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JOURNAL OF
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TECHNOLOGY

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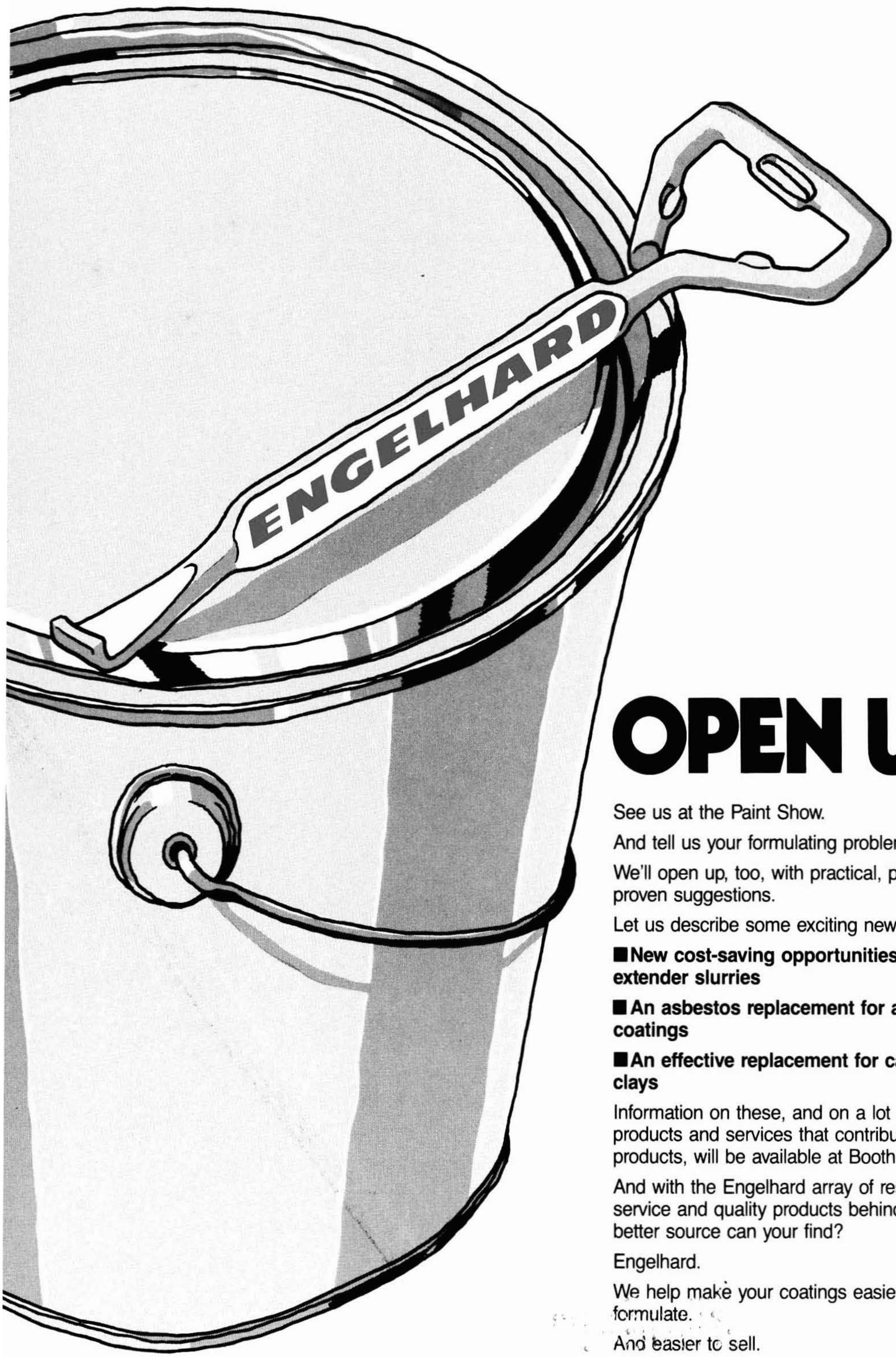
OCTOBER 1980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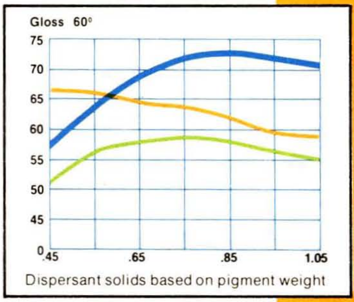

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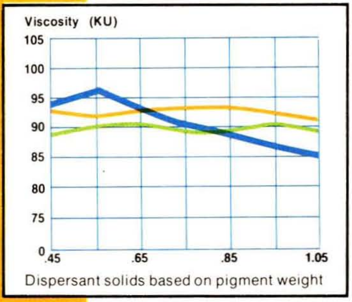
DISPERSANT DEMAND



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Booth No.'s
257, 259, 356, 358

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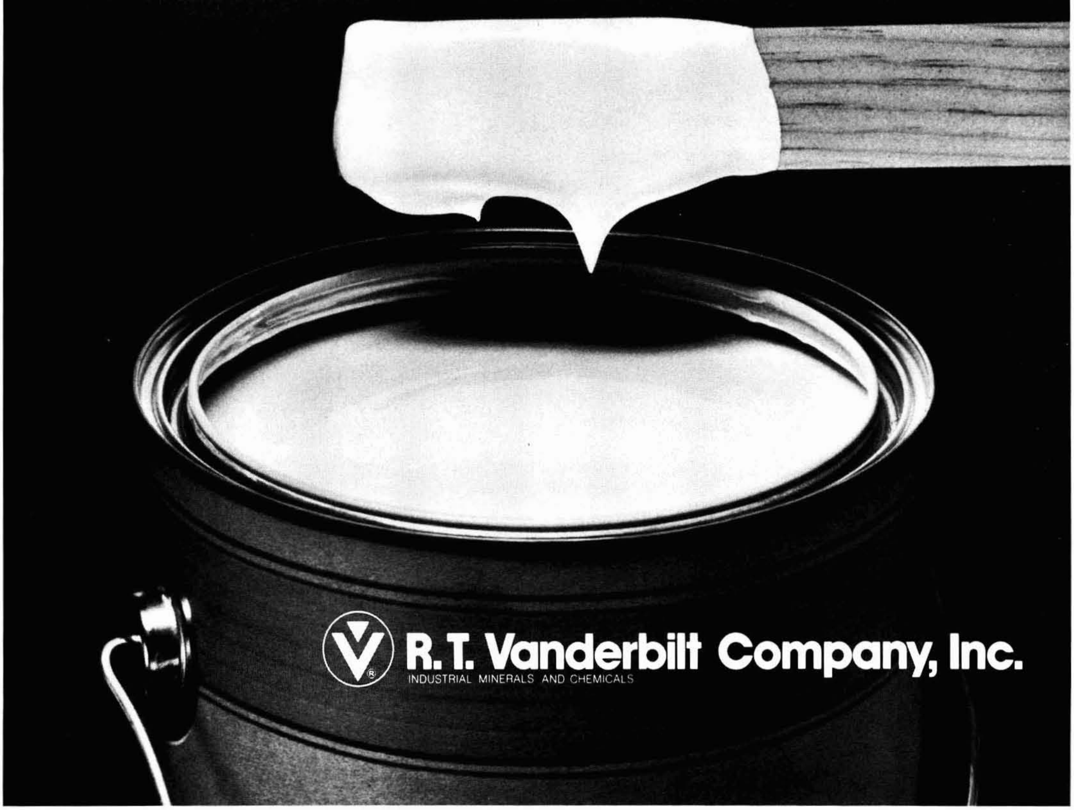

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INDUSTRIAL MINERALS AND CHEMICALS

OCTOBER 1980

jct JOURNAL OF COATINGS TECHNOLOGY

Volume 52 Number 669

Features

Departments

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Comment

Education—The Ongoing Commitment

One of the primary Federation objectives is the promotion of educational activities, and much effort continues to be directed to that end.

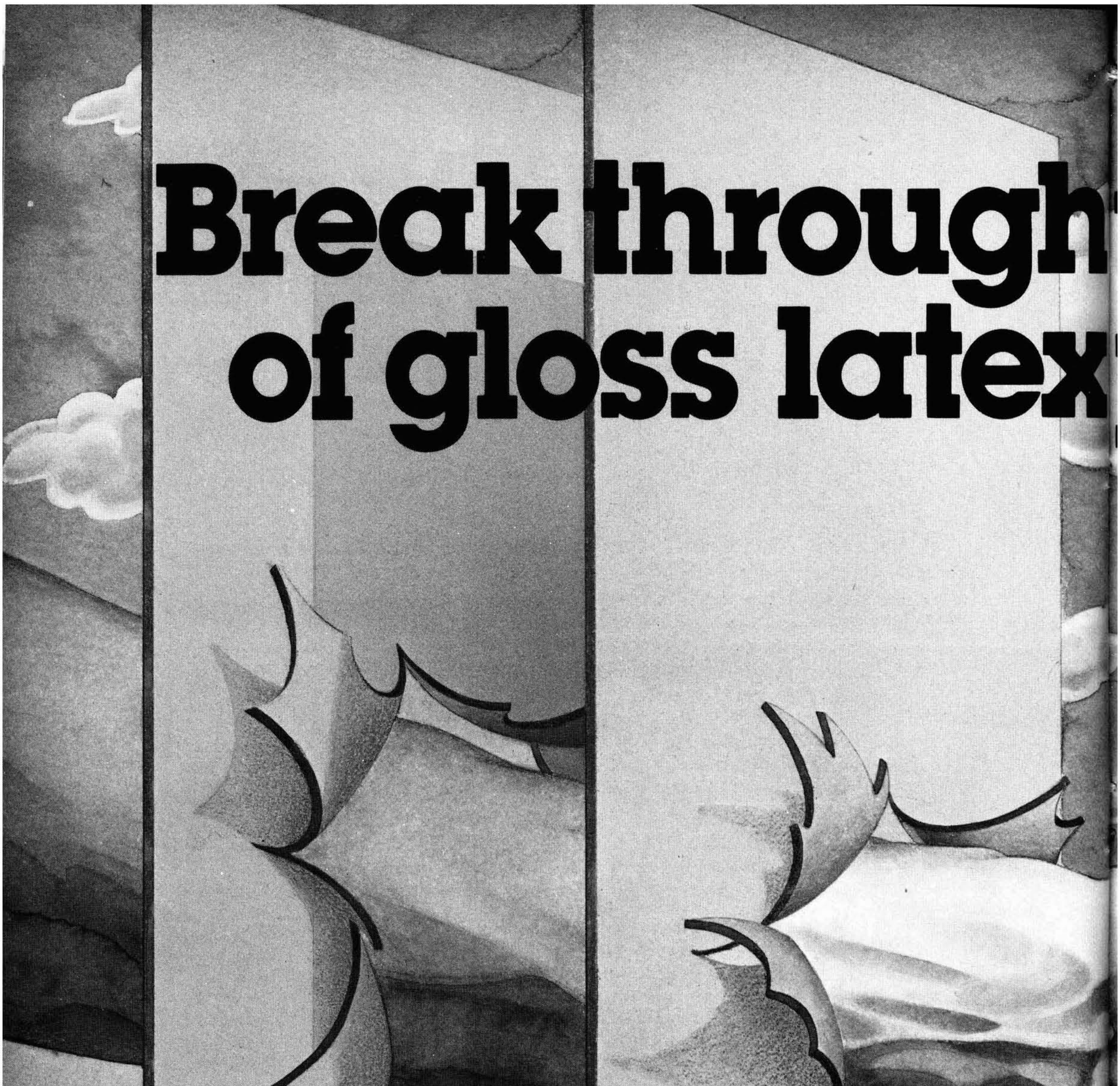
To help meet the demand for more technically trained people in the industry, scholarship funds will again be made available to five schools with coatings curriculum (University of Southern Mississippi, North Dakota State University, Kent State University, University of Missouri—Rolla, and University of Detroit); these funds are used as grants-in-aid for students in the coatings technology programs at each institution, with preference given to qualified scholarship applicants who are children of Federation members. Meanwhile, the Educational Committee is developing informational material on career opportunities in the coatings industry, to be used in presentations to student groups by Society personnel.

For those in the industry, the Federation is developing in conjunction with the University of Southern Mississippi a correspondence course on the Science and Technology of Surface Coatings for personnel who have not had the opportunity to receive specialized academic training in the science of coatings and polymers.

The popular Federation series of booklets on coatings technology is being revised and updated to reflect recent advances. And the audio/visual programs are being expanded: the Manufacturing Committee's presentation of "Introduction to Resin Operations," produced by the Toronto Society has just been introduced, and the Birmingham Club's production of "The Setaflash Tester" will be added to the Educational Committee's Training Series on Test Methods. Two other productions are nearing completion—"Measurement of Hiding" (Kansas City) and "Impact Resistance" (Western New York). These A/V programs provide a two-sided benefit, in that they not only fill a need as a learning tool but also serve as interesting, meaningful projects for Society members.

In the larger sense, of course, just about every area in which the Federation is involved is educational to some degree—including such activities as the publication of JCT, Paint/Coatings Dictionary, Infrared Spectroscopy Atlas, and other special publications, as well as sponsoring the Annual Meeting and Paint Industries' Show, and providing substantial funding in support of the Paint Research Institute.

Education is vital to our industry and our membership, and the Federation is committed to maintaining support of these activities wherever and however it can.—TAK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latex paint modified with
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Second generation epoxies for

These special Ciba-Geigy epoxy resins have been used for years in the electronics and electrical industries. Their outstanding properties make them ideal for new high-performance coatings as well as for upgrading existing coatings systems. We have recently built new, larger manufacturing facilities, and these special epoxies are now available to the coatings industry. We believe the suggested applications illustrated are only a few of many possible uses. Details? Ciba-Geigy, Resins Department, Ardsley, New York 10502. Phone (914) 478-3131.

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Increases high temperature stability.

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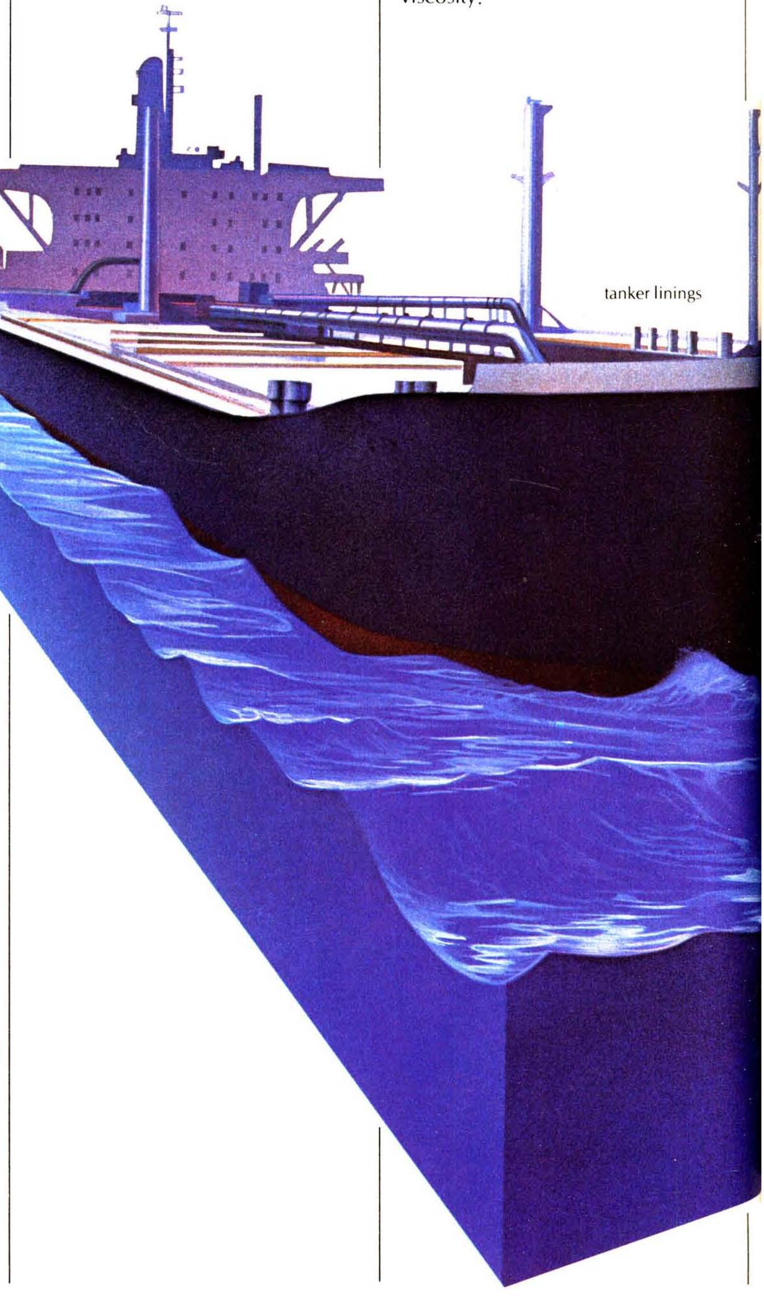
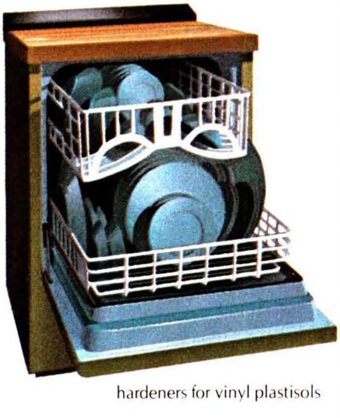


tanker linings



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Remain stable six months after formulating – high latency. Cure rapidly – five minutes at 100°C.

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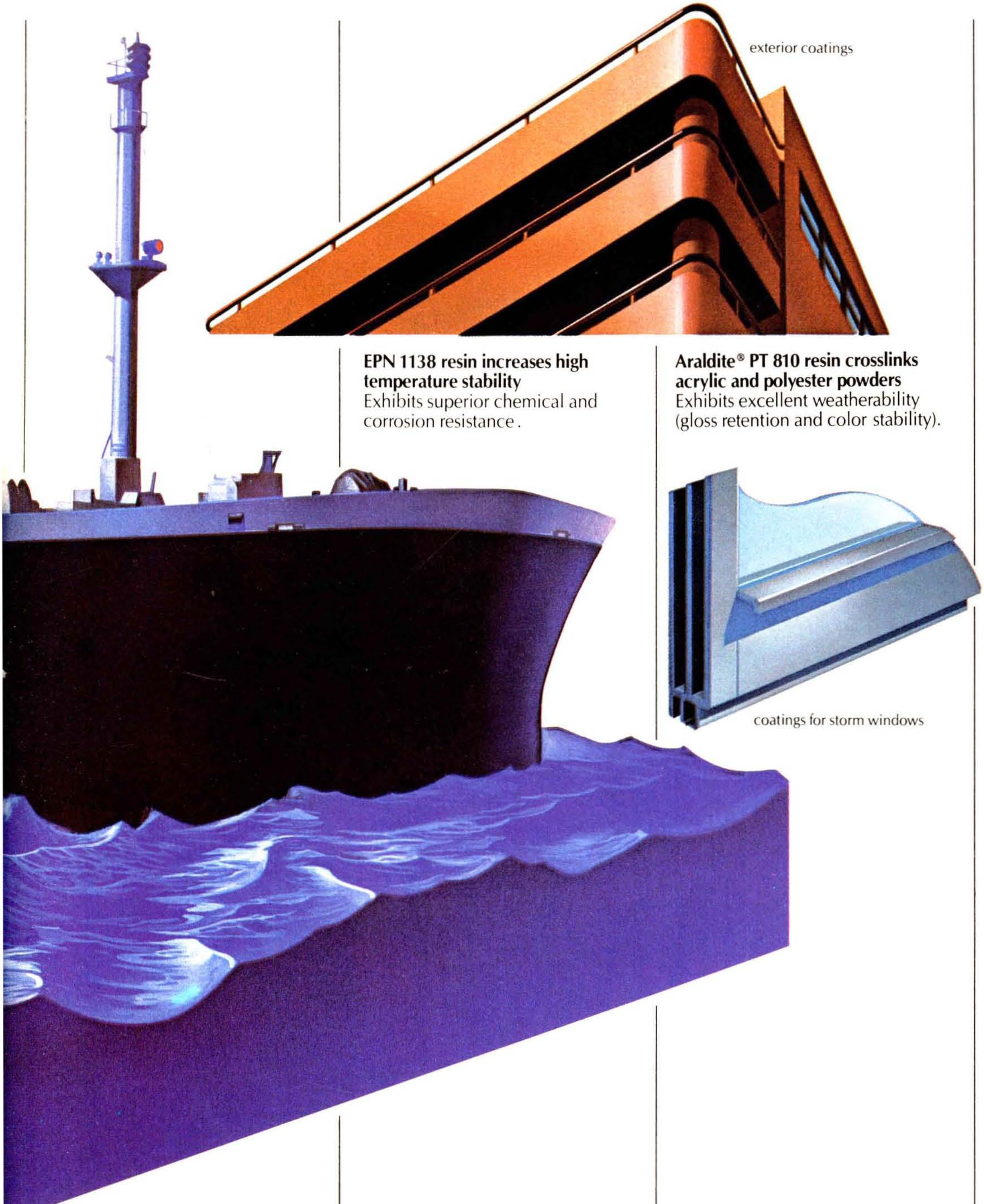

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coatings for storm windows

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Abstracts of Papers in This Issue

APPLICATION OF HYDRODYNAMIC VOLUME AND SWELLING THEORY TO THE CROSSLINKING OF LATEX PARTICLES—G.M. Crews, G.C. Wildman, J.R. Grawe, and B.G. Bufkin

Journal of Coatings Technology, 52, No. 669, 33 (Oct. 1980)

An *in situ* hydrodynamic approach to swelling ratios was investigated as a means of monitoring the degree of crosslinking of emulsion particles while in suspended form. The accuracy of the hydrodynamic technique was examined in comparison with a free film swelling technique using precrosslinked, butyl acrylate/diethylene glycol diacrylate (DEGDA) model emulsions. The results obtained from both methods supported the expected trend of a decrease in swelling ratio with increasing concentration of crosslinking agent.

The theoretical utility of the hydrodynamic technique was demonstrated by using the swelling ratio values to obtain the molecular weight of the polymer segments between crosslinks (M_c). Using the M_c values, the crosslinking efficiency of DEGDA was estimated to be 31% and 16% when employed at 0.42 and 3.15 mole % levels, respectively.

The applied utility of the technique was illustrated by using swelling ratios to monitor the migration rate of an external amine curing agent from the aqueous phase to an epoxy-containing polymer phase.

AMINE LOSS FROM WATER-SOLUBLE POLYMER SOLUTIONS EXPRESSED AS SIMPLE SOLVENT EVAPORATION—P. Kamarchik, Jr.

Journal of Coatings Technology, 52, No. 669, 41 (Oct. 1980)

The behavior of various amines in three types of drying paint films was investigated by preparing films of the neutralized polymer from which solvent and amine were allowed to evaporate for specified times. The films were then analyzed for residual amine by acid titration. By this method it was possible to construct amine concentration versus time graphs for various temperatures and film thicknesses. The behavior of these plots indicates the similarity between amine loss and simple solvent evaporation during the time that the film remains "wet." For the most commonly employed amines, this period accounts for most of the amine lost. Furthermore, the temperature variation of the initial loss rate leads to an activation energy of amine loss comparable to the heat of vaporization of the amine under consideration. The effects of base strength are shown to be minimal, at least in the early stages of the loss.

TRACE RESIDUAL MONOMER ANALYSIS BY CAPILLARY GAS CHROMATOGRAPHY—K.J. Rygle

Journal of Coatings Technology, 52, No. 669, 47 (Oct. 1980)

A method has been developed for the determination of trace amounts of residual monomers by capillary gas chromatography for application in the coatings industry. Most previously employed methods for these analyses made use of conventional packed columns. Recent discoveries of adverse health effects associated with some monomers have enhanced the importance of trace analysis. The most feasible method for performing these analyses when dealing with complex solvent systems is to take advantage of high resolution capillary columns. A method is described which uses a splitless injection technique, with a solvent effect being employed for the analysis. Varying solvent systems are considered for possible interferences in conjunction with the monomers of interest. A unique feature of the method is the use of a 50 meter, stainless steel capillary column. A performance evaluation of the stainless steel column is discussed in detail.

AN OVERVIEW OF QUALITY ASSURANCE—H.M. Werner

Journal of Coatings Technology, 52, No. 669, 53 (Oct. 1980)

Quality Assurance is extradepartmental in scope and responsibility. Quality begins with the setting of standards for the desired levels of excellence. People involvement and good communication are vital to the success of a good program. Assurance of quality is dependent upon optimum formulations with raw materials of known characteristics and practical specifications, close process control, documentation of procedures, monitoring of compliance to standards, and good basic practices. An established program of education and training for involved personnel is vital to a "preventive medicine" approach to "doing it right the first time!"


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OIL & COLOUR CHEMISTS' ASSOCIATION

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OCCA
33

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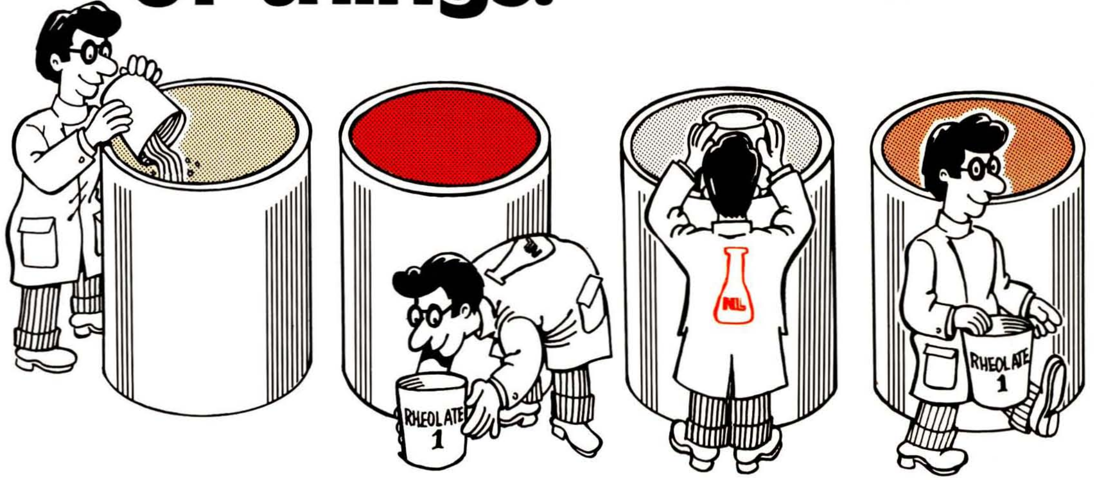

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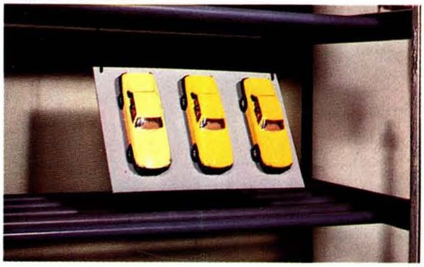
2 INNOVATIONS IN 1 PRODUCT

HEUCOTRON Yellow 5

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

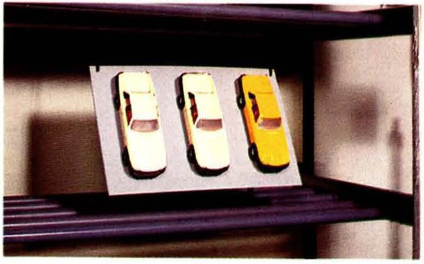
Reference sample

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.
With this 32-times-reduction of the acid soluble lead content in HEUCOTRON-Yellow 5 the above mentioned supposition of a potential hazard to human beings has lost its basis. This Factor should eliminate the substitution of chrome yellows by organic pigments.

High SO_2-Resistance
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

The Kesternich test has shown that HEUCOTRON-Yellow 5 possesses a significantly better resistance to SO_2 than the other two types. This characteristic predestinates HEUCOTRON-Yellow 5 for use in high quality paint systems such as those used for the final coat on cars and in other coating materials which are subjected to severe industrially induced atmospheric influences.

