Oil Chemists' Society. Fairmount Hotel, San Francisco, Calif. (James Lyon, Executive Director, American Oil Chemists' Society, 508 S. Sixth St., Champaign, Il. 61820).

(May 3-4)—International Symposium on "Flammability and Fire Retardants." Opryland Hotel, Nashville, Tenn. (V.M. Bhatnagar, Alena Enterprises of Canada, P.O. Box 1779, Cornwall, Ont. K6H 5V7, Canada).

(May 7-10)—Society of Plastics Engineers, 37th Annual Technical Conference, "Plastics—Efficient Use of Resources." Hyatt Regency Hotel, New Orleans, La. (SPE, Eugene E. Wilson, 656 W. Putnam Ave., Greenwich, Conn. 06830).

(May 10-11)—"Control of Volatile Organic Compound Emissions," sponsored by the U.S. Environmental Protection Agency, National Paint and Coatings Association, Association of Finishing Processors of the Society of Manufacturing Engineers, and Air Pollution Control Association. New Otani Hotel, Los Angeles, Calif. (Mr. Michael R. Taylor, JACA Corp., 550 Pinetown Rd., Fort Washington, Pa. 19034).

# Coming Events (Continued)

(May 15-17)—Powder and Bulk Solids Conference and Exhibition. The Civic Center, Philadelphia, Pa. (Industrial & Scientific Conference Management, Inc., 222 W. Adams St., Chicago, Ill. 60606).

(May 21-25)—"Colloids and Surfaces." Carnegie-Mellon University. (Mrs. Gerry Cohen, Course Coordinator, Post College Professional Education, Carnegie Institute of Technology, Carnegie-Mellon University, Schenley Park, Pittsburgh, Pa. 15213).

(May 21-25)—Polymer Conference Series, "Understanding Polymer Science: Preparation, Properties, and Applications." State University of New York—New Paltz. (Dr. A.V. Patsis, Dept. of Chemistry, SUNY, New Paltz, N.Y. 12562).

(June 4-8)—Polymer Conference Series, "Advances in Polymer Synthesis, Modification, and Characterization." State University of New York—New Paltz. (Dr. A.V. Patsis, Dept. of Chemistry, SUNY, New Paltz, N.Y. 12562).

(June 8-13, 1980)—XVth Congress of FATIPEC. RAI Congress Centre, Amsterdam, Netherlands. "Activities of the Coatings Industry in the Framework of Ecology, Energy, and Economy Problems." (Congress Secretary is C. Korf, Oostenrijklaan 43, Haarlem, Netherlands).

(June 10-13)—American Oil Chemists' Society short course on "Industrial Fatty Acids." Tamiment Resort, Tamiment, Pa. (James Lyon, Executive Director, American Oil Chemists' Society, 508 S. Sixth St., Champaign, Ill. 61820).

(June 10-13)—ASTM Committee D-1 on Paints and Related Coatings and Materials, Shoreham Hotel, Washington, D.C. (J.H. Bystrom, ASTM, 1916 Race St., Philadelphia, Pa. 19103.)

(June 10-13)—Industrial Fatty Acids Short Course sponsored by the American Oil Chemists' Society. Tamiment Resort and Country Club, Pocono Mountain Region, Pa. (Fatty Acids Short Course, American Oil Chemists' Society, 508 S. Sixth St., Champaign, Ill. 61820.

(June 11-15)—Polymer Confeence Series, "Polymers in Electronic Applications: Photopolymers, Photoconductors, Conductors, and Insulators." State University of New York—New Paltz. (Dr. A.V. Patsis, Dept. of Chemistry, SUNY, New Paltz, N.Y. 12562).

(June 14-15)—Society of Plastics Engineers, European Sections RETEC, "Lastest Improvements in the Development and Processing of Polyolefins." Ghent, Belgium, (Jacques de Craene, RIGI p.v.b.a. Noorderlaan 98/36 2030-Antwerpen-Belgium).

