jct JOURNAL OF COATINGS TECHNOLOGY

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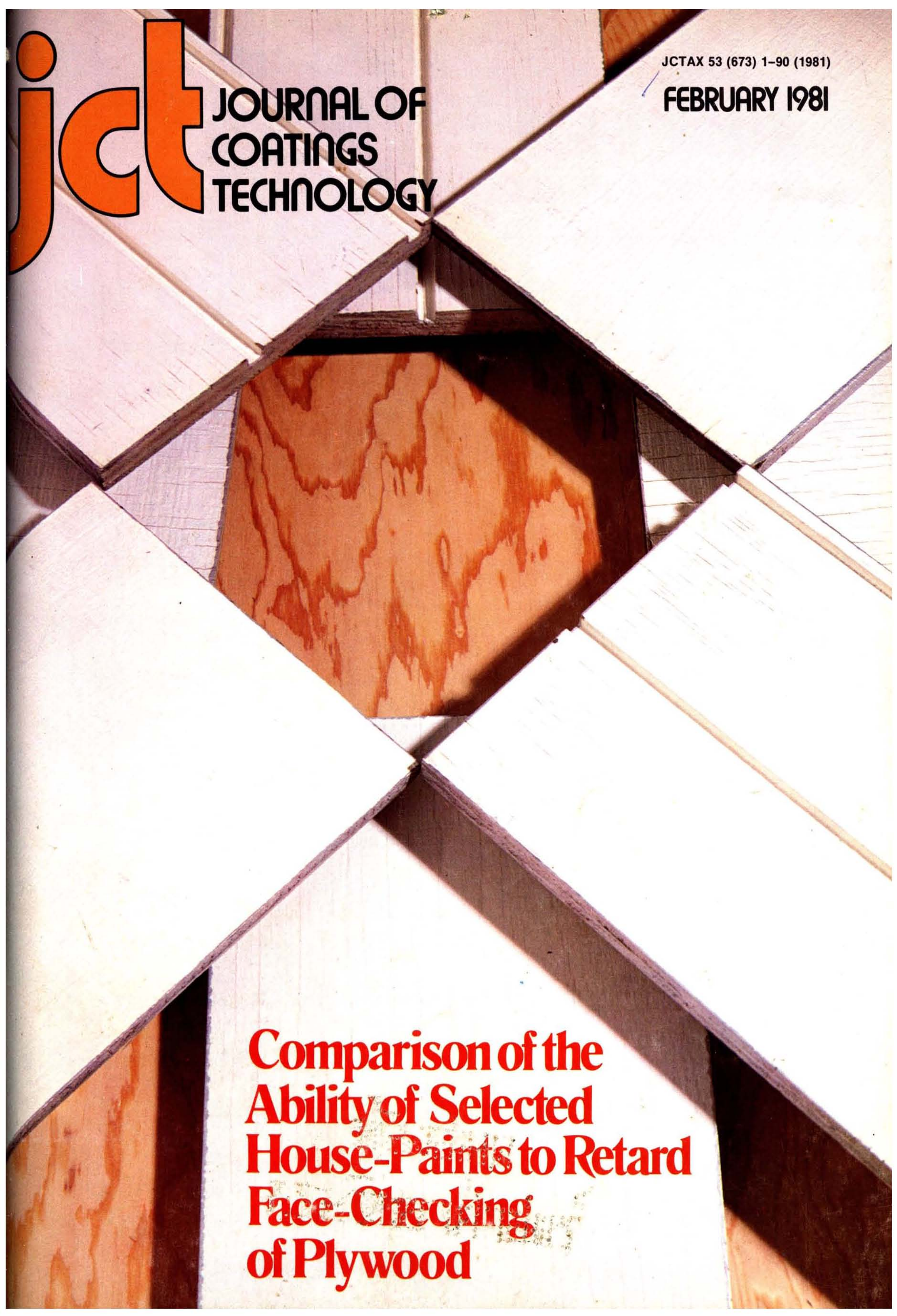

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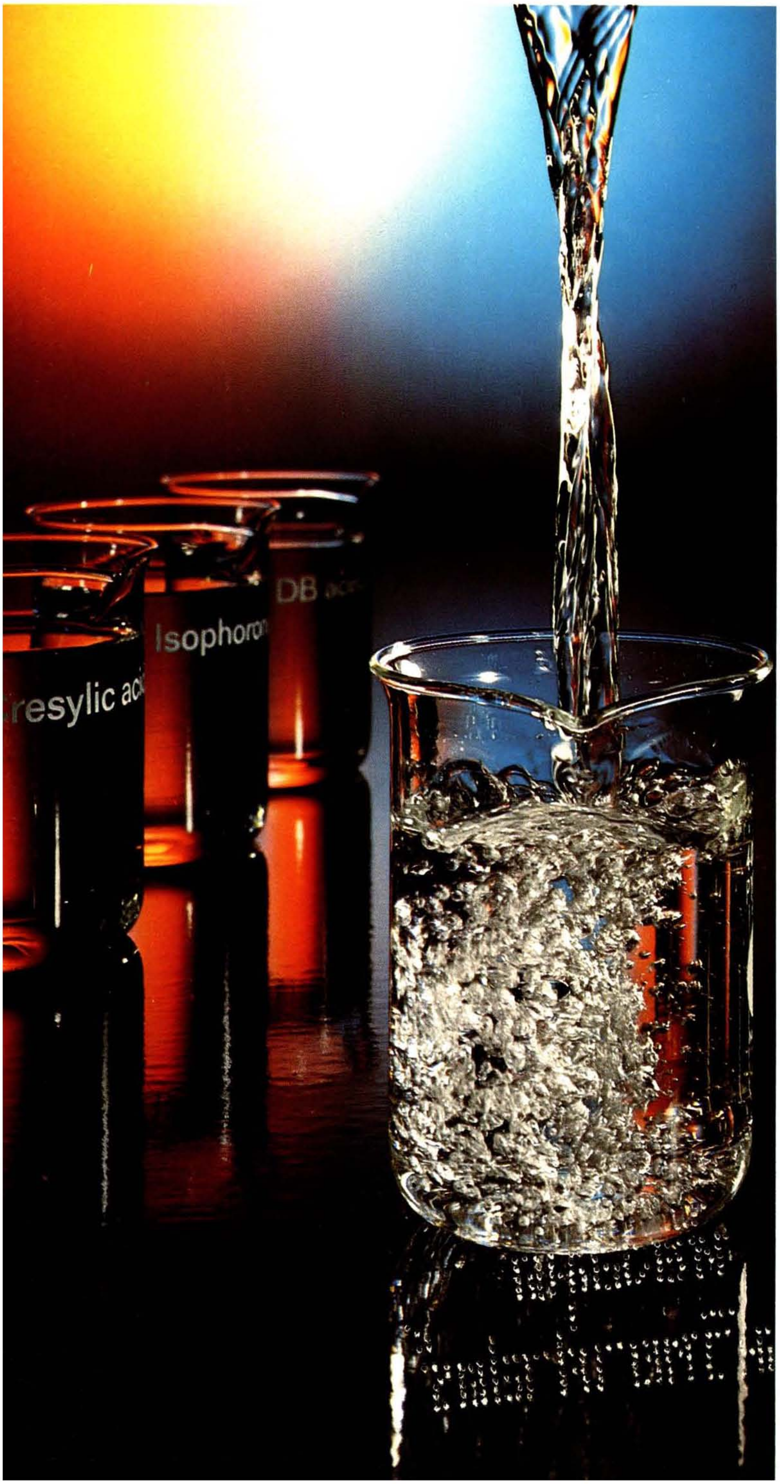

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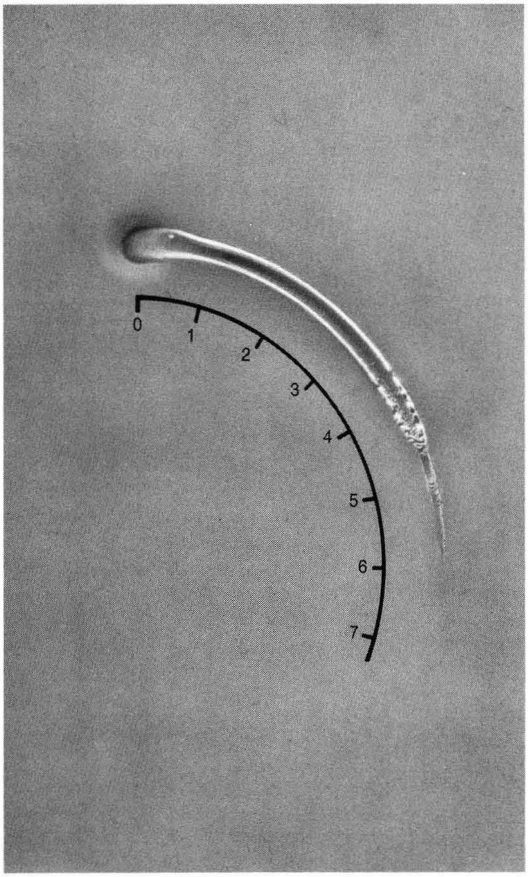
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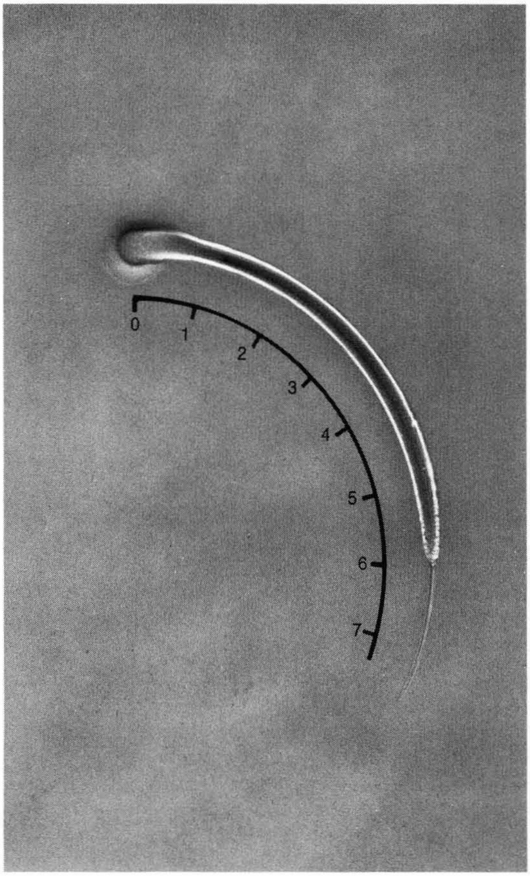
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FEBRUARY 1981

JOURNAL OF COATINGS TECHNOLOGY

Volume 53 Number 673

Features

21 REPORT OF THE RESEARCH DIRECTOR—R.R. Myers

35 SYNTHESIS OF BLOCKED MDI ADDUCTS, THEIR DSC EVALUATION AND EFFECT OF PIGMENTATION—T. Anagnostou and E. Jaul

49 USE OF THIIRANE-FUNCTIONAL MONOMERS AS A MEANS OF DEVELOPING CROSSLINKABLE EMULSIONS—R.M. O'Brien, S.A. Brown, B.G. Bufkin, and J.R. Grawe

61 COMPARISON OF THE ABILITY OF SELECTED HOUSE PAINTS TO RETARD FACE-CHECKING OF PLYWOOD—J.A. Emery

Federation Activities

16 1980–81 CONSTITUENT SOCIETY OFFICERS

21 PROCEEDINGS OF THE PAINT RESEARCH INSTITUTE NO. 141

47 THREE SOCIETIES WIN 1980 MMA AWARDS

Departments

7 Comment

11 Government and Industry

12 Abstracts

71 Society Meetings

73 Future Society Meetings

74 Elections

79 Meetings/Education

82 People

83 Literature

86 Book Review

86 Letters to the Editor

87 Coming Events

90 Humbug from Hillman

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Comment

Scholarships Lend a Helping Hand

One of the keen interests of our industry is encouraging students to pursue coatings careers, and the Federation continues to assist in this regard through its scholarship program.

These grants-in-aid have been awarded for many years to various colleges and universities who offer coatings technology curricula. The funds are awarded with the stipulation that preference be given to qualified scholarship applicants who are children of members of the Federation.

A total of $15,000 will be awarded for the 1981-82 academic year to the following institutions:

University of Southern Mississippi — $6,000
North Dakota State University — $4,000
University of Detroit — $2,000
Kent State University — $2,000
University of Missouri—Rolla — $1,000

Members who have children desiring to pursue a career in the coatings industry should apply for these funds promptly. Details are available from the Federation office.

To receive preference, the application from each child of a member must be received by the school of choice before April 1. This deadline affords sufficient time for recruiting efforts to encourage other qualified students to participate in the program.

The scholarship grants, which are a benefit of Federation membership, are also a means by which our organization makes a meaningful contribution to the well-being of our industry. By helping to attract and train students for careers in coatings, we are making an investment in our most important and irreplaceable resource—people.—TAK

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We built our reputation on superior service as well as products. Our technical staff is always here to work with you. To answer any questions you have. To help choose the precise formulation you need. And after you order, our multiple plant locations offer the benefits of reliable supply, fast delivery, and savings in shipping costs. So when it comes to Low-VOC coatings, get the help you need. And the answers you want. Contact one of our field representatives ... or our Marketing Services Dept., Independence Mall West, Philadelphia, PA 19105.

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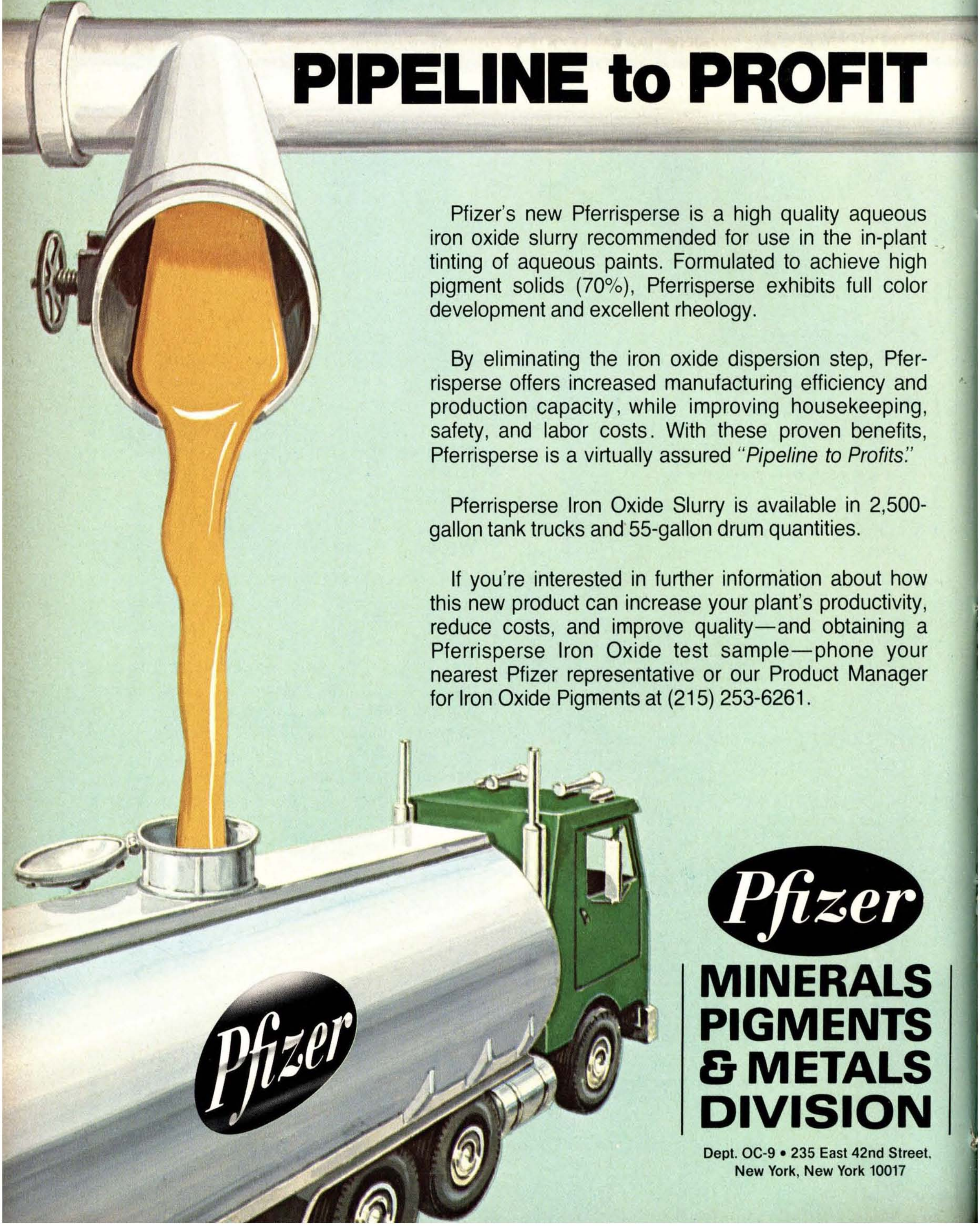


Pfizer

Government and Industry

Battelle Indicates U.S. R&D Funding Will Surpass $68 Billion This Year

Expenditures in calendar year 1981 for research and development in the United States are expected to reach \$68.6 billion, according to the annual forecast of Battelle's Columbus Laboratories. This represents an increase of \$8.2 billion (13.7%) over the \$60.4 billion that the National Science Foundation estimates was to be actually spent for R&D in 1980.

While most of the increase will be absorbed by continued inflation (estimated at 9.9% in 1981), Battelle forecasts a real increase in R&D expenditures of 3.8%. This is slightly higher than the seven-year average rate of 3.4% in real R&D effort that has been experienced since 1973.

In 1981, Battelle sees an increase of 14% in federal support for R&D, with funding expected to be about \$33.7 billion. This is 49.1% of the total R&D expenditures for 1981. Industrial funding is forecast to be \$32.4 billion, up 12.9% from 1980. This sector is expected to account for 47.2% of the total R&D funding. Funding by academic institutions is expected to be \$1.4 billion (2.1% of the total), and other nonprofit organizations will provide \$1.1 billion (1.6%).

While the federal government continues to be the dominant source of research funds, industry remains as the dominant performer. In 1981, performance of R&D by industry is expected to rise to \$48.0 billion, or 70.0% of all research performed. This compares with \$9 billion (13.1%) for the federal government, \$9.1 billion (13.3%) for academic institutions, and \$2.5 billion (3.6%) for other nonprofit organizations.

The Battelle forecast notes that federal funding supports research performance in all four sectors. Currently, about one-fourth goes to support R&D conducted by the government itself; almost half goes to industry; approximately one-fifth goes to colleges and universities; and the rest, less than one-twentieth, goes to other nonprofits.

Industry absorbs almost all of its own funds, either performing the R&D itself or contracting with other industrial performers. Its contracts and grants to colleges and universities slightly exceed those to other nonprofit institutions. Other nonprofits finance both themselves and the academic institutions about equally; colleges and universities use up all the funds they originate.

Four government agencies dominate the federal R&D scene and are expected to account for 86.7% of total federal R&D funding in 1981. These are the Department of Defense, 45.0%; the National Aeronautics and Space Administration, 15.7%; the Department of Energy, 15.1%; and Health and Human Services, 12.1%.

The forecast notes that national security, reflected in the Department of Defense budget, is a principal driving force in furthering R&D spending. Also, the continuing emphasis on energy, and the associated problems of balance of payments and insecure resources, will result in increased efforts in the research, development, demonstration, evaluation, and diffusion of new energy technologies.

Additionally, it is anticipated that R&D funds will continue to support the biological and "soft" science areas; educational research directed toward support of expanding technical areas; "impact" research that covers environmental, ecological, and socio-economic consequences of technological or related actions; and research on methods to comply with regulations.

Industrial support of research is growing in fields affected by regulations and in those fields most directly influenced by the need for more energy-efficient products and processes. R&D will be heavily self-funded in the manufacturing industries, where on the average, only 32.9% of the total will be supported by the federal government. The non-manufacturing industries do relatively little R&D, and support for this activity will be divided almost equally between federal and industrial support.

In addition, the forecast discusses the impact the new Administration and changes in congressional leadership are likely to have on R&D expenditures in 1981.

According to the report, it is anticipated that the new Administration's posture relative to business and industry may provide an atmosphere more conducive to industrial R&D. In particular, greater efforts will be directed toward both short-term and long-term R&D aimed at an improved competitive position vis-a-vis foreign competition. Furthermore, there are indications that industry may judge the overall government attitude to be more conducive to increased business profitability, causing industry to increase investment in R&D.

Government/Industry Continued on Page 78.

Abstracts of Papers in This Issue

SYNTHESIS OF BLOCKED MDI ADDUCTS, THEIR DSC EVALUATION AND EFFECT OF PIGMENTATION—T.J. Anagnostou and E. Jaul

Journal of Coatings Technology, 53, No. 673, 35 (Feb. 1981)

This study investigates the use of a differential scanning calorimeter (DSC) to designate the temperature range at which the unblocking of blocked isocyanate compounds will occur.

Three blocked versions of 4,4′-diphenyl methane diisocyanate (MDI) were synthesized. These were the adducts of methyl-ethyl ketoxime, ε-caprolactam, and benzotriazole. They were selected because they represented adducts which were capable of decomposing over a wide temperature range. Confirmation of their unblocking temperature by infrared (IR) spectroscopy techniques on samples baked at temperatures specified by the DSC studies was, in general, successful.

Changes were observed in the temperature range at which adduct unblocking occurs when a polymeric compound containing primary hydroxyl groups (polyoxyethylene glycol) is present. Addition of pigments to the mixture of blocked MDI adducts and polyoxyethylene glycol had minimal effect on unblocking of the adducts at the lower and medium temperature ranges. However, at high temperature ranges, significant changes were observed.

USE OF THIIRANE-FUNCTIONAL MONOMERS AS A MEANS OF DEVELOPING CROSSLINKABLE EMULSIONS—R.M. O'Brien, S.A. Brown, B. G. Bufkin, and J.R. Grawe

Journal of Coatings Technology, 53, No. 673, 49 (Feb. 1981)

A feasibility study was conducted which investigated the acceptability of using 2,3–epithiopropyl methacrylate (ETPM) as a means of developing crosslinkable emulsions.

To prevent premature reactivity of the thiirane functionality in the emulsion environment, the polymerizations were conducted at low temperatures using nonionic surfactants and a two-step, delayed addition of episulfide monomer. However, in spite of the preventative measures taken, compositions containing in excess of 6 mole percent ETPM possessed inordinately high degrees of premature crosslinking. Therefore, the 54.5/36.4/9.1–EA/MMA/ETPM copolymer emulsion (6 mole percent ETPM) was selected as the most reasonable candidate for further evaluations.

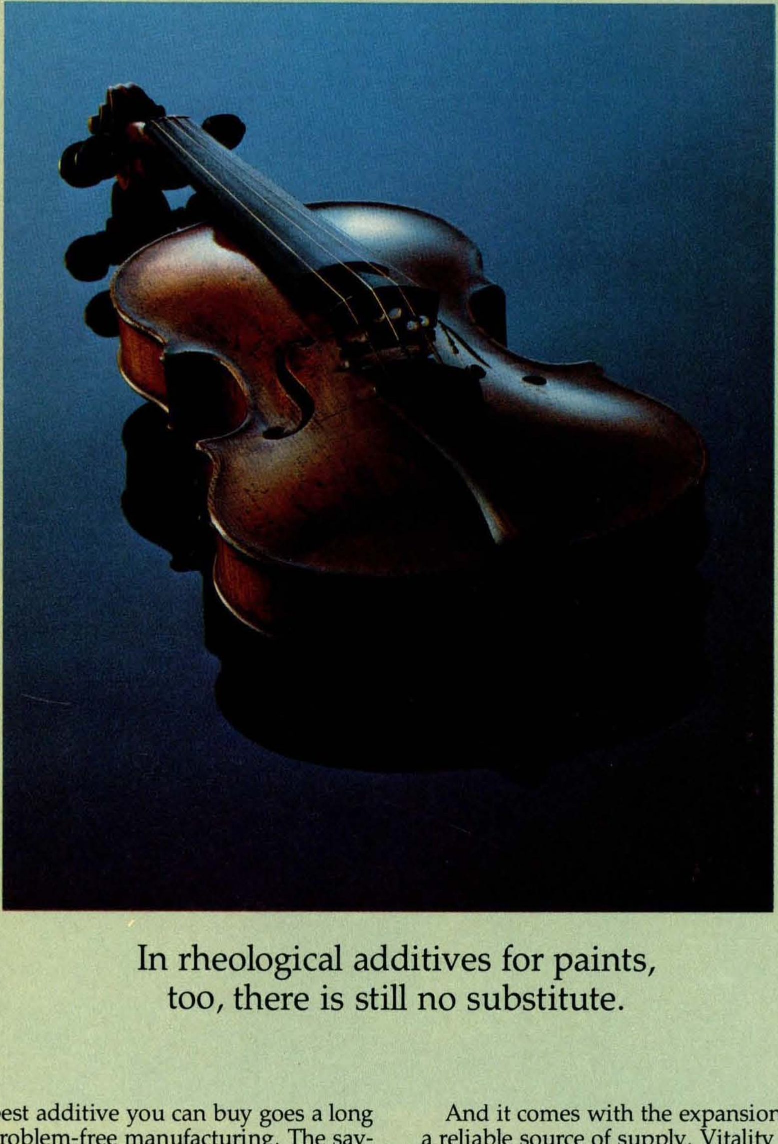
When the model emulsions containing 6 mole percent ETPM were combined with various chemically and structurally different curing agents, the samples containing piperazine displayed a 2.5 fold increase in tensile strength, a 2.3 fold increase in solvent resistance, a 2.7 fold decrease in elongation, decreased impact resistance, and increased film hardness (2H vs 2B) after thermal curing (30 min/65° C) as compared to samples of the same emulsion evaluated without a curing agent.

Coatings of the ETPM–containing emulsion were also found to crosslink at room temperature when piperazine was used as a curing agent.

COMPARISON OF THE ABILITY OF SELECTED HOUSE PAINTS TO RETARD FACE-CHECKING OF PLYWOOD—J.A. Emery

Journal of Coatings Technology, 53, No. 673, 61 (Feb. 1981)

Plywood specimens finished with various house paints were exposed outdoors for 5.5 years at four geographic locations and to an artificial laboratory test cycle. The performance of the paints during both outdoor and laboratory exposures indicates that certain all-acrylic latex paints decreased both the rate and the degree of face-checking relative to paints which used an organic solvent-thinned primer and acrylic or vinyl-acrylic latex topcoat. Although some all-acrylic latex systems performed better than others, this generic type of finish appears to offer advantages over paints with a solvent-thinned primer and a latex topcoat. Certain stain-blocking acrylic latex systems appeared to be especially effective in reducing face-checking.



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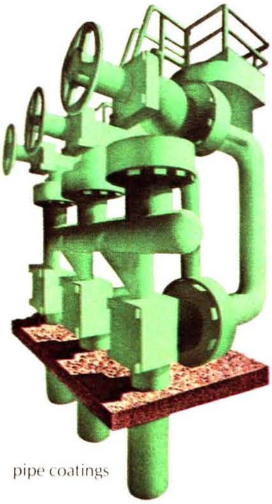
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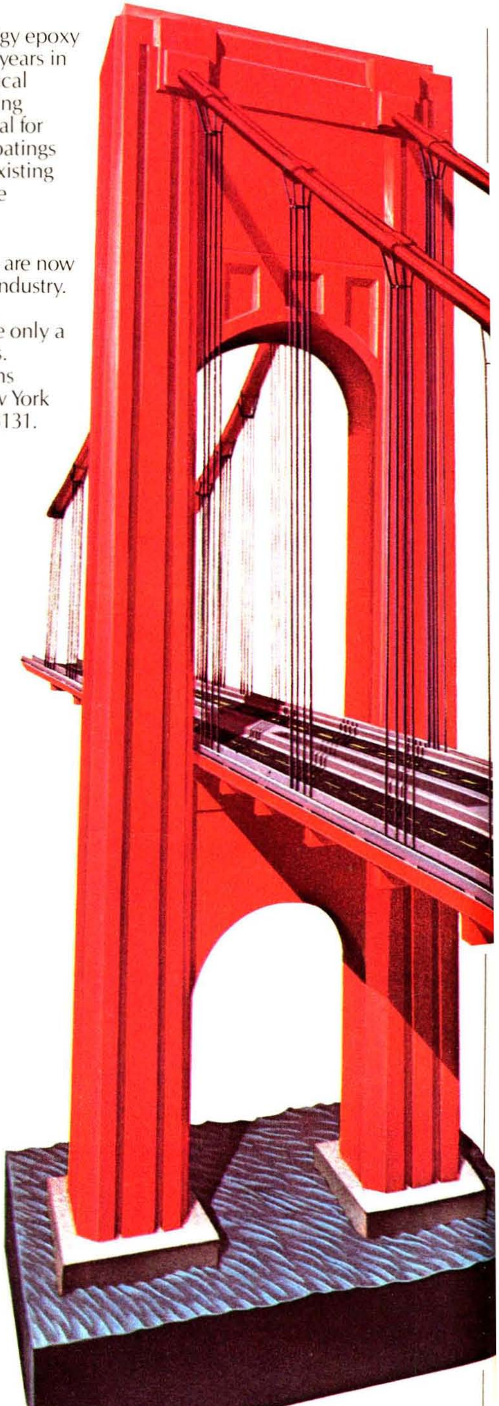

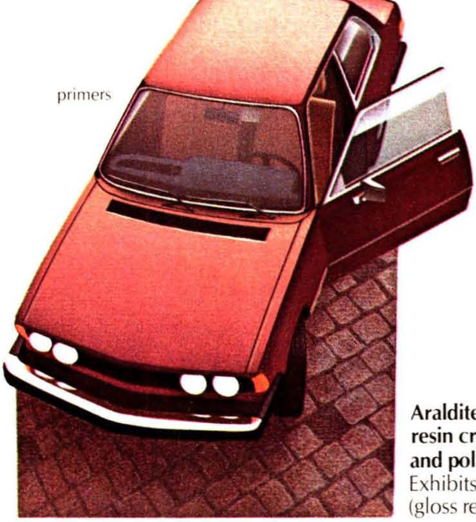
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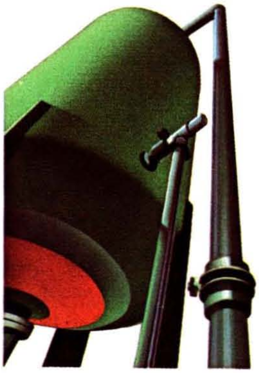

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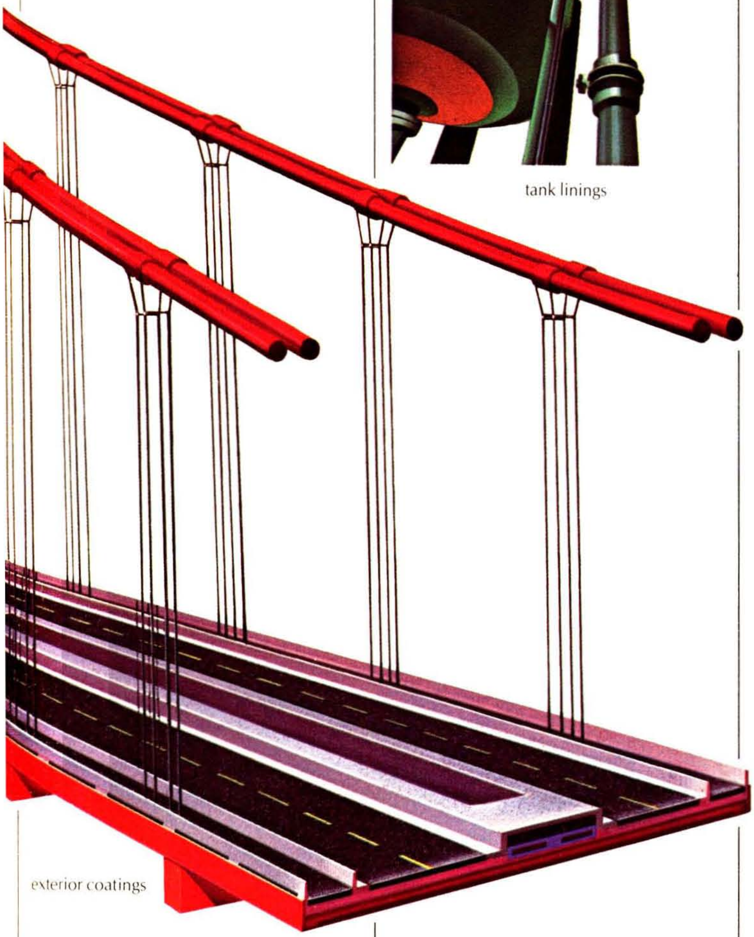

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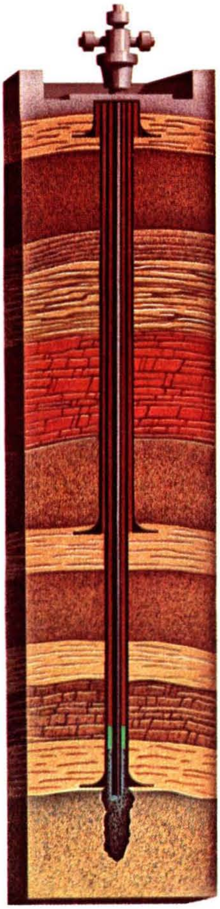

down hole piping coating

CIBA–GEIGY

PRI Paint Research Institute
of the Federation of Societies for Coatings Technology

PROCEEDINGS/NUMBER 141

Report of the Research Director

Raymond R. Myers*
Research Director, Paint Research Institute

Introduction

Six grants were active in 1980. Three concerned mildew defacement; two were part of the aqueous program; and one was launched in the area of high solids. Progress on these grants is covered.

Plans are laid for the 1981 symposium on Stability and Stabilization of Coatings Systems. Four half-day sessions will be held on May 4–5, 1981.

An encounter session has been scheduled for December 18, 1980 on the subject of High Solids Coatings.

A new service to Federation members has been started by PRI. Tutorials on the use of computers are planned after a canvass is made of the need perceived by a sampling of small companies. Software packages will be generated.

A critical survey on corrosion control has been commissioned.

Grant Progress in 1980

MILDEW CONSORTIUM

An analysis of how the consortium has progressed since he became program manager in March 1980 was presented by Dr. Charles C. Yeager at the Federation Annual Meeting in Atlanta.

A summary of the projects and an indication of how they interrelate follow the usual format: results of Drs. Pittman, Zabel, and Siehr are given along with a brief description of each grant.

Polymer-Anchored Mildewcides

PRINCIPAL:

Dr. C. U. Pittman, University of Alabama.

ABSTRACT:

Grafts of fungicides to acrylic polymers are made and paint systems are made from the resulting copolymer. Laboratory tests show that fungicide is active but does not leach from the coating.

RELEVANCE:

A fixed fungicide should be long-lasting and not toxic, even if it is soluble or volatile as an additive. Toxicity would be minimized if the fungicide were attached by a bond that resists salivary enzymes.

COMMENTS:

This largely synthetic program is concerned with copolymers in which fungicidal groups are attached to acrylic polymer backbones, utilizing such linkages as esters, amides, and ethers. Such a bound fungicide would be a permanent part of the binder until it is attacked by microorganisms (in the ideal sense) or by weathering (in the practical sense). It would neither vaporize nor leach and, therefore, the moiety could be selected from candidates that suffer from volatility or solubility.

In the early stages of the research the guiding principle was to create permanent bonds, mostly of the ester variety. A laboratory test for efficiency was developed wherein exposure of painted squares on agar to a test organism resulted in squares of inhibition rather than large areas of inhibition around the square. The latter would signify leaching had occurred, at least into the agar medium.

An improvement on the laboratory test was developed by Dr. Zabel and is described in the account of his grant. It is too difficult to prove that a restricted zone of inhibition is a necessary and sufficient condition for anchoring to be successful, other than to surmise that a wide zone achieved with a known fungicide implies little or no anchoring.

The fungicidal copolymers that were first synthesized from acrylates contained the ester linkage. Then the work

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*Kent State University, Chemistry Dept., Kent, OH 44242.

was extended to amides and ethers. Because a proper balance between hydrolysis and enzyme attack was used as a guiding principle, the nucleophilicity of the leaving group on hydrolysis was altered. These and other variables are being subjected to laboratory study as the first phase of the program.