HEUCOTRON-Yellow	1064	123	5
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Light fastness	↗	↑	↑
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● red lead ● litharge ● battery oxides

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heubach

2 INNOVATIONS
IN 1 PRODUCT

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

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With this 32-times-reduction of the acid soluble lead content in HEUCOTRON-Yellow 5 the above mentioned supposition of a potential hazard to human beings has lost its basis. This Factor should eliminate the substitution of chrome yellows by organic pigments.

High SO_2-Resistance

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

The Kesternich test has shown that HEUCOTRON-Yellow 5 possesses a significantly better resistance to SO_2 than the other two types. This characteristic predestinates HEUCOTRON-Yellow 5 for use in high quality paint systems such as those used for the final coat on cars and in other coating materials which are subjected to severe industrially induced atmospheric influences.

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

heubach

Dr. Hans Heubach GmbH & Co. KG
Heubachstraße 7
Postfach 1160
D-3394 Langelsheim 1
Telefon (05326) 52-0
Telex 09 57 726 heuba d
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Harlingerode works

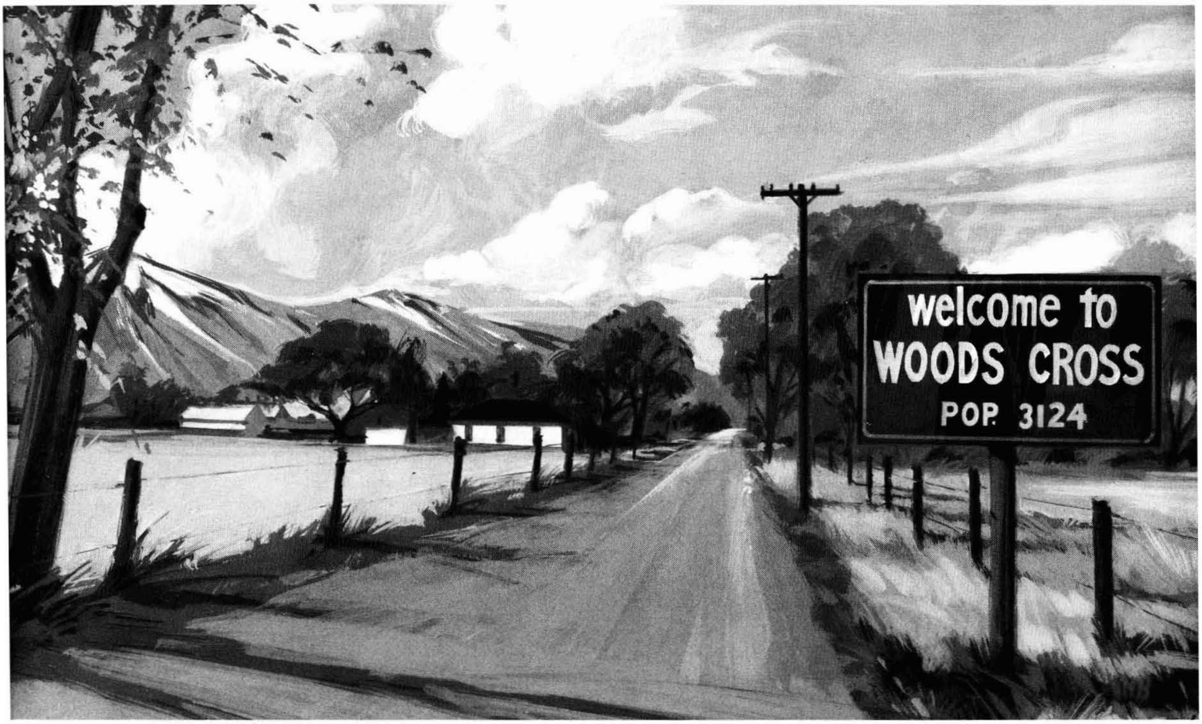
The companies hold a leading position on the German market in the following product areas:

- zinc oxide
- zinc dust
- zinc phosphate
- zinc chromate
- chrome yellow
- molybdate red
- special pigments
- red lead
- litharge
- battery oxides

Langelsheim works

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.

heubach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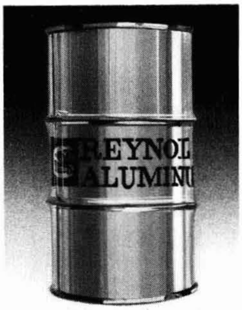
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NEOREZ R-900 polymers are colloidal dispersions of high molecular weight polyurethanes supplied in a water-reducible mixture. Both the aliphatics and aromatics can meet the toughest environmental and performance standards for industrial finishes. All are fully reacted and contain no free isocyanates, making them easy to handle. They can be applied by spray, roll or flow coat techniques and will air-dry to a hard, tack-free film.

R-940 A hard, tough aromatic polymer for primers and top coats for business machines and interior metal applications; excellent chemical, water and abrasion resistance.

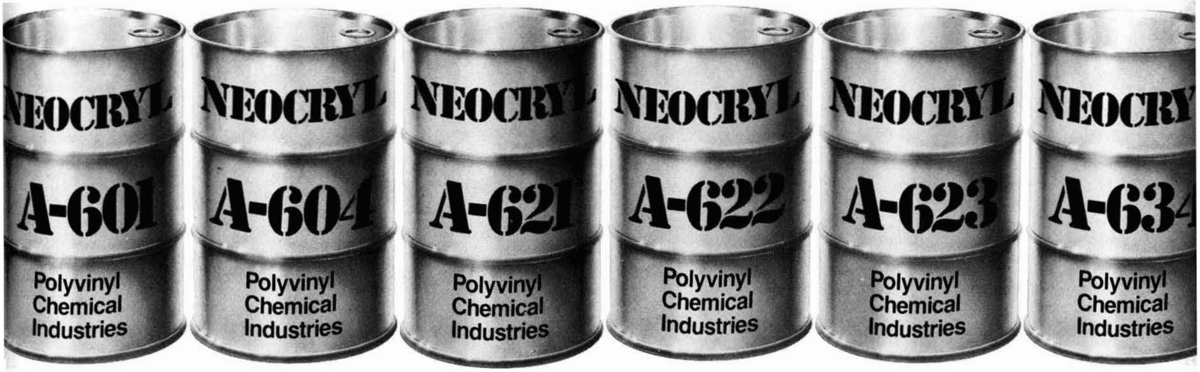
R-943 A general purpose, more flexible aromatic polymer with superior abrasion resistance for use on a wide variety of substrates; good impact, solvent and water resistance.

R-960 A high-performance aliphatic polymer with superior toughness for use on metal, wood and rigid and flexible plastics; exhibits excellent cold temperature flexibility; superior ultraviolet, chemical, water and abrasion resistance.

R-962 A very flexible aliphatic polymer with good adhesion for use in top coats for films and flexible foam; high elongation for forming soft extensible films; excellent abrasion and ultraviolet resistance.

R-963 A versatile aliphatic polymer with intermediate hardness and good chemical resistant properties for use on a wide variety of substrates; excellent water, ultraviolet and abrasion resistance.

R-966 A fast drying aliphatic polymer with good cold temperature flexibility for use on rigid and flexible substrates; hard; tough; excellent ultraviolet and abrasion resistance.


NEOCRYL
A-601
Polyvinyl Chemical Industries
NEOCRYL
A-604
Polyvinyl Chemical Industries
NEOCRYL
A-621
Polyvinyl Chemical Industries
NEOCRYL
A-622
Polyvinyl Chemical Industries
NEOCRYL
A-623
Polyvinyl Chemical Industries
NEOCRY
A-634
Polyvinyl Chemical Industries

Government / Industry

Hazardous Waste Site Selection Is Subject of Model Legislation

Model legislation to assist states in the handling of hazardous wastes has been prepared by the Chemical Manufacturers Association. It concerns site selection, treatment, storage, and disposal facilities.

Many states have encountered difficulties in finding locations for the construction of new or expanded disposal facilities. This legislation attempts to address the needs and concerns of the public by placing siting decisions in the hands of councils made up of state residents.

CMA's model bill would:

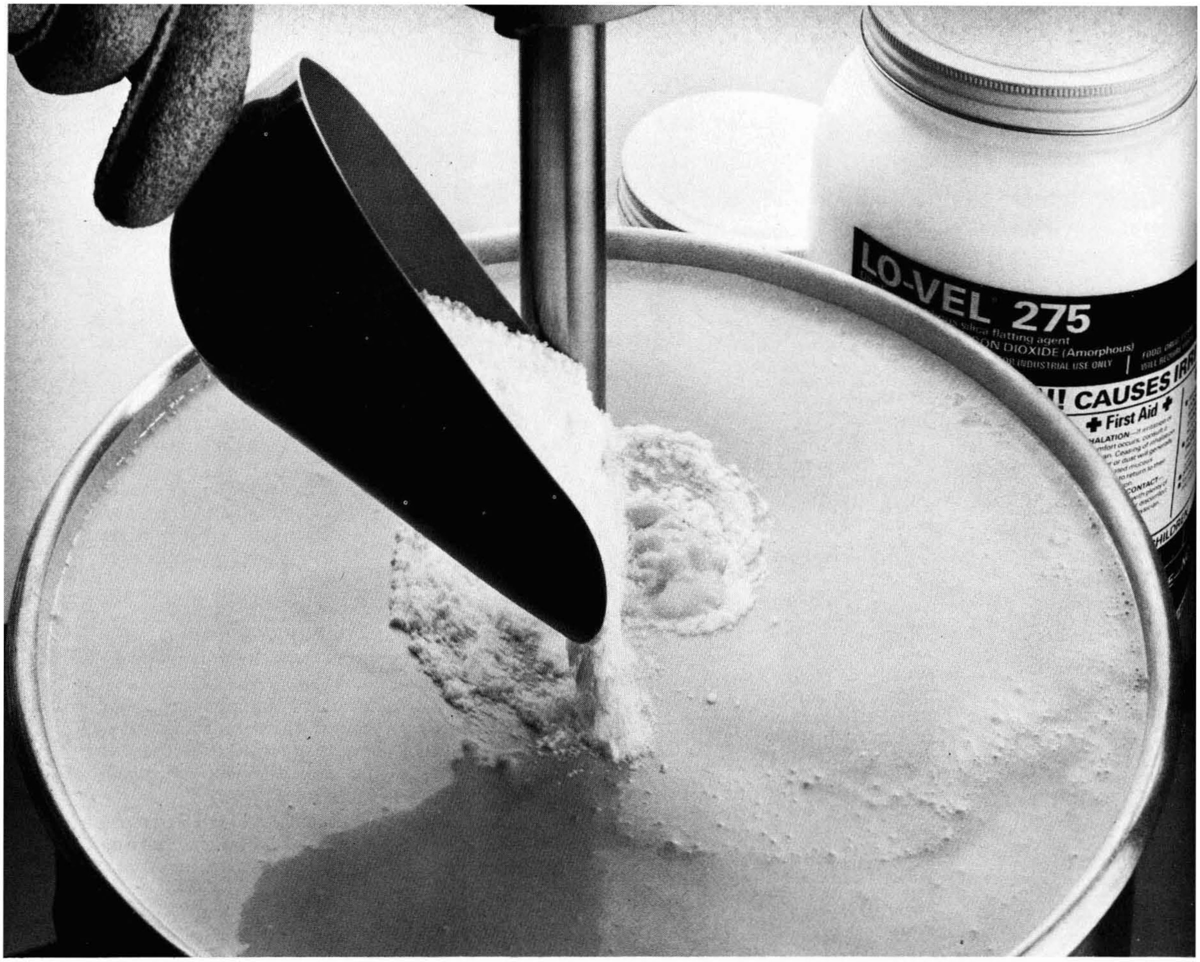
- Authorize the creation of a statewide Hazardous Waste Facilities Plan to locate a sufficient number of environmentally acceptable sites in a state.
- Require anyone proposing to site a new facility to obtain a certificate of environmental compatibility and public necessity. This would be in addition to required state and federal permits in order to assure that the facility would not impose unacceptable environmental risks.
- Provide that the legislation be administered by a Hazardous Waste Facility Siting Council made of permanent members from various sectors of the state and temporary members drawn from the locality of the proposed facility.
- Permit, as a last resort, council acquisition of land for a hazardous waste site and preemption of local zoning and other land-use control laws; and
- Create a state Hazardous Waste Facilities Corporation which would have the power, as a last resort, to acquire sites from the Hazardous Waste Facility Siting Council to either finance, construct or operate a hazardous waste treatment, disposal or storage facility or to lease sites to private operators for that purpose.

Hazardous Waste Facility Siting Councils would be established by Governors of the states. Each would be made up of seven permanent members serving three-year terms. The members could be reappointed to a second term. The councils, rather than being temporary ad hoc groups, would provide uniform policy, consistency of approach, continuity and the intimate knowledge necessary for equitable siting on a statewide basis.

In addition to the seven permanent members, two others would be appointed for each siting decision. These would be residents of the municipality or county in which the proposed disposal facility would be located, and be appointed by the chief executive officer of the city or county involved. Also, one of the permanent members would have to be a current or formerly elected official of either a municipal or county legislative body. Thus, three members would represent local viewpoints.

The remaining council members include one with knowledge of the industrial processes by which hazardous wastes are generated; one expert in environmental sciences; one with expertise in industrial planning, probably from a state agency; one engaged in the business of treatment or disposal of hazardous waste, and two members from the general public.

The final makeup of the council, then, would represent a balance of statewide interests.



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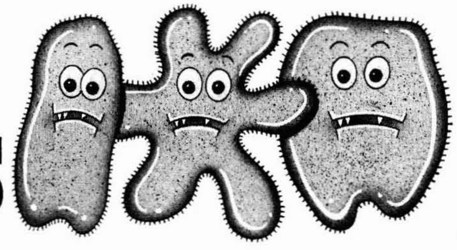
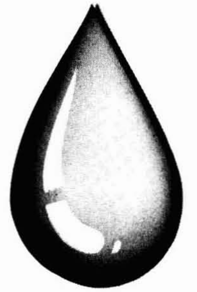
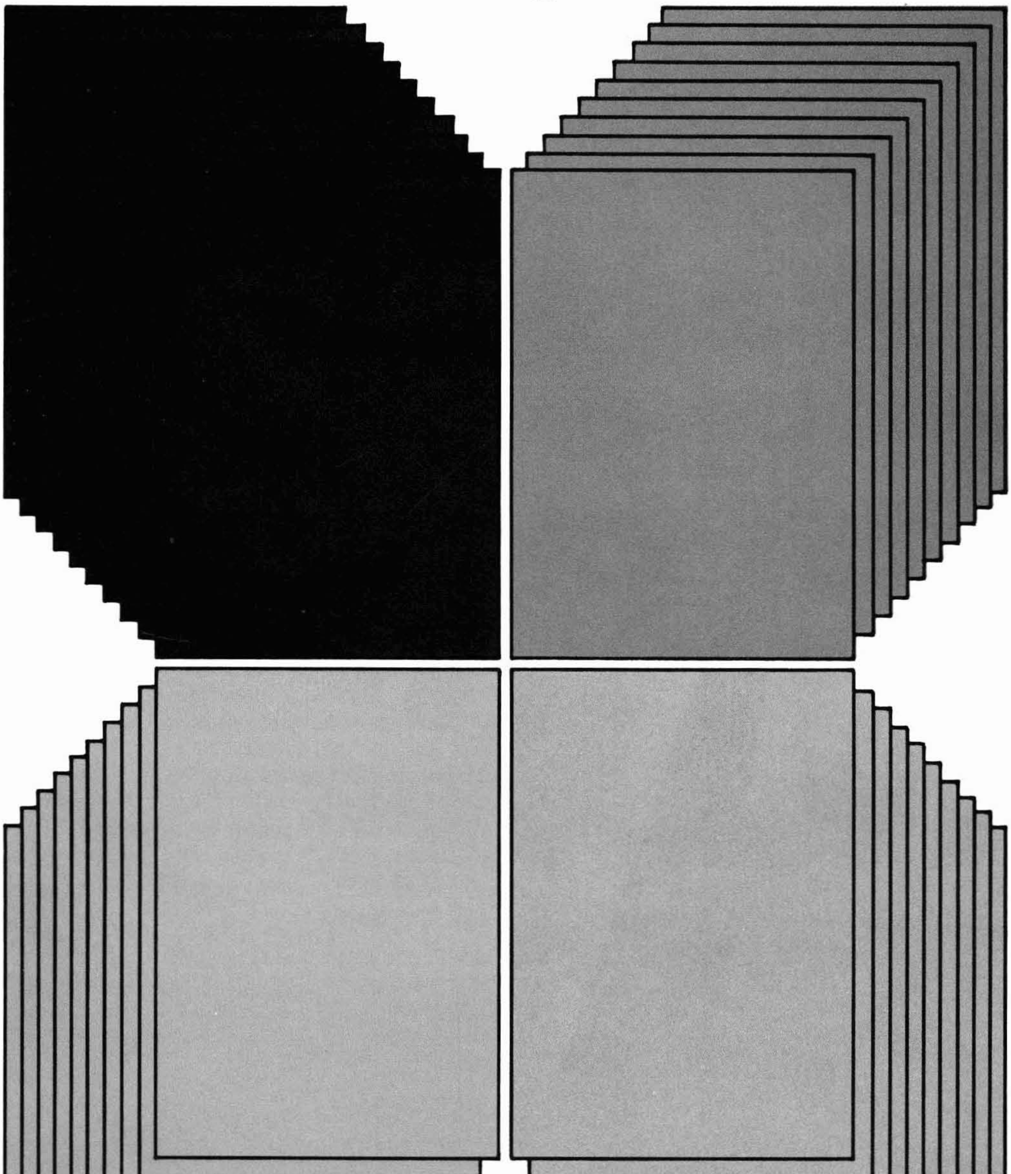




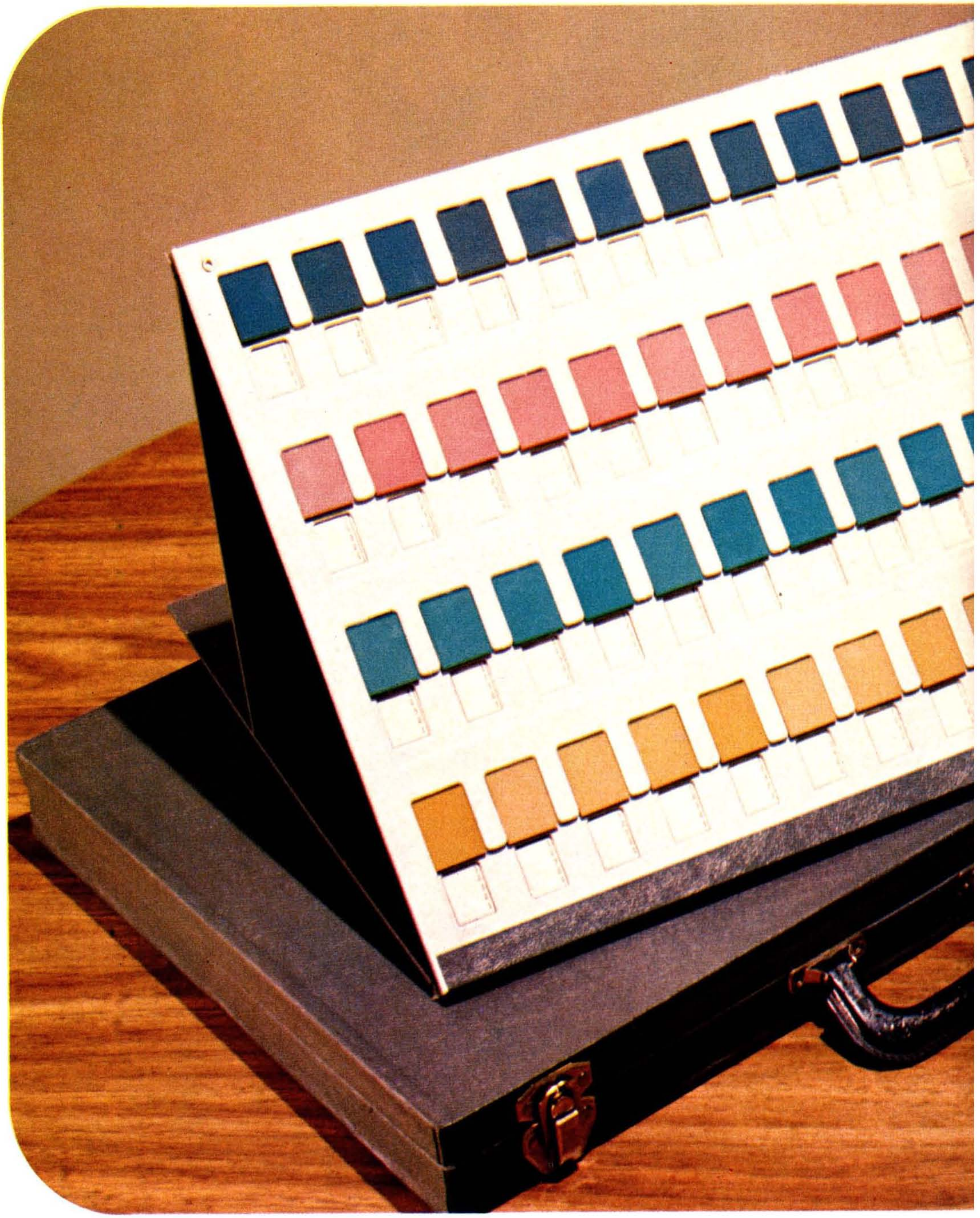
Federation of Societies for Coatings Technology

Color-matching Aptitude Test



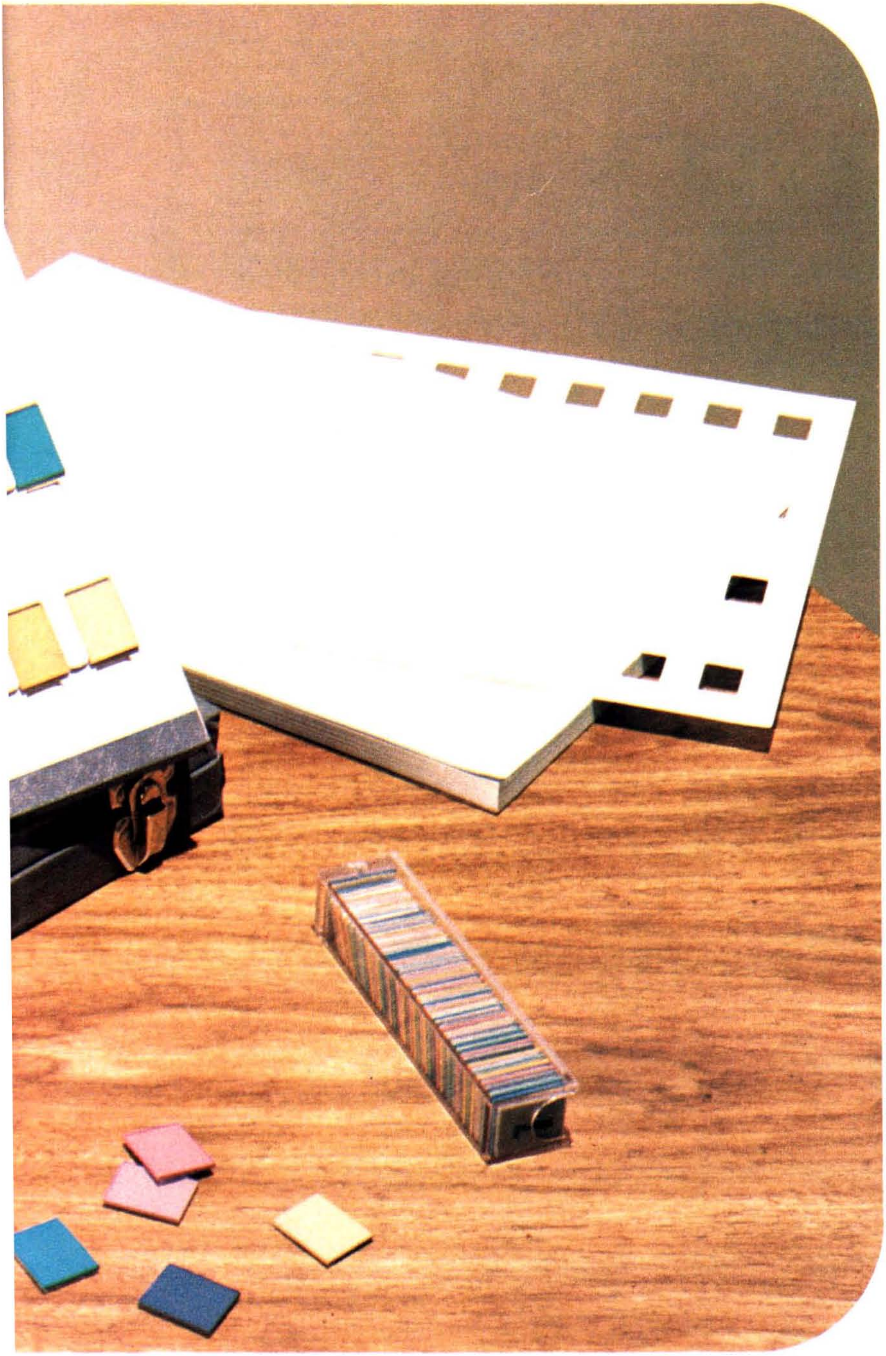
prepared by
The Color Test Evaluation Committee
of the INTER-SOCIETY Color Council

Color-matching Aptitude Test



The Color-matching Aptitude Test Set consists of a carrying case, an easel on which are mounted 48 color chips, a dispenser which holds 48 matching chips, score sheets, and a scoring key.

The 1978 edition



price
\$300.00
per set

plus shipping

were carefully established using spectrophotometric standards, and the intermediate steps produced by mixing the end-point colorants in proportions determined gravimetrically. The series thus produced were tested by 20 to 50 observers to make sure that they progressed in regular steps. Not until the series were found acceptable in this respect were they incorporated in the final Test.

LIGHTING

The test results are most satisfactory when the lighting is of the same spectral quality (average north-sky daylight with a correlated color temperature of about 6700 K) and of the same level of intensity (about 50 foot candles or 540 lux) as those used in standardizing the chips. Standard viewing booths or fluorescent lamps approximating daylight (with a color-rendering index of 85 or higher) should be satisfactory. Unfiltered incandescent (tungsten) lighting should be avoided because the chips so illuminated all appear more yellowish and close discrimination is more difficult.

The angle of the easel is set so that lighting from directly above the samples and perpendicular viewing will eliminate specular reflections from the chips into the viewer's eyes. Light from the illumination source should be shielded from the observer's eyes and no shadows should be allowed to fall on the Test easel. The chips should be viewed from a distance of ten inches or more.

STANDARD CONDITIONS

Although production of a proper set of chips is a prime essential, many other factors have to be controlled before a satisfactory test is achieved. If the Test is to measure gradations in ability to make color matches, subjects cannot be allowed to use their own special tricks to help them in deciding which colors match. In order that the matches may result in an adequate quantitative score the Test must be given under standard conditions. Therefore, one of the duplicate series of color chips which make up a Test set is mounted in a fixed order on a gray background with ½ inch strips between chips. The order is such that any two adjacent chips on the mounted field are perceptibly different and therefore one cannot obtain clues from a comparison of two of the mounted chips at the same time. Likewise the mounting leaves only one edge of the chip free for matching so that the judgment of match is always made with the samples in a standard orientation. Only one loose chip at a time can be taken by the matcher and he is allowed to make only the selection of the best match for that single chip. There will be many cases in which the subject is uncertain as to which is the best match. Nevertheless, he must decide before he can go on to the next match because he must return the chip to the holder in order to get the next one.

The chip holder also serves to present chips for matching in a standard order. The order is random so that the subject cannot relate any match to a previous match in the same hue series. In addition, the standard order assures that the Test is the same for everyone who takes it, and that everyone performs the same task in the same way.

STANDARDIZATION

Standardization of the 1964 edition was based on experience obtained from evaluation of a large number of scores contributed by users of the 1953 edition. The changes in the 1978 edition relative to the 1964 edition are not sufficient to require new standardization.

The Inter-Society Color Council Subcommittee for Problem 10, Color Aptitude Test, will appreciate receiving copies of Test scores, with an appropriate description of the level of experience of the individual tested in each case, and any pertinent comments and criticisms of the Test which the user wishes to make. It is only in terms of the user's evaluation of the usefulness of the Test that the Subcommittee can continue its work of providing tests which are meaningful.

