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JOURNAL OF
COATINGS
TECHNOLOGY

JCTAX 52 (670) 1-96 (1980)
NOVEMBER 1980

Technical Computer Applications In the Coatings Industry

INTRODUCING

BYK

VP-155

POLYMERIC DISPERSANT

A Non-Foaming Anionic Dispersant for Latex Systems

BYK VP-155, the latest development from Byk-Mallinckrodt, represents a remarkable breakthrough in dispersants for latex systems.

BYK VP-155 affords stable pigment dispersion which ultimately provides substantial gloss improvement, increased shelf life and minimum foaming. Due to its polymeric structure, it does not detract from film properties.

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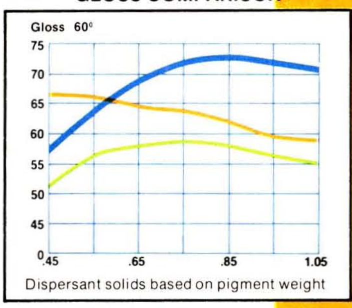
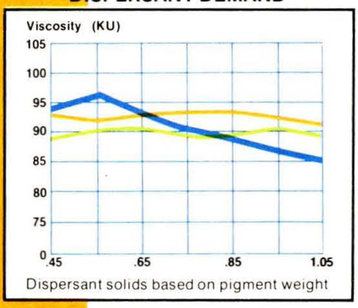


- BYK VP-155
- Competitor A
- Competitor B

- ***Substantial gloss improvement.***
- ***No foam produced during production or application.***
- ***Considerably improved stability on heat aging.***
- ***Effective with many prime pigments as well as most extenders and reactive pigments using a minimum dispersant level.***

DISPERSANT DEMAND



- BYK VP-155
- Competitor A
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NOVEMBER 1980

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Volume 52 Number 670

Features

Federation Activities

Departments

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Comment

A Study Guide to the Future

Not too many years ago, Data Banks might have been thought of as a chain of financial institutions. Today, the growth of science and technology is such that even youngsters recognize that the term refers to computers.

As recent developments have increased the complexity of computer systems, their functions have expanded to encompass the spectrum of industrial effort. The computerization of industry can, perhaps, then be considered as the second industrial revolution.

The coatings industry, of course, has not been left behind in a tangle of magnetic tape. Practically every aspect of paint manufacture is, or can be, accomplished or assisted via the computer. Analytical methods, testing equipment, color control, formulation, shipping, and marketing are only part of the litany of functions aided by the ubiquitous computer.

As part of its commitment to the International Coordinating Committee, the Federation, through the Technical Information Systems Committee, has compiled a bibliography, "Technical Applications of Computers in the Coatings Industry." Chairman Helen Skowronska and her committee searched through 12 years (1967–1979) of literature, selecting over 300 references in 10 categories. These, together with input from the French Association of FATIPEC, are contained in the work, which was discussed at this year's Annual Meeting, and is published in this issue of the JCT.

In a recent news item, experts predicted that within the next 10 years, U.S. industry will require over 4 million persons who are trained in the uses of computers. And further, that in the next 20 years, the ability to operate and program a simple computer will be a prerequisite for employment.

It is quite apparent that the highly competitive nature of business inextricably binds the languages of Industry and Science. The current bibliography stands as a "Study Guide" to the often used and seldom understood computer.—RFZ

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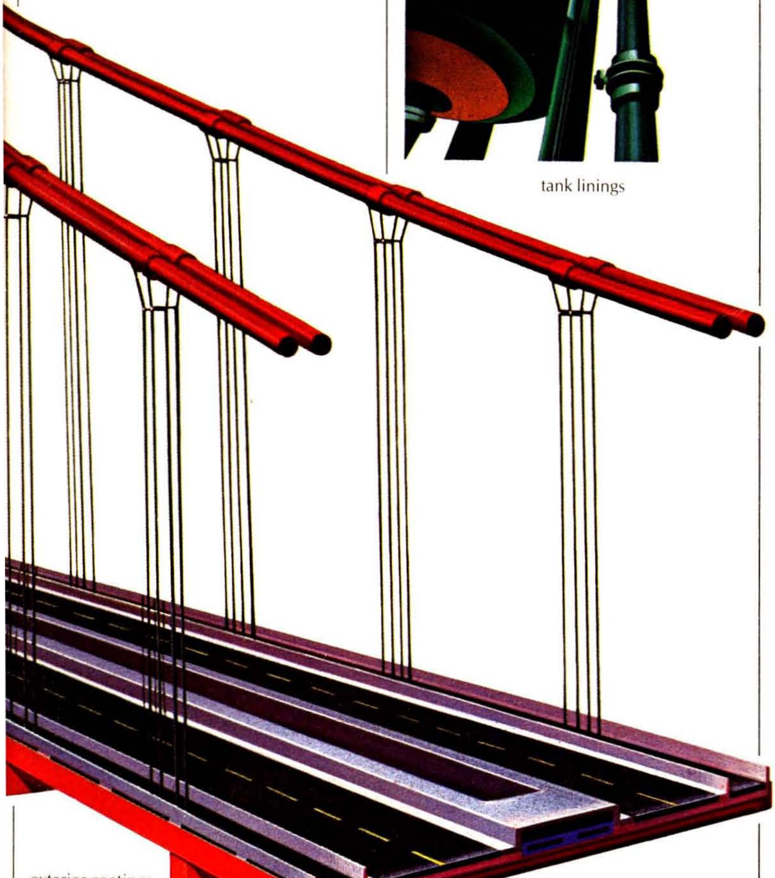
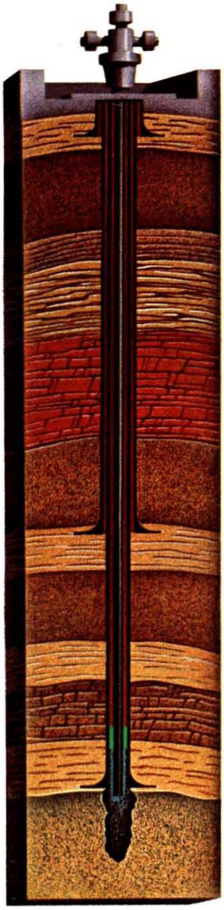


exterior coatings



down hole piping coating

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Abstracts of Papers in This Issue

A COMPUTER METHOD FOR PREDICTING EVAPORATION OF MULTICOMPONENT AQUEOUS SOLVENT BLENDS AT ANY HUMIDITY—A.L. Rocklin and D.C. Bonner

Journal of Coatings Technology, 52, No. 670, 27 (Nov. 1980)

Selection of cosolvents for water-reducible coatings is simplified by a computer program which predicts solvent balance and evaporation time of multicomponent water/solvent blends at any humidity. Conventional organic solvent blends are also accommodated, but without humidity correction. The computer method uses an additive equation incorporating activity coefficients calculated by the UNIFAC method. Operation is straightforward, and results are presented promptly at the computer terminal in tabular and graphic form. The method simplifies the problem of selecting blends which comply with air quality control regulations. Examples show how to choose among cosolvents, including those which are only partly miscible with water.

RADIOISOTOPE TRACER TECHNIQUE OF MEASURING ADSORPTION OF PAINT DRIERS BY PIGMENTS—E.S. Matsui

Journal of Coatings Technology, 52, No. 670, 39 (Nov. 1980)

A sensitive radioisotope tracer technique to measure the amount of paint drier adsorbed on paint pigments has been developed. This technique was found to be very sensitive and precise, and reliability and reproducibility of the procedure were very good. The laboratory results indicated that paint driers were adsorbed by pigments during storage, and the amount of adsorption varied among the paints.

ORGANO-SILANES AS ADHESION PROMOTERS FOR ORGANIC COATINGS—P. Walker

Journal of Coatings Technology, 52, No. 670, 49 (Nov. 1980)

Organofunctional silanes of the general formula $R\text{-}Si(OR^1)_3$ have been examined as adhesion promoters for organic surface coatings on metallic substrates. Torque spanner and direct pull-off adhesion measurements clearly show that dramatic improvements in the initial, water-soaked, and recovered adhesion of epoxide and polyurethane paints can be obtained on degreased and sandblasted surfaces when silanes are used as pretreatment primers. Investigations also show that similar improvements in adhesion can be obtained when selected silanes are incorporated into a paint in the single package, self-bonding concept.

The results of X-ray photoelectron spectroscopy (XPS) examination of the sites of adhesion failure are described, and the existing theories on the role and mechanism of silanes in increasing the adhesion of coatings to the substrate are reviewed in light of the observations recorded. Finally, the selection of silanes for use with particular formulations is discussed.

SURFACE ANALYSIS OF INTERFACIAL CHEMISTRY IN CORROSION-INDUCED PAINT ADHESION LOSS. II. EFFECTS OF RESIN CHEMISTRY AND SUBSTRATE PRETREATMENT ON ADHESION LOSS MECHANISM—J.W. Holubka, J.S. Hammond, J.E. DeVries, and R.A. Dickie

Journal of Coatings Technology, 52, No. 670, 63 (Nov. 1980)

The effect of changes in resin composition on the interfacial chemistry of corrosion-induced paint de-adhesion has been studied by X-ray photoelectron spectroscopy (XPS); the effect of a zinc phosphate conversion coating on interfacial chemistry has also been assessed in a preliminary way. For epoxy-urethane and epoxy-amine based coatings crosslinked with melamine-formaldehyde resin or mixtures of melamine-formaldehyde and urea-formaldehyde resins, it has been found that corrosion-induced de-adhesion is associated with hydroxide degradation of coating resin, typically by hydrolysis of urethane or, more slowly, of urea linkages. For an epoxy-ester based resin formulated with a mixture of melamine-formaldehyde and urea-formaldehyde resins and applied to bare steel, it was previously reported that corrosion-induced de-adhesion involves saponification of ester linkages by cathodically-generated hydroxide. For this coating applied over a zinc phosphate conversion coating, it is now found that there is again evidence of paint resin degradation by ester saponification. The surface composition of the conversion coating also changes significantly, but the locus of adhesion loss is evidently in the organic phase very near the organic-inorganic interface.

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Molyorange/BON Control	$1.50

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Pfizer Red (R-1599D)	27.3 lb.
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Pigment Cost/Gallon	$1.80
Molyorange/Per Red Control	$1.86

GREEN—SOLVENT & AQUEOUS SYSTEMS

Pfizer Yellow (YLO-2288D)	34.7 lb.
Phthalo Green	14.3 lb.
TiO_2	1.0 lb.
Pigment Level	50.0 lb./100 gal.
Pigment Cost/Gallon	$1.45
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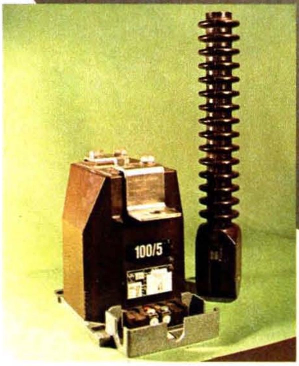
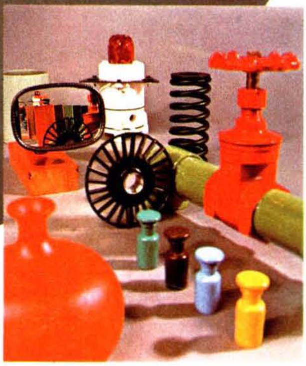


Anhydride based casting resins for the electrical industry

Filament-wound pipes based on anhydrides



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Anhydrides
HHPSA, THPSA and PMDA are white solids. MHHPSA is a low viscosity liquid.

Special hardeners
B 31, B 55, B 68, B 73 enable the production of highly reactive or normally curing powder coating systems with high-gloss or matt or dead-matt films.

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HHPSA = hexahydrophthalic anhydride
THPSA = tetrahydrophthalic anhydride
PMDA = pyromellitic dianhydride
MHHPSA = methyl-hexahydrophthalic anhydride

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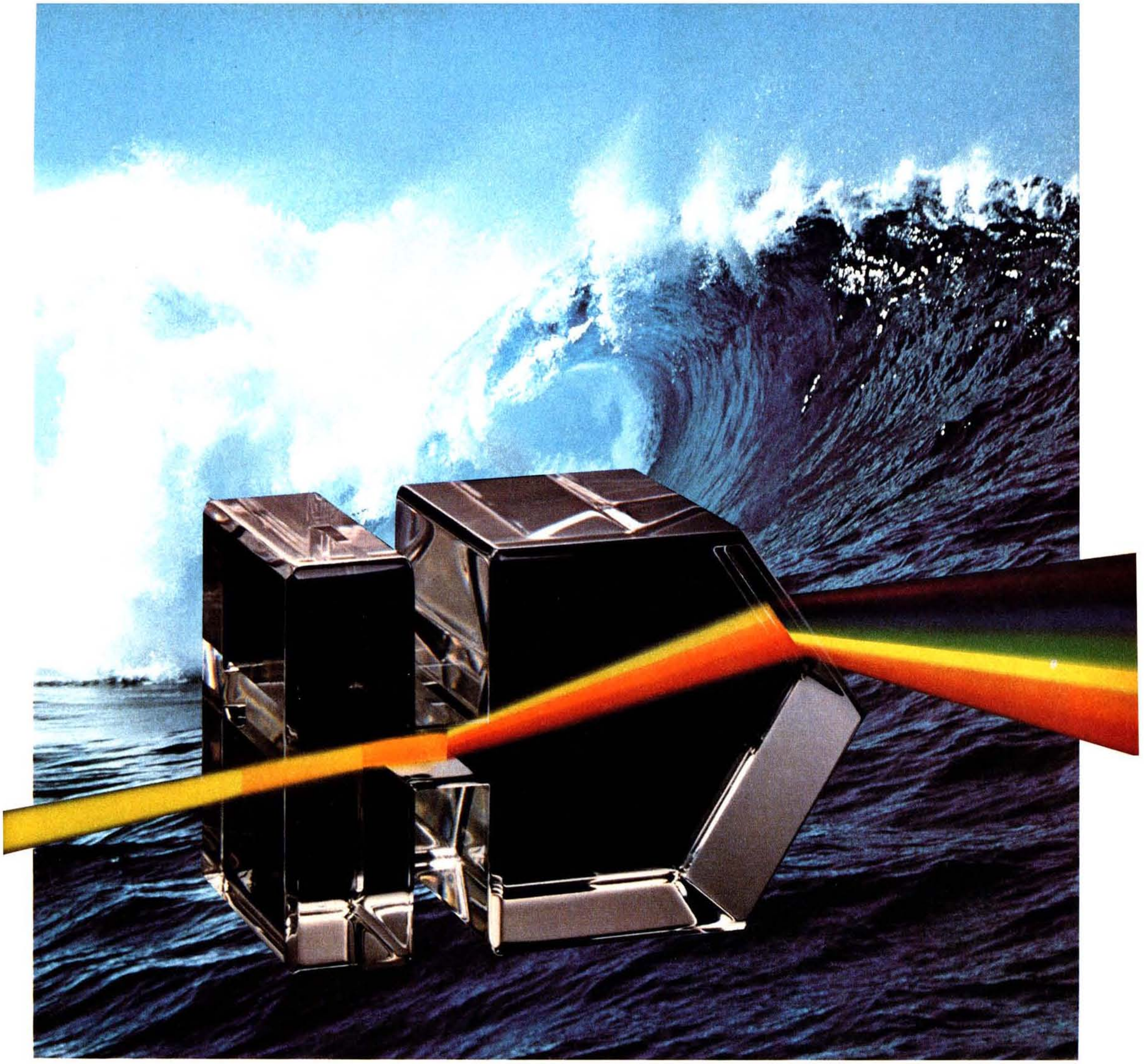
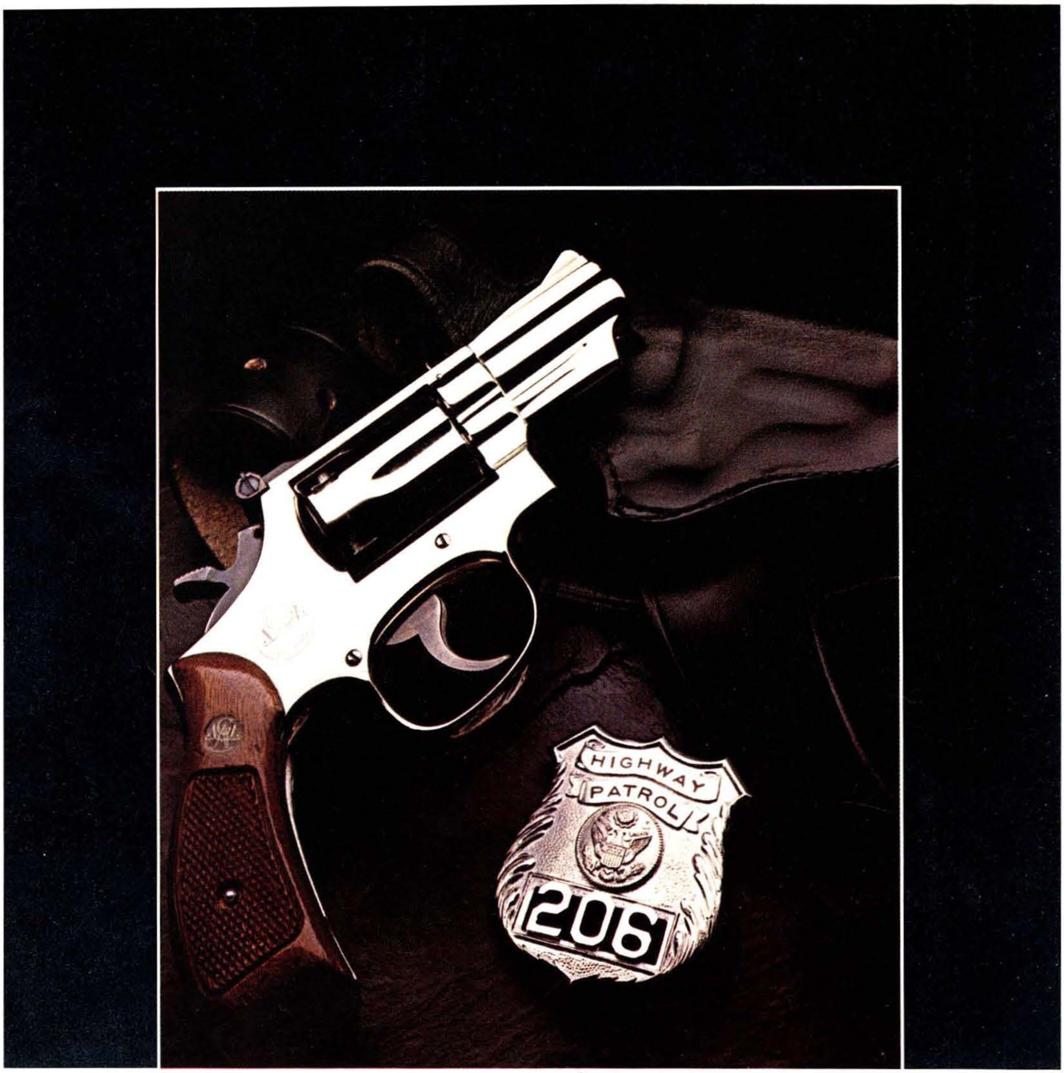


Surf off Maui, Hawaiian Islands



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- ■ 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
SO_2 resistance	↓	↘	↑
Light fastness	↗	↑	↑
Weathering fastness	↗	↑	↑
Hue steps	↑	↗	↗

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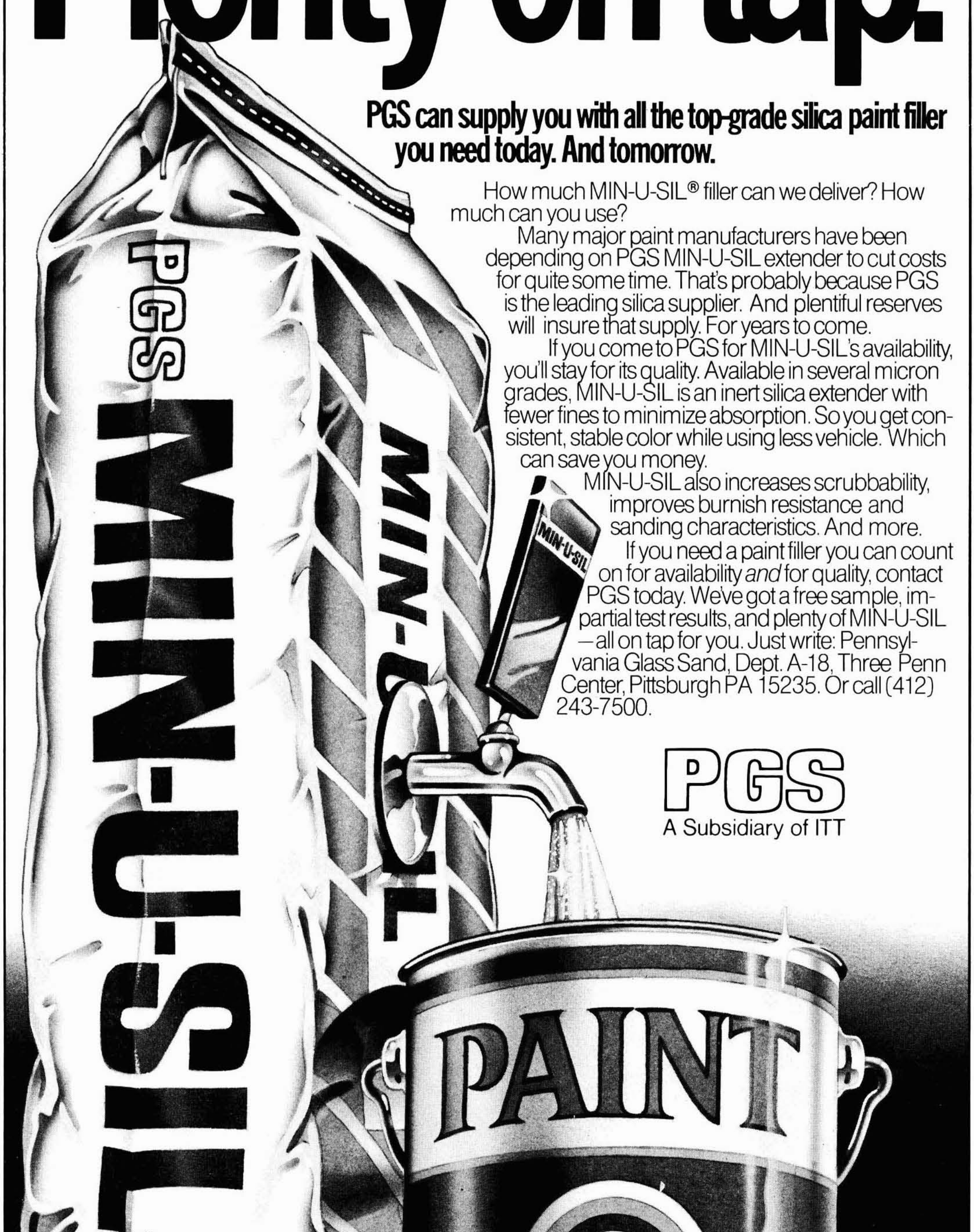
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William H. Ellis, of Los Angeles Society Becomes Fifty-Ninth Federation President

William H. Ellis, Senior Research Associate at Chevron Research Co., El Segundo, CA, became the 59th President of the Federation of Societies for Coatings Technology on October 31 at the Federation's Annual Meeting in Atlanta, GA.

Howard Jerome, of Spatz Paint Industries, Inc., St. Louis, MO, was named President-Elect, and A. Clarke Boyce, of Nacan Products Ltd., Toronto, Ont., was elected Treasurer.

President Ellis

Mr. Ellis is a Past-President of the Los Angeles Society and has served as Federation Council Representative. He was General Chairman of the 1972 West Coast Societies' Symposium and has served on numerous Society committees.

He has been a member of the Federation Board of Directors, and has also served on the Program and Publicity Committees as well as the Ad Hoc Committees on Education and Federation Restructuring.

A graduate of Stanford University, Mr. Ellis is a member of the American Chemical Society, American Association for the Advancement of Science, Sigma Xi, and the Air Pollution Control Association.

He and his wife, Bea, reside in El Segundo.

President-Elect Jerome

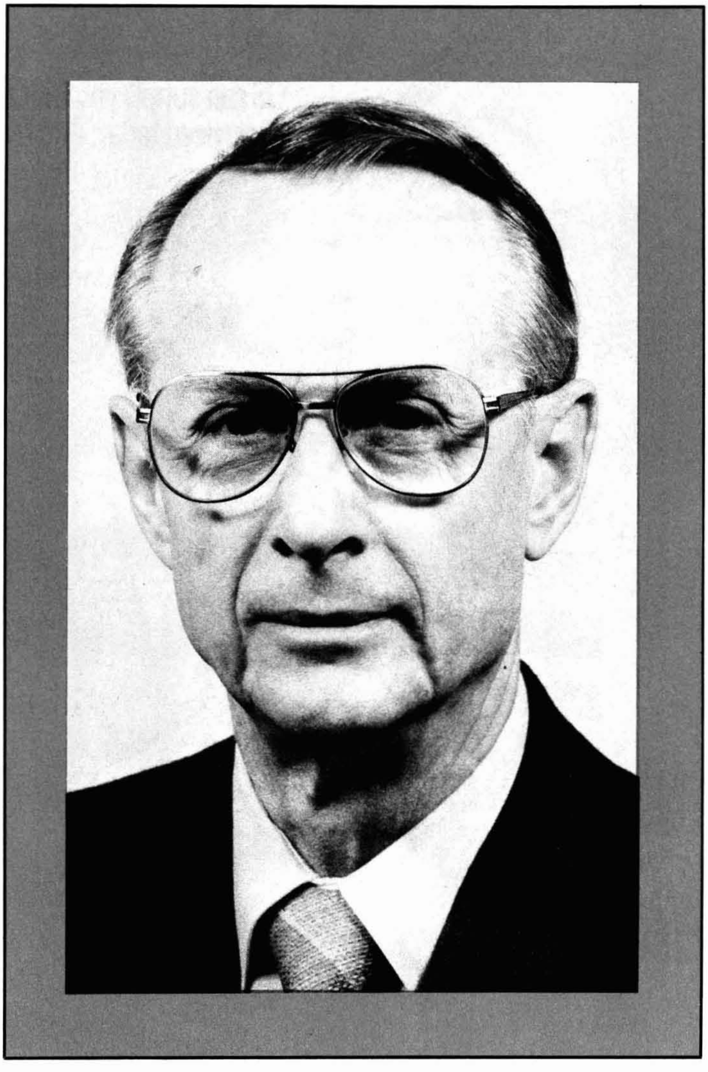
Mr. Jerome is a Past-President and Honorary Member of the New England Society (where he also served as Federation Council Representative), and most recently served as the St. Louis Society's Representative to the Federation's Board of Directors.

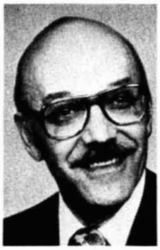
He has also served as Chairman of the Federation's Technical Advisory Committee (1969–70) and the By-Laws Committee.

Mr. Jerome is a graduate of Northeastern University.

Treasurer Boyce

Mr. Boyce is a Past-President of the Toronto Society. He most recently served as Society Representative to the Federation Board of Directors and as a member of the Federation Executive Committee.





H. Jerome

A.C. Boyce

'Challenge, Change, Opportunity' Is Theme of 1981 Annual Meeting

The 1981 Annual Meeting will have as its theme, "Challenge, Change, and Opportunity," it was announced by Program Chairman Thomas J. Miranda, of Whirlpool Corp., Benton Harbor, MI.

The Annual Meeting and Paint Show will be held at Cobo Hall, in Detroit, on October 28–30.

President Ellis Announces Committee Chairmen for 1980–81

Chairmen of the 30 committees of the Federation of Societies for Coatings Technology for 1980–81 have been announced by President William H. Ellis. A complete roster of all committees will be published in the 1981 Year Book.

An asterisk (*) indicates re-appointment for 1981.

A.F. Voss/American Paint & Coatings Journal Awards—Albert Seneker, Ameron Research & Development, South Gate, CA.

Bruning Award—Ruth Johnston-Feller, Pittsburgh, PA.*

By-Laws—Fred Schwab, Coatings Research Group, Cleveland, OH.*

Corrosion—Saul Spindel, D/L Labs, Inc., New York, NY.*

Definitions—Stanley LeSota, Rohm and Haas Co., Philadelphia, PA.*

Educational—John A. Gordon, Jr., University of Missouri—Rolla, Rolla, MO.*

Environmental Control—S. Leonard Davidson, N L Industries, Inc., Hightstown, NJ.*

Finance—Elder C. Larson, Shell Development Co., Houston, TX.

Heckel Award—Harold Spitzer

Host (Annual Meeting)—Jose Benavides, Ford Motor Co., Mt. Clemens, MI.

Inter-Society Color Council—Dennis Osmer, CIBA-GEIGY Corp., Ardsley, NY.

Investment—Herbert L. Fenburr, Columbus, OH.*

Liaison—John J. Oates, Troy Chemical Corp., Newark, NJ.*

Manufacturing—Donald Fritz, Superior Varnish & Drier Co., Merchantville, NJ.*

Mattiello Lecture—Felix P. Liberti, Chemray Coatings Corp., Middlesex, NJ.

MMA Awards—Terryl Johnson, Cook Paint & Varnish Co., Kansas City, MO.

Membership—A. Gordon Rook, O'Brien Corp., Fremont, CA.

Memorial—Carroll M. Scholle, Chicago, IL.*

Metric Systems—Ernest L. Humburger, Schenectady Chemicals Canada, Scarborough, Ont.*

Nominating—Elder C. Larson, Shell Development Co., Houston, TX.

Paint Industries' Show—Deryk R. Pawsey, Rohm and Haas Canada Ltd., Vancouver, B.C.*

Program—Thomas J. Miranda, Whirlpool Corp., Benton Harbor, MI.

Program Awards—Horace Philipp, Sherwin-Williams Co. of Canada Ltd., Montreal, Que.

Publications—Thomas J. Miranda, Whirlpool Corp., Benton Harbor, MI.*

Public Relations—Peter F. Decker, Union Carbide Corp., Atlanta, GA.

Roon Awards—Martin R. Hastings, Midland Div., Dexter Corp., Waukegan, IL.

Specifications—John A.J. Filchak, General Services Administration, Auburn, WA.*

Technical Advisory—William F. Holmes, DeSoto, Inc., Garland, TX.

Technical Information Services—Helen Skowronska, Cleveland, OH.*

Trigg Awards—Sharon Vadnais, E.T. Horn Co., Oakland, CA.