The second phase is the one which calls for some explanation. At least two Societies want to try out these new fungicidal polymers on test fences. The mildew consortium is reluctant to propose any of the experimental formulations because more data are necessary to ensure that the enzymolysis/hydrolysis balance has been achieved. The committee is striving to avoid the Edisonian approach of throwing a large group of candidates into the hopper and observing which ones survive, and the synthetic routes are so complicated that careful selection of the early candidates is necessary. The available samples are in short supply, and a carefully designed protocol for their testing has been worked out by the committee.

The Role of *Aureobasidium pullulans* In the Disfigurement and Deterioration of Latex Paint Films

PRINCIPAL:

Dr. R. A. Zabel, SUNY Syracuse (The Leo Roon Fellowship).

ABSTRACT:

Mildew growth on wood is revealed in early stages by controlled (axenic) culture. Nutrient and moisture requirements are determined.

RELEVANCE:

Growth requirements are a prelude to stunting mold growth. The assay procedure developed in this study is rapid, reliable, and needed by the industry.

COMMENTS:

The earliest finding in the mildew program was the detection by Dr. John Schmitt, of Ohio State University, of a symbiotic relation between *A. pullulans* and bacteria. This symbiosis was picked up by other researchers in the program and the concept grew until most of us thought that a succession of organisms prevailed in which Ap was the end point in a fungal succession.

Unfortunately, the idea began to circulate that a succession was mandatory and that Ap would not grow unless some other organism had paved the way. At this stage, we began to ask Dr. Zabel to delineate, once and for all, the nutritional requirements of Ap. Before he began his study, there was a general feeling that Ap would not grow on a fresh latex film and that enzymatic or hydrolytic attack had to precede successful colonization.

When Dr. Zabel developed a method to grow pure cultures of Ap on fresh latex films, the succession theory vanished. I feel that the consortium should not abolish attempts to observe successions because microecology has a role to play in the overall perspective. At any rate, no one is talking about succession these days.

An even more iconoclastic finding is the indication from microscopy that Ap does not digest paint. Our attempts to find a polymer that fights back when attacked by mildew is built on the surmise that enzymatic attack accompanies the growth of a mildew colony. It is known that exogenous carbon sources provide all the nutrient that is needed by fungi growing on exposed surfaces, so a variation on the anchored fungicide theme is needed. One of the variations suggested by this work is that of controlled hydrolysis of the anchored polymer, so that one need not rely on enzymatic attack to trigger the release of the fungicide.

The key to Ap's success appears to be the existence of detritus and moisture contents above certain levels. Whether or not these factors come as a surprise, they focus on formulation changes that are available to coatings manufacturers. Latexes should contain minimum amounts of humectant and should be nonporous. Among the compliance coatings under consideration are water-sensitized polymers that have been rendered soluble by neutralizers that may be humectants in their own right.

The continuing momentum on Dr. Zabel's grant involves the development of a laboratory procedure for facilitating rapid development of Ap on paint under laboratory conditions that simulate outdoor exposures, in every sense, except for the control which the experimenter exerts on the colonizing species. Routine tests have been established to determine optimum conditions for identified key factors (e.g., 28°C, sapwood over heartwood, hardwoods over conifers, 0.5% pollen extract). The procedure relies on reflectance of diffuse illumination, measured at 457 nm, as recommended by TAPPI.

Progress in 1980 occurred in narrowing the variables associated with the assay. Latex paints were applied to one side of a 3 × 7.5 × 0.5 cm sapwood substrate. Sterilization by steam was followed by insertion into square bottles containing Vermiculite moistened with a mineral/glucose medium. Ap was inoculated and reflectances were measured weekly.

Gravimetry revealed that the only weight loss of a paint film during Ap growth was confined to about 2% and to the first two weeks of incubation. This behavior was attributed to the leaching of low molecular weight material from the paint. Measurement of the fungal biomass was based on glucosamine.

Zabel's bioassay procedures are now in service. Samples of anchored fungicides, both weathered and unweathered, are in the chambers. Results will be available in three months.

Control of Cell-Wall Biosynthesis in *Aureobasidium pullulans*

PRINCIPAL:

Dr. Donald J. Siehr, University of Missouri, Rolla.

ABSTRACT:

A. pullulans protoplasts are made (free of cell walls) and the biochemistry involved in their manufacture of new walls is studied.

RELEVANCE:

Fungicides may be discovered by knowing how they interfere with synthesis of cell walls.

COMMENTS:

This grant fits in the overall scheme of the mildew program in the sense that a means to tamper with cell wall synthesis might provide a permanent solution to the control of mildew. If one cannot deny nutrient because of detritus, and if one does not have a mandated succession to attack at a weak point, then the only recourse may be to alter the life cycle by a potent component.

This component need not be a fungicide nor need it be toxic to humans. Our best chance to find it is to understand the biochemical pathway in wall synthesis and in the companion synthesis of the black pigment.

This kind of work proceeds slowly in the hands of researchers which generally work in large teams with ample funding. Results are even slower to emerge with limited programs. The first problem of producing viable protoplasts of Ap has just been solved, and now the focus has turned to their ability to regenerate cell walls. This problem may be solved by the time the grant terminates next June, but it is unlikely that inhibitors of either cell wall or pigment synthesis will be found by that time.

AQUEOUS COATINGS

Two grants are active in a program designed to study the physical chemistry of water-borne polymers. In a third grant which was phased out this year, a standard copolymer formulated with acrylic acid and butyl methacrylate was made available to the participants and was used in their sequence of studies.

The general theme of this program was spelled out in a prospectus appended to my last report and was accepted with enthusiasm by the Trustees. A program manager was authorized for the purpose of implementing the ideas in the prospectus; after this step has been taken a decision will be reached on whether two standard systems should be selected for the program or whether we should adhere to acrylics.

Rheology of Water-Soluble Acrylics

PRINCIPAL:

Dr. Zeno W. Wicks, Jr., North Dakota State University.*

ABSTRACT:

Standard copolymer was synthesized from acrylic acid and butyl methacrylate in t-butanol. Solutions of two different acid numbers were supplied to all principal investigators. Hill studied viscosity trends on dilution with water.

RELEVANCE:

Extensive characterization of a system resembling a practical aqueous coating is necessary in order to gain control of the physical properties that govern storage, application, and drying.

COMMENTS:

This grant terminated in June but has been kept active by Dr. Wicks. The current effort is to elucidate the effect of molecular weight on the location and magnitude of the enormous viscosity increase that occurs around 20–23% solids.

With a threefold variation in molecular weight at acid number 37, the copolymer, 75% neutralized by dimethylamino ethanol, showed fivefold increases in viscosity at 20% solids and 50-fold increases at 22% solids. Because of shear thinning, especially at 22% solids, these values are rough approximations.

A slight increase was observed in viscosity on aging of some of the samples.

Film Formation and Polymer Transitions

PRINCIPAL:

Dr. Raymond R. Myers, Kent State University.

ABSTRACT:

Water-borne acrylic acid copolymers dry at rates and to degrees that are determined by the neutralizer and degree of neutralization. Water retention is ascertained and related to clustering tendencies.

RELEVANCE:

Energy requirements and time lapses on drying are severe drawbacks, as is water sensitivity of the dried film. Formulation variables to maximize economy should be indicated by systematic study.

COMMENTS:

A paper was published on the drying of poly (acrylic acid) in *I & E Chem. Product Quarterly,* September 1980. A second paper on amine-neutralized PAA and the PRI copolymer has been written and will be submitted to JOURNAL OF COATINGS TECHNOLOGY.

In brief, the findings of these two papers are that neutralization of carboxylated polymers expands the molecule so that gelation sets in during drying at lower solids contents. Once this happens, subsequent water loss results in residual stresses that lead to film failure. Ways to ameliorate the problem were suggested by adjusting the hydrophilicity of the amine used to neutralize the polymer and also by using branched amines. The two effects are complementary in that branching reduced hydrophilicity in the studies performed here, and evidence is presented in the paper that the simple picture of steric hindrance with the amine group will not suffice. We suspect that intercalation (i.e., a spatial fit of the amine cation with the macromolecule) plays a role.

Committee recommendations that the effect of relative humidity be ascertained were responsible for a complete rebuilding of the apparatus housing and air delivery system. The new PRI student, Shen Ren Chuu, has built a transparent plastic chamber which will accommodate the impedometer and the weight loss panel, one above the other, in a current of collimated air of accurately

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*Original grant was made to Dr. Loren Hill, now in industry.

maintained relative humidity and controlled flow rate. Problems in controlling evaporation rates are expected to be solved by this new design.

Rheology is not the only aspect of the study of the carboxylated polymers. Evidence is presented in the pair of publications described above that a change occurs in poly(acrylic acid) at half neutralization, that may not be perceptible in copolymers of the type used in aqueous coatings. And yet, these changes portend a sudden increase in hydration capacity which would render coatings more retentive of water, as long as the neutralizing agent was present. Water analyses by Karl Fischer titration confirmed this situation.

Another adjunct to rheology is the measurement of dielectric constant and dielectric loss in a study of relaxations in regions where the polymers undergo transitions. Dielectric measurements are so sensitive to polar groups that they serve as sensitive probes of carboxyls and of the neutralizing groups adjacent to them. Dielectric properties are ultrasensitive to water, and, as a consequence, they offer more promise in studying the types of oligomers used in high solids than in studying aqueous solutions; however, their use in characterizing dry films is sufficiently promising to warrant investigation.

Publication of dielectric results is not yet ready. We are discovering anomalies that really should not be discussed until data can be presented depicting normal behavior, so that the anomalies can be interpreted with reference to the norm. Dry, homogeneous polymer provides the norm; whereas polymer with t-butanol, especially with water present, begins to show anomalies. We suspect adduct formation.

At present, we are looking into the conditions under which the cosolvent competes effectively with the neutralizer. On the surface, one would not expect competition to be beneficial. In case it is, we have preliminary data on dioxane as a cosolvent that suggest the participation of both ethereal oxygens in the molecule. Dioxane had been selected as a less polar solvent for dielectric studies, not because of practical considerations.

With the restructuring of the aqueous coatings program, it is likely that emphasis will be placed on factors other than relative humidity. Our interests run more toward the high solids program because it will be these systems that will provide the best fit of rheological and dielectric information. In both cases, one is concerned with dispersion (i.e., the dependence of the measured quantity on frequency). Each mechanical or dielectric dispersion region is associated with a group which becomes immobile as the temperature is reduced (or as the frequency is increased). If the group contains dipoles, a correspondence between dielectric and mechanical dispersions can be found.

Dielectric data are easily obtained with a high degree of precision, but they are difficult to interpret. Mechanical data are not as precise but they are amenable to interpretation. The combination of the two measurements has the proper ingredients of a rounded program.

Surface and Physical Properties of Water-Borne Coatings

PRINCIPAL:

Dr. Henry P. Schreiber, Ecole Polytechnique.

ABSTRACT:

Copolymers do not always produce films of uniform cross section. Surface tension indicates whether a particular group is oriented outward and reveals if formulation variables change this orientation and consequent wettability.

RELEVANCE:

Polar polymers used in water-borne coatings will most likely orient. If exploited, orientation could enhance adhesion and reduce water resistance of cured film.

COMMENTS:

This grant derives its utility from what it reveals during the aging of a film, as contrasted with the preceding study of what happens during film formation. By observing the spreading areas of liquids of various surface tension, rather than their contact angles, one can extrapolate to the critical surface tension of the film, γ_c.

The film formation process affects the γ_c initially read by this device. The two standard copolymers differed by less than 10% (3.7 dynes/cm out of 45), with the lower acid number polymer displaying the lower γ_c. This trend is expected when one decreases the number of polar groups.

A significant ancillary measurement was made in the form of intrinsic viscosities of solutions of the polymer in various mixtures of t-butanol and water. It showed that 70% water gave the greatest intrinsic viscosity and implied the greatest extension of the molecule. This was unexpected.

Unfortunately, the two measurements are not relatable. The value of γ_c did not depend on the composition of the solvent, and the trend in intrinsic viscosity was not particularly strong. There was evidence of an abrupt decrease in the molecular size on progressing from 30 to 35% cosolvent, and this shrinkage may confound the situation which exists in film formation.

Casting out of chloroform results in a lowered γ_c. This is an energetically favorable trend that allows one to declare, as Dr. Schreiber did, that any films which display larger γ_c values would change with time.

Earlier studies, reported before the standard copolymer became available, encountered small increases in γ_c on aging. These reverse trends have not been explained other than to surmise that surface oxidation had taken place.

Studies on the polymer-substrate interface are planned in order to reveal what segments of the polymer migrate to the metal-coating interface. Barrier properties will also be studied, as will the morphology as revealed by electron microscopy.

HIGH SOLIDS

Rheology of Concentrated Oligomer Solutions

PRINCIPAL:

Dr. Zeno W. Wicks, Jr., North Dakota State University.

ABSTRACT:

Selection of a model system containing terminal carboxyl groups will be made after preparation difficulties are resolved. Thereafter, characterization of the oligomers will be performed, with emphasis on application and curing of highly concentrated solutions.

RELEVANCE:

Insufficient information on the physical chemistry of low molecular weight polymers (oligomers) requires a standard preparation upon which various measurements will be made, starting with rheology.

COMMENTS:

This grant started on March 1, 1980. The first progress report emphasized the necessity of considering the glass transition temperature, Tg, in selecting the model. The rate of increase in viscosity η with concentration c depends on Tg in that log η does not long retain a linear dependence on concentration, and the point at which the knee appears shifts downward as Tg is approached.

Any factor that increases the polymer coil diameter renders it more difficult to stave off steep viscosity increases at high concentrations. Backbone rigidity, absence of branching, electrostatic repulsions of the type studied in the aqueous program, and steric hindrances would lead the list.

Butyl methacrylate was the intended polymer, to be made at a degree of polymerization below 17 by terminating the reaction by alcohol. The Tg of the resulting oligomer would be high enough to be practical, yet the value would permit considerable latitude in concentration before viscosity would get out of hand. Polymerization was not achieved, and the decision was reached to use methyl methacrylate which had been reported in the literature in oligomeric form. The product had a $\overline{M}_w$ of 760 and will be used in a study of the effect of different solvents on relative viscosity. This work will be done before a decision is reached on what should be the model compound. A certain amount of carboxyl termination is needed after the polymer backbone is made.

NEW DIRECTIONS IN RESEARCH

MILDEW

The mildew consortium is performing so well that the format will continue with outside support guaranteed until October 1, 1981.

There are seven supporters and a steering committee of nine (a representative from each supporter, Program Manager Yeager, and the PRI Research Director). During the past year quarterly meetings were held which resulted in a test protocol for the anchored fungicide copolymers and their latex formulations.

If additional support can be generated the committee will authorize expansion of the program, either in one of the directions recommended by Dr. Yeager, or along alternate routes such as extensive field testing of systems capable of controlled release upon hydrolysis. The laboratory test procedure is ready for round-robin evaluation by a technical committee.

I have recommended a definitive study of the nutritional requirements of mildew. I doubt that Ap assimilates pollen and wonder how much depolymerization is required in order for the extract to serve as nutrient. Since accidental nutrients will always plague us, we should know what constitutes bitesized chunks.

We expect, that in laboratories of most mildewcide producers, research is under way to incorporate fugitive species into film-forming polymers. The more candidates we can try the better off will be the coatings industry.

A plan is being formulated by John Oates, of the Mildew Consortium Steering Committee, whereby cooperation of Constituent Societies can be achieved through the Federation's Technical Advisory Committee, chaired by Colin Penny. Testing of various materials may not be the main effort of the TAC, for there is much synthetic work to be done, and there is need for assistance in formulating after certain polymers have been prepared. Not all monomers lend themselves to copolymerization as latexes.

AQUEOUS PROGRAM

Selection of Project Manager

Air-dry aqueous coatings differ from baking enamels. A project manager will be selected who can clarify which route to take, for our chosen standard copolymer fits only the baking enamel category. A different model must be selected for entry into the air-dry or even the forced dry domain.

Research by Prospectus

Research proposals will be solicited based on the prospectus approved by the Trustees. Currently, the two projects are moving along paths suggested by the steering committee. These paths may change, as indicated by the prospectus which is appended.

HIGH SOLIDS

An encounter session has been scheduled for December 18, 1980 in Kent, Ohio between three coatings scientists and a group made of physical scientists, suppliers, and users.

The purpose of encounter sessions is to arrive at a research prospectus which outlines, in terms acceptable to both sides, what research is needed. Participation by others outside the encounter group is not a vital feature, for there is no attempt at brainstorming. Nevertheless, occasional comments from others have been found useful, and therefore, a small audience is considered desirable.

The composition of the encounter group is:

Coatings Scientists

Joseph A. Antonelli, duPont Marshall Laboratory, Philadelphia, PA

Donald Emch, Valspar Corp., Minneapolis, MN

Garmond G. Schurr, Sherwin-Williams Co., Chicago, IL

Outside Experts
R. Drew Baker, Reichhold Chemicals, Inc., Detroit, MI
Harry Frisch, SUNY at Albany, NY
Robert M. Lukes, General Electric, Louisville, KY
Zeno W. Wicks, North Dakota State Univ., Fargo, ND

Moderator
Raymond R. Myers, Paint Research Institute

CORROSION CONTROL

Having declared on recent occasions that our attempt to make inroads on corrosion control was a failure, I can report positively on the step that was authorized by the Trustees at the July meeting.

With the earth-to-earth syndrome constantly reminding us that corrosion control will at best be a delaying action, we commissioned a critical survey on the subject, to be written by Dr. Henry Leidheiser, of Lehigh University.

The characteristics of a critical survey, as envisioned in my report to the NSF on how a coatings research institute would operate, are as follows:

(1) It would be slanted to the coatings chemist as the intended reader.
(2) It would contain input from interested and knowledgeable coatings scientists.
(3) It would be written by an individual who knows the field.

These ingredients are present in the critical survey commissioned under the tentative title: "The Mechanism of Corrosion-Inhibition Via Inhibitors in Organic Protective Coatings."

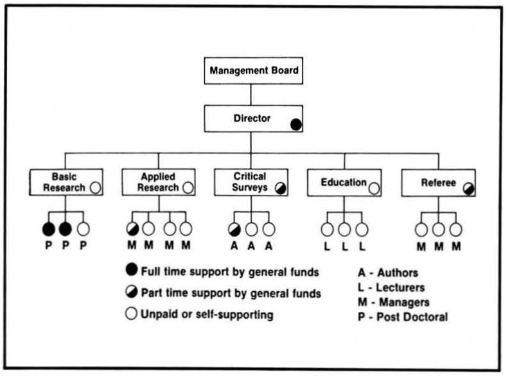

Figure 1—Coatings research institute table of organization (exit plan)

Other Services

LONG-RANGE PLAN

Results of NSF Grant

My report to the NSF contained some recommendations that were followed, in practice, by the Trustees in the July meeting. The recommendations are summarized in *Figure* 1, the exit plan for an expanded coatings research institute aided by a second NSF grant.

Figure 1 is not yet operational, for no application has been made for the supplementary grant. There is no evidence that the Federation will support a plan to enlarge PRI with the aid of federal funds. The Federation can support only a limited effort and all signs point toward the National Paint and Coatings Association not being ready for cooperative research. Normally, this situation would mean curtains for an expanded PRI effort; however, in this case, we have learned that the NPCA is considering a cooperative effort being organized by their Scientific Committee.

If significant support materializes, it will increase the pressures to engage in more visible activities than fundamental research. But there has to be a *quid pro quo*: practical projects require a more extensive commitment than does basic research.

One must assume that the more extensive commitment will not be forthcoming in the realm of equipment and personnel. PRI must continue to operate with contracts, grants, and commissions, using funds derived from the Federation and others. Some funds will be for specific efforts, which our shift to program managers is meant to emphasize.

Cautious Expansion of Effort

With minimal expense, PRI can engage in research-related services. Applied research may eventually become the main consideration, for there are few adherents to basic research these days. Critical surveys and education have now become part of the PRI portfolio. As long as the original PRI charter prevails, the block represented by "Referee" in *Figure* 1 is not transferrable to the PRI Table of Organization.

Other blocks are transferrable, as our moves into critical surveys and computer education testify. This cautious expansion of our domain should increase PRI's visibility and bring, in general, funds for the entire operation.

Plans for Phase II

The NSF grant was Phase I of a proposed two-part plan to supplement Federation funds invested in cooperative research. Unlike other government agencies NSF does not impose on grantee institutions. Their program on basic research is especially lenient and served as a model for the way PRI operated for 15 years. The NSF program in Industry-University cooperation is a bit more demanding of what kind of format they envision, possibly because of their third-party role. Since PRI can speak with a single voice representing both industry and a university, the problem of format can be resolved without complications.

A meeting of industry leaders who may cooperate in a second phase grant is in the planning stages.

In passing, it is worth mention that generic technology centers have been established by the Department of Commerce for research in powder metallurgy, welding and joining, and friction, wear, and lubrication.

The Technology Innovation Act of 1980 provides $169 million to industry over a five-year period. Unless more champions appear from the coatings sector, none of this appropriation will accrue to the PRI. In case anyone feels that NSF contacts are of questionable value, there is another move afoot to create a National Technology Foundation and place the NSF program, which supported our study, under the aegis of the NTF.

COMPUTER SOFTWARE

Trustee action at the July meeting initiated a new PRI service represented by the Education block of *Figure* 1. A committee consisting of Theodore Provder, Robert Bailey, and Colin Penny was formed to advise me on how to reach smaller paint companies on the subject of computer usage in research and production.

Their advice is to start with a questionnaire. This has been done, and a first version was submitted to the Trustees. Interested readers who did not receive a questionnaire are invited to write to me for a copy.

1981 SYMPOSIUM

Personnel have been contacted to appear at the 1981 Symposium on Stability and Stabilization of Coatings Systems. Dr. Richard Ruch, of Kent State University, will be the lead-off speaker, followed by Dr. Seymore Hochberg in the first session, which is tutorial. Succeeding sessions will be devoted to practical problems and an outline of needed research.

EMPLOYMENT OF PRI FELLOWS

We received superb cooperation from former PRI principal investigators in our effort to locate their PRI fellows.

Of the 105 fellows listed, plus 17 auxiliary personnel who were introduced to coatings via PRI grants, 50 are employed in the coatings industry. Forty-seven more have positions that are at least peripheral to coatings, counting 25 who have joined university faculties. See Appendix B.

This listing bears testimony that the PRI exposure improves the bias for graduate students to undertake careers related to coatings. It will be maintained on a biennial basis.

APPENDIX A

AQUEOUS COATINGS

Experimental Program
On the Physical Chemistry
Of Ion-Containing Polymers

OBJECTIVES

Supply basic knowledge of ion-containing polymers that will enable one to control their solution viscosities, their responses to humidity variations, and their mechanical properties. Understand the role of solubilizing agents and their effect on coatings application and film formation.

Explanation

One class of aqueous coatings is based on partial solubilization of the polymer by pendant groups such as COOH and OH. This technology, unlike that of latexes, leads to formulations that have high viscosity at low molecular weights. These characteristics and the sensitivity of polar groups to water are responsible for application difficulties and for unsatisfactory performance of the dried films under certain circumstances. High viscosity limits solids loading, hinders pumping, and affects application.

These characteristics arise from various sources, most of which involve the molecular structure or morphology of the polymer. Extension of the macromolecule by repulsion of ionic charges produced by neutralization accounts for some of the various effects. The presence of a molecule made up of a hydrophobic backbone is considered to be an important consideration, for molecular association displays many of the effects of crosslinking. Confounding the issue of what interactions are involved, is the ubiquitous presence of hydrogen bonds.

Basic research is needed to help the formulator cope with problems encountered in storing, pumping, pigment wetting, spraying, defoaming, leveling, drying, curing, and protecting surfaces. Idealized studies on model systems and on solutions at infinite dilution partially address these questions; but the essential feature of PRI's approach to the problem is to apply the results of model studies to the imperfect systems encountered in practice.

REASONS FOR AQUEOUS COATINGS RESEARCH

Potential Advantages

Ecology and the substitution of an economical solvent for organic solvents are the chief motivations for the use of aqueous systems. These advantages, plus the reduction in toxicity and flammability that accompany the use of water in place of organic diluents, were responsible for the acceptance of latexes, whose use in coatings preceded the introduction of ion-containing polymer formulations.

Table 1—Potential Advantages of Hydrophilic Polymers vs Latexes

Benefit	Reason
Freeze-thaw stability	Solution involved
Fewer MFT problems	Water plasticization
Better coalescence	No discrete phases
Less need for surfactant	No phases
Wetting of pigment by polymer	No separate water phases
Better gloss	No interstices
Higher pigment loading	100% mobile phase
Need for less cosolvent	Lower M.W.
Better adhesion	Functional groups

Advantages of Ion-Containing Polymers

Possible advantages of hydrophilic polymers over latexes are listed in *Table* 1. Not all of these advantages have been realized, of course; but all of the listed ones have a basis in theory, if not in fact.

These advantages supply the reasons for interest in hydrophilic polymer coatings. They contain insufficient background to justify a research program in that restricted area, however. There is no guarantee that this type of formulation will capture the segments of the market for which it is targeted. Some of the research that is needed in order to enable these interesting formulations to capture additional markets is listed in *Table* 2.

Table 2—Problems Associated With Hydrophilic Polymers

Limitation	Reason
During Formulation	
Restricted to low molecular weight	Solution rheology prevails
Volatile or fugitive solubilizer is needed	Polymer must be an electrolyte at first, not later
Synthesis has fewer degrees of freedom	Copolymer must be synthesized
During Application	
Electrostatic spraying is difficult	Charge is dissipated
High-speed application is difficult	Solutions are long and stringy
Viscosity is too high	Solution viscosity, coupled with association
Foam control is difficult	Polymer is surfactant
Thermal effects are significant	Viscosity and rigidity drop on heating
During Film Formation	
Relative humidity affects composition and film quality	Competitive evaporation rates vs cosolvents
Surface defects abound	Surface activity and high viscosity
Drying is retarded	Polymer is a humectant
Flexibility/hardness balance is elusive	Plasticization depends on numerous factors
Solvent retention is high; scrubbability is poor	Water sensitivity is high

Research Needs Involving Hydrophilic Polymers

Problems appeared with acrylic acid copolymer systems during attempts to apply them with conventional equipment. Their forerunners had worked in electrodeposition baths where the negative charge on the molecule was employed as a handle for deposition; but extension to spray systems and particularly to architectural coatings has not met with success.

In *Table* 2 the problems are listed according to whether they appear to be most prominent in the formulation, the application, or the drying and subsequent service of the coating.

BASIC STUDIES

The PRI program on the physical chemistry of aqueous coatings has four facets: Physical Chemical Principles; Mechanical Properties; Drying Behavior; and Blue Sky Approaches. Priorities have emerged during the course of this work, so that elucidation of the physical chemistry has become the most important item from the viewpoints of the principal investigators, whereas immediate application of these principles is the aim of the PRI. This proposal establishes the linkage between the two.

Physical Chemical Principles

Surface chemistry underlies the pigment wetting, adhesion, and defoaming referred to above. Our current approach is to measure the critical surface tension of dried films which underlies those properties which depend on the free surface of the dried film (in some cases the interface could be measured). Approaches responsive to industry's needs would concern themselves with high-speed application and foam control, in addition to quiescent properties of the free surface. Therefore, surface chemistry of formulations should be conducted dynamically; and, of films, in connection with other measures of performance.

Solubility is mainly a thermodynamic problem, tempered by the need to consider specific interactions, acid-base theory, and electrostatics in the case of ion-containing polymers. The academic investigator would elucidate the molecular conformations involved when formulations or drying conditions are changed; the industrial sponsor would prescribe the changes. In this collaboration, there should be no difficulty in getting together. Specific interactions between carboxyl and metal ions of differing charge density would be studied; proton acidity, also.

Dielectric properties are of direct practical interest as well as of academic interest. The problem in mounting a research offensive consists of how to measure them in aqueous systems, especially when they are spread out in a thin coating. Almost any rational measurement of electrical properties should reap a windfall of information of direct bearing on electrostatic spraying difficulties, solvent retention, and relative humidity effects. The massive polarization that occurs at an oil/water interface should provide a probe of the interesting borderline between solution and colloid behavior that occurs on dilution of these systems and the similar transition that evidently occurs on drying.

For proper performance as a film, water sensitivity must be lost by the polymer on curing. Critical surface tensions monitor this development only to a degree and only after the film has cured. Other measurements, emphasizing conditions at the interface, are needed. One such measurement (the Impedometer) is in use; it has not attracted wide attention. What appears to be needed is a way to formulate for water insensitivity, allowing each sponsor to decide how to measure it. Formulation principles can be studied according to the PRI charter, but not formulation trials.

Mechanical Properties

Industry has asked for concepts that will enable one to minimize viscosity on dilution. This goal may be realized by increasing the concentration at which a given viscosity level is reached. The steering committee for the aqueous program suggests decreased molecular weights, changed degree of neutralization, and other cosolvents (including reactive monomer). They also request a study of relative humidity effects, performed in conjunction with our systematic study of amine neutralizers.

The role of the solubilizing agent in affecting the system's mechanical properties should be clarified. These properties include the viscosity of the resin solutions and pigmented systems made therefrom, the gelation tendencies of the film and rigidities of the gel state containing various plasticizers and various amounts of plasticizer, especially water. Relation of rigidity to extensibility should be clarified.

In general, mechanical properties should be more clearly defined or prescribed. Films of highest practical rigidity are desired, yet attainment of high rigidity leads to poor extensibility and poor adhesion. Extensibility measurements require unsupported films, while adhesion measurements require a substrate. Differences in behavior between supported and unsupported films should be clarified.

The effect of MW and MWD should be investigated, especially in regard to how they control flow and leveling during application and how they determine the mechanical properties of the applied film. Because the critical surface tension measurements have shown that changes occur at the molecular level on aging, it would be of interest to learn if smaller oligomers migrate to one of the interfaces on aging. Migration would affect the free surface appearance and it would weaken the adhesion to the substrate, depending on which direction was taken.

The one area in which the coatings industry may have to dictate the direction of the research is in the study of pigmentation effects. Academic people are not likely to compound the handicap of working with a non-ideal polymer system by pigmenting this same system and reporting on interactions at the molecular level. It will be necessary to deal with materials-oriented scientists who willingly study systems of uncontrolled composition and adjust their sights to accept results that are not expressed in rational terms. Rheologists have done this for years. Not many of their papers appear in physical chemical journals, but practical solutions to compounding problems have resulted. Transport phenomena certainly can be studied in practical pigmented systems. Electrical properties, perhaps not.

Drying Behavior

Since the days of the Gardner drying time recorder, the coatings industry has needed to standardize on the best available means to measure drying. This method should provide information on the changing mechanical properties of the coating as it dries, and it certainly should supply more information than drying time.

Why our Impedometer has not enjoyed more popularity has escaped me. It is the workhorse of our operation, even more so than our Torsional Braid Analyzer which does enjoy wide acceptance. Both of these devices emerged as a direct result of the need to measure viscoelastic properties of fluids and soft solids while they were supported by a substrate of far overpowering mechanical properties. Both devices measure transitions of the type encountered in drying and curing. The Impedometer has the added advantage of detecting adhesive failure long before any noticeable effects of that failure have been observed, such as blistering or reactions involving the substrate.

My first recommendation is that a committee of industrial scientists select the best method for measuring drying behavior, based on their conception of what are the relevant questions to be asked about film formation. Their recommendations should be responsible for commercializing a version of the instrument selected. Volunteer evaluation of the drying behavior of a standard copolymer should then be conducted in various laboratories.

The actual study of drying behavior starts with relative humidity effects. The steering committee for the aqueous program recognized these as the most urgent ones to be investigated because of problems encountered on production lines where aqueous coatings are in use. Sagging occurs when the relative humidity is high, and popping spoils the film when the humidity is low. Air conditioning of the plant is an unsatisfactory solution of the problem.

Related to the humidity question is the effect of varying the degree of neutralization. Work soon to be published by us shows that the drying behavior of a given polymer, as a function of its degree of neutralization, is determined by the structure of the amine used to neutralize it. Our explanation of the differences in behavior is based on hygroscopicity.

Naturally, the acid value of the copolymer interacts as a variable, as does the ionic strength of the medium. Early results with minor variations in cosolvent indicate that major changes in cosolvent should be subjected to systematic study.

One "far-out" suggestion resulting from our work concerns the positioning of the carboxyl groups. If our hypothesis is correct, one would avoid situations in which more than two carboxylates can form next to each other. It is not necessary to accept this deduction, nor even to understand it, in order to see how basic research can impinge on the problem of formulation without becoming involved in the proprietary side of the industry. In this case, in fact, one would make a copolymer with the intention of proving it to be inferior. This would be more rewarding to the academic person than going after the intentionally superior product.

Rates become important when practical solutions to drying problems are sought. In the case of aqueous coatings the rate question involves a differential in rate, as well. The cosolvent/water ratio that is maintained during drying determines the viscosity trends of the solution, the onset of gelation, accommodations by the macromolecules after gelation, and the porosity of the final film. These features supplement the main objective in controlling evaporation rates, which is to dry the film in the shortest possible time with an optimum (or minimum) of applied energy.

Combination Approaches

Recognizing that the valuable features of latexes may be retained in formulations containing hydrophilic polymers, one should remain alert for combinations of the two technologies that show promise of being synergistic.

One possible approach has been suggested in connection with PRI research. Several years ago we recommended soft-sheathing of hard particles as a means of improving the mechanical properties of latex films. This idea worked, for most of the distortion during coalescence was confined to the soft sheaths.

A new thought is presented in connection with the aqueous program: sheaths can be made of hydrophilic polymer, the latex stabilized by the changes imparted on neutralization, the coalescence enhanced by gradual loss of the neutralizer, and final attainment of water insensitivity achieved by diffusion of the occasional carboxyl groups through the main body of the polymer through the well-established mechanism of further gradual coalescence.

Another combination approach, suggested by Marco Wismer, is to study means of stabilizing a system against viscosity drift. Such a study could be something as simple as repeated measurement of viscosity and hysteresis loops in a rotational viscometer or it would involve a non-destructive measurement of viscosity while the system is undergoing shear at various levels. There are expensive rheometers on the market that will make this measurement.

THE STANDARD SYSTEM

The first four investigators in the aqueous program worked with a standard copolymer of acrylic acid (7%) and butyl acrylate. A 75% solution in t-butanol was prepared by Loren Hill, formerly of NDSU, and supplied to the participants.

I see no need to depart from this formulation. Acid numbers from 35 to 70 and molecular weights from 10M to 70M are reasonable; the only negative feature appears to be the wide range of molecular weights ($M_w / M_n > 2$).

Variations from the standard system would be made by the individual investigator, although it would be preferable to commission one individual at NDSU or elsewhere to handle all of the syntheses. Some variations would require the synthesis of terpolymers, and all preparations should be characterized by the same individual.

Experimental design of the polymer series suggested by James Erickson adds hydroxyl groups to alter the hydrophilicity and takes into consideration the resulting polymer's Tg. Monomer-starved conditions would promote high instantaneous percentage conversion.

In order to base a complete program on a standard system, it is necessary to make significant characterizations of the system and to outline a meaningful program of research on the performance of the system and the variations based on it. The measurements currently employed consist of GPC determinations of molecular weight. In addition, one needs data on adhesion and other measures of coatings performance. While this need is reflected in the proposed research program, there is room in the program under the category of preliminary characterization of the polymer for test data. How do zero shear measurements relate to sag, brush mark leveling, gloss, and even to corrosion? What about rewetting? On residual stress development by the polymer on drying? Contributions in the area of test methods are urgently needed according to Marco Wismer; a program of this nature requires a standard system.

According to James Erickson, the problem with the waterborne program from the start has been the use of only one analytical technique to characterize the standard system. John Poist feels the same way. To elucidate the chemical-physical behavior of the polymer, it is mandatory that a more comprehensive program be developed for characterizing it. I classify these characteristics under morphology, and include MWD, crystallinity, branching, crosslink density, tacticity, head-to-head vs head-to-tail linkage, and (for good measure) the chemical functionality. All of these parameters influence the way the polymer will coil up when treated with solubilizers, cosolvents, and water.

Among the degrees of freedom that one has are monomer ratio, randomness, and block and graft possibilities. In waterborne coatings, one also can vary the electrostatic charges on the molecule, the hydrophicity of the polymer, and the acidity of the functional group.