Send results and comments either to (1) Color-matching Aptitude Test, Federation of Societies for Coatings Technology, 1315 Walnut Street, Philadelphia, Pennsylvania 19107, or to (2) Color-matching Aptitude Test, Office of the Secretary, F. W. Billmeyer, Jr., Inter-Society Color Council, Department of Chemistry, Rensselaer Polytechnic Institute, Troy, N.Y. 12181. Continued contributions of results from users of the Color-matching Aptitude Test will help to ensure that tests of value to persons involved in color, color-matching decisions, etc., will be created, refined, and made most useful.

order from
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FEDERATION OF SOCIETIES FOR COATINGS TECHNOLOGY
presents
COLOR-MATCHING APTITUDE TEST

1978 Edition
prepared by
THE INTER-SOCIETY COLOR COUNCIL
SUBCOMMITTEE FOR PROBLEM 10, COLOR APTITUDE TEST

COLOR-MATCHING APTITUDE TEST

In 1940, the Inter-Society Color Council appointed a committee to undertake the development of a color aptitude test. After preliminary trials, both in the production of materials for the Test and in the establishment of proper testing techniques, the Experimental Edition of 1944 was produced. The excellent performance of the 1944 edition has been described both in reports to the Council and in independent journals. With the financial support of the Federation of Societies for Coatings Technology, a member body of the Inter-Society Color Council, a second edition of the Color Aptitude Test was released in 1953, and a third edition in 1964. No major changes have been introduced in the Test since the 1944 edition. The present 1978 edition incorporates only minor refinements which bring it closer to the original plan and should make it a still more successful tool for evaluating color-matching skill. The name has been modified for the 1978 edition to indicate more clearly the purpose of the Test, to provide a means for estimating color-matching skill.

AIM OF TEST

The basic aim of the Test is to provide an objective measure that will aid in determining an individual's ability to perform color-matching tasks accurately. A person's color-matching skill may change over a period of time, improving due to training, experience, and motivation, or deteriorating from lack of practice or as a result of health disorders. Retesting at regular intervals will provide an indication of any such changes in an individual's color-matching ability.

The Test is not designed to indicate or measure "color-blindness" (anomalous color vision). Special tests designed for this purpose should be used.

DESCRIPTION

The Test consists of duplicate sets of samples, from which a subject must make forty color matches. Each set contains four groups of samples having constant hue and lightness but varying in saturation. Saturation series were chosen because in the preliminary work this dimension provided a satisfactory measure of matching skill and because series of equally spaced saturation steps can easily be produced with available industrial techniques. The total number of matches is limited, first by the number that an individual can be expected to make in one sitting, and second by the size of the field that can be properly lighted with simple equipment. The steps in the series have been made so small that rarely, if ever, will a subject make all matches correctly. This provision has two advantages. It places no upper limit on the score an individual can obtain, and it provides a basis for differentiation among persons in the higher brackets of color-matching ability.

In order that the forty matches furnish a good prediction of the subject's ability to match other colors, great care has been used in the selection and production of the color chips. To provide adequate coverage of the color range, matches must be made in each of the four basic hues, red, yellow, green, and blue, approximately Munsell hues 7.5 RP, 7.5 YR, 2.5 PB and 5.0 BG. The lightness of the samples is close to Munsell value 5/ and their saturation ranges from Munsell chroma /3 to /6.

The colorants used in the Test were chosen to assure permanence of the samples against fading. The color is applied as a coating to the back of plastic chips so that the surface is protected against abrasion. The resulting high gloss of the front surface of the chips requires the use of correct angles of illumination and viewing, specifically 45° from and along the normal (perpendicular) to the surface, respectively. When these angles are maintained there is no specular reflection from the chips into the subject's eyes. Since the subject must view a chip from directly in front, it may be necessary for him to shift his position from left to right as he works on the matching.

It is desirable that the steps in each series be equal. To assure this, the end-point colorants of each series

INSTRUCTIONS

for the Use and Care of Your

INTER-SOCIETY COLOR COUNCIL COLOR-MATCHING APTITUDE TEST

1978 EDITION

CONTENTS:

An easel with 48 fixed color chips, a chip dispenser with 48 corresponding loose chips, 100 scoring sheets, a scoring chart, and a carrying case.

PURPOSE:

This test evaluates a person's efficient use of color perception by means of the accuracy with which he makes color matches. The color gradations are chosen so that the average individual may be expected to choose one of several colors as a match for a given chip. Only one is an "accurate" match. No individual is expected to choose only the "accurate" matches.

The use of very fine color gradations was dictated by the fact that the Test is intended for individuals who must work accurately with colors. "Color-matching aptitude" scores will depend upon the inherent color interests of the subject, his motivation at the time of testing, his previous training with color discrimination and his native capacity for profiting by training.

PROCEDURE:

THE TEST MUST BE GIVEN UNDER STANDARDIZED CONDITIONS, otherwise the scores cannot be interpreted correctly. Most of the controls are incorporated in the Test itself: (1) the matching field has a fixed arrangement of colors, (2) order of matching is predetermined by the numbering of the chips, (3) matching comparisons are reduced to one at a time and a standard position for comparison is assured by the arrangement and the spacing of the mounted chips, (4) the angle of the easel provides perpendicular viewing.

ILLUMINATION:

The test has been designed and standardized for average daylight conditions, therefore it should be given under this type of illumination. The test materials have been measured spectrophotometrically and their color specifications have been determined for C.I.E. illuminant C (about 6700°K). Standard viewing booths with simulated daylight illumination or fluorescent lamps approximating daylight (with a color rendering index of 85 or higher) should be satisfactory. Natural north daylight or light from a moderately overcast sky should be satisfactory. Incandescent (tungsten) lighting should be avoided. Whether natural or artificial light is used, the illumination should be uniform over the test field at approximately 50 foot candles.

Light should not fall into the subject's eyes and no shadows should be cast on the "matching field." Because the surface of the chips is glossy, standard conditions of 45° illumination and perpendicular viewing must be met by placing the light source directly above the test. Illumination of the subject's face or clothing must be avoided to prevent objectionable reflections. The subject must not be permitted to view the chips from a distance of less than 10 inches.

CHIP NUMBERS AND THE ORDER OF PRESENTATION:

The numbers on the backs of the loose chips give the order of presentation of the chips for matching. The subject should begin with chip #1 and proceed through the numerical series to chip #48. The chip holder is loaded so that the chips will be obtained by the subject in this order. If for any reason the original arrangement should be disturbed, e.g., by several of the chips falling out of the chip holder, the proper order should be restored.

SCORING:

Scores for matches are indicated on the "Scoring Key" in terms of the serial numbers of the order of presentation.

Scores are given for only 40 chips—#9 to #48, inc. Chips numbered 1 to 8 are introduced to give the matcher initial practice in the matching task under test conditions. They are easier matches to make than the rest of the series and are not given scores. However, the person taking the test should do his best to match them correctly, because in so doing, he will improve his performance in the remainder of the test.

Scores for every match appear on the Scoring Key alongside the spaces in which the subject has written the numbers of the loose chips. Scores have been weighted according to their predictive value of the total score determined from the standardizing test scores.

INTERPRETATION OF SCORES:

From the probabilities inherent in the scoring system, the following "cut-off" scores have been estimated:

0 - 48	Poor
49 - 65	Fair
66 - 74	Average
75 - 83	Good
84 - 109	Excellent

In some 200 tests, on which standardization of the test is based, about 5% fell in the "Excellent" category. Test-retest reliability in a limited group proved to be good.

Because of the extreme difficulty of the discrimination judgments required in this test, score steps of less than 5 units probably have no meaning. Until more validation data become available it is best to use the verbal designations given above.

TIME FACTOR:

No time limit has been set because analysis of test data indicates that the 40 judgments constitute the minimum number that will give acceptable reliability. Instructions to the subject emphasize the need to work rapidly but not carelessly. In situations where information is desired concerning speed of matching, time should be recorded for matching the 40 chips beginning with #9 and including #48. Two speed factors may be calculated:

$$\text{time per match} = \frac{\text{total time}}{40}$$

$$\text{score per minute} = \frac{\text{total score}}{\text{total time}}$$

No standard time factors are available.

PLEASE SUBMIT TEST SCORES

The Inter-Society Color Council Subcommittee for Problem 10, Color-matching Aptitude Test, will appreciate receiving test scores of results obtained, with an appropriate description of the level of experience of the individual tested in each case, and any pertinent comments and criticisms of the Test which the user wishes to make. It is only in terms of the user's evaluation of the usefulness of the Test that the Subcommittee can continue its work of providing tests which are meaningful.

Send results and comments either to (1) Federation of Societies for Coatings Technology, 1315 Walnut Street, Philadelphia, Pennsylvania 19107, or to (2) CAT, Office of the Secretary, F. W. Billmeyer, Jr., Inter-Society Color Council, Department of Chemistry, Rensselaer Polytechnic Institute, Troy, N.Y. 12181. Continued contributions of results from users of the Color-matching Aptitude Test will help to ensure that tests of value to persons involved in color, color-matching decisions, etc., will be created, refined, and made most useful.

CARE OF MATERIALS:

In constructing the Color-matching Aptitude Test, materials were selected that would provide the maximum resistance to color change either from exposure to light or chemical fumes. The color chips, the chip holder and the vehicle in which the pigments are dispersed are made of acrylic plastic. The pigments themselves are the most permanent known to the art. The highly glossy surface was purposely selected to facilitate cleaning the chips. In most instances it will be necessary only to wipe them off with a soft, lint-free cloth or a piece of optical tissue. In instances where this does not suffice, a damp cloth and mild soap may be used. DO NOT USE SOLVENTS OF ANY KIND, OR ANY ABRASIVE CLEANERS. While the surface will stand considerable cleaning with water, and the adhesives used are of the waterproof type, care should be exercised to prevent water from swelling the felt backing and smudging the numerals on the loose chips. While the color chips will exhibit excellent light fastness, they should not be unduly exposed.

REPLACEMENT PARTS:

In the event any of the color chips become lost or damaged replacements may be ordered from the Philadelphia office of the Federation of Societies for Coatings Technology (Address below.) Chips from the holder may be ordered by number. In ordering chips for the easel, where the numbers are not known, send in a score sheet marked with an X for each position you wish to replace. Additional score sheets, scoring keys, or other parts may be ordered in the same manner.

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of the Federation of Societies for Coatings Technology

PROCEEDINGS/NUMBER 139

Application of Hydrodynamic Volume And Swelling Theory to the Crosslinking of Latex Particles

George M. Crews, Gary C. Wildman,
John R. Grawe† and B. George Bufkin
University of Southern Mississippi*

An *in situ* hydrodynamic approach to swelling ratios was investigated as a means of monitoring the degree of crosslinking of emulsion particles while in suspended form. The accuracy of the hydrodynamic technique was examined in comparison with a free film swelling technique using precrosslinked, butyl acrylate/diethylene glycol diacrylate (DEGDA) model emulsions. The results obtained from both methods supported the expected trend of a decrease in swelling ratio with increasing concentration of crosslinking agent.

The theoretical utility of the hydrodynamic technique was demonstrated by using the swelling ratio values to obtain the molecular weight of the polymer segments between crosslinks (M_c). Using the M_c values, the crosslinking efficiency of DEGDA was estimated to be 31% and 16% when employed at 0.42 and 3.15 mole % levels, respectively.

The applied utility of the technique was illustrated by using swelling ratios to monitor the migration rate of an external amine curing agent from the aqueous phase to an epoxy-containing polymer phase.

INTRODUCTION

Advantages of Crosslinking

Over the past decade, a growing awareness has emerged concerning the need to develop high-integrity, water-borne coatings which provide the application and performance characteristics of conventional solvent-borne systems. In response to this increasing need, the coatings industry has placed considerable emphasis on the research and development of an advanced generation of industrial-quality, water-borne vehicles. Unfortunately, the technology required for the development of the desired water-borne systems has been difficult to establish, and, as a result, several intermediate generation products have been introduced which represent an optimization of prior technology. For example, industrial maintenance coatings have been developed from conventional thermoplastic emulsion vehicles through an optimization of the surfactant and monomer composition and by increasing the molecular weight of the polymer. While these modifications significantly improve the performance characteristics of the resulting product as compared with the parent system, in general thermoplastic emulsions lack the adhesive and cohesive strength; abrasion and mar resistance; long term acid, alkali, and solvent resistance; corrosion and temperature resistance; and general durability associated with such industrial products as vinyl, chlorinated rubber, epoxy, and polyurethane solvent-borne systems. Fortunately, the integrity and performance capabilities of emulsion systems can be greatly improved by introducing functional groups which are capable of forming crosslinks.

The many advantages which emulsion coatings display upon the formation of a network structure have been corroborated using various types of crosslinkable functionality,[1] and the degree of crosslinking obtained in these systems is reflected qualitatively by increases in tensile strength and solvent resistance. The increased integrity that crosslinking provides is illustrated in *Table* 1 which compares the properties of uncrosslinked and crosslinked films of a halogen-functional emulsion.[2]

---

*Department of Polymer Science, Hattiesburg, MS 39401.
†Present address is Brevard College, Dept. of Chemistry, Brevard, NC 28712.

Table 1—Effects of Crosslinking on the Film Properties of a 60/32.7/7.3-EA/STY/CMS Copolymer Emulsion[a]

Crosslinking Agent[b]	Hardness Sward	Hardness Pencil	Impact Resistance Forward/Reverse (in.-lbs)	MEK[c] Double Rubs/Mil	MEK Insolubles (%)	Tensile Strength (Kg/cm²)	Elongation (%)
None	8	4B	160/160	24	5	51	1200
Disodium succinate	10	2B	100/70	135	60	100	920

(a) EA = ethyl acrylate, STY = styrene, CMS = p-chloromethyl styrene
(b) Films were cured 30 min at 250°F
(c) MEK = methyl ethyl ketone

More Quantitative Approaches To Crosslinking

Besides using qualitative methods to verify the formation of a network structure, the extent of crosslinking may also be monitored using volumetric free film swelling determinations.[3,4] Volumetric swelling ratios, which represent the volume ratio of the solvent swollen polymer to unswollen polymer, are commonly obtained by determining the unidimensional increase of a crosslinked film which has been immersed in and equilibrated with a suitable solvent.[5] By combining the equilibrium swelling ratio values, the polymer-solvent interaction parameter, and the molecular weight of the uncrosslinked polymer, the molecular weight between crosslinks for the network structure may be obtained by application of the Flory-Huggins theory,[6] i.e., $-[\ell_n(1-1/S) + 1/S + \chi/S^2] = (V_1/\bar{v}M_c)(1-2M_c/M)(1/S)^{1/3} - 1/2S$ where S = swelling ratio (swollen polymer volume/unswollen polymer volume), χ = polymer-solvent interaction parameter, V_1 = solvent molar volume, $\bar{v}$ = specific volume of polymer, M_c = molecular weight between crosslinks, and M = molecular weight of uncrosslinked polymer.

Swelling Ratio Theory As Applied to Emulsions

While swelling ratio values may be obtained using a gravimetric or volumetric swelling method performed on free films of the emulsion, an alternative approach has been developed, using viscosity theories, which characterizes the emulsion while in suspended form. Thus, one of the salient advantages of this technique lies in the ability to determine the degree of crosslinking which occurs prior to film formation.

Under ideal, although limiting, conditions, the viscosity of a suspension may be expressed as a function of the volume fraction of the internal phase, i.e., $\eta = \eta_0(1 + 2.5\ \phi)$ where η is the viscosity of the suspension, η_0 is the viscosity of the external phase, and ϕ is the volume fraction of the internal phase.[7] Since the Einstein equation is not valid for values of ϕ greater than 0.02, an expanded form has evolved which employs a power series in the volume concentration term, i.e., $\eta = \eta_0(1 + \propto_0\phi + \propto_1\phi^2)$. Using calculated values for the coefficients of ϕ, Guth, Simha,[8] and Gold[9] determined that $\propto_0 = 2.5$ and $\propto_1 = 14.1$ which extended the validity of the equation to a maximum volume fraction of 0.06. This value of ϕ provides a sufficient change in the viscosity of the suspension so that small changes in volume fraction, and, thus, crosslinking can be detected.

In order to obtain a working relationship between viscosity and the volume fraction of the internal phase, a study was conducted to examine the validity of the Einstein ($\propto_1 = 0$), Guth-Simha-Gold ($\propto_1 = 14.1$), and Oliver-Ward[10] ($\propto_1 = 6.25$) coefficients. The investigation utilized known volume fractions of polymer, ϕ, which were prepared by dispersing a precrosslinked

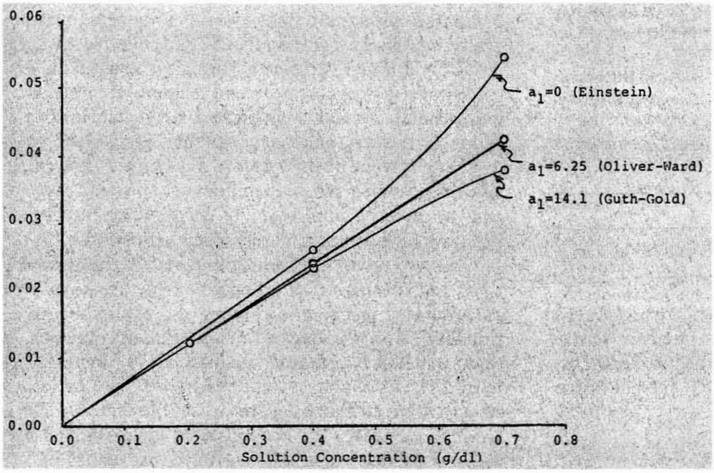

Figure 1—Evaluation of the power series coefficients

acrylic emulsion in an organic solvent (acetone). Viscosity measurements were conducted on both the continuous phase (η_0) and the resulting suspension (η). Using the Einstein, Guth-Simha-Gold, and Oliver-Ward constants in combination with the measured values for η and η_0, ϕ was calculated as a function of each coefficient. The results of this treatment are illustrated in *Figure* 1. In light of the linearity obtained with the Oliver-Ward coefficients, the expression

$$\eta = \eta_0(1 + 2.5\phi + 6.25\phi^2) \qquad (1)$$

was chosen as representing the most suitable relationship between the viscosity of the suspension, the viscosity of the continuous phase, and the volume fraction of the polymer. Using the Oliver-Ward constants, no variations from linearity were observed in the range of concentration used.

The Oliver-Ward power series [equation (1)] may be used to arrive at an expression which relates swelling ratios with measurable hydrodynamic quantities. For example, an expression for the internal phase volume can be obtained by rearranging equation (1) to set it equal to zero and applying the quadratic formula:

(1) Rearranging equation (1):

$$(6.25\eta_0)\phi^2 + (2.5\eta_0)\phi + (\eta_0 - \eta) = 0$$

(2) Applying the quadratic formula:

$$\phi = \frac{-2.5\eta_0 \pm \sqrt{6.25\eta_0^2 - 25\eta_0(\eta_0 - \eta)}}{12.5\eta_0}$$

(3) Cancelling the η_0 terms:

$$\phi = \frac{-2.5 \pm \sqrt{6.25 - 25(\eta_0 - \eta)/\eta_0}}{12.5}$$

(4) Rearranging the equation:

$$\phi = \frac{\sqrt{6.25 + 25(\eta - \eta_0)/\eta_0} - 2.5}{12.5} \qquad (2)$$

The volume of a dispersed polymer in a continuous phase is obtained through density measurements and may be expressed as a mass-density quotient, m/ρ_p, where m is the weight of particle solids per cubic centimeter of suspension, and ρ_p is the dry polymer density. Using these parameters and the volume fraction of the internal phase, the swelling ratio, S, can be expressed as:

$$S = \frac{\phi}{m/\rho_p} \quad \text{or} \quad \phi\rho_p/m \qquad (3)$$

By substituting ϕ from equation (2) into equation (3), a working expression is obtained which relates the swelling ratio of a suspended polymer to measurable hydrodynamic quantities, for example:

$$S = \frac{\rho_p[\sqrt{6.25 + 25(\eta - \eta_0)/\eta_0} - 2.5]}{12.5\ m} \qquad (4)$$

EXPERIMENTAL

The poly(butyl acrylate) emulsion and butyl acrylate/DEGDA copolymer emulsions used in the swelling ratio determinations were prepared according to the following sample procedure.

Preparation of a Poly(butyl acrylate) Emulsion

A 500 ml, four-neck flask was fitted with nitrogen inlet tubes, thermometer, agitator, and condenser. The apparatus was charged with 100 g of deionized water, and the system was deoxygenated by heating and sparging with nitrogen gas. After reaching 95° C, 1.87 g of Triton® X-200 (28% solids), 0.50 g of n-dodecyl mercaptan chain transfer agent, and 0.20 g of ammonium persulfate were added to the degassed water. After the addition was complete, the temperature of the mixture was adjusted to 80° C, and the rate of agitation was increased to a moderate level. After a short equilibration period, 100 g of butyl acrylate were added to the initiator-surfactant-water mixture at a rate of approximately 6 ml per min. Throughout the addition of the monomer, the pH was maintained at 7.0 with NH_4OH, and cooling was used to maintain the reaction temperature at 80° C. After complete addition of the monomer, 0.05 g of initiator were added, and the reaction temperature was maintained at 80° C for 4 hr in order to complete the polymerization. At the end of the gestation period, the temperature was increased to 90° C in order to insure complete conversion. The resulting emulsion was cooled to room temperature and filtered through cheese cloth. Only small amounts of coagulum developed on the thermometer, stirrer, and flask, and 98% conversion was obtained.

Preparation of a 68.3 / 31.7-Methyl Acrylate/Glycidyl

METHACRYLATE COPOLYMER EMULSION: The procedure used to prepare the poly(butyl acrylate) emulsion was also used for the preparation of a 68.3 / 31.7-methyl acrylate/glycidyl methacrylate (GMA) copolymer emulsion, except that the following ingredients were used in the order listed: 400 g of deionized water, 33 g of Triton X-200, 0.38 g of n-dodecyl mercaptan, 0.83 g of ammonium persulfate, and a monomer mixture consisting of 131 g of GMA and 282 g of methyl acrylate. The post-addition of initiator was not utilized.

Swelling Ratio Determination

The following example illustrates the procedure used to determine the swelling ratio of a 98/2-butyl acrylate/DEGDA copolymer emulsion. Using this latex which contained 43.7% solids, a 0.4 g/dl suspension in acetone was prepared by adding 0.91 g of the latex dropwise to 100 ml of acetone with agitation.

Prior to viscosity measurements on the suspensions, the viscometer constants for a Cannon-Fenske No. 50 viscometer were determined using a conventional procedure.[11] The viscometer was calibrated for centipoise units. Also, the density of the unswollen polymer was determined by either drying the latex and using a displacement-type determination, or by measuring the density of the undiluted latex and applying the percent solids factor. Excellent agreement was found using either of these methods. The following results were obtained using these techniques:

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Table 2—Comparison of Hydrodynamic And Bulk Swelling Ratios

DEGDA[a] Mole %	DEGDA[a] Weight %	Hydrodynamic Swelling Ratio	Free Film Swelling Ratio
0.06	0.1	Soluble	Soluble
0.42	0.7	14.5 ± 0.7	18 ± 5
0.60	1.0	10.8 ± 0.5	13 ± 4
2.16	3.5	6.7 ± 0.3	7 ± 2
3.15	5.0	6.6 ± 0.3	7 ± 2

(a) Diethylene glycol diacrylate

(1) Viscometer calibration factors: Calibration constant, A, = 3.45 × 10^{-3}; Kinetic energy constant, B, = −2.6316.

(2) Density of unswollen polymer: ρ_P = 1.058 g/ml

A detailed exposition of the swelling ratio determination is as follows:

(1) Suspension density determination: volume of pycnometer, V, at 25° C = 25.858 ml; tare weight of pycnometer, P_T, = 29.5508 g; gross weight of pycnometer and suspension, P_g, = 50.2921 g; net weight of suspension = 20.7413 g; density of air, ρ_a, = 0.001169 g/ml; correcting for buoyancy, $K = 1 - \rho_a/8.4$ and K = 0.99986. Using the expression, $\rho_s = K(P_g - P_T)/V + \rho_a$, the density of the suspension, ρ_s, = 0.8032 g/ml.

(2) Suspension solids determination: weight of aluminum dish = 1.3660 g; weight of syringe and suspension = 34.4640 g; weight of syringe after discharge = 22.0494 g; net suspension weight = 12.4146 g; weight of dish and dried suspension = 1.4286 g; net weight of suspension solids = 0.0626 g; and weight of solids per gram of suspension, W_s, = 0.0050 g.

(3) Suspension viscosity determination:

Trial No.	Flow time (sec)
1	155.6
2	155.7
3	155.65

Average time flow time, t_s, = 155.65 sec.

Applying the expression for the suspension viscosity, $\eta_s = (At_s - B/t_s)\rho_s - \rho_a)$ = 0.3937 cp.

To determine the characteristics of the continuous phase, the components of the suspension were separated by means of ultracentrifugation. The suspensions were centrifuged at 14,500 rpm for 45 min at a temperature of 10° C. The clear supernatant fluid was then drawn off and characterized for density, percent solids, and viscosity. The sample calculations are as follows:

Table 3—Molecular Weight Between Crosslinks And Crosslinker Efficiency of DEGDA

Conc. DEGDA %	Molecular Weight between Crosslinks[a] (M_c)	Crosslinker Efficiency (%)
0.7	41,300	31.4
1.0	30,000	30.0
3.5	14,800	21.0
5.0	13,600	16.0

(a) M_n determined by means of membrane osmometry.

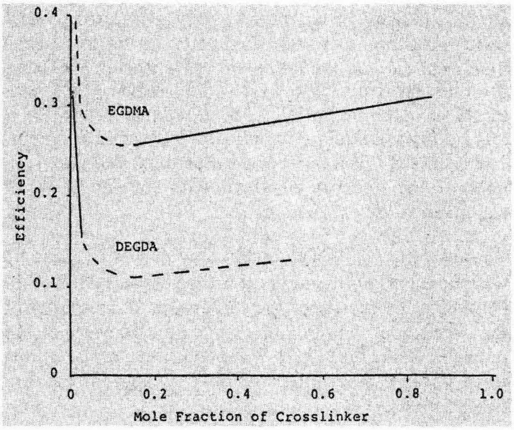

Figure 2—Crosslinking efficiency of diethylene glycol diacrylate and ethylene glycol diethacrylate. ——: measured; ....: predicted

(4) Continuous phase density determination: Repeating the steps outlined under suspension density determination and applying the relationship, $\rho_o = K(P_g - P_t)/V + \rho_a$, the density of the continuous phase, ρ_o = 0.7996 g/ml.

(5) Continuous phase solids determination: To determine the weight of solids per gram of suspension, W_o, the steps outlined under suspension solids determination were repeated. W_o = 0.00012 g.

(6) Continuous phase viscosity determination: The flow times for the continuous phase were determined as under suspension viscosity determination and from the relationship $\eta_o = At_o - B/t_o)(\rho_o - \rho_a)$, the viscosity of the continuous phase was obtained. η_o = 0.3656 cp and t_o = 144.40 sec.

(7) Calculating the mass of the particles in suspension: The mass, m, of the particles in the suspension is obtained from the relationship, $m = \rho_s(W_s - W_o)$. Thus, m = 0.80317 (0.0626 − 0.0012); m = 0.0031 g/ml.

(8) Determining the volume fraction, ϕ, of the internal phase: The volume fraction, ϕ, of the internal phase is obtained from equation (2); ϕ = 0.0286.

(9) Determining the swelling ratio: The swelling ratio, S, is obtained from equation (3). S = 0.0286 × 1.058 g/ml ÷ 0.0031 g/ml; S = 9.86.

Standard deviations were determined by measuring the swelling ratio values for a minimum of five samples.

