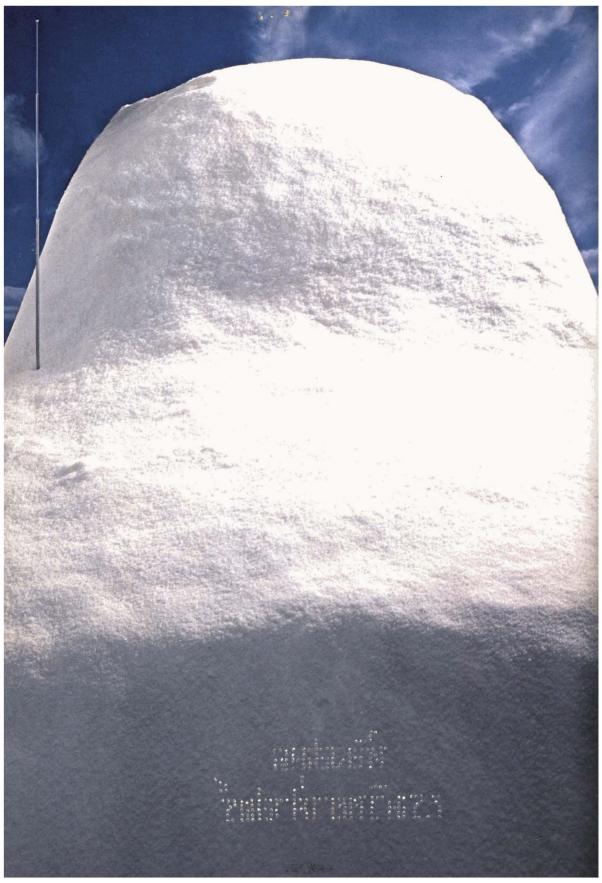
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October 1984

JOURNAL OF COATINGS TECHNOLOGY

Film Formation and Rheology of Powder Coatings



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Section 17

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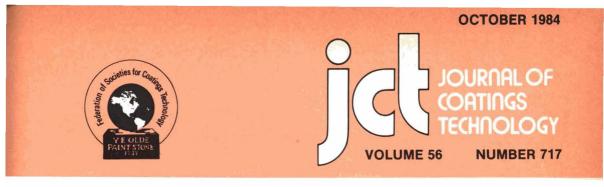
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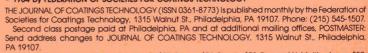
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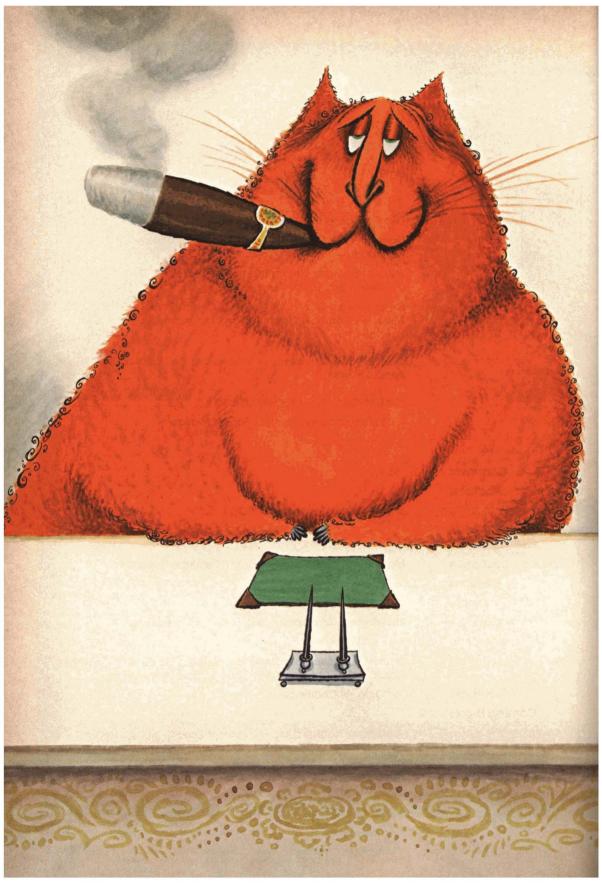
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Journal of Coatings Technology

Comment

55 Across

After receiving the August JCT, I reminisced and checked the same publication (Official Digest) 25 years ago.