All of these considerations must be decided by a steering committee for the project.

A communication from Marco Wismer suggests that some of the studies should include models with external crosslinkers such as Cymel 303 in the amount of 25% molar excess over hydroxyl group (in cases in which the polymer backbone contains hydroxyl).

APPENDIX B

PRI FELLOWS EMPLOYED IN INDUSTRY

R. L. Abrams—Columbus Coated Fabrics Div. of Borden Chemical Co., Columbus, OH

Pamela J. Bennett—Editor, Indiana Historical Society, Indianapolis, IN
S. Beret—Union Carbide Corp., S. Charleston, WV
K. J. Bhansali—Cabot Corp., Stellite Div., Kokomo, IN
D. C. Bonner—Shell Development Co., Houston, TX

G. D. Cheever—General Motors Res. Ctr., Warren, MI
S. Chiang—Fiberglas Canada Ltd., Sarnia, Ontario
Yohannes Chonde—Diamond Shamrock Corp., Painesville, OH
J. Cinkler—A Corrosion Research Lab in Yugoslavia
P. Clark—Dow Chemical Co.
Richard Cooper—Foster Grant Co.
Marshall H. Cohen—Res. Chemist, Polaroid Corp., Brockton, MA
George McNeny Crews—Melamine Chem. Corp., Donnalson, LA

J. G. Davidson—Macbeth Div. of Kollmorgen Corp., Newburgh, NY
James S. Dodge—B.F. Goodrich, Avon Lake, OH
R. G. Eager—Union Carbide, Tarrytown, NY
Richard R. Eley—Glidden-Durkee, Strongsville, OH
Hans Essler—Alexander Wacker, W. Germany

David P. Garner—GM Research Center, Warren, MI
P. Goetze—Raychem Corp., Palo Alto, CA
F. T. Golemba—Fiberglas Canada Ltd., Sarnia, Ontario

J. R. Hart—Champion International, Cattaraugus, NY
Kenneth Hartman—Allegheny Ballistics Lab
Donald Hunston—Naval Research Laboratory, Washington, DC
D. L. Hunt—DuPont Co., Waynesboro, VA

R. B. Jenkins—ICI, Runcorn, Cheshire, U.K.
H. K. Johnston—Tonecraft Ltd., Toronto, Ontario, Canada
Mike S.-D. Juang—Borg-Warner Corp., Des Plaines, IL

Sam Kalousdian—Central Research, 3M Company, St. Paul, MN
A. Kapadia—Girdler Corp.
L. K. Keys—Northern Bell Laboratories
John Klimek—Eberhard Faber

Leroy Lenhart—duPont Marshall Lab., Philadelphia, PA

Milton I. Levenberg—Abbott Laboratories, N. Chicago, IL
P. Li—Monsanto Chemical Co.
Li-Jen Liu—Cincinnati Milacron, Cincinnati, OH

Robert A. Meyers—Systems Group of TRW, Inc., Redondo Beach, CA
Richard Miller—Atco Performance & Chemicals, Houston, TX
D.F. Mitchell—National Research Council, Ottawa, Canada
Mohammed A. Mostafa—American Hoechst Corp., Leominster, MA

T. P. Nelson—Allied Chemical Co., Hopewell, VA
R. D. Newman—Gulf Oil Chemicals Co., Orange, TX
Tyan-Faung Niu—Xerox Corp., Webster, NY
M. Nozari—3M Co., St. Paul, MN

W. Olmsted—Englehardt Minerals Corp.

N. Patel—Standard Pressed Steel Co., Jenkintown, N. Philadelphia, PA
Robert Patterson—Texas Instruments, Dallas, TX
R. Penwell—Xerox Corp., Webster, NY
Klauss Plesske—Chemische Werke Albert, W. Germany
W. Prest—Xerox Corp., Webster, NY

Thomas Richert—Ferro Corp.
K. Raju—Cargill, Lynwood, CA
Richard Robison—Coatings Research, International Paper Co., Longview, WA

Madhu C. Samanta—PPG Paints, Toronto, Ontario
T. Serafini—NASA Lewis Research Ctr., Cleveland, OH
William Sherman—United Technology Corp., California
O. Smith—Union Carbide Corp.
J. Southern—Monsanto Co., Pensacola, FL
G. Allan Stahl—Phillips Petroleum, Bartlesville, OK

Koichi Tsutsui—Nippon Paint Co., Osaka, Japan

Harris Ulery—Jackson Lab, duPont

H. J. Wang—Standard Ultra Marine Co.
J. S. Wang—Monsanto Co., Pensacola, FL
L. Wang—Wilmington Enameling Co.
R. Wang—Wilmington Enameling Co.
C. Whitmarsh—Atomic Energy Commission
D. M. Williams—Foster Wheeler
P. W. T. Willmott—Ralph Symonds Pty., Ltd., Sydney, NSW Australia
David Wohleber—Alcoa
Martin E. Woods—Diamond Shamrock Corp., Painesville, OH
J. Wyhof—GAF Company

PRI FELLOWS IN ACADEME

E. G. Bell—Medical School at Syracuse, NY
Jesse Bernstein—High School teacher, Cleveland, OH
S. H. Bhattacharyya—Indian Assoc. Cultivation of Science, Calcutta, India
Graham Blake—Professor, Leicester Polytechnic, Leicester, England

Show-An Chen—Nat'l Tsing Hua U., Taiwan, Rep. of China
D. W. Cherry—Monash University, Victoria, Australia
Robin K. Cowling—School science teacher, Essex, U.K.

W. B. DeLauder—Dept. Chairman, N. Carolina A&T Univ., Greensboro, NC

G. Delmas—Assoc. Prof., Univ. of Quebec
A. P. deMendoza—Univ. of Mexico
C. S. Dickey—Lutheran High School, Cleveland, OH

N. Z. Erdi—Dentist

George E. Farley—Science teacher, Carlisle, PA

P. Grube—Mobile College, Alabama
M. Guidry—Seiler Lab., USAF Academy, Colorado

K. Harmon—Penn State
P. Anne Hiltner—Asst. Prof., Case Western Reserve Univ., Cleveland, OH

P. W. Jennings—Univ. of Montana

R. Kabler—U of Illinois, Urbana, IL
George Kennedy—Colorado School of Mines
Henryk Kolny—Acad. of Mining & Metallurgy, Krakow, Poland
F. Kosel—High school teacher, Philadelphia, PA

Walter E. Maust—Dentist, Hanover, PA

D. R. Owen—Assoc. Prof., Tulane University

Donald E. Padgett—U of N. Carolina, Wilmington, NC
F. H. Pinkerton—Carson Newman College, Johnston City, TN
N. Pisker-Trifunac—Med School, U of Southern California

Marc H. Schneider—Assoc. Prof., Coatings, U of New Brunswick, Canada
R. K. Schultz—Asst. Prof., Ursinus College
Leonard A. Smith—Coatings & Adhesives, SUNY, Syracuse, NY

K. Varadarajan—U of Illinois
G. C. Vogel—Ithaca College, NY

Helmut Werner—Inst. fur Anorganische Chemie, Wurzburg, W. Germany
Jerry W. Williams—U of Cincinnati

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Charles Chessher—Chemist, PPG Industries
Roy Christofel—Polymer chemist, Monsanto Chemical Co.
Anne Elting—Nalco Chemical
Jim Evans—Sylva Chem Corp., Panama City, FL
Isaac Hassid—Gulf Oil Co., Orange, TX
L. Earl Johnson—DuPont Co.
Climaco J. Losana—Polymer Chemist, Celanese, Bogota, Colombia
J. Sosa—Vulcan Chemical, Wichita, KS
Peter Tsang—Res. Group Leader, Bendix, Detroit MI
Ho Tung—Owner, Lacquer & Adhesives Concern, Kingston, Jamaica
Mike Yang—Industrial chemist, Houston, TX

PRI-RELATED GROUP IN ACADEME

Richard H. Albright—Dentist, Lancaster, PA
Hans-Peter Baer—Prof., U of Alberta, Edmonton, Alberta, Canada
S. C. Honeycutt—M.D., Gastonia, NC
Louise McGee—Chem instructor, San Jacinto College
K. Nihikawa—U of Tokyo
William Tinnerman—Prof., St. Thomas U, Houston, TX

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● red lead ● litharge ● battery oxides

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The history of the company began as far back as 1305 as the forefathers of the present owners founded a glassworks in the Thuringer forest. Porcelain was produced first in 1764, white lead in 1847. The works in East Germany were lost in 1945. Since that time the enterprise is existing in West Germany.

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HEUCOTRON Yellow 5

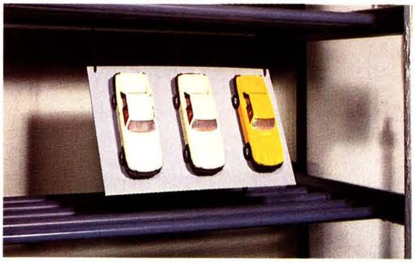
- ■ Less than 1 % Acid Soluble Lead
- ■ High SO_2-Resistance

Reference sample

1 cycle Kesternich

5 cycles Kesternich

10 cycles Kesternich



Less than 1 % Acid Soluble Lead
During recent years chrome yellow pigments have been partly substituted by organic yellow pigments as many authorities suspected the average content of 16 % acid soluble lead to be a potential hazard to human beings whereas there is no such danger with non-soluble lead and chrome combinations such as lead crystal or chrome steel.
HEUCOTRON-Yellow 5 is the first product of a newly developed generation of chromate pigments which contain hardly any acid soluble lead. According to the German DIN-Standard 55975 which simulates the solubility of lead in the human body, only 0.5 % acid soluble lead (less than 1 % is guaranteed) and no soluble chrome have been found in HEUCOTRON-Yellow 5.
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The test panels were uniformly coated with an alkyd-melamine resin varnish containing 20 % pigment and tested in accordance with DIN 50018 with 2 litres SO_2 in a Kesternich apparatus.
HEUCOTRON-Yellow 5 was compared with two high performance pigments from the highly and medium stabilized series in the same shade range:

HEUCOTRON-Yellow 1064 - medium stabilized
HEUCOTRON-Yellow 123 - highly stabilized
HEUCOTRON-Yellow 5 - SO_2-stabilized

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HEUCOTRON-Yellow	1064	123	5
SO_2 resistance	↓	↘	↑
Light fastness	↗	↑	↑
Weathering fastness	↗	↗	↑
Hue steps	↑	↗	↗

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THIS PAPER WAS AWARDED FOURTH PRIZE OF $200 IN THE 1980 ROON AWARDS COMPETITION

Synthesis of Blocked MDI Adducts, Their DSC Evaluation And Effect of Pigmentation

Taki Anagnostou
Wyandotte Paint Products Company*
and
Ernest Jaul
Union Carbide Corporation†

Blocked isocyanate compounds have been used in the coatings and related industries for many years. However, there is no convenient technique reported to indicate at which temperature level unblocking will occur. In addition, only with considerable experimentation can the effect additives and pigments have on this unblocking reaction be found. This study investigates the use of a differential scanning calorimeter (DSC) to designate this temperature range.

Three blocked versions of 4,4′-diphenyl methane diisocyanate (MDI) were synthesized. These were the adducts of methyl-ethyl ketoxime, ϵ-caprolactam, and benzotriazole. They were selected because they represented adducts which were capable of decomposing over a wide temperature range. Confirmation of this unblocking temperature by infrared (IR) spectroscopy techniques on samples baked at temperatures specified by the DSC studies was, in general, successful.

Changes were observed in the temperature range at which adduct unblocking occurs when a polymeric compound containing primary hydroxyl groups (polyoxyethylene glycol) is present. Addition of pigments to the mixture of blocked MDI adducts and polyoxyethylene glycol had minimal effect on unblocking of the adducts at the lower and medium temperature ranges. However, at high temperature ranges, significant changes were observed.

INTRODUCTION

The three blocking agents used in this study were specifically selected to unblock at low (methyl-ethyl ketoxime), medium (caprolactam), and high (benzotriazole) temperature levels. Their use as isocyanate blocking agents is known in the literature,[1] but the specific compounds of the 4,4′-diphenyl methane diisocyanate (MDI) blocked adducts have not been reported. For example, Frisch and co-workers[2] prepared and studied blocked MDI isocyanate terminated polyol prepolymers with ϵ-caprolactam and benzotriazole. However, they did not isolate and characterize the basic MDI adducts.

Blocked isocyanates are necessary to prepare stable, one package systems[1,3,4] and the knowledge for reactivating them is essential for their proper utilization. Numerous investigators[3,5,6] have evaluated techniques for determining the relative temperature range at which reactivation occurs. None, however, report the use of differential scanning calorimetry (DSC) to identify the temperature range at which unblocking can be expected to occur. If this technique were to prove successful, one simple scan for each blocked MDI combination could evaluate the effect of reactive diluents, copolymers, additives, solvents, and pigments. The need for such information, considering the chemistry of blocked isocyanates, is essential. Addition of a reactive polyol can reduce the unblocking temperature, as is shown by Griffin and Willwerth.[5] They determined that ϵ-caprolactam blocked toluene diisocyanate unblocks at about 125° C in paraffin oil, but, when a reactive polyol is added, the unblocking decreases to 108° C. This same study also revealed that the presence of solvent can affect the unblocking reaction. Hill[7] reviews this aspect of blocked isocyanate-containing one package systems. He points out that some solvents can actively participate in

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the unblocking process, thus leading to package instability, and suggests solvents which aid in the deprotonation of the blocked isocyanate would be expected to decrease stability. The importance of this phase of formulation is frequently overlooked since, in most coating compositions, the solvent is removed from the film prior to the filming reaction.

Finally, the catalytic activity of various compounds toward the isocyanate-hydroxyl reaction is well known.[8-10] The review of catalysts in isocyanate reactions by Frisch and Rumao[11] is especially helpful because it points out that these compounds play a role in polymerization of the isocyanate monomers to dimers and trimers in addition to the normal hydroxyl reaction. Therefore, the addition of pigments such as titanium dioxide, zinc oxide, calcium carbonate, etc., can have a profound effect, not only on the unblocking of the isocyanate, but also on the basic isocyanate hydroxyl reaction.

SYNTHESIS OF BLOCKED ADDUCTS

The chemicals used in the preparation of the adducts are listed in *Table* 1. Polyoxyethylene glycol was used and the molecular weight was determined to be 400 by hydroxyl value analytical techniques (ASTM-E326).

In the preparation of the adducts, equivalent quantities of active hydrogen reagent and NCO were used. Because of its tendency to dimerize or trimerize, the isocyanate MDI was analyzed prior to its use for percent available NCO groups. Based on this analysis and the molecular weight of the proton-containing compound, adjusted amounts were used. The MDI according to analysis[12] had a purity of 92.6%.

Caprolactam Adduct

The caprolactam was obtained as the 99% pure reactive agent. Caprolactam (2 moles) was reacted with 1 mole of MDI according to the following:

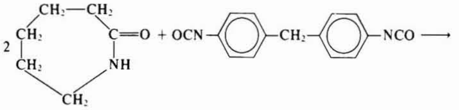
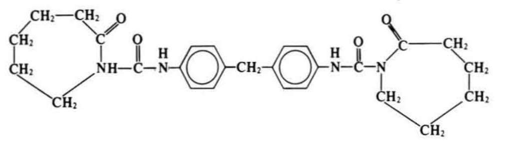


Table 1—Chemicals Used in the Preparation of Blocked MDI Adducts

Chemical Composition	Trade Name Grade	Source	Properties
4,4′ diphenyl methane diisocyanate	Multrathane M (MDI)—reagent	Mobay Chemical Co. Pittsburgh, PA	Avg. equ. wt. 125 Sp. gravity 1.9 Recrystallizing point min. 37°C 99% purity
hexahydro 2H azepin 2-one (ϵ-caprolactam)	ϵ-Caprolactam—reagent	Aldrich Chemical Corp. Inc. St. Paul, MN	99% purity, b.p./50 180°C m.p. 70°C mol. wt. 113.16[b]
(dibutyltin dilaurate) dibutyl bis (1-oxododecyl) oxystannane	T-12—Reagent	M&T Chemical Corp. Sub. of American Co. Rahway, NJ	tin (17.5–18.5%) pale yellow liquid mol. wt. 631.55[b]
2-methoxy ethyl acetate	Methyl Cellosolve acetate (urethane)	Union Carbide Corp. S. Charleston, WV	purity 99%[a] Sp. gravity 20°/20°C 1.0067 b.p. 145°C
Ethyl acetate	Ethyl acetate (urethane)	Union Carbide Corp. S. Charleston, WV	Mol. Wt. 88.11[a] Sp. gravity 20°/20°C 0.9018 b.p. 77.2°C
Benzotriazole	Cobratec 99—reagent flake X18BT 5566	Sherwin Williams Co. Cleveland, OH	99% purity[b] m.p. 98–99°C mol. wt. 119.13
2-butoxy ethyl acetate	Butyl Cellosolve acetate (urethane)	Union Carbide Corp. S. Charleston, WV	purity 99%[a] Sp. gravity 20°/20°C 0.9424 b.p. 192.3
Methyl-ethyl ketoxime	Methyl-ethyl ketoxime—Reagent	Dow Chemical Corp. Midland, MI	b.p. 152–153°C, purity 99%
Polyoxyethylene glycol 400 mol. wt.	Carbowax 400 (Commercial)	Union Carbide Corp. S. Charleston, WV	mol. wt. range 380–420[a] Sp. gravity 20°/20°C 1.1281 m.p. 4–8°C viscosity cst 7.3 at 100°C
1,4,-Diaza (2,2,2) bicyclo-octane (triethylene diamine)	Dabco	Houdry Chemical, Div. of Air Products Corp.	

(a) Chemicals & Physical Properties, 1979–80 Edition.
(b) Merck Index, 9th Edition, p. 1121.



Caprolactam 1.77 moles (200 g) and MDI $\frac{(1.77)}{(\ 2\ )}$ = .885 moles. $\left[0.885 \times \frac{250}{0.926} = 238.9\ \mathrm{g}\right]$ were reacted in the following manner:

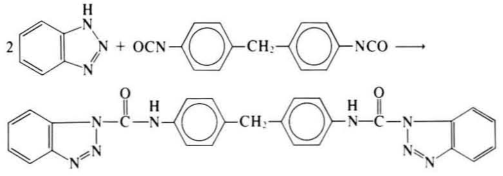
A 25% solution of MDI (239 g) in methyl Cellosolve® acetate (urethane grade) was added dropwise to a 50% solution of ϵ-caprolactam (200 g) in methyl Cellosolve acetate. The addition was carried out in a three-liter resin flask equipped with a stirrer, reflux condenser, dropping funnel, thermometer well, heating jacket, and an inlet tube to carry dry nitrogen to keep an inert atmosphere throughout the reaction. Dibutyltin dilaurate (0.005% based on ϵ-caprolactam) was used as catalyst. The gradual addition of the isocyanate was started at 23° C. During the addition process, mild heat was applied. When the mixture reached 65° C, a white precipitate started to form. Continual addition of the isocyanate mixture gradually raised the temperature to 80° C. When the addition was completed, the mixture was kept between 80–85° C for an additional two hours to insure completion of reaction. The white precipitate that formed was filtered using suction and the material recrystallized from ethyl acetate. The melting point of the product, using the Fisher-Johns apparatus, was 177–180° C. The blocked compound was carbon, hydrogen, and nitrogen, analyzed using the Perkin Elmer Model PE-240 Elemental Analyzer for determination of the elements.

	C	H	N
Theoretical, %	68.06	6.72	11.76
Actual, %	67.12	6.88	11.25

The melt point compares favorably with the one listed by Iwakura and Hayaski,[13] who synthesized this adduct and reported a melting of 172–174° C for this compound.

Benzotriazole Adduct

Benzotriazole (2 moles) was reacted with 1 mole of MDI according to the following:



$\left[\ 1 \times \frac{250}{.926} = 270\ \mathrm{g}\ \right]$ were reacted in the following manner:

A 50% solution of MDI (270 g) in methyl Cellosolve acetate was added dropwise to a 25% solution of benzotriazole (238 g). The addition was carried out in a three-liter resin flask equipped with a stirrer, reflux condenser, thermometer well, heating jacket, and an inlet tube to supply dry nitrogen to keep an inert atmosphere during the reaction. The reaction took place with such ease that no catalyst was used; however, a small amount of heat was applied to maintain 75–80° C throughout the 135 minute reaction period.

After nearly one-third of the isocyanate was added, the mixture became so bulky that the product had to be removed before the reaction could be continued. This procedure was repeated twice before all of the isocyanate monomer could be added.

The white precipitate was washed with ethyl acetate but not recrystallized, because a suitable solvent media could not be found. Ethyl acetate, butyl Cellosolve acetate, methyl Cellosolve acetate, toluene, dichlorobenzene, trichlorobenzene and acetone were evaluated.

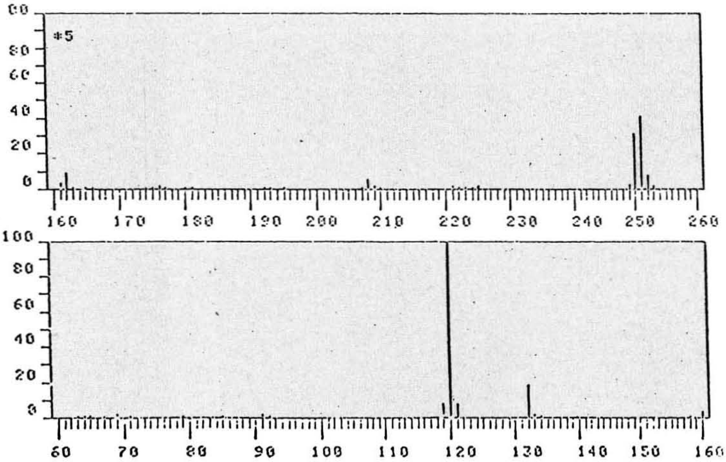
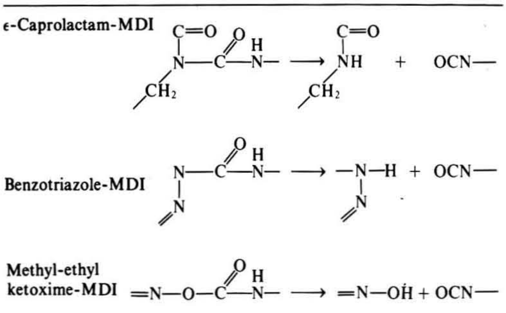


Figure 1—Structural analysis of benzotriazole-MDI adduct (probe 250° C)

Table 2—MDI—Adduct Unblocking Reaction Sites



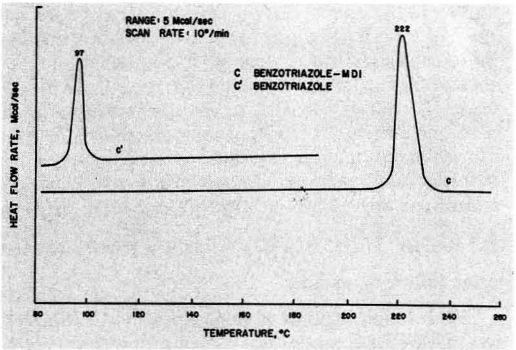


Figure 3—DSC scans of MDI adducts and the corresponding monomeric compound

All failed to properly dissolve the precipitate. The compound was soluble in hot dimethyl formamide; all efforts to crystallize the compound from the hot solution failed.

The Fisher-Johns melting point determination indicated sublimation began at 218°C with no clear melt occurring. The adduct elemental analysis yielded the following:

	C	H	N
Theoretical, %	66.39	4.09	22.95
Actual, %	66.24	4.14	22.79

Again, a review of the literature produced no references of a prior synthesis of this adduct. Thus, a comparison of melt performance could not be made.

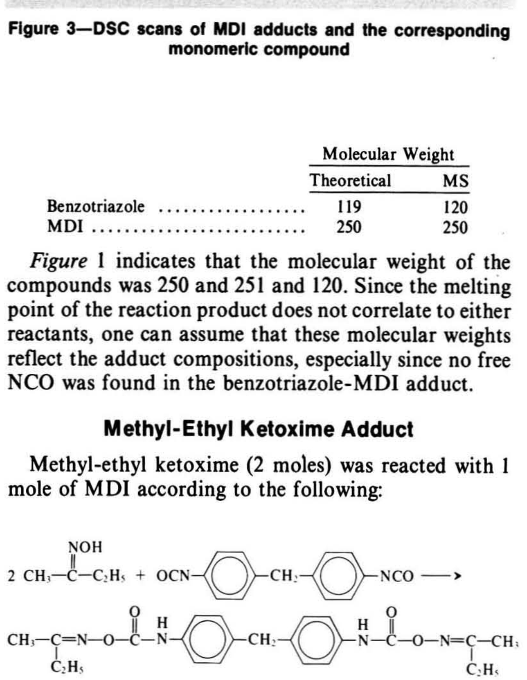
The structure of this adduct using the AEI Chemical Ionization Mass Spectrometer Model 3074 was further substantiated (*Figure* 1). The Probe analysis was carried out at 250°C. At this temperature, it was theorized that the adduct would split into its individual components. If this were the case, the Mass Spectrogram should identify two compounds of molecular weight 119 (benzotriazole) and 250 (MDI).

	Molecular Weight	
	Theoretical	MS
Benzotriazole	119	120
MDI	250	250

Figure 1 indicates that the molecular weight of the compounds was 250 and 251 and 120. Since the melting point of the reaction product does not correlate to either reactants, one can assume that these molecular weights reflect the adduct compositions, especially since no free NCO was found in the benzotriazole-MDI adduct.

Methyl-Ethyl Ketoxime Adduct

Methyl-ethyl ketoxime (2 moles) was reacted with 1 mole of MDI according to the following:



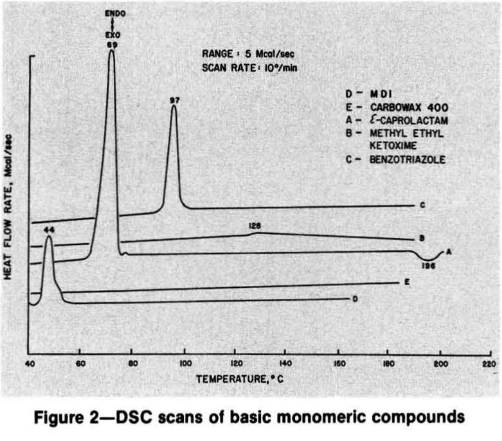


Figure 2—DSC scans of basic monomeric compounds

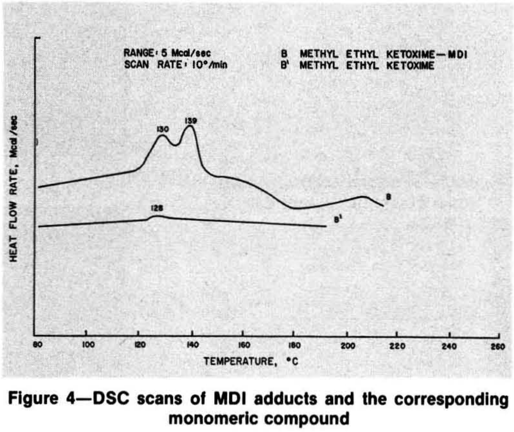


Figure 4—DSC scans of MDI adducts and the corresponding monomeric compound

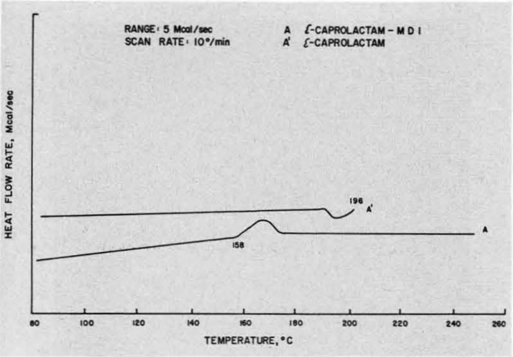


Figure 5—DSC scans of MDI adducts and the corresponding monomeric compound

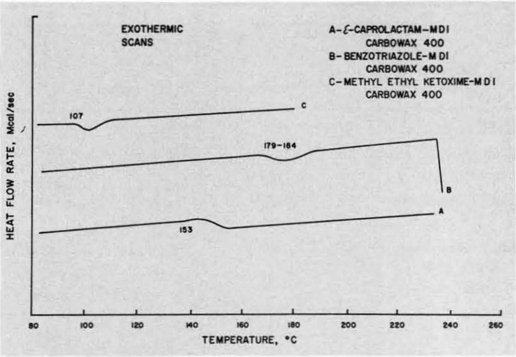


Figure 6—DSC scans of blocked MDI compounds and Carbowax 400 (1/1 ratio of NCO/OH)

The reagents, methyl-ethyl ketoxime (1.13 moles) and MDI (0.565 moles) were reacted in the following manner: A 25% solution of MDI (153 g) in butyl Cellosolve acetate (urethane grade) was added dropwise to a 50% solution of methyl-ethyl ketoxime, also in butyl Cellosolve acetate. The addition was carried out in a three-liter resin flask equipped with a stirrer, reflux condenser, thermometer well, dropping funnel, heating jacket, and an inlet tube to supply a blanket of dry nitrogen during the reaction. Triethylene diamine, 1%, was recommended for use as catalyst rather than dibutyltin dilaurate.[14] The reaction was carried out first at 60° C until the isocyanate addition was completed and was subsequently heated between 85-90° C for one hour to insure complete reaction. Total reaction time was 135 minutes. The product was soluble, unlike the other adducts which precipitated from solution when they formed.

The clear reactant solution was subsequently cooled in an alcohol dry ice bath. The adduct precipitated from a solution at a temperature range of −45° to −58° C. The precipitated adduct was washed and purified with cold butyl Cellosolve acetate. Washing the precipitate created a problem because the butyl Cellosolve acetate redissolved the adduct. However, when cooled to −45° C or below, the solubility was limited and the washing was successful, as evident by the sharp melting point.

The methyl-ethyl ketoxime-MDI adduct has a melting point of 142° C, and elemental analysis as follows:

	C	H	N
Theoretical, %	65.09	6.60	13.20
Actual, %	65.14	6.81	12.97

Again, no reference could be found in the literature and no counter check could be made on the melting point.

DIFFERENTIAL SCANNING CALORIMETRY DATA

DSC measures heat flow into or out of a sample over a specified temperature range. This technique was used because the compounds synthesized contain different adduct structures and, thus, should exhibit significant energy variations (endothermic) when they unblock (*Table* 2). In addition, when an active hydroxyl-containing compound is present upon unblocking, reaction of the free isocyanate will occur and exhibit an exotherm. These separate reactions should be easily verified with DSC.

Thermal Gravimetric Analysis (TGA), on the other hand, measures sample weight changes over a specified temperature range. Thus, since the compounds used may not exhibit volatility over the unblocking temperature range, TGA could not be used.

The DSC investigation was carried out using a Perkin-Elmer Standard Model 2. The rate of heating was set to 10° K per minute. Samples (1.5 mg ±0.1) were weighed on a Cahn Electrobalance and contained in aluminum

Table 3—Summary of Endotherm Temperatures in DSC Scans of Components and Blocked MDI

	Initial, °C	Maximum, °C
MDI	40	44.5
Carbowax 400	—	—
ε-caprolactam	63	69.5
Methyl-ethyl ketoxime	No meaningful curve	
Benzotriazole	92	97.4
ε-caprolactam-MDI	158	162
Methyl-ethyl ketoxime-MDI	120	130 and 139
Benzotriazole-MDI	214	222

Table 4—DSC Scans of Blocked MDI Compounds And Carbowax 400 (1/1 NCO/OH) (Exothermic Scan In All Cases)

	Initial, °C	Maximum, °C
ε-caprolactam-MDI	153	155
Methyl-ethyl ketoxime-MDI	107	Not defined
Benzotriazole-MDI	179	184

Table 5—Pigments

Chemical Composition	Trade Name	Source
$BaSO_4$—#1	Barytes	Pfizer, Inc.
Zinc phospho oxide complex	Nalzin SC-1	NL Industries, Inc.
$CaCO_3$ (natural)	Camel Wite	Harry T. Campbell Son's Corp.
Magnesium silicate MgO, 31%, Water, 4.8%; SiO_2, 63.5%' (3 MgO .4 SiO_2 H_2O)	Micro Talc HP-15-58	Pfizer, Inc.
Mica 3 $Al_2O_3 \cdot K_2O$.6 SiO_2.2H_2O	325 White Waterground P-81	English Mica Co.
Sb_2O_3		NL Industries, Inc.
ZnO	XX600 Acicular Type	New Jersey Zinc
TiO_2	Unitane OR-600 Rutile Type	American Cyanamid Co.
Fe_2O_3	1167 Black Oxide	Reichard-Coulston, Inc.

pans with sealable lids. All samples were run in a nitrogen atmosphere.

The DSC scans for the blocked adducts and the monomeric components are shown in *Figures* 2-5. They confirm the absence of monomeric components in the adduct structure.

Table 3 summarizes the temperature of the endothermic reaction. This indicates the point at which unblocking occurs. The accuracy of the DSC regarding this action is emphasized by the double endothermic peak (130° and 139°C) of the methyl-ethyl ketoxime-MDI adduct. Thus, verification of the presence of the two possible isomers which encompass the methyl-ethyl ketoxime structure (alpha and beta) was obtained.

Polyol was added to determine its effect on the unblocking of the adduct. The polyol was chosen because it has a negligible vapor pressure at the unblocking temperature, and it contains only primary hydroxyl groups. Therefore, polyethylene glycol of average molecular weight 400 (Carbowax 400) with the following structure was utilized.[15]

$$HO—CH_2—CH_2—(O—CH_2—CH_2)_x—O—CH_2—CH_2OH$$

In each case, a 1:1 molar ratio of —OH to —NCO was used. It would be expected that the endothermic nature of the reaction would change in the presence of the active —OH compound, since upon unblocking, immediate reaction would occur with the polymeric OH compound. *Table* 4 verifies this to be the case because the DSC scans in this series only exhibited an exothermic reaction. Significantly, in each case the exotherm occurs at a lower temperature than the original unblocking temperature (endotherm). The DSC scans in this series are also shown in *Figure* 6.