Delegates to Other Organizations

National Association of Corrosion Engineers—Thomas Ginsberg, Union Carbide Corp., Bound Brook, NJ.*

National Paint & Coatings Association, Scientific Committee—Ted Provder, Glidden Coatings & Resins Div., Strongsville, OH.*

Steel Structures Painting Council—Sid Levinson, D/L Labs., Inc., New York, NY.*

National Paint & Coatings Association and Government Agencies (Environmental Control)—S. Leonard Davidson, N L Industries, Inc., Hightstown, NJ.*

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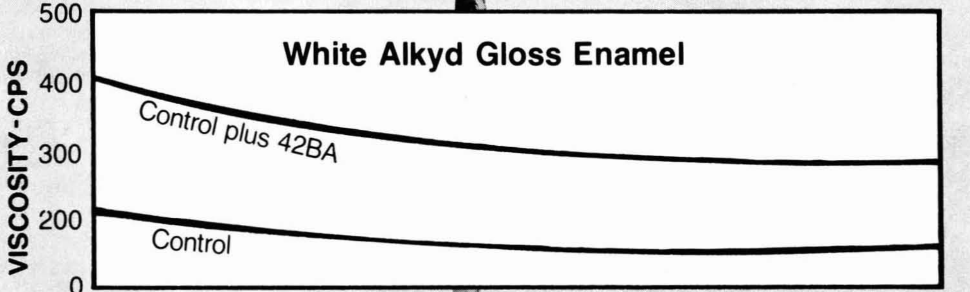

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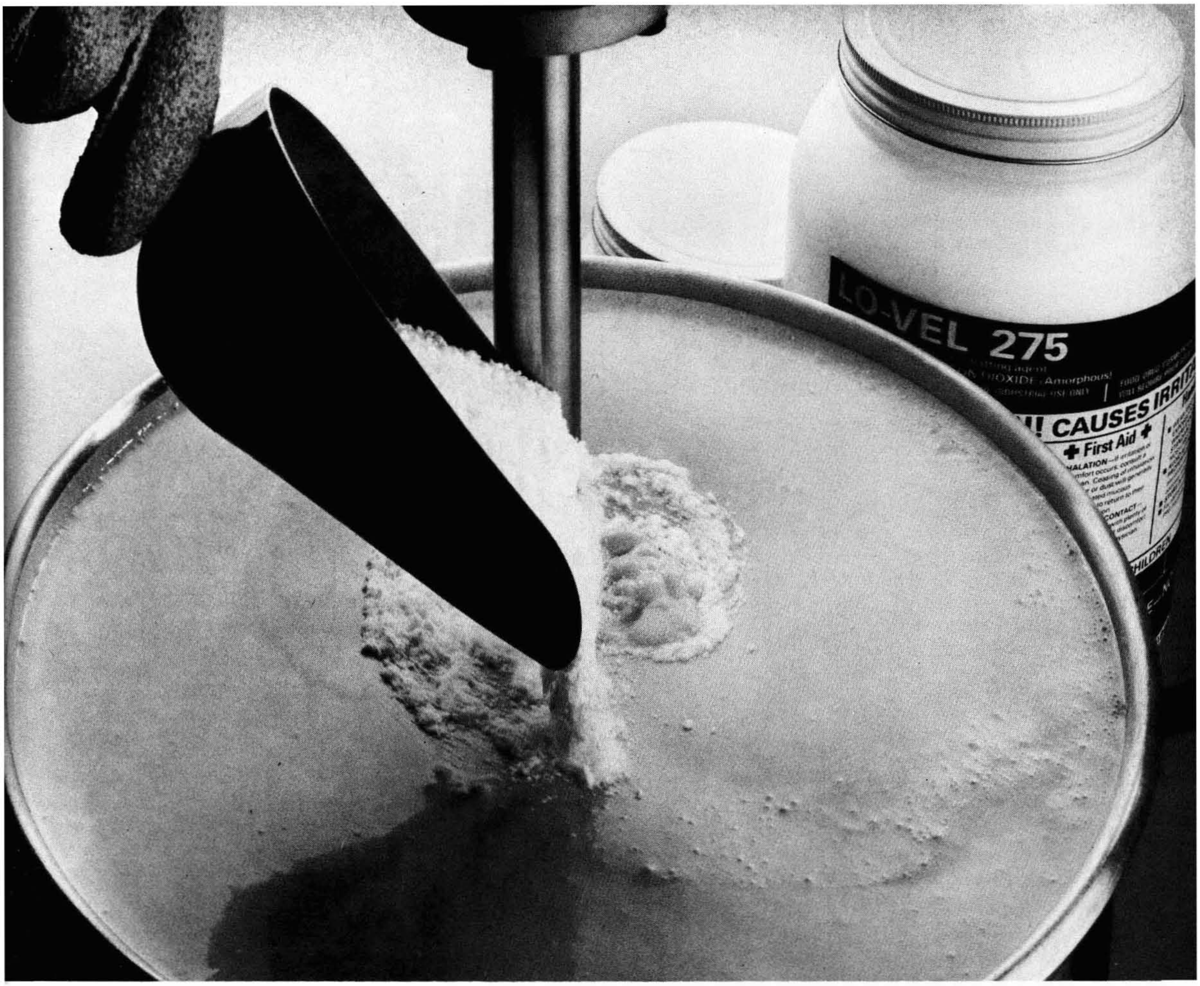

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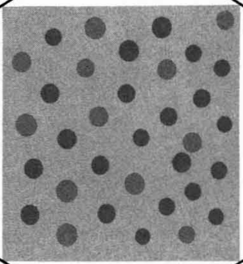

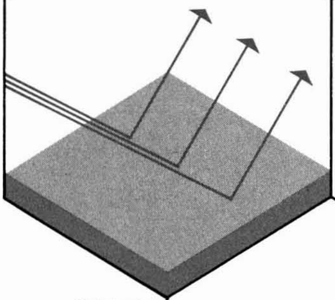

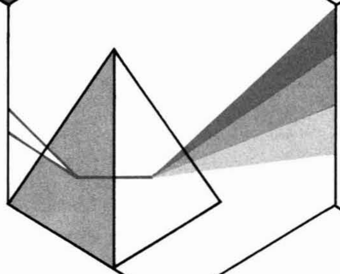

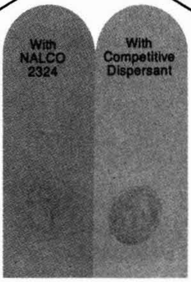
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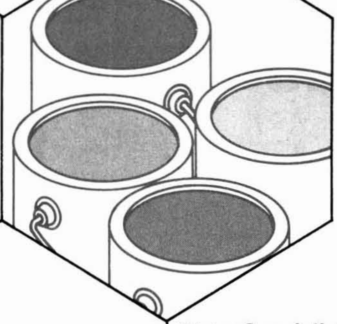

THIS PAPER WAS AWARDED SECOND PRIZE OF $450 IN THE 1980 ROON AWARDS COMPETITION

A Computer Method For Predicting Evaporation Of Multicomponent Aqueous Solvent Blends at Any Humidity

Albert L. Rocklin
Shell Development Company*
and
David C. Bonner
Shell Oil Company†

Selection of cosolvents for water-reducible coatings is simplified by a computer program which predicts solvent balance and evaporation time of multicomponent water/solvent blends at any humidity. Conventional organic solvent blends are also accommodated, but without humidity correction. The computer method uses an additive equation incorporating activity coefficients calculated by the UNIFAC method. Operation is straightforward, and results are presented promptly at the computer terminal in tabular and graphic form. The method simplifies the problem of selecting blends which comply with air quality control regulations. Examples show how to choose among cosolvents, including those which are only partly miscible with water.

INTRODUCTION

Humidity has a strong effect on the drying of a water-reducible coating. As shown in an earlier study,[1] humidity can drastically alter evaporation time and solvent balance. High humidity prolongs drying time by slowing down the rate of water evaporation from the solvent blend. This has the further effect of allowing the organic cosolvents to evaporate while the water is kept back. The result is a depletion of cosolvent and an enrichment of water. The effect becomes increasingly pronounced as humidity rises. Since film quality is affected by water-cosolvent balance from the moment of application through the final drying step, it is advantageous for the coatings technologist to be able to predict the effect of humidity on evaporation so that cosolvents can be selected for optimum performance.

Although previous studies had addressed the problem of the effect of humidity on water/solvent evaporation, their applicability is limited. The wide ranging papers by Dillon[2] and by Stratta, Dillon, and Semp[3] contain elaborate computer simulations of evaporation behavior and claim verification for typical ternary systems, but reveal data for only a few binary systems. In a further elaboration, Eaton and Willeboordse[4] use diffusion theory to account for evaporation through boundary layers, but treat only binary blends and single solvents. On the other hand, an earlier study[1] is replete with data, but is confined to a few ternary systems.

This paper describes a practical computer method for predicting evaporation time and solvent balance at any humidity during evaporation of aqueous blends containing any number of water-soluble organic solvents. It also predicts evaporation time and solvent balance for conventional organic solvent blends, but as with other solvent blend calculation methods, it ignores humidity. Calculations are performed by a FORTRAN program using data drawn from a computer file of pure solvent properties.

Calculation Method

To help coatings technologists select practical organic solvent replacement blends, Walsham and Edwards[5] had developed a useful computer program for predicting solvent blend evaporation time and solvent balance. The program calculates evaporation at 5% increments using activity coefficients computed from the solvent concen-

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Table 1—Evaporation Temperatures of Solvents
(from filter paper by ASTM D3539 in Shell Evaporometer with instrument set to 25.0° C)

Solvent	T_{min} °C
Water	15.6
Acetone	0.0
Methyl ethyl ketone	10.0
Ethanol	12.2
Isopropanol	14.7
Toluene	18.4
sec-Butanol	18.6
Methyl isobutyl ketone	20.0
n-Butanol	20.8
2-Ethoxyethanol	21.3
n-Butyl acetate	22.0
Xylene	22.5

trations by the method of Van Laar.[6] Although the method is successful with organic solvent blends, attempts to apply it to aqueous blends have failed. The method described in this paper retains some of the features of that program but overcomes the water failure by (1) using a different system to calculate activity coefficients and (2) allowing for evaporative cooling of solvent. The calculations apply to homogeneous solutions. If phase separation occurs, the predictions may not be valid.

As in the method of Walsham and Edwards,[5] the course of solvent evaporation is calculated by a repetitive procedure. The first step calculates the time to evaporate a small portion of the blend at a constant rate determined by the initial composition. At the end of this step, a new composition is computed and the process repeated using this new composition to calculate a new rate. At each step, the evaporation rate is calculated by adding up the rates of the individual components at their concentrations in the blend. The method takes into consideration the effect of evaporative cooling on evaporation rate, and adjusts the rates of the individual components according to the actual temperature of the blend as it evaporates. Activity coefficients are calculated at the actual evaporation temperature by the UNIFAC[7-9] group method. Humidity is accommodated by applying a correction factor to the water evaporation rate. It is assumed that diffusion of each component in the liquid-vapor boundary layer and in the vapor phase is not affected by diffusion of other components.

These concepts are incorporated in the following equation which computes the total mass rate of evaporation at each step as the sum of the evaporation contributions of each component:

$$r(t) = \sum_{i=i}^{n} x_i(t)\,\gamma_i\,(t, T_{sol})\,r_i^0\,(T_{sol}/T_{min_i})^{1/2} \qquad (1)$$

where $r(t)$ = total rate of evaporation from solution at time t, in grams/sec of the n components

$x_i(t)$ = mole fraction of component i in the blend at time t

$\gamma_i(t, T_{sol})$ = activity coefficient of component i at time t at the actual temperature of the blend during evaporation

T_{sol} = actual temperature of the evaporating blend

r_i^0 = rate of evaporation (g/s) of pure i, at its actual evaporation temperature (T_{min}) as a pure component in the evaporation instrument set to its standard temperature of 25°C, determined from the time required to evaporate 90% (wt) of a standard sample, assuming a linear evaporation rate to 90%.

The term $(T_{sol}/T_{min})^{1/2}$ is a temperature correction[12] to r_i^0 to account for the fact that each solvent is evaporating at solution temperature (T_{sol}) rather than at the temperature at which it evaporates as a pure solvent (T_{min}). This is discussed more fully in a later section.

Solvent blend evaporation times are useful in gauging drying times of formulated coatings, and in assessing effects of changes in solvent blend composition. The pure solvent 90% evaporation times employed in our computer program are published values[10] determined for evaporations from filter paper in the Shell Automatic Thin Film Evaporometer[11] at an instrument temperature of 25°C, using evaporation method ASTM D3539. The computer calculations predict the results of testing solvent blends in the instrument under those conditions. For aqueous blends, the predictions apply to tests performed at the specified humidity.

Correction for Relative Humidity

For aqueous solvent blends, the water evaporation rate is modified by a linear factor which takes into account the effect of relative humidity on the driving force. The correction is applied to water only because it alone is directly affected by humidity. What may appear to be a humidity effect on water-soluble solvents is merely a dilution effect relating to the concentrations and activity coefficients in aqueous solution: High humidity, which slows down water evaporation, will indirectly slow down cosolvent evaporation because it will prolong the time that the water diluent remains in solution.

For the water component of the blend, the evaporation rate term in equation (1) is multiplied by the linear humidity correction factor (1-RH/100) as follows:

$$r_{H_2O} = (1\text{-}RH/100)\; x_{H_2O}\,\gamma_{H_2O}\,(t, T_{sol})\; r_{H_2O}^0\,(T_{sol}/T_{min})^{1/2} \qquad (2)$$

where RH is relative humidity of the ambient air surrounding the evaporating blend and r_{H_2O} is the evaporation rate of water from the blend. The linear correction factor (1-RH/100) appears to be a good approximation to actual behavior.

Solution Temperature During Evaporation

The temperature correction factor for each solvent in equation (1) involves two quantities, T_{min}—the actual evaporation temperature of the pure solvent, and T_{sol}—the evaporation temperature of the solvent blend. T_{min} can be determined experimentally for each solvent and the values included in the computer data file. *Table* 1 lists T_{min} values for a few representative solvents. The cooling effect is very pronounced with the volatile solvents and is certainly significant with water and some of the more slowly evaporating solvents.

To calculate T_{sol}, a heat balance calculation is done based on the evaporative cooling of the individual solvents, i.e., on the T_{min} values. The method is not rigorous because several simplifying assumptions are made, yet it produces results that are adequate for the purpose. Among the simplifications are the assumptions that (1) heat of mixing is small compared with heat loss by evaporation, (2) heat capacities remain constant during the evaporation, and (3) solution temperature drops instantaneously to its steady state value, T_{sol}, and thereafter depends only on solution composition.

If heat of mixing is negligible, the same amount of heat is required to evaporate the blend as would be required to evaporate the same amounts of its unmixed components. This means that, in being cooled from instrument temperature to T_{sol}, the blend loses the same amount of heat as would be lost by its unmixed components if each were cooled to its own actual evaporation temperature, T_{min}. Since the heat loss of the blend is also the sum of the heat losses of its dissolved components all being cooled to the same temperature, T_{sol}, the following equation can be written equating total cooling effects of the unmixed and the dissolved solvents.

$$\Sigma n_i Cp_i (298.15 - T_{sol}) = \Sigma Cp_i (298.15 - T_{min,i}) n_i \qquad (3)$$

This assumes that, for each solvent, Cp_i, the molar heat capacity of component i at 298.15K, remains essentially constant during the small temperature drop caused by evaporation; n_i is moles of component i in solution.

Equation (3) can be rearranged to solve for T_{sol}.

$$T_{sol} = 298.15 - \frac{\Sigma Cp_i (298.15 - T_{min}) n_i}{\Sigma n_i Cp_i} \qquad (4)$$

Activity Coefficients

At each step in the incremental calculation procedure, the program computes activity coefficients at the actual evaporation temperature of the blend. Furthermore, the activity coefficient of each solvent depends on its concentration and molecular structure and also on the concentrations and structures of all the other solvents in the blend. The computations can become enormously complicated (and expensive) if an inappropriate method is chosen for assigning activity coefficients.

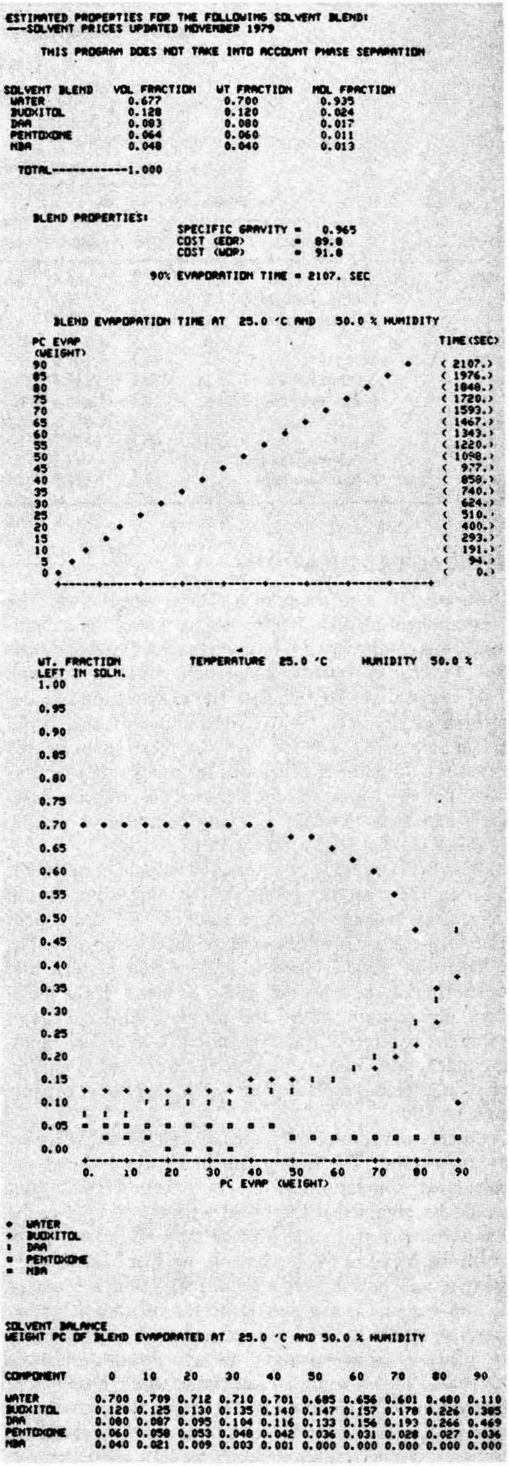
The UNIFAC method was chosen because it treats all molecules as collections of standard molecular fragments or groups for which activity calculations are readily performed for any given temperature.* For example, a molecule of the well-known solvent 2-ethoxyethanol having the structure,

$$HO{-}CH_2{-}CH_2{-}O{-}CH_2{-}CH_3$$

can be represented as the following collection of groups:

$$1\ OH,\ 2\ CH_2,\ 1\ O{-}CH_2,\ 1\ CH_3$$

With each component of a solution represented by the appropriate collection of groups, the computer uses the prescription in the UNIFAC method to calculate activity coefficients at the evaporation temperature of the blend.



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ESTIMATED PROPERTIES FOR THE FOLLOWING SOLVENT BLEND:
---SOLVENT PRICES UPDATED NOVEMBER 1979

    THIS PROGRAM DOES NOT TAKE INTO ACCOUNT PHASE SEPARATION

SOLVENT BLEND   VOL FRACTION   WT FRACTION   MOL FRACTION
 WATER             0.677          0.700         0.935
 BUOXITOL          0.128          0.120         0.024
 DAA               0.083          0.080         0.017
 PENTOXONE         0.064          0.060         0.011
 NBA               0.048          0.040         0.013

  TOTAL-----------1.000


   BLEND PROPERTIES:
                         SPECIFIC GRAVITY =   0.965
                         COST (EDR)       =  89.8
                         COST (WDP)       =  91.8

                    90% EVAPORATION TIME = 2107. SEC


     BLEND EVAPOPATION TIME AT  25.0 'C AND   50.0 % HUMIDITY

   PC EVAP                                                TIME(SEC)
   (WEIGHT)
   90                                                     ( 2107.)
   85                                                     ( 1976.)
   80                                                     ( 1848.)
   75                                                     ( 1720.)
   70                                                     ( 1593.)
   65                                                     ( 1467.)
   60                                                     ( 1343.)
   55                                                     ( 1220.)
   50                                                     ( 1098.)
   45                                                     (  977.)
   40                                                     (  858.)
   35                                                     (  740.)
   30                                                     (  624.)
   25                                                     (  510.)
   20                                                     (  400.)
   15                                                     (  293.)
   10                                                     (  191.)
    5                                                     (   94.)
    0                                                     (    0.)
     +-----+-----+-----+-----+-----+-----+-----+-----+-----+


   WT. FRACTION            TEMPERATURE  25.0 'C     HUMIDITY  50.0 %
   LEFT IN SOLN.
   1.00

   0.95

   0.90

   0.85

   0.80

   0.75

   0.70

   0.65

   0.60

   0.55

   0.50

   0.45

   0.40

   0.35

   0.30

   0.25

   0.20

   0.15

   0.10

   0.05

   0.00
       +-----+-----+-----+-----+-----+-----+-----+-----+-----+
       0.    10    20    30    40    50    60    70    80    90
                          PC EVAP (WEIGHT)