What a difference-and not only in size!

In the late 50's, the OD featured several technical papers and only a smattering of other editorial (Federation/Society news). There were few photos and little color. The only change on the front covers was the date/volume line beneath the five-inch Federation seal.

Time and "times" have brought many changes, all on the plus side. The creative, colorful, and clever front covers of the JCT have been acclaimed to be among the best of industry publications. Inside are (still) the finest technical material from authors around the world, and these departments: Comment Page, Abstracts, Government and Industry, Letters, Book Reviews, Literature, Meetings/Education, Coming Events, and lots of color everywhere.

Since September 1980 we have been fortunate to be the exclusive publishers of "Humbug from Hillman," the last word in many issues. Humbug is even more popular than Herb realizes. He has given our readers such a refreshing collection of anecdotes, quips, quotes, and quacky stories that I, for one, hope that he and Libby enjoy their retirement in Vermont for many years, and continue the writing and cooking which have made them famous.

The latest addition to the JCT is—of all things—a crossword puzzle, created by Earl Hill, of Erie, PA. Most of the words that will fill the blocks can be found in the Federation's Paint/Coatings Dictionary. (Thank you, Stan LeSota, and the Philadelphia Society). And thank you, Earl, for CrossLinks.

55 across in Earl's August puzzle was "has 'em all."

And that's how we feel about the JCT-has 'em all.

Except maybe a comic strip about "Joe, the Paint Chemist." Any cartoonists out there?

Quart & Fande Frank J. Borrelle.

Executive Vice-President

Ethylene vers glycol ethers Here are the fac

THE FACT IS that the Environmental Protection Agency has filed an Advanced Notice of Proposed Rulemaking (ANPR) on four ethylene-based glycol ethers and acetates: EGME, EGEE, EGMEA, and EGEEA. The ANPR was the result of toxicological tests on animals that indicate ethylene-based glycol ethers and acetates, even at relatively low levels, present a substantial risk of reproductive toxicity to both males and females.

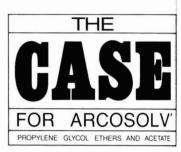
The ANPR gives notice that the EPA may impose a variety of restrictions including a ban on the use of these products.

THE FACT IS that toxicological research on ARCO Chemical Company's ARCOSOLV* propylene

glycol ethers and acetates, even at much higher exposure levels than the ethylene-based glycol ethers and acetates, show no evidence of reproductive organ toxicity.

THE FACT IS that even before the EPA acted, paint, cleaner and ink manufacturers in growing numbers were already moving away from ethylene-based ethers and acetates toward propylene-based products. Many have found ARCOSOLV solvents can directly replace the ethylene-based products they had been using.

THE FACT IS that in price and performance ARCOSOLV propylene glycol ethers and acetate are very competitive with ethylene-based products.



The CASE for ARCOSOLV Solvents

In view of these facts, ARCO Chemical Company can present a strong CASE for a switch from ethylenebased solvents to the ARCOSOLV propylene glycol family of products. Our CASE is this:

Typical Physical Pro	perties				
ARCO Chemical Nomenciature	Chemical Name	Molecular Weight	Evaporation Rate BuAc = 100	Solubility	
Glycol Ethers		Milliolation Michael	Bunchas In Design of DESTINATION	B Ministerielderidaut	A BULLIST
ARCOSOLV* PM	Propylene Glycol Methyl Ether (PGME)	90.1	66	10.4	x
ARCOSOLV® · DPM	Dipropylene Glycol Methyl Ether (DPGME)	148.2	2	9.6	œ
	Ethylene Glycol Methyl Ether (EGME)	76.1	47	10.8	œ
	Ethylene Glycol Ethyl Ether (EGEE)	90.1	32	9.9	œ
	Ethylene Glycol Butyl Ether (EGBE)	118.2	6	8.9	x
Glycol Ether Acetate	S				
ARCOSOLV* PMAC	Propylene Glycol Methyl Ether Acetate (PGMEA)	132.2	34	9.2	18.5/5.6
	Ethylene Glycol Ethyl Ether				
	Acetate (EGEEA)	132.2	20	9.4	23.8/6.5

and acetates. s: you decide.

liost

Because they are physically and structurally similar to E series products, ARCOSOLV solvents are a straightforward and economical eformulation choice. But to help ase the expense of reformulation, ARCO Chemical Company is announcing an important Temporary Reformulation Allowance (TRA) to be in effect over the next twelve nonths. The allowance will help our ustomers with the cost of the thangeover from E series to aRCOSOLV solvents.