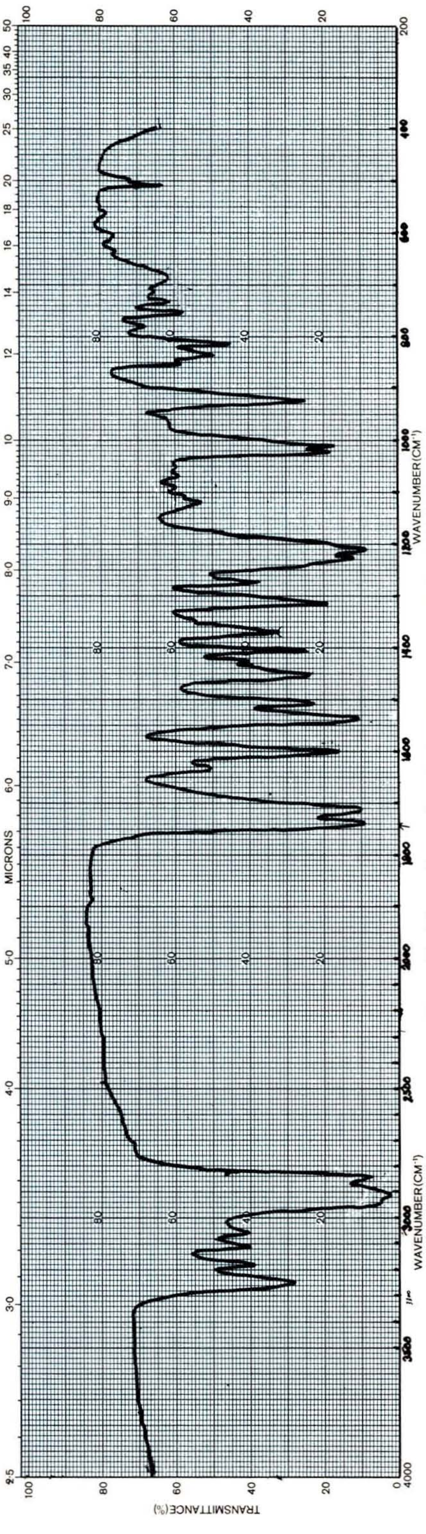


Figure 7—IR spectrum of methyl-ethyl ketoxime-MDI adduct

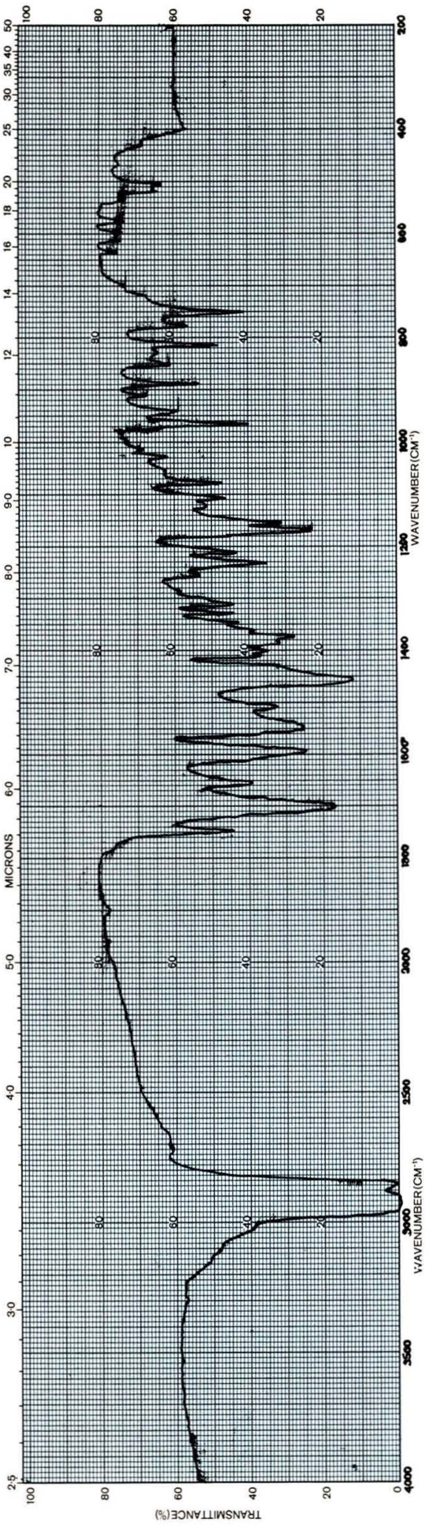


Figure 8—IR spectrum of ε-caprolactam-MDI adduct

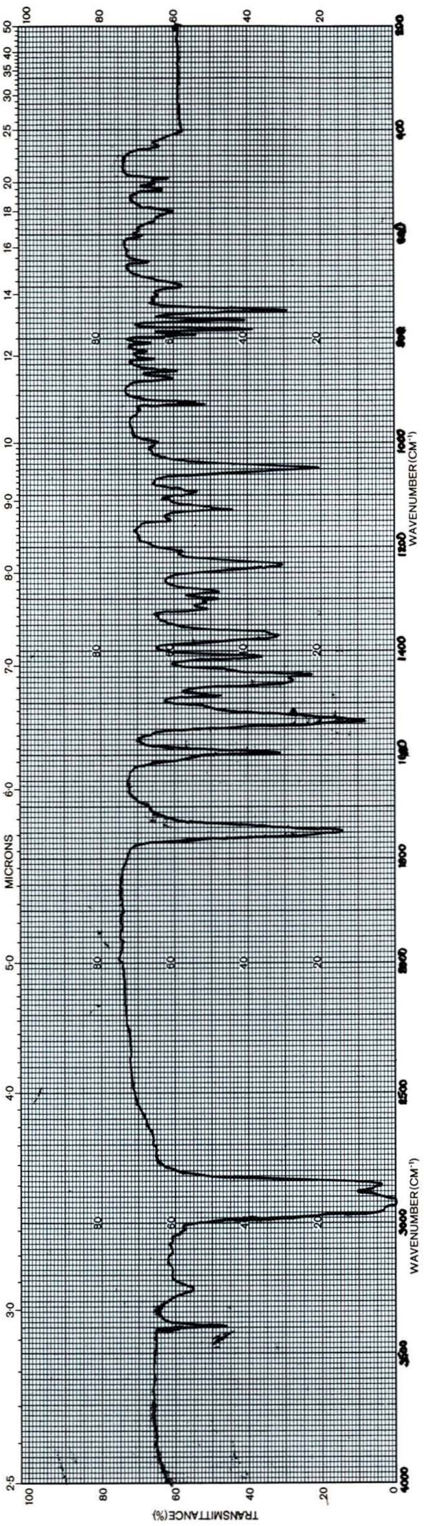


Figure 9—IR spectrum of benzotriazola-MDI adduct

Table 6—Summary of Exotherm Temperatures in DSC Scans of Pigmented Adduct and Carbowax 400 Mixtures

	Pigments								
Adduct	TiO_2, °C	ZnO, °C	$CaCO_3$, °C	Black Fe_2O_3, °C	Talc, °C	Mica, °C	Sb_2O_3, °C	Nalzin, °C	$BaSO_4$, °C
Benzotriazole-MDI (179°C) + Carbowax 400	151	154	151	—	157	155	182	162	157
ε-Caprolactam-MDI (153°C) +Carbowax 400	162	165	158	165	167	166	161	167	164
Methyl ethyl ketoxime-MDI +Carbowax 400 (107°C)	107	110	114	110	107	110	110	110	107

The next phase of the study was carried out with nine common pigments added to determine their effect on the DSC scans. Theoretically, if the pigment had a catalytic or inhibiting effect on the unblocking reaction, it should exhibit a distinct change in the DSC scans previously obtained. These pigments, listed in *Table* 5, were dried at 77°C for two hours and the DSC curves were run to establish their base line curves. When scanned alone in the DSC, none of the pigments show an endo- or exothermic peak, indicating complete stability in the studied temperature range.

Equal amounts by weight of the adduct, Carbowax 400, and pigments were mixed and the DSC curves run. *Table* 6 summarizes the pigment effect with each of the adducts.

INFRARED STUDIES

Infrared spectra of all samples were obtained using the oil-mull technique and potassium bromide as the sample matrix. Those areas in which absorption occurs due to the presence of nujol are shaded and should be discounted (2910-2990, 1430-1480, 1360-1390 cm^{-1}). A Perkin-Elmer, Model #399B IR spectrophotometer at a scan speed of 12 min was used.

Bakes were performed in a Despatch oven, model #LAC 1-38B, with the vent closed. Unless otherwise noted, all bakes were 20 min in length.

The spectra of the blocked adducts appear in *Figures* 7-9. These are included since two of them are new compounds and have not been previously reported and characterized in the literature. The ε-caprolactam adduct was previously reported,[13] however, no IR spectrograph was given in the reference. They all show a complete absence of absorption at the 2270 cm^{-1} range, thus reconfirming complete reaction of the MDI. However, in the case of the methyl-ethyl ketoxime adduct, a small peak occurred at 2220 cm^{-1}. This was close enough to cause suspicion; thus, the adduct was analyzed for free NCO via the Siggia[12] technique. None was found.

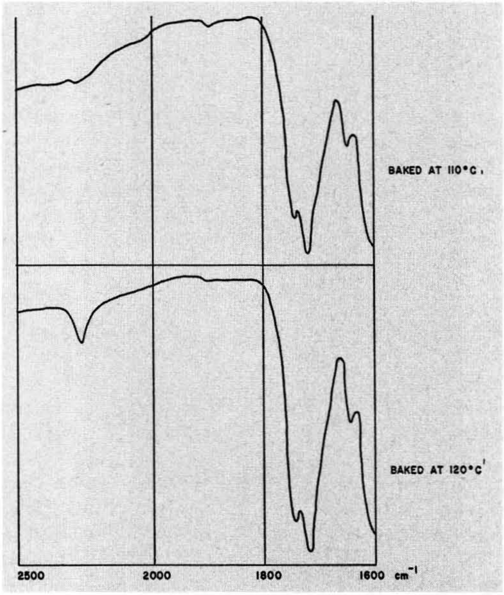


Figure 10—Infrared spectrograph of methyl-ethyl ketoxime-MDI of baked adduct (1600–2500 cm^{-1})

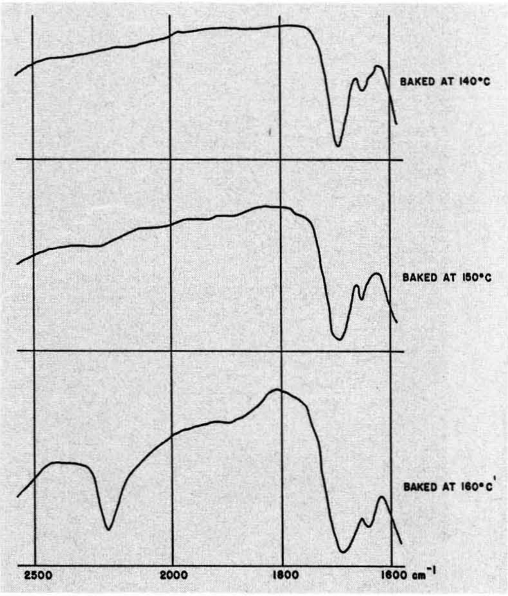


Figure 11—Infrared spectrograph of ε-caprolactam-MDI of baked adduct (1600–2500 cm^{-1})

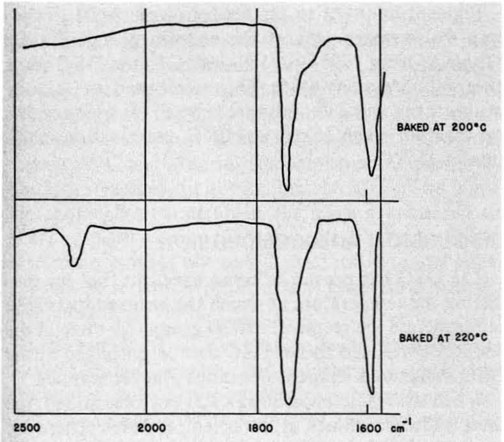
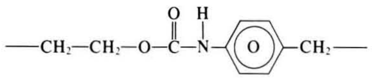


Figure 12—Infrared spectrograph of benzotriazole-MDI of baked adduct (1600–2500 cm^{-1})

Each adduct was baked at a temperature at which reaction was insured and the reacted compositions of the adducts and Carbowax 400 were IR scanned. When compared with the scans of the nonbaked compositions, four common changes occur in all three adducts: peak shift from 1700 to 1730 cm^{-1}; disappearance of band at 1510 cm^{-1}; disappearance of band at 1325 cm^{-1}; and appearance of band at 1215–1225 cm^{-1}.

Based on the chemistry of the reaction between the MDI and Carbowax 400 polyol, the following chemical structure should be present in all three adducts.



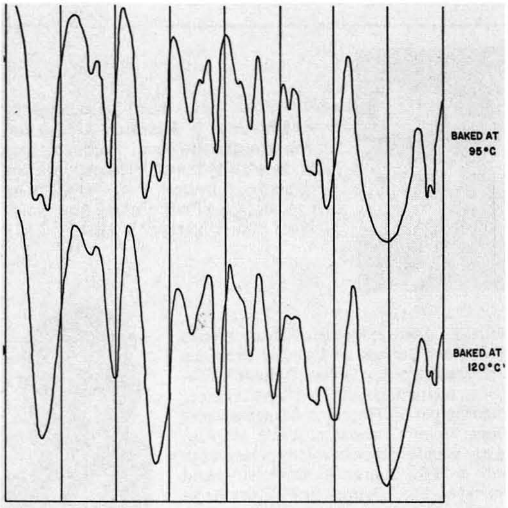


Figure 13—Infrared scans of baked methyl-ethyl ketoxime-MDI adduct, Carbowax 400 and ZnO

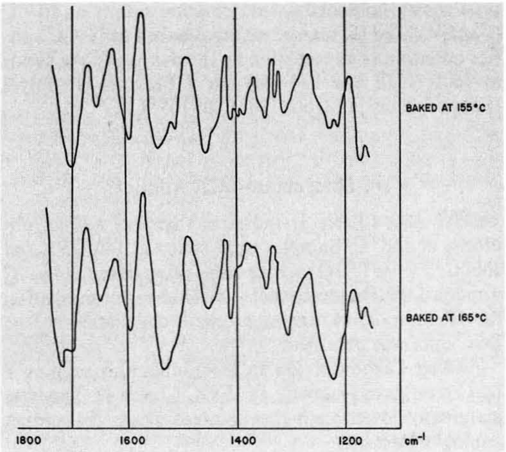


Figure 14—Infrared scans of baked ε-caprolactam-MDI Carbowax 400 and mica

However, the curves cannot be identical, since two of the blocking agents, ε-caprolactam and benzotriazole, are expected to remain in the baked material, whereas methyl-ethyl ketoxime should volatilize during the baking process. Therefore, the extent of the reaction of isocyanate with Carbowax can be validated using these changes in the IR spectra.

Methyl-Ethyl Ketoxime-MDI Adduct

DSC data (*Table* 3) indicates that this adduct begins to unblock at 120°C. Samples baked at 110°C and 120°C (*Figure* 10) verify the DSC data as the characteristic NCO peak at 2270 cm^{-1} appears only after the 120°C bake.

When Carbowax 400 is added to the adduct, the DSC

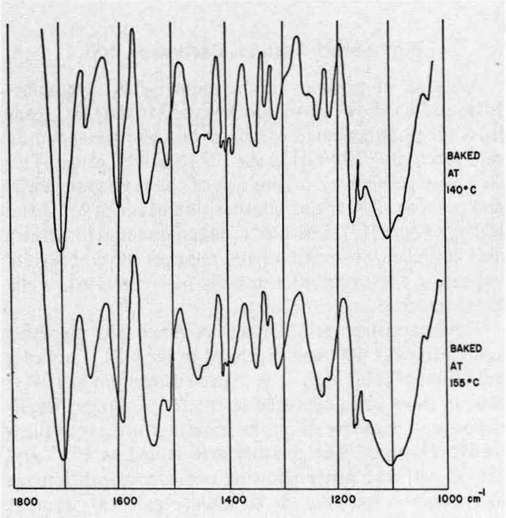


Figure 15—Infrared scans of baked benzotriazole-MDI Carbowax 400 and TiO$_2$

scan shows unblocking, and reaction occurs at 107°C (*Table* 4). The IR scan of material baked at 110°C verifies initiation of the reaction by the changes in the bands at 1600, 1270 and 930–960 cm^{-1}. Presence of polyol lowers unblocking from 120°C to 107°C.

ϵ-Caprolactam-MDI Adduct

DSC data (*Table* 3) indicates that this adduct unblocks at 158°C. Samples were baked at 140, 150, and 160°C (*Figure* 11). Only the composition baked at 160°C displayed the characteristic —NCO absorption band in the IR scan. Thus, again, excellent correlation to the DSC data was obtained.

Adding Carbowax 400 to the adduct and running a DSC scan gave reactivity at 153°C (*Table* 4). This was marginally lower than the point at which the adduct unblocks by itself.

Benzotriazole-MDI Adduct

DSC data (*Table* 3) indicates that this adduct begins to unblock at 214°C. IR scans of adduct baked at 200°C and 220°C show the NCO absorption peak only on the 220°C material (*Figure* 12), again giving excellent correlation with the DSC.

When Carbowax 400 is added to the adduct, the DSC scan indicates that the reactivity is activated at 179°C (*Table* 4). This is a considerable decrease from the adduct alone. Significantly, this adduct with Carbowax 400 is the only one which would not correlate with the IR on baked material. IR scans of material baked at 140°C confirmed no reaction had occurred, but when baked at 160° C, reaction was confirmed by changes in the peaks at 1600–1650, 1410, 1210-1230, 945 cm^{-1}. Theoretically, this should not have happened, since the DSC indicates stability at 179°C.

Pigmented-Adduct-Carbowax 400

Addition of pigment to the methyl-ethyl ketoxime-MDI adduct shows very little deviation in the DSC scans from the nonpigmented composition. All pigments indicate reactivity (*Table* 6) in the 107 to 114°C range. This data was verified by taking one of the pigments (ZnO) and running IR scans on composition baked at 95°C and 120°C (*Figure* 13). The 120°C baked material illustrates that sufficient absorption band changes have occurred, indicating the expected reactions have initiated, while 95°C material had no change.

The ϵ-caprolactam-MDI adduct mixed with pigments and Carbowax 400 when scanned in the DSC showed a mild level of inhibition. The expected reaction at 153°C was, in most cases, elevated to the 165°C range. Verification was made by using the mica mixture as the illustrative example. The mixture was baked at 155° and 165°C with the latter showing considerable difference in absorption bands in the IR scan (*Figure* 14), whereas the 155°C did not display the reactivity shown in the nonpigmented composition.

Pigment addition to the benzotriazole-MDI Carbowax 400 mixture reduces the endotherm significantly (*Table* 6) from 179°C to 150–160°C in the DSC scans except for antimony oxide. The sample used for IR study in this series was TiO_2 mixture (*Figure* 15) which shows no reaction when baked at 140° C and reaction initiation at 155° C.

DISCUSSION AND CONCLUSION

The DSC has proven to be an excellent tool for predicting the temperature at which the basic adducts split to regenerate the reactive —NCO group. All three of the adducts correlated to the DSC data when baked at the DSC designated temperature range and IR scanned.

When the reactive Carbowax 400 polyol is added, the three adducts unblock at lower temperatures. There are several possible explanations for this phenomena, as there could be a bimolecular reaction of the primary hydroxyl group on the blocked site or a solvating effect on the adduct itself. The solvating effect seems to be magnified at the high unblocking temperatures, as illustrated by the benzotriazole adduct which fell from 214°C to 179°C. This same phenomenon can possibly account for the poor correlation of the DSC vs bake-IR exhibited by the benzotriazole adduct, whereas the other two performed as predicted. Perhaps baking the adduct in the presence of the liquid Carbowax accelerated the unblocking reaction, especially when the duration of bake is as long as 20 min. The short scan time of the DSC may have hidden this observation. However, this phase of the study succeeds in illustrating the importance of the crosslinking agent in the unblocking reaction. It is very possible a more complex polymeric polyol may not have displayed this reduction in unblocking temperature.

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Pigment addition to the adduct-Carbowax 400 mixtures seems to be universal in effect, depending more upon the base adduct than upon the specific pigment. All the pigments used performed about the same, having no effect on the methyl-ethyl ketoxime adduct, raising the ϵ-caprolactam adduct unblocking reaction by approximately 10°C and lowering the benzotriazole adduct unblocking reaction by approximately 20°C. The higher the unblocking temperature, the more pronounced the effect of pigmentation. This indicates that the effect of pigmentation does not have a chemical catalytic effect. Instead, it would probably be a physical absorption phenomenon.

Sufficient evidence has been gathered to support the conclusion that some blocked adducts are susceptible to changes in unblocking temperatures by pigments and crosslinking additives. Addition of catalysts would no doubt alter much of the data presented in this investigation, but their inclusion may have detracted from the impact of our basic objectives and conclusions.

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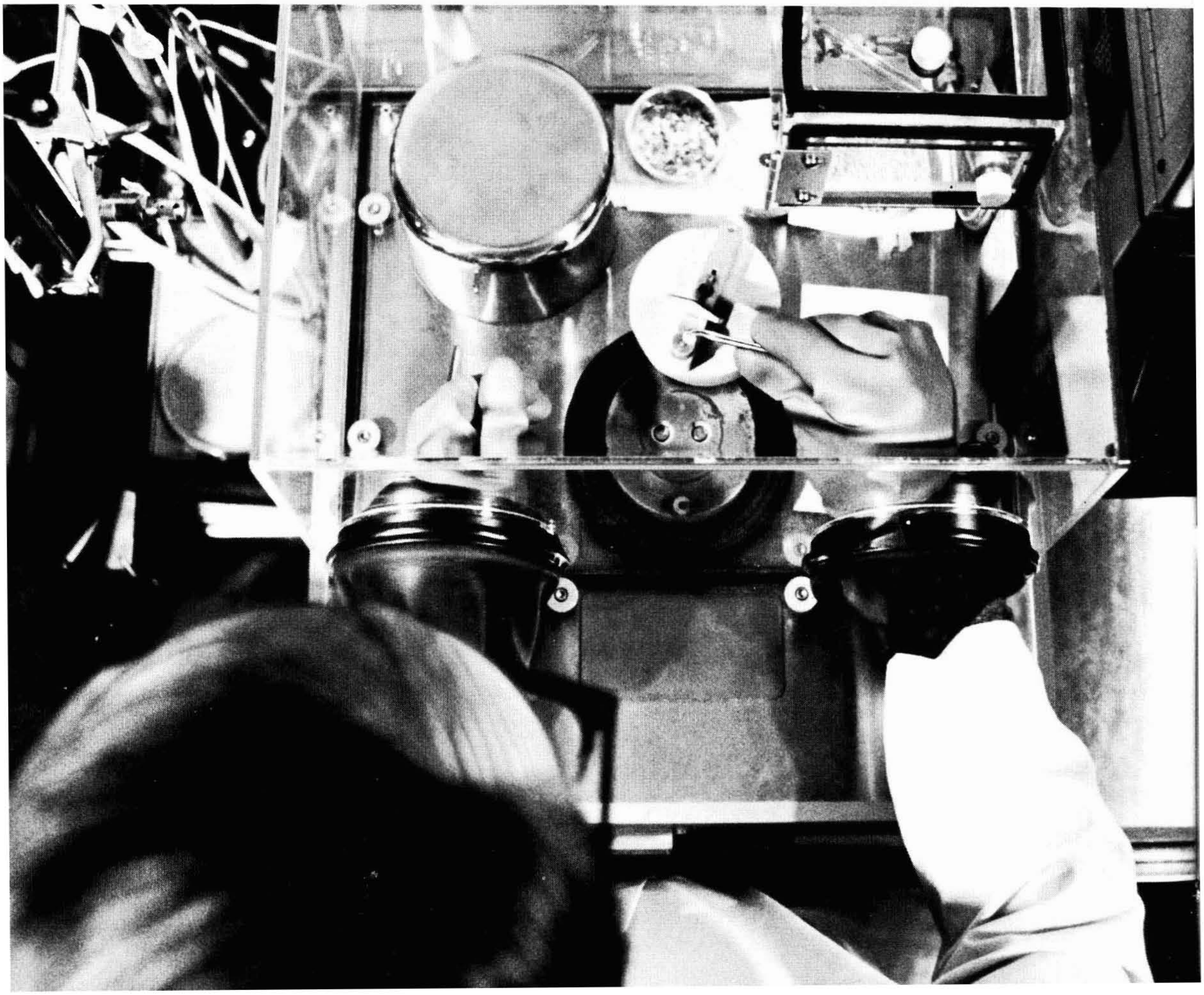
Errata

The article, "Skid Resistant Coatings for Aircraft Carrier Decks" (JCT, Dec. 1980, pp 65–69), was prepared by the authors, S.H. Davang and H. Dear, while employed at the David W. Taylor Naval Ship R&D Center, Annapolis, MD and *not* at Tracor, Inc., as it appears in the byline. Mr. Dear is a former employee of the Center and is now associated with Tracor, Inc. The Journal sincerely regrets the error.



Birmingham Club, Chicago and Louisville Societies Win MMA Awards for Notable Achievements

The 1980 MMA Awards for notable achievements by Constituent Societies of the Federation were won by the Birmingham Club and the Chicago and Louisville Societies. Presentation of the Awards was made at the recent Federation Annual Meeting in Atlanta.

Established in 1975 by Materials Marketing Associates, a national marketing group of manufacturers' representatives, the Awards recognize notable achievements by Constituent Societies (excluding those Society papers presented at the Federation Annual Meeting).

There are three categories of Awards, based on Society membership size.

Each winning Society receives $350 in cash plus a handsome plaque, appropriately engraved.

Material Marketing Associates Awards were presented by MMA Awards Committee Chairman Tom Cochran (right) and MMA Executive Director John Paul Taylor (left) to winners (left to right): John Vandeberg, for the Chicago Society; John Bauer, for the Louisville Society; and John Hitchin, for the Birmingham Club

Compilation Of Infrared Spectroscopy Atlas

The Chicago Society was cited for its work in compiling the *Infrared Spectroscopy Atlas for the Coatings Industry*. This 896-page book (published by the Federation) contains 1433 infrared spectra of materials commonly utilized in the coatings industry, fully indexed. Spectra of many recently marketed materials are included, as well as examples of Fourier transform infrared spectra.

The text includes a fundamental and comprehensive dissertation on the theory of infrared spectroscopy, and chapters on qualitative and quantitative analysis, IR instrumentation accessories, sample preparation and selected applications. A fully indexed literature survey contains over 1500 references and represents the most complete bibliography published in this type of text.

Excellence of Educational Program

The Louisville Society won an Award for educational activities, through both seminars and various college-level courses.

The Society has had a cooperative program for many years with the University of Louisville, and has sponsored courses on such topics as: basic and advanced coatings technology; instrumental analysis; resins; and quality control. These courses feature presentations by instructors and guest lecturers, all of whom are industry people. College credits are awarded to students who successfully complete the courses.

A special 1980 symposium was held in May, focusing on "Compliance with Government Regulations" and "Hazardous Waste Disposal," which featured the comments of speakers from industry, government, and academic institutions.

Industry Contributions Through Seminars and A/V Programs

The Birmingham Paint, Varnish, and Lacquer Club was cited for its preparation of audio/visual presentations on "The Setaflash Tester" and "An Introduction to the Paint Industry," sponsoring a symposium on "Effects of Current Legislation in the U.K. upon the Industry," and for developing procedures to serve as agents in the U.K. for marketing Federation literature and A/V programs.

"The Setaflash Tester" is an hour-long, 134-slide presentation on procedures for testing for flash point by both the go/no-go and definitive methods for different temperature ranges. "Introduction to the Paint Industry" explains the basics of paint technology and manufacture, its importance in society, and career opportunities in the industry.

The symposium on legislation focused on product liability, labeling, and occupational health and safety regulations.

Principles Governing Awards

The MMA Awards recognize notable achievements in the field of education, manufacture 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 (Terryl F. Johnson, Cook Paint & Varnish Co., P.O. Box 389, Kansas City, Missouri 64141).

A complete description of the Society activity to be considered in the competition must be submitted by the Society President to Chairman Johnson by July 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.; E. T. Horn Co., Los Angeles, Calif.; Lukens Chemical Co., Boston, Mass.; Matteson-Ridolfi, Inc., Detroit, Mich.; McCullough & Benton, Inc., Atlanta, Ga.; McNalley & Webber Co., Cleveland, Ohio; Ribelin Distributors, Inc., Dallas, Texas; 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.

Use of Thiirane-Functional Monomers as a Means of Developing Crosslinkable Emulsions

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B. George Bufkin,* and John R. Grawe*
University of Southern Mississippi†

A feasibility study was conducted which investigated the acceptability of using 2,3-epithiopropyl methacrylate (ETPM) as a means of developing crosslinkable emulsion.

To prevent premature reactivity of the thiirane functionality in the emulsion environment, the polymerizations were conducted at low temperatures using nonionic surfactants and a two-step, delayed addition of episulfide monomer. However, in spite of the preventative measures taken, compositions containing in excess of 6 mole percent ETPM possessed inordinately high degrees of premature crosslinking. Therefore, to avoid the baneful effects associated with excessive losses in thiirane functionality and to obtain coatings with an acceptable balance of physical properties, the 54.5/36.4/9.1-EA/MMA/ETPM copolymer emulsion (6 mole percent ETPM) was selected as the most reasonable candidate for further evaluations.

When the model emulsions containing 6 mole percent ETPM were combined with various chemically and structurally different curing agents, the samples containing piperazine displayed a 2.5 fold increase in tensile strength, a 2.3 fold increase in solvent resistance, a 2.7 fold decrease in elongation, decreased impact resistance, and increased film hardness (2H vs 2B) after thermal curing (30 min/65° C) as compared to samples of the same emulsion evaluated without a curing agent.

Coatings of the ETPM-containing emulsion were also found to crosslink at room temperature when piperazine was used as a curing agent.

INTRODUCTION

Crosslinking in emulsion systems is typically achieved by copolymerizing up to 10% by weight of a vinyl-type monomer which contains reactive functionality suitable for post-curing. Of the various crosslinkable monomers which have been investigated for the development of such products, the use of epoxy-functional, vinyl monomers represents one of the more effective and versatile approaches for obtaining low temperature curing, high integrity coatings.[1] While considerable effort has been placed on the development of oxirane-functional emulsions, relatively little attention has been given to examining the advantages and limitations associated with the development of epithio-functional emulsions (epithio, episulfide, or thiirane compounds are the sulfur counterparts of oxirane derivatives).

Qualitative Differences in the Reactivities Of Oxirane and Thiirane Compounds

In many respects, oxirane- and thiirane-functional compounds display quite similar modes of reactivity. For example, thiirane derivatives undergo nucleophilic ring opening reactions with alcohols,[2] phenols,[3] phosphites,[4] thiols,[5] primary and secondary amines,[6] and compounds capable of forming carbanions[7] while electrophilic ring opening reactions occur with hydrogen halides,[8] halogens,[9] carboxylic acid halides,[10] alkyl halides,[11] and halides of sulfur,[12] phosphorus,[13] and arsenic.[14]

However, in spite of the similarities, certain salient differences have been observed in the ring cleavage reactions of thiirane and oxirane compounds as a result of the weak bonding and antibonding nature of the C—S bond, together with the pronounced electrophilic character of the thiirane ring. For instance, the reaction between thiols and the oxirane ring in a nonpolar medium occurs only under the influence of base catalysis which implies that the formation of a trimolecular com-

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Table 1—Second Order Rate Constants and Activation Parameters for the Reaction of Aniline with Propylene Oxide and Propylene Sulfide

Propylene Sulfide		Propylene Oxide	
T (C)	$k \times 10^5$ ($1\ mole^{-1}S^{-1}$)	T (C)	$k \times 10^5$ ($1\ mole^{-1}S^{-1}$)
49.60	1.59	50.90	3.65
	1.52		3.55
41.05	0.815	39.08	2.30
	0.852		2.20
39.75	0.722		
30.60	0.330	30.50	1.72
	0.334		1.59

Activation Parameters

	E_A ($\Delta H^{\#}$) (kcal/mole)	log A	$\Delta S^{\#}$ (e.u.)
Propylene sulfide	14.64	5.10	−35.2
Propylene oxide	7.10	0.35	−56.8

Table 2—First Order Rate Constants and Activation Parameters For the Reaction of Acetic Acid with Propylene Oxide and Propylene Sulfide

		Experimental Rate Constants (S^{-1})			
Reactants	T(C)	$k \times 10^{5a}$	$k_N \times 10^{5b}$	$k_A \times 10^{5c}$	k_H/k_o
Propylene oxide and acetic acid	51.80	3.19	2.23	0.96	
Propylene oxide and acetic acid	70.00	14.70	10.29	4.41	
Propylene oxide and acetic acid	90.00	66.30	46.40	19.90	
Propylene oxide and d-acetic acid	51.80	1.65			1.93
Propylene oxide and d-acetic acid	70.00	9.96			1.43
Propylene oxide and d-acetic acid	90.00	43.70			1.52
Propylene sulfide and acetic acid	70.20	0.232	0.127	0.105	
Propylene sulfide and acetic acid	82.00	0.622	0.342	0.280	
Propylene sulfide and acetic acid	92.00	1.060	0.583	0.477	
Propylene sulfide and d-acetic acid	90.00	0.627			1.6

Activation Parameters

Reactants	$\Delta H^{\#}$ (kcal/mole)	$\Delta S^{\#}$ (cal/deg. mole)
Propylene oxide and acetic acid	18.5 ± 0.9	−24.3 ± 1.5
Propylene sulfide and acetic acid	15.7 ± 1.4	−40.6 ± 4.5

(a) Average for two determinations.
(b) k for attack at C-1 (Normal attack).
(c) k for attack at C-2 (Abnormal attack).

plex is necessary for the activation process.[15] However, the reaction of thiols or even amines with thiirane compounds occurs smoothly in nonpolar solvents without the need for catalysis. Moreover, the tertiary amine catalyzed reaction of thiophenols with ethylene sulfide proceeds with somewhat greater difficulty in nonpolar solvents than the same reaction conducted with ethylene oxide.[16] Thus, thiophenol and p-thiocresol react quantitatively with ethylene oxide at 20° C while the reaction with ethylene sulfide requires heating above 40° C.

Besides displaying slower reactivity as compared with oxirane compounds, the inclusion of sulfur has also been found to promote such anomalous effects as a greater tendency to undergo abnormal ring cleavage resulting in the formation of the anti-krasuskii product,[17] the elimination of sulfur through attack by metals[18] or amine nucleophiles,[19] the formation of thiiran 1,1-dioxide by oxidation by peroxides,[20] and the predilection towards poly(alkylene sulfide) formation resulting from the strong nucleophilicity of the thiolate anion.[21]

Quantitative Measurements Of the Reactivity Differences Between Oxirane and Thiirane Compounds

Concomitant with the many qualitative treatments which illustrate the anomalous responses associated with thiirane compounds, quantitative studies have been conducted which provide additional insight into the ability of the sulfur atom to alter the rate of reaction and mechanism of ring opening. For example, kinetic studies which monitored the rate of reaction of aniline with propylene sulfide and propylene oxide indicated that while both ring opening reactions proceeded in accord with second order kinetics, the inclusion of sulfur significantly altered the rate of ring cleavage and the various activation parameters associated with the process. The magnitude of these differences is illustrated by the data presented in *Table* 1.

According to these results, the substitution of the oxygen atom by sulfur resulted in a two to six fold decrease in the rate constant of the addition reaction. While cleavage of the C—S bond (55 kcal/mole) requires less energy than the C—O bond (75 kcal/mole), the higher degree of polarization inherent in the C—O bond provides a more favorable energy condition for attack by the approaching nucleophile. Consequently, the transition state for the less polarized C—S bond involves a smaller degree of bond formation between aniline and carbon and a greater degree of bond breaking between carbon and sulfur. Under these circumstances, the thiirane ring tends to display a lower degree of reactivity with a given nucleophile than does the analogous oxirane ring.

Besides the effects due to polarization, another factor contributing to a difference in reactivity between thiirane and oxirane compounds involves the strain energy inherent in the three-membered rings. The ring-strain energies of ethylene oxide and ethylene sulfide have been estimated by hydrogenation methods as being 14 and 9 kcal/mole, respectively. Thus, by opening the more highly strained oxirane ring, more energy is released to

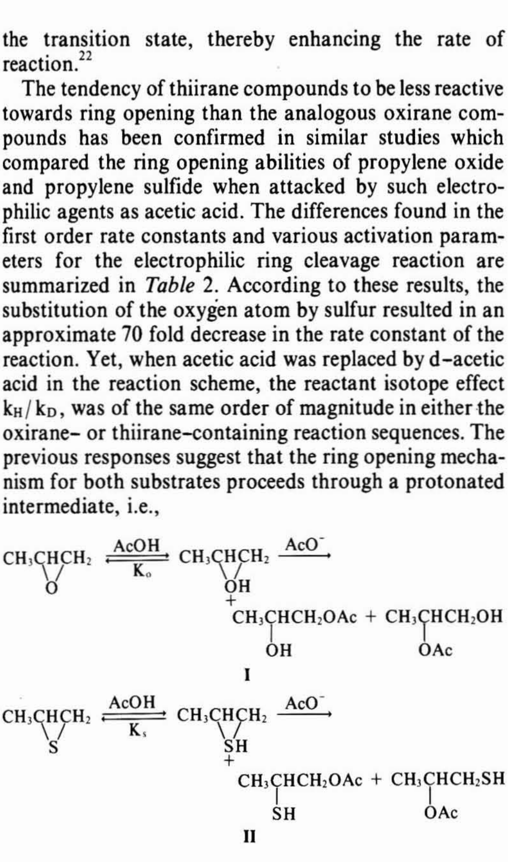
Table 3—Relationship Between the Basicity of the Amine and the Rate of Ring Opening[a]

Amine	pK_b	Rate constant (k_{exp}, 1 $mole^{-1}hr^{-1}$)	$-\log k_{exp}$	Δpk_b[b]	$\Delta(-\log k_{exp})$[b]
Morpholine	11.15	0.0246	2.6294	−0.85	−0.76
Piperidine	10.30	0.249	1.3962	0	0
Pyrrolidine	10.17	0.316	1.4997	0.13	0.096

(a) Kinetic values were determined with propylene sulfide at 60° C in anhydrous dioxane.
(b) Note the approximate 1 to 1 correlation between Δpk_b and $\Delta(-\log k_{exp})$.

the transition state, thereby enhancing the rate of reaction.[22]

The tendency of thiirane compounds to be less reactive towards ring opening than the analogous oxirane compounds has been confirmed in similar studies which compared the ring opening abilities of propylene oxide and propylene sulfide when attacked by such electrophilic agents as acetic acid. The differences found in the first order rate constants and various activation parameters for the electrophilic ring cleavage reaction are summarized in *Table* 2. According to these results, the substitution of the oxygen atom by sulfur resulted in an approximate 70 fold decrease in the rate constant of the reaction. Yet, when acetic acid was replaced by d-acetic acid in the reaction scheme, the reactant isotope effect k_H/k_D, was of the same order of magnitude in either the oxirane- or thiirane-containing reaction sequences. The previous responses suggest that the ring opening mechanism for both substrates proceeds through a protonated intermediate, i.e.,



I

II

Assuming that the rate of protonation is approximately the same for both heterocyclic rings ($k_o \simeq k_s$), the difference in reactivity between the thiirane and oxirane compounds towards nucleophilic attack presumably resulted from the greater stability of protonated species **II** compared to species **I**. If the protonated thiirane species possessed greater stability, the subsequent ring opening reaction would involve relatively significant S_N2 character. Conversely, if the protonated oxirane species was considerably less stable, the ring opening process of the oxirane compound would be relatively spontaneous and thus emulate greater S_N1 character.[23]

Effects of Amine Basicity and Steric Hindrance On the Reactivity of Thiirane Compounds

Besides the effects which the heteroatom has on the inherent reactivity of the three membered ring, kinetic studies have also uncovered several external factors which influence the ring opening ability of the thiirane compound. For example, a comparison of the second order rate constants with the pK_b values of various amines indicated that the rate of ring opening for propylene sulfide was a function of the basicity of the amine. These data are presented in *Table* 3.