 •  WATER
 ◆  BUOXITOL
 ⁝  DAA
 ▫  PENTOXONE
 ▪  NBA


SOLVENT BALANCE
WEIGHT PC OF BLEND EVAPORATED AT  25.0 'C AND 50.0 % HUMIDITY


COMPONENT       0     10     20     30     40     50     60     70     80     90

WATER       0.700 0.709 0.712 0.710 0.701 0.685 0.656 0.601 0.480 0.110
BUOXITOL    0.120 0.125 0.130 0.135 0.140 0.147 0.157 0.178 0.226 0.385
DAA         0.080 0.087 0.095 0.104 0.116 0.133 0.156 0.193 0.266 0.469
PENTOXONE   0.060 0.058 0.053 0.048 0.042 0.036 0.031 0.028 0.027 0.036
NBA         0.040 0.021 0.009 0.003 0.001 0.000 0.000 0.000 0.000 0.000
```

Figure 1—Computer printout of calculation for 5-component blend

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*For the UNIFAC method, see Fredenslund, et. al.[7-8]; for the parameter matrix, see Skjold-Jørgenssen, et. al.[9]

Table 2—Evaporation of 3-Component Aqueous Blend

Temperature 25°C

Starting Composition, %w: 70.0 Water
15.0 2-Ethoxyethanol
15.0 2-Butoxyethanol

		Composition at Indicated Stage of Evaporation, %w							
	%w Evaporated:	20		45		70		Time to 70% Evap, sec	
RH, %	Solvents	Obs	Calc	Obs	Calc	Obs	Calc	Obs	Calc
15	Water	60.2	66.6	50.3	58.3	28.9	35.4	1050	1061
	2-Ethoxyethanol	17.5	16.2	22.4	19.4	28.5	27.9		
	2-Butoxyethanol	22.3	17.2	27.5	22.4	42.6	36.7		
40	Water	64.2	68.0	57.7	62.8	51.1	48.2	1530	1412
	2-Ethoxyethanol	16.3	15.4	18.4	16.8	19.5	21.1		
	2-Butoxyethanol	19.5	16.6	23.9	20.4	29.4	30.7		
65	Water	68.3	70.7	70.4	72.2	75.4	76.4	2605	2071
	2-Ethoxyethanol	14.2	13.8	13.4	11.8	12.2	8.2		
	2-Butoxyethanol	17.5	15.5	16.2	16.0	12.4	15.4		

TYPICAL CALCULATION

Figure 1 is a printout of a typical calculation. The 5-component blend is 70% by weight water; the remaining 30% is composed of 2-butoxyethanol, diacetone alcohol, 4-methoxy-4methyl-2pentanone,* and n-butanol. The exercise was to calculate the evaporation characteristics at 50% RH. (With five components, this would be an impossible job for even the most sophisticated extension of methods developed for predicting evaporation of binary water/solvent blends. This program, however, can accommodate as many components as any coatings technologist would wish to consider.)

Whether the data were entered by volume or by weight fraction, the computer prints out the initial composition in volume, weight, and mole fraction. All subsequent compositions are printed out in weight fraction only. The calculations include specific gravity and eastern and western prices in cents per gallon of blend. If the blend does not contain water, the program also calculates solubility parameter, fractional polarity, hydrogen bonding index, and viscosity because these are useful parameters in selecting replacements for conventional organic solvent blends.

Calculation results are presented in a form that is easy to understand. The first graph in *Figure* 1 shows the calculated simulation of the evaporation curve that would be obtained if the blend were evaporated in the Evaporometer at the specified humidity. Times to evaporate the blend in 5% increments are listed in the right margin, and, as a convenience, the 90% time is repeated at top center. The graph is useful because it shows whether or not the blend evaporates at a uniform rate. In this case, the evaporation rate is fairly uniform. If the curve had shown an initial fast rise, it would have meant that much of the blend is lost during the early part of the evaporation. This may be good for some lacquers where fast set is important, but it may be bad for other coatings and application methods where dry spray, popping, and other such plagues must be avoided.

The second graph shows solvent balance of the blend during evaporation. The operator can tell at a glance which solvents are undergoing enrichment or depletion as the blend evaporates. Since the points on the graph are

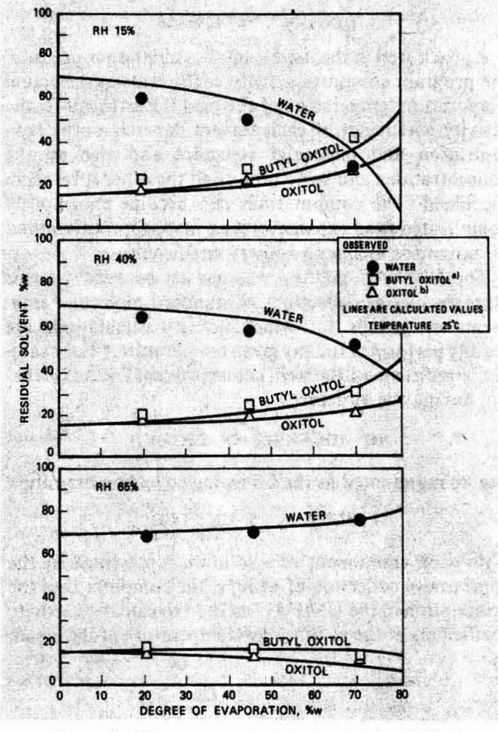

Figure 2—Three component aqueous solvent blend

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*Pentoxone® solvent. Pentoxone is a registered trademark of Shell Chemical Co.

Table 3—Evaporation of 4-Component Aqueous Blend

Temperature 25° C

Starting Composition, %w:
70.0 Water
10.0 Dimethylformamide (DMF)
10.0 2-Butoxyethanol
10.0 4-Methoxy-4-Methyl-2-Pentanone (PON)

		Composition at Indicated Stage of Evaporation, %w						Time to 70% Evap, sec	
	%w Evaporated:	20		45		70			
RH, %	Solvents	Obs	Calc	Obs	Calc	Obs	Calc	Obs	Calc
15	Water	64.7	67.6	56.0	61.3	34.2	43.1	1010	1123
	DMF	14.3	12.5	19.3	18.2	31.4	32.7		
	2-Butoxyethanol	14.0	10.8	18.1	12.6	28.2	16.9		
	PON	7.0	9.0	6.6	7.9	6.2	7.3		
40	Water	68.2	69.2	64.3	66.2	53.5	54.5	1670	1489
	DMF	13.7	12.5	18.4	18.2	27.8	32.6		
	2-Butoxyethanol	12.6	10.3	14.2	10.5	17.2	10.6		
	PON	5.5	8.0	3.1	5.1	1.5	2.4		
65	Water	73.9	72.1	74.9	74.1	71.4	66.5	2820	2376
	DMF	12.5	12.5	15.9	18.1	23.2	32.0		
	2-Butoxyethanol	10.5	9.2	8.4	6.2	5.4	1.6		
	PON	3.1	6.2	0.8	1.5	-0-	-0-		

typed by a computer terminal, their positions cannot be exact because they have to conform to typewriter spacing. Exact values are listed in the table.

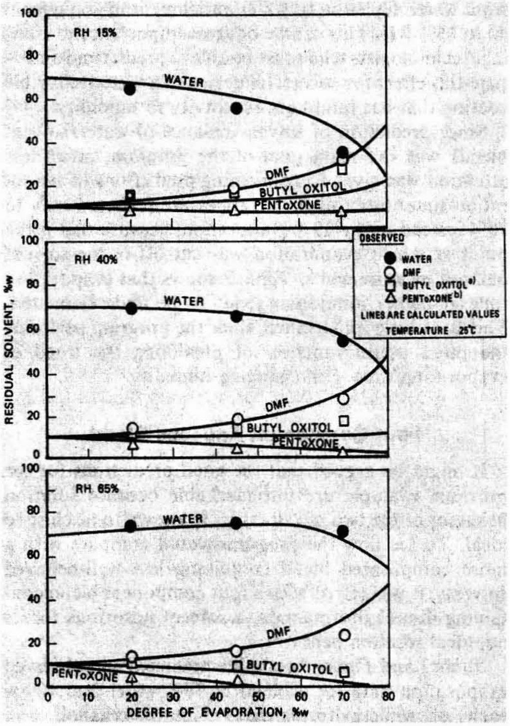


Figure 3—Four component aqueous solvent blend

EXPERIMENTAL

Evaporation measurements were made with the Evaporometer. For all measurements, the instrument was first adjusted to the standard setting of the ASTM-D3539 procedure. A 0.7 ml sample of n-butyl acetate is deposited on a 9 cm disc of filter paper suspended in the instrument at 25°C in a dry air stream set to a rate close to 21 l/min such that 90% of the sample evaporates in 468 sec. An earlier publication[1] describes humidity adjustment by mixing dry and wet air, humidity measurement, and determination of solvent concentration at different stages of evaporation. T_{min} was measured with a thermocouple placed directly in contact with the filter paper. In each case the temperature dropped almost instantaneously to the final value.

Solvents were reagent grade, usually at least 99% pure. Since water contamination has a significant effect on evaporation time, each solvent was checked for water by gas chromatography. Those with more than 0.1% water were dried over 3A molecular sieve.

RESULTS AND DISCUSSION

Three Component Aqueous Blend

Solvent balance was measured for a blend of 70%w water, 15%w 2-ethoxyethanol, and 15%w 2-butoxyethanol at three stages of evaporation at three relative humidities. *Table* 2 and *Figure* 2 compare observed and calculated concentrations of solvents in the residual blend when 20, 45, and 70% of the blend has evaporated at humidities of 15, 40, and 65%. *Table* 2 also compares the observed and calculated time to evaporate 70% of the blend.

Table 4—Solvent Balance During Evaporation of Organic Solvent Blend

Temperature 25° C

		Composition at Indicated Stage of Evaporation, %w					
	%w Evap:	0	25	50	60	70	80
Saturates	Obs[a]	30.5	30.2	28.6	23.7	17.2	9.3
	Calc		27.8	23.6	20.3	14.2	2.2
Toluene	Obs	13.2	14.8	16.2	15.1	13.2	8.3
	Calc		14.4	15.0	14.1	11.0	3.4
Xylene	Obs	8.9	10.7	14.5	16.7	19.4	19.8
	Calc		11.2	15.2	17.6	20.4	20.6
MEK	Obs	19.7	14.7	6.7	2.4	0.45	-0-
	Calc		14.7	7.3	3.5	0.3	-0-
IPA	Obs	9.7	6.4	1.7	0.3	-0-	-0-
	Calc		8.1	4.5	2.2	0.1	-0-
C Solv. AC	Obs	17.9	23.2	32.4	42.0	49.8	62.6
	Calc		23.7	34.5	42.5	54.0	73.8

(a) Evaporation from TAFID boat.

Agreement is certainly not exact and it is doubtful if it ever can be, but the calculations come satisfyingly close to predicting the main features of the evaporation behavior and of showing how these are affected by RH. The greatest deviation occurs at the lowest humidity, but even here the fit is fairly good and the calculation predicts the correct order of evaporation of the solvents and the approximate rate of depletion of water and of enrichment of the cosolvent. The fit at the higher humidities is remarkably good. It is noteworthy that the calculations successfully predict the change in evaporation behavior from water depletion to water enrichment in going from 40 to 65% RH. This can be of great importance to coatings technologists who must be able to predict and anticipate this effect for successful design of a water-reducible coating that has minimum sensitivity to humidity.

Table 5—Evaporation Time of Organic Solvent Blends

Solvent Blend Composition			Evaporation Time, sec	
Solvent	%v	%w Evap	Obs	Calc
n-Butyl acetate	10	10	15	17
Acetone	8	30	52	55
MIBK	2	50	96	97
IPA	15	70	145	147
Toluene	65	90	201	205
Isopropyl acetate	15	10	19	20
n-Butyl acetate	10	30	59	60
IPA	15	50	104	101
Toluene	60	70	151	147
		90	207	201
Acetone	20	10	9	11
Cellosolve® acetate	15	30	32	38
IPA	10	50	73	73
Toluene	12	70	141	136
Tolusol® 5	15	90	334	334
VMP-EC	22			
Xylene	6			
Shell Sol® 340 H	54	10	168	210
Butyl Oxitol®	46	30	576	633
		50	1080	1075
		70	1852	1701
		90	3100	3042

Cellosolve is a registered trademark of Union Carbide Corp.; Tolusol, Shell Sol, and Oxitol are registered trademarks of Shell Chemical Co.

Since prediction of solvent balance of water/solvent blends was the main goal of the program, much less attention was given to confirming predictions of evaporation time. For this blend, times are reported only to 70% instead of the 90% evaporation, because that is the point at which evaporation was cut off in the solvent balance measurements. *Table* 2 shows that evaporation time at higher humidities tends to be underestimated. This is of little importance, since the program performs the more useful function of predicting the trend in evaporation time with changing humidity.

Four Component Aqueous Blend

It might be argued that the good predictions for the previous example are unremarkable because solution behavior of the two glycol ethers is known to be close to ideal. To see how the program would compare with a more complicated blend containing less well-behaved solvents, it was tested with a four component blend containing dimethylformamide, a solvent notorious for its nonideal solution behavior.

Table 3 and *Figure* 3 compare predicted and observed evaporation data for a blend of 70% water and 10%w each of dimethylformamide, 2-butoxyethanol, and 4-methoxy-4-methyl-2-pentanone. The agreement both on solvent balance and on evaporation time is equally as good as for the previous example. In fact, the solvent balance prediction for the supposedly poorly behaved dimethylformamide is appreciably better than for the glycol ether.

Conventional Organic Solvent Blends

There is nothing in the program which confines it to aqueous systems. The method is equally applicable to organic blends except for the humidity feature which is not applied because that could involve consideration of phase separation. *Tables* 4 and 5 show close prediction of solvent balance and evaporation time for several typical organic blends.

APPLICATIONS

The computer program allows the coatings technologist to survey a large variety of options economically and efficiently. The following examples illustrate a few of those options.

Minimizing Effect of Humidity On Cosolvent Depletion

Cosolvents are put into a water-reducible system to do a certain job. If they are lost by evaporation before the job is done, the coating may be inferior. With the computer program, it is possible to examine evaporation of prospective formulations over a range of humidities and to optimize on compositions with minimum sensitivity to effect of humidity on cosolvent retention. Instead of performing the lengthy operation of repeating the complete solvent balance calculation over a range of humidities, it is more convenient and efficient to follow the water concentration only. For most cases, this will be adequate because the properties of a water-reducible coating tend to be more sensitive to water/solvent ratio than to the makeup of the organic solvent portion of the blend.

Figure 4 shows the effect of composition on the humidity sensitivity of water concentration in the blend at different stages of evaporation. *Figure* 4a shows the response of a typical water/solvent blend composed of 80%w water and 10%w each of butoxyethanol and sec-butanol (SBA). The dashed line shows the effect of humidity on the weight percent water remaining in the blend after 25% of the blend has evaporated. For example, if the blend is evaporated at 40% RH, point A shows that when 25% has evaporated, the water concentration in the remaining liquid is 90.5%w. If the evaporation is done at 70% RH, point B shows 93.0% water in the remaining liquid. Not surprisingly, residual water increases with increasing humidity. The broken line shows

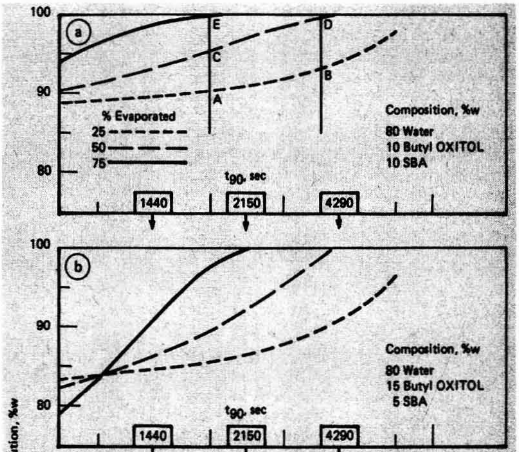


Figure 4—Effect of cosolvent composition on water retention at different humidities

Table 6—Residual Liquid Composition at 90% Evaporation

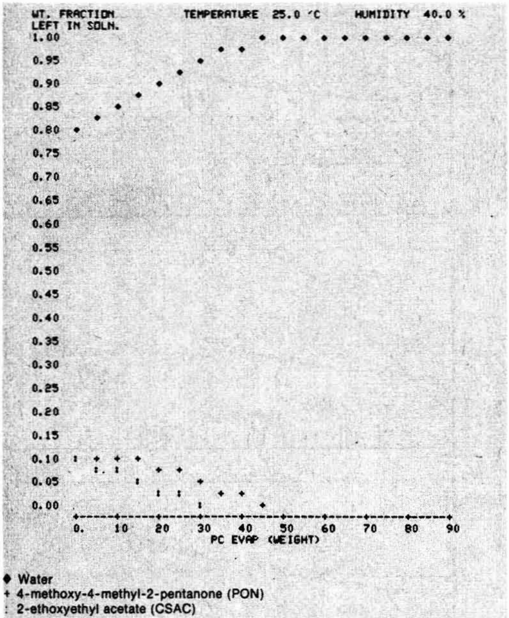
(Initial composition, %w: Water 90.0; Ethoxyethoxyethanol 5.0; Butoxyethoxyethanol 5.0)

	Solvent Concentrations, %w		
RH %	Water	Ethoxyethoxyethanol	Butoxyethoxyethanol
0	8.1	44.8	47.1
25	11.0	43.4	45.6
50	16.4	40.2	43.4
75	32.6	31.0	36.3

Table 7—Influence of Activity Coefficient on Evaporation Rate

(Composition, %w: Water 90.0; Ethoxyethoxyethanol 5.0; Butoxyethoxyethanol 5.0)

	Solvent	
	Ethoxyethoxyethanol	Butoxyethoxyethanol
t_{90}, sec.	27,840	150,000
γ in initial blend	15.1	80.0
$(\gamma/t_{90}) \times 10^4$	5.42	5.33



Solvent Balance
Weight pc of Blend Evaporated at 25.0°C and 40.0% Humidity

Component	0	10	20	30	40	50	60	70	80	90
Water	0.800	0.841	0.890	0.943	0.985	0.999	1.000	1.000	1.000	1.000
PON	0.100	0.094	0.081	0.053	0.015	0.001	0.000	0.000	0.000	0.000
CSAC	0.100	0.065	0.029	0.004	0.000	0.000	0.000	0.000	0.000	0.000

Figure 5—Competitive evaporation from an aqueous solvent blend

the water concentration at the half-way point of the evaporation. Point C shows 95.5% water at the half-way point when the blend is evaporated at 40% RH, but at 70% RH, point D shows the blend to be almost entirely water. Virtually all the organic solvent has been depleted. Point E on the solid line shows that less than 1% cosolvent remains in the blend when 75% has evapoarated at 40% RH. The line ACE shows the progressive increase in water concentration, and progressive decrease in cosolvent as the blend is evaporated at 40% RH. *Figure* 4a also lists the 90% evaporation times at 25, 50, and 75% RH.

The graph offers a guide as to how much cosolvent one can expect to find in the blend at different stages of evaporation at any given humidity. The blend is shown to lose cosolvent at all humidities. But, if the goal is to retain cosolvent, it would seem logical to increase the concentration of the slowly evaporating cosolvent, butoxyethanol, and decrease the fast evaporating solvent, SBA. *Figure* 4b shows the effect of this change. Cosolvent retention is greatly improved at the lower humidities but total depletion still occurs above 50% RH. It is interesting to note that, in this case, the change has not affected the 90% evaporation times.

An alternative approach is to replace some of the slow cosolvent with a slower one. *Figure* 4c shows the effect of modifying the blend of *Figure* 4a by replacing half of the butoxyethanol with the slower evaporating butoxyethoxyethanol. The new blend retains cosolvent at all humidities. Despite the addition of the much slower solvent, the effect on evaporation time is minimal. At the lower humidities, there is about a 6% increase in evaporation time, and at 75% RH the increase is less than 5%.

The graphs show that, for this system, cosolvent retention during evaporation is better achieved by changing to a slower cosolvent than by increasing the ratio of slow to fast cosolvent. An understanding of these effects is important where environmental controls limit the total cosolvent concentration.

Butoxyethoxyethanol has a very long evaporation time, (t_{90} = 150,000 sec). For some applications it may be preferable to use a faster evaporating solvent. An obvious choice is ethoxyethoxyethanol, which, though fairly slow (t_{90} = 27,800 sec) evaporates five times as fast. *Figure* 4d shows the effect of using it instead of the butyl glycol ether. The difference between *Figures* 4d and 4c is hardly noticeable. Both glycol ethers give blends with almost identical evaporation behavior. Although this unexpected result can be explained logically, it illustrates the power of the computer program to reveal behavior that is not obvious.

Choosing Between Two Cosolvents

The previous example showed that 5% of either of the two cosolvents, ethoxyethoxyethanol and butoxyethoxyethanol had almost the same effect on the evaporation of the blend. Their similar behavior could have been discovered more directly by calculating evaporation characteristics of a blend of 5% of each in water. Under these competitive conditions, differences between the two should show up clearly. *Table* 6 lists the solvent balance of that blend at the 90% evaporation point at four different humidities. Although the less volatile cosolvent has a bit of an edge on retention, the advantage it offers is not significant.

It can be argued that at the 5% level all high boiling cosolvents should have about the same effect, because their evaporation rates are so much slower than water that each functions as a nonvolatile tail, so that within the 0–90% evaporation range all that is being looked at is the evaporation of water. This is undoubtedly an important reason for the similarity of these two glycol ethers, but there is another factor at work that further narrows their differences. In aqueous solution, their activity coefficients are of inverse magnitude to their neat evaporation rates. *Table* 7 shows that neat ethoxyethoxyethanol evaporates about five times as fast as butoxyethoxyethanol, but its activity coefficient in aqueous solution is only one fifth as great. The effect is to equalize the evaporation rates from aqueous solvent blends. The bottom line shows the product of activity coefficient and reciprocal evaporation time. These are proportional to actual evaporation rates from aqueous solution and, as seen, are almost identical.

The technique of competitive evaporation is a useful tool in selecting one cosolvent in preference over another. It can show behavior that would not be obvious just from a consideration of evaporation times. Suppose a formulator has the option of using either 2-ethoxyethyl acetate (CSAC) or 4-methoxy-4-methyl-2-pentanone (PON) in an application where the slower evaporating solvent is favored. Based on the 90% evaporation times of the neat solvents, the winner would appear to be CSAC because its evaporation time is 2200 sec as compared with the shorter 1875 sec of PON. But in an aqueous blend, this pattern is reversed. *Figure* 5 shows the computed solvent balance for a blend of 10% each of the two competitive cosolvents in water. Of the two, PON would be the solvent of choice because it is retained for a much longer time. The same order of retention was seen at other humidities.

Using Partly Miscible Cosolvents

There is an understandable reluctance to use cosolvents that are only partly miscible with water. Though they can be solubilized with other cosolvents, there is the fear that differential evaporation with prior loss of the solubilizing cosolvent will bring on phase separation. Since it may not be obvious which order of evaporation will prevail, the fear has some justification. Yet, aside from miscibility, solvents such as n-butanol or sec-butanol may have just those characteristics that are needed for viscosity control or other film forming properties. With the computer program, solvent balance calculations can show whether or not a prospective solvent blend containing partly miscible solvents will remain within the safe homogeneous region as evaporation proceeds.

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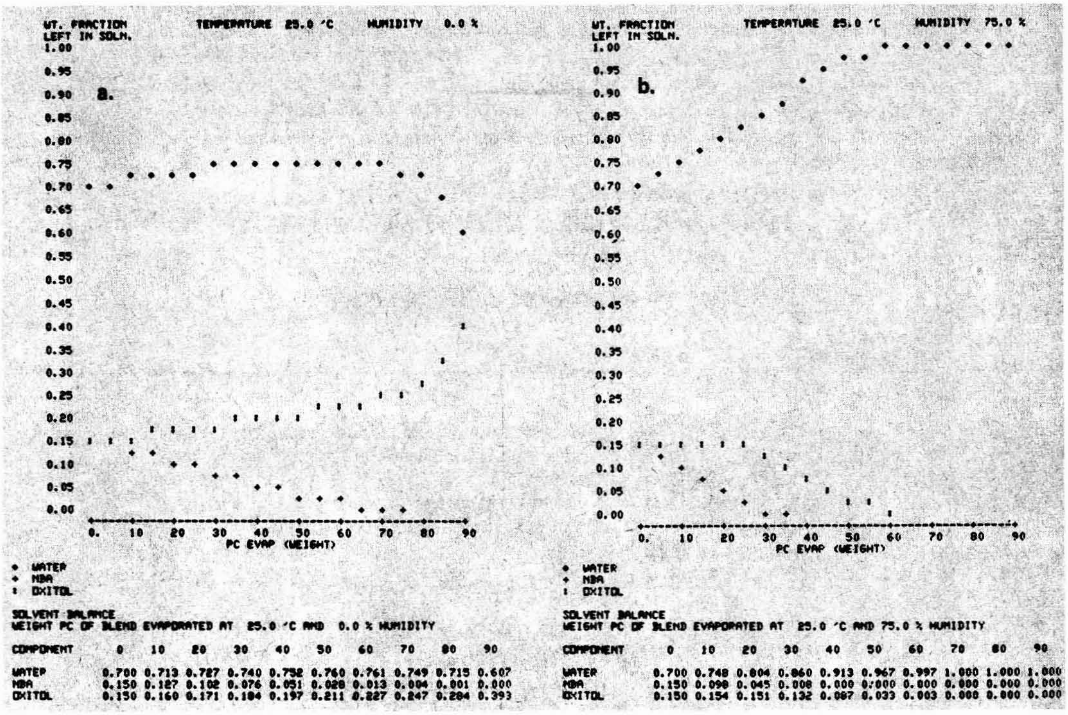
The solubility of n-butanol in water is less than 8%w, but a study on miscibility of ternary systems[13] shows that it can be raised to 15% by addition of an equal amount



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SOLVENT BALANCE
WEIGHT PC OF BLEND EVAPORATED AT  25.0 'C AND  0.0 % HUMIDITY

COMPONENT      0     10     20     30     40     50     60     70     80     90

WATER      0.700 0.713 0.727 0.740 0.752 0.760 0.761 0.749 0.715 0.607
NBA        0.150 0.127 0.102 0.076 0.051 0.028 0.013 0.004 0.001 0.000
OXITOL     0.150 0.160 0.171 0.184 0.197 0.211 0.227 0.247 0.284 0.393
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SOLVENT BALANCE
WEIGHT PC OF BLEND EVAPORATED AT  25.0 'C AND 75.0 % HUMIDITY

COMPONENT      0     10     20     30     40     50     60     70     80     90

WATER      0.700 0.748 0.804 0.860 0.913 0.967 0.997 1.000 1.000 1.000
NBA        0.150 0.098 0.045 0.008 0.000 0.000 0.000 0.000 0.000 0.000
OXITOL     0.150 0.154 0.151 0.132 0.087 0.033 0.003 0.000 0.000 0.000
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Figure 6—Evaporation of blend containing a partly miscible solvent

of the solubilizing cosolvent ethoxyethanol. Will a blend of 15% each of n-butanol and ethoxyethanol evaporate without phase separation? *Figure* 6a shows the predicted solvent balance for evaporation at zero humidity. From the very beginning, the concentration of n-butanol falls while that of ethoxyethanol rises, thereby maintaining conditions that guarantee miscibility. But what about evaporation at a higher humidity? *Figure* 6b shows that at 75% humidity the n-butanol concentration approaches zero long before any significant loss of ethoxyethanol has occurred. With this blend, n-butanol can be used at a 15% concentration without phase separation during evaporation.

Consideration of the use of partly miscible cosolvents need not be confined to ternary systems. If miscibility data are available, solvent balance can be calculated for any multicomponent system and used for predicting whether solubility limits will be exceeded during evaporation.

SUMMARY

The new calculation method does away with previous limitations confining predictions of water/solvent evaporation to binary systems only. Any number of cosolvents can be dealt with routinely at any humidity. This makes it easier to survey realistic formulations which will comply with air quality control regulations and have optimum evaporation performance and minimum sensitivity to humidity, and it can embolden the coatings technologist to consider partly miscible cosolvents.

ACKNOWLEDGMENTS

Evaporation temperatures of pure solvents were measured by Elder C. Larson. Water/solvent miscibility data were obtained by Mark F. Dante. We are indebted to Laurie S. Pipenur for writing for computer program.

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Radioisotope Tracer Technique Of Measuring Adsorption Of Paint Driers by Pigments

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A sensitive radioisotope tracer technique to measure the amount of paint drier adsorbed on paint pigments has been developed. This technique was found to be very sensitive and precise, and reliability and reproducibility of the procedure were very good. The laboratory results indicated that paint driers were adsorbed by pigments during storage, and the amount of adsorption varied among the paints.

INTRODUCTION

Corrosion and deterioration of U.S. military structures from the environment are major factors for concern in construction and maintenance. Painting is the most widely used method of protection for large surface areas because of the simplicity of application and the relatively low cost. Techniques and coatings need to be adapted to minimize deterioration and the resulting costly repairs. Excessively prolonged drying times can cause undesirable increases in downtime of an operation or premature failure of coatings.

To provide satisfactory, long-lasting protection, the coating must be able to (1) resist chemical and physical attacks from the environment, (2) maintain good adhesion with the surface for extended periods, and (3) form a hard impervious film with minimum mechanical defects.

Oleoresinous and modified oleoresinous (e.g., alkyd) paints are the most widely used coatings in the Naval Shore Establishment. An important factor which affects overall protective properties of such coatings is their loss of drying power during storage. Slow-drying films are vulnerable to chemical and physical attack from the environment during the prolonged drying process. This loss of paint drying power during storage is believed to be caused by adsorption of driers by the pigments. With this condition, the drier will not be available to the paint resin to accelerate the oxidation process necessary for curing.

Uncured films (i.e., soft) are not only easily damaged by external forces but are also more permeable to moisture or corrosive elements than cured films. Any impurities or corrosive elements trapped during the prolonged curing process will ultimately affect the overall protective properties of the films.

BACKGROUND

Absorption of paint driers by pigments during storage has long been suspected,[1-4] but verifying experimental data are lacking. No accurate and reproducible procedure for measuring small quantities of driers adsorbed by paint pigments has been developed because the amounts involved are usually too minute for accurate measurement by the usual analytical methods.

More recently, the Philadelphia Society for Coatings Technology has conducted series of drier studies[5-8] and reported that the most important parameter affecting the drying time was the type of vehicle used, followed by variation in temperature. The influence of the humidity and that of the organic radical portion of the driers were minimal. However, the Society also reported encountering puzzling results, stating that many aged finishes took longer to dry, while others dried more rapidly than they had originally. Adsorption of driers by pigments was not investigated. In fact, many facets of the loss of dry problem are still waiting for a thorough investigation.

The Civil Engineering Laboratory (CEL) undertook the task of developing a new technique for making these measurements. A radioisotope tracer technique was developed and applied to the investigation of factors that play an important role in drier adsorption on pigment surfaces. This technique was found to be very sensitive and precise, and reliability and reproducibility of the procedure were very good.

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Table 1—Coating Analysis

Paint No.	Paint Specification	Generic Type	Weight Per Gallon (lb)	Specific Gravity (gm/ml)	Viscosity (KU)	Total Solid Weight (%)	Pigment Weight (%)	Vehicle Solid Weight (%)
1	TT-E-489F	Alkyd	9.09	1.091	100	60.96	28.59	32.37
2	TT-P-105A	Oil	13.15	1.578	82	93.54	60.38	33.16
3	TT-P-81B	Oil-alkyd	10.93	1.312	78	82.29	44.51	37.78
4	TT-E-490E	Silicone-alkyd	9.79	1.175	91	61.64	32.43	29.21
5	Mil-E-699C	Phenolic-alkyd	10.39	1.247	85	70.30	42.63	27.67
6	Mil-P-16188B	Vinyl-alkyd	7.52	0.902	76	49.51	25.43	24.07

PREPARATION OF EXPERIMENTAL PAINTS

Synthesis of Paint Driers

Three soluble metal driers (cobalt, lead, and manganese naphthenates) were synthesized by reacting metallic salts (acetates) with a long-chain organic acid (naphthenic acid), as described by Gordon and Gordon.[9] This reaction is illustrated with cobalt naphthenate in the following equation:

$$Co(C_2H_3O_2)_2 \cdot 4H_2O + 2(R - COOH) \longrightarrow Co(R - COO)_2 + 2H(C_2H_3O_2) + 4H_2O \quad (1)$$

where R = naphthenic acid radical

Radioisotopically tagged driers were synthesized in exactly the same manner except that radioactive metallic salts (cobalt-60, lead-210, or manganese-54 acetates) were used in place of the nonradioactive salts as in the following equation:

$$Co^{60}(C_2H_3O_2)_2 \cdot 4H_2O + 2(R - COOH) \longrightarrow Co^{60}(R - COO)_2 + 2H(C_2H_3O_2) + 4H_2O \quad (2)$$

COBALT NAPHTHENATE: The radioactive and nonradioactive cobalt naphthenate were prepared in the same manner. Thirty grams of naphthenic acid were weighed in a 500-cc beaker and heated to 200°C. Added slowly were 12.5 g of cobalt acetate, allowing the reaction to subside between the addition and agitation. After the cobalt acetate was added, the temperature of the solution was kept at 200°C until the evolution of acetic acid and water was completed. The final product was diluted with 33 g of mineral spirits while stirring. The resulting cobalt metal concentration in the drier was 5.6% by weight.

LEAD NAPHTHENATE AND MANGANESE NAPHTHENATE: These driers were prepared in the same manner. There were, however, differences in the amounts of acetates and mineral spirits used. Nineteen grams each of lead acetate and manganese acetate were added to the respective drier

Table 2—Concentration of Radioactive and Nonradioactive Paint Driers in Experimental Paints

Paint No.	Paint Specification	Paint (gm)	Drier (gm)	Drier (% by wt)	Paint (gm)	Drier (gm)	Drier (% by wt)
		Co-60 Drier			Co Drier		
1	TT-E-489F	257.276	0.815	0.32	234.250	0.588	0.25
2	TT-P-105A	360.900	1.019	0.28	342.700	0.923	0.27
3	TT-P-81B	293.150	0.822	0.28	309.125	0.819	0.26
4	TT-E-490E	275.850	0.803	0.29	277.133	0.778	0.28
5	Mil-E-699C	294.050	0.824	0.28	275.300	0.706	0.26
6	Mil-P-16188B	248.725	0.714	0.29	233.750	0.839	0.36
		Pb-210 Drier			Pb Drier		
1	TT-E-489F	267.300	2.335	0.91	241.755	1.777	0.74
2	TT-P-105A	365.850	3.308	0.90	329.834	2.323	0.70
3	TT-P-81B	306.600	2.799	0.91	303.509	2.265	0.75
4	Mil-E-490E	265.725	2.420	0.91	277.500	1.980	0.71
5	Mil-E-699C	297.575	2.718	0.91	281.975	2.105	0.75
6	Mil-P-16188B	242.450	2.273	0.94	232.250	1.645	0.71
		Mn-54 Drier			Mn Drier		
1	TT-E-489F	257.275	0.258	0.10	251.775	0.334	0.13
2	TT-P-105A	362.525	0.363	0.10	335.125	0.373	0.11
3	TT-P-81B	300.300	0.308	0.10	301.275	0.278	0.09
4	TT-E-490E	270.250	0.320	0.12	263.125	0.293	0.11
5	Mil-E-699C	290.575	0.292	0.10	285.475	0.285	0.10
6	Mil-P-16188B	245.700	0.248	0.10	236.200	0.228	0.10

charges. Twenty-two grams of mineral spirits were added to each solution. The lead metal concentration in the drier was 17.71% and the manganese metal concentration was 7.09%.

Basic Paint Materials