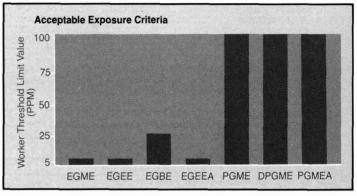
he TRA will be augmented by a echnical service program offering eformulation assistance where it is eeded. Ask us for details on these rograms.

Availability

Because of the company's strong raw materials position in propylene oxide (at 1.6 billion pounds per year, we are one of the nation's largest producers) and methanol (1.3 billion pounds per year), ARCO Chemical Company is able to serve the needs of the marketplace. And we have been producing propylene glycol ethers and acetate since 1981.

Safety

Considerable testing of propylenebased glycol ethers and acetates has demonstrated that P series products have much safer toxicological profiles than E series products. Research on propylene glycol methyl ethers has demonstrated no evidence of reproductive organ



TLV's of 5 ppm for EGME, EGEE and their acetate derivatives recommended by the American Conference of Government Industrial Hygienists (ACGIH) on June 10, 1982. Several glycol ether producers are recommending 2–5 ppm on EGME and 5 ppm on EGEE and EGEEA. ANPR could result in more severe restrictions. There is no standard established by ACGIH for PGMEA. Based on a comparison with PGME TLV a comfort level of 100 ppm would be recommended for PGMEA.

toxicity even at high exposure levels (up to 3,000 ppm PGME).

Effectiveness

ARCOSOLV propylene glycol ethers and acetate offer solvency power for a wide range of substances, a good range of evaporation rates, and excellent coupling ability. The chart at the left shows some of the important properties of ARCOSOLV solvents and how they compare with ethylene-based products.

Facts Make the CASE for ARCOSOLV Solvents

In Cost, Availability, Safety and Effectiveness, the case for ARCOSOLV ethers and acetate is a strong one. For more information—plus details on our Temporary Reformulation Allowance—call ARCO Chemical Company today, toll-free, at 1-800-354-1500. In Pennsylvania call 1-800-292-1500.

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Abstracts of Papers inThis Issue

FILM FORMATION AND RHEOLOGY OF POWDER COATINGS-P.G. de Lange

Journal of Coatings Technology, 56, No. 717, 23 (Oct. 1984)

Film formation and flow of thermosetting powder coatings are reviewed as functions of the two main parameters, namely, surface tension and melt viscosity.

The influence of flow additives, stoving temperature, stoving time, rate of heating during stoving, particle size, pigment concentration, and degree of pigment dispersion on these parameters is discussed. It is indicated that, in order to give a powder coating its optimum film flow, the powder should have minimum melt viscosity, lowest reactivity, and maximum surface tension, and the highest practicable heating rate should be used. Furthermore, particle size should be as small as possible, a high degree of pigment dispersion should be realized during manufacture, and pigment volume concentration should be lower than 20%.

It is shown, that very good flow can only be obtained, when the yield value in the shear stress/shear rate diagram (rheogram) of a molten coating powder (at stoving temperature) is less than 3 Pa. Finally, attention is paid to cluster formation and entrapped air, which both interfere with film formation. Ways are indicated to prevent these typical phenomena.

FLAMMABILITY OF CHLORINATED HYDROCARBONS AND HYDROCARBON ADMIXTURES—H.A. Wray

Journal of Coatings Technology, 56, No. 717, 37 (Oct. 1984)

Some of the misconceptions concerning the flammability properties of chlorinated hydrocarbons and their admixtures with aliphatic or aromatic hydrocarbons were investigated. Reported misunderstandings of the operation of the Setaflash Tester and misinterpretations of the results obtained from it were studied. Although some chlorinated hydrocarbons do not have a flash point in a standard flash point apparatus, they can burn under certain conditions. A flash point occurs only when a flame is propagated over the surface of a liquid. The presence of a blue halo or a greenish-yellow flame enlargement around the ignition flame should not be interpreted as a flash point. Admixtures of hydrocarbons and chlorinated hydrocarbons may have no flash point, or a lower or higher flash point than the hydrocarbon alone, depending on the composition of the admixture.