(June 17-20)—Dry Colors Manufacturers Association, Annual Meeting. The Greenbrier, White Sulpher Springs, W. Va. (J.L. Robinson, DCMA, Suite 100, 1117 N. 19th St., Arlington, Va. 22209).

(June 18-22)—Polymer Conference Series, "Advances in the Stabilization and Controlled Degradation of Polymers." State University of New York—New Paltz, (Dr. A.V. Patsis, Dept. of Chemistry, SUNY, New Paltz, N.Y. 12562).

(June 20-23)—Oil and Colour Chemists' Association Conference, "The Challenge to Coatings in a Changing World." Stratford Hilton Hotel, Stratford-on-Avon, England. (The Director and Secretary, OCCA, Priory House, 967 Harrow Rd., Wembley, Middlesex HAO 2SF, England).

(June 24-29)—Air Pollution Control Association 72nd Annual Meeting and Exhibition, Cincinnati Convention-Exposition Center, Cincinnati, Ohio. (Public Relations Dept., Air Pollution Control Association, P.O. Box 2861, Pittsburgh, Pa. 15230).

(June 25-26)—European Conference on Paints and Coatings, "Eurocoatings-79." Excelsior Hotel, Rome, Italy. (V.M. Bhatnagar, Alena Enterprises of Canada, P.O. Box 1779, Cornwall, Ont. K6H 5V7, Canada).

(June 25-29)—Polymer Conference Series, "Understanding and Assessing Paper and Fibrous Structures." State University of New York—New Paltz. (Dr. A.V. Patsis, Dept. of Chemistry, SUNY, New Paltz, N.Y. 12562).

(June 28-29)—3rd European Conference on "Flammability and Fire Retardants." Excelsior Hotel, Rome, Italy. (V.M. Bhatnagar, Alena Enterprises of Canada, P.O. Box 1779, Cornwal, Ont. K6H 5V7, Canada).

(July 2-3)—World Spectroscopy Conference. Sheraton Hotel, Lisbon, Portugal. (V.M. Bhatnagar, Alena Enterprises of Canada, P.O. Box 1779, Cornwall, Ont. K6H 5V7, Canada).

(July 5-6)—World Chromatography Conference. Sheraton Hotel, Lisbon, Portugal. (V.M. Bhatnagar, Alena Enterprises of Canada, P.O. Box 1779, Cornwall, Ont., K6H 5V7, Canada).

(July 16-20)—International Conference in Organic Coatings sponsored by the State University of New York and Greek Professional Societies. Athens, Greece. (Dr. A.V. Patsis, Conference Director, State University of New York at New Paltz, CSB 209, New Paltz, N.Y. 12562).

(July 17-20)—"Fifth Annual International Conference in Organic Coatings Science and Technology," Athens, Greece. (Angelos V. Patsis Institute in Science & Technolgoy, State University of New York, College at New Paltz, Department of Chemistry, New Paltz, N.Y. 12562).