Six different generic, government specification coatings were specially prepared by the paint manufacturers by eliminating paint driers. Details of the paints are given in Appendix A. All were analyzed by methods specified in References 10 through 12 to determine physical properties and chemical composition. The physical properties (weight per gallon, drying time, specific gravity, and consistency) and chemical properties (nonvolatile solids, total pigment, and nonvolatile vehicle) are listed in *Table* 1.

Paint Storage

Each of the synthesized paint driers was carefully added to each of the six paints and thoroughly mixed for storage and subsequent study. The amounts of driers, both radioactive and nonradioactive, added to the paints are given in *Table* 2. The paints were stored in inverted cans at 72° F (22° C) for specified periods.

Apparatus

Specialized equipment for this experiment included a centrifuge with a multi-unit head, a liquid scintillation counter, and Soxhlet extractors plus monitoring equipment.

LABORATORY PROCEDURES

Pigment Separation —Centrifuging and Extraction

Paint samples were removed from storage for analysis every three months. The pigment was separated from each paint by a centrifuge method.[10] The pigment cakes were wrapped in filter paper and placed in paper extraction thimbles.

Each thimble with a pigment cake was placed inside a Soxhlet extractor. Extraction with 100 ml of benzene was conducted for 4 hr to remove all traces of vehicle from the pigments. The assumption was that only strongly adsorbed drier remained with the pigment after this treatment. Each pigment was then removed from the thimble, oven-dried, and ground to a fine powder with a mechanical shaker.

Method of Counting Radioactivity

The amount of paint driers adsorbed on the pigment surface during storage was determined by counting radioactivity of the extracted pigments. A liquid scintillation spectrometer, considered the best system for counting soft beta rays, was used.

The scintillation solution in this study was a mixture of two fluors—PPO(2,5-diphenyloxazole) and POPOP (p-bis-[2-(5-phenyloxazolyl)]-benzene)—dissolved in toluene.

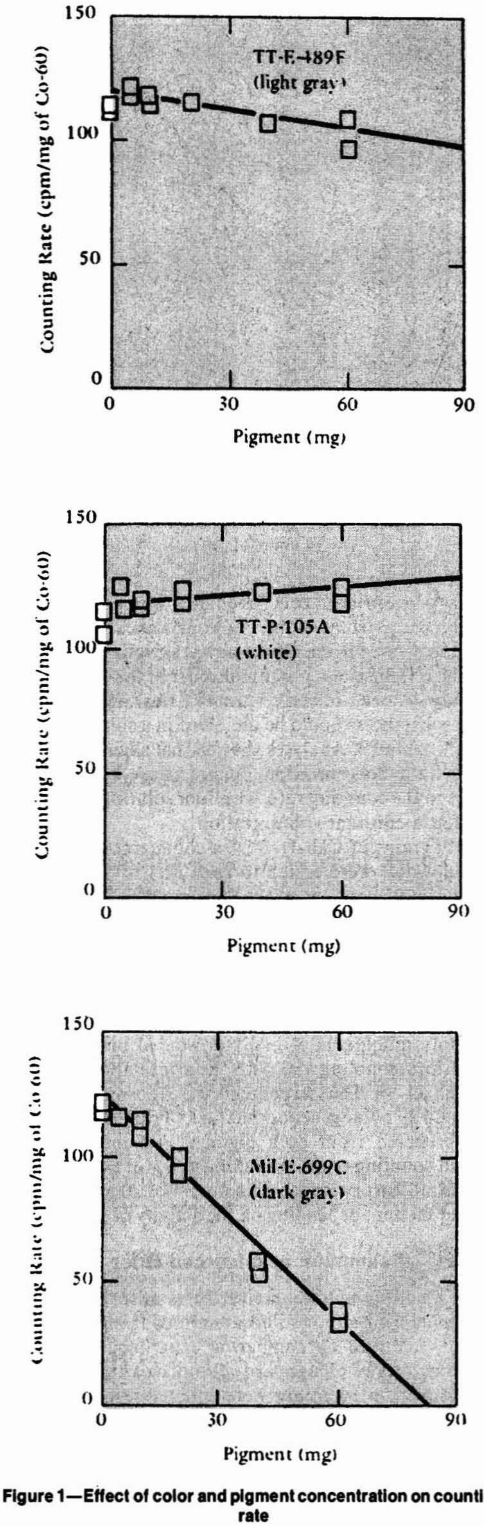

Figure 1—Effect of color and pigment concentration on counting rate

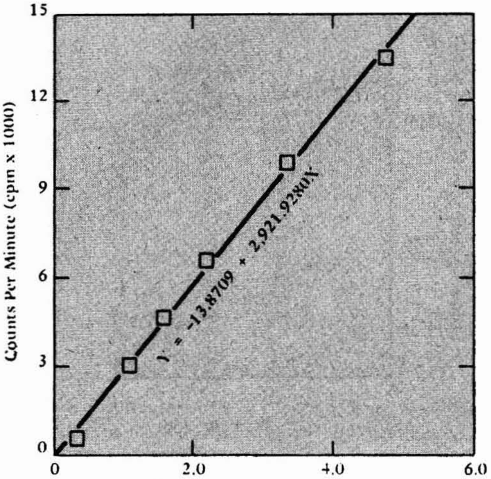

Figure 2—Regression line of sample Co-60 drier concentration vs counting rate

Fluors are fluorescent aromatic compounds that absorb energy from soluble molecules excited by particles. They re-emit energy at a wavelength compatible with the response of a photomultiplier of the scintillation counting system. For best results with this counter, radioactive substances should be dissolved in a solution of the fluor ("cocktail"). Analyses showed that slight variations of the fluor concentration do not cause a significant change in the counting rate. The fluor solution was maintained at a constant concentration.

Forty grams of Cab-O-Sil,® a commercial pyrogenic silica powder, were added to each liter of scintillation solution to suspend the ground pigments in the solution during the counting. Cab-O-Sil increases the viscosity of the "cocktail" solution to reduce the rate of settling.[13] Analyses indicated that variations of Cab-O-Sil concentration within the range tested did not significantly affect the counting rate.

Twenty milligrams of finely powdered pigment were placed in a counting vial with the scintillation solution and Cab-O-Sil. The vial contents were thoroughly mixed to obtain a homogeneous mixture. Triplicate samples were prepared from each pigment. To minimize the random counting error, the radioactivity of each vial was counted at least twice for a minimum of 20 and a maximum of 60 min, depending on the activity of the sample.

Estimation of Adsorbed Drier

The amounts of radioactive driers adsorbed on the pigments during storage were computed from regression equations derived by comparing experimental data to that from a series of standard solutions of known activities. Regression equations were updated prior to counting each sample to compensate for the loss of activity by radioactive decay of both samples and standards.

Table 3—Reliability and Reproducibility Of Sampling and Counting Method

Sample No.	Radioactivity Counting Rate (cpm) for Sample Counts — 1	2	3	4	Mean
1	357.1	352.2	353.6	344.9	351.9
2	357.3	353.7	344.4	358.3	353.4
3	351.6	346.8	353.6	347.2	349.8
Mean	355.3	350.9	350.5	350.1	351.7

Analysis-of-Variance Summary

Source of Variation	Degree-of-Freedom	Sum of Squares	Mean Square	Significant Difference?
Recounting	3	52.9125	17.6542	No
Sampling	2	26.5850	13.2925	No
Experimental error	6	179.7950	29.9658	
Total	11	259.3425		

Pigment Concentration and Color

The effect of pigment concentration and color on counting rate was determined by counting the activity of the driers mixed with varied pigment concentrations and colors. Only three pigments—white (TT-P-105A), light gray (TT-E-489F), and dark gray (MIL-E-699C)—were used (*Figure* 1). After the analyses, it was decided that all pigment samples were to be measured to a close tolerance of 20 ± 1 mg for each sampling to obtain reliable and reproducible data.

Drier Concentration Vs Counting Rate

The validity of the counting method used in this experiment was determined by counting the radioactivity in the Co-60, Pb-120, and Mn-54 naphthenate driers at various concentrations mixed with pigment from each paint sample. The resulting straight line in *Figure* 2 indicates that the counting rate is directly proportional to drier concentration. Statistical analysis showed a very highly significant relationship. From this, it was concluded that the counting method used to determine the amount of the drier was satisfactory for this experiment.

Regression equations were derived similarly for all other driers mixed with each pigment. The counting rates of all three driers were significantly affected by the presence of dark-colored pigment in comparison with those mixed with lighter colored pigment. These results showed that an appropriate regression equation for each drier and coating is required for accurate determination of adsorbed driers.

Reliability and Reproducibility

The reliability and reproducibility of the sampling and counting system were determined by counting each of the triplicate samples for 20 min (*Table* 3). No significant difference was noted among the triplicate samples nor did any significant change occur in the recounted rate. It

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Cab-O-Sil is a registered trademark of Cabot Corp.

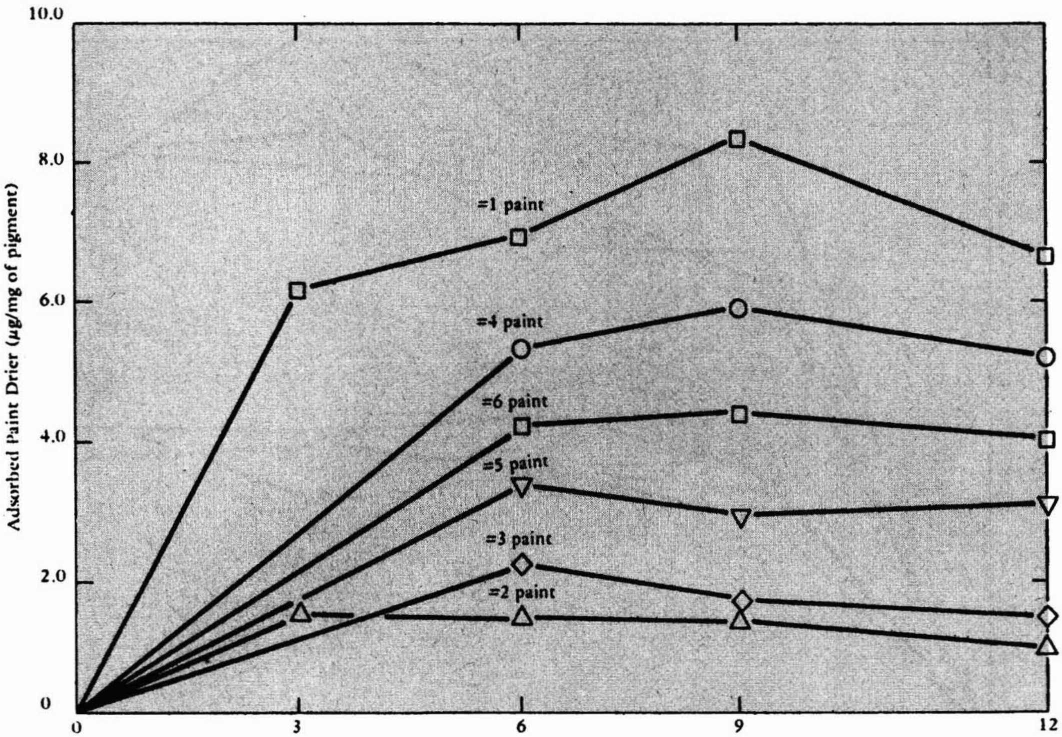

Figure 3—Adsorption of cobalt paint drier by pigments during storage

was concluded that the method of sampling and counting is reliable and reproducible and was satisfactory for use for this experiment.

LABORATORY RESULTS

The amounts of drier adsorbed by the pigments in the six radioactive experimental paints were determined by (1) separating the pigments from the paints by centrifuging and Soxhlet extraction and (2) counting their radioactivity by a liquid scintillation counter every three months during 12 months of storage.

Data for earlier than three months are not available since it was initially assumed that the adsorption is a slow process because loss of drying power of paints does not usually become apparent until after several months of storage. Unfortunately, the result indicated that the adsorption (chemisorption) took place much sooner than expected and, therefore, data for earlier stages are missing.

Appropriate regression equations were used to relate measured radioactivity to the amount of drier.

Cobalt, Lead, and Manganese Naphthenate Driers

The adsorption rate of the various driers differed from one generic type of paint to another. For the *cobalt* and *lead* driers, adsorption took place during the first three months of storage and changed only slightly thereafter. For the *manganese* drier, the pigment from phenolic-alkyd and vinyl-alkyd adsorbed the drier modestly during storage. The pigment from alkyd and silicon-alkyd completed their adsorption during the first three months of storage and the pigments from oil and oil-alkyd paints continued to adsorb the drier up to nine months, after which desorption of the drier to the three-month level was observed. The differences in the adsorption data among the paints and storage periods were not due to the standard deviation of individual measurements, but were true differences in drier adsorption.

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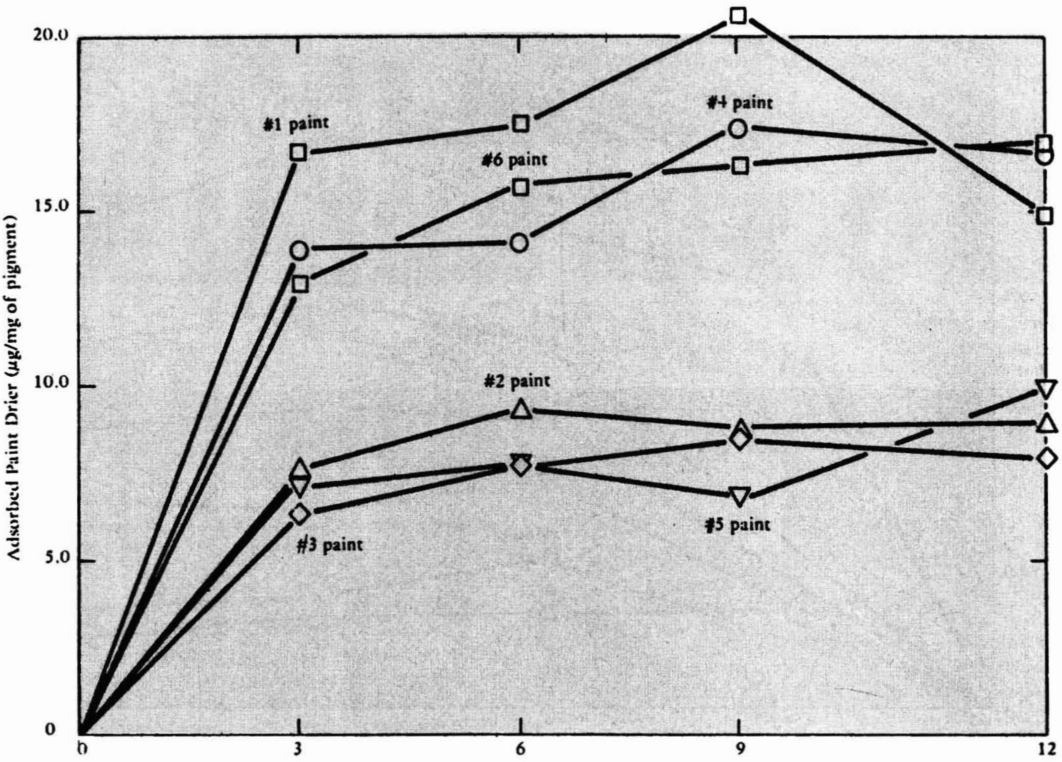

Figure 4—Adsorption of lead paint drier by pigments during storage

SUMMARY

A sensitive radioisotope tracer technique to measure the amount of paint drier adsorbed on paint pigments has been developed. Reliability and reproducibility of the procedure were very good; random error of the counting system was less than 2%.

The laboratory results clearly indicated that the amounts of the three driers adsorbed during storage varied among the paints. The pigment in the oil and oil-alkyd paints adsorbed less paint driers, and the pigments in the silicone-alkyd paint always adsorbed considerably more than other paints. The pigments in phenolic-alkyd and vinyl-alkyd paints adsorbed moderately in comparison. It appears that the type of vehicle possesses more control over adsorption of paint driers than the pigment itself.

These experimental results seem to indicate a strong sign of interaction between the generic vehicle and the type of pigment upon the adsorption of paint driers. (A series of experimental paints with two main isolated variables—vehicles and pigments—are required to determine the significance of interaction between them.)

Two types of adsorption processes, physical and chemical, take place during the storage of paints. Physical adsorption occurs where adhesional forces between adsorbate and adsorbent are relatively weak. The extent of the adsorption layer formed on the surface of the pigments by the adsorbate is dependent on the nature of the pigment-vehicle system. Physical adsorption takes place slowly and is readily reversible. Chemical adsorption (chemisorption) occurs where adhesional forces between adsorbate and adsorbent are relatively strong, and a chemical bond is formed. (This bond cannot extend beyond one molecular layer.)[14] Chemisorption takes place very rapidly and is either irreversible or reversible with great difficulty.

In this experiment, only driers strongly bonded by chemisorption were retained. Hence, *Figures* 3, 4, and 5 represent the amounts of chemically adsorbed driers. Chemisorption took place at an early stage of storage and rapidly reached its equilibrium by forming a monomolecular layer on the surface of the pigments. Earlier work[15] determined the drier content left in an aliquot part of the paint after separating the pigments. The driers were adsorbed by titanium dioxide as early as the first half hour of contact.

The limited experimental results indicate that the amount of physically adsorbed paint driers and their reaction to the surrounding factors (type of vehicle, temperature, and humidity) may have a more profound effect on drying potential of the paint than the chemisorbed drier.

Additional work will be necessary to provide detailed

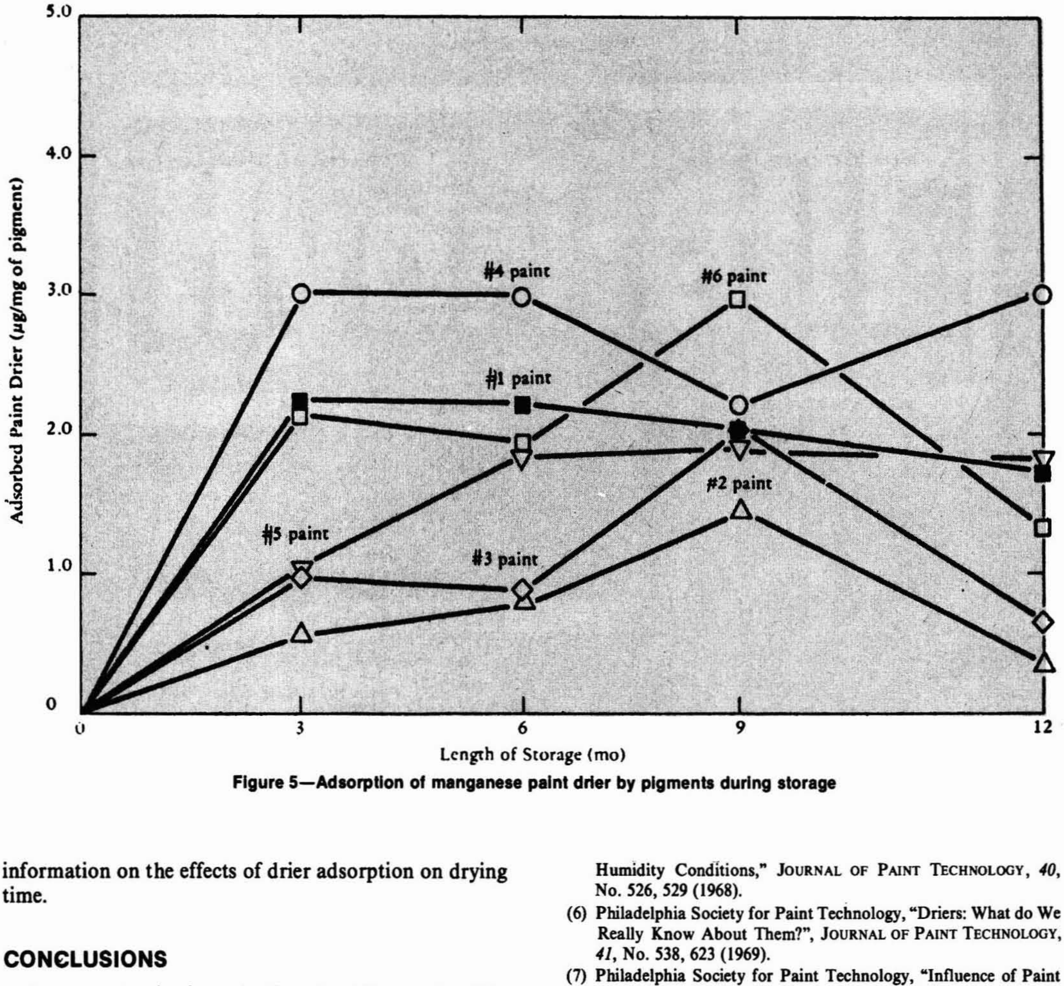

Figure 5—Adsorption of manganese paint drier by pigments during storage

information on the effects of drier adsorption on drying time.

CONCLUSIONS

A more extensive investigation should be made of the amount of drier adsorbed physically (rather than chemically) by the pigment during storage. In addition, basic research should be conducted to determine if surfaces of paint pigments can be precoated with a tightly bonded vehicle prior to introduction of drier into the paint so that no area is available on the pigment surface for adsorption of drier.

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APPENDIX A

Paint Formulations

The specifications for the six paints used in the tests follow.

Ingredients	Weight* (lb)
Paint No. 1, TT-E-489F, Light Gray	
Titanium Dioxide	250.0
Beckosol® 11-082	603.5
Bentone® #34	5.0
Alcohol	0.5
Nuact® Paste	1.5
Antiterra® U	4.0
Mineral Spirits	30.0
Hi-Flash Naphtha #114	14.5
Post-#4	4.0
Total Weight	913.0
Paint No. 2, TT-P-105A, White	
Busan® 11-M-1	280.0
Titanium Dioxide	200.0
15-15 Calcium Carbonate	320.0
Raw Linseed Oil	133.0
Z2 Bodied Linseed Oil	277.0
Mineral Spirits	123.0
Antiterra U	4.0
Total Weight	1,337.0
Paint No. 3, TT-P-81B, White	
Busan 11-M-1	250.0
Titanium Dioxide	100.0
15-15 Calcium Carbonate	250.0
Talc Hi-Fine 80	350.0
Antiterra U	2.0
Bentone #34	2.0
Isophthalic Alkyd	525.0
Q Linseed Oil	98.0
Z2 Linseed Oil	70.0
Alkali Ref-Linseed	209.0
Mineral Spirits	305.0
Total Weight	2,161.0

Ingredients	Weight* (lb)
Paint No. 4, TT-E-490E, Light Gray	
Titanium Dioxide	160.0
Talc Hi-Fine 80	206.0
Bentone #34	3.0
Alcohol	0.3
Antiterra U	4.0
McCloskey® 333-60	466.0
Hi-Flash Naphtha	36.0
Mineral Spirits	116.0
Post #4	4.0
M.C. Black	1.56
M.C. Yellow Oxide	0.38
M.C. Red Oxide	0.09
Total Weight	997.33
Paint No. 5, Mil-E-699C, Very Dark Gray	
Titanium Dioxide	36.0
Zinc Oxide	125.0
Lampblack	15.0
Magnesium Silicate	135.0
Silica, pulverized	100.0
Alkyd Resin solution	221.0
Phenolic Varnish	240.0
Petroleum Spirits	159.0
Total Weight	1,031.0
Paint No. 6, Mil-E-16188B, Dark Gray	
Titanium Dioxide	30.0
Zinc Oxide	110.0
Lampblack	2.4
Magnesium Silicate	80.0
Alkyd Resin	195.0
Vinyl Resin	75.0
Methyl Isobutyl Ketone	400.0
Xylene	40.0
Total Weight	932.4

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[*] Per batch

Beckosol is a registered trademark of Reichhold Chemicals; Bentone of NL Industries; Nuact of Tenneco Chemicals, Inc.; Antiterra of Byk-Gulden, Lomberg; Post-4 of The Baker Castor Co.; Busan of Buckman Laboratories, Inc.; and McCloskey of McCloskey Varnish Co.

Organo Silanes As Adhesion Promoters For Organic Coatings

Peter Walker
Ministry of Defence*

Organofunctional silanes of the general formula $R\text{-}Si(OR^1)_3$ have been examined as adhesion promoters for organic surface coatings on metallic substrates. Torque spanner and direct pull-off adhesion measurements clearly show that dramatic improvements in the initial, water-soaked, and recovered adhesion of epoxide and polyurethane paints can be obtained on degreased and sandblasted surfaces when silanes are used as pretreatment primers. Investigations also show that similar improvements in adhesion can be obtained when selected silanes are incorporated into a paint in the single package, self-bonding concept.

The results of X-ray photoelectron spectroscopy (XPS) examination of the sites of adhesion failure are described, and the existing theories on the role and mechanism of silanes in increasing the adhesion of coatings to the substrate are reviewed in light of the observations recorded. Finally, the selection of silanes for use with particular formulations is discussed.

INTRODUCTION

There are many references in literature to the use of reactive silane coupling agents as adhesion promoters in the technology of reinforced plastics[1-5] and adhesive bonding,[6-13] but relatively few on their use in or with organic coatings.[14-16] The patent literature is also largely silent on silanes used in conjunction with organic coatings. One patent[17] describes how the adhesion of organic polymers, e.g., polyesters, to wood may be improved by using a silane of specified general formula. The silane or its hydrolysate is suitably applied to the wood as a 0.10-10 wt % solution in ethanol containing 5% water. Another[18] relates to the treatment of inorganic substrates to enhance the bonding of organic coatings, more particularly to the adhesion of polyolefins. The silane and ethylene copolymer may be applied to the substrate separately or as a mixture. A more recent patent[19] discloses the use of compounds having an organic copolymer or terpolymer backbone with pendent hydrolizable organosilane groups to improve the gloss and weathering properties of a vinyl paint.

The literature on adhesive and reinforced plastic claims some improvement in the initial bond strength to metallic and glass substrates, but stresses the substantial increase in the wet strength of the bonds.[5] The Cleveland Society's work[16] indicates an improvement in the initial bond strength of a vinyl-based coating using N-beta-aminoethyl-aminopropyltrimethoxysilane as the adhesion promoter.

Improvements in both the initial and wet adhesion of organic coatings to metallic substrates would be of great interest to surface coating and metal finishing chemists alike, especially if improvements were to be obtained on surfaces given the minimum pretreatment, or on the more difficult metals to paint, e.g., cadmium plate and copper. Any improvement in wet adhesion would be welcomed, particularly in the light of work reported by Timmins,[20] who suggests that if adhesion is maintained, corrosion will not occur.

This present paper describes the results of a series of investigations into the use of organosilanes as adhesion promoters for epoxide and polyurethane coatings applied to aluminum, mild steel, cadmium, copper, and zinc substrates.

EXPERIMENTAL

Materials

PAINTS: Two paints were used throughout: a two-part epoxide paint pigmented with rutile titanium dioxide and

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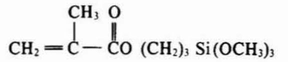
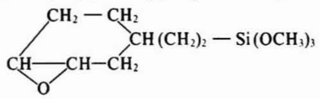
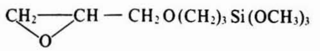
Presented by Mr. Walker at the 58th Annual Meeting of the Federation of Societies for Coatings Technology in Atlanta, GA, October 30, 1980.

*Atomic Weapons Research Establishment, Aldermaston, Reading RG7 4PR, England.

Table 1—Organosilanes

Chemical Name and Structure	Trade Name
γ Methacryloxy propltrimethylsilane  $CH_2=\overset{CH_3}{\overset{\vert}{C}}-\overset{O}{\overset{\Vert}{C}}O\ (CH_2)_3\ Si(OCH_3)_3$	A174®[a]
β-(3, 4 epoxycyclohexyl) ethyltrimethoxysilane 	A186®
γ-glycidoxypropyltrimethoxysilane 	A187®
γ-mercaptopropyltrimethoxysilane $HS(CH_2)_3\ Si(OCH_3)_3$	A189®
n-beta aminoethyl aminopropyltrimethoxysilane $H_2\ N(CH_2)_2\ NH(CH_2)_3\ Si(OCH_3)_3$	A1120®

(a) Union Carbide Corp.

cured with a polyamide resin, and a two-part aliphatic isocyanate adduct-cured polyester paint, also with rutile titanium dioxide pigmentation. The paints conformed to DTD 5555 and DTD 5580, respectively.

SILANES: Five organosilanes were examined and are presented in *Table* 1.

Substrates

The bulk of the reported work utilized aluminum and mild steel, but some additional work was carried out on cadmium plate, copper, and zinc.

Aluminum: 99.9% aluminum conforming to specification BS 1470 Grade SIC-H8

Mild Steel: BS1449, Steel Plate and strip, Part 1B CR3/FF

Cadmium Plate: Mild steel (as above) plated to DTD904A

Copper: BS 2870 C101 — ½H × 0.08 mm

Zinc: BS 849 (98.5%) × 1.00 mm

METHOD

Panel Preparation

All panels, each 150 × 100 mm (6 × 4 in.) were cut from the same batch of sheet stock and individually identified. The cadmium plating operation was carried out on individual panels, plated in one batch. Panels were degreased to the dictates of BS3900, section A3, and those selected for sandblasting were again degreased on the completion of this operation. The silanes were applied from a 2 w/w solution in a mixture of acetone and distilled water (4:1) by brush, and allowed to flash dry at ambient temperature before being rinsed in distilled water and dried at 40°C in an air-circulating oven. All paints were applied by spray as a single coat and allowed to cure for 14 days at room temperature and humidity before exposure or testing. Methods of testing are detailed in Appendix A.

EXPERIMENTAL RESULTS

Silanes Used as Pretreatment Primers

INITIAL ADHESION VALUES ON ALUMINUM: *Table* 2 shows the initial adhesion values of the polyurethane and epoxide paints on aluminum, as determined by the two separate methods. In all cases, the presence of the silane materially increased the adhesion of the polyurethane paint over that of the degreased silane-free panel when assessed by both methods. The increase in adhesion was dramatic when A174 and A1120 were present and, in

Table 2—Torque Shear and Direct Pull-Off Adhesion Values on Aluminum

		Torque Shear Adhesion		Direct Pull-Off Adhesion	
Paint	Silane/Treatment	MPa	psi/Area of Detachment	MPa	psi/Area of Detachment
Polyurethane	Degreased only	15.8	2300/100	11.4	1660/100
	A 174/Degreased	41.5	6030/0	33.1	4880/0-60
	A 186/Degreased	21.0	3050/20	18.5	2690/100
	A 187/Degreased	24.4	3540/100	17.0	2470/100
	A 189/Degreased	34.4	4990/0-80	19.8	2880/100
	A1120/Degreased	40.3	5850/0	23.2	3370/5-50
	Sandblasted only	36.7	5330/10	27.6	4000/10
Epoxide	Degreased only	27.9	4050/90	20.3	2950/50-100
	A 174/Degreased	42.2	6130/0	30.8	4470/0
	A 186/Degreased	44.2	6420/0	25.0	3630/50
	A 187/Degreased	43.9	6370/10	24.9	3610/80
	A 189/Degreased	42.2	6130/0	22.7	3300/30
	A1120/Degreased	41.5	6030/0	29.7	4310/0
	Sandblasted only	40.4	5870/5	26.3	3820/60

Table 3—Torque Shear and Direct Pull-Off Adhesion Values on Mild Steel

		Torque Shear Adhesion		Direct Pull-Off Adhesion	
Paint	Silane/Treatment	MPa	psi/Area of Detachment	MPa	psi/Area of Detachment
Polyurethane	Degreased only	22.6	3280/60	15.4	2230/10
	A 174/Degreased	38.7	5620/0-30	24.4	3550/80
	A 186/Degreased	37.7	5470/100	22.9	3330/30
	A 187/Degreased	47.1	6830/0	24.2	3520/60
	A 189/Degreased	36.4	5280/10	23.6	3430/40
	A1120/Degreased	48.0	6970/0	29.6	4300/2
	Sandblasted only	40.0	5800/10	24.9	3610/50
Epoxide	Degreased	36.2	5260/100	19.5	2830/40
	A 174/Degreased	39.7	5770/30	27.6	4000/30
	A 186/Degreased	36.2	5250/20	26.6	3860/20
	A 187/Degreased	35.7	5180/20	23.2	3370/0
	A 189/Degreased	42.8	6220/0	22.7	3290/60
	A1120/Degreased	45.0	6530/0	30.6	4400/0
	Sandblasted only	37.0	5370/0	21.8	3160/20

both cases, the measured adhesion was greater than that on the sandblasted control. A similar improvement was also apparent with the epoxide paint, with A174 and A1120 showing the greatest improvement in the direct pull-off test. The real extent of the improvement was masked by the number of failures to detach paint from the substrate; here, the values recorded were a minimum only. Once again, some of the values on the silane-treated and degreased panels were greater than those on the sandblasted panels.

Initial Adhesion Values on Mild Steel: *Table* 3 shows similar data obtained on mild steel. The adhesion of the polyurethane paint was materially improved by the presence of the silanes, and the most marked improvement occurred on the A174, A187, and A1120 treated panels, with the measured adhesion in some cases being greater than on the sandblasted panel.

The torque shear values for the epoxide paint also showed a considerable improvement for the panels treated with A189 and A1120, and all the values were close to or greater than those on the sandblasted panel. The direct pull-off values also showed an improvement on the silane-treated panels, with the greatest improvement occurring on those treated with A174, A186, and A1120. All values were in excess of those on the sandblasted panel.

These data show that there can be no doubt of the improvement in initial adhesion which can be obtained on aluminum or mild steel substrates by the use of a silane pretreatment.

Water-Soaked and Recovered Adhesion Values

It has been demonstrated that organic surface coatings show a marked loss of adhesion under water-soaked conditions, but may show some recovery of adhesion on drying.[23] Because the films dry out rapidly, the measurement of wet adhesion can be difficult. In the experiment, the coated panels were totally immersed in distilled water for 1500 hr and every effort was made to keep the panels wet during all subsequent operations. Individual panels were removed from the water, wiped

Table 4—Wet and Recovered Adhesion Values—Direct Pull-Off on Aluminum

		Control		Wet Adhesion		Recovered Adhesion	
Paint	Silane/Treatment	MPa	Area of psi/Detachment	MPa	Area of psi/Detachment	MPa	Area of psi/Detachment
Polyurethane	Degreased only	12.5	1820/100	3.8	550/100	9.9	1440/100
	A 174/Degreased	32.3	4690/30	10.1	1470/30	21.7	3160/30
	A1120/Degreased	26.3	3820/40	11.1	1610/30	22.8	3310/30
	Sandblasted only	28.6	4150/10	8.5	1230/100	13.6	1970/80
	A 174/Sandblasted	33.7	4890/0	13.0	1890/20	22.4	3250/40
	A1120/Sandblasted	34.0	4930/0	14.9	2160/20	22.0	3190/30
Epoxide	Degreased only	21.3	3100/90	5.6	820/100	11.2	1630/100
	A 174/Degreased	30.2	4380/0	12.0	1740/30	19.6	2850/50
	A1120/Degreased	31.1	4520/0	11.4	1660/30	20.5	2970/40
	Sandblasted only	28.4	4130/30	8.5	1230/100	13.7	1990/80
	A 174/Sandblasted	31.8	4610/0	13.3	1930/40	21.6	3140/50
	A1120/Sandblasted	32.6	4730/0	12.9	1880/40	24.9	3620/30

Table 5—Wet and Recovered Adhesion Values—Direct Pull-Off on Mild Steel

		Control		Wet Adhesion		Recovered Adhesion	
Paint	Silane/Treatment	MPa	Area of psi/Detachment	MPa	Area of psi/Detachment	MPa	Area of psi/Detachment
Polyurethane	Degreased only	16.7	2420/100	5.6	820/100	6.8	990/100
	A 189/Degreased	25.1	3650/30	5.4	780/100	12.1	1760/100
	A1120/Degreased	38.2	5540/0	7.4	1070/90	12.5	1820/90
	Sandblasted	25.6	3720/40	11.8	1710/95	20.8	3020/60
	A 189/Sandblasted	32.2	4680/5	23.6	3430/10	29.1	4230/0
	A1120/Sandblasted	36.6	5320/0	22.7	3300/30	29.1	4230/0
Epoxide	Degreased	19.9	2890/60	7.2	1050/100	10.9	1590/100
	A 186/Degreased	27.3	3970/20	17.3	2510/100	21.8	3160/90
	A 187/Degreased	30.0	4360/0	14.3	2070/100	15.6	2260/100
	A 189/Degreased	27.1	3930/40	10.9	1590/30	16.4	2380/100
	A1120/Degreased	32.0	4640/0	28.1	4080/100	29.2	4240/10
	Sandblasted	25.9	3760/40	9.2	1340/100	20.9	3040/100
	A 186/Sandblasted	30.4	4410/0	16.3	2360/30	31.7	4600/60
	A 187/Sandblasted	31.9	4630/0	8.3	1210/70	26.9	3910/100
	A 189/Sandblasted	31.1	4520/0	17.2	2500/10	30.0	4360/45
	A1120/Sandblasted	33.5	4870/0	26.3	3820/50	27.8	4040/40

dry, bonded with cyanoacrylate adhesive (IS 150) and cured in a chamber containing water-saturated air. Panels were removed after 2 hr and tested individually in the shortest time possible. Other specimens were allowed to dry out under laboratory conditions for 48 hr before testing.

ALUMINUM PANELS: *Table* 4 gives the direct pull-off values of those silanes showing the most promise in the initial tests. Comparison of the control values in *Table* 4 with those in *Table* 2 shows that extremely good agreement in the repeat experiments was obtained. Although not quoted in the tables, the coefficients of variation obtained were below 5%. With both paints it can be seen that there was a marked loss of adhesion under water-soaked conditions on all the panels, but that the loss on the silane-treated panels was considerably less than on the silane-free panels, both degreased and sandblasted. There was little difference between the two promoters, although A1120 was marginally better with the polyurethane, and A174 better with the epoxide. The recovered adhesion values on the silane-treated panels were considerably higher than on the silane-free panels.

MILD STEEL PANELS: In *Table* 5, the direct pull-off values obtained on mild steel are shown. The control values are similar to those shown in *Table* 4, with approximately the same coefficient of variation. The polyurethane coating on the degreased panels showed a marked loss of adhesion under water-soaked conditions, and the presence of the silanes gave little improvement. The recovery values, however, were much improved. The "wet" adhesion values were much higher on the sandblasted panels and the presence of the silanes resulted in even higher adhesion values. The recovered values were also appreciably higher.

The epoxide coating showed a marked loss of adhesion under water-soaked conditions, and the presence of the

Table 6—Initial Adhesion Values of Paints Containing Silanes—Aluminum

		Torque Shear Adhesion		Direct Pull-Off Adhesion	
Paint	% Silane/Treatment	MPa	psi/Area of Detachment	MPa	psi/Area of Detachment
Polyurethane	Degreased only	29.1	4230/100	8.8	1280/100
	0.1% A 189/Degreased	31.7	4600/80	21.8	3160/30
	0.4% A 189/Degreased	43.1	6250/40	20.8	3020/40
	1.0% A 189/Degreased	47.0	6820/30	21.4	3100/40
	0.1% A1120/Degreased	47.9	6960/0	20.5	2970/30
	0.4% A1120/Degreased	49.3	7150/0	27.8	4030/40
	1.0% A1120/Degreased	48.0	6970/0	29.2	4240/20
Epoxide	Degreased only	30.6	4440/100	31.1	4510/50
	0.1% A 189/Degreased	36.4	5280/30	30.0	4350/30
	0.4% A 189/Degreased	44.1	6400/0	43.0	6240/0
	1.0% A 189/Degreased	49.6	7200/0	43.8	6360/0
	0.1% A1120/Degreased	47.0	6820/5	30.0	4350/60
	0.4% A1120/Degreased	49.6	7200/0	32.1	4660/0
	1.0% A1120/Degreased	49.6	7200/0	31.3	4540/0

Table 7—Initial Adhesion Values of Paints Containing Silanes—Mild Steel

		Torque Shear Adhesion		Direct Pull-Off Adhesion	
Paint	% Silane/Treatment	MPa	psi/Area of Detachment	MPa	psi/Area of Detachment
Polyurethane	Degreased only	30.7	4460/60	23.8	3450/100
	0.1% A 189/Degreased	35.5	5160/50	31.2	4530/60
	0.4% A 189/Degreased	40.3	5850/20	32.5	4720/60
	1.0% A 189/Degreased	39.5	5730/90	33.3	4830/30
	0.1% A1120/Degreased	47.9	6960/20	41.2	5980/0
	0.4% A1120/Degreased	47.4	6880/80	43.1	6260/0
	1.0% A1120/Degreased	45.1	6550/20	41.9	6080/0
Epoxide	Degreased only	31.5	4570/100	22.1	3200/60
	0.1% A 189/Degreased	36.0	5230/50	27.4	3970/60
	0.4% A 189/Degreased	39.1	5680/50	29.1	4230/0
	1.0% A 189/Degreased	40.2	5820/30	31.8	4620/0
	0.1% A1120/Degreased	41.0	5960/30	23.8	3450/10
	0.4% A1120/Degreased	40.8	5920/50	28.8	4180/10
	1.0% A1120/Degreased	40.0	5800/40	30.0	4360/20

Table 8—Wet and Recovered Adhesion Values on Paints Containing Silanes—Direct Pull-Off on Aluminum

		Controls		Wet Adhesion		Recovered Adhesion	
Paint	% Silane/Treatment	MPa	psi/Area of Detachment	MPa	psi/Area of Detachment	MPa	psi/Area of Detachment
Polyurethane	Degreased only	8.8	1280/100	1.3	190/100	—	Paint peeled on drying out
	0.4% A 189/Degreased	20.6	2990/100	11.7	1700/100	13.8	2010/30
	0.2% A1120/Degreased	21.0	3060/100	9.5	1380/100	14.5	2100/100
	Sandblasted only	29.3	4260/10	10.4	1510/70	15.2	2210/40
	0.1% A1120/Sandblasted	33.6	4880/0	15.6	2260/70	25.2	3660/30
Epoxide	Degreased only	14.1	2050/100	2.2	320/100	12.9	1880/100
	0.2% A 189/Degreased	27.6	4000/0	26.3	3820/0	27.3	3960/20
	0.2% A1120/Degreased	31.5	4570/0	25.3	3680/0	26.9	3910/0
	Sandblasted only	21.0	3050/10	7.4	1070/100	22.2	3230/20
	0.2% A 189/Sandblasted	31.2	4530/0	25.1	3640/0	26.7	3870/5
	0.4% A1120/Sandblasted	28.2	4090/0	28.2	4090/0	28.7	4160/0