Since the ring opening mechanism presumably involved considerable S_N2 character, the steric factors associated with both reactants were expected to strongly influence the rate of ring cleavage. The relationship between the rate of reaction and the degree of steric hindrance possessed by the amine is illustrated in *Table* 4.

Since the amines selected for the investigation displayed nearly identical basicities, the loss of reactivity which occurred upon proceeding from diethylamine to di-n-butylamine was attributed to increased steric hindrance resulting from the bulk of the substituent on the nitrogen atom.

Steric hindrance was also found to play a prominent role in the reactivities of alkyl substituted thiirane compounds. The loss of reactivity of the thiirane ring upon substitution with methyl groups is presented in *Table* 5. Based upon the results for the series thiirane, 2-methylthiirane, and 2,2-dimethylthiirane where carbon 2 proceeds from primary to tertiary, the presence of a methyl group resulted in a 7 fold reduction in the rate constant for the reaction. Thus, the dimethyl substituted episulfide possessed 1/48.6 of the rate displayed by the unsubstituted episulfide compound.

In light of the various trends established in this latter rate study, the conclusion was drawn that the ring opening reaction of thiirane compounds with amines involved a S_N2 attack on the least substituted carbon atom. The nucleophilic attack by the amine was the rate determining step while the proton exchange between nitrogen and sulfur was the rapid step,[24] i.e.,

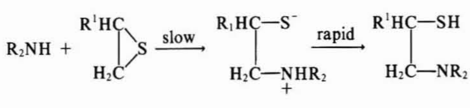


Table 4—Effects of Amine Substituents on the Rate of the Reaction

Amine	Substituent	Basicity $(pK_b)^a$	Rate Constant, (k_{exp}, 1 mole^{-1}hr^{-1})
diethylamine	ethyl	10.21	0.0194
di-n-propylamine	n-propyl	10.32	0.0138
di-n-butylamine	n-butyl	10.39	0.0132

(a) determined in anhydrous dioxane.

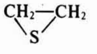
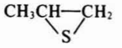
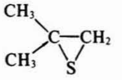
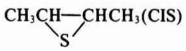
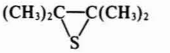
EXPERIMENTAL

The following procedure was used to prepare the thiirane-functional monomer:

Preparation of 2,3-Epithiopropyl Methacrylate

2,3-epithiopropyl methacrylate (ETPM) was prepared by reacting 897.3 g (6.31 moles) of distilled glycidyl methacrylate with 815.2 g (8.40 moles) of anhydrous potassium thiocyanate in 1500 ml of distilled acetone. Hydroquinone (0.7g) was used as the inhibitor. The thiocyanate exchange reaction was effected by refluxing the brown mixture for 16 hr while under a nitrogen atmosphere. At the end of the conversion period, the acetone carrier was removed by means of a distillation trap, and the resulting reaction mixture was allowed to cool to room temperature. The solid reactants and byproducts were removed by filtration, and the crude monomer was vacuum distilled three times to give an overall yield of 39% ETPM.

Table 5—Relationship Between the Rate of Reaction And the Substituents Present on the Thiirane Ring

Thiirane Compound	Rate Constant, (k_{exp}, 1 mole^{-1}hr^{-1})	Relative Reactivity^a
 $\underset{S}{\mathrm{CH_2{-}CH_2}}$	1.750	1
 $\underset{S}{\mathrm{CH_3CH{-}CH_2}}$	0.249	1/7.03
 $\underset{\mathrm{CH_3}\quad \mathrm{S}}{\overset{\mathrm{CH_3}}{\mathrm{C{-}CH_2}}}$	0.036	1/48.6
 $\underset{S}{\mathrm{CH_3CH{-}CHCH_3}}$(CIS)	0.0058	1/302
 $\underset{S}{\mathrm{CH_3CH{-}CHCH_3}}$(Trans)	0.0033	1/540
 $\underset{S}{\mathrm{(CH_3)_2C{-}C(CH_3)_2}}$	0.0004	1/4380

(a) rate of reaction relative to ethylene sulfide.

Preparation of a 54.56/36.37/9.07-Ethyl Acrylate/Methyl Methacrylate/ETPM Copolymer Emulsion (6 mole percent ETPM) Using a Delayed Monomer Addition Technique

The following sample procedure illustrates the general methodology used to prepare all the ETPM-containing model emulsions:

A 250 ml, 3-neck flask was fitted with stirrer, thermometer, condenser, nitrogen inlet, and two addition funnels. The apparatus was charged with 153 g of deionized water, and the contents were deoxygenated by heating to 95° C for five minutes while purging with nitrogen gas. After sparging, the deoxygenated water was cooled to 25° C, and 6 g of Triton® N-102 were added to the flask. The surfactant/water mixture was stirred for five minutes, and 20 g of a 60/40-ethyl acrylate (EA)/methyl methacrylate (MMA) monomer mix (20% by weight of the total monomer charge) were added to the agitated mixture. After a five minute equilibration period, 5.0 g of a 0.15% aqueous solution of ferrous sulfate and 0.3 g of sodium metabisulfite were added to the water-surfactant/monomer mixture as part of the redox initiator system. After a five-minute gestation period, a solution consisting of 0.3 g of ammonium persulfate in 30 g of deionized water was added dropwise to the agitated reaction mixture. After the initial exotherm subsided, 60 g of a 60/40-EA/MMA monomer charge (60% by weight of the total monomer charge) were added separately from but simultaneously with the remaining initiator feed. When the addition of the intermediate feed was complete, an overlay charge consisting of 10.93 g of the 60/40-EA/MMA monomer mix and 9.07 g of ETPM (20% by weight of the total monomer charge) was added simultaneously with the remaining initiator feed. The reaction temperature was maintained at 25° C by controlling the rate of monmer addition and by using external cooling. The addition of the remaining initiator feed usually continued for approximately 15 min past complete addition of the monomers, and the resulting emulsion was maintained at 25° C for an additional two hours in order to obtain a high degree of conversion. The emulsion was then filtered to remove the agglomerated material which formed.

Test Methods Employed in Characterization of the Emulsions

Thiirane Content Determination: Because of the propensity of the thiirane ring to undergo polymerization as compared to ring addition, many of the common techniques employed for the detection of residual oxirane content were found to be inappropriate for determining the residual thiirane content. The analytical techniques found to be unsuited for determination of the thiirane content included the pyridinium chloride method,[25] the potassium iodide method,[26] the acetic acid/hydrogen bromide method,[26] the hydrogen bromide/silver nitrate/potassium chromate method (ASTM D 1652), and addition by acetic acid.[23]

In light of this impasse, an analytical technique was developed based upon the addition of acetyl chloride with the thiirane ring.[27] An exposition of this procedure follows.

SAMPLE PREPARATION: The thiirane-containing emulsion (100 to 125 g) was coagulated with acetone, and the extracted polymer was washed four times with deionized water, pulverized, and dried under vacuum for 24 hr at ambient temperature. The dried polymer was then dissolved in a 3/1 mixture of acetone/toluene, and 3A molecular sieves were added to the solution to ensure complete dehydration. The solids content was determined in the conventional manner.

ANALYTICAL PROCEDURE: Approximately 340 g of the above polymer solution (3.2 meq of thiirane functionality) was weighed into a 3000 ml, round bottom flask which had been previously flame dried and purged with nitrogen gas. The flask was sealed and heated with stirring to 40° C. Upon equilibration at 40° C, 6.2 meq of acetyl chloride were added to the polymer solution by means of a syringe. After addition, the reaction temperature was raised to 45° C and this temperature was maintained for 12 hr. At the end of the acetyl chloride/ETPM reaction period, 50 ml of a 0.3 N solution of n-butylamine in toluene (15 meq of n-butylamine) were introduced into the reaction mixture in order to scavenge the unreacted acetyl chloride. After a 30 min reaction period, 800 ml of isopropanol were added to the flask, and the amount of unreacted n-butylamine was determined by titration to a bromophenol blue end-point with 0.5 N HCl. All thiirane determinations were conducted in duplicate and inherent errors were corrected by means of a blank.

Table 6—Characteristics of Epithiopropyl Methacrylate

	Experimental	Reported
Boiling point	47° C at 0.1 mm Hg	59° C at 1.4 mm Hg
Index of refraction	1.4950	1.4942
Density, 25° C, g/ml	1.1009	1.1005
	Calculated	**Experimental**
Elemental analysis		
% C	53.16	53.36
% H	6.33	6.48
% S	20.25	20.07
% O	20.25	20.09

Monomer purity = 99.+%

Solubility, 25° C, 50/50 mixture	
water	Less than 0.03 g/100 g H_2O
Benzene	Soluble
Carbon tetrachloride	Soluble
Methanol	Soluble
Ethyl acrylate	Soluble
Styrene	Soluble
Vinyl acetate	Soluble

SWELLING RATIO DETERMINATION: Hydrodynamic swelling ratios were conducted on samples in suspended form using the procedure outlined by Crews.[28] Swelling ratio represents the volume ratio of the solvent swollen polymer to unswollen polymer.

PERCENT INSOLUBLES: The percent insolubles were determined using a soxhlet extractor with methyl ethyl ketone (MEK) solvent.

Test Procedures Used to Physically Characterize the Latex Films

Physical test determinations such as Sward rocker hardness, impact resistance, flexibility, adhesion, gloss, and solvent resistance were conducted according to standard procedures outlined in the *Paint Testing Manual* [15 ed., G. G. Sward, ed., ASTM Technical Publication

Table 7—Stability of ETPM in an Emulsion Environment[a]

	Analysis of ETPM After 48 Hrs at:					
	25°C		45°C		65°C	
% Surfactant	EA[b]	SH[c]	EA	SH	EA	SH
None	99%	None	99%	None	99%	None
2% Dupanol WAQE	99%	None	99%	None	Polymer[d] @40 Hr	None[e]
2% Gafac RE-870	99%	None	99%	None	Polymer @44 Hr	None[e]
2% NH_4 Laurate	99%	None	99%	None	Polymer @39 Hr	None[e]
2% Triton X-405	99%	None	99%	None	98%	None

(a) Monomer to water ratio—50/50.
(b) EA indicates monomer purity based upon elemental analysis.
(c) SH indicates presence of SH absorption by infrared analysis.
(d) Indicates the formation of polymer.
(e) Vinyl unsaturation found unaffected by infrared analysis even though polymer was formed.

Table 8—Determining the Optimum Curing Conditions^a

Property	Temperature of cure cycle^b (°C)				
	35°	65°	93°^{c}	121°^{c}	149°^{c}
Tensile strength (Kg/cm^2)	119	175	141	121	154
Elongation (%)	1230	701	460	380	320
Acetone insolubles (%)	7	16	21	19	23

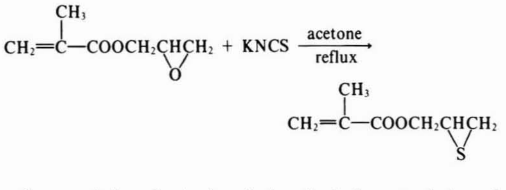
(a) All samples contained the stoichiometric amount of piperazine.
(b) All samples were baked for 30 min at each respective temperature.
(c) Considerable film yellowing developed at these temperatures.

500, 1972]. Tensile strength and percent elongation were determined on an Instron 1130 Tensile Tester, using a crosshead speed of 5.0 cm/min.

DISCUSSION, RESULTS, AND CONCLUSIONS

Preparation of a Thiirane-functional Monomer

Thiirane-functional compounds are conveniently prepared from the analogous oxirane derivatives through a sulfur-oxygen exchange reaction with thiourea[29] or potassium thiocyanate.[30] Thus, 1.0 mole of glycidyl methacrylate (GMA) was reacted with 1.33 moles of potassium thiocyanate for 16 hr to achieve a 39% yield of 2,3-epithiopropyl methacrylate (ETPM).



Some of the physical and chemical characteristics of ETPM are presented in *Table* 6. In general, ETPM was found to be soluble in common solvents and monomers but was insoluble in water. Thus, because of its propensity to remain in the organic phase, ETPM was expected to generate emulsion systems in which the crosslinkable functionality were statistically distributed throughout the volume of the polymer particles.

Choice of Surfactants

When anionic surfactants are used to effect the emulsion polymerization of epoxy-functional monomers, approximately 25% of the available oxirane groups are consumed during the reaction process.[31] Therefore, because of the similar reactivities displayed by oxirane and epithio compounds, the preliminary studies centered on determining the most suitable surfactant for preventing excessive loss of the thiirane functionality. The investigation utilized simulated emulsion conditions whereby 2%, based upon weight of the monomer, of a given surfactant was combined with a 50/50-water/ETPM mixture, and the resulting three-component blend was heated at various reaction temperatures.

The surfactants were selected as representing categories of different acid strengths, i.e. sulfates (Duponol® WAQE), phosphoric acid esters (Gafac® RE-870), carboxylate (ammonium laurate), and poly(oxyethylene) alcohol (Triton X-405), and the extent of thiirane loss was monitored by means of elemental analysis and infrared spectroscopy. The results of the investigation are presented in *Table* 7.

While virtually no premature ring opening was detected in any of the simulated emulsions at 25 and 45° C, ETPM was found to undergo ring opening polymerization at 65° C when in the presence of anionic surfactants. The use of nonionic surfactants provided the greatest resistance to premature reactivity of the thiirane ring, and aging studies on the water/nonionic surfactant/ETPM emulsion indicated that side reactions or polymerization did not occur after storage for three months at ambient conditions. Therefore, to avoid premature loss of the thiirane functionality, all subsequent studies utilized emulsions prepared with nonionic surfactants.

Determining the Optimum Curing Conditions

Prior to investigating the numerous reaction variables and performance characteristics associated with thiirane-functional emulsions, a curing profile study was conducted to determine the optimum conditions required for efficient crosslinking. The investigation utilized a 56.3/37.6/6.1-ethyl acrylate (EA)/methyl methacrylate (MMA)/ETPM copolymer emulsion which was catalyzed with the stoichiometric amount of piperazine curing agent, and the degree of crosslinking in the samples was monitored by means of tensile strength, percent elongation, and percent insolubles measurements. Based on the data presented in *Table* 8, a curing profile consisting of 30 min at 65° C (150 F) was judged as providing the most optimal conditions for developing efficient crosslinking.

Interestingly, curing temperatures in excess of 65° C resulted in a reduction in tensile strength and also the development of considerable film yellowing. This response may emanate from an undesirable side reaction (i.e., sulfur elimination or sulfide oxidation) which occurs at higher temperatures.

Coatings containing ETPM were found to be sensitive to heat and spontaneously crosslinked in the absence of a curing agent when heated at elevated temperatures. Thus, films of the 56.3/37.6/6.1-EA/MMA/ETPM copolymer emulsion which did not contain a curing agent demonstrated the following increases in tensile strength upon baking for 30 min at the respective temperatures: 65° C = 71 Kg/cm^2, 93° C = 95 Kg/cm^2, 121° C = 96 Kg/cm^2, and 148° C = 131 Kg/cm^2. This response was similar to that displayed by GMA-containing systems which also self crosslink upon heating.[32]

Duponol is a registered trademark of E. I. DuPont deNemours & Co. Gafac is a registered trademark of GAF Corp. Triton is a registered trademark of Rohm and Haas Co.

Table 9—Effect of Increasing the ETPM Concentration on the Physical And Functional Characteristics of the Emulsion

Amount of ETPM (Mole Percent)	Polymer Composition (Weight %)	Yield (%)	Coagulum (%)	Thiirane Content (% initial)	Swelling Ratio	Sulfur Content	
						Theoretical	Actual
0	60/40 EA/MMA	98.9	0.40	—	—	—	—
2	58.1/38.8/3.1 EA/MMA/ETPM	98.1	0.38	100	228	0.63	0.75
4	56.3/37.6/6.1 EA/MMA/ETPM	98.8	0.33	100	60	1.24	1.19
6	54.5/36.4/9.1 EA/MMA/ETPM	98.4	0.24	89	45	1.84	1.57
8	52.8/35.2/12.0 EA/MMA/ETPM	96.5	0.26	71	24	2.42	1.93
10	51.1/34.1/14/8 EA/MMA/ETPM	97.1	0.19	74	15	2.99	2.38
12	49.4/33.0/17.6 EA/MMA/ETPM	96.5	0.28	67	12	3.56	2.71

Determination of the Optimum Concentration of ETPM

The optimum concentration for structurally different crosslinkable monomers may vary considerably as a result of balancing the properties emanating from the physical and chemical characteristics of the monomer, with those properties resulting from the formation of a crosslinked network. Therefore, to determine the level of a crosslinkable monomer required for the development of suitable physical properties, a ladder study was conducted in which the concentration of ETPM in the polymer composition was varied in increments of 2% from 0 to 12 mole percent. In each case, the noncrosslinkable monomer composition consisted of a 60/40 weight ratio of EA to MMA to obtain general application-type coatings properties, and 6%, based on weight of total monomer, of Triton N-101 (a nonylphenoxy polyethoxy ethanol nonionic surfactant with a HLB of 13.8) was used as the polymerization surfactant.

In anticipation of a possible premature loss of the thiirane functionality, a two-step, delayed monomer addition technique was investigated to minimize the residence time that ETPM experienced in the polymerization mixture. The procedure involved dividing the monomer feed into two portions. The first portion, representing 80% of the total monomer charge, contained only conventional, acrylate-type monomers and served as the "seed" latex for the second portion. The second portion of the divided feed, representing 20% of the total monomer charge, contained the remainder of the acrylate-type monomer feed comingled with the total ETPM charge. The more salient features of the emulsions produced in this study are presented in *Table* 9.

Thus, in spite of the preventative measures taken, these data indicate that as the concentration of ETPM increased in the polymer composition, the degree of crosslinking resulting from premature ring opening of the thiirane functionality also increased. The emulsion containing 8 mole percent of ETPM displayed a 29% loss in thiirane content as a result of premature ring opening, and the resulting emulsion particles demonstrated a moderate degree of crosslinking as indicated by a swelling ratio value of 24. The tendency of ETPM to undergo ring opening in the emulsion environment presumably resulted from the presence of sulfate initiator fragments since simulated emulsions of water/nonionic surfactant/ETPM demonstrated no propensity towards premature gelation while under the same conditions.

To establish the concentration of ETPM required for optimum physical properties, the ladder series of ETPM-containing emulsions was combined with the stoichiometric amount of piperazine curing agent (based on the residual thiirane content), mixed for 10 min, applied to glass and Bonderite 37 steel substrates at approximately 2-3 mils dry film thickness, dried at ambient conditions for 24 hr, cured for 30 min at 65° C, and evaluated by means of a battery of typical coatings tests. A portion of the more relevant data for this series of ETPM-containing emulsions is presented in *Table* 10.

While all concentration levels of ETPM demonstrated some property improvement as a result of crosslinking, in general, these results indicate that such properties as film hardness, solvent resistance, and tensile strength passed through a fairly pronounced maximum at approximately 6 mole percent ETPM. The subsequent diminution in physical properties with increasing ETPM concentration was attributed, in part, to the effects associated with higher levels of premature crosslinking. A similar response has been noted in other studies in which coatings with poor integrity were obtained from emulsions crosslinked while in suspended form.[33] As expected, such properties as percent elongation and impact resistance decreased and percent insolubility increased as the concentration of ETPM increased.

As compared to the 60/40-EA/MMA control, films of the emulsion containing 6 mole percent ETPM displayed a 2.5 fold increase in tensile strength, a 3.8 fold increase in solvent resistance, a 2.2 fold decrease in elon-

Table 10—Effects of Increasing the ETPM Concentration On the Physical Properties of Latex Films

Amount ETPM (Mole Percent)	Impact Resistance Forward/Reverse (in-lbs)	Hardness		MEK Double Rubs/Mil	Acetone Insolubles (%)	Elong. (%)	Tensile Strength (Kg/cm^2) after 30 min. Immersion in		
		Sward	Pencil				Initial	H_2O	IPA[a]
0	160/160	10	3B	10-12	0	1359	83	37	NA
2	160/140	10	2B	26	12	775	104	45	9.4
4	120/70	12	B	44	16	701	175	68	9.1
6	80/30	14	2H	46	36	610	213	88	9.0
8	30/15	15	2H	38	45	401	195	94	10.6
10	15/4	14	H	28	52	411	175	78	22.6
12	12/4	14	H	31	54	332	169	72	32.1

(a) IPA = Isopropyl alcohol.

gation, reduced flexibility, and increased film hardness (2H vs 3B). While only minor differences were apparent in the performance characteristics of coatings containing from 4 to 8 mole percent ETPM, the emulsion containing 6 mole percent ETPM was selected as being the most suitable candidate for further evaluation.

Determination of the Most Effective Curing Agent

To investigate the effects that compositionally different crosslinking agents had on the physical properties of the system, and to partially optimize the system for further evaluation, several chemically and structurally different curing agents were evaluated at the stoichiometric level (based upon the remaining thiirane content) using the emulsion containing 6 mole percent ETPM. The samples were processed in an identical manner to those of the previous study, and the results of the evaluation are presented in *Table* 11.

Of the test measurements employed in this evaluation, the formation of a network structure is reflected best by increases in tensile strength, solvent resistance, and to a lesser extent film hardness. Based upon these criteria, the ability of the various curing agents to promote effective crosslinking decreased according to the following order: piperazine > diethylenetriamine > 1,6-hexanediamine ≃ Jeffamine D-230 > none ≃ terephthalic acid ≃ adipic acid.

Interestingly, the order of effectiveness among the various amine curing agents supported the conclusion drawn from kinetic studies where the rate of thiirane ring cleavage was found to be a function of the basicity of the amine. Thus, piperazine demonstrated the greatest increases in those properties which reflect the formation of a network structure. As compared to the identical emulsion tested without a curing agent, the samples containing piperazine displayed a 2.5 fold increase in tensile strength, a 2.3 fold increase in solvent resistance, a 2.7 fold decrease in elongation, decreased impact resistance, and increased film hardness (2H vs 2B).

Besides being effective at elevated temperatures, piperazine also possessed sufficient activity to promote crosslinking at ambient conditions. For example, samples containing the stoichiometric amount of piperazine were found to develop the following tensile strengths as a function of time: 62 Kg/cm^2 after one day, 97 Kg/cm^2 after three days, 132 Kg/cm^2 after five days, and 144 Kg/cm^2 after eight days. Thus, after drying at ambient conditions for eight days, films containing piperazine displayed a 2.3 fold increase in tensile strength.

Table 11—Effects of Various Crosslinking Agents on the Physical Properties of ETPM-Containing Emulsions[a]

Curing Agent	Impact Resist. Forward/Reverse (in-lbs)	Hardness: Sward Initial	Hardness: Sward After H_2O Immersion[b]	Hardness: Pencil Initial	Hardness: Pencil After H_2O Immersion[b]	Crosshatch Adhesion (%) Initial	Crosshatch Adhesion (%) After H_2O Immersion[b]	MEK Double Rubs/mil	Tensile Strength (Kg/cm^2)	Elong. (%)
None	160/160	8	8	2B	6B	100	0	20	72	1162
Adipic acid	160/160	8	8	H	6B	29	0	24	43	577
Terephthalic acid	160/160	6	6	3B	6B	100	0	20	65	542
Jeffamine® D-230[c]	160/155	8	6	B	6B	100	0	30	115	513
1,6-hexanediamine	160/160	12	6	H	4B	100	0	38	104	467
Diethylenetriamine	160/155	10	4	HB	5B	100	5	40	143	557
Piperazine	80/30	14	8	2H	B	100	0	46	182	422

(a) All samples were cured 30 min at 65°C.
(b) Samples were immersed for 1 hr.
(c) Jeffamine D-230 is a polyoxypropylenediamine with 2.6 oxypropylene units per molecule, total acetylatables of 8.75 meq/g, and a primary amine value of 8.30 meq/g. Jeffamine is a registered trademark of the Jefferson Chemical Co. Inc.

Table 12—Comparison of the Properties of ETPM-and GMA-containing Emulsions[a]

				Hardness				Crosshatch Adhesion (%)				
				Sward		Pencil						
Cross-linkable Monomer	**Distribution of Cross-linking sites**	**Curing Agent**	**Impact Resist. Forward/ Reverse (in-lbs)**	**Initial**	**After H_2O Immersion[b]**	**Initial**	**After H_2O Immersion[b]**	**Initial**	**After H_2O Immersion[b]**	**MEK Double Rubs/ mil**	**Tensile Strength (kg/cm²)**	**Elong. (%)**
ETPM	Shell area	Piperazine	80/30	14	8	2H	B	100	0	46	182	422
	Random throughout	Piperazine	160/145	10	7	B	6B	100	0	57	143	320
	Shell area	DETA	160/155	10	4	HB	5B	100	5	40	143	557
	Random throughout	DETA	160/160	8	6	3B	6B	100	0	40	124	674
GMA	Shell area	Piperazine	160/160	10	4	2B	6B	0	0	27	191	510
	Random throughout	Piperazine	160/140	10	6	4B	6B	0	0	32	170	352
	Shell area	DETA	160/160	5	4	4B	6B	0	0	14	135	646
	Random throughout	DETA	160/160	6	4	5B	6B	0	0	62	122	840

(a) All samples were cured 30 min at 65° C.
(b) Samples were immersed for 1 hr.

Finally, the physical properties evaluation indicated that little or no crosslinking took place when dicarboxylic acids were used as the curing agents. While acetic acid does add to the thiirane ring, the reaction proceeds at a slower rate than with aliphatic amines.[23]

Comparison of Thiirane- and Oxirane-containing Emulsions

To obtain greater insight into the various advantages and limitations possessed by thiirane-functional emulsions, a comparative study was conducted in which the crosslinking effectiveness of similar oxirane- and thiirane-containing emulsions was examined by monitoring the physical property changes which occurred upon curing. The study utilized emulsions containing 6 mole percent ETPM or GMA, respectively, and the remaining monomer composition consisted of a 60/40 weight ratio of EA to MMA. To ascertain the effects associated with the topographical location of the crosslinking segments, the emulsions were prepared using either a two-step, delayed monomer addition technique (which yielded particles possessing a shell region enriched with crosslinkable functionality) or a continuous, single feed technique (which yielded particles displaying a more or less statistical distribution of crosslinkable functionality throughout the volume of the particle). The samples contained the stoichiometric amounts of piperazine on diethylenetriamine (DETA) curing agents and were processed in a manner identical with the previous studies. A portion of the more relevant data is presented in *Table* 12.

While both sets of crosslinkable emulsions displayed similar increases in those performance characteristics which reflected the formation of a network structure, in general, films of the ETPM-containing emulsions tended to display somewhat lower tensile strengths and elongation but greater solvent resistance and hardness than coatings of the GMA-containing emulsions. For example, utilizing those data which illustrate the greatest differences between the two systems, films of the ETPM-containing emulsions demonstrated approximately 16% lower tensile strength, approximately 20% lower elongation, 285% greater solvent resistance, and considerably greater hardness (2H vs 2B) than films of the GMA-containing emulsions. Aside from these results, the most apparent property differences associated with the two methods of crosslinking involved the tendency of films containing ETPM to yellow upon baking, together with the propensity of the thiirane-containing monomer to exhibit a greater degree of ring opening during the polymerization process. Conversely, films of the GMA-containing emulsions remained water white upon baking, and the oxirane-containing monomer displayed greater resistance to premature crosslinking during the synthesis process. Interestingly, once prepared, emulsions containing either GMA or ETPM displayed similar resistance to premature crosslinking upon aging *Table* 13. Therefore, based upon its similar reaction and performance capabilities together with its propensity towards

Table 13—Aging Characteristics of GMA- and ETPM-Functional Emulsions

	Swelling Ratios	
Aging Period (days)	**6% GMA**	**6% ETPM**
1	71	60
7	70	63
14	56	69
28	62	59

yellowing and premature crosslinking, ETPM was judged as being somewhat inferior to GMA in its capacity to generate a product with the characteristics required by the industrial coatings industry.

PROJECT OVERVIEW

In summary, a feasibility study was conducted which investigated the suitability of using thiirane-functional monomers as a means of developing crosslinking emulsions. To obtain polymer with pendent thiirane functionality, 2,3-epithiopropyl methacrylate (ETPM) was prepared in 39% yield using the oxygen-sulfur exchange reaction between glycidyl methacrylate (GMA) and potassium thiocyanate.

To prevent premature loss of the thiirane functionality, the model emulsions were prepared at low temperatures using nonionic surfactants and a two-step, delayed addition of thiirane monomer. However, in spite of the preventative measures taken, analysis for the remaining thiirane content indicated that as the concentration of ETPM increased in the polymer composition, the degree of premature crosslinking also increased. To avoid the undesirable effects associated with higher degrees of premature crosslinking and to obtain coatings with an optimum balance of physical properties, the 54.5/36.4/9.1-EA/MMA/ETPM copolymer emulsion (6 mole percent ETPM) was chosen as the most suitable candidate for further evaluations.

While emulsion coatings containing ETPM were found to crosslink at room temperature when piperazine was used as a curing agent, more suitable physical properties were obtained by thermally treating the samples for 30 min at 65° C.

When the model emulsions containing 6 mole percent ETPM were combined with various dicarboxylic acid and polyamine curing agents, the coatings cured with piperazine demonstrated a 2.5 fold increase in tensile strength, a 2.3 fold increase in solvent resistance, a 2.7 fold decrease in elongation, decreased impact resistance, and increased film hardness (2H vs 2B).

While coatings of ETPM-containing emulsions displayed certain advantages over similar coatings derived from GMA-containing emulsions, ETPM was rated as being somewhat inferior to GMA as a result of its tendency towards yellowing and premature crosslinking during preparation of the emulsions.

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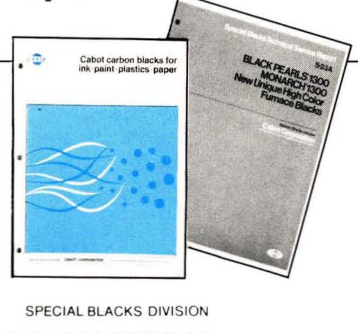

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CSB 105A

Comparison of the Ability Of Selected House Paints To Retard Face-Checking of Plywood

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American Plywood Association*

Plywood specimens finished with various house paints were exposed outdoors for 5.5 years at four geographic locations and to an artificial laboratory test cycle. The performance of the paints during both outdoor and laboratory exposures indicates that certain all-acrylic latex paints decreased both the rate and the degree of face-checking relative to paints which used an organic solvent-thinned primer and acrylic or vinyl-acrylic latex topcoat. Although some all-acrylic latex systems performed better than others, this generic type of finish appears to offer advantages over paints with a solvent-thinned primer and a latex topcoat. Certain stain-blocking acrylic latex systems appeared to be especially effective in reducing face-checking.

INTRODUCTION

Plywood for exterior use is commonly finished in the field with various architectural finishes. Stains, both semi-transparent and opaque, as well as house paints, are used to protect the wood and achieve various aesthetic effects. The degree of protection afforded by these types of finishes varies, with the semi-transparent stains offering the least and two- or three-coat paints the most protection.

Textured plywood accounts for about 20% of the surface area of all siding materials used for new residential construction. Sanded plywood is very commonly used for soffits and for other miscellaneous exterior applications. Thus, paint systems must perform on both types of surfaces.

One of the characteristics of plywood which limits certain applications is its tendency to face-check and crack the finish. Face-checks are small separations between wood fibers in the face veneer, in a direction roughly parallel to the grain.† They are caused by swelling and shrinking stresses which result from variations in moisture content. While they do not affect the structural integrity of plywood in any way, face-checks can sometimes detract from the appearance of the finished product. Rough-sawn surfaces with a stain finish are often used for a more-or-less rustic appearance. Face-checks, in this case, are generally not considered objectionable, since they blend with the texture. However, they can be quite noticeable on surfaces painted with very light or very dark-colored finishes. In these cases, the face-checks contrast in color with the surrounding surface and are quite apparent.

If a consumer desires plywood which is relatively free of face-checks, a Medium Density Overlay is normally recommended. However, there is a need for finishes which will prevent or greatly retard face-checking, since most plywood which is used for exterior applications is not overlaid. Research conducted by the American Plywood Association (APA) indicates that certain all-acrylic latex house paints can greatly retard face-checking relative to other finishes. This report summarizes the results of that research.

METHODS AND MATERIALS

Specimen Preparation and Exposure

Outdoor Exposure: Specimens for outdoor exposure were cut from panels of rough-sawn and sanded plywood of two wood species: Douglas-fir and southern pine.

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† The term "face-check" has traditionally been used by the plywood industry, and it actually applies only to the substrate. It should not be confused with checking of a coating, which is a break that does not completely penetrate the film. However, the face-checks are not usually visible unless they cause the finish to crack. Thus, for the purposes of this report, the term face-check is used to indicate both cracking of the finish and face-checking of the substrate.

Table 1—Description of the House Paint Systems Studied[a]

Finish No.	Finish Manufacturer	Exposure	Description of Finish
1	A	outdoors	Self-priming stain-resistant acrylic latex, two coats.
2	B	outdoors	Stain-blocking acrylic latex primer, one coat; acrylic latex topcoat, two coats.
3	B	outdoors	Two coats of the topcoat described for #2 above.
4	B	outdoors	Same primer and topcoat described for #2 above, but two prime coats and one topcoat were used.
5	B	lab	Same primer and topcoat described for #2 above, but only one coat of each.
6	D	lab	Stain-blocking acrylic latex primer, one coat; acrylic latex topcoat, one coat.
7	E	lab	Acrylic latex primer, one coat; acrylic latex topcoat, one coat.
8	C	outdoors	Long-oil alkyd primer, one coat; acrylic latex topcoat, one coat. Primer contains lead and is no longer available.
9	C	outdoors and lab	Same as #8, but the primer was reformulated to exclude lead.
10	C	outdoors and lab	Linseed oil-base primer, one coat; vinyl-acrylic latex topcoat, one coat. Both primer and topcoat were specially formulated to provide an example of an inferior paint system which might be commercially available.
11	F	lab	Long-oil alkyd primer, one coat; acrylic latex topcoat, one coat.
12	F	lab	Same as #11, but the formulations of both the topcoat and primer were modified to meet the specifications of a large retail store.

(a) All primers and topcoats were white, except the topcoat for finish No. 10 which was beige.

Panels were selected to provide nearly defect-free surfaces. The three-ply rough-sawn plywood was ⅜-in. thick. The five-ply sanded plywood was ⅝-in. thick and was grooved in a Texture 1-11 pattern, grooves 4 in. on center. The surfaces of all panels representing each surface texture were machined with the same equipment.