Furthermore, the need to follow the standard method for operating the Setaflash is emphasized to prevent the quenching effect of combustion or decomposition products developed during a flash within the tester.

EFFECT OF PIGMENTATION ON INTERNAL STRESS IN LATEX COATINGS—D.Y. Perera and D. Vanden Eynde

Journal of Coatings Technology, 56, No. 717, 47 (Oct. 1984)

The internal stress (S) in filled latex coatings was studied. The formulations investigated consisted of single filler systems (one binder/one filler) and mixtures of fillers. The experimental results show that the development of S depends on pigmentation (pigment volume concentration (PVC), type of filler).

With increasing PVC, for PVC < CPVC (critical pigment volume concentration) S increases, and for PVC > CPVC, S decreases. The CPVC-values obtained from S-measurements are in good agreement with those obtained by other methods. The shape of the curve describing S versus PVC was attributed to relaxation processes (important at low PVC's but restricted at higher PVC's). Close to or above the CPVC, relief processes can occur and affect the coating characteristics. The magnitude of S-values is mainly determined by the way the filler/binder interaction (reinforcing) affects the elastic modulus, one of the S-components.

Mathematical relationships were established to predict approximately the S and the CPVC for paints containing a mixture of fillers.

ENVIRONMENTAL CONTROL OR COATINGS COMPO-SITION AS A MEANS FOR REDUCING CATHODIC DELAMINATION OF ORGANIC COATINGS—H. Leidheiser, Jr., et al.

Journal of Coatings Technology, 56, No. 717, 55 (Oct. 1984)

Described are observations which indicate that environmental variables change the rate of cathodic delamination. The rate of delamination was observed for both a polybutadiene coating and a fluorocarbon coating on abraded steel substrate. The conclusion was reached that since the coating that adjoins the metal is the environment in which the delamination occurs, it is this environment that the coatings formulator can control.

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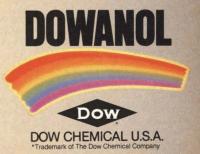
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(Continued on page 16)

Journal of Coatings Technology

GLOSS O MEASURE owder coating hardeners or high-gloss to dead-mat coatings – ithout additives

shion trends and many application ditions call for different degrees gloss – of powder coatings, too. selecting one or a combination tur special hardeners B 31, B 55, 8 or B 73, coatings, from highss to mat, can be achieved withtadditives for epoxy powders or ends (hybrids). And this with good an and pigment compatibility.

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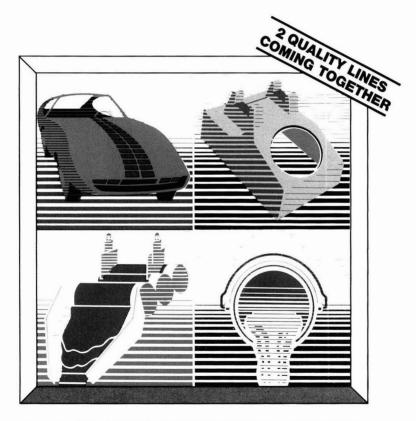
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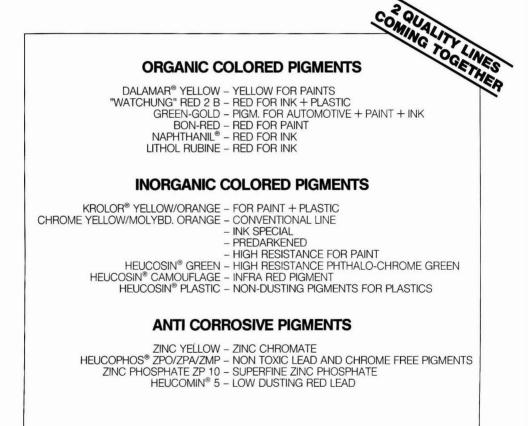
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SRI International Predicts Bright Outlook for TiO₂ Minerals and Pigments

Most companies that produce titanium minerals and pigments (TiO₂) can expect better times, according to a new multiclient study by SRI International, Menlo Park, CA.