Free Film Swelling Measurements

Free film swelling determinations were performed on a relatively thick film cast from the latex which was equilibrated in a solvent (acetone). The uptake of solvent was determined by weighing the swollen film. The swelling ratio was obtained from the relationship, $S = 1 + (Z - 1)\ \rho_P/\rho_s$ where Z is the ratio of solvent

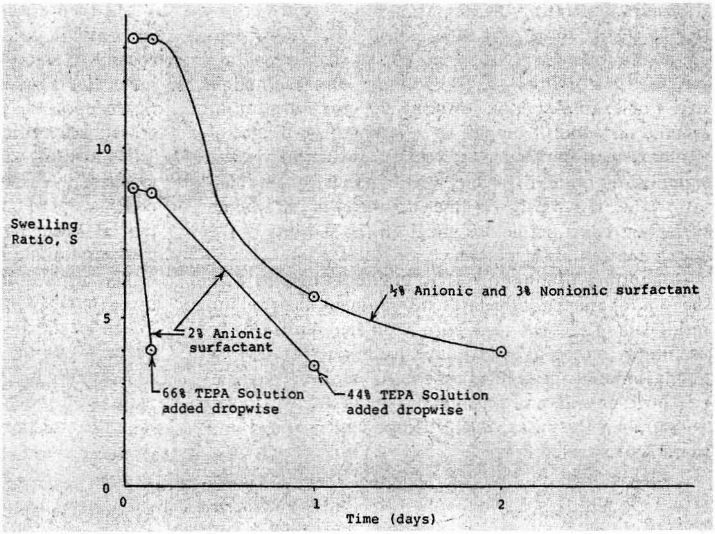

Figure 3—Swelling ratios of methyl acrylate-glycidyl methacrylate emulsion containing tetraethylenepentamine curing agent

swollen film weight to dry film weight, and ρ_s and ρ_p are the densities of the solvent and dry polymer, respectively.[12] Standard deviations were determined by measuring the swelling ratio values for a minimum of ten samples.

DISCUSSION AND RESULTS

The usefulness of the hydrodynamic technique to estimate the degree of crosslinking was investigated using precrosslinked, poly(butyl acrylate) emulsions containing 0.1, 0.7, 1.0, 3.5, and 5.0 weight percent of DEGDA. Both hydrodynamic and free film swelling techniques were performed, and the results of this study are presented in *Table* 2.

In general, both techniques reflected the predicted trend that an increase in crosslink density results in a subsequent decrease in swelling ratio. In addition, excellent agreement was obtained between the two techniques for determining the swelling ratio at higher degrees of crosslinking. However, some disparity between the two methods becomes more prominent at lower degrees of crosslinking. Conceivably, deviations from ideality may occur as a result of nonequilibrium solvation in the free film method or partial dissolution of lightly crosslinked polymer and non-Newtonian behavior in the hydrodynamic method.

The magnitude of the swelling ratio data for either method also suggests that less than 100% crosslinking efficiency was achieved using DEGDA. By combining the swelling ratio data of *Table* 2 with the polymer-solvent interaction parameter, $\chi = 0.469$, obtained from membrane osmometry, the crosslinking efficiency can be estimated from differences between the predicted and measured values of M_c. The results of such a comparison are presented in *Table* 3.

These data indicate that the crosslinking efficiency of DEGDA was approximately 31% and 16% when employed at 0.42 and 3.15 mole %, respectively. This trend somewhat contrasts previous findings which predicted that, at low concentrations, the crosslinking efficiency would approach 100%. Moreover, when ethylene glycol dimethacrylate (EGDMA) was utilized at 15 and 85 mole % levels, the crosslinking efficiency was found to be 26% and 31%, respectively.[13] A comparison of the crosslinking efficiency of DEGDA and EGDMA is presented in *Figure* 2. Conceivably, greater agreement between the crosslinking efficiencies of DEGDA and EGDMA could be obtained by using identical comonomer and reaction conditions and by employing empirically determined χ values.

In order to illustrate the practical applications for which the hydrodynamic swelling ratio technique is particularly suited, the migration of an external, amine crosslinking agent was followed by means of swelling ratio values for a 68.3 / 31.7-methyl acrylate/GMA copolymer emulsion. The experimental design included the use of several different crosslinking agents, variations in the method of addition of the curing agent, and the effect engendered by the ionic character of the surfactant. The swelling measurements were carried out in N,N-dimethylformamide solvent, and the swelling ratios were determined for the emulsion system every 24 hr. A portion of the results of this study using the stoichiometric amount of tetraethylenepentamine (TEPA) curing agent are presented in *Figure* 3.

In general, these data indicate that the migration of a water soluble crosslinking agent into the latex particle is quite rapid. The last measurement for each curve represents the point where a suspension could no longer be made because of a high degree of crosslinking. At this point, film coalescence was poor and resulted in very brittle coatings which cracked severely on drying. The minimum swelling ratio that could be determined for this system and solvent combination was approximately 3.5 and very brittle films resulted at swelling ratio values below 4.5.

Besides illustrating the migration ability of the crosslinking agent, these data also indicate that the hydrodynamic swelling ratio technique can be used to evaluate changes in formulation composition and methodology. For example, upon lowering the concentration of anionic surfactant from 2% to ½%, based on weight of the monomer, the degree of premature crosslinking was significantly reduced, as indicated by the larger swelling ratio value. Obviously, the nucleophilicity of the anionic surfactant has a pernicious effect on the stability of the epoxy content of the system.

In addition to monitoring the effects associated with the ionic character of the latex environment, the swelling ratio data presented in *Figure* 3 further illustrate the usefulness of the hydrodynamic technique for investigating some of the applied aspects of emulsion systems, i.e., both pot-life and through-dry times are greatly reduced when the curing agent component is added in a more concentrated form.

Thus, in light of the data obtained from both the DEGDA- and GMA- functional emulsion systems, the hydrodynamic swelling ratio technique was found to be a useful method for examining both the theoretical and applied aspects of crosslinkable emulsions.

SUMMARY

Measurement of the degree of crosslinking in emulsion systems was investigated using swelling ratio data obtained by application of a hydrodynamic technique. The approach was derived, in part, from a power series expansion of the Einstein equation modified with the Oliver-Ward coefficients to obtain an expression relating swelling ratios with measurable hydrodynamic qualities.

The accuracy of the method was examined in comparison with the conventional, free film swelling technique using a series of poly(butyl acrylate) model emulsions containing progressively increasing amounts of DEGDA crosslinking agent. While both techniques corroborated the expected trend of a decrease in swelling ratio with increasing concentration of difunctional acrylate monomer, some difference in agreement between the two methods was ostensible at low levels of crosslinking. However, a comparison of the standard deviation differences between the two methods indicate that the hydrodynamic technique possesses inherently greater reproducibility than does the free film swelling technique.

The theoretical utility of the technique was illustrated by combining the swelling ratio data with the polymer-solvent interaction parameter, χ, in order to obtain the molecular weight of the polymer between crosslinks, M_c. Based upon the predicted and calculated M_c values, the crosslinking efficiency of DEGDA was estimated to be 31% and 16% when employed at 0.42 and 3.15 mole %, respectively. The low crosslinking efficiency of DEGDA contrasts predictions from other studies which indicated that at lower concentrations of difunctional monomer the crosslinking efficiency would approach 100%.

The applied utility of the technique was illustrated using a GMA-containing emulsion which was crosslinked by the post addition of an external, amine curing agent. Using the hydrodynamic technique, the extent of crosslinking was monitored as a function of such parameters as the ionic strength of the surfactant and the rate of migration of the curing agent into the latex particle.

ACKNOWLEDGMENT

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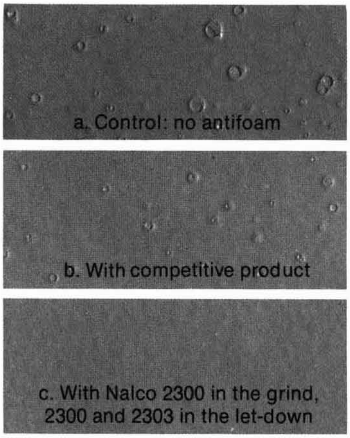

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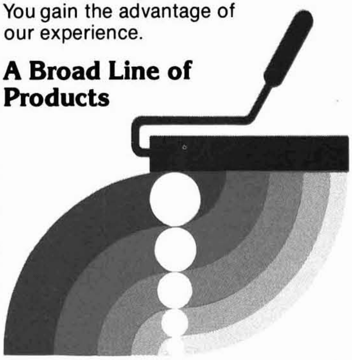

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Amine Loss from Water-Soluble Polymer Solutions Expressed As Simple Solvent Evaporation

Peter Kamarchik, Jr.
PPG Industries, Incorporated*

The behavior of various amines in three types of drying paint films was investigated by preparing films of the neutralized polymer from which solvent and amine were allowed to evaporate for specified times. The films were than analyzed for residual amine by acid titration. By this method it was possible to construct amine concentration versus time graphs for various temperatures and film thicknesses. The behavior of these plots indicates the similarity between amine loss and simple solvent evaporation during the time that the film remains "wet." For the most commonly employed amines, this period accounts for most of the amine lost. Furthermore, the temperature variation of the initial loss rate leads to an activation energy of amine loss comparable to the heat of vaporization of the amine under consideration. The effects of base strength are shown to be minimal, at least in the early stages of the loss.

INTRODUCTION

One of the major responses to the restrictions placed on solvent emissions in the paint industry is the widespread use of amines to effect the water solubility of polymers containing carboxylic acid functionality. The use of amines has allowed the development of a wide variety of water-soluble alkyds, acrylics, and polyesters.[1] The amines serve as proton acceptors for the carboxylic acid groups in the polymer backbone, resulting in the production of anionic sites which provide a relatively strong ion-dipole interaction between the polymer and water. The chemistry of amine salt formation has been extensively studied,[1-4] including the effects of the concentration of acid functionality, molecular weight, amine base strength, and cosolvent level of the system. These factors determine the solubilization efficiency[1] of an amine in a particular system and will, therefore, affect the choice of amine in the formulation. Another basis for deciding which amine to use in a given formulation is the behavior of that amine in a drying paint film. The presence of amine in a dried film is known to impart water sensitivity to the film. Generally, the lowest residual amine content possible is desired; however, to control effects such as wrinkling and solvent popping, the rate of loss may be required to be relatively low in certain stages of the drying process. Reports of some work on amine loss have already appeared in the literature,[5-7] but considerably more information is needed. This study was undertaken to more fully categorize the factors controlling the rate of amine loss and the factors controlling the level of residual amine from high acid content copolymers.

EXPERIMENTAL

Amines included in this study are ammonia (obtained as 15M ammonium hydroxide), triethylamine (TEA), triethanolamine (TEOLA), diethanolamine (DEA), and dimethylethanolamine (DMEA). All were obtained from Fisher Scientific as reagent grade and were used without further purification. The three resins solutions used in this study were: #1—an acrylic acid copolymer containing acrylates and styrene, acid value 1.58 meq/gm (on solids); #2—a copolymer of methacrylic acid and several acrylates, acid value 2.73 meq/gm (on solids); and #3—an alkyd with an acid value of 0.82 meq/gm (on solids). Unless otherwise noted, the solutions from which films of the resins were cast were prepared according to the following formulation: resin–150 gm; water–100 gm; amine–as stoichometrically required for 100% neutralization of acid functionality; butyl cellosolve–250 gm; and tributyl phosphate–3 gm.

The butyl cellosolve was added to improve flow-out characteristics when casting films and the tributyl phosphate was added to retard skinning. This prepara-

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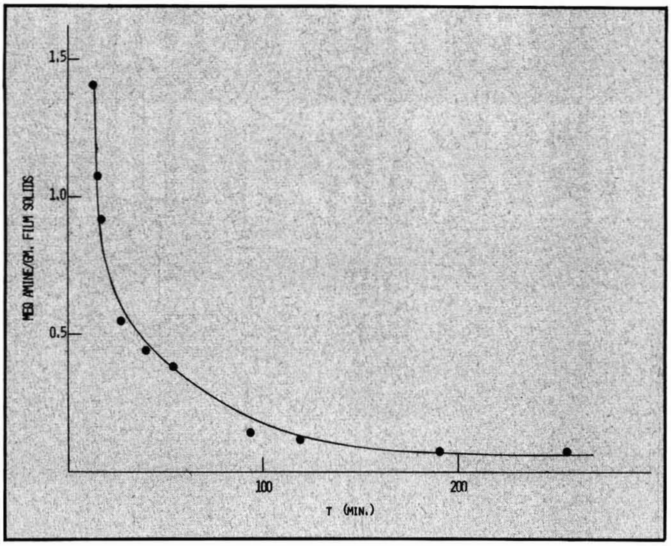


Figure 1—Loss of TEA from resin #1 at 73°C

tion resulted in films of approximately 10% resin solids and intitial neutralization of 100%.

Films were cast by weighing a quantity of the solution into 2 in. diameter aluminum weighing dishes. Two film thicknesses were generally prepared. The exact dry film thickness depends on the solids content and the particular amount of solution used. This was calculated for each film and used in subsequent calculations. These thicknesses were approximately 0.10 and 0.15 mm. The dishes were then placed in a controlled temperature oven on a steel plate which had previously been heated to the desired temperature. The good thermal contact between the steel plate and the aluminum dish resulted in rapid heat-up times. Similarly, when samples were removed from the oven, they were immediately placed on a cold surface to inhibit the loss process. The films or solutions (depending on bake duration) were then solubilized in a mixture of 20% propylene glycol/80% tetrahydrofuran and potentiometrically titrated with 0.1N aqueous HCl to determine the amine content. A back titration with 0.1N methanolic KOH then determined the total amine plus acid content. The data were tabulated as the number of milliequivalents of amine or acid per gram of resin solids.

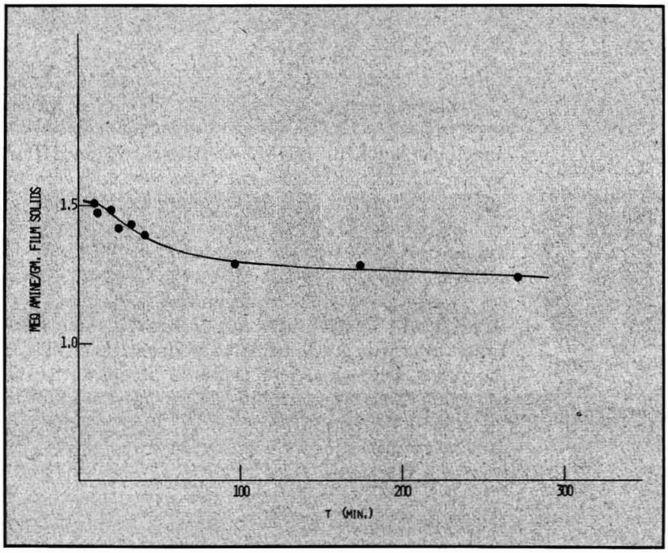


Figure 2—Loss of DEA from resin #1 at 73°C

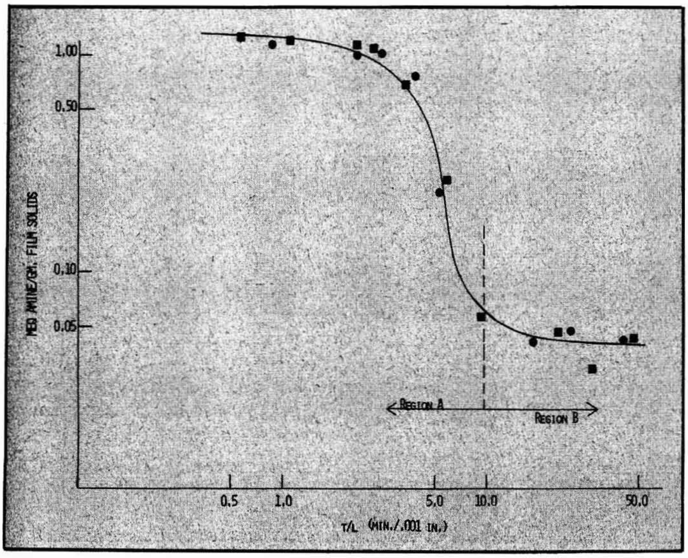


Figure 3—Log-Log plot of the loss of ammonia from resin #1 at 73°C. Legend: • 0.10 mm; ■ 0.15 mm

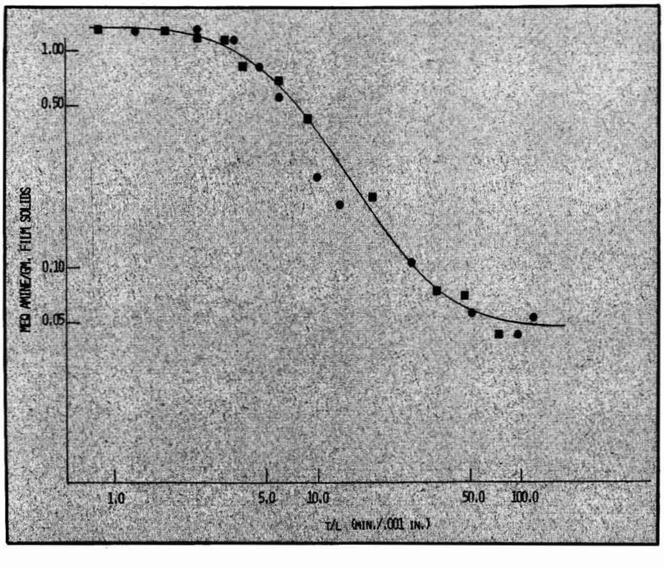
RESULTS AND DISCUSSION

Comparison with Solvent Evaporation

The amine loss curve for TEA from resin #1 is shown in *Figure* 1 and is given as the number of meq's of amine/gm solids vs time. The general shape is a curve of constantly decreasing slope; however, a simple log function is not a good fit to this curve. The general behavior exhibited by this sample is also shown for the loss of ammonia and DMEA, all three relatively volatile amines. The behavior of the nonvolatile amines is shown in *Figure* 2. This is exhibited by DEA and TEOLA.

Another graphic presentation of the amine-loss data is a plot of the log of the amine concentration vs the log of the time divided by the film thickness. These curves are presented in *Figures* 3 through 6. In all of these experiments, the bake temperature was set at 73°C±1°C. Hansen has shown [8-10] for solvent evaporation, that log-log plots of remaining solvent vs t/L and plots vs t/L^2, where t is time and L is dry film thickness, provide a means of distinguishing between boundary layer resistance and internal diffusion resistance as controlling factors of loss rate. It was shown that, if data for several different film thicknesses were plotted on the same graph,

Figure 4—Log-Log plot of the loss of TEA from resin #1 at 73° C. Legend: See Figure 3



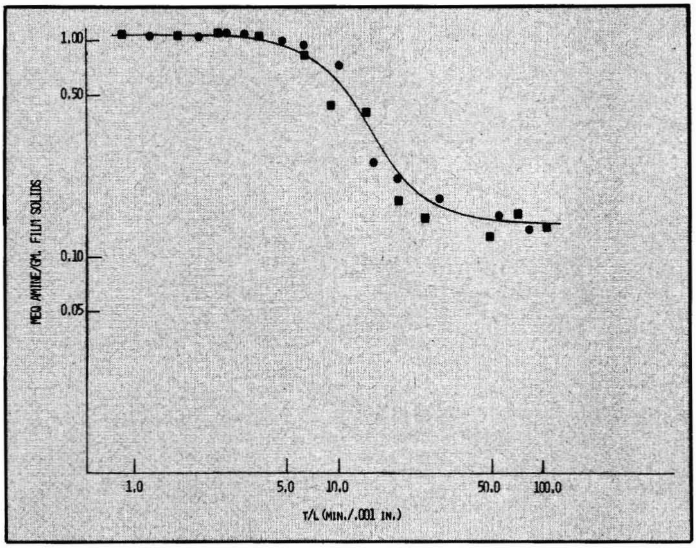


Figure 5—Log-Log plot of the loss of DMEA from resin #1 at 73°C. Legend: See Figure 3

one master curve would be followed on a t/L plot for boundary layer resistance to solvent loss while the curves for the different thicknesses would be seperate if the controlling factor was internal diffusion resistance. On a t/L^2 plot, a single master curve is followed if the controlling factor is internal diffusion resistance while the curves become distinct for boundary layer control. It should be noted though, that for curves with slopes sufficiently close to zero, a single master curve will appear to be followed on both t/L and t/L^2 plots regardless of the rate controlling mechanism. All amine-loss curves plotted in this manner are of the same general character as the curves obtained for solvent evaporation. The t/L plots show a single curve for all film thicknesses through region A (indicated on *Figure* 3) of the amine loss. However, in region B the slope is sufficiently small and the data scatter too great to distinguish whether a t/L or t/L^2 plot would better represent the data as a single line. This observation suggests that through region A which corresponded to films that were still fluid, the controlling factor for amine loss is the transport of amine across the liquid/vapor boundary layer. Furthermore, this wet stage has been observed to account for up to 50% of the amine loss. This is also in complete agreement with recent work by Hansen which demonstrated that through region A the loss of solvent is governed by the rate of evaporation of the pure solvent, while in region B other factors become rate controlling.[11]

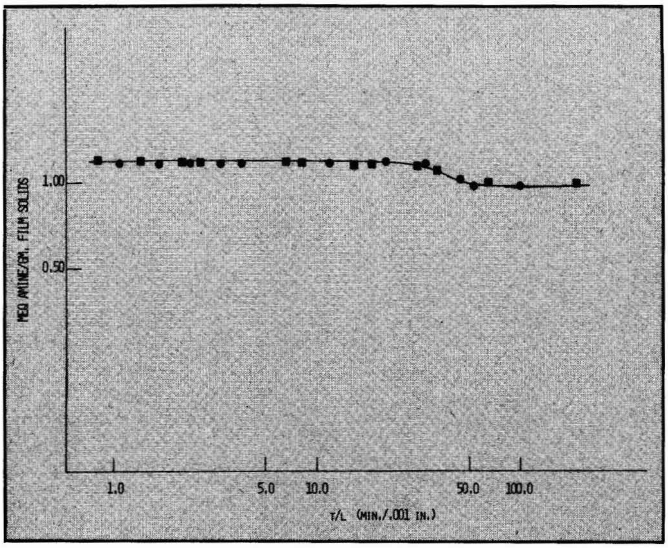


Figure 6—Log-Log plot of the loss of DEA from resin #1 at 73°C. Legend: See Figure 3

Table 1—Relative Volatilities of Some Amines[a]

	^{T}10% Loss	% Amine at 10 min	% Amine at 30 min
NH_3	7 min.	82.2	26.7
TEA	10	84.8	27.1
DMEA	16	98.5	59.1
DEA	136	99.7	99.1
TEOLA	412	99.3	93.0
EDA	36	98.8	96.1
TEDA	123	99.6	98.8

(a) Resin #1 at 73°C.

Table 3— Activation Energy Data for Amine Loss from Resin #1

Amine	T (K)	$\frac{1}{T} \times 10^3$	Rate (%/min)	Ln. Rate
NH_3	294	3.40	0.23	-1.46
NH_3	346	2.89	0.75	-0.29
NH_3	363	2.76	0.98	-0.02
TEA	346	2.89	0.33	-1.10
TEA	370	2.70	0.58	-0.53
TEA	383	2.61	0.74	-0.30
DMEA	346	2.89	0.17	-1.76
DMEA	363	2.76	0.33	-1.10
DMEA	383	2.61	0.56	-0.59

The data in *Table* 1 include several approaches to a measurement of the relative volatilities of the amines from the resin film. Boiling points and base strengths are given in *Table* 2. The amines included in this study show complete agreement between boiling point ordering and the ordering of relative volatility given in *Table* 1. The same behavior was observed by Wicks,[6] who also used NH_3, TEA, and DMEA as well as some amines not included in this work. These results show that the rate of amine loss follow the order NH_3>NEM>TEA>DMEA. It can be seen that, with the exception of N-ethylmorpholine (NEM), the controlling factor for the loss of amine from a high acid value acrylic polymer is the boiling point of the amine. The large difference in pKa values between NEM and the other amines presumably accounts for the reordering of volatility in these systems. These experiments suggest that salt formation has little effect on loss rate during the wet stage. This could be explained by reasoning that the equilibrium between salt and acid/base pair is fast compared to the rate of transport from liquid to vapor phase.

Activation Energy for the Amine Loss Mechanism

The rates of amine loss from films prepared from resin #1 neutralized with NH_3, TEA, DMEA, and DEA were observed at several different temepratures. The loss rate, determined as the tangent to the concentration vs time curve, was measured at the time of 20% loss of amine. These data are presented in *Table* 3 and in *Figure* 7. The value 20% was chosen in order to use a rate from the early stage of the drying process to eliminate problems of any "skinning" or solidification of the film. However, it is desirable that the percentage loss chosen should correspond to a dry time of several minutes to insure that the heat-up interval is small with respect to the time at loss rate measurement.

Table 2—Boiling Point and pKa's of Various Amines

	Boiling Point (°C)	pKa
NH_3	-33.4	9.40
TEA	88.8	10.88
DMEA	135	9.31
DEA	270	—
TEOLA	360	—
NEM	138.6	7.78

An Arrhenius plot, which displays the natural logarithm of rate vs the reciprocal of the absolute temperature, for the ammonia system gave an activation energy for ammonia loss of 4.5 kcal/mole. The activation energy is obtained by multiplying the slope of the Arrhenius plot by R, the universal gas constant. The heat of vaporization of ammonia is 4.7 kcal/mole. These values are in good agreement and tend to confirm transport from the liquid to vapor phase, as Hansen observed for solvents, as the rate controlling step in amine loss. Similar calculations were performed for TEA and DMEA. These resulted in E_a values of 6.0 kcal/mole and 8.3 kcal/mole, respectively. The heats of vaporization for these two amines are

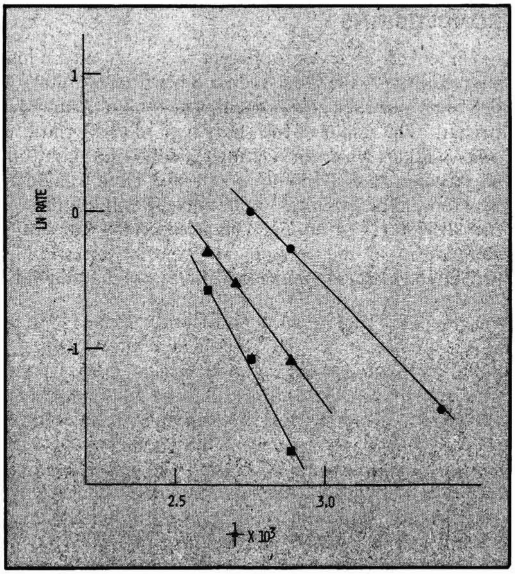


Figure 7—Arrhenius plots for NH_3, TEA, and DMEA. Legend: ●NH_3; ▲ TEA; ■ DMEA

Table 4—Activation Energies

Amine	Observed E_A	Literature ΔH_v
NH_3	4.5 kcal/mole	4.7 kcal/mole
TEA	6.0 kcal/mole	5.8 kcal/mole
DMEA	8.3 kcal/mole	9.4 kcal/mole

5.8 kcal/mole and 9.4 kcal/mole, respectively. These data are summarized in *Table* 4. The very low rate of loss and the scatter of data points make it impossible to do this calculation with any reliability for DEA and TEOLA. The effect of temperature on these amines, as well as on the other samples, can be observed in *Table* 1.

Variations in Polymer Acid Content

Samples of resins #2 and #3 neutralized with TEA were prepared and analyzed in the manner described for resin #1. Although these polymers differ considerably in concentration of acid functionality, both systems show virtually the same curve shape and loss rate behavior with respect to film thickness as the resin #1 system. Wicks and Chin[6] also found little variation of amine loss behavior with changes in resin except where the acid value was changed to essentially zero. For the system containing a zero-acid functionality polymer, an enhanced loss rate of amine was observed. This does not necessarily indicate that salt formation is responsible for the lower rate of loss in the other systems. Since the polymer containing no acid functionality could not be dissolved in water, ethanol was chosen as the solvent. Ethanol is a relatively volatile solvent and, indeed, simple mixtures of amine and solvents will show faster loss from more volatile mixtures than from less volatile ones.

CONCLUSIONS

The effects of several different factors on amine loss characteristics have been studied. These include intrinsic volatility, polymer acid content, temperature, and base strength. The results of this study tend to verify those of other workers when considering the solid film stage, but go beyond this in pointing out the close similarity between amine loss and simple solvent evaporation for the early stages of drying. This is especially true in the comparison of the enthalpy of vaporization and the activation energy for amine loss and by casting the amine loss data into the format outlined by Hansen. These results suggest that, during the early stages of drying, the attainment of equilibrium between salt and acid and base is fast when compared with the rate of transport of amine across the vapor/liquid boundary. The effects of base strength in this region appear to be minimal. Therefore, since the amine loss rate in the diffusion controlled region or second drying stage is minimal, its is important to insure that the amine present is reduced to very near the desired final level before the diffusion controlled level is reached. This necessitates formulating based on amine volatility rather than base strength or other characteristics except possibly where extremely large differences in pKa are being considered.