Finishes were brush-applied to 4 ft × 4 ft plywood panels which were finished in a vertical position to help assure typical application conditions and spread rates. The amount of finish applied to each panel was measured by weighing the paint container and brush before and after finishing.

The outdoor exposure specimens measured 8 in. × 12 in. and were cut from the finished panels. Two replicate specimens from separate panels were prepared for each species/finish/texture condition for each of four exposure sites. All specimens were edge sealed with two coats of an aluminum pigmented urethane. They were exposed during October, 1973 at the following locations: Tacoma, WA; Prosser, WA; Phoenix, AZ; Miami, FL. All specimens were exposed vertically facing south and were inspected every six months for the first 4.5 years and annually after that.

LABORATORY EXPOSURE: Specimens for artificial weathering in the laboratory were prepared from two Douglas-fir plywood panels which were wire brushed to remove the earlywood, leaving only the latewood exposed on the surface. The face veneer of one of these panels represented a moderately fast-growth tree (approximately 16 rings per in.), while that for the other panel represented a slow-growth tree (approximately 50 rings per in.). Specimens measuring 2-⅝ in. × 9 in. were randomly selected from each panel for each paint system. All paints were brush-applied at a spread rate which was within the range suggested by the manufacturer.

The specimens were exposed to the following weekly test cycle: Five days of repeated wetting and drying in a Weather-Ometer® using a four-hour cycle consisting of 18 min water spray and irradiation followed by 222 min of irradiation only; 0.5 hr soak in ice water under 25 in. Hg vacuum; 23.5 hr soak in water at 110 ± 5°F; 24 hr drying by irradiation in the Weather-Ometer. The Weather-Ometer was a 6,500-watt Xenon Arc model equipped with borosilicate inner and outer filters. The maximum ambient drying temperature reached was 150° ± 5° F and the black-panel temperature was 185-190°F. The Weather-Ometer was modified to provide very low humidities during drying by draining all water from the reservoirs in the humidity chamber and by facilitating a rapid draining of all spray water in the exposure chamber. The irradiation level was maintained at 50 ± 1 W/M^2/nm at 340nm. Exposure continued for approximately 12 weeks (1,800 hr in the Weather-Ometer plus a total of 10 days of soaking.)

Test Finishes

Table 1 provides a summary of the characteristics of the finishes which were evaluated. Of the seven finishes which were exposed outdoors, all but acrylic latex system No. 4 were used on all the substrates. Finish No. 4 was applied only to the rough-sawn specimens of both wood species. All but finishes No. 8 and 10 were commercially available and were manufactured by large, well-known paint companies. The primer for finish No. 8 contained lead which had to be removed from architectural finishes due to government regulations. Finish No. 10 was formulated specially for APA to provide an example of a poor-quality finish which might be found on the market.

The Weather-Ometer is a registered tradename of Atlas Electric Devices Co.

Specimen Inspections

The amount of face-checking on the exposure specimens was quantified visually according to a 0-10 scale which is similar to ASTM D-661-44, "Standard Method of Evaluating Degree of Cracking of Exterior Paints"[1]. The higher the number on this scale, the less face-checking; an evaluation of 10 indicates no face-checking whatsoever.

RESULTS AND DISCUSSION

The specimens which were exposed outdoors represented part of a large study which was described previously.[2] The outdoor exposures actually included six wood species, but this report considers only Douglas-fir and southern pine. These two species were selected because they are the dominant wood species which are used for plywood that is finished for exterior exposure. Moreover, they are the most difficult to finish and they illustrate the same trends found for all other wood species.

The four outdoor exposure sites were selected to provide a wide range of exposure conditions. Tacoma is characterized by mild temperatures and lengthy periods of rain, but this exposure also includes cyclic freezing and thawing under very wet conditions. Prosser represents an arid climate with hot, dry summers and cold winters. Phoenix provides high temperatures, high levels of solar radiation, and infrequent periods of heavy rainfall. Miami provides a humid sub-tropical environment.

Outdoor Exposure

Degree of Face-Checking: The face-checking data in *Tables* 2 and 3 compare the various finish systems after 5.5 years of exposure at each of the four outdoor test sites. *Table* 2 provides data for sanded plywood, while the data in *Table* 3 represent the rough-sawn plywood. Each value represents an average of four specimens, two replicates of each wood species.

Table 2—Degree of Face-Checking on Sanded Plywood After 5.5 Years of Exposure[a]

	Finish System					
	All-Acrylic Primer and Topcoats(s)			Solvent-Thinned Primer/Latex Topcoat		
Exposure Site	1	2	3	8	9	10
Tacoma, WA	9.0[b]	9.5[b]	7.5	5.0	5.5	5.8
Prosser, WA	7.8[b]	9.0[b]	6.0	4.2	5.5	5.8
Miami, FL	9.2[b]	8.5[b]	5.8	4.8	4.8	6.8
Phoenix, AZ	7.2[c]	8.2[c]	5.0	3.2	4.5	4.2
Average, all sites	8.3	8.8	6.1	4.3	5.1	5.6

(a) Severity of face-checking is rated on a 0–10 scale, with a value of 10 indicating no checks (see text). Each value represents an average of four specimens, two for each wood species.
(b) Face-checks were observed only on the top and bottom edges of all specimens.
(c) Face-checks were observed only on the top and bottom edges of two of the four specimens.

Regardless of the exposure condition or the surface texture of the plywood, the data in both tables indicate that the all-acrylic latex* paint systems generally reduced the amount of face-checking relative to paints which used a solvent-thinned prime coat. Although the seven finishes which were studied in the outdoor exposures represent only three manufacturers, the laboratory test data which are presented later, as well as other outdoor exposure studies which are currently in progress, indicate the same trend for house paints from several other manufacturers. Moreover, other reports have indicated that all-acrylic latex finishes reduce the degree of face-checking on lumber, relative to solvent-thinned finishes.[3,4,5] Acrylic paint films are generally more flexible than solvent-thinned films, especially after weathering.[5,6,7,8] The acrylic films can, thus, better withstand the dimensional changes in the substrate, and this partially explains their better resistance to face-checking and cracking. *Figure* 1 illustrates the typical appearance of two of the paints after 5.5 years of exposure in Tacoma.

Table 3—Degree of Face-Checking on Rough-Sawn Plywood After 5.5 Years of Exposure[a]

	Finish System						
	All-Acrylic Primer(s) and Topcoat(s)				Solvent-Thinned Primer/Latex Topcoat		
Exposure Site	1	2	3	4	8	9	10
Tacoma, WA	9.0[b]	9.5[c]	9.2[d]	9.8[c]	4.4	4.8	5.8
Prosser, WA	9.2[c]	9.5[c]	8.8[d]	9.5[d]	3.8	5.0	5.2
Miami, FL	9.0[b]	8.8[c]	6.8[e]	9.2[c]	4.0	4.5	6.0
Phoenix, AZ	8.0	8.5	6.5	8.5	3.5	4.0	4.2
Average, all sites	8.8	9.1	7.8	9.2	3.9	4.6	5.3

(a) Severity of face-checking is rated on a 0–10 scale, with a value of 10 indicating no checks (see text). Each value represents an average of four specimens, two for each wood species.
(b) One of the specimens had no face-checks, and face-checking was primarily confined to the top and bottom edges of the others.
(c) Two of the specimens had no face-checks, and face-checking was primarily confined to the top and bottom edges of the others.
(d) Face-checking was observed primarily on the top and bottom edges of all specimens.
(e) Face-checking was fairly severe for two of the specimens (average rating of 4), but was confined to the top and bottom edges of the other two.

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*The term "all-acrylic" is used here to indicate latex paints which used true acrylic polymers for the bulk of the resin in both primer and topcoat. These latexes are to be distinguished from those which use polymers containing vinyl monomers or other non-acrylic monomers. Such latex systems were not evaluated in this study.

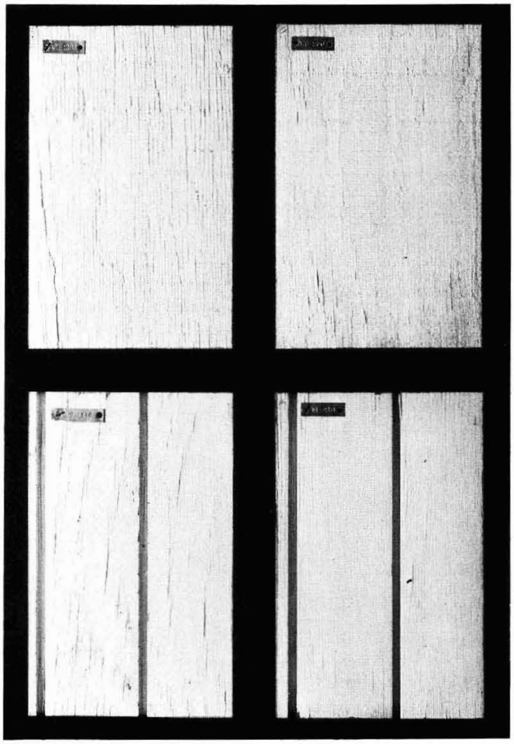

Figure 1—Typical differences between all-acrylic paint system No. 1 and alkyd-primed system No. 9 after exposure for 5.5 years in Tacoma. Finish No. 9 is shown in the left-hand photographs

The exposure data reveal that three of the four all-acrylic latex paints (Nos. 1, 2, 4) were particularly effective in resisting face-checking. In fact, face-checking on most of the specimens which were finished with these three paints was generally confined to the top and bottom portions of the specimens, even after 5.5 years of exposure. The urethane edge seal on the specimens began failing by severe flaking after about three years at all exposure sites. When this failure occurred, the end-grain of the veneers in the specimens was exposed to wetting. The exposed end-grain rapidly absorbs water even during a brief shower or when dew forms. The failure of the sealer probably explains why face-checking occurred first at the top and bottom edges of the specimens. After 5.5 years of exposure, some face-checks in the substrate were visible under the finish in the center portions of some of the specimens which were finished with these all-acrylic systems, but the finish films had not cracked.

The data in *Tables* 2 and 3 indicate rather wide variations in the performance of the acrylic latex paint systems. All-acrylic paint system No. 3 did not perform as well as the other acrylics which were exposed outdoors. While still considerably better in retarding face-checking than the paints with solvent-thinned primers, finish No. 3 allowed considerable face-checking in the center portions of the specimens, as well as near the top and bottom ends. This two-coat finish was the only acrylic system included in the outdoor exposures which was not resistant to staining by water-soluble wood extractives, suggesting that certain physical properties of these stain-resistant latexes may be important in retarding face-checking. Finish No. 1 used two coats of a stain-resistant latex, and it was far superior to finish No. 3. Moreover, three-coat system No. 4 was slightly superior to three-coat system No. 2, *Table* 3, even though both finishes were from the same manufacturer and differed only in the number of prime coats and topcoats; finish No. 4 used two stain-resistant prime coats and one topcoat, while finish No. 2 consisted of one prime coat and two topcoats. The laboratory tests described later provide further indications that stain-resistant acrylic latex finishes are more resistant to face-checking than those formulations which are not resistant to staining.

The ability of the stain-blocking all-acrylic latex paint systems to retard face-checks may relate to the flexibility and continuity of the finish films. Other outdoor exposure studies which are currently in progress also indicate this same trend.

The data in *Tables* 2 and 3 also indicate that face-checking tended to be somewhat more severe in Phoenix than at the other test locations. While this result may be due, in part, to the hot, dry climatic conditions at that site, it may also be due to the particular method which was used to hold the specimens on the test fence. Aluminum channels were used to hold the specimens at their top and bottom edges, and the lower channel trapped water during rainy periods. The bottoms of the test specimens were, therefore, subjected to a very severe wetting condition, and most of the face-checking has occurred on the lower half of the specimens. Drain holes were drilled in these channels after about three years of exposure, but water still collected in the channels because the specimens tended to block water flow.

It is also important to note that the specimens at the Prosser test site were frequently subjected to lengthy periods of water spray from irrigation, resulting in a very harsh exposure from the standpoint of wetting and drying.

INITIATION OF FACE-CHECKING: *Tables* 4 and 5 provide a comparison between finishes relative to their ability to affect the rate of face-checking during outdoor exposure. The data in these tables indicate the interval, in years, which was required to develop some face-checking on all four of the specimens for each finish at each exposure site. Since the specimens were inspected every six months during the first 4.5 years of exposure, the exact time that the first face-check occurred was not recorded. Therefore, if no face-checks were found on any specimens for a given finish at 1.5 years, but all of the specimens had checked by 2.5 years, the data would indicate an interval of 1.5 - 2.5 years.

It is important to note that no face-checking was observed for any of the specimens finished with the all-acrylic systems until more than 1.5 years of exposure had elapsed, even at the most severe test sites. The paint systems which used the solvent-thinned primers, on the

Table 4—Time Interval, in Years, in Which at Least One Face-Check Occurred on All Specimens of Sanded Plywood

	Finish System					
	All-Acrylic Primer and Topcoat(s)			Solvent-Thinned Primer/Latex Topcoat		
Exposure Site	**1**	**2**	**3**	**8**	**9**	**10**
Tacoma, WA	2.0–4.5	4.0–4.5	1.5–3.5	1.0–2.5	0.5–2.5	1.0–2.5
Prosser, WA	2.0–3.0	2.0–3.0	1.5–2.5	0.0–1.0	0.0–1.0	0.0–1.5
Miami, FL	3.0–4.0	3.5–4.0	2.5–3.0	1.0–2.0	1.5–2.0	1.5–2.5
Phoenix, AZ	2.5–3.0	2.5–3.0	2.5–3.0	<2.0[a]	<2.0[a]	<2.0[a]
All test sites	2.0–4.5	2.0–4.5	1.5–3.5	0.0–2.5	0.0–2.5	0.0–2.5

(a) Data were not collected on face-checking at this test site for the first two years. All specimens were heavily face-checked at the two-year inspection.

other hand, all exhibited face-checking within 0 to 3 years of exposure, and all but one specimen had face-checks by 2.5 years. Moreover, the first face-checks on the all-acrylic paints were virtually always observed at the top or bottom of the specimen; whereas, early checking in the center of the specimen also was very common for the other paint systems. The face-checking of the all-acrylic latex systems near the ends of the specimens was probably the result of failure of the edge sealer, as discussed previously.

The face-checking resistance offered by the stain-resistant all-acrylic latex house paints (Nos. 1, 2 and 4) is again evident in the data in *Tables* 4 and 5. With the exception of one specimen at Prosser, Washington, no face-checking was observed for these three paints until more than two years had elapsed. Many of the rough-sawn specimens finished with stain-resistant acrylics still had not face-checked after 5.5 years of exposure at three of the four test sites. In fact, of those specimens on which the first face-check was noted after two to three years, most still did not show face-checking in the center portion of the specimen after 5.5 years.

SUBSTRATE EFFECTS: *Table* 6 shows the average amount of each finish which was applied to the rough-sawn and sanded plywood surfaces for exterior exposure (amount given is the reciprocal of spread rate in square feet per gallon). It is obvious from the data that the rough-sawn surfaces took much more finish. Since the smooth plywood contained grooves 4 in. on center, more finish was applied than would have been if no grooves were present. The grooves in Texture 1-11 plywood are ¼in. deep and ⅜ in. wide and they extend into the center veneer. Thus, the actual differences in spread rate between smooth and rough surfaces are probably even greater than indicated by the data. It is also apparent that much of the extra finish on the rough-sawn surfaces was applied in the first coat. The amount of finish applied for subsequent coats became more similar between the rough-sawn and smooth surfaces. The first coat evidently fills the roughened areas and makes the surface more smooth for following coats.

A comparison of the data in *Tables* 2 and 3 and in *Tables* 4 and 5 indicates that face-checking was retarded better by the all-acrylic paint systems on the rough-sawn surfaces than it was on the sanded and grooved surfaces. These paints probably provide more protection to the rough surfaces because a greater amount of finish is required to adequately cover them. Moreover, the grooves in the sanded surfaces provide a great deal of "edge effect," since they extend through two veneers and into the center veneer. The "edge effect" may hasten face-check formation by allowing more moisture to move into

Table 5—Time Interval, in Years, in Which at Least One Face-Check Occurred on All Specimens of Rough-Sawn Plywood

	Finish System						
	All-Acrylic Primer(s) and Topcoat(s)				Solvent-Thinned Primer/Latex Topcoat		
Exposure Site	**1**	**2**	**3**	**4**	**8**	**9**	**10**
Tacoma, WA	2.5–5.5+[a]	5.0–5.5+[b]	2.5–3.5	5.0–5.5+[b]	0.5–1.5	0.5–1.5	1.0–2.5
Prosser, WA	1.5–5.5+[b]	3.5–5.5+[b]	2.5–3.5	3.0–4.0	0.0–1.0	0.0–1.0	0.0–1.0
Miami, FL	3.0–5.5+[a]	2.5–5.5+[b]	1.5–4.0	2.5–5.5+[b]	0.0–1.0	0.0–2.5	0.0–3.0[c]
Phoenix, AZ	2.5–3.0	2.5–4.5	2.5–3.0	2.5–3.0	<2.0[d]	<2.0[d]	<2.0[d]
All test sites	1.5–5.5+	2.5–5.5+	1.5–4.0	2.5–5.5+	0.0–1.5	0.0–2.5	0.0–3.0[c]

(a) One specimen still had no face-checks after 5.5 years.
(b) Two specimens still had no face-checks after 5.5 years.
(c) One specimen did not face-check until sometime between 2.5 and 3.0 years; all others had face-checked by 2.5 years.
(d) Data were not collected on face-checking at this test site for the first two years. All specimens were heavily face-checked at the two-year inspection.

Table 6—Average Amount of Finish Applied to Sanded and Rough-Sawn Plywood Surfaces[a]

Finish No.	Type of Finish	Sanded Surface First Coat	Sanded Surface Second Coat	Sanded Surface Third Coat	Sanded Surface Total	Rough-Sawn Surface First Coat	Rough-Sawn Surface Second Coat	Rough-Sawn Surface Third Coat	Rough-Sawn Surface Total
		Amount of Finish Applied, gal./1000 ft^2							
1	Acrylic/acrylic	3.77	2.52	—	6.29	5.03	2.55	—	7.58
2	Acrylic/acrylic/acrylic	3.20	2.26	1.95	7.41	4.90	2.66	1.95	9.51
3	Acrylic/acrylic	3.73	2.15	—	5.88	5.34	2.17	—	7.51
4	Acrylic/acrylic/acrylic	—	—	—	—	5.99	3.11	2.37	11.47
8	Alkyd/acrylic	3.30	2.26	—	5.56	4.67	2.68	—	7.35
9	Alkyd/acrylic	4.06	2.53	—	6.59	5.85	3.42	—	9.27
10	Linseed/vinyl-acrylic	4.13	3.00	—	7.13	5.12	2.84	—	7.96

(a) Each value is the average of four measurements: two replications (different plywood panels) for each of two wood species.

and out of the specimens. It is more difficult to seal the edges of plywood than the face because the end grain of some of the veneers is exposed.

The data in *Tables* 2 and 3 indicate that, when solvent-thinned primers were used, the degree of face-checking on the rough-sawn plywood after 5.5 years is slightly more severe than on the sanded plywood. The additional amount of solvent-thinned primer on the rough-sawn surface is evidently not advantageous, and may even be detrimental. Indeed, general observations indicate that many solvent-thinned systems become more unstable on plywood as more finish is applied. Although the greater amount of solvent-thinned primer might retard moisture movement better, the resultant film evidently becomes too brittle during weathering to accommodate dimensional changes in the substrate, resulting in cracking of the film and face-checking of the plywood. The data in *Tables* 4 and 5 show no significant differences between roughsawn and sanded surfaces for the solvent-thinned primer systems, however, indicating that the surface characteristics had little effect on the time required to form the first face-checks.

Laboratory Tests

Table 7 provides face-checking data for the laboratory tests after 600 hr and 1800 hr of exposure in the Weather-Ometer. As with the outdoor studies, the laboratory test data indicate that the all-acrylic latex paints are generally superior to those which employ a solvent-thinned primer. Moreover, the variablility between the all-acrylic systems is again evident, since finish No. 7 was inferior to acrylic systems No. 5 and No. 6. Finish No. 7 did not utilize a stain-blocking acrylic latex primer, while the other two finishes did, again suggesting that such primers may be very effective in reducing face-checking.

A general comparison of the outdoor exposure data in *Tables* 2 and 3 for finishes 9 and 10 with the laboratory test data in *Table* 7 for the same two finishes suggests that 600 hr of exposure in the laboratory test cycle is roughly equivalent to 5.5 years outdoors for face-checking. The weekly 24-hr wetting/24-hr drying periods used in this test provide a very harsh exposure, since the wood swells and shrinks to nearly the maximum degree. The laboratory test specimens were not edge-sealed or back-sealed, making the exposure particularly severe.

SUMMARY AND CONCLUSIONS

The results from both outdoor exposures and artificial weathering tests indicate that certain all-acrylic latex house paints can greatly retard the development of visible face-checks on plywood relative to house paints which use an organic solvent-thinned primer and a latex topcoat. While some acrylic latex paints performed

Table 7—Degree of Face-Checking On Brushed Douglas Fir Plywood After Exposure to the Laboratory Test Cycle[a]

Exposure Time (hr in Weather-Ometer)	All-Acrylic Primer and Topcoat 5	All-Acrylic Primer and Topcoat 6	All-Acrylic Primer and Topcoat 7	Solvent-Thinned Primer/Latex Topcoat 9	Solvent-Thinned Primer/Latex Topcoat 10	Solvent-Thinned Primer/Latex Topcoat 11	Solvent-Thinned Primer/Latex Topcoat 12
	Finish System						
600	9.5	8.5	6.0	5	3.5	3.5	3.5
1800	7.5	8.0	4.0	2.5	2.0	1.5	3.0

(a) Each value represents an average of two replicate specimens.

DR. JOHN EMERY is a Senior Scientist, Panel Technology Department, Technical Services Division, American Plywood Association. He earned the Ph.D. Degree in Wood Chemistry at Colorado State University in 1972. He is currently conducting research on architectural finishes in order to develop laboratory test methods which will serve as the basis for a performance standard for finishes on plywood sidings.

better than others, and none completely eliminated face-checking, all the systems studied were definitely superior to paints which used a solvent-thinned primer and a latex topcoat. It appears that the degree of face-checking resistance offered by an all-acrylic house paint depends upon the surface characteristics of the plywood and upon certain properties of the paint, such as film elasticity and continuity. The particular stain-blocking acrylic latexes which were studied appeared to be especially effective in reducing face-checking.

More finish was required to cover the rough-sawn than the grooved and sanded plywood. The rough-sawn plywood also showed less face checking when all-acrylic latex paints were used. The increased face-checking of the grooved and sanded plywood relates both to edge effects caused by the grooves and to the lesser amount of finish applied. On the other hand, when paints with solvent-thinned primers were used, the rough-sawn plywood tended to face-check more than the grooved and sanded plywood. This difference may result from the more brittle nature of the thicker solvent-thinned primer on the rough surfaces.

References

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(3) "Acrylic Latex Paints Improve Paintability of Wood," *Southern Lumberman*, September 15, 1971.

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(5) Zeicherman, J. B. and Thomas, R. J., "Scanning Electron Microscopy of Weathered Coatings on Wood," JOURNAL OF PAINT TECHNOLOGY, *44*, No. 570, 88 (1972).

(6) Allyn, G., "A Guide to the Use of Acrylic Paints, Part I: Paint Composition, Definitions, Types," *Architectural Record*, November, 1965.

(7) Schurr, G. G., "Exterior House Paint," *Federation Series on Coatings Technology, Unit 24*, Federation of Societies for Coatings Technology, Philadelphia, PA 1977.

(8) Shur, E. G. and H. Rubin, "Accelerated Testing of Finishes on Hardboards," JOURNAL OF PAINT TECHNOLOGY, *41*, No. 537, 537, (1969).

United Kingdom Orders of FSCT Educational Literature

In the United Kingdom, Federation publications and other industry aides are available from the Birmingham Paint, Varnish and Lacquer Club. Please contact: Mr. Ray Tennant, Carrs Paints Limited, Westminster Works, Alvechurch Rd., Birmingham B31 3PG, England.

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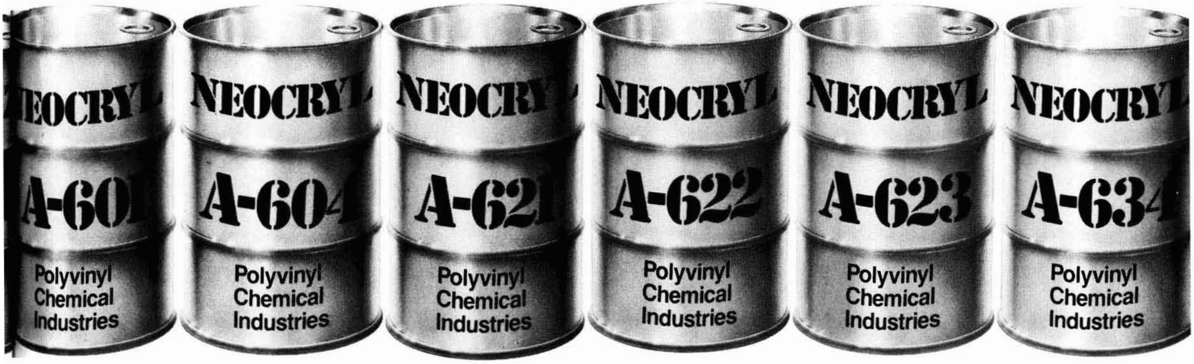
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Society Meetings

Houston Nov. 12

Elio Cohn of Daniels Products Company spoke on "FACTORS GOVERNING TINTER PERFORMANCE."

He discussed the choice of the appropriate dispersing medium, determination of the optimum pigment, solvent selection for the tinter, effect of dispersion equipment, and the interaction of tinter with the tint base. He concluded by reminding the user of the properties of the tinter which govern its performance. These include a wide range of compatibility, no effect upon the drying or performance characteristics of the tinted paint, fineness of grind, maximum color development, and high pigment loading.

K.D. JACOBSON, *Secretary*

Northwestern Nov. 11

Doug Grossman, of The Q-Panel Co., discussed "KNOW THE ENEMY: THE WEATHER."

G. DALE ERNST, *Secretary*

Northwestern (Winnipeg Section) Oct. 21

The speaker was William D. Meadows of Cyprus Industrial Minerals Co. His talk was entitled, "CHLORITE AND TALC AND THEIR COMPARISON TO OTHER EXTENDER PIGMENTS."

FRANCOIS LAFORTUNE, *Secretary*

Dallas Society Officers for the year 1980–81. (Left to right): Society Representative—Carlos Dorris, Jones Blair Co.; Treasurer—T. Leon Everett, Dan-Tex Paint Mfg. Co.; President—Richard Williamson, Trinity Coatings Co.; Vice-President—Ray Marett, Western Specialty Coatings Co.; Secretary—William Wentworth, Jones Blair Co.

New York Nov. 11

Richard Max and Irwin Young, of the Manufacturing Committee, presented a slide presentation on the batch type mini media mill, which will be submitted to the Federation.

The speaker was Ken Haagenson, of Buchman Laboratories. His topic was "COMPETITIVE NON-LEAD, NON-CHROMATE CORROSION INHIBITIVE PIGMENTS IN LATEX PAINTS."

He discussed corrosion and its causes, and non-lead, non-corrosion inhibitors in water thinned coatings in both accelerated and environmental exposures. Using slides he compared different chemicals and their results between laboratory and exterior exposures.

TED YOUNG, *Secretary*

Northwestern Society Executive Committee for the year 1980–81. Seated (left to right): Immediate Past-President—Al Heitkamp; President—Roger Anderson; Vice-President—Dale Ernst; Society Representative—Lowell Wood. Standing (left to right): Treasurer—Herbert Davidson; Secretary—Don Emch; Technical Chairman—Les Ware; Membership Chairman—Lee Sveum; and Rick Brandt

Pacific Northwest Nov. 20

J. Physick, of the Environmental Committee, gave an in-depth report concerning Bill C-18, "Transportation of Dangerous Goods Act", passed in the House of Commons, July 16, 1980. He also discussed Kinetic Contaminants Ltd. and their proposed waste treatment facility to serve Western Canada.

Edward Antonucci, of Drew Chemical Corp., presented "A FORMULATOR'S GUIDE TO SILICONE DEFOAMERS."

ART BRAGG, *Secretary*

Pittsburgh Dec. 1

Jack Cherry, of Rohm and Haas Co., spoke on "BACTERIAL PROBLEMS IN MANUFACTURING AND HANDLING LATEX EMULSIONS."

Mr. Cherry stated that contamination of latex emulsions occurs mainly in two ways: via water-borne and air-borne bacteria. Laboratory methods for testing contamination are all right, he said.

However, there is also a need for filed testing, such as heat aging 10 days at 60°C for preservative stability, 24 week testing at ambient temperature to simulate bulk storage, and also chemical analysis, said Cherry.

He then suggested some of the essential equipment needed for bulk storage of acrylic emulsions and stated the critical elements needed to minimize bacterial contamination of storage facilities. Good housekeeping, he said, is imperative in eliminating contamination. Among his suggestions were: clean tanks twice per year; regular equipment maintenance; back flushing of pumps and lines; periodic sanitization of hose lines and pumps; and quarterly audits of all possible contamination sources.

Q. Would sloped storage tanks be better, for drainage, etc.?

A. Definitely, and, at the same time, agitation is sometimes recommended.

Q. Are high solids emulsions more prone to contamination?

A. Solids should not make a difference if the package is properly designed for the system.

W. Cibulas, *Secretary*

Piedmont Dec. 16

Ronald Rowland of OMYA, Inc. presented, "New Concepts in Calcium Carbonates for the 1980's."

He began with a discussion of the basic concepts of titanium dioxide and how particle size and particle spacing relate to hiding power. He introduced the use of particle space extenders (i.e. calcium carbonates) to present "pigment packing" while retaining efficient hiding power. Slides were used demonstrating typical formulations utilizing calcium carbonates. He presented two noteworthy examples: (1) a water-based styrene-acrylic baking enamel where substitution of a known volume of titanuim dioxide with the carbonate did not sacrifice gloss, hardness, and dry brightness of the cured film; (2) a high-solids catalyzed alkyd-amine baking enamel utilizing a treated ultra-fine carbonate which overcomes problems associated with PTSA acid catalysis and untreated carbonates.

Q. Is the speaker talking about slurry calcium carbonates?

A. No. This talk pertains to dry carbonate only.

Q. What is the price of ultra-fine carbonates in comparison to 3 micron carbonate extenders?

A. Cost is 8 to 11¢ per pound dependent on location in the U.S.

Sara Robinson, *Secretary*

Constituent Society Meetings and Secretaries

BALTIMORE (Third Thursday—Eudowood Gardens, Towson). Donald Keegan, Valspar Corp., 1401 Severn St., Baltimore, MD 21230.

BIRMINGHAM (First Thursday—Warwickshire County Cricket Club). B. J. Addenbrooke, Croda Paints Ltd., Bordesley Green Rd., Birmingham B9 4TE, England.

CHICAGO (First Monday—meeting sites in various suburban locations). Layton F. Kinney, Sherwin-Williams Co., 10909 S. Cottage Grove, Chicago, IL 60628.

C-D-I-C (Second Monday—Sept., Nov., Jan., Mar., May in Columbus; Oct., Dec., Feb., Apr., in Cincinnati, Kings Island Inn). Nelson W. Barnhill, Inland Div., G.M.C., P.O. Box 1224, Dayton, OH 45401.

CLEVELAND (Second Tuesday—meeting sites vary). Carl J. Knauss, Kent State University, Kent, OH 44242.

DALLAS (Thursday following second Tuesday—Vic's Gallery Restaurant). William A. Wentworth, Jones-Blair Co., P.O. Box 35286, Dallas, TX 75235

DETROIT (Fourth Tuesday—meeting sites vary). Mike King, U.S. Army TACOM, 765 Dellwood Dr., Ann Arbor, MI 48103.

GOLDEN GATE (Monday before third Wednesday—Sabella's Restaurant, San Francisco). F. Robb Holt, Sherwin-Williams Co., P.O. Box 23505, Oakland, CA 94623.

HOUSTON (Second Wednesday—Sonny Look's, South Main). Klebert Jacobson, Cron Chemical Corp., P.O. Box 14042, Houston, TX 77021.

KANSAS CITY (Second Thursday—Cordon Bleu). Mike Bauer, Tnemec Co., Inc., P.O. Box 1749, Kansas City, MO 64141.

LOS ANGELES (Second Wednesday—Steven's Steak House). Lloyd Haanstra, Ameritone Paint Corp., P.O. Box 190, Long Beach, CA 90801.

LOUISVILLE (Third Wednesday—Essex House). John Lanning, Porter Paint Co., 400 S. 13th St., Louisville, KY 40203.

MEXICO (Fourth Thursday—meeting sites vary). Juan Irazabal, Grafex De Mexico, S.A., Calz. Tulychaulco 4615, Mexico, D.F., Mexico.

MONTREAL (First Wednesday—Bill Wong's Restaurant). G.L. Simpson, Sico, Inc., 3280 Blvd. St. Anne, Quebec, P.Q., Canada G1E 3K9.

NEW ENGLAND (Third Thursday—Fantasia Restaurant, Cambridge). Fran Koebert, Kyanize Paints, Inc., Second & Boston Sts., Everett, MA 02149.

NEW YORK (Second Tuesday—Landmark II, East Rutherford, NJ). Ted Young, Jesse S. Young Co., Inc., P.O. Box 275, Hewlett, NY 11557.

NORTHWESTERN (Tuesday after first Monday—Edgewater Inn). Don Emch, Valspar Corp., 1101 3rd St. S., Minneapolis, MN 55415.

PACIFIC NORTHWEST (Portland Section—Tuesday following second Wednesday; Seattle Section—the day after Portland; British Columbia Section—the day after Seattle). Robert Miller, Imperial Paint Co., 2526 N.W. Yeon, Portland, OR 97210.

PHILADELPHIA (Second Thursday—Valle's Steak House). Wayne Kraus, Lawrence-McFadden Co., 7430 State Rd., Philadelphia, PA 19136.

PIEDMONT (Third Wednesday—Howard Johnson's Coliseum, Greensboro, NC). Sara M. Robinson, Union Oil Co. of Calif., P.O. Box 7129, Charlotte, NC 28217.

PITTSBURGH (First Monday—Skibo Hall, Carnegie Mellon Univ.). William Cibulas, Mobay Chemical Co., Penn Lincoln Pkwy. W., Pittsburgh, PA 15205.

ROCKY MOUNTAIN (Monday prior to second Wednesday—Gusthaus Ridgeview, Wheatridge, CO). Donald R. Bagge, George C. Brandt, Inc., 6500 Stapleton Dr. S., Denver, CO 80216.

ST. LOUIS (Third Tuesday—Salad Bowl Restaurant). Joseph J. Wrobel, Jr., CIBA-GEIGY Corp., P.O. Box 26653, St. Louis, MO 63122.

SOUTHERN (Gulf Coast Section—Second Tuesday; Central Florida Section—Thursday after third Monday; Atlanta Section—Third Thursday; Memphis Section—Second Tuesday). William G. Early, Piedmont Paint Mfg. Co., P.O. Box 6623, Station B, Greensville, SC 29606.

TORONTO (Second Monday—Town and Country Restaurant). A. Suk, K-G Packaging Ltd., P.O. Box 658, Concord, Ontario, Canada L4K 1C7.

WESTERN NEW YORK (Second Tuesday—Holiday Inn, Cheektowaga, NY). Charles S. Glinski, Pierce & Stevens Chemical Corp., 710 Ohio St., Buffalo, NY 14240.

Future Society Meetings

Baltimore

(Mar. 12)—Manufacturing Committee program.

(Mar. 13)—MINITRADE SHOW. Pikesville Hilton.

(Apr. 16)—Education Committee program.

(May 21)—Technical Steering Committee program on PAINT APPLICATION METHODS.

Birmingham

(Mar. 5)—"MARKETING IN THE EIGHTIES"—P. Harland, Croda Paints Ltd.

(Apr. 2)—"SOME ASPECTS OF AMINO RESINS"—J.E. Sreeves, B.I.P. Ltd, Chemical Div.

(May 7)—"COATING WINDOW FRAMES"—G.W. Rothwell, Building Research Establishment.