In the study, entitled, "Outlook for TiO₂," SRI foresees a balance in mineral supply and demand, with likely pigment plant operating rates of 85 to 90% worldwide. With increases in demand of 2 to 4% per year after 1987, new mineral operations and TiO₂ pigment plants will be needed to meet anticipated demand before 1992, according to industrial economist Alan Ferguson.

The SRI forecasts come as good news to titanium minerals and pigments producers. The past five years have been difficult for such companies, just as they have been for many other minerals and

Jacksonville Site of SCM's New Application Development Lab

Construction of a new application development laboratory in Jacksonville, FL, has been announced by SCM Organic Chemicals Group, of SCM Corp. The million-dollar facility, to be shared by SCM Specialty Chemicals and SCM's wholly-owned subsidiary. Sylvachem Corp., will contain approximately 10,000 square feet, and will be furnished with state-of-the-art equipment for the adhesives and plastics industries.

SCM Specialty Chemicals provides individualized contract research, custom synthesis and research chemicals, and produces a line of silanes, terpenes, and organoflourines which are marketed to the coatings, adhesives, foundry, composite materials, and electronic industries.

Syvachem Corp., a supplier of tall oil-based rosin tackifiers used primarily in hot melt and pressure sensitive adhesives, markets a line of upgraded rosins, dimer acids, naval stores, reactive and non-reactive polyamides, distilled tall oils, and specialty blends to the coatings and adhesives industries.

The new development laboratory is expected to be operational in the fall of 1985.

chemicals producers. Several TiO_2 mineral operations have been forced to close, and rationalization has taken place in pigment production as well.

The general result of these changes is a leaner, more profitable industry, ready to meet the challenges of the future, says Ferguson. SRI's study points out these challenges, examines each pigment and mineral market in detail, and gives pigment consumption data for 80 countries.

For more information about the study and the TiO_2 multiclient program, contact Alan Ferguson, Project Manager, SRI International, 333 Ravenswood Ave., Menlo Park, CA.

CPCA to Develop Labeling Guidelines

A recent meeting of the Industrial Labeling Subcommittee of the Canadian Paint & Coatings Association Occupational Health and Safety Committee resulted in a decision to proceed with the development of industrial labeling guidelines. In researching this decision, 11 representatives from CPCA member companies were briefed on the status of the Federal Workplace Hazardous Materials Information System (WHMIS). They also had the opportunity to hear a presentation on the revision to the industrial labeling section of the National Paint and Coatings Association (NPCA) "Labeling Guide." NPCA's General Counsel, Bruce Hamill, described the revision's status which will include use of the Hazardous Material Identification System on finished products.

The Labeling Subcommittee then determined that CPCA should base the development of CPCA guidelines on the NPCA revision. Copies of the current draft were distributed to the Subcommittee who will investigate and, where appropriate, make changes to reflect provincial requirements.

A target date for late winter/early spring 1985 was established for the production of the labeling guidelines.

PPG Initiates Expansion of Lake Charles Facility

PPG Industries, Pittsburgh, PA, has begun a five-year, \$35 million expansion of the capacity of high-purity silicas at its chemical complex in Lake Charles, LA.

According to Chemicals Group Vice-President, Robert D. Cuncan, the first phase of the project is expected to increase production capacity by 20%. In addition to installing highly energy efficient processing equipment, this phase will give the facility a capacity three times larger than any other existing or planned domestic facility for precipitated silicas production.

PPG's program to reinforce the company's commitment to the silicas business began last year when they started producing silicas in Taiwan and embarked on the modernization of the Lake Charles plant. A faster-than-expected recovery in the demand for silicas prompted PPG to speed up work on the modernization. This project is designed to allow PPG to shift more of its silicas production to designed products for special high-technology applications.

Valspar Completes Acquisition Of Mobil Chemical's U.S. and Canadian Coatings

The Valspar Corp., Minneapolis, MN, has reported that its \$95 million acquisition of Mobil Chemical Co.'s domestic and Canadian coatings business is completed. Added to Valspar's 11 facilities are nine Mobil coatings plants in the U.S. and one in Toronto.