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Trace Residual Monomer Analysis By Capillary Gas Chromatography

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A method has been developed for the determination of trace amounts of residual monomers by capillary gas chromatography for application in the coatings industry. Most previously employed methods for these analyses made use of conventional packed columns. Recent discoveries of adverse health effects associated with some monomers have enhanced the importance of trace analysis. The most feasible method for performing these analyses when dealing with complex solvent systems is to take advantage of high resolution capillary columns. A method is described which uses a splitless injection technique, with a solvent effect being employed for the analysis. Varying solvent systems are considered for possible interferences in conjunction with the monomers of interest. A unique feature of the method is the use of a 50 meter, stainless steel capillary column. A performance evaluation of the stainless steel column is discussed in detail.

INTRODUCTION

The method described in this paper involves the detection of trace amounts of low boiling monomers in complex solvent systems. The analysis is accomplished through the use of capillary gas chromatography with emphasis placed on the use of a stainless steel capillary column.

One of the problems many chromatographers are faced with is the task of redeveloping currently existing methods to achieve lower detection limits. While doing this it is also advantageous to reduce analysis time by reducing the number of columns required for a given analysis. Along with these two factors is the necessity of developing a method which can be implemented into a plant situation where the conditions are far from ideal. All of the criteria just mentioned were taken into consideration in the development of this method.

The determination of trace amounts of residual monomers in polymers has always been of importance to the analytical chemist involved in the coatings industry. In the past a major necessity of this determination was for quality control. Recent discoveries of adverse health effects associated with some monomers have enhanced the importance of trace analysis. The following method is an adaptation of previously used packed column methods. It is shown that the transition from packed to capillary methods can be accomplished with a nominal amount of change.

SCOPE AND PACKED COLUMN METHODS

The monomers for which the method has been developed are listed in *Table* 1, along with their physical properties.

It seems necessary to first describe briefly the packed column methods previously used for the analysis of low boiling monomers. When analyzing for the six monomers mentioned, three different methods, each employing a different column, must be used. These three methods are described in *Table* 2. The first method is for the analysis of low boiling monomers in samples which do not contain aromatic naphthas or interfering solvents. Besides aromatic naphthas, there are two major solvent interferences on this column: methyl and amyl ketone (MAK) masks butyl acrylate (BA), and 2-ethoxyethyl acetate masks styrene.

The second method, using packed columns, is for samples which contain aromatic naphthas and/or other interfering solvents. An exception to this method is that it cannot be used for samples which contain ethyl acrylate (EA) or methyl methacrylate (MMA), due to interferences. In a case such as this, a third method must be used to determine these two monomers. This is also described in *Table* 2.

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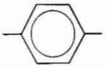
Table 1—Physical Properties

Name	Formula	Formula Weight	Boiling Point Range, °C
Acrylic Esters			
Ethyl acrylate	$CH_2 = CHCO_2C_2H_5$	100.13	98.8-99.8
Butyl acrylate	$CH_2 = CHCO_2C_4H_9$	128.17	145.7-148
Methacrylate Esters			
Methyl methacrylate	$CH_2 = C(CH_3)CO_2CH_3$	100.13	100.3-100.8
Butyl methacrylate	$CH_2 = C(CH_3)CO_2(CH_2)_3CH_3$	142.20	160
Isobutyl methacrylate	$CH_2 = C(CH_3)CO_2CH_2CH(CH_3)_2$	142.20	155
Aromatic			
Styrene	 $CH_2 = CH_2$	104.15	145-146

These analyses were studied, and it was decided that by choosing the UCON liquid phase and achieving better resolution of components, it would be possible to achieve the analysis of samples both with and without the interfering solvents mentioned above on one column.

EXPERIMENTAL

After some consideration, a 50 meter × 0.25 mm ID stainless steel, wall-coated column (Analabs, Inc., North Haven, CT) was chosen for the analysis. The liquid phase used is the same as for the packed column, that is UCON LB550X (chemical type, polyalkylene glycol).

Table 3 lists the chromatographic conditions decided upon for the capillary method. The instrumentation used is a Hewlett Packard Model 5840 gas chromatograph equipped with capillary option 18835, microprocessor, and automatic sampler. The column is installed using Graphlok® ferrels from SGE which are suitable for producing a tight seal.

A splitless injection technique[1,2] was used, employing a solvent effect[3] for the analysis. To achieve a solvent effect, the starting temperature for the analysis is 40°C. This temperature is held for four minutes and then increased slowly at a rate of 2°C/min to 150°C. A septum purge is activated 30 sec after injection.[4,5]

Using the packed column method, samples are prepared of approximately 8% resin sample in tetrahydrofuran (THF). When using the capillary method, only 1 to 1.5% sample in THF is needed. When concen-

Table 2—Method for the Analysis of Low Boiling Monomers In Samples Which Do Not Contain Aromatic Naphthas or Other Interfering Solvents

Column:	15′, ⅛″, SS, 10% Ucon® LB-550-X, 100-120 mesh Chromosorb® W(NAW)	
Column Temp.:	Temperature Programmed	
	Temp. 1	110°C
	Time 1	4 min
	Rate	4°C/min
	Temp. 2	130°C
Method:	Internal Standard	
Interfering solvents:	methyl amyl ketone masks butyl acrylate, cellosolve acetate masks styrene	

Methods for the Analysis of Low Boiling Monomers In Samples Which Contain Aromatic Naphthas and/or Other Interfering Solvents

(A) Method for the analysis of BA, Styrene, IBMA, BMA

Column:	9′, ⅛″, SS, 10% (TCEP) 1,2,3-Tris(2-cyanoethoxy)propane 100/120 mesh Chromosorb PAW
Column temp.:	130°C, Isothermal
Method:	Internal Standard

(B) Method for the analysis of ethyl acrylate and methyl methacrylate in samples which contain an aromatic naphtha.

Column:	10′, ⅛″, SS Porapak®Q, 80-100 mesh
Column temp.:	250°C
Method:	External Standard

Table 3—Chromatographic Conditions

Carrier Gas	Helium	
Pressure	22 psi	
Carrier gas flow	26 cm/sec	
Make-up gas	Helium	
Make-up gas flow	25 ml/min	
Septum purge flow	0.6 ml/min	
Inlet purge flow	60 ml/min	
Detector	Flame Ionization	
Air	240 ml/min	
Hydrogen fuel	30 ml/min	
Injection port temp.	250°C	
Detector temp.	190°C	
Column temp.	Temp. Programmed	
	Temp. 1	40°C
	Time 1	4 min
	Rate	2°C/min
	Temp. 2	150°C
	Time 2	30 min
Sample size injection	0.5 microliter	
Attenuation	X16	
Septum purge		

The septum purge is deactivated before each run and reactivated 30 sec after each injection.

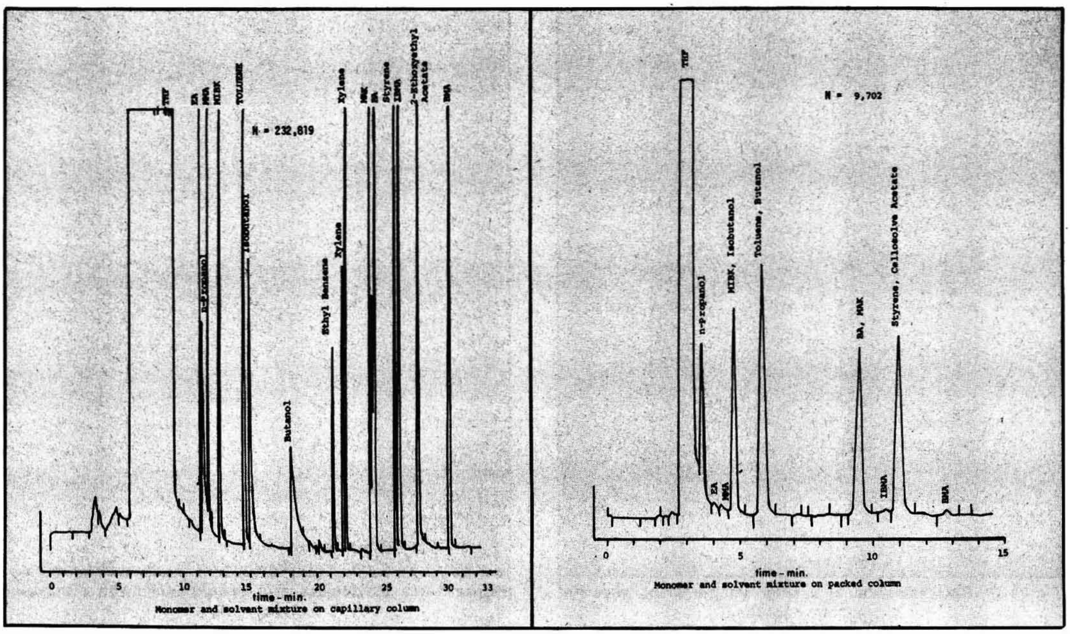


Figure 1—Comparison chromatograms of solvent and monomer mixtures on capillary and packed columns

trations below 50 ppm are sought, the percent of sample may have to be doubled. For the capillary method, an internal standard method is used in which the standard is one of the monomers for which the method has been developed, but which is not in the sample in question. The sample should be run prior to the addition of the internal standard to insure that there are no interferences. A standard is made up which contains all the monomers in question and the internal standard in concentrations which are compatible with levels which are present in the sample.

Calculations

The reporting integrator is programmed to perform the Internal Standard calculation as illustrated:

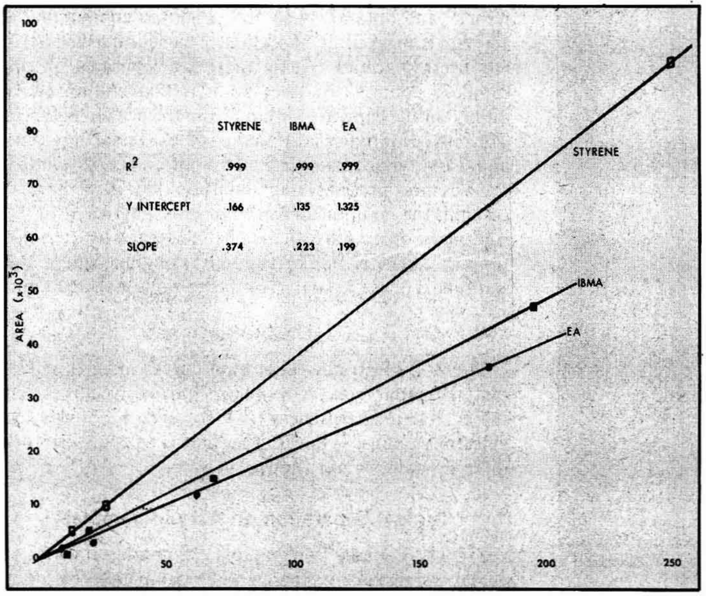


Figure 2—Calibration curves

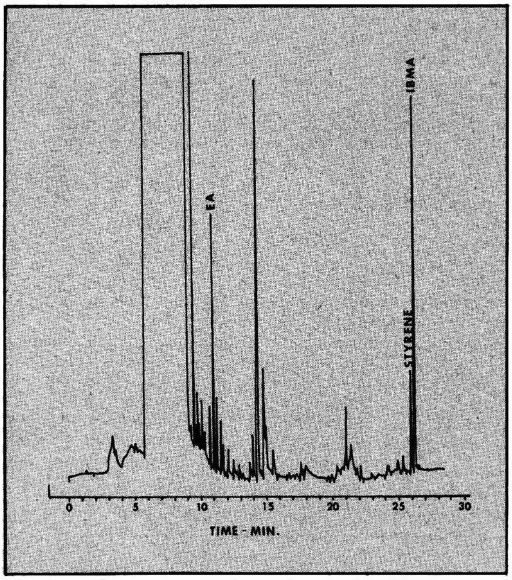


Figure 3—Chromatogram of a resin sample which does not contain a naphtha

$$\text{Amount y} = \frac{(\text{Area y})\ (\text{Response y})}{(\text{Area IS})\ (\text{Response IS})}\ (\text{IS Amount})\ (\text{XF Factor})$$

where:

y = Unknown
IS = Internal Standard
Response = Amount/Area in Standard

$$\text{XF Factor} = \frac{100}{\text{Sample Weight}}$$

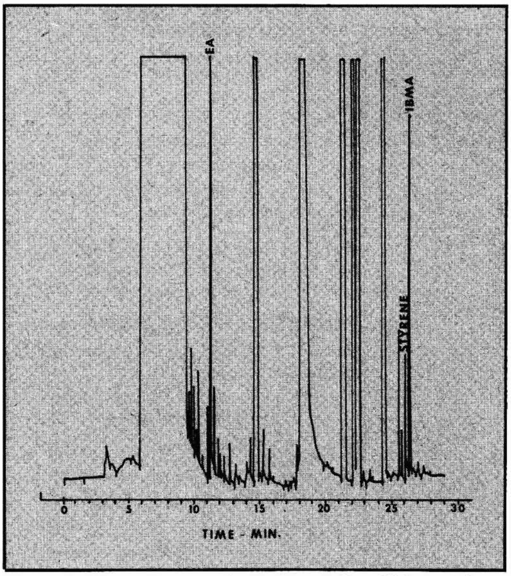


Figure 4—Chromatogram of a resin sample which contains a naphtha

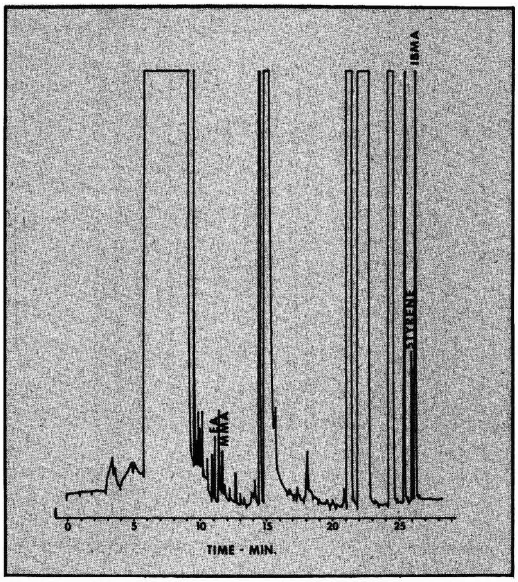


Figure 5—Chromatogram of a resin sample with a complex solvent system

RESULTS

Capillary Column Separation

Figure 1 shows comparison chromatograms on capillary and packed columns of a mixture of the monomers in question and solvents commonly encountered. The components in both mixtures are in the 150-200 ppm range. It can be seen that much greater sensitivity can be achieved using the capillary column. It can also be seen that methyl isobutyl ketone (MIBK) and isobutanol coelute are separated on the capillary column, as are toluene and butanol. Also on the packed column, butyl acrylate is masked by MAK and styrene is masked by 2-ethoxyethyl acetate. Again these are separated on the capillary column. Another point is that elution order differs on the capillary column from the packed column. The most pronounced difference is the reversal of styrene and isobutyl methacrylate. This difference in elution order is most probably due to the fact that, in the packed column, the components will not only interact with the stationary phase but will also be influenced by the solid support. This is unlike capillary columns where the stationary phase is coated onto the wall of the column.

Calibration

Figure 2 illustrates calibration curves of three representative monomers. As can be seen from the curves and the correlation coefficient, (r^2), for each monomer the detector response is linear. The same is true for the other three monomers, not shown.

Typical Separation on Acrylic Samples

Figures 3, 4 and 5 are sample chromatograms run of actual acrylic resin samples. The sample in *Figure* 3

Table 4—Retention Time Reproducibility (min)

Component	EA	MMA	BA	Styrene	IBMA	BMA
Run						
1	9.97	10.63	24.39	25.99	26.22	29.89
2	9.97	10.62	24.36	25.95	26.24	29.85
3	9.97	10.61	24.37	25.97	26.20	29.86
4	9.98	10.61	24.33	25.93	26.19	29.88
5	9.99	10.61	24.42	26.00	26.20	29.85
6	9.98	10.62	24.43	26.00	26.22	29.89
$\overline{X}$	9.98	10.62	24.38	25.97	26.21	29.87
σ	±0.01	±0.01	±0.04	±0.03	±0.02	±0.02
σ rel. %	0.10	0.09	0.16	0.12	0.08	0.07

contains the monomer EA, MMA, and styrene. IBMA is used as an internal standard, as it is in all examples. All monomer concentrations are less than 50 ppm. The sample contains no aromatic naphthas, but as can be seen, is complicated by trace contaminants either introduced by solvents or as by-products of the reaction.

Figure 4 represents a sample which contains approximately 100 ppm EA and less than 50 ppm styrene. This sample contains an aromatic naphtha, and other solvents. The complexity of a sample such as this and that illustrated in *Figure* 5 supports the use of a capillary method.

Figure 5 is a chromatogram of a sample which does not contain an aromatic naphtha but has a very complex solvent system.

Reproducibility

A consideration of data reproducibility and column performance is worth noting. A study of retention time reproducibility was considered and is summarized in *Table* 4. Six different runs are listed with retention times for each monomer. These runs were made after the column had been used approximately two months. The automatic sampler was used for the injections. Relative percent standard deviation of retention times ranged from 0.07 to 0.16%. *Table* 4 shows the excellent reproducibility of retention times which can be obtained. This is very important when using capillary columns since most chromatograms are very complex. Also, it is especially important when coupling the gas chromatograph with an autosampler and running it unattended.

Table 5—Area % Reproducibility

Component	EA	MMA	BA	Styrene	IBMA	BMA
Run						
1	11.37	12.12	15.80	29.36	16.40	14.99
2	11.33	12.12	15.81	29.34	16.35	14.92
3	11.41	12.09	15.85	29.37	16.38	14.96
4	11.40	12.07	15.84	29.38	16.34	14.94
5	11.33	12.13	15.82	29.37	16.37	14.92
6	11.30	12.10	15.84	29.34	16.38	14.92
$\overline{X}$	11.36	12.11	15.83	29.36	16.37	14.94
σ	±0.04	±0.02	±0.02	±0.02	±0.02	±0.03
σ rel. %	0.35	0.17	0.13	0.07	0.12	0.20

Table 6—Weight % Repeatibility

	Day 1		Day 2			
	Run 1	Run 2	Run 1	Run 2	Average	Standard Deviation
Sample 1						
EA	0.15	0.15	0.13	0.14	0.14	±0.01
Styrene .	0.10	0.10	0.09	0.09	0.095	±0.006
Sample 2						
EA	0.21	0.21	0.19	0.19	0.20	±0.012
Styrene .	0.10	0.12	0.10	0.11	0.108	±0.01

A study of area percent reproducibility also generated consistent results as seen in *Table* 5. Relative percent standard deviations ranged from 0.07 to 0.35%.

The above was complemented by a study of the reproducibility of weight percentage of solvents in the samples, and the data is presented in *Table* 6. These samples were also run on packed columns and it was found that the packed column method reaches the lowest level of detection at about the 100 ppm.

Column Stability

The chromatogram in *Figure* 6 was run after the column had been in use approximately four months. It was suggested by Grob[6] that 2,3-butanediol be used to test for absorption of hydroxyl groups for glycol type stationary phases instead of 1-octanol, which is commonly used. Decreased peak size and tailing indicate absorption. The shape of 2,6-dimethylaniline as com-

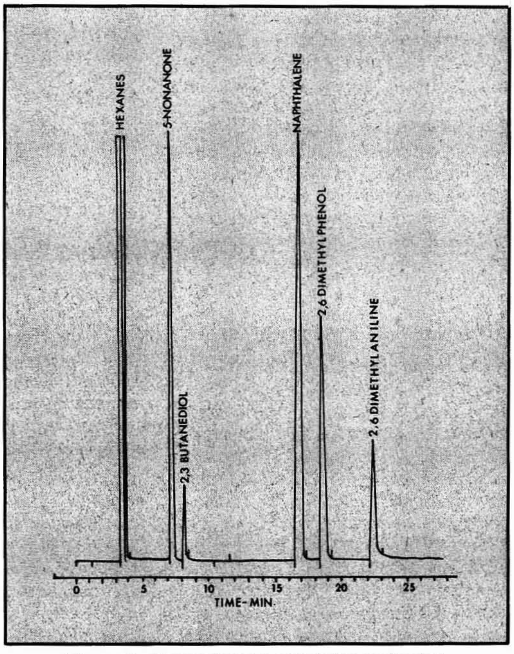


Figure 6—Chromatogram of column evaluation

Table 7—Test of Column Efficiency Over a Period of Time[a]

	Time (months)		
	1	3	6
N	232,819	170,221	127,575
HETP	0.21	0.29	0.39
k′	6.7	6.7	6.9

(a) EA peak used for these calculations.

pared to 2,6-dimethylphenol show the column is becoming slightly acidic. The ketone, 5-nonanone, seems to indicate no interaction with Lewis acid sites and the naphthalene peak shows no interaction with metallic adsorption sites.

In *Table* 7, the results of a test of column efficiency calculations[7] over a period of time are listed. This is a very critical test for this particular method. This is because a splitless injection is being used and a large amount of relatively polar solvent (tetrahydrofuran) is being put through the wall coated column. It was found that the column could be used regularly for a period of about five months before resolution and results began to suffer. After five months resolution began to decrease drastically and retention times began to increase as the liquid phase was washed from the column.

SUMMARY

A method has been developed to analyze for trace residual amounts of the low boiling monomers: ethyl acrylate, methyl methacrylate, butyl acrylate, styrene, isobutyl methacrylate, and butyl methacrylate using capillary gas chromatography. It is shown that a splitless injection technique may be employed (using a fairly polar solvent) and one may still achieve up to five months utility from the stainless steel capillary column. Monomer levels of 10–150 ppm may be analyzed in samples where the only sample preparation necessary is dilution with the solvent (tetrahydrofuran) and addition of an internal standard. The method may be adapted to lower concentrations by reducing the sample dilution or to higher levels by using a split injection (discussions of which are not covered in this paper). Included in this method is a discussion of the complexity of solvent systems which may be encountered and chromatograms showing the resolution which can be obtained.

In conclusion, the ease in handling of this column is obvious and studies of this method show it to be useful for the analysis of low boiling monomers in complex solvent systems.

References

(1) Freeman, R. R. and Wisniewski, J. V., "Splitless Injection On Open Tubular Columns," Hewlett-Packard Application Note: 4-76.
(2) Yang, F. J., Brown, A. C. III, and Cram, S. P., *J. Chrom.*, 158 (1978) 91–109.
(3) Grob, K. and Grob, K. Jr., *J. High Resolution Chrom & Chrom. Comm.*, July, 1978.
(4) Rooney, T. A., "Optimizing Analysis Using Splitless Injection on Capillary Columns," Hewlett-Packard Application Note: 228-5, 1977.
(5) Freeman, R. R., "Quantitative Analysis Using a Purged Splitless Injection Technique," Hewlett-Packard Application Note: 7-76.
(6) Grob, K. Jr., Grob, G. and Grob K., *J. Chrom.*, 156 1 (1978).
(7) Jennings, W., "Gas Chromatography with Glass Capillary Columns," p 4-7, Academic Press, 1978.

United Kingdom Orders of FSCT Educational Literature

Mr. Ray Tennant, of the Birmingham Society, will act as a source of Federation educational literature (Color-matching Aptitude Test Set; Infrared Spectroscopy; Paint/Coatings Dictionary) for United Kingdom customers. Anyone interested in receiving these items is urged to contact Mr. Tennant. His address is: Carrs Paints Limited, Westminster Works, Alvechurch Road, Birmingham B31 3PG, England.

An Overview of Quality Assurance

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Quality Assurance is extradepartmental in scope and responsibility. Quality begins with the setting of standards for the desired levels of excellence. People involvement and good communication are vital to the success of a good program. Assurance of quality is dependent upon optimum formulations with raw materials of known characteristics and practical specifications, close process control, documentation of procedures, monitoring of compliance to standards, and good basic practices. An established program of education and training for involved personnel is vital to a "preventive medicine" approach to "doing it right the first time!"

Introduction

"What is jazz?" someone once asked the late, great trumpet player, Louis Armstrong. "If you gotta ask, you'll never know," he replied. "Ol' Satchmo" wasn't putting down the questioner or trying to evade the question. Either you know what it is or you don't!

The same sort of thing might be said of quality, for there is just no single definition that will encompass everyone's understanding of what quality is. Many people do not appreciate the meaning and wide implications of true Quality Assurance as opposed to the narrow area of quality control. Control implies a standard against which to compare the results of an experiment or defined procedure. It is a restraining influence, or a check.

The verb "assure" means to make sure or secure; to establish firmly; to make something certain; to give confidence to, or to insure, as against loss. Assurance is, of course, the act of assuring or the state of being assured. It is a positive term, intended to provide confidence and encouragement; firmness of mind; certainty.

An ideal Quality Assurance program will recommend, develop, control, monitor and audit standard policies, procedures and responsibilities for achievement of the desired degree of excellence and will limit losses of material, time and money, with a resultant contribution to profit. Its organization must be extradepartmental in scope and authority, and interdepartmental in desire and cooperation for attainment of its goals.

A Desire for Excellence

The dictionary defines quality as "excellence; the degree of excellence; relative goodness, or grade." Quality begins with the setting of standards for the desired level of excellence. The establishment of quality standards is a complicated task. People differ widely in their ideas of quality. There must be a full understanding that quality is relative, not an absolute. Because quality is relative, especially in an industry such as ours, standards must be set in respect to every product, every order, and every customer's needs and desires—at any given time. If this basic premise is understood, it becomes obvious that quality, high or low, is not just something that results from manufacturing processes or control.

At the very base of quality is the desire for excellence, and this desire needs to be given life by responsible people. When a person finds out what is fitting and needed, and uses his skills to supply it, he is on the right road to excellence. Seeing the need and having the desire for excellence, the next thing to do is to act to achieve it. What is this excellence, and how high is the quality that is desired? Standards need to be defined and procedures must be documented, and they must be communicated to the people responsible for the actions needed to assure compliance.

People are a Vital Key

People are vital to the success of any good program. A total quality program must stress personal involvement of all employees. As the workers become involved in problem solving, the quality professional must become more of a teacher and facilitator. The notion that only the Quality Assurance Manager and his people can deal with quality issues is all wrong. The knowledge and skills required to improve and maintain quality are shared by large segments of people. The concept of quality must be in the forefront of each person's daily activity.

Presented at the Philadelphia Society for Coatings Technology Quality Assurance Seminar, Philadelphia, PA, May 15, 1980.

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However, involving employees in problem solving will not automatically improve quality. A total well-structured system must evolve which begins with an analysis of the types of changes needed within the current system to meet the needs.

Some years ago, in order to improve their national image and product quality, the Japanese turned to the quality control circle concept in which a major portion of the workers assumed responsibility for quality in their own work areas. We are well aware of the progress made in quality improvement of Japanese products in the world market and its impact on the U.S. commercial markets.

A more limited approach is that of the use of quality coordinating committees with representation from each department—Marketing, Technical and Manufacturing—that meet regularly to discuss major problems and devise approaches to their solutions. Members can rotate so that there is a wider exposure to the problems and to expand the forum for new ideas. There is a synergistic effect from this interdepartmental approach, which is lost when problem discussions are confined to the department having the problem.

Recent articles explain the "quality of work life" concept which has been identified as a process to unlock the creative potential of people by involving them in decisions affecting their work lives. This is a concept aimed at improvement of productivity by providing an opportunity to maximize human resources. Such concepts need to be studied and considered for their potential impact on product quality.

Table 1—Manufacturing Operations Guide

Standard Terms

Rate of Addition

ADD—indicates these materials should be added at a normal rate (about 150 lb/min for dry materials; 20 gal/min for liquids).

ADD SLOWLY—indicates a rate of addition of dry materials at a rate of about 50 lb/min. If more than one item is to be added at this slower rate, the statement must precede each item.

SIFT IN—indicates a rate of addition of dry materials at a rate of about 25 lb/min. The term must precede each item to be added at this rate.

Premixes

PREMIX—indicates that the next items listed will be mixed in a separate container prior to adding to the main batch.