Table 9—Wet and Recovered Adhesion Values on Paints Containing Silanes—Direct Pull-Off on Mild Steel

		Controls		Wet Adhesion		Recovered Adhesion	
Paint	% Silane/Treatment	MPa	psi/Area of Detachment	MPa	psi/Area of Detachment	MPa	psi/Area of Detachment
Polyurethane	Degreased only	10.1	1470/100	5.1	740/100	3.2	460/100
	0.4% A 189/Degreased	20.8	3020/40	7.8	1130/100	12.8	1860/100
	0.4% A1120/Degreased	27.8	4030/40	9.8	1420/60	20.2	2930/100
	Sandblasted only	28.6	4150/20	17.8	2590/50	22.5	3260/40
	0.4% A 189/Sandblasted	36.3	5270/0	20.9	3040/30	24.7	3580/20
	0.4% A1120/Sandblasted	36.0	5230/0	21.3	3090/20	29.8	4320/0
Epoxide	Degreased only	22.1	3200/40	14.7	2130/70	19.1	2770/50
	0.4% A 189/Degreased	29.1	4230/0	24.1	3500/0	29.7	4130/0
	0.2% A1120/Degreased	31.4	4560/20	17.7	2570/10	26.6	3860/20
	Sandblasted only	28.7	4160/10	19.1	2770/40	22.9	3330/100
	0.1% A1120/Sandblasted	35.7	5180/0	21.6	3130/10	27.5	3990/40
	0.2% A1120/Sandblasted	34.8	5050/0	23.8	3450/0	25.3	3680/20

silane resulted in appreciably higher "wet" adhesion values. The greatest improvement occurred on the degreased panels treated with A186 and A1120, and with A186, A189, and A1120 on the sandblasted panels. In every case, the recovered adhesion values on the silane-treated panels were greater than on the silane-free panels.

Undoubtedly, the presence of the silane adhesion promoter improved both the "wet" and "recovered" adhesion of both paints on aluminum and mild steel to a considerable extent.

Silanes Incorporated Into Paints

To determine whether the silanes function as adhesion promoters when present in the paint, additions of A189 and A1120 were made to both the polyurethane and epoxide coating by mechanical stirring shortly before application. All subsequent tests were carried out on degreased panels only.

Effect of Concentration

ALUMINUM PANELS: The initial adhesion values obtained by both the torque and direct pull-off methods are shown in *Table* 6. It is apparent that the A189 and A1120 were effective in both the polyurethane and epoxide paints and major improvements in adhesion were observed with both the torque spanner and direct pull-off tests. The complete extent of this improvement was masked by an appreciable number of cases in which no detachment of the film was observed. In general, A1120 appeared to be effective at lower concentrations than A189.

MILD STEEL PANELS: In *Table* 7, the values recorded on mild steel are given. Again, both silanes were effective in increasing the measured adhesion, and A1120 gave higher values at lower concentrations. In no case was there any apparent advantage in using an addition at more than 0.4% by weight, and the A1120 appeared to be effective at 0.1% by weight.

WATER-SOAKED AND RECOVERED ADHESION VALUES ON PAINTS CONTAINING SILANES

These experiments were performed in the manner described earlier and the direct pull-off values are shown in *Tables* 8 and 9.

Aluminum Panels

Table 8 shows that the initial results were very similar to those in *Table* 6 and they indicate a real improvement in adhesion. After water-soaking, the measured adhesion of all the polyurethane systems fell, but the retained adhesion of the paints containing silane was much higher. After drying out, the degreased silane-free panel showed no recovery and, in fact, failed completely in adhesion. The remainder of the recovered adhesion values were appreciably higher than the water-soaked values.

The wet adhesion of the epoxide paints was markedly greater for the silane-containing paint on both the degreased and sandblasted panels and there was little to choose between the two silanes. On drying out, the recovered adhesion of the silane-containing paints was greater than the silane-free systems.

Mild Steel Panels

Table 9 gives the direct pull-off adhesion values on mild steel. The initial values of all the silane-treated paints were markedly greater than the silane-free paints. The water-soaked values were also higher in every case, although the differences were not as great as on the aluminum panels. The recovered adhesion was greater on all panels, except the degreased silane-free polyurethane panels, which showed an apparent fall in adhesion. In this respect, it showed the same behavior as the aluminum panel. In each case the recovered adhesion of the silane-treated paints was greater than the silane-free paints.

If the results shown in *Tables* 3 and 4 (silane as a pretreatment) are compared, where possible, with those in *Tables* 8 and 9 (silane incorporated into the paint) it seems that, on the aluminum panels, there is a marginal advantage in incorporating silane into the paint. On mild steel, however, the values are so close that neither method seems to offer any major advantage. Certainly, both sets of results illustrate marked improvements to be gained in "wet" and "recovered" adhesion by the use of silanes.

POSSIBLE DIFFICULTIES ASSOCIATED WITH THE ONE-PART CONCEPT

The obvious advantages of the single part self-bonding concept, in which the silane is incorporated into

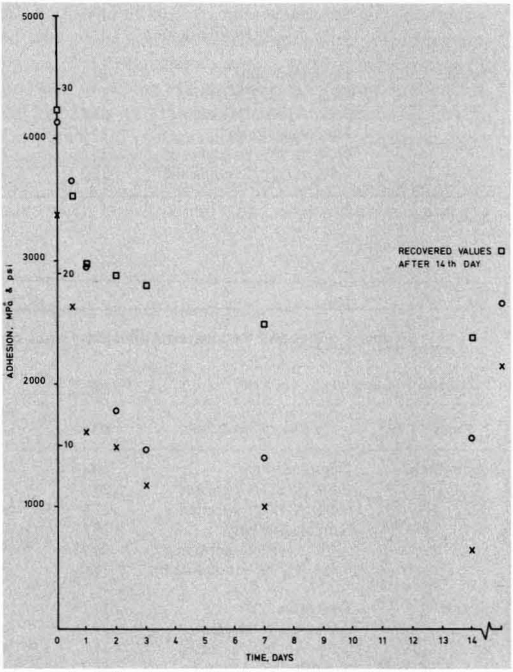


Figure 1—Rate of loss of adhesion of epoxide paints (water immersion). X—nonsilane control; ○—A1120 on surface; □—A189 in paint

Table 10—Results of Long-Term Storage Tests on Paints Containing Silanes (Degreased Aluminum—Torque Spanner Test)

		Initial Adhesion		3 Months Storage		6 Months Storage		9 Months Storage	
Paint	Drying Agent/Silane	MPa	psi/Area of Detachment	MPa	psi/Area of Detachment	MPa	psi/Area of Detachment	MPa	psi/Area of Detachment
Epoxide	None/None	24.1	3500/10	22.4	3250/50	23.2	3360/80	23.8	3450/50
	1% TL/A 174	30.7	4450/5	37.1	5380/30	36.4	5280/30	33.8	4900/30
	2% TL/OP/A 174	32.9	4770/0	34.1	4950/30	32.5	4720/40	35.1	5100/50
	2% TL/A 174	30.7	4450/10	30.5	4430/80	33.2	4820/80	32.7	4750/50
Polyurethane	None/None	11.8	1710/100	13.6	1980/90	15.1	2190/90	24.1	3500/50
	1% TL/A1890	26.7	3880/50	31.3	4550/30	32.2	4680/30	40.0	5800/70
	2% TL/OP/A189	26.0	3770/80	31.3	4550/40	30.5	4430/30	39.8	5770/60
	2% TL/A189	25.2	3660/60	32.0	4650/90	34.1	4950/80	40.0	5800/60

a paint during the manufacturing stage, are evident. However, there are possible disadvantages in the approach concerning the effects of short or long term storage. The silanes are readily hydrolyzed—reaction (1)—

$$\mathrm{R\,Si\,X_3 + 3H_2O \xrightarrow[\text{catalyst}]{\text{pH}} R-Si(OH)_3 + 3\,HX} \qquad (1)$$

HX = (usually an alcohol or acid)

and, almost immediately, silane triols begin to condense with each other to form higher molecular weight oligomers which are complex silanols. Generally, it is desirable to retain the silane as monomer or dimer to retain coupling agent activity.[24] Thus, the silanes are likely to lose their ability to act as adhesion promoters as they age in the presence of water. The presence of water in the paint is a distinct possibility which may arise from commercial solvents and pigments. Thus, if silane-containing paints are to maintain their improved adhesion characteristics during storage, the paint must be "dry," either "dry" components must be used or a water scavenger must be incorporated.

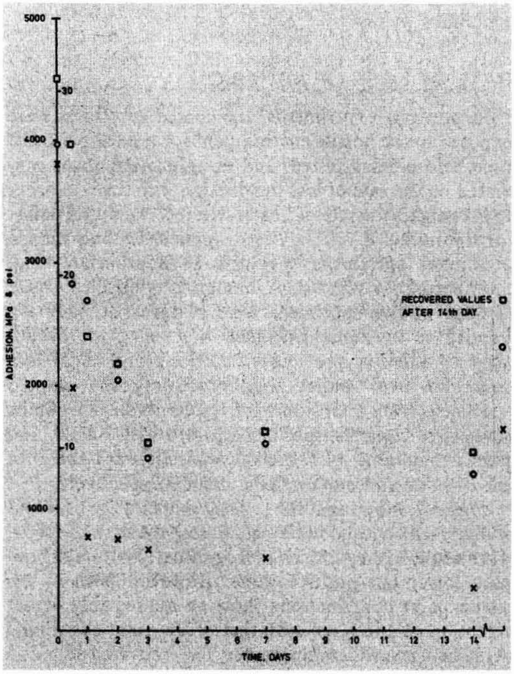
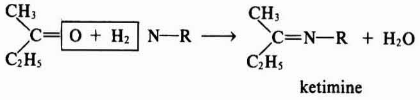


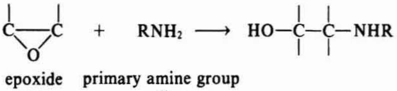
Figure 2—Rate of loss of adhesion of polyurethane paints (water immersion). X—nonsilane control; ○—A1120 on surface; □—A189 in paint

A second potential source of instability is a possible solvent-silane interaction. Amino-silanes are known to be reactive with oxygenated solvents such as ketones, and these are commonly used in polyurethane and epoxide paint formulations. For example, γ-aminopropyltrimethoxy silane will react with methyl ethyl ketone to form the ketimine plus highly undesirable water, as in reaction (2):



$$\mathrm{(CH_3)(C_2H_5)C{=}\boxed{O + H_2}\,N{-}R \longrightarrow (CH_3)(C_2H_5)C{=}N{-}R + H_2O} \qquad (2)$$

A third source of instability is the possible reaction of silanes with the polymeric components of the paint, either base resin or curing agent. An example of this is given in reaction (3):



$$\mathrm{C(-O-)C + RNH_2 \longrightarrow HO{-}C{-}C{-}NHR} \qquad (3)$$

This may not prove to be an intractable problem as the silane may be added to either component of a two-part system. A further possibility is reaction with, or adsorption on, the pigment particles.[24]

In order to pursue the self-bonding concept, considerable care will be necessary to eliminate water and to select a silane which will not react to the detriment of its coupling properties with any component of the paint.

LONG TERM STORAGE OF PAINTS CONTAINING SILANE

To examine the possibility of formulating paints containing silanes which would be stable for long periods, A189 was added to the pigmented portion of the epoxide

Table 11—Direct Pull-Off Adhesion Values on Cadmium, Copper, and Zinc

	Controls		After 1000 hr Accelerated Weathering	
Paint/Silane/Substrate	MPa	psi/Area of Detachment	MPa	psi/Area of Detachment
Polyurethane/None/Cadmium	23.4	3400/60	18.5	2680/60
Polyurethane/A174/Cadmium	29.8	4320/0	24.1	3500/5
Polyurethane/A189/Cadmium	29.1	4220/0	30.0	4360/0
Polyurethane/A1120/Cadmium	29.1	4220/0	16.9	2450/30
Epoxide/None/Cadmium	25.1	3640/30	17.4	2520/40
Epoxide/A174/Cadmium	29.1	4220/0	25.0	3630/10
Epoxide/A189/Cadmium	28.2	4090/5	29.8	4320/0
Epoxide/A1120/Cadmium	28.2	4090/0	30.4	4410/0
Polyurethane/None/Copper	27.9	4050/20	28.4	4120/20
Polyurethane/A174/Copper	29.7	4310/0	33.9	4920/0
Polyurethane/A189/Copper	29.3	4260/10	34.5	5000/0
Polyurethane/A1120/Copper	29.7	4310/0	34.3	4980/0
Epoxide/None/Copper	27.3	3970/30	20.2	2930/20
Epoxide/A174/Copper	27.4	3980/30	21.9	3180/30
Epoxide/A189/Copper	29.7	4310/0	23.8	3450/30
Epoxide/A1120/Copper	29.4	4270/0	20.7	3000/10
Polyurethane/None/Zinc	29.5	4280/50	14.6	2120/40
Polyurethane/A174/Zinc	22.3	3240/50	13.4	1950/50
Polyurethane/A189/Zinc	27.1	3940/20	30.4	4410/0
Polyurethane/A1120/Zinc	28.7	4170/80	13.2	1920/90
Epoxide/None/Zinc	20.5	2970/80	13.4	1950/30
Epoxide/A174/Zinc	20.5	2980/70	11.6	1690/80
Epoxide/A189/Zinc	31.0	4500/0	29.1	4230/0
Epoxide/A1120/Zinc	23.1	3360/60	23.1	3360/10

and A174 to the polyurethane paint, both of which had been previously reacted with a mono-functional isocyanate (Additive TL from Bayer UK Ltd.) and an ethyl ester of a o-formic acid (Additive OF from Bayer UK Ltd.) used singly and in combination (TL at 1 and 2% by weight on the paint and the TL/OP at a 2:1 ratio at 1%). Samples of the paints were tested after 3, 6, and 9 months storage.

The adhesion of both paints when applied to degreased aluminum panels and measured by the torque spanner method after 14 days cure at room temperature and humidity is shown in *Table* 10. In the case of the epoxide formulation there was, quite clearly, no change in the adhesion characteristics of any of the four paints during storage and all three of the paints containing silane had improved adhesion, which was maintained throughout the test period (9 months). With the polyurethane formulations, there appears to be a consistent improvement in adhesion of the paints throughout the storage period. A real improvement in adhesion with time seems highly unlikely, as a detailed examination of *Table* 10 shows that the control paint, as well as the silane-containing paints, exhibited an apparent increase in adhesion. The only explanation is that there was an improvement in the panel degreasing operation rather than any improvement in the paint adhesion. It should be remembered that the polyurethane paint was shown to be less tolerant of surface preparation than the epoxide (*Tables* 3–4). Certainly there is no evidence of a loss of adhesive properties during storage.

The results obtained indicate that certain silane/paint combinations retain their improved adhesion characteristics over a period of at least nine months, and the possibility therefore exists of producing stable silane-containing paints.

RATE OF LOSS OF ADHESION

In previous experiments, the coated substrates were exposed to the hostile environment for a fixed period of time before being tested, and no information was generated on the rate of loss in adhesion with time. In order to investigate the rate of loss, a selected silane, and paints incorporating a silane, were applied to bolt-shaped aluminum specimens,[23] coated as appropriate and, after curing for seven days, immersed in distilled water. Groups of five specimens were removed at intervals, bonded to a similar uncoated bolt using the cyanoacrylate adhesive previously described, and cured in a container over water; thus preventing the painted specimens from drying out. The results obtained from the silane-free controls, specimens coated with A1120, and paints containing A189 are shown in *Figures* 1 and 2 for the epoxide and polyurethane paints, respectively.

Figure 1 shows that the general shape of the curves is similar, with a rapid initial loss during the first 12–24 hr. This is followed by a much more gradual loss, leveling out at a lower but positive value. However, both of the silane/paint combinations show an appreciably higher retention of adhesion, with that of the paint containing A189 being considerably greater. The recovered adhesion values after 14 days are also much higher.

In *Figure* 2 it can be seen that the basic shape of the curves is similar to those in *Figure* 1, with a pronounced loss of adhesion during the first 12–24 hr and a gradual leveling off over the remainder of the immersion period. The loss was particularly rapid in the case of the silane-free control. There was little difference between the A1120 on the surface and the A189 in the paint. Again, the recovered adhesion values of the silane-treated specimens were appreciably higher.

EXPERIMENTS ON CADMIUM, COPPER, AND ZINC SUBSTRATES

A limited experiment in which A174, A189, and A1120 were applied as a pretreatment to cadmium plate, copper, and zinc substrates and exposed to an accelerated weathering cycle to BS3900 Method F3, confirmed the value of silanes on substrates other than aluminum and steel.

Accelerated weathering was chosen as the aging environment since it was considered that reaction of contaminants and moisture with the substrate is a major factor in the loss of adhesion to these metals. The results obtained are shown in *Table* 11.

From *Table* 11 it can be seen that both the initial values and those measured after recovery from the accelerated weathering cycle showed that the adhesion on all three substrates was materially improved by the use of a silane. Of the three silanes tested, A189 was the most effective all-round adhesion promoter for both the epoxide and polyurethane paints on all three substrates. In general, the greatest change in adhesion occurred on the zinc substrate and the least on the copper.

The finding that A189 was superior to the other silanes in retaining adhesion is at variance with the findings on aluminum and steel, on which A174 and A1120 appeared to be most effective. This suggests that the silanes may show some degree of specificity for metals, as well as for the polymeric binder.

ANALYSIS OF DATA

The results quoted are the average of four separate determinations for the direct pull-off method, and five for the torque spanner. Although not quoted for individual results, the coefficients of variation were, in general, below 7%, except where recovered adhesion was determined. Then coefficients rose to as high as 12% in some cases. A partial statistical analysis of the results given in *Tables* 3–5 showed that the difference between the silane-free and silane-treated panels is highly significant. In general, the difference in values for the panels was large and the standard deviations low, so that detailed statistical analyses were unnecessary. A major difficulty in carrying out a full statistical analysis is the failure in many cases to detach paint from the surface; thus, the values recorded do not represent the actual adhesion, but rather, a minimum value.

SITES OF FAILURE

Throughout this paper, the sites of failure have been recorded as "percentage detachment." This represents an apparent adhesive failure from the substrate. The remainder of the area may be cohesive failure within the paint film or the failure may have been associated with the adhesive used, i.e., adhesion failure from the paint film, cohesive failure within the adhesive layer, or detachment from the reverse side of the panel or the bonded-on test specimen. The percentage detachment is, then, the area on the test panel over which the paint has apparently been removed. There is an implication that the area of detachment is an actual failure in adhesion to the substrate and, for practical purposes, this may be regarded as being the case. The rationale for this is that if there is no paint present to the naked eye, for all practical purposes a failure in adhesion has occurred, since no corrosion protection will be afforded. However, from the point of view of understanding the role of silanes as adhesion promoters, a knowledge of the "true" as opposed to the "apparent" site of failure is absolutely essential.

In order to determine the true site of failure, a limited examination using X-ray Photoelectron Spectroscopy (XPS) was undertaken.

XPS Investigation

XPS is one of a group of newer analytical techniques including Auger and Raman spectroscopy, which basically measures the core binding energies in atoms and molecules. Chemical shifts occur in the core electron binding energies as the atomic electron density varies. These chemical shifts in organic compounds correlate with the charge on the atom. Thus, as the chemical nature of an atom varies its XPS peak shifts, it is possible to distinguish between different types of atom or of the same atom in the same molecule. XPS is sensitive to all atoms heavier than helium and it is particularly useful in this type of study. Because the technique primarily examines the top 20–50Å of the surface, the sensitivity is good, particularly in thin films.

The XPS spectra were run on a UG ESCA III spectrometer with a Mg(Ka) radiation and the intensities (measured as peak heights) were converted to elemental compositions.

Specimens showing an apparently clean adhesive failure were examined, and were selected from those used in the determination of the rate of failure tests. Both fracture surfaces were investigated and are described as "metal surface" and "paint surface," with the paint surface representing the underside of the film removed during the adhesion test. The work to date has been confined to samples on which A1120 was applied to the surface as a pretreatment primer.

The chemical composition of the fracture surfaces of the epoxide coated specimens is shown in *Table* 12. It should be noted that hydrogen cannot be detected by XPS and the values reported represent the atom fraction of the elements other than hydrogen present in the film. From the results in *Table* 12 several observations can be made. The most striking of these is that in no case was the aluminum substrate detected, whether in the presence or absence of silane, in water or in air, or on the metal or paint surface. Quite clearly the paint film failed cohesively, and there was no element of clean adhesive failure. A less obvious observation is that no titanium

Table 12—XPS Data on Fracture Surfaces of Epoxide Paint

		Metal Surface							Paint Surface						
System	Storage Condition	C^1	C^2	N	O^1	O^2	Si	Al	C^1	C^2	N	O^1	O^2	Si	Al
Epoxide paint (non-fracture surface	In air								0.68	—	0.11	0.16	—	0.03	—
A1120 Empirical formula		0.57	—	0.14	0.21	—	0.071	—							
Al/Epoxide	In air	0.50	0.15	0.10	0.06	0.18	—	—	0.50	0.19	0.10	0.17	—	0.010	—
Al/Epoxide	In water	0.50	0.19	0.13	0.16	—	0.008		0.51	0.17	0.12	0.17	—	0.016	—
Al/A1120/Epoxide	In air	0.51	0.19	0.11	0.17	—	0.002	—	0.51	0.18	0.11	0.18	—	0.003	—
Al/A1120/Epoxide	In water	0.50	0.20	0.12	0.18	—	0.005	—	0.52	0.17	0.12	0.18	—	0.016	—

was detected; therefore, the failure occurred within an *unpigmented* boundary layer at the paint/metal interface. As a subsidiary experiment, the fracture surfaces were ion-etched using argon and a cold cathode ion gun operating at 2 KeV whereupon aluminum was detected on the "metal" surface and titanium on the "paint" surface. Since nothing is known of the rates of removal by ion etching, no information is available on the thickness of the unpigmented layer. Bailey and Castle, however, report values of 37-46Å for escape depths[25] and it must be assumed that the layers are at least as thick as this, giving a total thickness of at least 74-92Å.

A second observation is that the chemical composition of the fracture layer is different from that of the upper exposed surface and, therefore, possibly different from the bulk composition of the polymeric binder. There is, of course, no reason why the boundary layers should be of the same composition as the bulk polymer.

Bell[26] has clearly demonstrated that the infrared spectra of fracture surfaces and the bulk paint are different. This finding is reinforced by the present work which indicates the presence in the fracture surfaces of a second carbon peak (C^2) at a higher binding energy (possibly C=C, C=O) not present in the unfractured surface. A further observation, partially obscured by the presence of silicon in the paint, is that the atom percent values for silicon are much lower than that given by the empirical formula of A1120; and it may, therefore, be argued that the failure is not within a layer of silane on the surface. The sites of failure in air and water are interesting in that although exposure to water produces a dramatic fall in the measured adhesion, the composition of the fracture surfaces is broadly similar despite differences in the silicon content.

Similar data are shown in *Table* 13 for the polyurethane paint and, in general, the observations are the same.

There was no evidence of a clean adhesion failure either in water or in air; an organic layer was present in all cases since no aluminum was detected. Again, there was no detection of titanium, indicating a break within an unpigmented layer at the metal/paint surface. Unlike the epoxide paint, the composition of the various fracture surfaces was almost identical in air and water in the absence of the silane. The second C^2 peak was again detected in these surfaces, although it was absent in the nonfracture surface. Unlike the epoxide, no silicon was detected in the paint surface either in air or water, indicating failure at a different and possibly greater depth in the paint.

The failure to detect titanium dioxide near the paint/metal interface—indicating that sedimentation had not occurred—is surprising and worthy of further investigation.

DISCUSSION

Before discussing the practical results obtained by the use of silanes, it may be beneficial to consider the existing theories on how silanes function as adhesion promoters. The mechanism by which organosilanes bond to

Table 13—XPS Data on Fracture Surfaces of Polyurethane

		Metal Surface						Paint Surface					
System	Storage Condition	C^1	C^2	N	O^1	Si	Al	C^1	C^2	N	O^1	Si	Al
Polyurethane paint (non-fracture surface)	In air							0.51	0.18	0.13	0.18	—	—
A1120 Empirical formula		0.57	—	0.14	0.21	0.071	—						
Al/Polyurethane	In air	0.52	0.18	0.12	0.18	—	—	0.51	0.18	0.13	0.18	—	—
Al/Polyurethane	In water	0.51	0.18	0.13	0.18	—	—	0.52	0.17	0.12	0.19	—	—
Al/A1120/Polyurethane	In air	0.51	0.17	0.12	0.17	0.002	—	0.51	0.17	0.12	0.17	—	—
Al/A1120/Polyurethane	In water	0.55	0.14	0.11	0.18	0.009	—	0.52	0.18	0.11	0.18	—	—

metal and glass substrates has attracted a great deal of attention. Reflecting the early technological interest in glass reinforced plastics, the major research effort has been on the mechanism of bonding to glass.

The oldest and most widely known theory is the "Chemical Bond Theory,"[27] in which it is postulated that trialkoxysilanes chemically bond to the substrate through interaction of the alkoxy group with surface silanols in the glass and also bond to the organic present by reaction of the organol functional group with reactive species contained in the organic. The strong interfacial bonds thus formed are of the order 50–100 k cal/mol.[28] The difficulties with this theory are that some silanes are effective although they are chemically nonreactive with the polymer and it does not necessarily explain the improved wet strength since metallosiloxane bonds to metal surfaces are susceptible to hydrolysis.

The "Deformable Layer Theory"[27] postulates a plastic interface providing a zone in which stresses between resin and glass may be relieved without bond rupture, thus minimizing internal stress. However, it has been suggested that the amount of silane present in a typical finish is too thin a layer to relax in the time required.

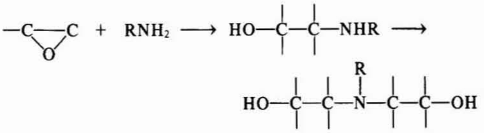
The "Surface Wettability Theory,"[27] applied initially to filled systems, argues that complete wetting of the filler particles by the resin would improve the adhesion of the polymer to the filler by physical adsorption. Although the increased adhesive strength would exceed the cohesive strength of the polymer, it is difficult to see how, in the competition with water and other weakly bonded layers, physical adsorption is likely to provide much reinforcement to a bond where chemical bonding is also present.

In the "Restrained Layer Theory"[27] it is postulated that a region of intermediate modulus is formed to transfer stress from the high modulus filler or surface, to the relatively low modulus polymer. Silanes function by "tightening up" the polymer structure at the interface while simultaneously providing silanol groups for bonding to the substrate. The need for stress relaxation due to thermal shrinkage between polymer and filler is not accounted for.[29] In effect, this theory postulates a similar reaction between polymer and substrate as the "Chemical Bond Theory" while advancing a different mechanism for its observed action.

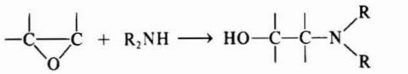
The "Reversible Hydrolytic Bond Mechanism Theory" has been proposed by several authors[27, 28, 29] and is largely a combination theory combining features of the chemical bonding theory with the rigid interface of the restrained layer, and allowing for the stress relaxation properties of the deformable layer theory. It proposes the reversible breaking and reformation of stressed bonds between the coupling agent and the substrate which allows stress relaxation without loss of adhesion in the presence of water. Since SiOH has a strong hydrogen bonding capacity, water is eliminated from the surface and a high initial bond strength is achieved. It has also been argued that when Si-O-Metal bonds are broken by the intrusion of water, the bond is capable of reforming with some recovery in adhesion.[24]

In this paper, it has been demonstrated that silanes materially improve the initial adhesion of epoxide and polyurethane coatings to a variety of metallic substrates. Further, their use results in a similar improvement in wet adhesion and also in recovered adhesion. In the experiments on aluminum and mild steel, it was demonstrated that all five of the silanes examined produced an improvement in adhesion.

If the possibility of chemical reaction between the silanes and some component of the polymeric binder is examined, it can be seen that the n-beta-aminoethylaminopropyltrimethoxysilane (A1120), $NH_2(CH_2)_2NH(CH_2)_3Si(OCH_3)_3$, may be expected to react with the epoxide resin via the epoxide group and the terminal primary amine group on the silane according to:


(4)

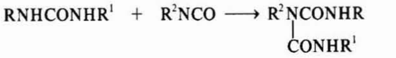
and possibly via the secondary amine group on the silane


(5)

In the case of the polyurethane paint, A1120 might be expected to react with the isocyanate group in the isocyanate adduct curing agent to form initially a substituted urea

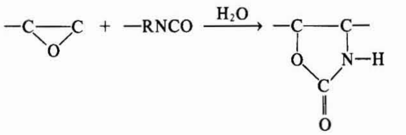
$$R{-}NH_2 + R^1{-}NCO \longrightarrow RNHCONHR \qquad (6)$$

with the possibility of a further reaction between the substituted urea and a further isocyanate group to form a biuret


(7)

$$RNHCONHR^1 + R^2NCO \longrightarrow R^2N(CONHR^1)CONHR \qquad (7)$$

(3-4 epoxycyclohexyl) ethyltrimethoxysilane (A186) may be expected to react with the amine groups on the polyamide curing agent according to reaction (4). With the polyurethane paint, a reaction between the isocyanate group and the epoxide to produce an oxazolidone appears to be a distinct possibility.

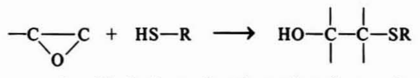

(8)

γ-glycidoxypropyltrimethoxysilane (A187) may be expected to react with the polyamide curing agent in the epoxide system as in reaction (3), and the isocyanate adduct curing agent in the polyurethane system as in reaction (5).

In the case of γ mercaptopropyl trimethoxysilane (A189), $HS-(CH_2)_3Si(OCH_3)_3$, a reaction with an isocyanate group in the polyurethane system via the terminal — HS group functioning as an —OH reactive site according to reaction (9) appears possible.

$$R^1\text{—NCO} + \text{HS—R} \longrightarrow R^1\text{NHCOSR} \qquad (9)$$

The—HS group may also react with an epoxide group in the epoxide system according to reaction (10).



(10)

It seems less likely that γ(methacryloxy) propylmethoxysilane (A174),

$$CH_2\text{—}\overset{CH_3}{\overset{|}{C}}\ \text{—}\overset{O}{\overset{\|}{C}}O(CH_2)_3\,Si(OCH_3)_3,$$

will react with any component of either the epoxide or polyurethane system but the presence of the polar $\overset{O}{\overset{\|}{C}}$ group introduces the possibility of a hydrogen bonding mechanism with the epoxide and the possibility of anionic addition at the double bond in the isocyanate group may exist.

It appears that some chemical reaction between the silanes and components of both paints (with the possible exception of A174) exists. However, the course of any individual reaction must depend on the structure of the resin and curing agent present, the solvents present, and the presence or otherwise of catalytic agents. Although a reaction is possible, it is not necessarily probable.