In connection with the acquisition, the following Mobil officers were elected: James D. Bennett, Vice-President— Maintenance and Wood Finishes; O.W. Huggard, Vice-President—Packaging Coatings; and David C. Olfe, Vice-President—International Operations.

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Federation News

FSCT Manufacturing Seminar Papers Now Available

Papers presented at the seminar on "Producing Paint Efficiently, Safely, and Economically," sponsored by the Federation of Societies for Coatings Technology and held in Louisville. May 15-16, are available (as a set) in limited quantities.

The package includes all 11 presentations:

- "The Importance of the Manufacturing Function"-J.R. Pickering, President, Lilly Industrial Coatings, Inc. "Employee Involvement in Action"-
- J. F. Tripoli, Manager, Manufacturing and Plant Engineering-Paint Operations, Ford Motor Co.
- "Resins and Polymers"-H. J. Lanson, President, Lanchem Corp.
- "Production Planning and Scheduling"-C. Yates, Manager of Manufacturing, Porter Paint Co.
- "Dispersion"-E. E. Baumhart, President, Coatings Engineering and Systems Co.
- "Storage and Distribution of Liquid Raw Materials"-M. P. Kenes, President, Straubing, Rubin and Kenes, Inc.

- "Instrumental Color Control-How to Keep It Out of the Red"-N. Auge, Technical Sales Representative, and J. Cave, Manager of Color Control Systems, Reliance Universal, Inc.
- "Paint Filling and Packaging"-R. W. Zimmerman, Vice President-Engineering, The Enterprise Companies
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- "Managing Your Safety, Health and Loss Control Program"-H. Russell, Vice President-Manufacturing, Safety, Health & Environment, Grow Group, Inc.
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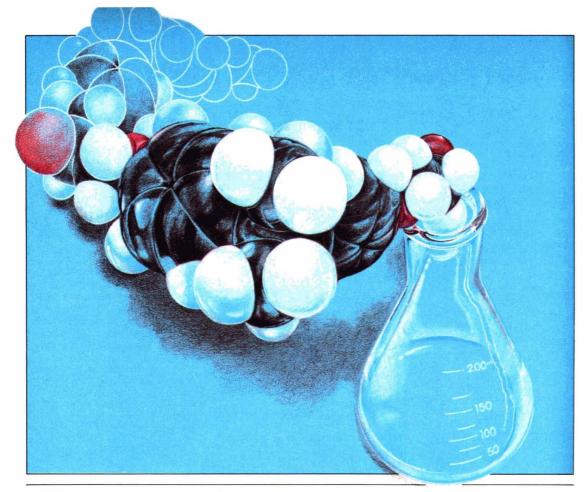
Cost of the complete set of seminar papers is \$75. To order, please contact Ms. Kathleen Wikiera, FSCT, 1315 Walnut St., Suite 832, Philadelphia, PA 19107 (215) 545-1506.

Federation Staff Welcomes Visiting Members



MEXICO SOCIETY MEMBERS George Carrington (left) and Jesus Ortega visited Federation headquarters on a recent trip "north of the border."





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Film Formation And Rheology of Powder Coatings

Peter G. de Lange Consultant*

Film formation and flow of thermosetting powder coatings are reviewed as functions of the two main parameters, namely, surface tension and melt viscosity.

The influence of flow additives, stoving temperature, stoving time, rate of heating during stoving, particle size, pigment concentration, and degree of pigment dispersion on these parameters is discussed. It is indicated that, in order to give a powder coating its optimum film flow, the powder should have minimum melt viscosity, lowest reactivity, and maximum surface tension, and the highest practicable heating rate should be used. Furthermore, particle size should be as small as possible, a high degree of pigment dispersion should be realized during manufacture, and pigment volume concentration should be lower than 20%.

It is shown, that very good flow can only be obtained, when the yield value in the shear stress/ shear rate diagram (rheogram) of a molten coating powder (at stoving temperature) is less than 3 Pa. Finally, attention is paid to cluster formation and entrapped air, which both interfere with film formation. Ways are indicated to prevent these typical phenomena.