Chicago

(Mar. 2)—"HARD LATEX POLYMER MODIFICATION OF WATER-SOLUBLE ALKYDS"—Nick Roman, Rohm and Hass Co.; "FSCT CORRESPONDENCE COURSE"—John Gordon, Univ. of Missouri—Rolla.

(Apr. 6)—FSCT SLIDE SHOW—Federation Officers; "MULTI-FUNCTIONAL EPOXIDES"—M. Gaschke, CIBA-Geigy Corp.

(May 9)—AWARDS NIGHT. Installation of 1981-82 Officers.

C-D-I-C

(Mar. 9)—FSCT SLIDE SHOW, Federation Officers.

(Apr. 13)—PLANT TOUR of Battelle Memorial Institute.

(May 11)—"PIGMENT DISPERSION AND MILLING IN THE PAINT AND RELATED INDUSTRIES"—Edward Carter, Morehouse Industries, Inc.

Golden Gate

(Mar. 16)—"SHORT TERM EVALUATION TECHNIQUE FOR LIQUID AND PARTICLE BEHAVIOR IN COATING SYSTEMS"—Frank Zurlo, Byk-Mallinckrodt Chem Prod.

(Apr. 13)—"POLYMERIC ORGANIC DISPERSANTS FOR PIGMENTS—PRINCIPLES AND PRACTICES"—Robert D. Athey, Jr., Swedlow, Inc.

(May 18)—"PLANT DESIGN—BUILD NEW, REDESIGN OLD"—Jim White, Hockmeyer Equipment Corp.

(June 15)—Manufacturing Committee Seminar, "SAFETY AND GOVERNMENT REGULATIONS."

Houston

(Mar. 11)—PAST-PRESIDENTS' NIGHT

(Apr. 8)—"CHLORITE: A NEW FAMILY OF PAINT EXTENDERS"—William Meadows, Cyprus Industrial Minerals.

Los Angeles

(Mar. 11)—"SHORT TIME EVALUATION TECHNIQUE FOR LIQUID AND PARTICLE BEHAVIOR IN COATINGS SYSTEMS"—Frank Zurlo, Byk Mallinckrodt, Chem Prod. GmbH.

(Apr. 8)—"POLYMERIC ORGANIC DISPERSANTS FOR PIGMENTS: PRINCIPLES AND PRACTICES"—Robert D. Athey, Jr., Swedlow, Inc.

(May 12)—"PLANT DESIGN: BUILD NEW—REDESIGN OLD"—Jim White, Hockmeyer Equipment Corp.

Montreal

(Mar. 4)—"SELLING TECHNIQUES"—Graham Oliver, McGill Univ.

(May 7)—PAST-PRESIDENTS' NIGHT

(May 9)—50TH ANNIVERSARY BALL. Ritz Carlton Hotel

New England

(Mar. 19)—JOINT INDUSTRY MEETING.

(Mar. 24)—JOINT MEETING with Society of Plastic Engineers; "MICROBIOLOGICAL DETERIORATION OF PLASTIC AND COATINGS"—Susan Tamborini, Ventron.

New York

(Mar. 10)—MINIWORKSHOPS ON "ZERO EFFLUENCE"; "TYPES AND FUNCTIONS OF ADDITIVES"; "ADHESION OF LATEX PAINTS"; SALES AND MANAGEMENT TOPIC; and "COMPUTER AS A TOOL IN PRODUCTION."

(Apr. 14)—"DISPERSION EQUIPMENT"—Panel Discussion.

(May 12)—PAVAC AWARD NIGHT.

Philadelphia

Technical Committee Meetings

(Mar. 5)—"NEW GENERATION COATINGS—APPLICATION WORKSHOP: PART I—WATER-BORNE AND HIGH SOLIDS"—speakers from Cargill Chemical Products Div. and Nordson Corp.

(Apr. 2)—"NEW GENERATION COATINGS—APPLICATION WORKSHOP: PART II—TWO COMPONENT AND POWDER"—speakers from Mobay Chemical, Polymer Corp., and Nordson Corp.

(May)—Society Technical Committee Seminar on "INTERFACES REVISITED."

Pittsburgh

(Mar. 2)—"PIGMENT DISPERSION—WHAT DOES IT MEAN?"—James D. Easton, Harshaw Chemical Co.

(Apr. 6)—"KNOW YOUR ENEMY—THE WEATHER"—George W. Grossman, The Q-Panel Co.

(June 1)—"ART AND ALCHEMY"—John C. Pavlik, Fisher Scientific Co.

Nominees Solicited for 'Golden Impeller Award'

Morehouse Industries, Inc., Fullerton, CA, announces a continuation of the Golden Impeller Award. This award is presented yearly to an individual in recognition and appreciation for outstanding service to the chemical processing industry for innovative achievements in the field of dispersion technology.

An independent board of judges will review nominees for the 1981 Golden Impeller Award which will be presented during the Annual Meeting of the Federation of Societies for Coatings Technology, October 28–30, 1981 at Cobo Hall in Detroit. On the board of judges for the current year are Albert Seneker, of Ameron; Michael W. Malaga, of Glidden Coatings & Resins Div.; Dr. Zeno W. Wicks, Jr., of North Dakota State University; Frederick K. Daniel, of Daniel Products Co.; and Temple C. Patton, retired from NL Industries, Inc. Previous winners of the Golden Impeller Award are Hugh F. Purcell (1978), Frederick K. Daniel (1979), and Temple C. Patton (1980).

Anyone wishing to nominate a candidate for the award may do so by submitting the person's qualifications to Mr. Albert Seneker, Ameron Research and Development, 4813 Firestone Blvd., South Gate, CA 90280, by April 1, 1981.

Elections

BALTIMORE

Active

Burke, John—Duron, Inc., Beltsville, MD
Kamasinski, Leo J.—Lenmar, Inc., Baltimore, MD
Lee, Oswald H.—Duron, Inc., Beltsville
Lenzi, Patrick A.—McCorquodale Colorcard, Whiteford, MD
Sorrow, Steven J.—Hanline Paints, Baltimore.
Welsh, Martin—Duron, Inc., Beltsville.

Associate

Gubicza, Andrew J.—Alco Chemical Corp., Philadelphia, PA

CHICAGO

Active

Bellini, Frank—Rusco Perma Coatings, Melrose Park, IL
Bromley, Ray W.—Pearl Paints of N.A. Ltd., Chicago, IL
Cook, Robert L.—Sun Chemical Corp., Northlake, IL
Lyyski, Jon M.—Midland Dexter, Waukegan, IL
Nieder, Scott E.—Pioneer Paint Prod, Inc., Melrose Park.
Plaisance, Thomas H.—DeSoto, Inc., Des Plaines, IL
Shah, Ashok A.—Valspar Corp., Chicago.
Sikorski, Ursula—DeSoto, Inc., Des Plaines.

Associate

Daniels, Jack—Cargill, Inc., Carpentersville, IL
Dichter, Andy—Spencer Kellogg Div., Des Plaines, IL
Hopson, William S.—Whittaker Clark & Daniels, Willowbrook, IL
Juggins, John E.—American Cyanamid Co., Bolingbrook, IL
Larsen, Diane—Cargill, Inc., Carpentersville, IL
McCarthy, David—G.R. O'Shea Company, Westchester, IL
Talbot, John P.—Morehouse Industries, Lombard, IL
Tolle, Thomas M.—Burgess Pigment Co., Geneva, IL

DALLAS

Active

Morgan, William E.—Southwestern Petroleum, Fort Worth, TX
Suarez, Rick—DeSoto, Inc., Dallas, TX
Wilson, Lloyd—Gillespie Coatings, Inc., Longview, TX

Associate

Walraven, Wally—Ashland Chemical, Forney, TX

HOUSTON

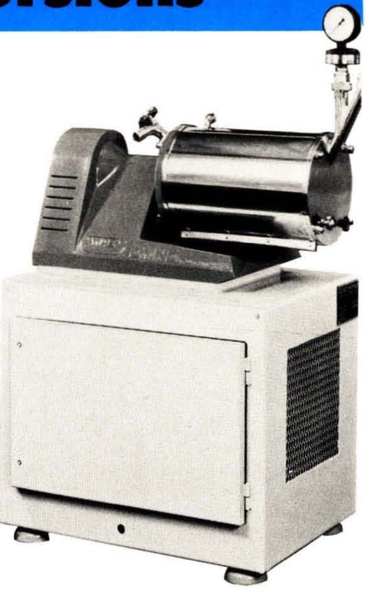
Active

Raska, Joseph G.—Tex. Dept. Hwys. Pub. Tr., Austin, TX
White, Henry E. Jr.—Cook Paint & Varnish, Houston, TX

KANSAS CITY

Active

Svoboda, James P.—Cook Paint & Varnish Co., Kansas City, MO



LOUISVILLE

Active

Brawner, Chas. R.—Devoe & Raynolds Co., Louisville, KY

Craig, Wallace M., Jr.—Louisville Varnish Co., Louisville.

Kimmel, Stanley—Progress Paint Co., Louisville.

Moody, Keith M.—Reliance Universal, Inc., Louisville.

Sturtzel, Patricia H.—Porter Paint Co., Louisville.

Associate

Baltaragis, Ray—Borden Chemical, Inc., Illiopolis, IL

Sheets, Phil—Penn Color Corp., Prospect, KY

Wamelink, John—Borden Chemical, Brecksville, OH

Williams, John P.—Genpak Corp., Cincinnati, OH

NEW ENGLAND

Active

Conti, Michael—Durant Paint Co., Revere, MA

Hanninen, Robert—Raffi and Swanson, Wilmington, MA

Hendriks, Eugene P.—AW Chesterton Co., Groveland, MA

Kapasi, Vikram C.—Compo Ind., Bradford Div., Lowell, MA

Kiezulas, Margaret P.—Aguadilla, PR

Villani, Dante J.—General Electric Co., Chelsea, MA

Associate

Delack, Robert J.—Delack Associates, Wayland, MA

Williams, Bronwen P.—Ferro Chemical, Amherst, NH

NEW YORK

Active

Accamando, Thomas J.—Everseal Mfg., Ridgefield, NJ

Associate

Bergen, Joseph F.—Reichard-Coulston, Inc., New York, NY

Blacker, Roger P.—Whittaker Clark & Daniels, South Plainfield, NJ

Boreham, Malcolm D. Jr.—Sun Chemical Corp., Staten Island, NY

Craffey, James F.—Pfizer, Inc., MPM Div., Clifton, NJ

Hasel, Craig A.—Cellofilm Corp., Wood-ridge, NJ

Nassberg, Bernard—Reichard-Coulston, Inc., Matawan, NJ

Palmer, Joseph M.—Modern Paint & Coatings, New York.

Vyskocil, Emil J.—Peltz Rowley Co., Inc., Rockville Centre, NY

PACIFIC NORTHWEST

Active

Aquila, Aldo—Synkoloid of Canada, Vancouver, B.C., Can.

Berg, Roald K.—Associated Chemists, Inc., Portland, OR

Foster, Terry—Drep Fmo Esquimalt, Victoria, B.C., Can.

Lo, Vernon—Stuart Plastics Ltd., New Westminster, B.C., Can.

Metheany, Jon A.—Associated Chemists, Inc., Portland, OR

Nishi, Tatsuo—Asahipen America, Inc., Seattle, WA

Park, David W.—The Kirk Co., Puyallup, WA

Petty, Don—Willamette Valley Co., Eugene, OR

Pizarro, Rodrigo A. Jr.—Forrest Paint Co., Eugene.

Walker, Maurice—Walker Bros. Ltd., Burnaby, B.C., Can.

Associate

Cettie, Ron—Abco Rainier, Mukilteo, WA

Galbraith, Ian T.—Wilson & George Meyer & Co., Beaverton, OR

Hagedorn, Allen T.—Union Chemicals Div., Vancouver, WA

King, Allison A.—Ashland Chemical Co., Beaverton.

Lakin, Dennis R.—J. F. Shelton Co., Portland, OR

Norton, Stephen R.—Tenneco Chemicals, Portland.

White, Kenneth Ray—Nalco Chemical Co., Concord, CA

PHILADELPHIA

Active

Caldwell, Richard A.—N L Industries, Inc., Hightstown, NJ

PITTSBURGH

Active

Andritz, Joseph M.—PPG Industries, Springdale, PA
Bucek, George E.—Sterling Div., RCI, Sewickley, PA
Good, Annette L.—PPG Industries, Allison Park, PA
Kelso, Robert G.—Mobay Chemical Corp., Pittsburgh, PA
Taylor, Michelle—PPG Industries, Pittsburgh.

Associate

Gibbs, John W.—Glidden Pigments, Hudson, OH
Strunk, Jay H.—Mobay Chemical Corp., Pittsburgh, PA

ROCKY MOUNTAIN

Active

Dillon, David E.—Kelly-Moore Paint Co., Denver, CO
O'Connor, Cory—Ashland Chemical, Englewood, CO

Associate

Halvorsen, Fredalyn—Chemical Sales Co., Denver, CO
Klail, Al—Celanese Plastics & Specialties, San Ramon, CA
Peckham, Ray—Oil & Solvent Process, Adams City, CO

ST. LOUIS

Active

Reckel, William L.—Amteco, Inc., St. Louis, MO
Sandro, Amerio—Ideal Stencil Mach., Belleville, IL

Associate

Held, Merle D.—Cyprus Ind. Minerals, Clayton, MO

TORONTO

Active

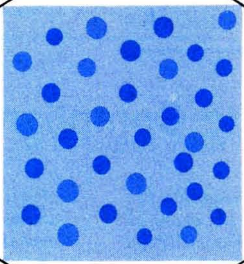
Agostino, James—K.G. Packaging Ltd., Concord, Ont., Can.
Amiel, Pat—Lorcon, Inc., Resins Div., Milton, Ont., Can.
Bailey, Rusty—Abrex Specialty Coatings Ltd., Oakville, Ont., Can.
Birkett, Arleish M.—C-I-L Paints, Inc., Toronto, Ont., Can.
Briston, Richard—K-G Packaging, Ltd., Concord.
Campbell, Donald H.—Para Paints Ltd., Toronto.
Chau, Francis—K-G Packaging Ltd., Concord.
Chincholi, Basappa—C-I-L Paints, Inc., Weston, Ont., Can.
Chung, Yoon-Sang—C-I-L Paints, Inc., Concord.
Debling, Gary A.—Custom Colour Paints, Brampton, Ont., Can.
Fortier, Michael—Monarch Coatings Ltd., Rexdale, Ont., Can.
Gowan, Gerald—C-I-L Paints, Inc., Toronto.
Gruber, Alex—Reichhold Ltd., Toronto.
Hambly, Jim—Phillips Paint Ind., Ajax, Ont., Can.
Hazen, Michael W.—L.V. Lomas Chemical Co., Mississauga, Ont., Can.
Henderson, J.B.—duPont Canada Ltd., Whitby, Ont. Can.
Johnston, Dennis J.—Allcolour Paint & Chem., Oakville, Ont., Can.
Johnstone, John A.—C-I-L Paint, Inc., Concord.
Kohler, Robert—Abrex Specialty Coatings, Oakville.
Laughlin, Bert A.—Sterling Varnish Co. (Canada) Ltd., St. Catherines, Ont., Can.
Madonia, Paul—C-I-L-Paint, Inc., Toronto.
Marshall, Larry—Phillips Paints Ind., Ajax.
Martin, Kenneth—K-G Packaging Ltd., Toronto.
Mason, Colin P.—Bate Chemical Co. Ltd., Don Mills, Ont., Can.
Noseworthy, Tom—Almatex Chemical Coatings, London, Ont., Can.
Palleschi, Sandy—Serif Coatings & Chemicals, Inc., Mississauga.
Perlus, Tibor G.—Consultant, Islington, Ont., Can.
Rana, Vikram—Ashland Chemical, Mississauga.
Rowe, Peter G.—P.G. Rowe, Consultant, Scarborough, Ont., Can.
Shillingford, William—Abrex Specialty Coatings, Oakville.
Siple, Steve—Phillips Paint Ind., Ajax.
Tappen, Harry—PPG Industries (Canada), Toronto.
Wason, Robin—XYZ Protective Coatings, Cambridge, Ont., Can.
Wong, Thomas S.—XYZ Protective Coatings, Cambridge.

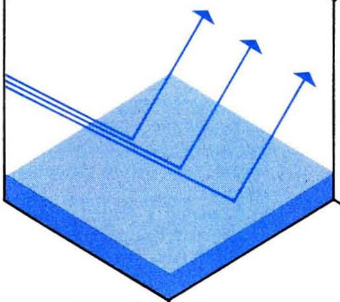
Associate

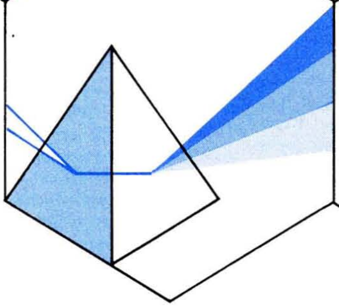
Baker, Michael J.—GAF (Canada) Ltd., Mississauga, Ont., Can.
Collins, Chuck—Ashland Chemicals, Mississauga.
Durocher, Jean P.—Esso Chemical, LaSalle, Que., Can.
Graham, William R.—Chemroy Chemicals Ltd., Mississauga.
Jenner, Rick—Canada Colours & Chemicals, Toronto, Ont., Can.
Maraghi, R.—Bayer (Canada), Inc., Mississauga.
Philpott, Cory—Tioxide of Canada, Inc., Etobicoke, Ont., Can.
Smith, Ian H.—C-I-L Paints, Inc., Willowdale, Ont., Can.
Trapman, Peter—Dominion Colour Co. Ltd., Toronto.
Wig, Vicki L.—Esso Chemical Canada, Toronto.

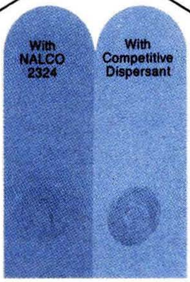
Educator and Student

Alkin, Zafer—Humber College, Rexdale, Ont., Can.
Tieu, Franklin—Ryerson Polytechnic Institute, Toronto, Ont., Can.
Zymiak, Barbara—Ryerson Polytechnic Institute, Toronto.








With NALCO 2324
With Competitive Dispersant

Checklist for Hazardous Waste Compliance

For those who are hazardous waste generators, transporters, or owners/operators of a hazardous waste treatment, storage, or disposal (TSD) facility and are not exempted by the Federal Regulation, the following checklists are suggested by National Paint and Coatings Association as a quick reference for the major action that must be taken in order to be in compliance with this new federal program.

Generators

- ☐ Must have notified EPA of hazardous waste activity and obtained an EPA ID number.
- ☐ Must utilize a manifest for all hazardous wastes transported off site.
- ☐ Must comply with all EPA and DOT requirements for packaging, labeling, marking and placarding before off-site transport of hazardous waste and on-site storage.
- ☐ Must have established recordkeeping system for manifests with three-year retention time.
- ☐ Must determine status of waste if they do not receive a signed copy of the manifest from Owner/Operator (O/O) of designated facility within 35 days from date waste was accepted by original transporter.
- ☐ Must submit Exemption Report to EPA if they do not receive a signed copy of the manifest from O/O of designated facility within 35 days from date waste was accepted by original transporter. Exemption Report must be kept on file for three years.
- ☐ Must keep test results, waste analysis, or other determinations made in accordance with regulations for at least three years.
- ☐ Must submit Annual Report for preceding calendar year by March 1 of each year. Annual Report must be kept on file for three years.

Transporters

- ☐ Must have obtained an EPA identification number.
- ☐ Must utilize manifest system for all hazardous waste shipments except those meeting small quantity generator exemption.
- ☐ Must deliver waste to next designated transporter or designated facility.
- ☐ Must establish recordkeeping system for manifest (or shipping papers with three-year retention time.)
- ☐ Must give notice of any discharge as required by regulations.

Owners/Operators Of Hazardous Waste TSD Facility

- ☐ Must have established interim status by notifying EPA and filing Part A of permit application*. This must have been accomplished by November 19*.
- ☐ Must obtain EPA identification number.
- ☐ Must utilize manifest system for wastes received from off-site generators.
- ☐ Must establish recordkeeping system for manifest (or shipping papers) with three-year retention time.
- ☐ Must submit Annual Report for preceding calendar year by March 1 of each year. Annual Report must be kept on file for three years.

Must meet the following interim status standards:

- ☐ Develop a written waste analysis plan, and before treating, disposing or storing waste, must obtain a detailed analysis of a representative sample of the waste.
- ☐ Comply with security requirements.
- ☐ Develop and follow a written schedule for inspection, maintain an inspection log and establish recordkeeping system for inspection logs with three-year retention time.
- ☐ Establish recordkeeping system documenting training of facility personnel.
- ☐ Implement precautions for ignitable, reactive, or incompatible wastes.
- ☐ Comply with preparedness and prevention requirements, including arrangements made with local authorities to handle emergencies.
- ☐ Prepare contingency plan and submit it to all State and local authorities that may be called upon to provide emergency services.
- ☐ Designate an emergency coordinator to coordinate emergency response measures.
- ☐ Keep a written operating record at the facility.
- ☐ Requirements for a written closure plan and a written estimate of cost of closing the facility have been amended. Plans and cost estimates now are due on May 19, 1981.
- ☐ Must also comply with interim status standards applicable to their specific types of facilities: containers, tanks, surface impoundments, incinerators, etc.
- ☐ Must notify National Response Center of on-scene government coordinator if release, fire, or explosion occurs which could threaten human health, or the environment, outside the facility. Must comply with post-release rules before resuming operation.
- ☐ Must submit a written report on the incident to Regional Administrator within 15 days of release.
- ☐ Must report unresolved significant discrepancies between manifest and shipment to Regional Administrator within 15 days of receiving waste.
- ☐ Must submit copy of operating records to Regional Administrator upon closure of the facility.
- ☐ Must report receipt of unmanifested waste within 15 days.
- ☐ Must submit closure and postclosure plan to Regional Administrator 180 days before closure.
- ☐ Must submit certification of closure upon completion of closure.
- ☐ Must submit survey plan to local land authority and Regional Administrator ninety days after closure is completed.
- ☐ Must, on each anniversary of the effective date, adjust closure and postclosure cost estimate using a specified inflation factor.

*EPA has temporarily suspended permit requirements for certain wastewater treatment units and certain neutralization units. EPA has proposed an amendment to the regulations to permit these facilities by rule only.

Parties who are interested in obtaining interim status for a treatment or storage facility that will handle only paint manufacturing and user wastes which were listed in July 16, 1980 Federal Register but missed the November 19, 1980 deadline had until January 16, 1981 to file Part "A" of permit application and still be eligible for an interim status.

EPA is also planning on issuing amendments to rules concerning establishment of interim status which may help some parties who missed the November 19 filing deadline.

Meetings/Education

Cleveland Society to Present 24th 'Advances in Coatings Technology'

The Education Committee of the Cleveland Society for Coatings Technology will present its 24th Annual Technical Conference, "Advances in Coatings Technology," on March 24-25, at Baldwin-Wallace College in Berea, Ohio.

Chairman George R. Pilcher has announced that the conference will be divided into the following four, half-day symposia: "Application Processes for Emerging Technologies"; "Advances in Consumer Coatings"; "Advanced Concepts"; and "Corrosion Characteristics and Control."

The first morning session, "Application Processes for Emerging Technologies," will be chaired by Charles K. Beck, Premier Industrial Corp.; the afternoon session, "Advances in Consumer Coatings," by Dr. Theodore Provder, Glidden Coatings & Resins Division, SCM Corp.

For the second day, the morning session, "Advanced Concepts," will be chaired by Dr. John C. Weaver, Case-Western Reserve University; Sister Jeanmarie DeChant, Ph.D., Notre Dame College, will chair the afternoon session, "Corrosion Characteristics and Control."

The following papers will be presented:

March 24
Application Processes
For Emerging Technologies

"Vehicle Paint Transfer . . . A New Tool to Compare Solvent Emissions"—D.R. Hays, General Motors Corp.

"Principles of Atomization & Application Using High-Speed Bells and Discs"—H.R. Powers, Sherwin-Williams Co.

"Applications of Coatings in the Magnetic Tape Industry"—R.J. Deffeyes, Graham Magnetics, Inc.

Advances in
Consumer Coatings

"Preparation of Latexes by Emulsification"—Dr. J.W. Vanderhoff, Lehigh University, Emulsion Polymers Institute.

"Microbiological Spoilage of Latex Emulsions: Causes and Prevention"—J.A. Jakubowski, Merck & Co., Inc.

"A Simplified CPVC Calculation for Latex Paints, and its Application to Ultrafine Calcium Carbonate"—R.H. Rowland, Omya, Inc.

"Plastic Pigment III: Influence of Extenders and Coalescents"—F.L. Floyd, Glidden Coatings and Resins Division, SCM Corp.

March 25
Symposium on
Advanced Concepts

"Multifunctional Epoxy Resins: A Second Generation of Epoxies for Coatings"—C.Y. Zendig, CIBA-GEIGY Corp.

"Advances in the Thermochemical Analysis of Organic Coatings"—Dr. C.K. Schoff, PPG Industries, Inc.

"The Thermosetting Process: Analytical and Predictive Capability by Chemorheology"—Dr. R.R. Eley, Glidden Coatings & Resins Division, SCM Corp.

Corrosion Characteristics
and Control

"Surface Composition of Zinc Coatings on Steel Strip"—Dr. F.M. Kilbane, Armco, Inc.

"Facile Method for Measuring the Surface Quality for Steel"—Dr. K.R. Buser, E.I. duPont de Nemours & Co.

"An Introduction to Cathodic Protection"—B. Husock, Harco Corp.

"Salt Spray Performance of Water-based Maintenance Paints: A Multiple Correlation Study"—Dr. C.M. Frey, Glidden Coatings & Resins Division, SCM Corp.

For additional information, write the Education Committee Chairman, George R. Pilcher, The Sherwin-Williams Co., Cleveland Technical Center, 601 Canal Road, Cleveland, OH 44113.

WPTG to Hold 21st Symposium April 13–14

The Washington Paint Technical Group will hold its 21st Annual Symposium, entitled "New Decade, New Ideas," at the Marriott Twin Bridges Motel in Washington, D. C., April 13–14.

The keynote speaker will be Dr. George E. F. Brewer, Coating Consultant, who will speak on "Productivity, Invention and Innovation in the Coating Industry."

The program also includes the following speakers and topics:

"Fusion Bonded Epoxy Coatings"—James J. McDermott, of 3M Company.

"Low Cost Solar Selective Paints"—Raymond Lin, of Honeywell Systems and Research Center.

"Fire Retardant Coatings and Mastics, An Overview"—William Wass, of Ocean Chemicals.

"New Polymer Based Anti-Fouling Paints"—Robert Hartley, of International Paint Co.

"Automobile Coatings—New Era"—Melvin Camelon, of Ford Motor Co.

"Coatings Research and Applications in the Air Force"—Sidney Childers and/or Gary E. Stevenson, of Wright Patterson Air Force Base.

"The State of the Art of Chemical Coatings Used in Coil Coatings"—R.R. Robertson, of Valspar Corp.

"Heat Shrinkable Plastic Sheathing for Wooden Piles"—Ted Dowd, of NAVSEA.

"New Advances in Color Formulation and Back Shadows"—Dr. Eugene Allen, of Lehigh University.

"Forensic Paint Analysis"—James E. Corby, of the FBI

"Thermal Control Paints Used in Aerospace Environment"—Ben Seidenberg, of Goddard Space Flight Center.

For further information write Washington Paint Technical Group, P.O. Box 12025, Washington, DC 20005; or call Mildred Post at 301-530-1664 or Bernard Appleman at 703-557-5204.

Polymer Blends is Subject Of Lehigh Short Course

Lehigh University will present a short course program, "Polymer Blends and IPN's," May 31-June 5, 1981, at the Bethlehem, Pa. campus. The program will include lectures by several speakers who will present the fundamentals of polymer blends, their miscibility, behavior, industrial application, and relation to other materials.

Laboratory demonstrations will be featured along with presentations on electron microscopy and mechanical techniques. Tuition cost is $675.

For additional information, contact Ms. Jone Svirzofsky, Lehigh University, Bethlehem, PA 18015.

Paint Research Institute Programs Supported by 1980 Contributors

The list of Corporations/Organizations/Individuals which made financial contributions to the Federation's Paint Research Institute in 1980 (in addition to the annual grant from the Federation and the continuing Leo Roon Endowment Fund) is as follows:

1980 Contributors to PRI

Societies

Cleveland; Golden Gate; Houston; Kansas City; Louisville; Montreal; Pacific Northwest; Southern.

Corporations and Foundations

Canada Color & Chemicals Ltd.; Canadian General Electric Co. Ltd.; Celanese Canada Ltd.; CIL Paints, Inc.; Cook Paint & Varnish Co.; Daniel Products Co.; Davies Can Co.; Dexter Midland Div.; Dominion Colour Co. Ltd.; Dow Chemical USA; Drew Chemical Corp.; Glidden Coatings & Resins, SCM Corp.; Grow Group, Inc.; Hanna Chemical Coatings Corp.; Inmont Canada Ltd.; Kalcor Coatings Co.; David Litter Labs., Inc.; L.V. Lomas Chemical Co. Ltd.; Mobil Canada Ltd.; Monarch Coatings Co., Ltd.; Monsanto Co.; Nacan Products Ltd.; N L Industries Foundation; Polyvinyl Chemicals, Inc.; Q-Panel Co.; Henry Reichhold Foundation; Reichhold Chemicals Ltd.; Rohm and Haas Co.; Sherwin-Williams Foundation; Shell Chemical Co.; Silberline Mfg. Co.; Tioxide of Canada Ltd.; Union Carbide Corp.; Whirlpool Corp.

Also D.A. Campbell & Co.; Chemcentral; Dar-Tech, Inc.; Ferro Corp.; Harshaw Chemical Co.; Hughson Chemicals; Jamestown Paint & Varnish Co.; Mameco International; Mooney Chemicals, Inc.; Benjamin Moore & Co., Ltd.; Ontario Paint Association; Palmer Supplies Co.; Pfizer, Inc.; Seegott, Inc.; Selectone Paints Ltd.; Tenneco Chemicals, Inc.

Corporations Comprising The Mildew Consortium

Buckman Labs, Inc.; Calgon Corp.; Cosan Chemical Co.; du Pont Co., Inc.; Glidden Coatings & Resins, SCM Corp.; National Paint and Coatings Assn.; PPG Industries, Inc.; Troy Chemical Corp.

Individuals

James W. Broggini; William Dunn; Everett B. Euchner; Ross Galati; David M. Gans, Alexander J. Gibson; David Grund; E. William Henschel; M/M Fleming Kress; Mrs. Deborah Lass; Joseph W. Marchbank; Joseph McCarthy; Raymond R. Myers; Leonard Schaeffer; Fred G. Schwab; Anonymous.

Hazardous Wastes is Topic Of Battelle Conference

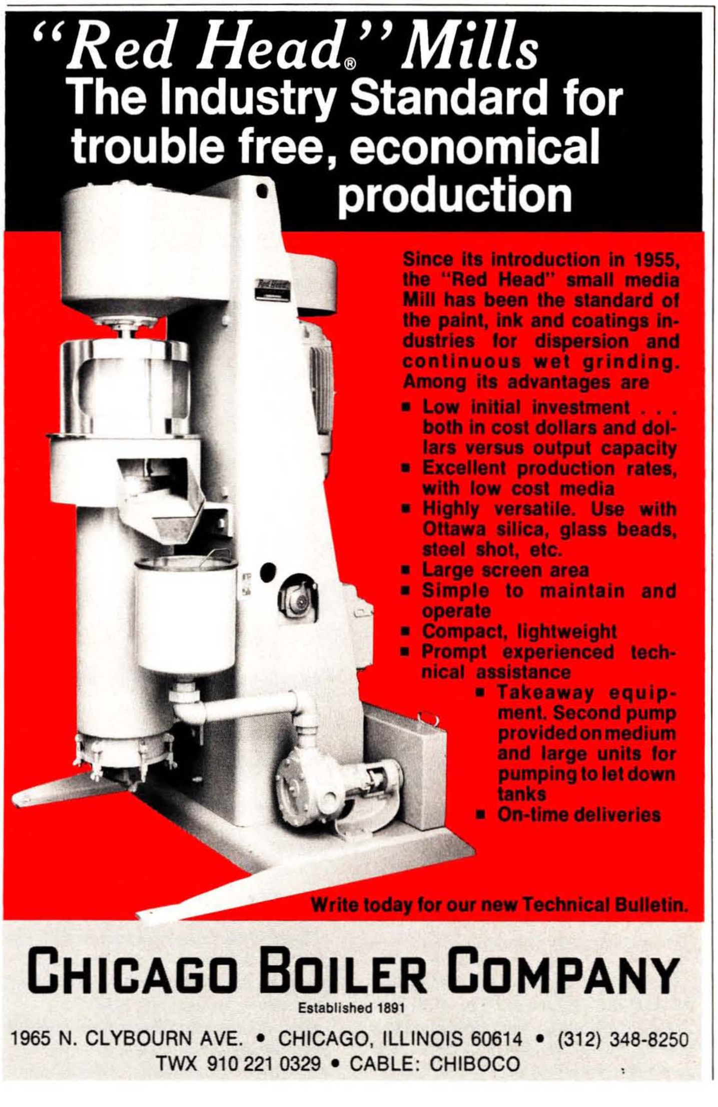
"Better Use of Technology to Manage Hazardous Wastes," will be discussed at a conference March 18–19 at Battelle's Columbus Laboratories, Ohio.

Designed to outline various options to consider when establishing new waste management programs, the conference will be of particular interest to upper- and middle-level managers responsible for hazardous wastes.

Topics to be discussed include the state-of-the-art in hazardous waste technology as well as future regulatory, economic, and technical trends associated with the field.

Presentations will be made by experts from industry and Battelle who have developed and established hazardous waste management systems. Representing such fields as resource management, economics, physical engineering, and biological sciences, speakers will be available for private discussions with attendees during the conference.

Additional information is available from Phil Wells, Battelle's Columbus Laboratories, 505 King Ave., Columbus, OH 43201, telephone 614-424-7249.



Western Societies Announce Program of 15th Biennial Symposium

Western Coatings Societies will present their 15th Biennial Symposium and Show on March 4-6, at the Disneyland Hotel and Convention Center, Anaheim, CA.

The program will include 24 papers of technical interest, as well as three workshops. The following papers are scheduled for presentation:

MARCH 4

"Government Regulation Affecting the Technology of Our Industry"—R. McFedries, Dow Chemical Co.

"Identifying Surprises or Irregularities in Waterborne and High Solids Industrial Coatings"—Al Heitkamp, Cargill, Inc.

"One Package Zinc-Rich Coatings-A Faster Route to Success"—T. Ginsburg and Al G. Kaufman, Union Carbide Corp.

"Rating Pigment Dispersion Quality from Light Transmission Measurements"—Dwight A. Holtzen, Dupont Corp.

"Biopolymers—Novel Additives for Waterborne Coatings"—P.W. Winston, Kelco, Div., Merk & Co.

MARCH 5

"Organic Pigments: Past, Present, and Future"—Dr. Hugh M. Smith, Sun Chemical Corp.

"Innovative Resins for Trade Sales Products Meeting CARB Regulations"—George H. Wilhelm, Spencer Kellogg Div., Textron, Inc.

"Surface Flow in Clear and Pigmented Resin Systems"—Robert Vash, Byk-Mallinckrodt Chem, Prod. GmbH.

"Optimal Pigmentation of Emulsion Paints for Steel in Aggressive Environments"—Roger H. Penne, Cryprus Ind. Minerals.

"Relationship of Thermal Analytical Characteristics and Application Properties of Solvent-Free Coatings"—Marcel M. Gaschke, Ciba-Geigy Corp.

"Paint Biocides—Future E.P.A. Restrictions of Effluence and Waste Disposal"—Paul Maker, Diamond Shamrock Corp.

"High Solids Polyester Spray Primers Designed for Reduced Levels of Volatile Organic Compounds"—William T. Sade, Eastman Chem. Products, Inc.

"The Utilization of Active Inhibitive Pigments in Water Based Maintenance Systems"—Jay Austin, Halox Pigments Div., Hammond Lead Products, Inc.

"Aluminum Organic Compounds in High Solids Alkyd Coatings"—D.J. Love, Manchem Limited.