Perhaps some degree of specificity in the role of a particular silane/organic binder combination may be appreciated, but it is more difficult to see why an individual silane should give better results on a particular metallic substrate. It may be speculated that the degree of bonding, and therefore, the measured adhesive strength, is a function of such factors as the degree of hydration of the surface, the number of OH groups per unit area, the basicity of the oxide present, and the possibility of catalysis of the reaction by organic groups, e.g., amines present in the silanes. Certainly some silanes are more effective on aluminum than on mild steel, and the effects of A189 on cadmium, copper, and zinc have already been mentioned.

The XPS data shed some light on the role of the silanes in improving the adhesion of the coatings examined. Bikerman[30] has postulated on theoretical grounds that true adhesive failures cannot occur; observed apparent adhesion failures are, in fact, failures within a boundary layer at the paint/metal interface. Support for this view is given in the present investigation where an organic layer has been detected on the "metal" fracture surface. Thus, the failure is one of cohesion within the paint film. An important point here is that the same mode of failure is apparent on specimens fractured after storage in air *and* after storage in water, and broken while still water soaked. The type of failure remains unchanged by the ingress of water, although there is some evidence that the "site" of failure is changed in the case of the epoxide paint. The adhesion tests clearly indicate an improvement in bond strength in the presence of the silanes, but it is argued that this improvement is not obtained by an improvement in the adhesion to the metal surface since no adhesion failure was observed in the absence of the silane. Rather, the increase in bond strength was a result of a change in the cohesive strength of a boundary layer within the paint or in a diffuse silane/polymer layer. The net result is a boundary layer less susceptible to the effects of water.

The quantities of silicon detected do not seem to be consistent with a failure in a silane-rich layer. At least in the case of the polyurethane paint, failure would seem to be at a silane/paint interface since no silicon was detected on the "paint" fracture surface. In this case, the full potential of the water resistance of the silane has not been realized. The observations appear to be consistent with the requirements of the reversible hydrolytic bond theory, but much more work is required to fully elucidate the role of silanes in improving initial and wet adhesion.

In considering the use of silanes either as pretreatments for metallic substrates or as additives to an epoxide or polyurethane coating, different considerations apply. In the former, long-term stability is not a problem and a silane showing the best overall performance on both aluminum and mild steel can be selected, or, alternatively, a specific silane for a particular metal or metal/paint combination. From the results presented it would appear that (methacryloxy) propyltrimethylsilane (A174) and n-beta aminoethyl aminopropyltrimethoxysilane (A1120) are particularly effective on aluminum with both epoxide and polyurethane paints, while γ-mercaptopropyl trimethoxysilane (A189) and A1120 are preferred for mild steel. However, there is very little difference in the performance under any given set of conditions, and A1120 provides an excellent all-around performance when used as a pretreatment primer.

In the latter case, the options. are more limited and great care must be exercised in the final choice of silane to be used as an additive. Although a particular silane may be selected for use with a particular paint, no such choice seems to exist in the matter of substrate, since it would be impractical to manufacture a range of silane-modified paints for use specifically on aluminum or mild steel, or indeed on any other metallic substrate. Although A1120 gave good results in both the epoxide and polyurethane paints, it is known to react with oxygenated solvents present in most, if not all, paints of this type and its use must therefore be discounted. A174 and A189 have been shown to be stable in the epoxide and polyurethane, respectively, when the paints have been suitably "dried." Both are worthy of further investigation.

It must be stressed that the use of any silane as an additive to a particular formulation should be thoroughly investigated for long-term stability and a study on depletion rates should be carried out. Such a study could be made by the use of gas liquid chromotography (GLC).[31]

A factor not considered in this paper is the financial aspect of using silanes as adhesion promoters in surface coatings technology. It may be argued that any improvement in the adhesion characteristics of a paint is worthy of consideration, even if it involves increased expense. In view of the improved adhesion to degreased substrates it may well be that silanes could replace the use of a wash primer or a relatively costly surface preparation in situations where sandblasting is not essential.

CONCLUSIONS

From the work reported it may be concluded that:

(1) The use of organofunctional silanes as pretreatment primers for epoxide and polyurethane paints results in a dramatic increase in initial, wet, and recovered adhesion on aluminum and mild steel substrates.

(2) The use of organofunctional silanes as pretreatment primers on copper, cadmium, and zinc substrates results in an improvement in initial adhesion and in the long-term retention of adhesion.

(3) Organofunctional silanes may be used as additives to epoxide and polyurethane paints to materially improve their initial, wet, and recovered adhesion to aluminum and mild steel.

(4) Suitably formulated paints containing silanes are stable over a period of at least nine months.

(5) XPS studies reveal that the mechanism by which silanes improve the adhesion characteristics of paints may be adequately explained by the existing "reversible hydrolytic bond theory."

POSSIBILITIES OF FURTHER STUDY

The reported work reveals several areas in which further work would be valuable in both exploring the uses to which silanes may be put in improving the adhesion characteristics of organic surface coatings and determining the precise mechanisms by which the undoubted improvements occur. Suggested areas for future work are a more detailed examination of the sites of failure on fracture surfaces in order to more fully understand the role of silanes as adhesion promoters, in particular the composition and depth of the boundary layers; the use of silanes in coatings cured by other mechanisms, e.g., oxidative cure or solvent evaporation; and a more detailed investigation of the single package, self-bonding concept.

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APPENDIX A

Adhesion Tests

DIRECT PULL-OFF: Four squares, 31 × 31 mm (1¼ × 1¼ in.) were cut from each test panel, avoiding the panel edges and were sandwiched between cylindrical test specimens using a two part polyamide cured epoxide adhesive (Versamid 115/Epikote 828 50/50 w/w). After 24 hr in an alignment jig under pressure of 45N (10 psi), the resulting doublets were broken in a Hounsfield Tensometer at a cross-head speed of 2 mm/min (1/16 in/min).[21]

TORQUE SPANNER: Cylindrical specimens of known diameter were bonded to the test surface using the two-part epoxide adhesive previously detailed. After curing for 24 hr, the specimens were twisted off by using a recording torque spanner. Five specimens were used on each panel.[22]

The two separate adhesion tests were carried out on the same panel. In some experiments, a rapid curing cyanoacrylate adhesive IS150 (Loctite Corp.) was used.

Environmental Tests

HUMIDITY UNDER CONDENSATION CONDITIONS: The test was carried out to the dictates of BS3900, Method F2, in which the panel is subjected to humidity under condensation conditions cycling between 42 and 48°C. The period of exposure was 1000 hr.

WATER IMMERSION: The panels were totally immersed in distilled water at room temperature. The period of exposure was 1500 hr. Unless otherwise stated, all panels were allowed to dry out at ambient temperature and humidity for 48 hr prior to adhesion testing.

ACCELERATED WEATHERING: The test was carried out to the dictates of BS3900, Method F3 in which the painted panels are exposed to a 1600 watt enclosed carbon arc with water spray. The period of exposure was 1000 hr.

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Surface Analysis of Interfacial Chemistry In Corrosion-Induced Paint Adhesion Loss

II. Effects of Resin Chemistry and Substrate Pretreatment on Adhesion Loss Mechanism

J.W. Holubka, J.S. Hammond, J.E. DeVries, and R.A. Dickie
Ford Motor Company*

The effect of changes in resin composition on the interfacial chemistry of corrosion-induced paint de-adhesion has been studied by X-ray photoelectron spectroscopy (XPS); the effect of a zinc phosphate conversion coating on interfacial chemistry has also been assessed in a preliminary way. For epoxy-urethane and epoxy-amine based coatings crosslinked with melamine-formaldehyde resin or mixtures of melamine-formaldehyde and urea-formaldehyde resins, it has been found that corrosion-induced de-adhesion is associated with hydroxide degradation of coating resin, typically by hydrolysis of urethane or, more slowly, of urea linkages. For an epoxy-ester based resin formulated with a mixture of melamine-formaldehyde and urea-formaldehyde resins and applied to bare steel, it was previously reported that corrosion-induced de-adhesion involves saponification of ester linkages by cathodically-generated hydroxide. For this coating applied over a zinc phosphate conversion coating, it is now found that there is again evidence of paint resin degradation by ester saponification. The surface composition of the conversion coating also changes significantly, but the locus of adhesion loss is evidently in the organic phase very near the organic-inorganic interface.

INTRODUCTION

Loss of paint adhesion adjacent to sites of corrosion on painted steel is known to involve disruption of the paint-metal interface by corrosion-generated hydroxide ions.[1-6] X-ray photoelectron spectroscopy (XPS) has been used to study the interfacial chemistry of corrosion-induced adhesion loss, and in a previous paper,[7] results have been presented on an epoxy ester coating applied to bare steel. In that work, it was concluded that a significant pathway for adhesion loss was saponification of the coating resin adjacent to the coating steel interface. In this paper, results on additional paint resin systems and preliminary data on the epoxy ester coating applied to a conversion coated substrate are presented.

EXPERIMENTAL

Materials

Three hydroxy-functional coating resins were prepared based on epichlorohydrin-bisphenol A epoxies: the first was a conventional tall oil epoxy ester; the second, a urethane oligomer prepared from a diol-extended epoxy and toluene diisocyanate; and the third, an epoxy-alkanol amine resin. Each of these resins was formulated as a conventional solvent-based coating with either a butylated melamine-formaldehyde crosslinker or a mixture of butylated melamine-formaldehyde and urea formaldehyde crosslinkers, as is later noted. No pigments, surfactants, or other additives were employed. In the following discussion, these coatings are referred to as epoxy-ester, epoxy-urethane, and epoxy-amine coatings, respectively.

Unpolished bare SAE 1010 cold-rolled steel and Bonderite® R40 phosphated test panels were used. The bare steel panels were cleaned with xylene and acetone; the phosphated panels were reconditioned by baking at 150°C for 15 min just prior to application of organic coatings.

Presented by Dr. Holubka at the 58th Annual Meeting of the Federation of Societies for Coatings Technology in Atlanta, GA, October 29, 1980.

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Bonderite is a registered trademark of Parker Rust Proof Co.

Table 1—Elemental Intensity Ratios and Binding Energy Separations of Reference Compounds

Compound	Binding Energies[a]	Elemental Intensity Ratios	Binding Energy Separations
NaCl	(Na 1s) = 1072.5 eV (Cl 2p) = 199.3 eV	$\frac{(\mathrm{Na\ 1s})}{(\mathrm{Cl\ 2p})} = 0.91$	(Na 1s) − (Cl 2p) = 873.2 eV
Na_2CO_3	(Na 1s) = 1072.3 eV (C 1s:CO_3) = 289.6 eV	$\frac{(\mathrm{Na\ 1s})}{(\mathrm{C\ 1s})} = 1.7$	(Na 1s) − (C 1s) = 782.7 eV
	(O 1s) = 531.7 eV	$\frac{(\mathrm{Na\ 1s})}{(\mathrm{O\ 1s})} = 0.69$	(Na 1s) − (O 1s) = 540.6 eV
$NaCO_3CH_3$	(Na 1s) = 1071.9 (C 1s:CH_3) = 285.0	$\frac{(\mathrm{Na\ 1s})}{(\mathrm{C\ 1s})} = 0.38$	(Na 1s) − (C 1s:RCO_2^-) = 783.5 eV
	(C 1s:RCO_2^-) = 288.4	$\frac{(\mathrm{Na\ 1s})}{(\mathrm{O\ 1s})} = 0.50$	(Na 1s) − (O 1s) = 541.2 eV
	(O 1s) = 530.7		

(a) Referenced to methylene - methyl C 1s at 285.0 eV.

All organic coatings were applied by air atomized spray and were crosslinked by baking at 140°C for 20 min, or, in the case of the epoxy-amine films, by baking at 180°C for 20 min.

Corrosion Tests

Prior to exposure to the corrosion test, the edges and back of the panel were masked with air dry lacquer. Two lines in the form of an 'X' were scribed to bare metal on the front panel surface. Two essentially equivalent corrosion test methods were used. In the first procedure, developed by Wiggle, et al.[8] and used in a previous study on the epoxy-ester coating applied to bare steel,[7] the test panel was coupled to a zinc electrode and immersed in oxygen saturated 5% aqueous NaCl. The test panel was thus maintained at a potential of about − 1050 mv(vs. SCE); the anodic corrosion reaction was suppressed at the painted test panel, thereby allowing the influence of cathodically generated hydroxide to be determined independently of the anodic dissolution of iron. For the epoxy-ester-based coating applied to bare steel, extensive failure occurred within a few hours. With some of the new coatings which are reported on in this paper, substantially longer test times are required to achieve an equivalent degree of adhesion loss. To avoid possible problems from contamination of the bath by zinc ions, the test was performed using a platinum counter electrode and a potentiostat; the potential was maintained at about - 1070 mv (vs. SCE).

X-Ray Photoelectron Spectroscopy

The surface analysis methods were the same as reported previously.[7] The application of XPS to corrosion-induced coating adhesion loss has been discussed, and references to general reviews of XPS and its applications have been given.[7]

Following completion of the corrosion test, the test panel was removed from the salt solution, rinsed with

Table 2—Surface Composition-Epoxy-Urethane Coating on Bare Steel
(atomic %)

Sample	C	O	N	Na	Fe	Cl
Calculated	80.2	12.7	7.1	—	—	—
Before Testing						
coating surface	77.9	13.5	8.6	—	—	—
After Testing						
interfacial coating surface						
before water wash	66.9	23.1	4.3	4.9	0.5	0.3
after water wash	77.6	16.6	6.7	0.3	0.8	—
interfacial substrate surface						
before water wash	51.0	37.6	1.6	6.9	2.4	0.5
after water wash	47.8	40.2	1.9	—	9.8	0.5

Table 3—Surface Composition-Epoxy-Amine Coating on Bare Steel
(atomic %)

Sample	C	O	N	Na	Fe	Cl
Calculated	78.2	16.7	5.1	—	—	—
Before Testing						
coating surface	71.3	10.5	18.2	—	—	—
After Testing						
interfacial coating surface						
before water wash	66.2	22.4	1.7	8.0	—	1.7
after water wash	85.2	12.1	2.1	0.5	—	0.1
interfacial substrate surface						
before water wash	35.5	40.7	0.6	17.1	3.1	3.0
after water wash	64.0	24.1	2.0	0.8	8.7	0.4

Table 4—Surface Composition-Epoxy-Ester Coating on Zinc Phosphate Conversion Coated Steel (atomic %)

Sample	C	O	N	P	Zn	Fe	Na	Cl
Before Testing								
coating surface	86.9	10.6	2.5	—	—	—	—	—
After Testing								
interfacial coating surface	75.1	19.8	—	—	0.1	—	4.4	0.6
interfacial substrate surface	51.6	34.7	—	0.4	1.7	0.6	10.1	0.9

distilled water, and quickly wiped dry. The coating film adjacent to the scribed lines exhibited reduced adhesion to the underlying metal surface, and was removed with adhesive tape. The interfacial surface of the primer film was examined in the spectrometer without further treatment. A portion of the metal substrate was also cut and mounted for analysis.

X-ray photoelectron spectra were obtained using a Vacuum Generators ESCA 3 electron spectrometer interfaced to a Vacuum Generators 3040 data system. An aluminum anode x-ray source (1486.6 eV) was used to obtain the spectra. The instrument was operated with a resolution of 1.25 eV FWHM (full width at half maximum peak height) on the Au 4f 7/2 line. The binding energies for the spectra were referenced to the hydrocarbon contamination (present on all of the samples tested) and alkane carbon line at 285.0 eV. Elemental intensities were calculated using Scofield photoionization cross sections[9] and the formalism of Wagner.[10] Relative error in measurement of spectral intensity from the de-adhesion samples was estimated at 10%; the elemental intensities thus provided a semi-quantitative analysis of surface chemistry.

Molecular identification of compounds was based on comparison of elemental intensity ratios and absolute binding energy values with values obtained on standard compounds. Uniform sample charging (allowing use of the methyl hydrocarbon C1s line as an internal standard at 285.0 eV) can be assumed for most of the work reported here. In some cases, differential charging (a phenomenon in which different chemical phases adopt different surface potentials) precludes the use of a single internal binding energy standard. In these cases, molecular identification is based on comparison of binding energy separation values for pairs of elements of a given compound. Binding energy separation and relative intensities are summarized in *Table* 1 for three reference compounds.

RESULTS AND DISCUSSION

Epoxy-Ester Coating on Bare Steel

In the previous work on the corrosion-induced adhesion failure of an epoxy-ester based coating on bare steel, a significant surface layer of organic material was found on the interfacial metal surface in the region of corrosion-induced adhesion failure. The carbon, oxygen, and sodium spectra of the interfacial metal and polymer surfaces were indicative of polymer degradation products on both surfaces. The presence of carboxylate residues on the interfacial polymer surface was confirmed by derivatization with aqueous $AgNO_3$ to form silver carboxylate. It was concluded that cathodically generated hydroxide degraded the polymer near the interface by saponification of the epoxy ester, and that the locus of apparent adhesion failure was in the saponification weakened polymer near the metal-polymer interface.

Epoxy-Urethane Coating on Bare Steel

Melamine-formaldehyde resin crosslinked epoxy-urethane films tested under cathodic polarization conditions were slightly more resistant to corrosion-induced de-adhesion than similarly formulated epoxy-ester coatings: about four hours' exposure were required to achieve the same extent of de-adhesion (ca. 4 mm) observed with the epoxy ester in two to three hours.

Elemental surface compositions of the untested epoxy-urethane coating and of the interfacial coating and substrate surfaces following testing are given in *Table* 2. Experimental values for surface composition were in excellent agreement with calculated values, indicating that neither surface contamination nor surface segregation effects were in evidence for these samples. The interfacial coating surface showed several interesting differences relative to the untested surface: the oxygen concentration was higher, and the nitrogen concentration was lower; nearly 5 atomic % sodium was incorporated which could not be accounted for by the low level of chlorine (0.3 atomic %) present. There was also a trace of iron. Upon washing the interfacial coating surface with water, the sodium residue was largely removed and the oxygen concentration was substantially reduced. Evidently, there was a water-soluble sodium salt on the interfacial coating surface. A water-soluble sodium salt was also found on the interfacial substrate surface; removal of this salt resulted in an increase in relative iron concentration. There was a nitrogen-containing residue on the interfacial substrate surface both before and after water wash.

The water-soluble sodium salt was confirmed to be sodium carbonate by examination of high resolution spectra, as discussed in detail elsewhere,[11] and by comparison of binding energy differences with standard compounds. Thus, the sodium-carbon binding energy difference on the coating interfacial surface was 782.8 eV, in good agreement with the value for Na_2CO_3 given in *Table* 1.

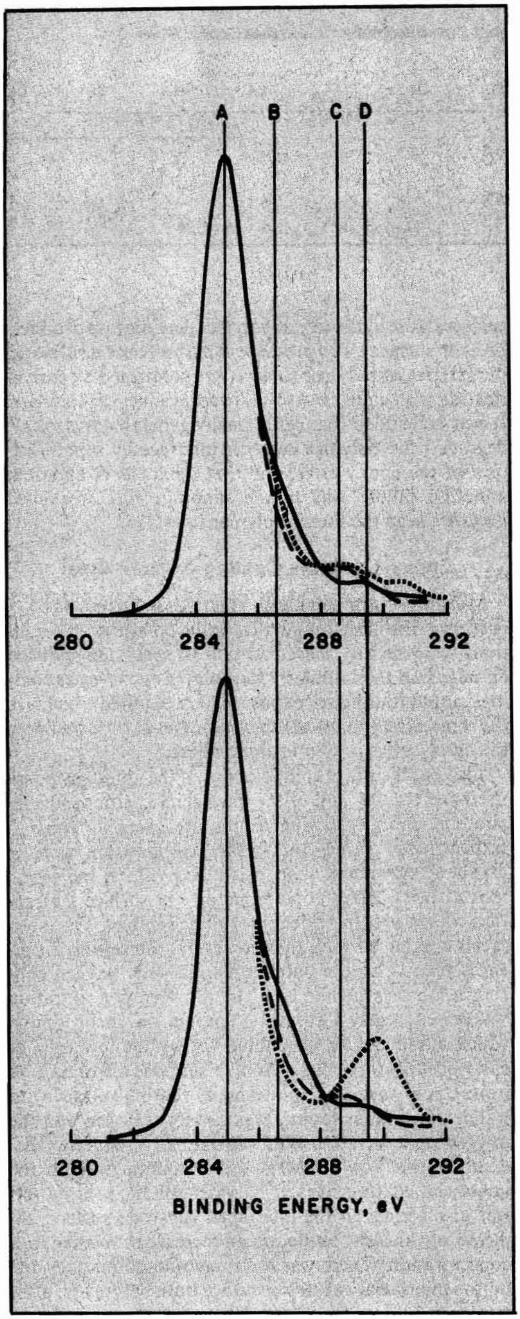
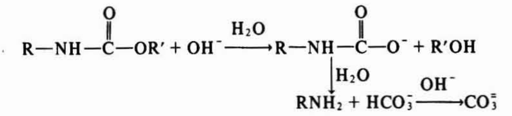


Figure 1—XPS C 1s spectra for epoxy-ester-based coating before corrosion test (solid line, top and bottom); interfacial polymer surface from corrosion tests on epoxy-ester-based coating applied to bare steel (long dashes, top) and to conversion coated steel (short dashes, top); interfacial metal surface from corrosion tests on epoxy-ester-based coating applied to bare steel (long dashes, bottom) and to conversion coated steel (short dashes, bottom). Spectral components A, B, C, and D correspond, respectively, to alkane contamination, ether, carboxylate, and carbonate

The low nitrogen concentration on the interfacial coating surface both before and after water wash (N/O was 0.64 for the untested surface compared with 0.19 or 0.40 for the unwashed and washed polymer surfaces, respectively) was consistent with a degradation of polymer with loss of nitrogen-containing species during corrosion. The presence of a nitrogen-containing residue on the interfacial metal surface was, on the other hand, evidence for a polymer residue on that side of the interface.

The presence of sodium carbonate on the interfacial surfaces after cathodic polarization testing can be rationalized in terms of a degradation of the urethane linkages of the coating by cathodically generated hydroxide:



$$\mathrm{R{-}NH{-}\overset{\overset{O}{\|}}{C}{-}OR' + OH^- \xrightarrow{H_2O} R{-}NH{-}\overset{\overset{O}{\|}}{C}{-}O^- + R'OH}$$
$$\downarrow \mathrm{H_2O}$$
$$\mathrm{RNH_2 + HCO_3^- \xrightarrow{OH^-} CO_3^{=}}$$

The low nitrogen content of the two surfaces can also be rationalized in terms of this degradation mechanism, since the low molecular weight diamine formed by hydrolysis of adjacent urethane linkages was likely washed away or volatilized from the surface after cathodic polarization testing.

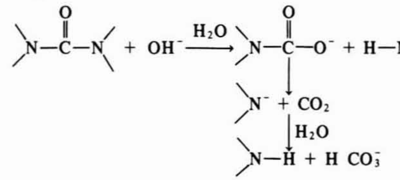
Epoxy-Amine Coating on Bare Steel

The epoxy-amine resin formulated with melamine-formaldehyde and urea-formaldehyde crosslinkers afforded coatings with substantially more resistance to cathodically generated hydroxide than either the epoxy-ester or epoxy-urethane resins. About 240 hr exposure to cathodic polarization test conditions were required to achieve the same degree of adhesion loss observed in four hours or less with the ester and urethane resins. *Table* 3 presents surface elemental compositions for the epoxy-amine coating before and after testing; calculated values for the bulk polymer composition are also included.

The untested coating surface showed an unexpectedly high nitrogen concentration, most likely reflecting surface segregation of one of the nitrogen-rich crosslinking resins. After cathodic polarization testing, the coating interfacial surface showed higher oxygen and lower nitrogen content than expected from either calculated bulk composition or observed untested surface composition. Also present was a significant sodium residue not explainable in terms of residual sodium chloride. As on the epoxy-urethane interfacial surface, most of the sodium salt residue was water-soluble, and, again, as on the epoxy-urethane surface, the residue was identified as sodium carbonate both by examination of high resolution spectra[11] and by comparison of binding energy values with standard compounds. In this case, the binding energy separation between sodium and carbon was 782.6 eV compared with 782.7 eV for Na_2CO_3 and 782.8 eV for the residue on the epoxy-urethane surface.

The interfacial substrate surface showed a similar water-soluble residue also identified as Na_2CO_3. In addition, the interfacial substrate surface showed a significant nitrogen containing residue characteristic of coating resin.

These observations are consistent with degradation of the urea crosslinker of the coating by cathodically generated hydroxide:



Degradation of the condensed urea structure characteristic of the crosslinking resin would likely yield water-soluble or volatile compounds, consistent with the observed low surface. nitrogen content. (The observed surface segregation of melamine crosslinker also may contribute to the lower interfacial nitrogen concentration, but the relative contribution of the two effects cannot be assessed readily).

Corrosion-induced de-adhesion of the epoxy-amine based coating was slower than for either the epoxy-ester or epoxy-urethane coatings, both because the urea linkage was inherently more resistant to hydrolysis than ester and urethane linkages and because the concentration of saponifiable groups was substantially lower. The epoxy-amine resin contained no saponifiable groups, in contrast to the epoxy-ester and epoxy-urethane resins; thus, the only source of saponifiable groups in this formulation was the relatively small proportion of urea-formaldehyde crosslinker. Thus, it is concluded that although the time scale was different, the mechanism of de-adhesion in each case probably involved polymer degradation by corrosion-generated hydroxide. The polymer degradation products were ionic species, and it seems likely that one consequence of the degradation was more facile swelling of the coating adjacent to the interface by water and electrolyte, thus extending the corrosion cell as well as weakening the coating-substrate bond.

Epoxy-Ester Coating On Conversion Coated Steel

Inorganic conversion coatings can substantially increase the time required for a given degree of de-adhesion under cathodic polarization conditions. For the epoxy-ester coating applied to a commercial zinc phosphate coating, the time to achieve 4–5 mm de-adhesion from the scribe is increased from 2–3 hr to about 48 hr.

Elemental compositions of the surfaces are compared in *Table* 4. There was again, as observed for this coating over bare steel as well as for the epoxy-urethane and epoxy-amine coatings, a substantial nonchloride ionic residue on both the interfacial coating surface and the interfacial substrate surface. In addition, surface oxygen concentration was higher and observed nitrogen concentration was lower than for the untested coating surface. It is of particular interest that there was almost no trace of the conversion coating on the interfacial film surface, while there was substantial organic residue on the interfacial substrate surface.

XPS carbon (C 1s) and oxygen (O 1s) spectra are compared in *Figures* 1 and 2 for the epoxy-ester formulation applied over bare and conversion-coated substrates. A

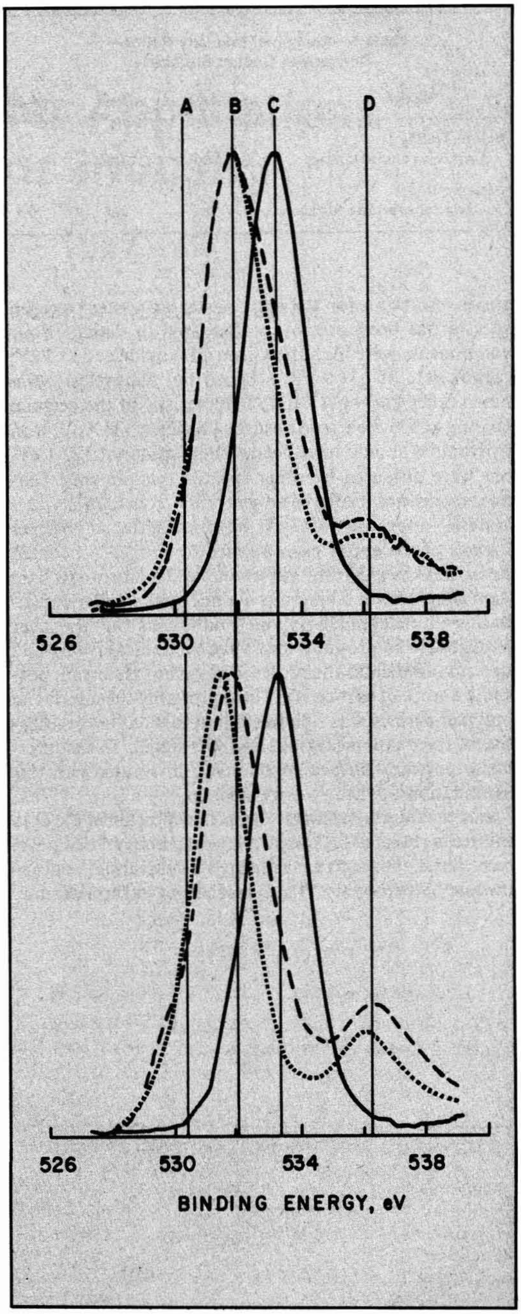


Figure 2—XPS O 1s spectra for epoxy-ester-based coating before corrosion test (solid line, top and bottom); interfacial polymer surface from corrosion tests on epoxy-ester-based coating applied to bare steel (long dashes, top) and to conversion coated steel (short dashes, top); interfacial metal surface from corrosion tests on epoxy-ester-based coating applied to bare steel (long dashes, bottom), and to conversion coated steel (short dashes, bottom). Spectral components, A, B, C, and D correspond, respectively, to iron oxide, carbonate-carboxylate, untested polymer, and water

Table 5—Elemental Intensity Ratios—Conversion Coated Surfaces

Sample	Fe/P	Zn/P	Fe/Zn
Before Testing			
conversion coated surface	0.7	0.3	2.4
After Testing			
interfacial substrate surface	1.7	4.4	0.4

curve resolution for the epoxy-ester/bare steel oxygen spectra has been previously discussed in detail.[7] Four components were identified: iron (III) oxide at 530.3 eV; carboxylate at 531.9 eV; a broad but featureless combined ether-carbonyl band characteristic of the original coating at 533.3 eV; and absorbed water at 536.4 eV. Iron hydroxides should have been evident at about 532.1 eV, but were obscured by other spectral components. Carbonates are not readily distinguishable from carboxylates and they appear at 531.7 eV. For the interfacial polymer surface of the epoxy ester applied to conversion coated steel, the O 1s spectrum was similar in structure to its bare steel counterpart. There was a somewhat smaller contribution of undegraded polymer, indicating that the layer of degradation products may have been thicker. The C 1s spectrum indicates the presence of carboxylate and, possibly, a trace of carbonate. Thus, probably the interfacial polymer surface was rich in sodium carboxylate, indicative of ester saponification, and was similar to the interfacial polymer surface encountered in results with this formulation applied to bare steel.

For the interfacial metal surface, inspection of the O 1s spectrum revealed little sign of residual undegraded polymer. The C 1s spectrum indicated a substantial concentration of carbonate. The presence of carbonate may have been due to degradation of urea crosslinker in the longer test period required for the epoxy ester when applied over a conversion coating. Alternatively, it could reflect CO_2 absorption by residual OH^- in the conversion coating.

Relatively little zinc, iron, or phosphorous were observed on the substrate surface due to the substantial carbonate overlayer. Nevertheless, the surface of the conversion coating was affected in some way by the corrosion test: the elemental ratios of iron and zinc to phosphorous were significantly larger than initial values characteristic of the conversion coating itself (cf. *Table* 5). These results indicate that, although corrosion-induced de-adhesion of the epoxy-ester coating over the zinc phosphate conversion coating involved degradation of the polymer at or near the organic inorganic interface, it was accompanied by changes in coating surface composition. The locus of de-adhesion was evidently near the interface, but in the organic phase.