ADD PREMIX—use of this term denotes not only that the premix is to be added, but, also, that this ends the premix.

ADD PREMIX AT A RATE OF ___ GALS/MINUTE—indicates the premix addition rate.

These standard terms must start the sentence of any instruction. They will be used to determine process costs applied to each specific manufacturing phase.

Other modifiers can be used with these terms; e.g., ADD to tank; ADD SLOWLY to avoid lumping; SIFT IN to vortex; PREMIX until clear; ADD PREMIX under agitation

Quality Comes at a Price

It also must be understood that quality comes at a price. An effective Quality Assurance program requires adequate staffing and funding, and the visible support of top management.

Quality Starts with the Consumer

Assuming the presence of these basics, where does it all start? It starts with the needs and requirements of the ultimate consumer, which in turn must be defined by the producer of the ultimate product. The standards of quality of an automotive coating are determined by the levels of performance accepted by the buyers and users of automobiles. The coatings standards must be set by a process of evaluation and testing in the marketplace and a cooperative effort between the automobile and coatings producers. Such efforts must be carefully coordinated by the Marketing Departments and accurately relayed to the Technical Departments responsible for product design and the setting of control test standards. The building in of quality must begin long before the order gets into the plant.

Quality standards for house paints and other coatings sold directly to the ultimate consumer must be determined through carefully controlled laboratory and field testing of formulations that can be marketed competitively. The final design will result from input of Market Research, Sales, Technical, Customer Service and Manufacturing departments. Ultimately, it will be a formulation that (1) will meet a need in the marketplace, (2) uses available raw materials of uniform quality, (3) can be manufactured on available equipment on a practical time schedule, and (4) can be sold at a competitive price that results in a reasonable profit.

Communication—Another Vital Key

After performance quality is defined and test standards set, they must be communicated properly to those responsible for product development. Communication is another vital key to successfully meeting the quality standards and needs. Means of communication include not only the spoken word, through telephone and written correspondence, but also proper, understandable and uniformly applied terminology (*Table* 1); precise documentation of formula information, and testing procedures in standard format.

Standard terminology should be used on both formulas and batch slips, with both following similar formats. There can be some critical and embarrassing errors made because of misunderstandings in communication.

Formulation Quality Is Essential

After the standards are set, the formulation process begins. Raw materials must be selected, and this introduces a set of specific problems and questions. Some materials will be those in current use, the uniformity and performance of which is fairly well-known. Others will be new in the marketplace or new to the formulators involved.

Some of the questions to be answered at this point include the following:

(1) Can the formulator ascertain the sources of supply and specific lot numbers of lab materials in current stock? If so, how can it be determined that they meet the required standards?

If sources or lot numbers of raw materials used in formulation are not known, or if they do not meet standard specifications, it is virtually impossible to determine the cause of variances from laboratory to plant during scale-up.

(2) Are the test methods available to test these raw materials? If so, are they significant tests? If not, what tests will be significant?

Too often typical analyses or physical constants supplied by the manufacturer are accepted as purchase specifications with little thought given to their significance in relation to finished product performance. Indeed, it is often difficult to establish such significant tests, making it necessary, or at least expedient, to purchase on certification by the supplier in order to provide future materials "equal to" the initial approved lot.

(3) On new raw materials, what should the specifications be and how shall they be tested for uniformity from lot-to-lot? What tolerance does the formula have for variances from the approved standard?

Testing of a number of consecutive production samples provided by the supplier, along with his quality control test data, in a series of lab batches with all other raw materials held constant, provides an initial indication of probable material uniformity.

(4) If the source and lot numbers are available and lab tests show lot-to-lot uniformity, how can it be assured that materials used in production meet the standards? What quality control, if any, is needed on in-coming shipments to the plants?

Standard quality control tests, especially for new or "critical" materials, must be established prior to plant manufacture. These tests must be practical ones that can be run by the quality control personnel with end points as objective as possible.

(5) What is the precision of the test methods used? Are those who will run the tests properly trained? Is test equipment functioning properly? What procedures must be used for calibration? How often should the instruments be checked?

These last questions introduce an entirely separate phase of the Quality Assurance program. It requires review of ASTM methods and precisions, possible round robins for determination of repeatability and reproducibility. It also entails an investigation into proper equipment operations and manufacturer's instructions. Training on use of test instruments is also necessary.

Quality of manufactured products cannot be assured if the formulation quality itself is not assured first.

Assume Nothing

In my early days of varnish formulation, I had a boss who had the exasperating knack of thoroughly humbling me when I proudly submitted a new varnish-coated test panel for him to scratch. The film appeared to me to be practically mar-proof and tightly bound to the substrate, but when subjected to his thumbnail, a far tougher "instrument" than any other normally used, it flaked off quite readily in all directions! This ego-shattering moment was followed by his comment, "Never be too proud of your work to thoroughly abuse it. If you don't, someone else will!"

This lesson was so thoroughly learned that I credit it with a good part of the reason for my ending up in Quality Assurance. Over the subsequent years, under this man's tutelage, I acquired a technical attitude of inquiry as to the less than obvious areas of potential weakness or failure that could result in less than the desired level of excellence. My most basic standard is summed up in two words—*assume nothing!*

"It's the little things that count!"

Leonardo da Vinci wrote in his notebook that when a drop of water falls into the sea the whole surface of the sea is raised imperceptibly. A "drop in the bucket" is not unimportant. This type of philosophy should, of course, be carried through into the other areas that are basic to assurance of uniformity of quality.

Process Development and Control—Important to Quality

Another very vital quality matter is that of "process development." This phase of product development is too often overlooked or minimized. An early interplay with manufacturing personnel in prototype development will greatly reduce the potential for processing problems or delays. Knowledge of processing equipment or plant configuration will often have a bearing on optimizing quality and final product costs. Especially in larger organizations, scale-up from laboratory batches to full-scale production, in a formalized step-wise procedure, by technically trained personnel knowledgeable in equipment operation, will minimize start-up problems and potential losses and maximize the chances for meeting quality criteria.

Development of a Manufacturing Operations Guide containing standards for terminology, equipment operating procedures, and other corollary information for use by both Technical and Manufacturing personnel is an excellent communication and training aid. *Table* 2 gives an example of the contents of such a guide.

There is an ever increasing need for instrumentation for in-process control, not only in the production of resins and latices, but also in paint processing. Use of ammeters, tachometers, and temperature recorders on dispersion equipment, as well as accurate measuring devices for flow control and metering of liquids, can contribute enormously to assurance of reproducibility of quality from batch to batch. A scheduled check on accuracy and cleanliness of scales and other measuring devices should be a way of life.

Table 2—Manufacturing Operations Guide

Table of Contents

Manufacturing Terminology
Rate of Addition
Premixes
Mixing and Dispersion
Thindown and Tank Agitation
Cleaning
Heating and Cooling
Other Dispersion Instructions
Adjusting
Shading
Testing
Filling
Miscellaneous

High Speed Dispersion
Standard Format
  Formula Card Examples
  Production Slip Examples
Standard Operating Procedures
Cowles Speed Charts

Sandmilling
Standard Format
  Formula Card Examples
  Production Slip Examples
Standard Operating Procedures

Steel Balls and Pebble Mills
Standard Format
  Formula Card Examples
  Production Slip Examples
Mill Cleaning Format and Procedure
  For Formulator
  For Scheduler
Standard Operating Procedures

Tanks
Standard Operating Procedures
  Mixing
  Agitation Before Filling

Miscellaneous Equipment
Standard Operating Procedures
  Resin Meters
  Solvent Meters
  Dust Collectors
  Bag Baler

Finished Product Quality Control

Quality control testing of the finished product is, of course, a very important phase of Quality Assurance. However, it is only the inspection part of the process, not the most important part of Quality Assurance, as some people tend to believe. The critical part of quality control is that of assuring that the testing done determines compliance with the standard specifications. The normal quality control tests run on the finished product will provide information only on physical constants or appearance relative to quality control standards for a decision on filling out the batch. At this point it must be assumed that these tests are sufficient to determine that the product is

Table 3—Quality Assurance Manual

Table of Contents

- I. Scope, Purpose and Manual Organization
- II. The Quality Assurance Program
  - A. Philosophy of Quality Assurance
  - B. Formula and Process Development
  - C. Education and Training
  - D. Loss Control, General
- III. Quality Control
  - A. Testing
    - 1. In-process testing
    - 2. Finished goods testing
- IV. Color Control
  - A. Formula review
  - B. In-process
  - C. Pigment concentrates
  - D. Field tinting
- V. Raw Materials and Containers
  - A. Testing programs
  - B. Receiving, storage and handling
- VI. Basic Plant Practices
  - A. Housekeeping
  - B. Bacteria control
  - C. Weighing, measuring and metering
  - D. Order processing
  - E. Production slips
  - F. Sampling procedures
- VII. Finished Goods
  - A. Warehousing and shipping
    - 1. Packaging and coding
    - 2. Over-age inventory
- VIII. Product Control: Trade-Maintenance
  - A. General program
    - 1. Specific products
- IX. Product Control: Industrial Coatings
  - A. General program
    - 1. Specific products
- X. Process Control: Paint
  - A. Dispersion and mixing
  - B. Thinning
  - C. Adjusting and color matching
  - D. Filtering and filling
- XI. Process Control: Vehicles
  - A. Polymer processing
  - B. Thinning
  - C. Adjusting
  - D. Filtering and filling
- XII. Supplemental Material

satisfactory for its intended use and that the raw materials used and the processing done were according to approved standards.

Therefore, it can be seen that all that went on before this phase is more important than the final product testing. It is also important that the formulator has predetermined that the quality control tests run will indicate a satisfactory product.

Training and Education

Establishment of a basically sound program, accurate formulas, significant test methods, good techniques, and standard equipment operating procedures will not be sufficient to provide the desired levels of excellence, or needed quality, unless the people responsible for each phase are properly trained and educated in the use of them and the importance of following instructions exactly. Every person is interested in doing a good job, but he needs to know not only how, but why it is necessary to follow prescribed instructions.

A formalized program for the training and orientation of new personnel, and use as a refresher for existing workers, is worth many times the value of the manhours spent for its development and presentation. Sessions on specific subjects, augmented by audio-visual tools, provide greater job interest and motivation. They can serve as a forum in which the workers can get answers to questions and offer suggestions for operational improvements. These sessions can provide good opportunities to administer a dose of the "preventive medicine" approach to Quality Assurance. The best and least expensive way to do a job is to "do it right the first time!"

Basic Practices Need Emphasis

There are, of course, many basic practices that need attention and which will contribute greatly to the "preventive medicine" approach. These include proper coding, receiving, handling and storage of raw materials; cleaning of equipment; scheduling of batches in proper size equipment; color control with practical standards; container selection and inspection; and close attention to final filtration—that important last phase in the plant operation prior to shipping the product to the customer. Failure to monitor this final operation, resulting in the customer receiving a "dirty" product, will undo all the fine effort put into the process up to that point—an unforgiveable act! Each of these basic practices alone could be the subject of an article.

Quality Assurance Manual

Documentation of all policies and procedures relative to a Quality Assurance program is essential. A well organized manual containing details of all phases of the program, showing approvals by all department heads involved, is the "bible" and authoritative source for all Quality Assurance action.

This manual should be a living instrument under constant scrutiny for needed revisions and updating when necessary. It should provide guidelines and checklists that are significant and useful by everyone involved, from the supervisory staff to the worker on the plant floor. *Table* 3 shows an example of the content of such a manual.

Continuous Monitoring Needed

All types of monitoring, including quality control testing, equipment inspection and maintenance, and a regular audit of all phases of the Quality Assurance program, are a continuing need vital to ultimate success. These actions tell us where we are at.

> *"Would you tell me, please, which way I ought to go from here?" asked Alice. "That depends a good deal on where you want to get to," said the cat. "I don't much care," said Alice. "Then it doesn't matter which way you go," said the cat.*

If this conversation from Lewis Carroll's "Alice in Wonderland" describes your approach to Quality Assurance, then you need to chart a clearer course of action.

Standardize, inspect, control, monitor, audit, and "assume nothing!"

June 1980 Subcommittee Reports of ASTM Committee D-1

DIVISION 30 PAINT MATERIALS

SUBCOMMITTEE D01.31 PIGMENT SPECIFICATIONS

C.W. Fuller, Chairman

Pigment and extender specifications that were balloted in the Spring of 1980 were reviewed for resolution of negatives and comments. Specifications D80, Leaded Zinc Oxide; D82, Basic Sulfate White Lead; and D405, Blue Lead Basic Sulfate were approved unanimously in main committee ballot for withdrawal. Specification D210 for Bone Black received one negative regarding paste in oil as being obsolete. Bone Black is still being used in artists' colors and the negative was voted non-persuasive. The balance of the negative was considered persuasive and "Number of Tests" will be changed to "Sampling," D1208, part 9 and 10, will be included to determine the pigment content of oil pastes and D215, part 10, will be included to determine the nonvolatile vehicle portion. Other editorial comments will be incorporated into the revised specification. A comment to change "purchaser and seller" to "producer and user" was considered non-persuasive on the basis that not all sellers of pigments are the producers. This also applies to Specifications D210, D763, D769, and D1199.

Specification D763 for Raw and Burnt Umber received a negative containing four items. The first item requested the iron content ($\%Fe_2O_3$) be increased based on NPCA values. The negative was voted non-persuasive since umbers are produced from variable raw materials and $\%Fe_2O_3$ does not necessarily relate to color value. On the other items, footnote "a" concerning manganese content will be deleted, 5.1.2 should be changed to D185 and 5.1.3 should be D1208. Also corrections should be made in 5.1.4 to delete balance of paragraph.

Specification D768 for Yellow Iron Oxide received a seven part negative. "Disintegrated" will be changed to "powdered." The portion concerning a reduction in $\%Fe_2O_3$ was voted non-persuasive because the reduction was approved previously and ASTM should not be constrained by NPCA values since all synthetic yellows are not listed by NPCA. On other items, ASTM will be deleted in section 5, "In Pigments" will be added in title of 5.3 and 5.4, sections 5.7 and 5.8 will be added, D562 added to Applicable Documents, and D1280 changed to D1208.

Specification D769 for Black Synthetic Iron Oxide received a four part negative. It was agreed to add D50, D3872, D280 and D185 as part of Applicable Documents. Unnecessary number 1 will be eliminated from 5.1.1, 5.1.2, etc. 5.1.1 will be changed to 5.2 and Method D1208, Part 5.1.2, changed to D280 for moisture and D185 for coarse particles under appropriate 5—numbers.

Specification D1199 for Calcium Carbonate Pigments received negatives from three different voters. One negative will be resolved by correcting typographical error "BFG" to "BET" the correct term for surface area by nitrogen absorption. Two other negatives will be resolved by correcting particle size definition of new Superfine grade V limestone. A comment that ISO grade "d" specifies a 0.01% max on 45 μm sieve was considered non-persuasive. Other comments from the same voter concerning the elimination of unnecessary tertiary numbers and the inclusion of Method D3360 were adopted.

Another comment questioned the inclusion of Method D718. Precipated Calcium Carbonate oversize is determined by a procedure very similar to D718. Editorial change to correct typographical error in types from 4 to 5 will be made in 1.2. Also unnecessary ASTM in test methods will be deleted.

The following specifications that are up for review and balloting in 1980 were discussed and the following action taken:

D261, Iron Blue; D262, Ultramarine Blue; D656, Toluidine Red Toner; and D963, Copper Phthalocyanine Blue were approved with minor changes in Applicable Documents and color and strength statements for concurrent subcommittee and main committee balloting. On D963, footnote 3 will be changed to read: "Aroplaz 7310-X-50, available from Spencer Kellogg Div. of Textron, Inc., P.O. Box 807, Buffalo, N.Y. 14240, a short oil alkyd soya type, would be suitable for this purpose."

Specifications D85, Ochre, and D602, Barium Sulfate, will be submitted for subcommittee and main committee concurrent ballot. Changes made will include correct spelling of ochre, change "Tests" to "Sampling," add applicable documents, and add appropriate test methods.

Extender pigment specifications D604, Diatomaceous Silica; D607, Wet Ground Mica; and D867, Pumice, will be submitted for concurrent subcommittee and main committee balloting after making appropriate editorial changes to include applicable documents, tests to sampling, and the inclusion of particle size test D3360.

Practice and procedure specifications D153, Specific Gravity, and D1366, Reporting Particle Size, will be submitted for concurrent balloting after making minor editorial changes.

Specifications D912, Cuprous Oxide, and D478, Zinc Yellow, will be submitted for concurrent subcommittee and main committee balloting after appropriate editorial revision.

Specification D911, Mercuric Oxide, will be modified to include a safety precaution as part of the scope. The statement will read: "This standard contains a known toxic material and it is the responsibility of whoever uses the standard to employ proper safety and handling practices and evaluate regulatory limitations before use." Editorial changes regarding applicable documents will be made and D911 submitted for concurrent balloting.

Specifications D209, Lampblack; D263, Chrome Oxide Green; D267, Gold Bronze Powder; and D1648 Basic Lead Silico Chromate, will be modified to incorporate appropriate editorial changes and submitted for concurrent balloting.

On D1648 for Basic Lead Silico Chromate, the same safety statement as developed for mercuric oxide will be added to the scope.

In response to the three negatives from the Committee on Standards on final approval of Specification D962, safety aspects of packaged aluminum pigment pastes were reviewed with aluminum producers. They proposed a safety statement which was modified to read: "Aluminum paste should be packaged in a polyvent container which can be safely punctured because of a possible pressure build up." This statement will be added under a new packaging section to D962 and the revised specification will be submitted for reballoting.

A request from a distributor for a specification for a talc-like product was discussed. Since the product does not meet D605 requirements, the originator of the request will be contacted and asked to follow standard procedures and officially request a specification with supporting data. It was reported that a formal request will be submitted for development of a specification for calcium borosilicate pigment.

SUBCOMMITTEE D01.32 DRYING OILS

P.C. Stievater, Chairman

A negative vote on Method D1983, "Standard Test Method for Fatty Acid Composition by Gas Chromatography of Methyl Esters," was found to be persuasive. It was agreed that the method is unclear in its reference to "standard solutions" and that certain sections would be rewritten for the voters approval before submitting the method for Society ballot.

The planned withdrawal of Method D1953, "Standard Test Methods for Drying Properties of Drying Oils," received no negatives or comments. It will go to Society ballot for withdrawal, to be replaced by Method D1640," Standard Test Methods for Drying, Curing or Film Formation of Organic Coatings at Room Temperature." This method, under the jurisdiction of Sub. D01.33, is being revised and balloted concurrently.

Five other methods, D12 on Specification for Raw Tung Oil, D961 on Specification for Dehydrated Castor Oil, D2071 on Testing of Fatty Nitrogen Products, D2800 on Preparation of Methyl Esters from Oils for Determination of the Fatty Acid Composition by Gas Chromatography, and D3457 on Preparation of Methyl Esters from Fatty Acids for Determination of the Fatty Acid Composition by GLC, received only editorial comments. Revised drafts of these methods will be forwarded to ASTM Headquarters with most of the suggested comments included.

SUBCOMMITTEE D01.33 VARNISH & RESINS, INCLUDING SHELLAC

W. C. Golton, Chairman

Group 1: Varnishes. H.A. Ball, Chairman, discussed negative votes received on recent ballots. Two negatives were received on Method D479, "Reactivity of Paint Liquids." One negative was found persuasive and the method will be balloted for withdrawal. The second negative was also found persuasive but action on the comments in the negative will be withheld pending the outcome of the ballot for withdrawal. Method D1643, "Gas Checking and Draft Test of Varnish Films," had one negative which was persuasive. It was voted to place the method on ballot for withdrawal. A second negative will be withheld pending the outcome of the ballot for withdrawal. Comments on Method D1639, "Acid Value of Organic Coatings," and Method D1647, "Resistance to Water and Alkali of Dried Films of Varnishes," will be incorporated and the methods submitted for a main committee ballot.

Group 6: Drying Time. H.M. Werner, Chairman, reported that two negatives had been received on Method D1640, "Test Method for Drying, Curing or Film Formation of Organic Coatings at Room Temperature." One was found nonpersuasive because no suggestion was made as to specific improvement in the method and existence of other Society standards are not sufficient grounds for a change. The other negative was found persuasive, three parts of which were considered technical. It was agreed to accept the temperature change in section 3.1 to 23 ± 2° C. The parts on section 5.8 and Table I were withdrawn pending additional work by the subcommittee. Various editorial changes will be made according to suggestions from four voters. The revised draft will be resubmitted for subcommittee letter ballot.

Group 12: Urethanes. R.A. Foss, Chairman, reported that Method D2615, "Test for Free Toluene Diisocyanate in Urethane Polymers by Distillation," received no negatives on balloting for withdrawal. This method was replaced by Method D3432, "Free Toluene Diisocyanate in Urethane Prepolymers and Coatings by Gas Chromatography." The initial testing for isophorone dissocyanate in urethane prepolymers by gas chromatography is very encouraging. More extensive round robin participation will be required. A new method for free isocyanate determination by liquid chromatography will be submitted to evaluate toluene diisocyanate, isophorone diisocyanate, and hexamethylene diisocyanate.

Group 14: Alkyds. R. Malek, Chairman, reported that Method D1544, "Color," had passed Society ballot and COS review and will be published as revised in 1981. Five methods are in for Society ballot at this time: D563, "Test for Phthalic Anhydride Content of Alkyd Resins and Resin Solutions"; D1306, "Test for Phthalic Anhydride Content of Alkyd Resins and Esters Containing Other Dibasic Acids (Gravimetric)"; D1397, "Test for Unsaponifiable Matter in Alkyd Resins and Resin Solutions"; D2689, "Recommended Practices for Testing Alkyd Resins"; and D2690, "Test for Isophthalic Acid in Alkyd and Polyester Resins." Two overdue test methods, D2455 on "Identification of Carboxylic Acids in Alkyd Resins," and D2456 on "Test for Polyhydric Alcohols in Alkyd Resins," should have been balloted for reapproval in 1979 and will be placed on the next main committee letter ballot. Method D1615, "Test for Glycerol, Ethylene Glycol and Pentaerythritol in Alkyd Resins," and D2998, "Test for Determination of Polyhydric Alcohols in Alkyd Resins," should be reviewed in time for the next ballot. A limited study of a proposed method for determination of ortho phthalic acid anhydride and fatty acids in alkyds by gas chromatography is underway. If certain problems can be resolved, a round robin will be conducted. It was suggested that NMR methods pertaining to alkyds be solicited for possible task group action.

Group 23: Epoxy Resins. P. Kamarchik, Chairman, reported that no negatives were received on balloting for reapproval of Methods D1652, "Test for Epoxy Content of Epoxy Resins"; D1726, "Test for Hydrolyzable Chlorine Content of Liquid Epoxy Resins"; and D1847, "Test for Total Chlorine Content of Epoxy Resins." A number of editorial changes based on suggested comments will be made and the methods issued as corrected. In response to concern that D1847 is an obsolete method, the PPG method, which utilizes sodium biphenyl, will be evaluated by round robin. Consideration is being given to developing a recommended practice for characterization of epoxy resins.

Group 24: Nitrogen Resins. J. Smith, Chairman, reported that there were two negatives on the main committee ballot for Method D1013, "Test for Total Nitrogen in Resins and Plastics." One negative was withdrawn when it was agreed to make the editorial changes requested by the voter. The second negative felt that mercury should be replaced by selenium as a catalyst for safety reasons. This negative was voted nonpersuasive because (1) mercury and its compounds are still acceptable for analytical use, (2) a change to selenium would not eliminate the health hazard, (3) a statement on hazard and disposal will be placed in the method, and (4) the supporting data indicates that a method based on selenium would not be accurate enough. A suggestion to develop a recommended practice for nitrogen resins will be considered. It was decided to ballot Methods D1597, "Test for Melamine Content of Nitrogen Resins," and D1727, "Test for Urea Content of D1727, "Test for Urea Content of Nitrogen Resins," for withdrawal. Method D1198, "Solvent Tolerance of Amine Resins," will be balloted for reapproval.

Group 26: Emulsions. D. Brown, Chairman, reported there were three negatives on the subcommittee ballot on the proposed new method for nonvolatile content. One negative was withdrawn upon agreement to clarify the scope. A second negative was withdrawn upon agreement to include a statement by the voter in the scope. The third negative was found to be persuasive in that the precision data should have been calculated differently. Various editorial suggestions were made and many adopted. The method will be revised including a new precision statement, and reballoted. Work on freeze-thaw and heat-age stability will be completed in time for the next meeting.

Nonvolatile Methods—Method D1644, "Nonvolatile Content of Varnishes," received a negative ballot on the basis that there were problems when applied driers. It was voted to find this argument non-persuasive on the grounds that driers are not mentioned in the scope. Some of the editorial suggestion will be incorporated. Method D1259, "Nonvolatile Content of Resin Solutions," received a negative vote. It was agreed to make all of the editorial changes and the voter agreed to withdraw his negative. It was decided to solicit methods for improving and consolidating the above test methods. A new task group will be formed for this purpose.

Miscellaneous Methods—Method D1156, "Determination of Total Chlorine in Polyvinyl Chloride Polymers and Copolymers Used for Surface Coatings," will be balloted for withdrawal because it is no longer used in the industry and an alternate method, D1243, exists. A recommended practice for testing of vinyl resins and coatings will be compiled. It was voted to place on Society ballot for reapproval without change the following methods: D1469, "Total Rosin Acids Content of Coating Vehicles"; D1542, "Qualitative Tests for Rosin in Varnishes"; D1546, "Performance Tests of Clear Floor Sealers"; and D1641, "Exterior Durability of Varnishes."

SUBCOMMITTEE D01.35 SOLVENTS, PLASTICIZERS AND CHEMICAL INTERMEDIATES

L.R. Thurman, Chairman

One negative from the November 1979 main committee ballot was resolved at this time. This negative on D600, "Specification for Liquid Paint Driers," was carried over from the last meeting. The negative was based on the fact that the scope of the specification was missing. It had been accidently left out when the revision was retyped and this editorial oversight was corrected in the January meeting by adding the original scope statement. For this reason the negative was no longer considered to be valid.

Group 1: Hydrocarbon Solvents. W.J. Frost, Acting Chairman, is continuing development of a test method for benzene in hydrocarbon solvents. Data from several laboratories were not received in time for discussion at this meeting. D235, "Specification for Petroleum Spirits," and D484, "Specification for Hydrocarbon Dry Cleaning Solvents," will be combined into a revised D235 for balloting. D484 will be withdrawn. The "Doctor Test" described in D484 will be prepared as a test method. D363, "Specification for Industrial Grade Xylene," was assigned for review.

Group 2: Oxygenated Solvents. T.H. Golson, Chairman, will continue study on low water in urethane solvents. Several proposed gas chromatography methods will be reviewed. A proposed specification combining all of the present ASTM specifications for ketones will be balloted in the subcommittee for the January 1981 meeting. There will be a total of 13 standards for subcommittee ballot from this group.

Group 3: Chemical Intermediates. J.M.Kauffman, Chairman, will ballot a new proposed specification for glacial acrylic acid listing dimer content at 1.0% maximum level. A new specification for 2-ethylhexanol will be revised to include a gas chromatographic determination for assay and a sulfuric acid color test. There will be six standards for subcommittee ballot and seven standards were assigned for review.

DIVISION 40 PAINT PRODUCTS APPLIED ON SITE

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

R.A. Brown, Chairman

Group 1: Purchase Criteria for Latex Flat Paints. J. Csernica, Chairman, is conducting a round robin test program to determine minimum number of criteria needed for procurement purposes. Twelve brands of white latex flat wall paint were procured through the cooperative effort of the State of Virginia. Samples of each brand were distributed to nine laboratories who will test them for: opacity (hiding power) by Method

D2805, 85° gloss (appearance) by Method D523, washability by Method D3450, and scrubbability by Method D2486. The cooperating laboratories are State of Virginia, State of Florida, DL Laboratories, General Services Administration, DeSoto, Sherwin-Williams, Glidden, DuPont, and Consumers Union. Some test results have been received and the program will be completed prior to the next D-1 meeting in January 1981. General Services Administration will use the test results in aiding them to write a Commercial Item Description (CID) for the purchase by government of commercial flat latex wall paints.