INTRODUCTION

Commercially, electrostatic thermosetting powder coatings technology is a rapidly expanding field. It represents an important potentiality for keeping a "clean" environment by efficient use of the coating material, very low or nil volatilization during curing cycles, and a healthy work

*P.O. Box 6. 4847 ZG Teteringen, The Netherlands.

climate for the coater. It is also a field in which our basic understanding of the behavior of these systems has not kept pace with their commercial acceptance.

Due to the unusual nature of a deposited but unbaked powder coating, relative to conventional solvent-containing coatings, the factors affecting the flow of such a powder coating during baking are quite different. One major difference from conventional coatings is that an unbaked powder coating cannot be regarded as a continuous layer. It consists of particles whose major dimensions are of the order of magnitude of the final coating thickness. Therefore one is dealing with macroscopic chunks, larger by far than the individual resin molecules and/or pigment particles.

To obtain a more fundamental understanding of the flow and film formation of various thermosetting powder coating types, the roles played by viscosity, surface tension, stoving temperature, stoving time, flow additives, particle size, and pigment loading are described in this paper.

Finally, some unusual features—cluster formation, and air or vapor entrapment, which are unique to powder coatings, are discussed.

MECHANISM OF FLOW

By design, powder coatings must melt, flow, and fuse through the application of heat to develop coating characteristics. When the flow process of a powder coating layer is considered, two phases can be distinguished: the *coalescence* of molten individual powder particles to form a continuous film (*Figure* 1) and the *flow* of that continuous film (*Figure* 2).

It is assumed and adequately supported in literature, ^{1,2,6} that surface tension is the major driving force (at a given temperature) to cause flow. Gravity is only

Presented by Dr. de Lange at the 61st Annual Meeting of the Federation of Societies for Coatings Technology in Montreal, Que., Canada, October 13, 1983, on behalf of NVVT, the Dutch Section of FATIPEC (rederation of Associations of Technicians in the Paint, Varnish, Lacquer, and Printing Ink Industries of Continental Europe).

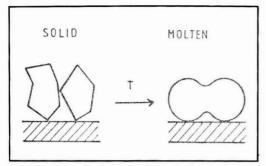


Figure 1—Schematic design of the coalescence of molten powder particles

important for films thicker than 100 microns. (Such film thicknesses are normally not applied on non-preheated metal objects). The only resistance to flow is the viscosity of the molten coating at baking conditions. If surface tension is the cause of the flow, the radius of curvature of the particles should be important, since the pressure causing two spherical particles to flow together is proportional to the surface tension of the coating, divided by the particle radius.

The time, t, required for two powder particles to coalesce can therefore, according to Nix and Dodge,¹ be expressed by the equation:

$$t = f\left(\frac{\eta \cdot R_c}{\gamma}\right) \tag{1}$$

where η is the viscosity, R_c is the average radius of the curvature (the mean particle radius would be a first approximation), and γ is the surface tension. The *kinetics* of coalescence of two spherical particles can more exactly be expressed by Frenkel's² relation (See also Figure 3.)

$$\left(\frac{x}{R}\right)^2 = \frac{3\gamma \cdot t}{2R \cdot \eta} \tag{2}$$

in which x = radius of contact area between the two particles

R = radius of particles $\gamma =$ surface tension

- t = time
- n = viscositv

For the coalescence of powder coatings it must be realized, that one is not dealing with single spherical particles, but with many very irregularly shaped particles of different size. Therefore such simplified particle models can only serve as starting points for considerations, allowing a more profound understanding of possible phenomena during film formation. Nevertheless, experi-

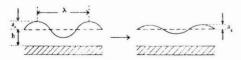


Figure 2—Stylized diagram of the flow of the sinusoidal surface of the continuous fused film⁶

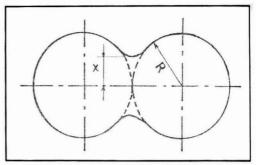


Figure 3—Model for kinetics of coalescence of two spherical particles

mental results from literature show that those simple models are acceptable by approximation. For the coalescence of spherical epoxy resin particles (at constant temperature), Frenkel's equation (2) is a little bit too simple. Therefore, Geguzin³ used the more general equation

$$\left(\frac{x}{R}\right)^2 = f(t) \tag{3}$$

which is illustrated in Figure 4.