"The Use of Organic Pigments with Controlled Particle Size Distribution in Coatings"—Al Meda, American Hoechst Corp.

MARCH 6

"Color Pigments in a Regulated World"—John Dickenson, Harshaw Chemical Co.

"The Effects of Metal Catalysts in a Regulated World"—Robert C. Middlemiss, Mooney Chem., Inc.

"Effects of Particle Size Distribution Upon the Optical and Rheological Properties of White Pigments"—W.S. Castor, Jr., Gulf & Western, Natural Resources Group.

"A Freedom of Choice Using High Performance Water-Borne Systems"—C.F. Dukes, Celanese Plastics & Spec. Co.

"The Effects of Cellulosic Thickeners on the Rheology of Latex Paints"—Debora F. Massouda, Hercules Incorporated.

"Improved Productivity and Corrosion Performance in Coatings with Titanate Coupling Agents"—G. Sugarman and S.J. Monte, Kenrich Petrochemicals, Inc.

"High Solids Technology with Polyurethanes"—William Cibulas, Mobay Chemical Corp.

"Effective Treatment of Paint Waste"—Jules A. Feledy, Drew Chemical Corp.

"Maintaining or Improving Paint Quality Without Increasing Raw Material Costs"—Ronald H. Roland, Omya, Inc.

Workshops scheduled for March 6 include: "Waste Control", moderated by Kenneth J. O'Morrow of Oil & Solvent Process; "Air Pollution—Formulation Aspects", moderated by Frank H. Martin; and "Labeling" moderated by James R. Elliott of Elliott Enterprises.

For additional information write, Western Coatings Symposium and Show, 11911 South Woodruff Avenue, Downey, CA 90241.

ANNUAL MEETING AND
PAINT INDUSTRIES SHOW
OCTOBER 28, 29, 30
COBO HALL
DETROIT, MICHIGAN

People

George M. Hausman has been appointed Technical Services Manager for Cellomer Corp., Newark, N.J. He will be responsible for applications development and technical services associated with the firm's alkyd and polyester resins and their line of high-solid, water-based resins. Mr. Hausman is a member of the New York Society.

Witco Chemical Corp. has appointed **Robert I. Wishnick II** Assistant General Manager of its Isocyanate Products Division. Mr. Wishnick will be relocating to the division's offices in New Castle, DE.

Dr. Joseph M. Sandri, of Alcolac, Inc., has been named Vice-President. Prior to his promotion, Dr. Sandri served as Director of Research and Development.

The membership of the Canadian Paint Manufacturers Association changed the association's name to Canadian Paint and Coatings Association at the organization's 68th annual meeting.

The transition from CPMA to CPCA will be complete with the development and institution of a new logo.

J.G. McWade, President and General Manager of Inmont Canada Ltd., was elected President of the association. **M.G. Bradley,** President of CIL Paints, Inc., was elected President-Elect.

McWade replaces **R.D. Wood,** Vice-President and general manager of the Coatings and Resins Div. of PPG Industries Ltd., as CPCA's chief executive officer. Bradley, who has been a member of the association's executive committee, has served as chairman of CPCA's Management Information Committee.

The 1981 Annual Convention will be held Sept. 13–16 in Vancouver, B.C.

A new Sherwin-Williams Business Unit is being inaugurated by the company's Chemical Coatings Div. To head the new unit are **D.D. Moit,** Coil Business Unit Manager; **T.E. Ballway,** Coil Unit Sales Manager; **J.D. Pontius,** Coil Unit Technical Director; and **J.J. Gadwood,** Coil Unit Production Manager.

New officers have been elected by the National Association of Corrosion Engineers (NACE). They include: **D.A. Dutton,** President; **W.K. Boyd,** Vice-President; **H.E. Rossy, Jr.,** Secretary-Treasurer. The new directors are: **R. Baboian, C. Christ,** and **J.D. Trim.**

G.H. Hausman

G.E.F. Brewer

R.E. Swett

F.H. Meyers

The Affiliate Council of the Engineering Society of Detroit has elected **Dr. George E.F. Brewer** as the recipient of the 1981 Gold Award, an annual presentation honoring an outstanding Detroit-area engineer, scientist, or technician.

Renowned for his development of the electrocoating process, Dr. Brewer is owner of Coatings Consultants in Brighton, Mich. Dr. Brewer is especially noted for his work in the area of anticorrosion and anti-pollution.

H. Gregory Wold has been appointed Plant Manager of Ford Motor Company's Plastics, Paint and Vinyl Division, at their Mt. Clemens Paint Plant, Mich. In his new position, Mr. Wold has responsibility for one of the largest paint plants in the world. Mr. Wold is a member of the Detroit Society.

PPG Industries, Coatings and Resins Division, announced the following appointments in their Research and Development Department at their Allison Park and Springdale, Pa. laboratories:

Dr. Neil H. Frick, has been named to the position of Manager, Industrial Coatings Development. **Mr. Ralph C. Gray** was appointed Manager, Coil & Container Coatings Development. **Dr. Gerald W. Gruber** has been named Manager, Radiation and Appliance Coatings Development. Dr. Frick and Mr. Gray are members of the Pittsburgh Society.

Nalco Chemical Co. has announced the election of several new officers for 1981. **Roland W. Graff** has been elected Executive Vice-President of the Corp. Two other promotions, within the International Division, were also announced. **Richard N. Brammer** has been promoted to President, Nalco Pacific; and **Aristide Cosani** has been promoted to President, Nalco Europe.

Roland E. Swett has been appointed Corporate Senior Vice-President of Morehouse Industries, Inc. Mr. Swett is a member of the Los Angeles Society. The firm has also appointed **Francis H. Meyers** Manager of Technical Services, Fullerton, Calif. division. Mr. Meyers is a member of the Rocky Mountain Society.

A major restructuring of the operating organization of the Hilton-Davis Chemical Group and resultant new executive assignments have been announced.

Three vice-presidents have been named to head the line functions. **Edwin B. Bergdoll** has been promoted to Vice-President of Manufacturing. **Dr. Paul J. Schmidt** has been named Vice-President of Research & Development. **Douglas H. Threader** has been appointed Vice-President of Marketing and Sales.

Named to head the staff functions are **Paul J. Nolan** as Director of Industrial and Public Relations; **Robert P. Huth** as Director of Quality Control; **Edwin H. Comrey,** Director of Materials Management; and **Paul J. Ernschwender** as Director of Forward Planning and Administrative services.

The following executive appointments have also been announced by the firm. **Dr. Raymond E. Werner** has been named Vice-President of New Technology and **Robert K. Johnson** has been promoted to Director of Quality Standards. **P. Ralph Rice** has been appointed Cincinnati Plant Manager and **Harold H. Hornbeck** has been named the Hilton-Davis Group Manager of Engineering and Services.

Donald R. Nadeau has joined Hughson Chemicals, as Senior Technical Sales Representative. He will be responsible for the territory encompassing Michigan, especially in the Greater Detroit area, selling Hughson's adhesives and coatings to the auto industry.

Columbia Paint Co. has named **Jose Siy** to the position of Senior Research and Development Chemist and **Julie Kathleen Johnson** to the position of Chemist at their Spokane, Washington laboratories.

Carl P. Izzo, a Past-President of the Pittsburgh Society, has been elected President of the Association for Finishing Processes of the Society of Manufacturing Engineers for 1980–81. Mr. Izzo, who has 25 years experience in the field of organic polymers for coatings, is a Senior Engineer with the Westinghouse Electric Corp.

Cheng H. Lee recently joined Rohm and Haas Co. as a Scientist at the Spring House (Pa.) Research Laboratories.

Devoe & Raynolds has appointed **Arthur E. Davis,** Technical Manager at their Trade Sales Laboratory in Louisville, KY.

DeSoto, Inc. has promoted **Joseph Stanton** to Senior Research Associate and **Steve Schmid** to the position of Research Chemist. Mr. Schmid is a member of the Chicago Society.

Hal Surface III, of Surface Paints, Inc., Kansas City, MO, has been elected President of the National Decorating Products Association. Other officers elected were: Vice-President—**Anthony N. Stephanie,** of Paulson's Paint and Wallpaper Co., Chicago; and Treasurer—**Murray Gladstone,** of Gladstone & Sons, Inc., Woodmere, NY.

Mark P. Morse, Research Associate for Marshall Laboratory, E.I. duPont de Nemours and Co., Inc., was named the 1980 recipient of the Henry A. Gardner Award by the American Society for Testing and Materials. Mr. Morse was cited for his outstanding contributions as chairman of three major subcommittees of ASTM Committee. D-1.

Edwin B. Gienger, Jr. has been named to the newly created position of Executive Vice-President/Eastern Operations for Reichhold Chemicals, Inc. Mr. Gienger will continue as Assistant to the Chief Executive Officer and will work closely with the president, regional managers, and financial officers of the firm. He will also have direct responsibility for 10 Eastern RCI facilities.

Literature

Techno-Economic Marketing Study

"Coatings, III", a multi-client, techno-economic marketing study has recently been completed. This 800-page study covers information concerning technical, economical, and marketing data on raw materials and end products, statistics on materials, end uses, and new coating methods, analysis of coatings producers, and controls on the environment. A free brochure, with Table of Contents, is available from Skeist Laboratories, Inc., 112 Naylon Ave., Livingston, NJ 07039.

Rinse Tank Behavior

A two-page article, "Equilibrium and Nonequilibrium Rinsing," for electroplating engineers and platers describes the experimental behavior of a rinse tank and compares it to theoretical calculations. The article provides step-by-step calculations on how to determine the total flow of rinse water and the quantities of pollutants in the water. For a copy, contact Mr. Rich Koretz, Beckman Instruments, Inc., Cedar Grove Operations, 89 Commerce Rd., Cedar Grove, NJ 07009.

SCAI

Symposium
on
Color and Appearance Instrumentation

March 24-26, 1981

Executive West Louisville, KY

Sponsored by

Federation of Societies for Coatings Technology
Manufacturers Council on Color and Appearance
Inter-Society Color Council

The 2½-day program will be divided between general sessions and "hands-on" equipment workshop sessions on instrumentation for measuring color and appearance in the coatings industry.

The format is designed to offer a "working meeting" environment, and registrants are invited to bring samples with them.

Manufacturing, production, quality control, and research and development personnel should all benefit from this definitive update on the various aspects of such topics as color measurement, color formulation, gloss and other appearance, and sample preparation.

For complete program details and information on registering, contact FSCT headquarters.

Federation of Societies for Coatings Technology,
1315 Walnut St., Suite 832, Philadelphia, PA 19107.
(215) 545-1506

Roll Filter Medias

A new eight-page brochure contains data on a complete line of fiberglass and synthetic roll filter medias. Included are illustrated descriptions of progressive density media construction, high compression strength, high tensile strength, and other product features. For a copy of RFD-1-112, contact American Air Filter Co., 215 Central Ave., Louisville, KY 40277.

Fatty Acids Study

A comprehensive review of the fatty acid and fatty acid derivative business has been published. This study provides an appraisal of the current status, as well as the future prospects of the industry. To obtain a copy of the Table of Contents and Table of Exhibits, write Mr. Alan K. Mackenzie, Hull & Co., 5 Oak St., P.O. Box 4250, Greenwich, CT 06830.

Coating Thickness Gauge

An electronic coating thickness gauge is the subject of recent literature. This pocket-sized nondestructive instrument is designed for the measurement of surface coatings on ferrous metals. For complete information, contact KTA Instruments, Div. of KTA-Tator Associates, Inc., 2020 Montour St., Coraopolis, PA 15108.

Glossmeter

A hand-held, one-piece glossmeter with 20° or 60° geometries for measurement of specular reflectance of nonmetalic surfaces of paints, plastics, and other materials is the subject of a recent brochure. For details, contact Hunter Associates Laboratory, Inc., 11495 Sunset Hills Rd., Reston, VA 22090.

Defoamers

A unique series of antifoams are the subject of recently published literature. These antifoams are formulated without the use of oils, waxes, silicas, and solvents. For additional information, contact Specialty Chemicals Division, Drew Chemical Corp., One Drew Chemical Plaza, Boonton, NJ 07005.

Measurement of Ultrapure Water

Details of monitoring and control systems which will provide a continuous indication of the water quality in a mixed-bed de-mineralization are given in a recently published application note. For a free copy of the resistivity monitoring application note, write, Foxboro Analytical, A Division of The Foxboro Co., P.O. Box 5449, South Norwalk, CT 06856.

Instrument Catalog

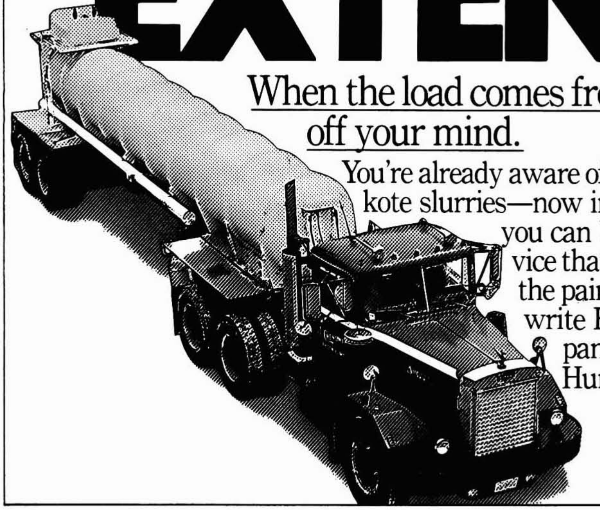
A new condensed catalog which describes major instrumentation has been prepared. Divided into two sections, entitled Appearance Measurement and Physical Testing, the catalog provides information on such materials as paints, textiles, plastics, adhesives, and inks. For additional details, contact Reva I. Goldman, Advertising and Sales Promotion, Gardner Laboratory Div., Pacific Scientific, P.O. Box 5728, 5521 Landy Lane, Bethesda, MD 20014.

Computerized Color Technology

A computerized color technology system used for the development and control of specially colored engineering materials is the focus of a new product bulletin. For complete details, write M.A. Delia, Delia Associates, P.O. Box 338, Whitehouse, NJ 08888.

Polymers

A recently published eight-page brochure details polymers for use in aqueous systems, including nonwovens, paper coatings, paints, adhesives, and caulks. For a free copy of the brochure, write to H.B. Fuller Co., Polymer Div., 4450 Malsbary Rd., Blue Ash, OH 45242.



Book Review

PAINT FLOW AND PIGMENT DISPERSION

by
Temple C. Patton 2nd Edition

Published by

John Wiley and Sons, Inc.
New York, NY
1979 (640 pages) $42.00

Reviewed by
Percy E. Pierce
PPG Industries, Inc.
Allison Park, PA

If the value of a book can be judged by its rate of disappearance from library shelves, then the first edition of "Paint Flow and Pigment Dispersion" must have set a new record, at least in our research library. The second edition extensively revised and updated should, I judge, receive an even more enthusiastic acceptance.

The author has attempted to provide engineers in the coating, ink, and allied industries with a practical exposition of the more important aspects of the technology relating to pigment dispersion, the rheology of coating flow, and other topics related to these phenomena. The author steers a middle course between a highly theoretical presentation and an unduly elementary treatment. Objective problems are worked throughout the text to demonstrate the practical use of equations derived from theoretical concepts. Each chapter has been thoroughly reworked and documented with original references, including much new work which has appeared in the past fifteen years.

The basic concepts of viscosity are clearly and concisely explained with emphasis on the types of rheological behavior important to coatings and inks. Common viscometers utilized by our industry are analyzed and compared in terms of basic concepts. The means for interconversion of viscosity units and comparison of results from different instruments are described in detail. The effect of temperature, binder concentration, solvent viscosity, and molecular size on viscosity are discussed.

Pigment/Binder geometry and its consequences to system performance are presented, followed by in-depth discussions of oil absorption and critical pigment volume concentration (CPVC) for both solvent and latex systems. The up to date review and critique of methods for CPVC determination is especially valuable and timely.

The fundamentals of surface tension, adhesion, wetting, and spreading are examined, along with means for measuring and estimating surface tensions. The work of dispersion, work of transfer; and work of flocculation are developed and applied to the pigment dispersion process. The concept of capillarity and its practical implications to hold out and film formation are developed.

The theoretical aspects of pigment dispersion stability and pigment flocculation are presented in a most understandable way. Interface activity, surfactants, and dispersants are discussed with clear explanations of terms, the physical roles and processes involved.

The solubility and interaction parameter concepts are explained. Different solubility parameter approaches are compared and applied to practical coating and ink problems. Solvent and water evaporation is analyzed, both from neat solvent mixtures and paints. Factors affecting evaporation and solvent retention, along with practical considerations, are presented.

Paint flow relationships with application to system structure and application performance are discussed.

An extensive discussion of pigment grinding, including an analysis of roller mills, ball and pebble mills, attributors, sand and shot mills, high speed dispensers, stone and colloid mills, and high speed impingement mills represent a unique feature of this excellent book. Millbase formulation principles, a description of the equipment, detailed process analyses, practical hints for successful operation scale up concepts are all combined together to provide a most useful discussion.

The problems of measuring pigment dispersion, millbase letdown, and pigment settling are extensively discussed with many useful suggestions for dealing with common problems encountered by the formulator.

Application problems such as leveling, sagging, floating, flooding, cratering, foaming, and spattering are discussed. The chapter on film applicators will also prove useful to all those who face the task of preparing reproducible, uniform films.

In summary, reading Temple Patton's revised 2nd edition of "Paint Flow and Pigment Dispersion" is a must for anyone seriously involved in the coating, ink, and allied industries. I would recommend this book for inclusion in your own personal library. It is a most useful and understandable source of relevant information for the coating engineer.

—P.E.P.

Letters to the Editor

Chairman Asks for Improved Communication

To The Editor:

I am constantly amazed, but more often annoyed, by the seeming lack of a sense of responsibility among people in the Paint Industry who don't reply to legitimate requests both written and verbal. It would appear that if one sends out a group of letters requesting replies, he would do well if he got a 10% response. While I have specific reference to Federation work, this statement will apply equally well to internal Society work.

Communication is the "glue" that binds this Federation together, so if, at times, we seem to be coming "unglued", it is usually because of the lack of effective communications. I realize that writing letters is becoming a lost art, but phone calls are not all that difficult to make. If one does not have time to write a letter, surely he could pick up the phone and pass on the required information in the matter of a few minutes—it certainly would be a big help to the person called.

I have purposely avoided mentioning groups, committees, people, etc. This sad situation is too wide spread to point a finger at one person or even group. I would hope that each person who reads this would reflect on his or her own situation and improve it accordingly. I do hope too, that those who owe me letters will respond to this and write me!

Joseph H. Boatwright
Chairman, Technical Committee
Chicago Society

Coming Events

FEDERATION MEETINGS

(Mar. 24–26)—"Symposium on Color and Appearance Instrumentation." Executive West, Louisville, KY. Jointly sponsored by Federation of Societies for Coatings Technology, Manufacturers Council on Color and Appearance, and Inter-Society Color Council. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, Pa. 19107).

(May 14–15)—Spring Meetings. Society Officers on 14th; Board of Directors on 15th. Hilton Hotel, Denver, CO. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, PA 19107).

(Oct. 28–30)—59th Annual Meeting and 46th Paint Industries' Show. Cobo Hall, Detroit, MI. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, PA 19107).

1982

(Apr. 29–30)—Spring Meetings. Society Officers on 29th; Board of Directors on 30th. Boston, MA. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, PA 19107).

(Nov. 3–5)—60th Annual Meeting and 47th Paint Industries' Show. Sheraton Washington Hotel, Washington, D.C. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, PA 19107).

PAINT RESEARCH INSTITUTE MEETING

(May 4–5)—Paint Research Institute Symposium on "Stability and Stabilization of Coatings Systems," Battelle Memorial Institute, Columbus, Ohio. (Dr. Raymond R. Myers, Chemistry Dept., Kent State University, Kent, Ohio 44242).

SPECIAL SOCIETY MEETINGS

(Feb. 25–27)—Eighth Annual Water-Borne and Higher-Solids Coatings Symposium Hyatt Regency Hotel, New Orleans, LA. Sponsored by Southern Society and University of Southern Mississippi. (Dept. of Polymer Science, Univ. of Southern Mississippi, Southern Station, Box 10076, Hattiesburg, MS 39401).

(Mar. 4–6)—Western Coatings Societies Symposium and Show. Disneyland Hotel, Anaheim, CA. (Richard C. Sutherland, E.T. Horn, Co., 16141 Heron Ave., La Mirada, CA 90638.)

(Mar. 11–13)—Southern Society. 45th Annual Meeting. Birmingham Hilton Inn and Conference Center, Birmingham, AL Peter F. Decker, Union Carbide Corp., 17 Executive Park Drive, N.E. Atlanta, GA 30359).

(Mar. 13)—Baltimore Society. Coatings Show. Hilton Inn, Baltimore, MD. (Dick Chodnicki, Stern & Marsh Co., 31 Alleghany Ave., Towson, MD 21204).

(Mar. 24–25)—Cleveland Society 24th Annual Conference, "Advances in Coatings Technology." Baldwin-Wallace College, Berea, OH. (George R. Pilcher, Sherwin-Williams Co., 601 Canal Rd., Cleveland, OH 44113.)

(Apr. 21–22)—Chicago Society. SYMCO '81. "Impact of Environment, Cost, and Technology in 1981." Fountain Blue Restaurant, Des Plaines, IL. (Dolores Thomas, Chicago PCA, Suite 1936, 35 E. Wacker Dr., Chicago, Il 60601).

(May 1–2)—Pacific Northwest Society. Annual Symposium. Washington Plaza Hotel, Seattle, WA.

(May 9)—Montreal Society. Fiftieth Anniversary dinner-dance, Montreal Que.

(June 15)—Golden Gate Society Seminar, "Safety and Government Regulations." (Louie Sanguinetti, Jasco Chemical Corp., P.O. Drawer J, Mountain View, CA 94042).

1982

(Mar. 10–12)—Southern Society Annual Meeting. Savannah, Ga.

(Apr. 22–24)—Southwestern Paint Convention. Shamrock Hilton Hotel, Houston, TX.

(May 6–8)—Pacific Northwest Society. Annual Symposium. Vancouver, B.C.

1983

(Feb. 23–25)—16th Biennial Western Coatings Societies' Symposium and Show. Hyatt Regency, San Francisco, CA.

OTHER ORGANIZATIONS

(Mar. 2–4)—"Adhesion Science and Technology" Short Course. The Center for Professional Advancement, Central NJ. (The Center for Professional Advancement, P.O. Box H, East Brunswick, NJ 08816).

(Mar. 3–5)—"Refresher for Painting Contractors, Maintenance Engineers and Inspectors" Short Course. Sheraton Anaheim Hotel, Anaheim, CA. (Norma Fleming, Coordinator, Arts & Sciences Continuing Education, University of Missouri-Rolla, Rolla, MO 65401).

(Mar. 9–11)—"Fundamentals of Adhesion: Theory, Practice and Applications" Course. State University of New York, New Paltz, NY. (Dr. Angelos V. Patsis, Chairman, Dept. of Chemistry, CSB 209, State University of New York, New Paltz, NY 12561).

(Mar. 23–27)—"Introductory Short Course on Composition of Coatings." University of Missouri-Rolla, Rolla, MO. (Norma Fleming, Coordinator, Arts & Sciences Continuing Education, University of Missouri-Rolla, Rolla, MO 65401).

(Mar. 23–27)—"Spray Finishing Technology" Workshop. Bowling Green State University & DeVilbiss Co., Toledo, OH. (Dr. R.A. Kruppa, School of Technology, Bowling Green State University, Bowling Green, OH 43403).

(Mar. 25–26)—Air Pollution Control Association, Ninth Government Affairs Seminar. Sheration Washington Hotel, Washington, D.C. (Public Relations Dept., Air Pollution Control Association, P.O. Box 2861, Pittsburgh, PA 15230).

(Apr. 6–10)—"Introduction to Paint Formulation" Short Course. University of Missouri–Rolla, Rolla, MO. (Norma Fleming, Coordinator, Arts & Sciences Continuing Education, University of Missouri–Rolla, Rolla, MO 65401).

(Apr. 6–10)—National Association of Corrosion Engineers "Corrosion/81." Sheraton-Centre and Hotel Toronto, Toronto, Ontario, Canada. (Conference Coordinator, NACE, P.O. Box 218340, Houston, TX 77218).

(Apr. 7–8)—NACE T6 Symposium on Underfilm Corrosion. Sheraton Centre Hotel, Toronto, Canada. (National Association of Corrosion Engineers, 1440 South Creek, Houston, TX 77084.)

(Apr. 13–14)—Washington Paint Technical Group Annual Symposium. "New Decade, New Ideas." Marriott Twin Bridges Motel, Washington, D.C. (WPTG, P.O. Box 12025, Washington, D.C. 20005.)

(Apr. 13–17)—"Advanced Paint Formulation" Short Course. University of Missouri-Rolla, Rolla, MO (Norma Fleming, Coordinator, Arts & Sciences Continuing Education, University of Missouri-Rolla, Rolla, MO 65401).

(Apr. 15–17)— Hunterlab Workshop, "Appearance Properties of Materials." Reston, VA. (Hunterlab Associates Laboratory, Inc., 11495 Sunset Hills Road, Reston, VA 22090).

(Apr. 26–28)—Inter-Society Color Council Annual Meeting. Roosevelt Hotel, NY. (Dr. Fred W. Billmeyer, Secretary, ISCC, Rensselaer Polytechnic Institute, Troy, NY 12181.)

(Apr. 28–30)—OCCA-32. Oil and Colour Chemists' Association 32nd Annual Technical Exhibition. Cunard International Hotel, London, England. (The Director and Secretary, OCCA, Priory House, 967 Harrow Rd., Wembley, Middlesex HAO 2SF, England.)

(May 10–12)—"Refresher for Contractors, Maintenance Engineers and Inspectors" Short Course. Sheraton-Valley Forge Hotel, Philadelphia, PA (Norma Fleming, Coordinator, Arts & Sciences Continuing Education, University of Missouri-Rolla, Rolla, MO 65401).

(May 17–21)—American Oil Chemists' Society 72nd Annual Meeting. Fairmont Hotel, New Orleans, LA. (American Oil Chemists' Society, 508 S. Sixth St., Champaign, IL 61820).

(May 18–22)—"Dispersion of Pigments and Resins in Fluid Media" Short Course. Kent State University, Kent, OH. (Carl J. Knauss, Chemistry Dept., Kent State University, Kent, OH 44242).

(May 19–20)—"Practical Waste Management for the Coatings Industry" Short Course. Granada Royale, Kansas City, MO (Norma Fleming, Coordinator, Arts & Sciences Continuing Education, University of Missouri-Rolla, Rolla, MO 65401).

(May 31–June 5)—"Polymer Blends and IPN's" Short Course. Lehigh University, Bethlehem, PA (Jone Svirzofsky, Lehigh University, Bethlehem, PA 18015).

(June 8–12)—"Adhesion Principles and Practice for Coatings and Polymer Scientist" Short Course. Kent State University, Kent, OH. (Carl J. Knauss, Chemistry Dept., Kent State University, Kent, OH 44242).

(June 14–17)—Dry Color Manufacturers' Association Annual Meeting. The Greenbriar, White Sulphur Springs, WV. (P.L. Lehr, DCMA, Suite 100, 1117 North 19th St., Arlington, (Rosslyn) VA 22209.)

(June 15–19)—"Applied Rheology for Industrial Chemists" Short Course. Kent State University, Kent, OH. (Carl J. Knauss, Chemistry Dept., Kent State University, Kent, OH 44242).

(June 17–20)—Oil and Colour Chemists' Association Conference, "Alternative Téchnologies in Coatings." Beaufort Hotel, Bath, England. (The Director and Secretary, OCCA, Priory House, 967 Harrow Rd., Wembley, Middlesex HA0 2SF, England.)

(June 21–26)—Air Polution Control Association 74th Annual Meeting & Exhibition. Philadelphia Civic Center, Philadelphia, PA (Public Relations Dept., Air Pollution Control Association, P.O. Box 2861, Pittsburgh, PA 15230).

(Aug. 10–14)—"Introductory Short Course on Composition of Coatings." University of Missouri-Rolla, Rolla, MO (Norma Fleming, Coordinator, Arts & Sciences Continuing Education, University of Missouri-Rolla, Rolla, MO 65401).

(Aug. 31–Sept. 4)—"Introduction to Paint Formulation" Short Course. University of Missouri-Rolla, Rolla, MO (Norma Fleming, Coordinator, Arts & Sciences Continuing Education, University of Missouri-Rolla, Rolla, MO 65401).

(Sept. 7–11)—"Advanced Paint Formulation" Short Course. University of Missouri-Rolla, Rolla, MO (Norma Fleming, Coordinator, Arts & Sciences Continuing Education, University of Missouri-Rolla, Rolla, MO 65401).

(Sept. 13–16)—Canadian Paint Manufacturers Association. Four Seasons Hotel, Vancouver, B.C. (Lydia Palazzi, Canadian Paint Manufacturers Assn., 2050 Mansfield, Montreal, Que., Canada H3A 1Y9.)

(Sept. 20–25)—4th Congress of the Association Internationale de la Couleur, "COLOR 81." International Congress Centre (ICC), Berlin (West), Germany. (Prof. Dr. Heinz Terstiege, (AIC COLOR 81), Bundesanstalt fur Materialprufung (BAM), Unter den Eichen 87, D-1000 Berlin 45, Federal Republic of Germany).

(Sept. 21–22)—"Techniques and Mechanics of Marketing Specialty Chemicals" Short Course. Hilton Plaza Inn, Kansas City, MO (Norma Fleming, Coordinator, Arts & Sciences Continuing Education, University of Missouri–Rolla, Rolla, MO 65401).

(Sept. 23–24)—"Managing for Innovation in Coatings" Short Course. Hilton Plaza Inn, Kansas City, MO (Norma Fleming, Coordinator, Arts & Sciences Continuing Education, University of Missouri–Rolla, Rolla, MO 65401).

(Sept. 28–29)—Golden Jubilee of Colour in the CIE. The Colour Group (Great Britain). Imperial College, London, England. (Ms. M.B. Halstead, Thorn Lighting Ltd., Great Cambridge Rd., Enfield, Middlesex EN1 1UL, England).

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CABOT CORP.	**59**
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CIBA-GEIGY CORP.	**18–19**
DuPONT CO., DBE SOLVENTS	**Cover 2**
ECLIPSE SYSTEMS, INC.	**87**
ENGLISH MICA CO.	**83**
FLINTKOTE STONE PRODUCTS CO.	**85**
HALOX PIGMENTS	**48**
DR. HANS HEUBACH & CO.	**33–34**
NALCO CHEMICALS CO.	**77**
NL CHEMICALS/NL INDUSTRIES, INC.	**13**
PENNSYLVANIA GLASS SAND CORP.	**70**
PETROLITE CORP., BARECO DIV.	**46**
PFIZER, INC., MPM DIV.	**10–11**
POLYVINYL CHEMICAL INDUSTRIES	**68–69**
PURITY ZINC METALS CO., LTD.	**Cover 3**
ROHM AND HAAS CO.	**8–9, 14–15**
SCHOLD MACHINE CO.	**88**
SPENCER KELLOGG DIV., TEXTRON, INC.	**Cover 4**
THIBAULT & WALKER CO., INC.	**32**
UNION CARBIDE CORP.	**4–5, 60**
R.T. VANDERBILT CO., INC.	**2**

CALL FOR PAPERS

Color and Appearance in Changing Times

6TH ANNUAL **FOCUS** CONFERENCE
FUTURE OF COATINGS UNDER STUDY

Detroit Society for Coatings Technology
May 5, 1981
Management Education Center
Troy, Michigan

Conference will FOCUS on those parameters which contribute to the buyer's appeal for an automotive finish. Solicited papers should cover the following areas:

- Substrate influence on appearance.
- New resins and coating systems.
- Enhancing appearance through new methods of application.
- Apparatus used to measure appearance qualities.
- New and improved color pigments.
- Quality control considerations for color.

Those wishing to participate are urged to submit a letter of intent including a tentative title of the paper as soon as possible and an abstract of about 200 words by February 28, 1981. Abstracts on any subjects related to color and appearance would be welcomed. Please forward all communications to Bohdan Melnyk, The Detroit Society for Coatings Technology, 765 Dellwood Drive, Ann Arbor, Michigan 48103. Telephone (313) 956-1256.

'Humbug' from Hillman

I have publicly confessed, in the past, my humble respect for those in our industry who voraciously devour, with complete understanding, all the articles in this fine periodical. I admit, further, to my inability to cope with the table of contents. Even the titles throw me, to my chagrin and embarrassment.

Some months back, one David Jackson said it so well for me in the *Wall Street Journal.* viz—

I sometimes feel that I am inert
Inept and also inane
The problems are innate I'm told
To worry is in vain.
The object very simply is
To change one's way of sin
To be more ert, ept, ane and nate
And stop from being done - "in".

In the meantime, I will get my giggles from both ends of the book—an occasional editorial from Borrelle and the tid bits such as those that follow below from friends who generously have offered to share appropriate pieces of wisdom.

Howard Jerome, our President-Elect, has foolishly elected to reveal his intellectual weaknesses along with his esteemed colleagues.

—Herb Hillman

Finagle's laws

1st Law: If anything can go wrong with an experiment, it will.
2nd Law: No matter what result is anticipated, there is always someone willing to fake it.
3rd Law: No matter what the result, there is always someone eager to misinterpret it.
4th Law: No matter what occurs, there is always someone who believes it happened to his pet theory.

Compendium of Ground Rules for Laboratory Workers

A. When you don't know what you are doing, do it neatly.
B. Experiments must be reproducible. They should all fail the same way.
C. First draw your curves, then plot the data.
D. Experience is directly proportional to the equipment ruined.
E. A record of data is essential. It indicates you have been working.
F. To study a subject best, understand it thoroughly before you start.
G. In case of doubt, make it sound convincing.
H. Do not believe in miracles: rely on them.
I. Teamwork is essential in the laboratory. It allows you to blame someone else.

I have more—

The Law of the Too Solid Goof

In any collection of data, the figure that is most obviously correct, beyond all need of checking, is the mistake.

COROLLARY 1. No one whom you ask for help will see the mistake either.

COROLLARY 2. Everyone who stops by with unsought advice will see it immediately.

—Howard Jerome

What Webster overlooked

WATER—*"An ingredient used to support both the paint and the manufacturer."* (Do you have to explain that to the younger paint chemist?)
ENAMEL—*"An enamel is a cross between a paint and a varnish. If the cross shows, it is not an enamel."*

—Royal Brown

The nomenclature of administration

a program: Any assignment that can't be completed by one telephone call.
to expedite: To confound confusion with commotion.
channels: The trail left by inter-office memos.
co-ordinator: The guy who has a desk between two expeditors.
consultant: Any ordinary guy more than 50 miles from home.
to activate: To make carbons and add more names to the memo.
to implement a program: Hire more people and expand the office.
under consideration: Never heard of it.
under active consideration: We're looking in the files for it.
a meeting: a mass milling by master-minds.
a conference: A place where conversation is substituted for the dreariness of labor and the loneliness of thought.
will advise in due course: If we figure it out, we'll let you know.
reorientation: Getting used to working again.
reliable source: The guy you just met.
informed source: The guy who told the guy you just met.
unimpeachable source: The guy who originally started the rumor.
a clarification: To fill in the background with so many details that the foreground goes underground.
we are making a survey: We need more time to think of an answer.
note and initial: Let's spread the responsibility for this.
see me, or let's discuss: Come down to my office; I'm lonesome.
give us the benefit of your present thinking: We'll listen to what you say as long as it doesn't interfere with what we decided to do.
to give someone the picture: A long confused inaccurate statement to a newcomer.
to negotiate: To seek a meeting of minds without knocking together of heads.
point up the issue: To expand one page to fifteen pages.

—Don Brody

A coating is only as good as its base elements. That's right where we come in. With Zinc Dust made only from virgin zinc. Dust with a purity level of up to 99.99% plus where necessary. So you can finally eliminate one major area of product inconsistency.

Because your products' quality is judged on a daily basis, every order we produce undergoes a thorough physical and chemical analysis before packaging.

Call for the latest technical data concerning zinc dust and its applications. Samples of Purity Zinc Dust are available to chemists and engineers for product testing, or put our technical staff to work on your product development.

Purity Zinc Dust. A lasting partner through quality and performance.

PZM

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