Group 2: Standard Guide for State and Institutional Purchase of Paint. W.V. Moseley, Chairman, reported that draft #10 of the Standard Guide for State and Institutional Purchasing of Paint has been approved and is in the process of being printed as an ASTM Standard. It has been given the number D3927. This culminates several years of work by the group and has established a new basis for procurement of paint products by States and other interested groups.

SUBCOMMITTEE D01.42 ARCHITECTURAL PAINTS

R.S. Armstrong, Chairman

Four negative ballots were received on a new "Standard Method of Test for Color Compatability." The objections from one voter were editorial in nature. These changes will be made and the voter asked to withdraw his negative vote. The second negative consisted of some corrections and editorial suggestions which were considered to be persuasive. He also suggested that the title be changed and did not think that section 6.1 was clear. These two objections were also considered to be persuasive. The third negative objected to the use of the Kubelka-Munk equation D/S for this method. The subcommittee members agreed with this objection and the method will be changed to use color readings instead of reflectance readings and any changes in color due to shear will be reported as ΔE values. The fourth negative stated that the use of a spectrophotometer should also be mentioned in the method. The use of ΔE measurements will refer to Method D2244 which includes the use of a spectrophotometer. Both of these negatives on color measurement were considered to be persuasive.

Two negative ballots were received on a new method to evaluate leveling. One negative consisted of editorial changes. The subcommittee was agreed to these changes and the voter will be asked to withdraw his negative if these changes are made. The second negative had to do with a more complete description and use of the test apparatus. These objections were found to be persuasive and the method will be modified to reflect these changes.

A new method for Evaluation of Scrub Resistance (by weight loss) received one negative vote on a subcommittee letter ballot. The objection was considered to be persuasive by the group but the subcommittee found this objection to be non-persuasive.

Group 1 will make some changes in the method for Scrub Resistance and submit it for subcommittee and main committee ballot.

Group 2 is starting to work on developing a method to evaluate Practical Stain Removal.

Group 3 will make some changes in the method to evaluate Leveling and submit it for subcommittee and main committee ballot.

Group 4 is starting to work again on a method to evaluate Wet Adhesion.

Group 5 is starting to work on a new method to evaluate Chalk Resistance.

Group 6 was formed to work on a "Recommended Practice for Testing Latex Semi-Gloss Paints" with S. LeSota as Chairman.

Group 7 was formed to develop a method to evaluate Leveling with L. Schaeffer as Chairman.

Group 9 will make some changes in the method to evaluate Color Compatibility and resubmit it for subcommittee and main committee ballot.

Group 16 is still attempting to develop a method to evaluate Practical Hiding but the high cost of large test panels is a problem.

Group 17 is developing a method to evaluate Roller Spatter. Results are promising and the method could be submitted for ballot soon.

SUBCOMMITTEE D01.44 TRAFFIC COATINGS

W.V. Moseley, Chairman

Six standards up for reapproval in 1979 and seven for reapproval in 1980 will be submitted for simultaneous subcommittee and main committee letter ballot.

Group 1: Thermoplastics. D. Miller, Chairman, is currently conducting round robins on bond strength and percent reflectance. Proposed round robins are planned for abrasion resistance and viscosity.

Group 2: Traffic Paints. E. B. Countryman, Chairman, reviewed the final report of the round robin on Method D711," "Dry to No Pick Up Time." The prescribed ASTM procedure for running round robins had not been followed and another round robin will be started. Round robins currently underway are D1309, "Settling Properties of Traffic Paints on Storage," and D869, "Evaluating Degree of Settling of Paint." Round robins were proposed for D868, "Evaluating Degree of Bleeding of Traffic Paints," and D969, "Laboratory Test for Degree of Bleeding of Traffic Paint." An experimental test method on "Abrasion Resistance of Organic Coatings by the Air Blast Abrasion Method" will be investigated for use with traffic paints.

Group 3: Night Visibility. J. Ritter, Chairman, received detailed copies of the test results of a field test on three types of retroreflectometers conducted by the Pennsylvania Department of Transportation. A special task force was formed to review and update the following test methods: D1155, "Percent Rounds of Glass Spheres," D1213, "Crushing Strength of Glass Spheres," and D1214, "Size Gradation of Glass Spheres."

Group 4: Catalyzed Coatings for Pavement Markings. D. Miller, Chairman, advised that 15 test methods were suggested for consideration. No round robins are planned at this time.

Group 5: Water Borne Coatings for Pavement Markings. E.B. Countryman, Chairman, discussed actual road test adhesion failures caused by early rainfall and traffic. It was agreed that work should be done to develop a test method which would duplicate this failure on an asphaltic substrate.

SUBCOMMITTEE D01.45 MARINE COATINGS

L. S. Birnbaum, Chairman

It was reported that 12 people, eight from industry and four from government, had responded to the April questionnaire on future Sub. D01.45 activities. Based on the results, it was decided to put Group 3, Intermittent Submergence, and Group 4, Marine Atmosphere, on an inactive status due to lack of support. Efforts will be concentrated on Group 5, Algae Control; Group 6, Dynamic Testing; and Group 7, Antifouling Rating. All those interested in algae control will be sent a detailed questionnaire to get recommended approaches. Those interested in dynamic testing will be contacted by A. Freeman, group chairman. The proposed new antifouling rating method will be circulated for review and comment before starting the round robin.

SUBCOMMITTEE D01.46 INDUSTRIAL PROTECTIVE PAINTING

D.M. Berger, Chairman

At the January 1980 meeting, a list of topics for future work by the subcommittee was compiled. The list was put into a questionnaire form and distributed to members of the industry. Based on approximately 100 returns, the top four priority items were selected for immediate work and the subcommittee reorganized to undertake the new responsibilities.

Group 2: Surface Preparation. Negative votes and comments on D610 and D2200 from the recent D-1 letter ballot were reviewed. On reapproval of D610, "Evaluating Degree of Rusting on Painted Steel Surfaces," one negative and one affirmative with comments were received. The negative was deemed to be editorial. It requested English units as alternates to metric and advised that ASTM follow ISO precedent on various items. It was felt that this would be more appropriately handled by the editorial group within ASTM, rather than by subcommittee members. The affirmative with comments was reviewed and editorial changes made. On reapproval of D2200, "Pictorial Surface Preparation Standards for Painting Steel Surfaces," the affirmative with comments was editorial and appropriate changes made. On the group of Specifications covering preparation of metal surfaces for painting, it was agreed to submit D1730 (aluminum), D1731 (hot dip aluminum), D1732 (magnesium), and D2092 (zinc coated steel) for reapproval without change.

As a result of the questionnaire, there was a great deal of interest in a guide for painting over galvanized steel. E. Horvick will chair this group with its new number (.10). It was agreed that much of the information compiled for SSPC could be consolidated into an ASTM recommended practice. The guide will address the painting over new galvanized, aged galvanized, and include a chart of generic coating types that can be used over galvanized substrates. A draft will be prepared and distributed to all subcommittee members prior to the next meeting.

Group 3: Painting Practice for Field Applied Coatings. As a result of the letter questionnaire, it was determined that a need for a method to identify old coatings which had been previously applied to structures was first priority and should be combined with a practice for selection of subsequent systems which would be compatible. Because this problem addresses itself to other field applied coatings as well as highway and railroad types, the latter two groups (.06) and (.08), will be consolidated into this group for these new tasks. D. Noxon will chair the group which will address the preparation of two separate documents. One will be a standard method for field identification of coating films. The second will be a recommended practice for selecting coatings that will be compatible with existing coated surfaces. Future work may include guidelines for the extent of repainting required on existing structures. Copies of the Navy CEL publication on identification of existing systems and a draft of the proposed work outline will be distributed to all members prior to the January meeting.

Group 7: Inspection and Elcometer Adhesion Test. The results of the D-1 letter ballot on D3276, "Guide for Paint Inspectors," were reviewed. One negative and one affirmative with comments were received. The negative was discussed and ultimately withdrawn upon agreement to an editorial revision. Some of the comments attached to the affirmative vote have been incorporated into the document. While the guide is being resubmitted for ballot, the subcommittee recognizes the need for a major revision to D3276. In particular, the guide often references "economic considerations" which are valuable from an information standpoint, but are not part of an inspector's responsibility. While the recent draft makes this clear, the ultimate goal is to develop a guide in which "economic considerations" or compromises are not included. R. Martell will draft the major revision.

Based on the questionnaire, there was determined to be a considerable need for a standard method for pull-off adhesion testing in the field. A draft of a proposed test method that had been prepared two years ago for this purpose was briefly discussed. A. Cunningham will serve as chairman and make changes in the draft for review prior to the next meeting. Included with the revised draft will be a list of questions regarding variables on the use and calibration of the instruments to assess the most common techniques used in the industry, and any problems encountered.

Group 9: Topcoating Zinc-Rich Primers. The results of the questionnaire indicated considerable interest in the topcoating of zinc-rich primers. The group discussed reasons for cohesive failure of zinc-rich coatings with regard to freeze/thaw cycles, improper ambient and humidity variables, porosity of the zinc-rich and the low coefficient of thermal expansion of cured inorganic zinc-rich versus that of the basic steel. As a result of these and other problems, it was decided there was a need for a guide for topcoating zinc-rich primers. M. Tellor will be chairman and initial work will address zinc-rich primers only. A proposed outline for the guide was prepared and it is hoped that the items in the outline can be filled in and a draft sent to all subcommittee members before the January meeting.

DIVISION 50 PAINTS FOR FACTORY APPLICATION

SUBCOMMITTEE D01.51 POWDER COATINGS

R.C. Kissler, Chairman

One method, D3451, "Practice for Testing Polymeric Powders and Powder Coatings," is due for review in 1980. Minor revisions and additions are projected and it is planned to submit a revised draft by September 1980. Two methods, one for determination of color of powder coating resin solutions using the platinum-cobalt method, and another using the tristimulus method, are being submitted for simultaneous Sub. D01.51 and D-1 letter ballots. A method for measuring powder compatability will be submitted for Sub. D01.51 and D-1 ballot.

Work is continuing on viscosity measurements and hiding power. Availability of equipment for determining texture is being investigated.

SUBCOMMITTEE D01.52 FACTORY-COATED WOOD PRODUCTS

R.C. Marck, Chairman

A negative vote was received on D2830, "Test for Durability and Compatibility of Factory-Primed Wood Products," during a recent main committee ballot. It concerned seven revisions largely updating documents and was resolved by making the necessary changes. D2793, "Block Resistance of Organic Coatings on Wood Substrates," was revised editorially as suggested by one voter. A round robin will be undertaken to provide the required precision statement for this standard. D2336, "Practice for Specifying Properties of Paint from the Liquid State Through the Curing Stage for Factory Applied Coatings on Wood Products," and D2691, "Microscopical Measurement of Dry Film Thickness of Coatings on Wood Products," have been revised and will be submitted for concurrent balloting.

Group B: Hardboard. A five year correlation of field exposures to accelerated weathering tests on prefinished hardboard siding has been completed. The 16-page final report was issued to all members present.

Group 12: Textured Paneling. A round robin is planned to develop a test method for determining the durability of finishes on textured paneling by using a soil, clean, soil technique.

Group 52/55 on UV Curing Coatings. J.M. Behrle, Acting Chairman, discussed a recently distributed draft of a "Standard Practice for Determining Package Stability of Coatings for Ultraviolet Curing Coatings." It was agreed that a statement be added to par. 5.1.1 indicating a relationship exists between storage life and the ratio of the area of liquid-air surface to the liquid volume. The draft will be submitted to Subs. D01.52 and D01.55 for letter ballot before the next meeting.

SUBCOMMITTEE D01.53 FACTORY-COATED STRIP METAL

E.E. Haney, Chairman

Group 1: Formability. W.H. Gunn, Chairman, reviewed a rough draft on a "Standard Method for Formability Characteristics of a Zinc Rich Primer/Chromate Complex Coating on Steel."

Group 2: Cure. K.E. Luyk, Chairman, reviewed round robin data on "Cure Determination by Solvent Rub (Finger Method)." A discussion was held concerning the poor reproducibility of this method and the need to emphasize this in a precision statement.

Group 3: Pretreatments. R.L. Williamson, Chairman, reviewed round robin data on determining coating weights on zinc coated steel. A revised draft will be submitted for subcommittee review.

Group 5: T-Bend Flexibility. R. De Graaff, Acting Chairman, is seeking a new chairman. The initial draft for "T-Bend Flexibility on Aluminum Substrate" was distributed for review.

Group 6: Drawdowns. R. Reinsel, Chairman, reviewed a "Recommended Practice for Applying Coatings Using the Wire Wound Draw Down Rod." It will be submitted for subcommittee ballot and editorial review.

A liaison report covering National Coil Coaters Association work was given. It should be noted that three years exposure has been completed on the coil series. When the data has been summarized, it should be made available to Sub. D01.27.

SUBCOMMITTEE D01.55 FACTORY-APPLIED COATINGS ON PREFORMED PRODUCTS

F.J. Steslow, Chairman

Methods D2091, "Print Resistance of Lacquers," and D2620, "Light Stability of Clear Coatings," have been reviewed and approved for Society ballot without change. Methods D1308, "Effect of Household Chemicals on Clear and Pigmented Organic Finishes;" D2571, "Standard Practice for Testing Wood Lacquers;" and D3459, "Humid-Dry Cycle Testing," have been reviewed and approved for Society ballot with editorial changes. Two standards are scheduled for reapproval in 1981. These are D3023, "Resistance of Factory Applied Coating on Wood Products to Stains and Reagents," and D3002, "Coating for Plastic." The latter should be reviewed for incorporation of water reducible coatings as pertaining to wood finishing. Subcommittee members were also asked to report back on electrostatic application and its applicability to methods currently in place.

Group 7: Water Thinnable Industrial Coatings. F.J. Steslow, Acting Chairman, advised that the initial draft of the Recommended Practice is in a general format. Books 27, 28 and 29 have been reviewed and all test methods and practices as applicable to the Practice incorporated. These will be carefully reviewed and selected for inclusion in five sections headed by the following task group chairman: Section 1—Liquid Coating Properties, C.K. Schoff; Section 2—Application and Film Formation, R. Huddleston; Section 3—Appearance of Dry Film, L. Schaeffer; Section 4—Properties of Dry Film, J.M. Behrle; and Section 5—Analysis of Liquid Coating, F.J. Steslow. All sections are to be completed and returned to the Subcommittee Chairman by Sept. 1. The chairman will review and collect Sections into a single document by October 1. The document will then be sent out to Sub. D01.55 members and Editorial Subcommittee as soon as possible after October 1. All recipients are to submit comments, suggestions, and any recommended changes by November 3 to the Chairman for preparation of final document for January 1981 meeting.

SUBCOMMITTEE D01.56 PRINTING INKS

F.J. Fetsko, Chairman

Group 1: Viscosity. F.J. Fetsko, Chairman, reviewed the results of the subcommittee ballot on the proposed method for "Viscosity of Vehicles and Printing Inks by the Falling Rod Viscometer." The three negatives were resolved by deleting the appendix. Many editorial comments were incorporated. A method of calculation for viscosity against shear rate will be added to the method annex. A method of calculation for the power law equation will also be added. All other comments were discussed and most will be implemented. The revised draft will be submitted for a D-1 ballot.

Group 6: Tack of Printing Inks. W. Gerlach, Chairman, reviewed a draft method for "Relative Tack of Printing Inks by the Inkometer." A considerable number of comments and questions were presented on each section. C. Shepard will prepare a second draft to reflect this additional information and have it ready for a subcommittee letter ballot.

New Business—Considerable interest developed in a smoke test. A task group with the assignment of developing a laboratory test method for predicting smoke emissions from a stack was created with J. Coleman as chairman. Some interest in measuring the relative tinting strength of ink color developed. A. Scarlotti was appointed task group chairman to review an existing NAPRI method and report its suitability.

SUBCOMMITTEE D01.57 ARTIST PAINTS AND RELATED MATERIALS

J.T. Luke, Chairman

This subcommittee is working on a specification for Artists' Paints and on test methods needed to determine conformance to the specification. Results are needed from a set of lightfastness tests before the specification can be completed. This data will not be available for another year; therefore, other sections of the specification are being agreed to separately by the subcommittee in order to speed final approval. The section of the specification dealing with requirements for labeling was balloted at the subcommittee level. There were no negatives. Balloting at the main committee and Society level will wait until all sections are approved by the subcommittee.

Group 1: Preparation of Samples for Colorimetric Determination. H.W. Levison, Chairman, noted that color difference data from the round robin conducted using the proposed method for preparing films of artists' tube paints for colorimetric determination, has indicated that only an 87.5% precision for repeatability could be achieved. Oil colors encountered lower repeatability than acrylics, and cobalt blue oil color had the lowest repeatability among the colors. Further analysis of the data will be made.

Group 2: Lightfastness of Pigments. H.W. Levison, Chairman, circulated a draft outlining the test methods and specific pigments which will be used in tests soon to be exposed. Most pigments will be tested in both oil and acrylic vehicles. Exposure will be under glass for 50,000 langleys in the sun, and to equivalent exposure by xenon-arc and by daylight fluorescent lamps.

Group 3: Tinting Strength of Pigments. I. Shack, Chairman, reported work is progressing in an attempt to develop a method for determining the tinting strength of an artists' paint. A report was issued on work done on the tinting strength of chromium oxide green and cadmium red light paints using log K/S and K/S data. More pigments will be examined.

Group 4: Investigation of Test Methods Applicable to the Subcommittee. A.L. Spizzo, Chairman, reviewed revisions in the draft of the "Standard Specification for Artists' Paints," upon which agreement was reached at the June 22 special meeting. Revisions will be incorporated into the next draft. Additional information will be included in the specification about the type of draw-down blade to be used. Manufacturers' literature would be required to include additional pigment information beyond that required on the tube.

Group 5: Pigment Identification. T. Pamer, Chairman, reviewed possible formats that could be used in compiling methods for pigment identification. Methods for both organic and inorganic pigments will be included.

Group 6: Definitions of Terms. T. Vonderbrink, Chairman, continued work on defining terms needed for the specification. Some of the terms will be sent to the attention of the *Paint/Coatings Dictionary.* The need for a general glossary was discussed.

Group 7A: Vehicle, Lightfastness and Distensibility. H.W. Levinson, Chairman, reviewed a draft of a proposed method for testing the physical properties of artists' vehicles and varnishes. A presentation on the tests performed was given. Flexibility and distensibility were discussed and it was decided that a test method is needed for each property.

AD HOC SUBCOMMITTEE COATINGS FOR ENCAPSULATION OF ASBESTOS FIBERS

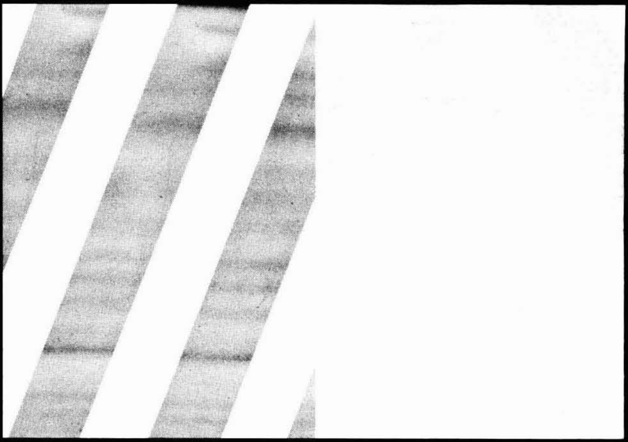
R.A. Brown, Acting Chairman

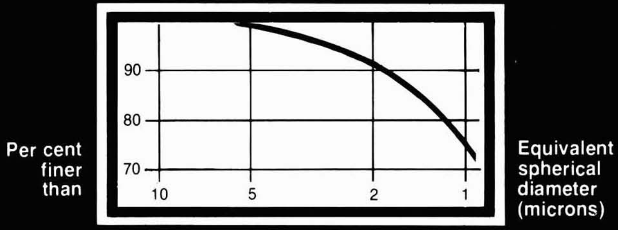
The subcommittee was given an update on W. Mirick's work at Battelle Laboratories in evaluating coatings for asbestos containing substrates. Battelle is now working on their third grant from EPA. A report on the second study involving some 60 coating products is due in about one month.

There was considerable interest in the problem as it is estimated that more than 10 billion square feet of surfaces exist in United States which contain asbestos, and from which fibers can escape into the air. These surfaces need proper coating systems applied to prevent health hazards from asbestos inhalation. These surfaces exist in schools, airports, office buildings, public buildings and industrial plants.

The Ad Hoc Subcommittee main effort should be to develop a performance specification which will describe the properties of an effective coating system. Committee E-6 on Buildings and Construction is also interested in helping to solve this problem. It was felt that the chairman should call a meeting of this Ad Hoc Subcommittee at Battelle Laboratories in Columbus, Ohio prior to the January 1981 meeting of Committee D-1.

Subcommittee reports of ASTM Committee D-1 Divisions 1 through 20 were published in the September issue of the JCT—Ed.




90
80
70
10
5
2
1
Per cent finer than
Equivalent spherical diameter (microns)

Elections

BALTIMORE

Active

Aichroth, Jeffrey T.—Dutch Boy, Inc., Baltimore, MD.

Grumbine, Theodore J.—Hanline Bros., Inc., Baltimore.

Stewart, Jr., Fred—Duron, Inc., Deanwood Park, MD.

Associate

Bender, Thomas E.—Steeltin Can Corp., Baltimore, MD.

BIRMINGHAM

Active

Eddowes, Derek—Industrial Newspapers Ltd., Redhill, Surrey, Eng.

Hegarty, Michael T.—Newtown, Ind. Finishes, Atherstone, Warwickshire, Eng.

Waite, Philip Steven—Hoechst UK Ltd., Burleys Way, Leicester, Eng.

Whyley, Ian—B.I.P. Chemicals Ltd., Oldbury Warley, West Mid., Eng.

HOUSTON

Active

Bounds, Lee—Cook Paint & Varnish, Houston, TX.

Hayley, Julianne—Cook Paint & Varnish, Houston.

Jacobson, K.D. Jake—Cron Chemical, Houston.

Zermeno, Nash—Cook Paint & Varnish, Houston.

Associate

Faulkner, Stanley—CIBA-GEIGY Corp., Denton, TX.

Martin, Ron—Nalco Chemical Co., Sugar Land, TX.

LOS ANGELES

Active

Butin, Miguel A.—Zolatone Process, Inc., Van Nuys, CA.

Dubble, William H.—Reichhold Chemicals, Inc., Azusa, CA.

Lander, Arthur G.—Custom Building Products, Bell, CA.

Associate

Brede, Wallace E.—Spencer Kellogg, Div., Textron, Long Beach, CA.

Cotton, Bruce—Pluess Staufer, Lucerne Valley, CA.

Taylor, Christopher L.—Buckman Laboratories, Santa Ana, CA.

SOUTHERN

Active

Fischer, Joseph W.—Guardsman Chemicals, Thomasville, GA.

Graves, Joe E.—Witco Chemical, Enterprise, AL.

Horne, William Russ—Pen Paints, Inc., Sanford, FL.

McMahon, John P.—Majemac Enterprise, Clearwater, FL.

Mistalski, Robert—Artex Hobby Products, Inc., Lexington, SC.

Moes, Philip—Guardsman Chemicals, Thomasville.

Morando, Charles—Balder & Morando, Inc., Boca Raton, FL.

Myers, Harry S.—MCI Corp., Tampa, FL.

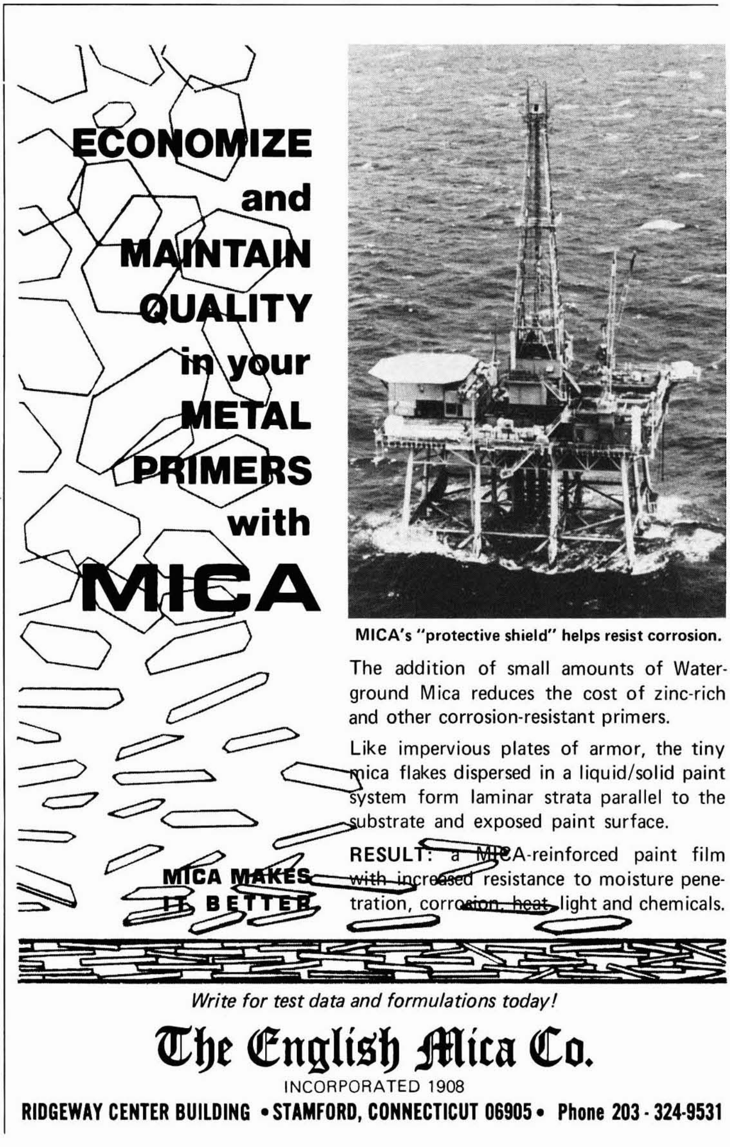
Patel, Natu P.—Coronado Paint Co., Edgewater, FL.

Rabley, Bill—Colormaid Mfg. Co., Lexington.

Rutherford, Henry S.—PPG Industries, Inc., East Point, GA.

Weeks, Fulton—MCI Corp., Tampa.

Wood, Thomas L.—PPG Industries, Inc., East Point.



Future Society Meetings

Baltimore

(Nov. 20)—JOINT MEETING with Baltimore PCA. AWARDS NIGHT featuring Herman Shugar Award.

(Jan. 15)—FSCT SLIDE PRESENTATION—Federation Officers.

(Feb. 13)—LADIES' NIGHT.

(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.

C-D-I-C

(Nov. 10)—PLANT TOUR of Armco Steel.

(Jan. 12)—"FORMULATION & APPLICATION OF INDUSTRIAL LOW SOLVENT FOR THE 80's"—Jim Larson, Cargill, Inc.

(Feb. 9)—"WASH SOLVENT RECLAMATION"—Larry Durr, DCI Corp.

(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.

Chicago

(Nov. 10)—"CARBON BLACK"—speaker from Columbia Chemicals; "AEROSOLS"—Gus Leep, Seymour of Sycamore, Inc.

(Jan. 5)—"SOLVENT ALKYD REPLACEMENT"—Dr. Andrew Mercurio, Rohm and Haas Co.; "ENZYMATIC RESINS"—Edward Glass, North Dakota State Univ.

Golden Gate

(Nov. 17)—"A FORMULATOR'S GUIDE TO SILICONE DEFOAMERS"—Edward M. Antonucci, Drew Chemical; LIBRARY NIGHT—Representatives to attend from Redwood City Library.

Houston

(Nov. 12)—"FACTORS GOVERNING TINTER PERFORMANCE"—Elio Cohen, Daniel Products Co.

(Dec. 10)—"THE DIFFICULT AND REWARDING ASPECTS OF SELLING FROM A SALESPERSON'S VIEW"—Sandra Collins, Hilton Davis Chemical Co.

(Jan. 14)—Education Committee Symposium.

(Mar. 11)—PAST-PRESIDENTS' NIGHT

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

Los Angeles

(Nov. 12)—"A FORMULATOR'S GUIDE TO SILICONE DEFOAMERS"—Edward M. Antonucci, Drew Chemical.