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Committee Activities

Technical Computer Applications In the Coatings Industry

A Bibliography—1967–79

By the FSCT Technical Information Systems Committee
(In cooperation with the International Coordinating Committee)

This bibliography had its beginning at a meeting of the International Coordinating Committee (ICC) on October 4, 1979, during the Federation's Annual Meeting in St. Louis.

The ICC, which was formally established on June 23, 1979, in Paris, is composed of the President and one other representative from the following coatings industry organizations:

FSCT—Federation of Societies for Coatings Technology
FATIPEC—Federation of the Associations of Technicians in the Paint, Varnish, Lacquer, and Printing Ink Industries of Continental Europe
OCCA-UK—Oil and Colour Chemists' Association of the United Kingdom
OCCA-AU—Oil and Colour Chemists' Association of Australia
JSCM—Japan Society of Colour Material
SLF—Scandinavian Federation of Paint and Varnish Technologists

At the St. Louis meeting, it was decided by the ICC that the preparation of such a bibliography would be a worthwhile initial project for the new international organization. The FSCT volunteered to undertake the work and assigned the search to its Technical Information Systems Committee, Helen Skowronska, Chairperson.

Jacques Roire, a Past-President of FATIPEC, also submitted 63 references to the bibliography, as a contribution from the French Association of FATIPEC.

The Federation expresses its gratitude to Ms. Skowronska and the Technical Information Systems Committee, for doing such a fine job in compiling this bibliography. We also acknowledge, with thanks, the special assistance of The Sherwin-Williams Co., Glidden Coatings & Resins Div. of SCM Corp., PPG Industries, Inc., and Mr. Roire.

INTRODUCTION

The references included in this bibliography are arranged under ten broad headings, or sections, listed below:

AG: Applications—General
AM: Analytical Methods
CO: Color and Optical Properties
EP: Evaluation; Performance
FR: Formulation
MP: Manufacturing and Plant
PA: Pretreatment; Application; Finishing
PI: Printing Inks
PR: Polymers/Resins
TS: Testing

Each reference is listed only under one heading. The arrangement within a given section is alphabetical by the author's surname.

As is customary, each reference or citation consists of the following information: author(s) name(s), title of article, paper, or book, name of periodical or journal in which the paper was published, and any other pertinent information in the abstracting/indexing publication. The abbreviations shown for the titles of the periodicals are those used by the abstracting/indexing publication.

Since most of the references were located by searching the Subject Indexes of CHEMICAL ABSTRACTS and/or REVIEW OF CURRENT LITERATURE/WORLD SURFACE COATINGS ABSTRACTS, the pertinent reference to abstracting/indexing publication appears in parentheses after the standard citation details. The following abbreviations are used to designate abstracting/indexing publications:

CAS—Chemical Abstracts
RCL—Review of Current Literature (now WSCA)
WSCA—World Surface Coatings Abstracts.

In order to find references on a given subject, it is advisable to scan one or more of the broad headings rather than just the most obvious one.

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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.

Society Meetings

Dallas Sept. 11

Von Pederson, of Tioxide of Canada, spoke on "THE INFLUENCE OF TITANIUM DIOXIDE ON THE FLOW AND LEVELING OF GLOSS LATEX PAINTS."

Mr. Pedersen introduced his presentation with an explanation of the hiding power of titanium dioxide. Several graphs were displayed to show different aspects of pigmentation. Purity in titanium dioxide pigment is important in that 100 ppm iron will impart a yellow color, he said. Mr. Pedersen maintained that a pigment concentration best for good milling in a ball mill is 40% or better. He exhibited a diagram showing the most useful PVC ranges for a high speed impeller mill.

WILLIAM A. WENTWORTH, *Secretary*

Golden Gate Sept. 15

A moment of silence was observed in memory of Jim Spratt, Al Luebkeman, Travis Rankin and Jack Heymes, who died recently.

Bob Cole presented the Tenneco Gavel to President Sharon Vadnais, of E.T. Horn Co.

A Past-President's pin was then presented to outgoing President Ted Favata.

In addition, Alfred Apfel Memorial Scholarships were presented to Kenneth Adkins, Melanie Shaw, and Tracie Waldron in recognition of their outstanding scholarship achievements.

Al Heitkamp, of Cargill, Inc., spoke on "HIGH SOLIDS COATINGS, A SMORGASBORD APPROACH."

Mr. Heitkamp's presentation covered the performance characteristics of four polyester high solids white baking enamels: conventional, high solids high performance, high solids general purpose, and very high solids. The four enamels contained the same levels of titanium dioxide and each enamel was incorporated with an amine-blocked acid catalyst to insure proper cure, he said.

Mr. Heitkamp explained that the enamels were then examined for similarities and differences in physical application and performance properties. As a result of this investigation, it was found that baking high solids coatings can be formulated at up to 90% volume solids, with film properties similar to those conventional solids coatings. In addition, the coatings formulator has a choice of VOC range to meet specific state and user requirements. According to the speaker, high solids coatings are generally easier to formulate than water-borne and powder coatings. He ended his presentation by stating that the extra operational costs of applying high solids coatings can in many cases be offset by reduced materials and shipping costs.

W. WAYNE COCHRAN, JR., *Secretary*

Kansas City Sept. 11

A moment of silence was observed in the memory of Harold Kicker, Society Honorary Member, who died recently.

The Tenneco Gavel was presented to President Dick Warren by Ted Terherst.

Karl King, of Valspar Corp. was nominated for, and elected Vice-President of the Society.

Lee Raden, of Fabricated Metals, spoke on "SEMI-BULK HANDLING SYSTEMS FOR PAINT AND RAW MATERIALS."

MIKE BAUER, *Secretary*

Los Angeles Sept. 10

A moment of silence was observed in memory of E.J. Murphy, of Old Colony Paint; D. Copenhaver, of Calsol, Inc.; R.S. Haley, of Emery Industries; and G. Halverson, of Pacific Coast Chemicals, who died recently.

The following officers of the 1980-81 year were introduced: President—Donald I. Jordan, of Cargill, Inc.; Vice-President—Jan P. Van Zelm, of Byk Mallinckrodt; Treasurer—Romer E. Johnson, of Dorsett & Jackson; Secretary—L. Lloyd Haanstra, of Ameritone Paint Corp.; and Council Representative—Gerald L. West, of Devoe & Raynolds Co.

Robert Gutknecht, of Tenneco Corp., presented President Jordan with the Nuodex Gavel.

President Jordan then presented Al Seneker with the Past-Presidents pin.

Al Heitkamp, of Cargill, Inc., spoke on "HIGH SOLIDS COATINGS, A SMORGASBORD APPROACH."

Aided by a slide presentation, Mr. Heitkamp discussed many aspects of industrial high solids coatings, including formulations, viscosity, pattern, pigmentation, stability, performance, and

Los Angeles Society Officers for 1980-81. (Left to right): President—Donald I. Jordon, Cargill, Inc.; Vice-President—Jan Van Zelm, Byk-Mallinckrodt; Treasurer—Romer E. Johnson, Dorsett & Jackson, Inc.; and Secretary—Lloyd Haanstra, Ameritone Paint Corp.

cost. He revealed that, as solids are raised, cost effectiveness is gained and solvent emissions are lowered. However, some performance properties are lost.

Mr. Heitkamp discussed the "bubble concept," the allowance of credit for solvent recovery or incineration which leads to variable allowable solvent emissions by industrial coatings operations, which is now adopted in State Implementation Plans. He showed how this concept can be applied to the high solids approach by offsetting the emission of one production line versus another.

L. LLOYD HAANSTRA, *Secretary*

Louisville Sept. 17

J. Kirk Menefee, of Hy-Klas Paint Co., was presented with the traditional gavel upon his installation as Society President.

The Executive Committee voted to present the Paint Research Institute and the University of Louisville with checks for $500 each.

Don Collier, of Porter Paint Co., spoke on "EXTENDER PIGMENTS IN LATEX WALL PAINTS."

Mr. Collier discussed the importance of choosing the correct extender pigments. He recommended selecting those extenders which will achieve the best balance of properties.

JOHN A. LANNING, *Secretary*

New England Sept. 18

The following officers were introduced for the year 1980-1981: President—Dan Toobs, of Lukens Chemical Co.; Vice-President—Bob Modrak, of Benjamin Moore & Co., Inc.; Treasurer—John Fitzwater, of Polyvinyl Chemical Industries; and Secretary—Fran Koebert, of Kyanize Paints, Inc.

The Tenneco Gavel was presented to President Toobs by Don Aikman, of D.H. Litter Co., Inc.

Sam Belletiere, of Tenneco, spoke on "DRIERS FOR WATER-BORNE COATINGS."

Mr. Belletiere reviewed some of the basic aspects of drier technology. Metal soaps (driers) have been used for many years in oleoresinous and alkyd vehicles to aid in the "drying" process, i.e., crosslinking of unsaturated groups in the oil, he explained. Metal soaps are differentiated by metal type and acid carrier, and are grouped as either "active" or "auxiliary" driers.

Mr. Belletiere concluded with an explanation of the three major classes of water-borne systems: alkyd modification of a latex paint; water-dispensible alkyds and urethanes; and water-soluble alkyds.

FRAN KOEBERT, *Secretary*

New York Sept. 9

A moment of silence was observed in memory of David H. Litter, Founder of D.H. Litter Co., who died recently.

Marvin J. Schnall, of Troy Chemical Corp., was presented the Nuodex Gavel upon his installation as Society President for 1980-1981.

Other Society officers installed were: Vice-President—Donald E. Brody, of Skeist Laboratories, Inc.; Secretary—Ted Young of Jesse S. Young & Co.; Treasurer—Herb Ellis, of D.H. Litter Co., Inc.; and Society Representative—S. Leonard Davidson, of NL Chemical/NL Industries, Inc.

Sidney J. Rubin, of Empire State Varnish Co. was presented with the Past-President's pin.

The President's Service Award was given to Joseph Cantor, of Fyn Paint & Lacquer Co., Inc., in recognition of his years of service to the Society.

John J. Oates, of Troy Chemical Corp., spoke on "THE PLACE OF ADDITIVES IN MODERN COATING SYSTEMS."

Mr. Oates defined an additive as a chemical or a surfactant that, when added to paints in small amounts, has a significant effect on appearance, durability, or stability of the system. He discussed different ways of formulating with or without additives, citing the examples of medium oil alkyds and their molecular weights. According to Mr. Oates, additives are necessary, but they must be tested for all properties.

TED YOUNG, *Secretary*

Executive Committee of the Toronto Society for the year 1980-81. Seated (left to right): Membership Chairman—Ted Stevenson; Program Chairman—Gord Major; Publicity Chairman—Larry Ham; President—Jan Grodzinski; President-Elect—Paul Cooper. Standing (left to right): Past-President—Helmet Zapfe; Secretary—Albert Suk; Technical Chairman—Peter Hiscocks; Education Chairman—Radi Narancic; By-Laws and Specifications Chairman—Frank Reckless; and Manufacturing Chairman—Ray Reynolds. Not present are: Treasurer—Frank LaRoche; FSCT Board Representative—A. Clarke Boyce; Environment Chairman—Steve Balmer; Membership Chairman—Hans Wittman; Entertainment Chairman—Jim Provan; and Metric Chairman—Ernie Humburger.

Northwestern Sept. 9

Gerland Sprinkle, Jr., of Eastman Chemical Products, Inc., spoke on "SOLVENTS—THEIR RELATIONSHIP TO HIGH SOLIDS COATING PARAMETERS."

Five parameters of solvents and their effect on high solids were discussed: density of solvent, solvent activity, electrical properties, surface tension, and boiling point and evaporation rate.

To emphasize the importance of density of solvent in high solids, Mr. Sprinkle considered the volatile organic compound (VOC) restrictions being proposed for various end-users. He stated that solvents range from light to heavy in the following approximate order: aliphatic hydrocarbons, lower molecular weight alcohols, lower molecular weight ketones, aromatics, glycol ethers, and glycol ether esters.

Mr. Sprinkle pointed out that ketones are among the most active solvents known for organic film formers, since they have relatively low density and high solvency.

DON EMCH, *Secretary*

Philadelphia Sept. 18

A moment of silence was observed in memory of Roger Garland, of New Jersey Zinc Co., who died recently.

Honored guests in attendance included Federation Executive Vice-President, Frank J. Borrelle, and Director of Communications, Thomas A. Kocis.

The following officers for 1980-1981 were installed: President—Barry Oppenheim, of McCloskey Varnish Co.; President-Elect—Ellis C. Moore, of Neville Chemical Co.; Secretary—Wayne A. Kraus, of The Lawrence-McFadden Co.; and Treasurer—Willis A. Johnston, of Atlantic Richfield Co.

Bill Meadows, of Cyprus Industrial Minerals Co., spoke on "TALC—AN OLD MINERAL WITH NEW USES."

Mr. Meadows described an ultrafine platey magnesium alumino-silicate pigment which is processed from the mineral chlorite and which combines the desirable properties of kaolin clay and talc in a single extender. He related the results of a series of tests which compared magnesium alumino-silicate with calcined and delaminated clays in typical vinyl acrylic interior latex formulations at PVCs ranging from 30 to 80%. Among the advantages of the magnesium alumino-silicate he cited were: equal or superior opacity when compared with clays; superior holdout and stain resistance when compared with calcined clay; superior scrub resistance when compared with delaminated clay; and excellent low sheen characteristics. When tested against acidic clays, the basic magnesium alumino-silicate displayed higher pH in latex paints. Also observed were outstanding sheen, color uniformity, and dispersibility in water with minimum surfactant requirements, he concluded.

WAYNE A. KRAUS, *Secretary*

Piedmont Sept. 17

The following officers for 1980-1981 were introduced: President—William J. Cunane, of Sherwin-Williams Co.; Vice-President—James N. Albright, Jr., of The Lilly Co.; Secretary—Sara M. Robinson, of Union Oil Co. of California; and Treasurer—James Husted, of Mobil Chemical Co.

The Tenneco Gavel was presented to President Cunane by Bill Bell, of Tenneco.

Dr. Emil Sagovac, of Carolina Coatings, was presented with a 25-Year pin, commemorating his years of service to the Federation.

Daniel Adrien, of Burgess Pigment Co., spoke on "EFFICIENT FORMULATING TECHNIQUES FOR THE 80'S."

In his presentation, Mr. Adrien dealt with the use of clays in trade sale

Constituent Society Meetings and Secretaries

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

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

CHICAGO (First Monday—meeting sites in various suburban locations). LAYTON F. KINNEY, Standard T Chemical Co., Inc., 10th & Washington Ave., Chicago Heights, IL 60411.

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

formulations and in industrial coatings. He discussed how crude clay is further refined into calcined clays. Through the use of slides, the speaker demonstrated the structure of a calcined clay particle, as well as the economics of using calcined clays as extenders with titanium dioxide.

Mr. Adrien showed the improved characteristics of coating films using comparative "draw-downs." In trade sale paints, Mr. Adrien pointed out that savings of ten to eighteen cents per gallon could be realized without sacrificing stain resistance and scrub and burnish resistance. In the industrial coatings market, clays have been gaining commercial acceptance during the last two years, he stated. One area of particular interest is water-based electrodeposition coatings. According to Mr. Adrien, film strength and cold check will not be adversely affected when clays are used to reduce the amounts of titanium dioxide normally required.

SARA M. ROBINSON, *Secretary*

Pittsburgh Sept. 8

The following officers were installed for the year 1980–1981: President—Raymond C. Uhlig, of Technical Coatings Co.; Vice-President—Richard Trudel, of Mobil Chemical Co.; Secretary—William Cibulas, of Mobay Chemical Co.; and Treasurer—Michael Gillen, of Van Horn Metz & Co.

Alex Siegel, of Silberline Manufacturing Co., spoke on "THE USE AND MISUSE OF ALUMINUM PIGMENTS IN SURFACE COATINGS."

Tracing the history of aluminum pigments, Mr. Siegel stated that they date back approximately 80 years and were rarely used in coatings at the time. He defined the terminology of leafing and nonleafing. In the treatment of aluminum, stearic acid is used for leafing and oleic acid for nonleafing, he explained. Leafing aluminums were introduced around 1930.

Aluminum is a reactive metallic flake which is processed in a liquid paste form to prevent possible explosion, Mr. Siegel continued. Gas formation can occur when in contact with moisture. Because of this, 0.15% is the maximum amount of moisture allowed in preparation. Mr. Siegel recommended the use of cobalt and zirco driers as catalysts, since others can cause deleafing. Because of seeding of the aluminum paste, he cautioned against using polar solvent, high acid value vehicles, high speed dissolves and cuno falters. Mr. Siegel concluded his presentation with a discussion of the problem of stability in the use of water-dispersed aluminum paste over a period of time.

WILLIAM CIBULAS, *Secretary*

Rocky Mountain Sept. 8

Al Heitkamp, of Cargill, Inc., spoke on "HIGH SOLIDS COATINGS, A SMORGASBORD APPROACH."

DONALD R. BAGGE, *Secretary*

St. Louis Sept. 16

Incoming President Floyd Thomas, Jr., of Thomas & English, Inc., presented Herbert S. Rosenblatt with the Past-President's pin and Frank Wagner, of Mozel, presented President Thomas with the Tenneco Gavel.

Alex Elbrechter, of Degussa Corp., spoke on "CARBON BLACKS."

Describing the production of carbon blacks within his company, Mr. Elbrechter explained the three processes used: furnace, lamp and channel. Channel black is no longer produced in the U.S. because the small particle size of the black makes it a pollutant, he said. A channel black has been made, however, which meets European EPA standards.

According to Mr. Elbrechter, channel blacks are the strongest of the three types and they have a blue undertone in masstone. By complexing or reacting these with oxygen, a series of channel blacks have been produced with stability in the vehicle, ease of dispersion, improved rheology, and very jet black masstone color.

Q. Does the oxygen complexing of channel black interfere with surface area measurements?

A. Only the external surface of the carbon particle is important in formulation work. The interior of the particle does not absorb any of the vehicle.

JOSEPH J. WROBEL, JR., *Secretary*

Southern Society—Memphis Section Sept. 9

Hugh M. Smith, of Sun Chemical Co., spoke on "GOVERNMENT AND ENVIRONMENTAL GROUP PRESSURE IN THE COATINGS INDUSTRY."

BRIAN L. BUDZIEN, *Secretary*

Future Society Meetings

Baltimore

(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.

Chicago

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

(Feb. 2)—"LATEX PARTICLE INTERACTIONS"—C.T. Havens, Sherwin-Williams Co.; "NEW WATER-REDUCIBLE POLYMERS"—Henry Kasprzak, Spencer Kellogg Div.

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

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

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

C-D-I-C

(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.

Houston

(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

(Jan. 14)—"EXTENDERS, THE INORGANIC BACKBONE OF FLATS AND PRIMERS"—Thomas G. Brown, Engelhard Minerals & Chemical Corp.

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

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

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

Montreal

(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 England

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

(Feb. 19)—"AMINO CROSSLINKERS: RELATIONSHIP 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.

New York

(Jan. 13)—"VENTILATION IN A PAINT PLANT"—Gabe Malkin, Jesse S. Young Co.

(Feb. 5)—"LEGISLATIVE UPDATE"; JOINT MEETING with New York PCA.

(Mar. 10)—MINIWORKSHOPS.

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

(May 12)—PAVAC AWARD NIGHT.

Philadelphia

Technical Committee Meetings

(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

(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.

Technical Articles in Other Publications

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

Farbe und Lack
(In German)

Published by Curt R. Vincentz Verlag, Schiffgraben 41-43,
3000 Hannover 1, Postfach 6247.

Vol. 86 No. 7 July 1980

Grau, C.A.—"The Situation in the Paints and Pigments Industry"; 587.
Potente, H. and Kruger, R.—"Mechanical Properties of Coated Amorphous Thermoplastics. Some Aspects of How the Mechanical Properties are Influenced Using a Scratch Resistant Finish Based on PMMA as an Example"; 588-591.
Reichert, K.-H.W. and Donnebrink, G.—"Modified Form of Thermomechanical Analysis for Measuring the Temperature Dependent Properties of Coating Films"; 591-594.
Hoffman, K.—"Digital Data Recording with the Elrepho Colour Measuring Apparatus"; 595-597.
Heinze, P.—"Requirements of Solvent-Free Coatings"; 597.
Brushwell, W.—"Improvements in the Product Quality of Alkyd Resins"; 598-602.
Kolar, O.—"Are Bonding Agents which are Poor in Solvents Absolutely Necessary in the Production of Industrial Lacquers?"; 603-606.

Vol. 86 No. 8 August 1980

Vincentz, K.W.—"Formula 80—A New Start in Exhibitions for the Lacquer Industry?"; 685.
Wolski, W. and Wolniewicz, A.—"Physical-Chemical Processes Occurring During Processing of Ferrite Yellow for Magnetic Tape"; 686-689.
Mleziva, J.—"New Developments in Unsaturated Polyester Resins"; 689-696.
Marquardt, W. and Gempeler, H.—"Outdoor Durability of Powder Paints Based on Oil-Free Polyesters Containing Carboxyl Groups and Triglycidyl Isocyanurate"; 696-698.
Besold, R. "Metal Pigments for Automobile Finishes—From Manufacture to Application"; 699-701.
John, W.—"From Separating Sieve to Stroke Separator"; 702-705.
Brushwell, W.—"Applications and Modification of Acrylate Resins"; 706-710.

Organic Coatings and Plastics Chemistry Preprints

Preprints of papers presented before ACS Division of Organic Coatings and Plastics Chemistry at ACS 180th National Meeting,
Las Vegas, Nevada

Vol. 43 August 1980

New Concepts in Applied Polymer Science—Polymer Blends (7 papers)
Nonpolluting Paint Technology (13 papers)
Recent Aspects of Polymer Flammability (18 papers)
Review and Preview of Polymer Science—Salute to H.F. Mark (9 papers)
Emulsion Polymerization (49 papers)
Polymer Materials for Electronic Application (24 papers)
Polymers and Coal
  Depolymerization of coal (6 papers)
  Structure of coal (5 papers)
Advances in Coating Metals for Corrosion Protection (12 papers)
Conductive Polymers (24 papers)
New Concepts in Applied Polymer Science (15 papers)
Macromolecular Secretariat Symposium on Polyurethanes (21 papers)

For information regarding the price and availability of this publication, please contact Dr. John Lupinski Research & Development Center, Bldg. K 1, General Electric Co., P.O. Box 8, Schenectady, N.Y. 12301.

Paint Manufacture

Published by Wheatland Journals Ltd., 157 Hagden Lane, Watford
WD1 8LW, U.K.

Vol. 50 No. 2 March 1980

Anon.—"More Legislation for the Paint Industry"; 16-18.
Anon.—"Plastic Pails for Paint"; 14-15, 25.

Vol. 50 No. 3 April 1980

Krishnamurti, N.—"Accelerated Corrosion Tests and their Reliability"; 20-23.
Ellinger, M.L.—"Anticorrosion and Marine Paints" (Literature review); 45-46.

Vol. 50 No. 4 May 1980

Lower, E.S.—"Laurate Metal Soaps"; 22,24,26-27.

Polymer Preprints

Preprints of papers presented before ACS Division of Polymer Chemistry at ACS 180th National Meeting, Las Vegas, Nevada

Vol. 21 No. 2 August 1980

Nonequilibrium Behavior of Polymeric Glasses (22 papers)
Mitchell Shen Memorial Symposium
  Viscoelasticity and Structure (7 papers)
  Plasma Polymerization, Fluorescence and ORD (7 papers)
  Siloxanes, Ionomers, Degradation and Biomedical (7 papers)

Polymeric Separation Media
  Transport in Polymeric Media (6 papers)
  Functionalized Polymers as Separation Media (6 papers)
  Polymeric Membranes as Separation Media (6 papers)
  Novel Polymeric Separation Media, Structure and Properties (7 papers)

Nonurethane Polymers from Isocyanates (7 papers)

Emulsion Polymerization (48 papers—abstracts only)

Macromolecular Secretariat—Urethane Chemistry and Applications (7 papers out of 50 listed)
Special Topics Poster Session (76 papers)

For information regarding the price and availability of this publication, please contact Stanley Israel, Chemistry Dept., University of Lowell, Lowell, Mass. 01854.

Progress in Organic Coatings
(In French, German or English)

Published by Elsevier Sequoia S.A., P.O. Box 851,
Lausanne, Switzerland

Vol. 8 No. 2 1980

Ramsbotham, J.—"Solvent Formulation for Surface Coatings"; p. 113.
Sato, K.—"Internal Stress of Coating Films"; p. 143.
Hill, L.W. and Wicks, Z.W., Jr.—"Amine Solubilizers for Water-Soluble Acrylic Baking Enamels"; p. 161.
Perera, D.Y.—"Hygric Aspects of Coated Porous Building Materials"; p. 183.

Meetings/Education

Color Symposium to Feature Paper Presentations, Workshops, and Equipment Displays

A symposium on instrumentation for measuring color and appearance will be held March 24-26, 1981 at the Executive West, Louisville, Ky., under the joint sponsorship of the Federation of Societies for Coatings Technology, Manufacturers Council on Color and Appearance, and Inter-Society Color Council.

The program will be divided between general paper presentations and "hands-on" workshop sessions.

Among the featured speakers will be Dr. David Wright, of England, who will talk about the experimental origins of the 1931 CIE system of colorimetry.

Other paper presentations will focus on such topics as: color communication; industrial metamerism; colorant formulation; measurement of appearance; goniospectrophotometry; on-line color control; tinting strength assessment; color difference assessment; sample preparation; and education in the instrumental measurement of color and appearance.

There will be four workshops: three will feature equipment displays for color formulation, color measurement, and gloss and other appearance measurement; the fourth is a sample-preparation-and-presentation tutorial.

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

Attendance will be limited so that registrants will have maximum opportunity for discussions.

General program sessions are under the direction of Dennis Osmer, of CIBA-GEIGY Corp., Ardsley, N.Y., who chairs the Federation's Inter-Society Color Council Committee. Arrangements for the workshops and instrument displays are being handled by Charles Leete, Executive Director of the Manufacturers Council on Color and Appearance.

To obtain complete program and registration information, contact Federation of Societies for Coatings Technology, 1315 Walnut St., Suite 832, Philadelphia, PA 19107 (215) 545-1506.

Baltimore Society Forms "Virginia Section"

To meet the needs of local companies manufacturing paints, lacquers, printing inks, adhesives and related raw materials, the Baltimore Society has formed a "Virginia Section" which conducts its own meetings and technical presentations. Members of this group are included within the Federation of Societies for Coatings Technology.

The Virginia Section held its first meeting on October 15 in Richmond. Officers of the Baltimore Society presided and honored guests in attendance included Federation Executive Vice-President, Frank J. Borrelle and Director of Communications and Field Services, Thomas Kocis. Future meetings will be held every other month in various parts of the state.

For additional information on the group, contact C. Herbert Pund, III, Acting Secretary, Haskell Chemical Co., P.O. Box 9515, Richmond, VA 23228.

Macbeth Color Seminars Are Scheduled for 1981

Macbeth, a Div. of Kollmorgen Corp., Newburgh, NY, will again hold its seminar, "The ABC's of Color," at eight locations across the United States during the first six months of 1981. Held for the third straight year, the seminar will be divided into two one-day sessions.

The first day is devoted to lectures and practical demonstrations. The fee for the first day is $125 and includes reference materials, workbooks, and lunch. The second day, for which there is no fee, is an informal session of hands-on use of visual standards, lighting control, and instrumentation.

The locations and dates of the 1981 schedule are: Detroit, Jan. 22-23; Cherry Hill, NJ, Feb. 19-20; Columbus, OH, Mar. 19-20; Cambridge, MA, April 20-21; Chicago, Apr. 23-24; Denver, May 18-19; Los Angeles, May 21-22; and Portland, OR, June 11-12.

Participants at the 50th annual FOCUS Symposium of the Detroit Society. (Top row, left to right): Co-chairman G.M. Sastry; Society President Walter C. Stuecken; Moderator K.A. Kinzler; and Co-chairman Stephen Peng. (Second row): Elaine M. Sloan; T. John Young; Donald R. Hays; Michael A. Tersillo; and Richard L. Dalton. (Bottom row): R. Henry; Gary K. Hulburt; J. David Nordstrom; R.J. Caiola; George E.F. Brewer; and S.P. Levine

University of Minnesota Short Course to Focus on Colloids

A short course on "Colloids and Surface Science" will be held May 18–22, 1981, at the University of Minnesota Minneapolis campus. The course is designed for those working with grinding, adhesives, sealants, wetting agents, emulsions and films, pigments, powders, printing inks, and soils and clays. Lectures, laboratory demonstrations, and problem solving groups will be utilized to cover such topics as methods of measuring surface tension, flocculation of colloids, electrophoresis, colloid size, and the rheology of colloids.