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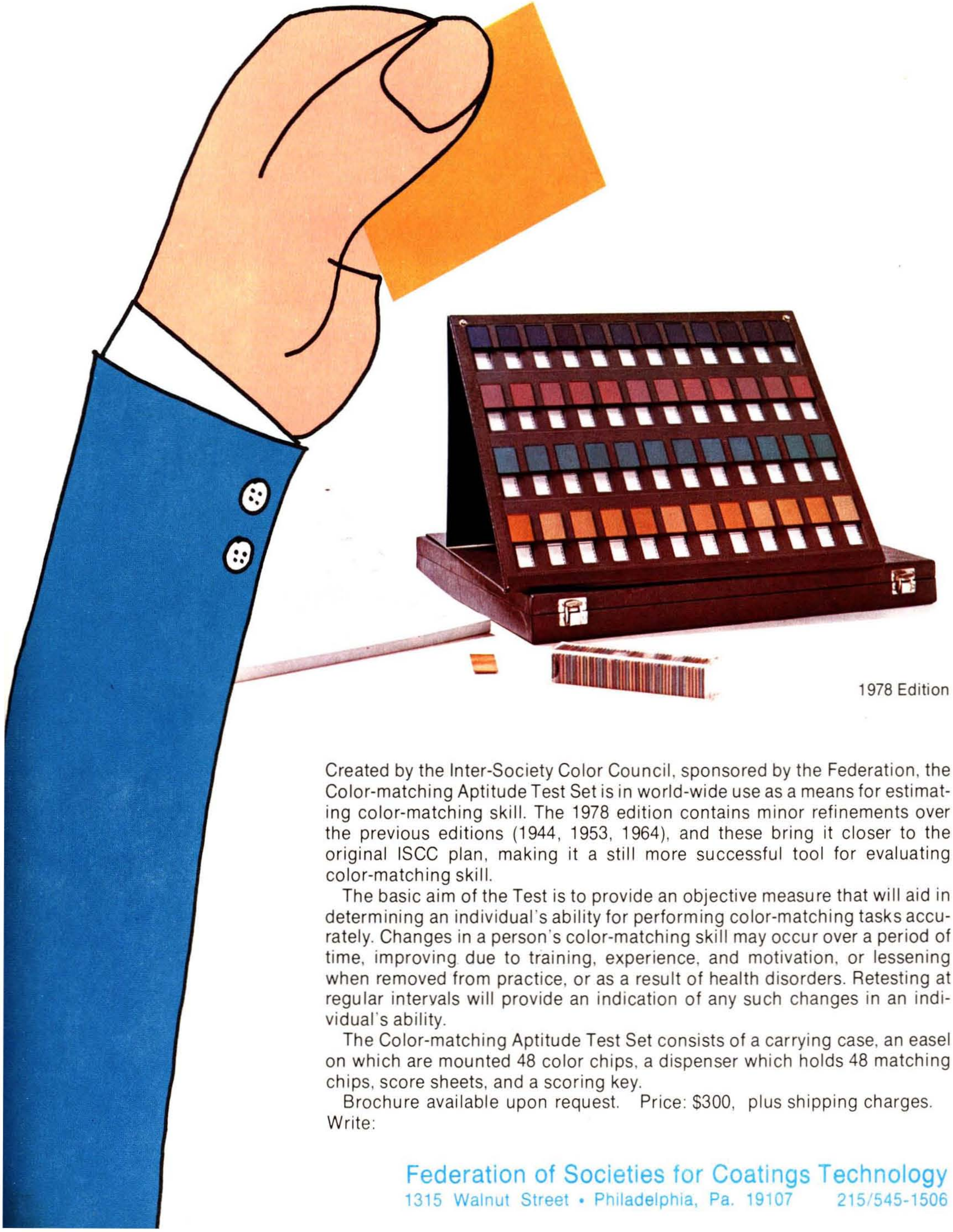
# Color-matching Aptitude Test Set



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Created by the Inter-Society Color Council, sponsored by the Federation, the Color-matching Aptitude Test Set is in world-wide use as a means for estimating color-matching skill. The 1978 edition contains minor refinements over the previous editions (1944, 1953, 1964), and these bring it closer to the original ISCC plan, making it a still more successful tool for evaluating color-matching skill.

The basic aim of the Test is to provide an objective measure that will aid in determining an individual's ability for performing color-matching tasks accurately. Changes in a person's color-matching skill may occur over a period of time, improving due to training, experience, and motivation, or lessening when removed from practice, or as a result of health disorders. Retesting at regular intervals will provide an indication of any such changes in an individual's ability.

The Color-matching Aptitude Test Set consists of a carrying case, an easel on which are mounted 48 color chips, a dispenser which holds 48 matching chips, score sheets, and a scoring key.

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