Montreal

(Nov. 5)—"STATISTICAL DESIGN APPROACH TO FORMULATING HIGHER SOLIDS ACRYLIC COATINGS"—R.R. Kuhn, Rohm and Haas Ltd.

(Dec. 3)—"INDUSTRIAL WASTES"—speaker from Eco Research Ltd.

(Jan. 7)—"OBSERVATIONS ON THE EXPERIMENTAL DETERMINATION OF CPVC"—Peter Hiscocks, CIL Paints Inc.

(Feb. 4)—Manufacturing Committee program.

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

(May 7)—PAST-PRESIDENTS' NIGHT

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

New York

(Nov. 11)—"NONLEAD, NONCHROMATE, CORROSION-RESISTANT PIGMENTS"—K.A. Haagenson, Buckman Laboratories.

New England

(Nov. 20)—"COMPUTERS AND THEIR RELATIONSHIP TO THE LABORATORY, OFFICE, PRODUCTION, SALES AND THE BOSS"—Harold Small, Marblehead Testing Laboratories.

(Jan. 15)—JOINT MEETING with New England PCA; "EFFECTIVE R&D MANAGEMENT/PRODUCT EVALUATION"— E. Bruce Euchner, Glidden Coatings & Resins.

(Feb. 19)—"AMINO CROSSLINKERS: RELATIONSHIPS OF STRUCTURE TO COATINGS PROPERTIES AND PERFORMANCE"—Dennis E. Erickson, Monsanto Plastics & Resins; "THE USE OF TITANATE COUPLING AGENTS IN COATINGS"—David Busker, Kenrich Petrochemicals.

(Mar. 19)—JOINT INDUSTRY MEETING.

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

Philadelphia

Technical Committee Meetings

(Nov. 6)—"A PROFESSIONAL VIEWPOINT OF SELECTING MICROCOMPUTERS"—Marty Becker.

(Dec. 4)—"USE OF HYDROGEN BONDING SOLVENTS FOR SUSPENSION OF UNTREATED SILICA FLATTING PIGMENTS IN PAINT"—Hubert Golden, PPG Industries, Inc.

(Jan. 15)—"CEMENTITIOUS COATINGS"—Dr. Joseph Lavelle, Rohm and Haas Co.

(Feb. 5)—"CONVERSION ENAMELS-ENERGY SAVERS"—Paul W. McCurdy, Reichhold Chemicals, Inc.

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

(Nov. 3)—"CURRENT STATUS OF VOC REGULATIONS AS THEY APPLY TO PROTECTIVE COATINGS"—Henry Alexander, Pa. Dept. of Environmental Research.

(Dec. 1)—"BACTERIAL PROBLEMS IN MANUFACTURING AND HANDLING LATEX EMULSIONS"—Jack Cherry, Rohm and Haas Co.

(Jan. 5)—JOINT MEETING with Pittsburgh PCA.

(Feb. 2)—"INDUSTRIAL HYGIENE AND URETHANE COATINGS"—Paul D. Ziegler, Mobay Chemical

(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.

Western New York

(Nov. 11)—"AN INVESTIGATION OF ABRASION RESISTANCE"—William B. Neuberg, Shamrock Chemicals Corp.

NPCA Changes Campaign Theme; Logo Approved

The National Paint and Coatings Association's new national public relations/promotional campaign is now strengthened by a new theme and logo, after formal approval by the NPCA Executive Committee. The Committee's approval came after a presentation of campaign progress to date and a demonstration of membership support for the program and its funding arrangement.

The Executive Committee determined that the campaign would be initially funded from NPCA resources, in the amount of $151,000, to carry to forward through December 1980. The Committee adopted a schedule for a Special Promotional Fee to be paid by benefiting members to fund the campaign thereafter. The total budget for the program has been set at $250,000 for the first year, $275,000 for the second year, and $305,000 for the third year.

The campaign's new slogan, "Picture It Painted," emerged after market research concluded that the tentative slogan, "Paint America Beautiful," was inappropriate for the campaign's objectives. The research, which consisted of separate consumer and retailer focus groups and interviews with paint manufacturer executives, was conducted by the Market Research Bureau, an affiliate of Henry J. Kaufman & Associates, the advertising/public relations agency selected to implement the campaign.

"Picture It Painted" will be the theme used for consumer publicity in magazines and newspapers, home show displays, consumer contests, and National Paint Month to be held annually in May. It is anticipated that participating paint manufacturers will feature the slogan and logo in their own individual advertising and promotional materials to further amplify this industry-wide sales promotional effort. The logo is unique in that it will graphically illustrate the color versatility of paint.

CARB Seeks Low VOC Finishes

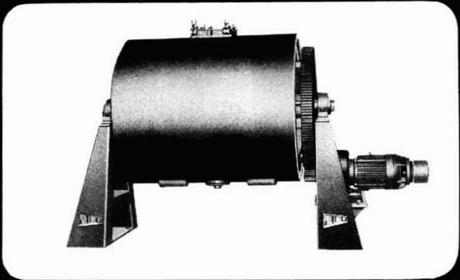
The California Air Resources Board (CARB), as part of its research program to investigate the current status of coating technology, is seeking industrial finishes for metal parts and products which meet its solvent limitation requirements. Consequently, CARB has contracted with D/L Laboratories to locate and evaluate the relative performance, vs. conventional industrial finishes, of commercial or developmental coatings (or formulations from raw material suppliers), which contain no more than 340 grams of volatile organic compounds (VOC) per liter of coatings, less water.

These coatings may be either water-based, high solids or powder. Any organic solvents may be used within the VOC limits. It is not necessary to meet Rule 66 or any of its variations.

The coatings should be intended for use on metal parts or products and can be cured by bake, air dry or force dry.

Selected submitted coatings will be evaluated vs. equivalent conventional solvent-thinned products. All products will be coded, no names will be used in the report, and all cooperators will receive a copy of the report with their code numbers.

Your cooperation is solicited. If you wish to have your products (or formulations) included in this program, please call or write Sidney B. Levinson, President, D/L Laboratories, 116 E. 16th St., New York, NY 10003 (212)777-4410.



CORROSION/81 to be Held in Toronto, April 6–10

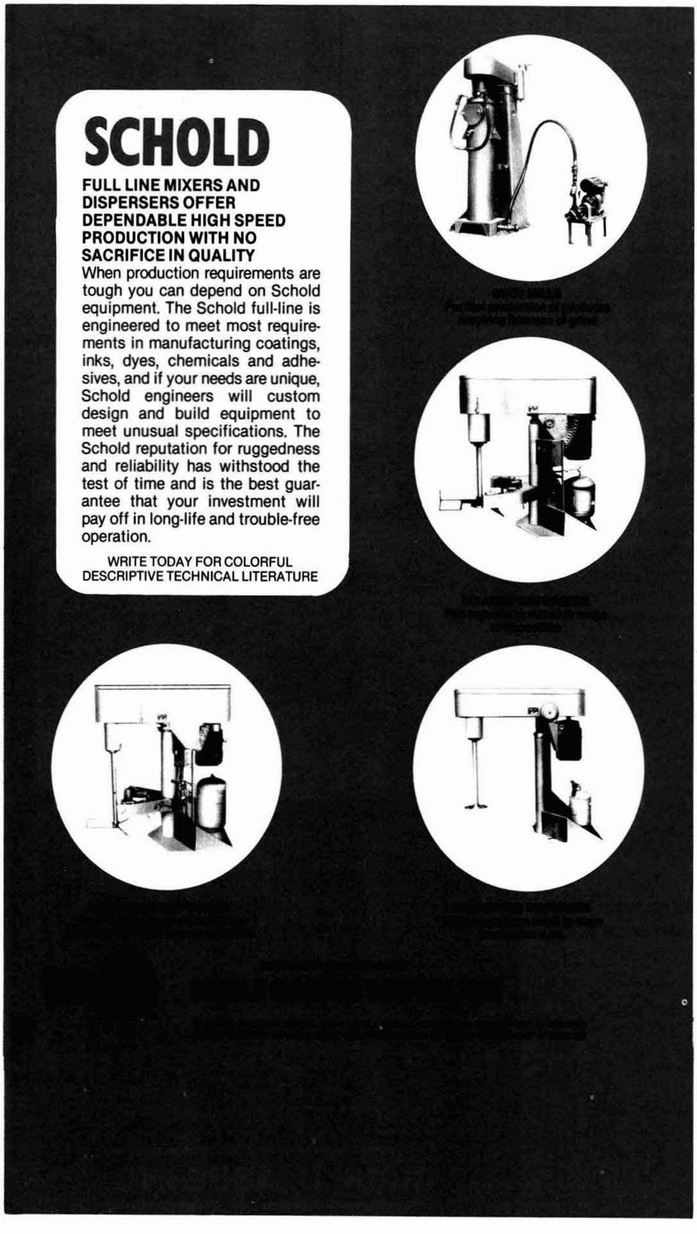
The National Association of Corrosion Engineers will hold its annual conference, CORROSION/81, at the Sheraton Centre Hotel, Toronto, Canada, April 6–10, 1981.

Approximately 3,500 engineers, technicians, and scientists are expected to attend the meeting, which will be held concurrently with the 1981 Materials Performance and Corrosion Show.

In addition to the 40 technical symposia scheduled, the Corrosion Research Conference will present papers dealing with the theme "Recent Contributions of Electrochemistry to Corrosion Research."

The 1981 Plenary Lecture will be presented by Dr. Hugh P. Godard who will speak on "The Performance of Aluminum in Corrosive Environments."

To obtain advance programs or additional information, contact NACE, P.O. Box 218340, Houston, TX 77218.



NACE Committee Sponsoring April 1981 Symposium On Underfilm Corrosion

A Symposium on Underfilm Corrosion, sponsored by Group Committee T-6 of the National Association of Corrosion Engineeres, will be held April 7–8, 1981 at the Sheraton Centre Hotel, Toronto, Canada.

Symposium Chairman D. M. Berger, of Gilbert/Commonwealth Associates, Reading, Pa., has announced the following presentations:

"Influence of Abrasive Cleaning on the Corrosion Resistance of Coated Steel—R. P. Wenz and J. J. Claus, of 3M Co., St. Paul, Minn.

"Coatings/Substrate Analysis by SEM and EDXRA"—Richard M. Holsworth, of Glidden Coatings and Resins Div., SCM Corp., Strongsville, Ohio.

"Are Modern Coatings and Their Methods of Application Giving Optimum Performance?"—D. A. Bayliss and H. Bray, of B. I. E. Anti-Corrosion Ltd., Watford Herts, England.

"Solid State Chemistry Principles as Applied to Corrosion-Induced Delamination"—Henry Leidheiser, Jr., of Lehigh University, Bethlehem, Pa.

"New Developments in Anti-Corrosion Protection and Repair Techniques to Offshore Structures"—Ken Lowrey, of Colebrands, Ltd., Lancashire, Enland.

"Performance of Coatings Applied Under Adverse Conditions"—Thomas Ginsberg, of Union Carbide Corp., Bound Brook, N. J.

"Underfilm Cutting and Permeation in Hostile Environments"—William J. Curren, of Foxboro Co., Foxboro, Mass.

"Corrosion Prevention by Increased Adhesion and Impermeability of Coating Films"—I. M. Winkeler, of Sigma Coatings Inc., Harvey, La.

"Underfilm Corrosion Control of Ferrous Substrate by a One-Component Moisture Curing Isocyanate Prepolymer"—Ronald Eritano, of Mobay Chemical Co., Pittsburgh, Pa.

"A New Dimension in Corrosion Protection"—Walter Sniff, of Corrosion Resistance Systems, Orrville, Ohio.

For complete program details and to register, contact National Association of Corrosion Engineers, 1440 South Creek, Houston, Texas 77084 (713) 492-0535.

People

Engelhard Minerals & Chemicals Div., Menlo Park, NJ, has named **C. David Richardson** Director of Sales, U.S. and Canada, for the firm's Pigments and Extenders Group. In his new position, Mr. Richardson will have responsibility for direct sales as well as distributor sales of nonmetallic minerals in North America.

Harmon Colors Corp. will combine with the Dyestuff Div. of Mobay Chemical Corp. to form Mobay Dyes and Pigments Div. This new division will be headed by **Dr. Heinz W. Machatzke** as Vice-President and General Manager. Harmon Colors will operate in the new division as a department, with **H. Prochaska** serving as its General Manager. **H. Richter-Oldekop,** formerly Director of Sales for the Dyestuff Div., will assume the position of General Manager for the new division's Dyes Dept.

Harry A. Wray, Consultant and Owner of Harry A. Wray Associates, Swarthmore, PA, has been named a 1980 recipient of the Award of Merit by the American Society for Testing and Materials (ASTM). Mr. Wray was cited for his work as the first Chairman of the ASTM Ad Hoc Committee on Flash Point Methodology and Government Response and for his promotion of ASTM interaction with government departments, agencies, and bureaus.

PPG Industries, Pittsburgh, PA, has elected **Richard H. Peake, Jr.** Vice-President for Government and Public Affairs. His new responsibilities include identifying public policy issues and proposed governmental actions of importance to the company and assuring the formulation of corporate policies and strategies that address them.

Pratt & Lambert, Inc., Buffalo, has appointed **Timothy E. Newerski** Chemist in its Powder Coatings Div. He was formerly employed by Whittaker Corp.

The firm has also named **Robert T. Richard** Sales Engineer in its Chemical Coatings Div. He will be based in Houston.

Frank Slater, Jr. has joined the Organic Chemicals Div. of W.R. Grace Co. as Senior Sales Representative. He will be based in Houston.

C. Richardson

D. Smith

S. Gonge

D. Holvey

As part of management reorganization under its new ownership, Troy Chemical Corp., Newark, NJ, has named **Scott Gonge** President and **Daryl Smith** Executive Vice-President. Former President, **Elias Singer,** will remain active with the company as Chairman Emeritus.

Kerr-McGee Corp., Oklahoma City, OK, has elected **Frank A. McPherson** President. In his more than 20 years with the company, Mr. McPherson has worked in and managed virtually every phase of the firm's petroleum, uranium, chemical, and coal activities.

Witco Chemical Corp. has promoted **Gerald Spiegelman** to Assistant Director of Corporate Research and Development. In his new position, Dr. Spiegelman will be involved in the administration of the company's Technical Center in Oakland, NJ.

Meanwhile, **Michael H. Fisch** has been named Manager of Research and Development for the firm. He will direct a wide range of technical projects.

Michael J. Carme, Donald E. Kaimann, Randy J. LaMotte and **Chastian Taurman** have joined Thiokol Corp.'s Specialty Chemicals Div. as Regional Sales Managers. They will be responsible for sales of polysulfide insulating glass sealants for the company.

Glidden Coatings & Resins, Div. of SCM Corp., has appointed **Richard Laurence** as Electrocoating Manager and **Robert Christiansen** as High Solids Manager. These technology managers will serve the company's markets in the appliance, automotive, farm and construction equipment, aluminum extrusion, and general metals industries.

The company has also named **Roy B. Watts** Quality Control manager for its Charlotte, NC powder coatings unit.

Sherwin-Williams Co., Cleveland, has appointed **David L. Parsons** Market Manager for private labels in the Consumer Div. and **David T. Holvey** Market Manager for its Martin-Senour brand.

Meanwhile, **Donovan H. Lumby** has joined the Specialty Products Div. of the company as Director of Product Engineering. He will report to **Charles Kumins,** Vice-President of Research and Development for the division. Mr. Kumins is a member of the Cleveland Society.

The Central Region of Glidden Coatings & Resins, Div. of SCM Corp., has announced the following promotions: **John T. Rebar**—Manager, Technical Services and **Lee W. Winters**—Field Sales Manager. In addition, **Dwight W. Kreuter** has been appointed Personnel Manager at the company's Midwest Consolidated Plant in Huron, OH.

Rohm and Haas Co., Philadelphia, has appointed **Gary W. Grindrod** as a District Manager in its Polymers, Resins & Monomers North America Business Team. He will be based in Bristol, PA.

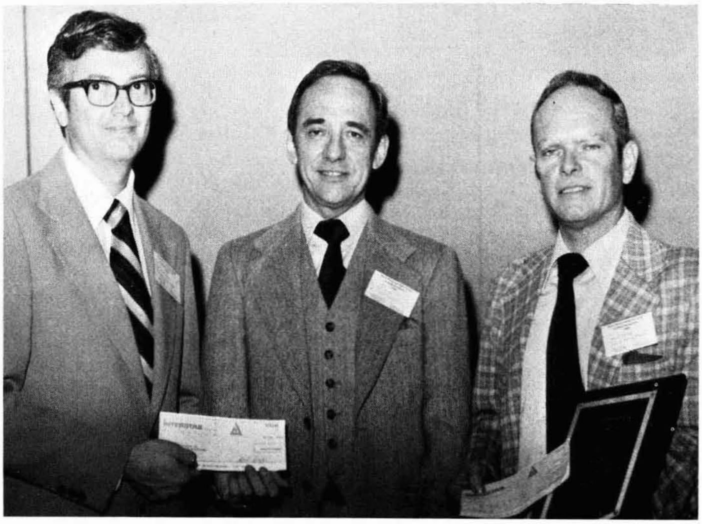
Mr. Grindrod is succeeding **J. Donald Urquhart,** who has retired after 40 years of service with the firm. Mr. Urquhart is a member of the Philadelphia Society.

Dr. Eric Baer, Dean of Case Institute of Technology of Case Western Reserve University, Cleveland, was named recipient of the 1981 American Chemical Society Award in the Chemistry of Plastics and Coatings, sponsored by Borden Foundation, Inc. Dr. Baer has edited several advanced treatises on the solid state of polymers, and serves on the editorial board of several scientific journals. The award will be presented at the ACS national meeting, March 1981, in Atlanta.

The Federation has announced the appointment of **Robert F. Ziegler** as Editor of the JOURNAL OF COATINGS TECHNOLOGY. Joining the FSCT staff in 1976 as Assistant Editor, he has served as Managing Editor since 1977.

Union Carbide Corp., New York, has promoted **Davida Barrett** to Sales Representative in its Coatings Materials Division sales force. Her territorial responsibilities will include Arkansas, Oklahoma, and north central and northeast Texas.

In a series of promotions, Midland Div., Dexter Corp., Waukegan, IL, has named **Edward A. McCarthy**—Supervisor of Beer and Beverage Interior Coatings Development Group; **Linda L. Icelic**—Senior Coatings Chemist in the Packaging Research Group; **Robert M. Baker** and **Roger W. Hoch**—Project Chemists in the Packaging Research Group; **Irene Flores**—Senior Research Technician of the General Line, Product Development Laboratory; and **Kanti V. Gandhi**—Staff Scientist in the General Line, Product Research Group. Messrs. McCarthy, Baker, Hoch, and Gandhi are members of the Chicago Society.



Dr. H. Arnoldy, President of Interstab Chemical Co., presents the Interstab Award and $500 check to Dr. P.G. Schmidt (right), of Du Pont Co., at the 1980 Water-Borne and Higher Solids Coatings Symposium in New Orleans. The award is given for the paper which represents the most significant contribution to science and technology in the field during the period between March 1979–1980. A $300 check was presented to Dr. B.G. Bufkin (left), of the Southern Society, to be used for an undergraduate scholarship in the Dept. of Polymer Science at the Univ. of Southern Mississippi.

Hunter Associates Laboratory, Reston, VA, has promoted **Howard I. McDevitt, Jr.** to the position of Vice-President of Engineering. To this position, Mr. McDevitt brings 17 years of diversified experience in engineering and management.

Richard Oglesbee has been named to the newly created post of Product Manager, Urethane Foam Coatings, for Gaco Western, Inc., Seattle. He will direct activities in new product development testing and marketing.

George L. Richon has been promoted to Technical Manager of the John C. Dolph Co., Monmouth Junction, NJ. A member of the New York Society, Mr. Richon will direct all activities of the firm's varnish and resin laboratories.

In a series of promotions at its Corporate Technical Center in Oakland, NJ, Witco Chemical Corp. has named **Clifford L. Hilton**—Senior Research Associate; **Richard D. Cowell**—Group Supervisor for Polymers and Polymer Applications in Corporate Research; **Koei-Liang Liauw**—Project Leader in Corporate Research; and **Barbara Hegranes, Robert Goldsmith, Louise Isbitsky,** and **Raymond F. Harron**—Research Chemists in Corporate Research and Development.

In addition, **Hal Mark** has been appointed to the position of Product Manager of a newly formed specialty catalyst group in the Organics division of the company.

Nalco Chemical Co., Oak Brook, IL, has promoted **Keith V. Davis** to the position of Industrial Division Regional Sales Manager for the Midwest. He previously served as District Manager, headquartered in Beaumont, TX.

Drew Chemical Corp., Boonton, NJ, has appointed **Dr. David R. Sexsmith** to the post of Vice-President of Technology. He will be responsible for the acquisition of new technology and products.

Meanwhile, **Dr. Elliott J. Levi** has been named Director of Research and Development for the firm. The author of numerous papers, Dr. Levi has an extensive background in the water and waste treatment and marine industries.

Rohm and Haas Co., Philadelphia, has assigned **Maurice I. Seifer** to the position of Senior Technical Sales Representative and **Rosemary J. Kunz** to Technical Sales Representative for its Polymers, Resins and Monomers North America Business Team.

Stanley J. Thompson, Jr. has been appointed Group Vice-President—Operations for Wyandotte Paint Products Co., Troy, MI. Mr. Thompson will assume responsibility for sales, research and development, and manufacturing.

In addition, the company has named **Peter T. Maxwell** Marketing Manager. His duties will include advertising and promotion, commercial development, and expansion of all current and future product lines.

Obituary

David H. Litter, Founder of the D.H. Litter Co., died July 27. His years of service to the coatings and coating materials industries began in 1914, when he joined Calco Chemical Co. as Traffic Manager, was promoted to Purchasing Agent, and was later named General Coordinator. After new management acquired Calco, Mr. Litter formed a partnership with Jack Allen, of Du Pont, in the Litter and Allen Co., as sales agents for chemical companies. When Mr. Allen left the partnership, Mr. Litter founded the D.H. Litter Co. He later began a technical service laboratory, which assumed the name of D/L Laboratories, Inc. During his lifetime, Mr. Litter was active in humanitarian organizations.

Hy Kredentser, an Honorary Member of the Montreal Society, died August 30 at the age of 55. Associated with both the Sherwin-Williams Co. of Canada Ltd. and the Montreal Society for 25 years, he retired from Sherwin-Williams in January 1979. In the Society, Mr. Kredentser served as President and Society Representative. His Federation activities included service as a member of the Board, Program Committee Chairman, and delegate to the National Association of Corrosion Engineers.

Coming Events

FEDERATION MEETINGS

(Oct. 28)—Federation Board of Directors Meeting. Hyatt Regency Hotel, Atlanta, Ga. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, Pa. 19107).

(Oct. 29–31)—58th Annual Meeting and 45th Paint Industries' Show. Atlanta Civic Center, Atlanta, Ga. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, Pa. 19107).

(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

(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. Plaza South Hotel, Birmingham, AL (Peter F. Decker, Union Carbide Corp., 17 Executive Park Drive, N.E. Atlanta, GA 30359).

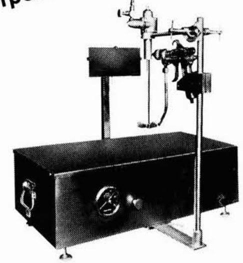
(Mar. 24–25)—Cleveland Society 24th Annual Conference, "Advances in Coatings Technology." Baldwin-Wallace College, Berea, OH. (George R. Pilcher, Sherwin-Williams Co., 601 Canal Rd., Cleveland, OH 44113.)

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

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

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.

OTHER ORGANIZATIONS

(Nov. 5-6)—Third Resins & Pigments Exhibition. Hotel Nikko, Dusseldorf, Germany.

(Nov. 7-9)—University of Waterloo Short Course, "Engineering Aspects of Polymer Systems." Hotel Toronto, Canada (Prof. A. Rudin, Dept. of Chemistry, University of Waterloo, Waterloo, Ontario, Canada).

(Nov. 10-12)—"Protective Coatings and Lining Work for Power Generation Facilities," sponsored by American Society for Testing and Materials Committee D-33, Atlanta, GA. (ASTM, 1916 Race St., Philadelphia, PA 19103.)

(Nov. 10-14)—"High Performance Coating Procedures," Houston, Tex. (Institute of Applied Technology, Jean L. Kaplan, 1200 - 17th St., N.W., Suite 406, Washington, D.C. 20036.)

(Nov. 10-21)—Paint Short Courses at University of Missouri-Rolla. Refresher Course for Maintenance Engineers, Contractors, and Painting Inspectors—Nov. 10-14; Job Estimating Workshop for Painting Contractors—Nov. 17-21. (Norma Fleming, Continuing Education, University of Missouri-Rolla, 501 W. 11th St., Rolla, MO.)

(Nov. 11-13)—4th International Conference of Paint Research Association, Excelsior Hotel, London Heathrow Airport. (PRA, Waldegrave Rd., Teddington, Middlesex TW11 8LD, England.)

(Nov. 17-Dec. 17)—Short Courses for the Chemical and Process Industries, The Center for Professional Advancement. "Water-Borne Industrial Coatings: Application"—Nov. 17-18; "Water-Borne Industrial Coatings: Chemistry and Formulation" —Nov. 19-20; "Organic Coatings Technology"—Dec. 3-5 and 15-17. Sheraton Motor Inn, East Brunswick, NJ (Rosanne Razzano, Dept. NR, The Center for Professional Advancement, P.O. Box H, East Brunswick, NJ 08816.)

(Nov. 19-21)—3rd Annual Western Plastics Exposition. Anaheim Convention Center, Anaheim, Calif. (Western Plastics Exposition, 1625 17th St., Santa Monica, CA 90404.)

(Nov. 21-23)—Annual National Decorating Products Association Convention and Show, H. Roe Bartle Convention Center, Kansas City, Mo. (NDPA, 9334 Dielman Industrial Dr., St. Louis, Mo. 63132.)

(Dec. 9-11)—Plant Engineering and Maintenance Show and Conference/West. Convention Center, Anaheim, CA. (Clapp & Poliak, Inc., 245 Park Ave., New York)

(Jan. 21)—ASTM Symposium, "Regiments for Predicting Permanence of Decorative and Protective Surfaces." Orlando, FL. (Symposium Chairman Garmond Schurr, Sherwin-Williams Co., 10909 S. Cottage Grove Ave., Chicago, IL 60628).

STATEMENT OF OWNERSHIP, MANAGEMENT AND CIRCULATION

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Federation Officers:
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  President-Elect—William H. Ellis, P.O. Box 97, El Segundo, CA 90246
  Treasurer—Howard Jerome, 1601 N. Broadway, St. Louis, MO 63102
  Executive Vice-President—Frank J. Borrelle, 1315 Walnut St., Philadelphia, PA 19107

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I certify that the statements made by me above are correct and complete. (signed) Robert F. Ziegler, Editor

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Advertisers Index

PAUL O. ABBE, INC.	**67**
ALCOA	**22-23**
AMERICAN CYANAMID CO.	**40**
AMERICAN HOECHST CORP.	**13**
ATLAS ELECTRIC DEVICES CO.	**71**
BYK-MALLINCKRODT CHEM. PROD. GMBH	**1**
CIBA-GEIGY CORP.	**10-11**
DEGUSSA CORP.	**25**
DOW CORNING CORP.	**Cover 3**
ECLIPSE SYSTEMS, INC.	**71**
ENGELHARD MINERALS & CHEMICALS CORP.	**Cover 2**
ENGLISH MICA CO.	**65**
FLINTKOTE STONE PRODUCTS CO.	**64**
DR. HANS HEUBACH & CO.	**17-18**
ICI AMERICAS, INC.	**26**
NALCO CHEMICALS CO.	**39**
NL CHEMICALS/NL INDUSTRIES, INC.	**16**
PFIZER, INC. MPM DIV.	**14-15**
PPG INDUSTRIES, INC.	**24**
POLYVINYL CHEMICALS INDUSTRIES	**20-21**
REYNOLDS METALS CO.	**19**
ROHM AND HAAS CO.	**8-9**
SCHOLD MACHINE CO.	**68**
SOUTH FLORIDA TEST SERVICE, INC.	**71**
SPENCER KELLOGG DIV., TEXTRON, INC.	**Cover 4**
UNION CARBIDE CORP.	**4-5**
R.T. VANDERBILT CO., INC.	**2**

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