Chairman of the course is Dr. D. Fennel Evans and Assisting Instructor is Dr. H.T. Davis. Both are staff members of the Department of Chemical Engineering and Materials Science, Institute of Technology of the university.

For further information, contact John Vollum, Program Director, Department of Conferences, 210 Nolte Center, 315 Pillsbury Drive S.E., University of Minnesota, Minneapolis, MN 55455.

Organic Coatings to be Subject of XVIth FATIPEC Congress

The XVIth Congress of the Federation of the Associations of Technicians in the Paint, Varnish, Lacquer and Printing Ink Industries of Continental Europe (FATIPEC) will be held from May 9 to 14, in Liège, Belgium. President of FATIPEC, A. Toussaint, has announced that the theme of the Congress will be "Evaluation and Foresight of Film Properties of Organic Coatings Through the Use of Modern Analytical Methods."

An exhibition of raw materials, finished products and materials, named FATIPEXPO, will also be featured at the Congress. This exhibit is designed to allow attendants to both learn about new developments and to establish direct contact with specialists and representatives concerning specific problems, as well as general information.

Calif. Rule 1113 Exemptions Delayed

A six-month extension of the deadline on California's Rule 1113 exemptions was voted by the South Coast Air Quality Management District's Board at its September 5 meeting.

The delay until March 2, 1981 will provide paint industry representatives and the South Coast District staff with more time to assess recent scientific information.

Representing the industry at the hearing were Al Aronow of the Sinclair Paint Co. and Michael Campilongo of National Paint and Coatings Association's legal staff.

The rule, which had been set to go into effect for the Los Angeles region on September 2, would reduce the solvent content of interior surface coatings from 350 gram/liter to 250 grams/liter.

In a related development, NPCA is moving forward with its application for a class variance in the Bay Area Air Quality Management District. It has been necessary to seek temporary relief similar to the kind provided in the South Coast District by applying for a limited variance from the Bay Area's version of the architectural surface coatings rule.

People

NL Chemicals/NL Industries has appointed **George C. Dager** to the position of Advertising Manager for the NL Chemicals operation. He recently held a field sales position in the Chicago area, marketing rheological additives and TiO_2 to the paint, ink, sealant, adhesive, polyester, and grease industries. Mr. Dager is a member of the St. Louis Society.

Fred King has been appointed Production Manager for the Consumer Products Div. of McCloskey Varnish Co. of the West, based in Los Angeles. Mr. King will be responsible for all processing, manufacturing, packaging, and distribution for the firm. He is a member of the Los Angeles Society.

James C. Healy has been named Director of Research and Development, elastomers, latex and additives, and specialty chemicals for B.F. Goodrich Chemical Group in Cleveland. He had served as Director of elastomers research and development since 1975.

American Hoechst Corp., Somerville, NJ, has named **Peter Blum** President of its Industrial Chemicals Div. Mr. Blum previously served as Director of the Hoechst AG Corporate Marketing Dept. in West Germany. He succeeds **Dieter Poetzsch**, who has been elected President of Hoechst Canada, Inc., in Montreal.

J. Barry Smith has joined Deeks & Company, Cincinnati, as Vice-President, Marketing. He previously served as Marketing Manager for Reichard-Coulston and as Marketing Director for Guest Industrials Limited, Kent, England.

Buckman Laboratories, Inc., Memphis, has appointed **Thomas White Macy** to the position of Area Sales Representative. Responsible for distributor sales in the northeastern U.S., Mr. Macy will be based in Concord, MA. He is a member of the New England Society.

Harland B. Kinzley has been appointed Product Manager of Coil Coatings for Cook Paint and Varnish Co., Kansas City. He will bring to the position more than 30 years of managerial experience in coil coating marketing and sales.

G.C. Dager

F. King

Richard M. Podhajny has been appointed Technical Director of the Printing Ink Div., of Borden Chemical, in Cincinnati, OH. Dr. Podhajny will assume responsibility in the research and development areas for coatings and printing inks.

Albert J. Moreau has been named Vice-President of Western Region Sales for Armstrong Containers, Inc. of Westchester, IL. In addition to creating a strong marketing base in the western U.S., Mr. Moreau will continue to be National Sales Manager for the firm.

Mark Stoops has joined Geocel Corp., Elkhart, IN, as Product Manager. In his new position, he will be responsible for new products and sales promotions on established lines.

Lilly Industrial Coatings, Indianapolis, IN, has appointed **Daniel H. Monroe** Director of Corporate Purchases to serve in a supervisory capacity of purchasing for all the company's plants. Mr. Monroe is a Past-President of the Piedmont Society.

In a series of appointments, Kerr-McGee Chemical Corp., Oklahoma City, has named **E. C. O'Conner**—Director of Marketing; **D.F. Schiesz**—Director of Manufacturing; and **I.L. Denny**—Manager of New Products, Pigments Div.

R.M. Campbell, Jr., has been named Executive Vice-President, Domestic Operation, of Manchem Limited, which has recently been formed as a separate corporation to handle the marketing, distribution, and production of the products of Manchem and affiliated companies. Mr. Campbell will be located at the headquarters office in Princeton, NJ.

R.M. Podhajny

J.H. Owens

John H. Owens has been promoted to General Manager, Color Operations, for the Thermoplastics Div. of Ferro Corp. in Cleveland, OH. In this position, Mr. Owens will assume responsibility for the manufacturing and marketing of the division's complete line of thermoplastic colorants and color services.

The American Society for Testing and Materials (ASTM) has named **William T. Cavanaugh** President. He last served as Managing Director and Chief Executive Officer for the Society.

Edward T. Baker has been appointed Senior Sales Engineer for the Plating Div. of M&T Chemicals, Inc. He will be headquartered in the company's Rahway general offices and will handle the sale of plating products in Connecticut and Rhode Island.

William P. Shilleci has joined Engelhard Minerals & Chemicals Div. as Sales Representative for sorbents and chemicals in the Catalysts and Chemicals Group. Based in Atlanta, Mr. Shilleci will assume responsibility for established trade relationships and for the development of new accounts for the division's activated bauxite and attapulgite products.

Harmon Colors Corp., Hawthorne, NJ, has appointed **Brian Stott** Sales Representative for the Midwest region. He will handle sales of the firm's pigments in the Michigan and northern Ohio areas.

Peter W. Shidler has been named National Accounts Manager for E Z Paintr Corp. of Milwaukee. He previously served as Area Manager for the Midwestern region.

S.L. Kopald, Jr. has been elected Corporate Vice-President and General Manager of Witco Chemical Corp.'s Humko Chemical Div. In charge of Humko's operations prior to its recent acquisition by Witco, Mr. Kopald will continue in that capacity. He had served as President of the division for the past seven years.

Rafael G. Aviles and **William A. Harvey, III,** have joined Rohm and Haas Co.'s Spring House (PA.) Laboratories. They will be responsible for scientific research in the industrial coatings field.

Dr. Roy T. Gottesman has been promoted to Vice-President, Environment & Regulatory Affairs of Tenneco Chemicals, Saddle Brook, NJ. Dr. Gottesman most recently served as Director of Chemical Development and Director of Environment & Regulatory Affairs for the company.

Spencer Kellogg Div. of Textron Inc., Buffalo, NY, has appointed **Paul C. Stievater** to the position of Technical Sales Manager. In this newly created position, he will be responsible for divisional technical training and all promotional and advertising activities.

W.R. Grace & Co. has promoted **Peter I. Kiziuk** to Western Regional Sales Manager for the Organic Chemicals Div. Previously, Mr. Kiziuk served as Senior Chemical Salesman for the mid-Atlantic states.

Robert D. Athey, Jr. has joined Swedlow, Inc. as Manager of New Programs in the Research and Development department. He is a member of the Pittsburgh Society.

Lester P. Johnson has been promoted to Research Chemist in the Packaging Group of DeSoto, Inc., Des Plaines, IL.

Frank Rueter has been appointed to the position of Vice-President, Marketing, for Zorelco Ltd., Cleveland, OH. In this position, he will retain overall responsibility for sales and advertising and will assume responsibility for the planning and development of world-wide distribution of the company's measuring and testing intrumentation.

Obituary

Jean P. Teas, Past-President of the Federation, died in Cape Elizabeth, Maine, Sept. 26, following an extended cancer illness. He was 69 years old.

Retiring from The Flood Co., Hudson, Ohio, as Technical Director in 1976, Mr. Teas relocated to Maine to live with his son Scott, who survives. Prior to joining The Flood Co. in 1956, he worked in engineering and administrative positions with Union Carbide Corp. Olin-Mathieson Corp., and Thompson, Ramo, Woolridge, Inc.

Mr. Teas was a graduate of Cornell University and did graduate work at Case Institute and Kent State University. During World War II he served with the U.S. Army as a Captain in the Ordnance Dept. and received the Legion of Merit Citation for field testing and combat instruction of special ordnance material.

Mr. Teas served as President of the Federation in 1971–72, and was the first chairman of the Federation's Technical Information Services Committee in 1967. He was also a former trustee of the Paint Research Institute and was an Honorary Member of the Cleveland Society.

In addition to his son, he is survived by a daughter, Mrs. Debbie Lass, of Grosse Pointe, Mich. Mr. Teas' wife, Mrs. Hazel Scott Teas, died in 1973.

Literature

Defoamer

A new technical bulletin features a defoamer for water-reducible coating systems. This product can be used in amine-neutralized air or baking alkyd systems, oil-free polyester, and epoxy coatings. For additional information, write Byk Mallinckrodt, 734 Walt Whitman Rd., Melville NY 11747.

Chromatography Newsletter

The newest issue in a continuing series of Chromatography Newsletters is now available. Included in the issue are ten articles. Free copies may be obtained by requesting Order No. CHN-16, from the Perkin-Elmer Corp., Instrument Div., Main Ave., Mail Station 12, Norwalk, CT 06856.

Methyl Cellulose

A newly developed brand of methyl cellulose ethers has been introduced in literature. These ethers offer a broad range of applications requiring organic solubility, film forming, or controlled viscosity. Further information concerning Culminal ® methyl cellulose may be obtained from the Water Soluble Polymers Div., Henkel Corp., 4620 West 77th St., Minneapolis, MN 55435.

Powder Coating Resins

Specifications for powder coating resins and additives that offer energy savings are now available in a newly published leaflet. These resins and additives are intended for use with paints, varnishes, enamels, lacquers, industrial primers, sealing compounds, and printing inks. For further information, contact American Hoechst Corp., Industrial Chemicals Div., Wax, Resins & Coatings Dept., P.O. Box 2500, Somerville, NJ 08876.

Batch Correction

A batch correction system which is based on Kubelka-Munk relationship of absorption and scattering to compute the rate of change of each product color with changes in concentration of each colorant is the topic of a new product brochure. Additional information on this colorimeter-based microprocessor-controlled system is available from Hunter Associates Laboratory, Inc., 11495 Sunset Hills Rd., Reston, VA 22090.

Flatting Agents

New information on silica flatting agents is available in a recently published brochure. In addition to a discussion of recent developments for improved low gloss coatings, applications and specific silica grade recommendations are provided. For a copy of PA-40, write Industrial Chemicals Dept., W.R. Grace & Co., Davison Chemical Div., P.O. Box 2117, Baltimore, MD 21203.

Specialty Asphalts

Specialty asphalts suited to a range of design uses are described in a recently published four-page color brochure. Among the properties discussed are resistance to corrosion, moisture, extreme heat or cold, adhesion and low cost. Copies of "Witco Specialty Asphalts: A Design Material with Hundreds of Uses" may be obtained from Witco Chemical Corp., Pioneer Div., 802 Ash St., Lawrenceville, IL 62439.

Urethane Resins

Information has been released which details three new water-reducible urethane resins developed for use in sealers, clear varnishes, and stains for wood floors and furniture. Designed for rapid drying, high gloss finish, and hardness/abrasion resistance, each resin features distinct performance characteristics. Additional information may be obtained by writing Spencer Kellogg, Div. of Textron, Inc., P.O. Box 807, Buffalo, NY 14240.

Low Viscosity Adapter

A redesigned accessory for measurement of ultra-low viscosity materials is the subject of technical literature. This adapter consists of a precision cylindrical spindle rotating inside an accurately machined tube. More information can be obtained from Brookfield Engineering Laboratories, Inc., Dept. NR 34, 240 Cushing St., Stoughton, MA 02072.

Urethane Latex

A light-stable urethane latex that can be frothed into a foam or employed as an energy-efficient saturant or binder has been introduced. This water-based product is suited for frothing foam used as an interlayer or coating for textiles, paper and nonwovens, as well as urethane, vinyl, and other flexible plastic materials. Further information on Witcobond W-290H may be obtained from Witco Chemical Corp., Organics Div., 400 N. Michigan Ave., Chicago, IL 60611.

Tristimulus Colorimeter

Newly published literature highlights the applications and features of a tristimulus colorimeter system. Features include 40 product standard memory locations, instantaneous calibration, and automatic zero compensation. For additional information, contact Gardner Laboratory Div., Pacific Scientific P.O. Box 5728, 5521 Landy Lane, Bethesda, MD 20014.

Infrared Spectrophotometer

A recently released applications bulletin describes the use of an infrared spectrophotometer for the measurement of emission spectra obtained from samples at moderate temperatures. Results are ratioed against those from a black body to calculate emissivity by using a newly developed data station. For a copy of Order No. IRB-75, write the Perkin-Elmer Corp., Main Ave., Mail Station 12, Norwalk, CT 06856.

Block Polymers

Block polymers of ethylene oxide and propylene oxide are the subject of a new technical bulletin. Used as emulsifiers, demulsifiers, dispersants, wetting agents, and rinse aids, these polymers can be applied in petroleum production, paints, cleaning compounds, and textile processing. For complete details, contact Hodag Chemical Corp., 7247 N. Central Park Ave., Skokie, IL 60076.

Latex Paint Thickener

A 24-page booklet on a nonionic, water-soluble polymer for latex paint thickening is now available. The booklet includes a discussion of four high-range viscosity grades of the polymer used for paint thickening, a table showing aqueous solution viscosities, and a thickener selection guide. Copies of booklet F-41612B are available from Union Carbide Corp., Coatings Materials Div., Dept. JLS, 270 Park Ave., New York, NY 10017.

Corrosion Engineer's Reference

The National Association of Corrosion Engineers (NACE) has published "The NACE Corrosion Engineer's Reference Book," a compilation of researched material, which gives the physical, mechanical, chemical, and performance properties of metals and nonmetals used in certain environments and industries. Subject areas include conversion tables, physical and chemical data, engineering data, metals and alloys, and nonmetallic materials and coatings. The cost of the book is $25 for NACE members, $30 for nonmembers. For additional details, contact Amy Montoni, NACE, P.O. Box 218340, Houston, TX 77218.

Infrared Option

Newly published information describes an infrared measurement option for a color spectrophotometer. This option extends the instrument's standard range of 380–700 nanometers to 380–1000 nanometers. Complete details are available from Mr. Dennis Tompkins, Color Products Marketing Manager, Diano Corp., P.O. Box 1005, 8 Commonwealth Ave., Woburn, MA 01801.

Urethane Dispersions

Two colloidal urethane dispersions that can be formulated into highly protective finishes for difficult-to-adhere substrates, such as vinyls, glass, metals, and woods are discussed in a new product bulletin. For details, write Witco Chemical Corp., 277 Park Ave., New York, NY 10017.

Epoxy Resin

A four-page technical bulletin details a solid bisphenol A/epichlorohydrin epoxy resin designed for thin-film functional and decorative epoxy powder applications. Included are storage and handling information, specifications, and properties data. For a copy of Technical Bulletin SC: 235-80.202, write Shell Chemical Co., Manager/Chemical Communications, One Shell Plaza, Houston, TX 77002.

Textured Compliance Finish

Literature has been released describing a water-borne, textured business machine finish suitable for application to both metal and plastic substrates, which meets present EPA requirements. For additional information, contact Whittaker Corp., Coatings & Chemicals Group, 10880 Wilshire Blvd., Los Angeles, CA 90024.

Blast Primers Standards

A report prepared by the Paint Research Institute provides performance standards for blast primers for both land and marine applications. The work also points the way to a code of practice for the satisfactory use of blast primers and to further improvement in these paints. Copies may be obtained from Mrs. Caroline Veitch, Publications Coordinator, Paint Research Association, Waldegrave Rd., Teddington, Middlesex England TW11 8LD.

Differential Scanning Calorimeter

A new differential scanning calorimeter designed for ease of operation and based upon the patented DSC principle is described in newly published literature. The instrument offers true power compensation for high accuracy, as well as excellent baseline performance. For further information, contact Perkin-Elmer Corp., Main Ave., Mail Station 12, Norwalk, CT 06856.

Diaphragms

Two additions to a line of air-operated double diaphragm pumps have been introduced. Available in three sizes, these pumps have capacities from less than one gallon per-minute to over 6000 gallons per-hour. Additional information may be obtained from Wilden Pump and Engineering, 22069 Van Buren, Colton, CA 92324.

Batch Correction

Literature has been released which describes a new system for automated batch corrections. Designed for pigment applications utilizing colorimeters for color measurement, this system is compatible with X Y Z tristimulus measurement data. For more information, contact Applied Color Systems, Inc., P.O. Box 5800, U.S. Hwy. One, Princeton, NJ 08540.

Flexible Foam Intermediates

In a new, 68-page booklet, intermediates for flexible urethane foam are described. The range of products includes polyether polyols, polymer polyols, silicone surfactants, blowing agents, catalysts, and a group of specialty materials. The brochure includes tables of typical properties and charts that show the effect of temperature on specific gravity and viscosity. To obtain a copy of booklet F-45581A, contact Union Carbide Corp., Silicones and Urethane Intermediates Div., Dept. JSW, 19th Fl., 270 Park Ave., New York, NY 10017.

Defoamer

A new 100% active, silicone oil-free anti-foaming agent has been introduced in recent literature. Designed for the reduction or elimination of foam in the production and application of water-based coatings, this product is said to be especially effective in vinyl acrylic and acrylic emulsions. For further details, contact Interstab Chemicals, Inc., Technical Service Dept., 500 Jersey Ave., P.O. Box 638, New Brunswick, NJ 08903.

Wet Film Thickness Gauge

A new instrument which measures the wet film thickness of coatings or films applied to various size and shape surfaces is the subject of a recent brochure. For information, contact Paul N. Gardner Co., Inc., 218-D Commercial Blvd. Lauderdale-by-the-Sea, FL 33308.

Copper and Zinc Naphthenate

Information is now available which details the advantages of a new water-dispersible copper and zinc naphthenate. For complete details, write Jim Leopold, Mooney Chemicals, Inc., 2301 Scranton Rd., Cleveland, OH 44113.

Matting Agent

A newly published brochure has been prepared which discusses a matting agent for the varnish industry. For further information, write American Hoechst Corp., Industrial Chemicals Div., Wax, Resins, & Coatings Dept., Route 202-206 North, Somerville, NJ 08876.

Colors

Six new colors in a line of industrial spray coatings are the subject of recent literature. These coatings are designed for use on metal, wood, concrete, and masonry surfaces. For Publication 7928, contact Rust-Oleum Corp., 11 Hawthorn Pkwy., Vernon Hill, IL 60061.

Data Station

A new full-color brochure describes data stations for "computer-aided chemistry." These data stations are featured as either stand-alone computers to speed interpretation of instrument data, or as terminals in a distributed processing network for direct access to a host system's data base. For a copy of Order No. L-650, write the Perkin-Elmer Corp., Main Ave., Mail Station 12, Norwalk, CT 06856.

Corrosion Problems

The National Association of Corrosion Engineers (NACE) has published a new book, "Proceedings: Materials and Corrosion Problems in Energy Sources," which details corrosion problems in a variety of energy sources industries including petroleum, natural gas, coal, solar, nuclear, geo-thermal, ocean-thermal, and wind energy. This publication is a compilation of seventeen papers presented at the NACE-sponsored "Materials and Corrosion Problems in Energy Systems Symposia" held during the February 1980 Energy Sources Technology Conference. Cost of the book is $30 and papers may be purchased individually for $5. For information, contact NACE, P.O. Box 218340, Houston, TX 77218.

Antifoams

A technical bulletin provides complete details on a basic group of antifoams for use with almost any type of adhesive or emulsion coating. Special-purpose formulations are also given to overcome specific foam problems. For a copy, contact Hodag Chemical Corp., 7247 N. Central Park Ave., Skokie, IL 60076.

Book Review

MACROMOLECULES
An Introduction To Polymer Science

Written by
Bovey and Winslow

Published by
Academic Press
1979 (540 pages) $29.50

Reviewed by
Dr. Henry K. Wren
Faribo Industries, Inc.
Faribault, Minnesota

Here is a very scholarly book on polymer chemistry which I feel is second only to Flory's. While somewhat shorter than that work, it does give an indepth treatment of the subject, beginning with an introduction to polymer science which is coupled with a brief history of the various types of polymers.

The second chapter deals with the formation of macromolecules. The various methods of polymerization utilized in the formation of macromolecules are thoroughly covered, along with molecular weight, its distribution and control. Polyfunctional monomers and gel points are also covered in depth. The factors involved in step-reaction polymerizations are enumerated. Some three-stage reactions, particularly those relating to formaldehyde in resin formation, are given a brief but thorough treatment.

Spectroscopic conformation of macromolecules forms the beginning of the next section. Various spectrophotometric methods are explored. The areas of stereochemical configurations and geometrical isomerism are concisely covered, while ring openings, branches and defect structures, chains, and chain conformation are dealt with in detail.

Chapter 4 covers macromolecules in solution, and a discussion of the Flory-Huggins theory on solubility of macromolecules is given a good overview. Molecular weight measurements are discussed quite extensively, as is viscosity as a method of ascertaining molecular weight. Information about polyelectrolytes, as well as gel permeation chromatography and its relation to molecular weight determinations, is provided.

Chapters covering the crystalline or solid state of macromolecules and the physical behavior of macromolecules are quite informative. The physical behavior section includes three main areas of consideration: the rubbery state, the glassy or crystalline state, and transport. Additional chapters include biopolymers and reactions of macromolecules.

The book is well written and contains numerous references. I sincerely recommend this volume to all students and practicing polymer chemists.

Letters to the Editor

Reader Objects to JCT Cover

TO THE EDITOR:

Your cover on the July 1980 issue of the JCT and the comments on page 7 of the same issue suggest that the Federation is made up of Societies in the United States.

I suggest that the Canadian membership of around 600 people have been slighted to say nothing of the Mexico and Birmingham (England) Societies.

At the very least you could have said, "Paint North America Beautiful." But then, why not "The World."

I realize that it is the NPCA which has launched this campaign but I think that a few comments by FJB which echo my thoughts might have been in order.

RICHARD P. STEWART
President-Elect
Pacific Northwest Society
Burnaby, B.C., Canada

There was no intent to slight the Federation's valued members from the Societies in Canada, the U.K., and Mexico. Our objective was to lend a cooperative hand to the NPCA in its promotional effort to boost consumer use of the industry's products . . . wherever they may be sold. Our apologies to Mr. Stewart and the Pacific Northwest Society.—FJB

CLASSIFIED ADVERTISING

Coming Events

FEDERATION MEETINGS

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

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

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

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

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

PAINT RESEARCH INSTITUTE MEETING

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

SPECIAL SOCIETY MEETINGS

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

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

(Mar. 11–13)—Southern Society. 45th Annual Meeting. 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.

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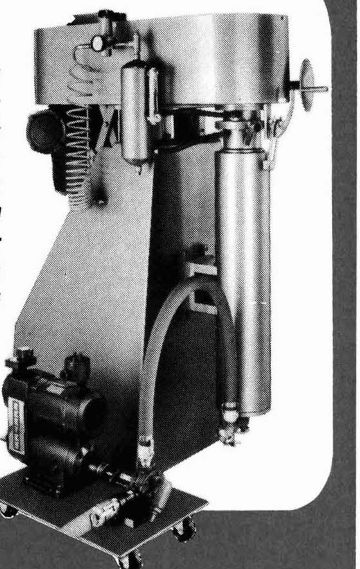
(Mar. 10–12)—Southern Society Annual Meeting. Savannah, Ga.

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

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



OTHER ORGANIZATIONS

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

(Jan. 7–9)—"Thermoanalytical Methods" Short Course. The Center for Professional Advancement, Central NJ. (The Center for Professional Advancement, P.O. Box H, East Brunswick, NJ 08816).

(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).

(Feb. 9–11)—Inter-Society Color Council. Williamsburg Conference, Williamsburg, VA. (Dr. Fred W. Billmeyer, Secretary, ISCC, Rensselaer Polytechnic Institute, Troy, NY 12181.)

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

(Apr. 6–10)—National Association of Corrosion Engineers "Corrosion/81." Sheraton-Centre and Hotel Toronto, Toronto, Ontario, Canada. (Conference Coordinator, NACE, P.O. Box 218340, Houston, TX 77218).

(Apr. 7–8)—NACE T6 Symposium on Underfilm Corrosion. Sheraton Centre Hotel, Toronto, Canada. (National Association of Corrosion Engineers, 1440 South Creek, Houston, TX 77084.)

(Apr. 27–28)—Inter-Society Color Council Annual Meeting. Roosevelt Hotel, NY. (Dr. Fred W. Billmeyer, Secretary, ISCC, Rensselaer Polytechnic Institute, Troy, NY 12181.)

(Apr. 28–30)—OCCA-32. Oil and Colour Chemists' Association 33rd Annual Technical Exhibition. Cunard International Hotel, London, England. (The Director and Secretary, OCCA, Priory House, 967 Harrow Rd., Wembley, Middlesex HAO 2SF, England.)

(June 14–17)—Dry Color Manufacturers' Association Annual Meeting. The Greenbriar, White Sulphur Springs, WV. (P.L. Lehr, DCMA, Suite 100, 1117 North 19th St., Arlington, (Rosslyn) VA 22209.)

(June 17–20)—Oil and Colour Chemists' Association Conference, "Alternative Technologies in Coatings." Beaufort Hotel, Bath, England. (The Director and Secretary, OCCA, Priory House, 967 Harrow Rd., Wembley, Middlesex HAO 2SF, England.)

(Sept. 13–16)—Canadian Paint Manufacturers Association. Four Seasons Hotel, Vancouver, B.C. (Lydia Palazzi, Canadian Paint Manufacturers Assn., 2050 Mansfield, Montreal, Que., Canada H3A 1Y9.)

(Sept. 20–25)—4th Congress of the Association Internationale de la Colueur, "COLOR 81." International Congress Centre (ICC), Berlin (West), Germany. (Prof. Dr. Heinz Terstiege, (AIC COLOR 81), Bundesanstalt fur Materialprufung (BAM), Unter den Eichen 87, D-1000 Berlin 45, Federal Republic of Germany).

(Oct. 13–15)—Association for Finishing Processes of the Society of Manufacturing Engineers. "Finishing '81" Conference and Exposition, "Economics, Compliance, and Energy." Cobo Hall, Detroit, MI. (William J. Yeates, Executive Director AFP/SME, One SME Dr., P.O. Box 930, Dearborn, MI 48128.)

(Dec. 9)—American Society for Testing and Materials Symposium on Selection and Use of Wear Tests for Coatings. Phoenix, AZ. (ASTM, 1916 Race St., Philadelphia, PA 19103.)

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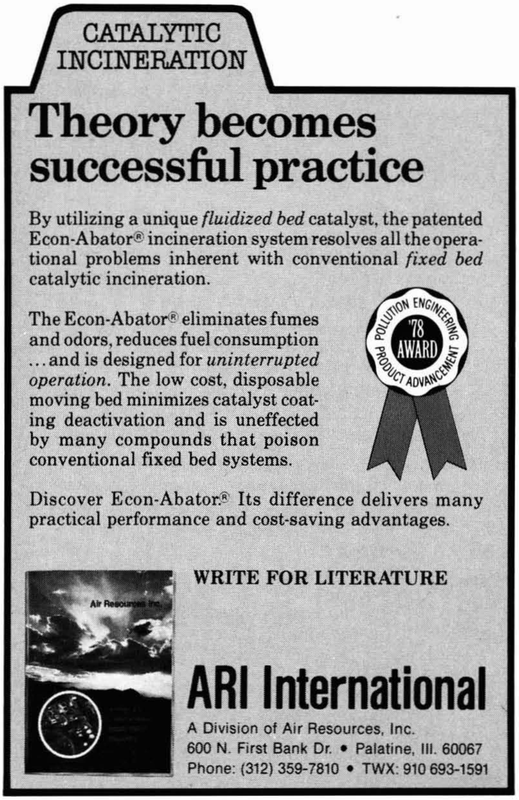
(Feb. 7–10)—Inter-Society Color Council. Williamsburg Conference, Williamsburg, VA.

(May 9–14)—XVIth Congress of FATIPEC, Brussels, Belgium.



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