Taking *temperature* into account, Kuczynski, et al.⁴ suggested the following equation:

$$\left(\frac{x}{R}\right)^{n} = A(T) \cdot t \tag{4}$$

in which A depends on type of resin and on particle geometry. Exponent n varies with temperature. So Kuczynski found for polymethylmethacrylate (spherical) particles: n = 5 (at 127°C) and n = 0.5 (at 207°C).

Apart from the coalescence of resin particles one should also consider the *spreading* of the molten particles on the substrate. The *spreading rate* of spherical resin drops can be expressed, neglecting gravitational forces (mass of drops < 100 mg), according to Hahn and Jonach,⁵ by the equation:

$$\frac{\mathrm{d}y}{\mathrm{d}t} = \frac{\mathrm{B}}{\mathrm{y}^3} \tag{5}$$

in which y = radius of contact area between particle and substrate

- t = time
- B = factor, which depends on changes in free $surface energy \Delta E_s, mass m of drop,$ $specific gravity <math>\rho$ and melt viscosity η

$$B \sim \frac{\Delta E_s \cdot m}{\rho \cdot \eta}$$
(6)

Considering the *second phase* of *flow* of an irregular surface into a smooth surface, Rhodes and Orchard⁶ showed that for a thin film with an idealized sinusoidal surface the following relation can be given:

$$\ln \frac{a_i}{a_o} = -\frac{16 \pi^4 h^3}{3 \lambda^4} \int_0^t \frac{\gamma}{\eta} \cdot dt$$
 (7)

Journal of Coatings Technology

where η and γ are, respectively, the viscosity and surface tension of the system, h is the average film thickness, λ is the wavelength, and a its amplitude (see *Figure 2*). This equation is only valid, when $\lambda > h$.

If γ and η are constant, the levelling speed is

$$\ln \frac{a_o}{a_t} = k \cdot \frac{h^3 \cdot \gamma}{\lambda^4 \cdot \eta} \int_{\Omega}^{t} dt$$
 (7a)

This means that large values of the quotient a_o/a_t represent good levelling (or flow) of a powder system. This can be realized, according to equations (7) and (7a) by

- · large film thickness h
- high surface tension γ
- low melt viscosity η
- small λ (at which λ should always remain greater than h), which may be attained by small particle sizes.

From the equations described above, it is now clear that if reactivity and particle size are neglected, the *total flow* of the powder coating is mainly determined by the surface tension and the viscosity of the system. Therefore it is of interest to have a more profound knowledge of the influence of surface tension and viscosity on film flow.

Surface tension of a liquid (or molten powder) arises from molecular forces on the surface of that liquid, to give to the liquid a minimum surface area. If one considers, for instance, a liquid in contact with its vapor, then a molecule in the middle of the liquid is in equilibrium in a uniform field, but a molecule at the surface of the liquid undergoes an attraction from the liquid phase. The attraction from the more dispersed vapor phase is not able to compensate the former and the molecules at the surface have a higher free energy.

To reach an equilibrium, the system will tend to minimize its Helmholtz free energy per unit area and consequently to contract its surface to a minimum. It results in a surface tension parallel to the surface, preventing any attempt to increase the interface.

According to Fowkes⁷ the surface tension is the sum of two forces, namely the non-polar intermolecular attractions (or dispersion tension γ_d) and the polar intermolecular attractions (or association tension γ_p), or:

$$\gamma = \gamma_{\rm d} = \gamma_{\rm p} \tag{8}$$

By increasing temperature γ decreases, as the intermolecular attraction forces diminish.

Interfacial tension is defined as the result of the surface tensions of two materials in contact with each other, resulting from the intermolecular attractions of these two materials at their common interface (see *Figure 5*). The equilibrium between these tensions is governed by Young's equation:

$$\gamma_{sv} = \gamma_{pv} \cdot \cos \theta + \gamma_{sp} \text{ or } \cos \theta = \frac{\gamma_{sv} - \gamma_{sp}}{\gamma_{pv}}$$
(9)

in which $\gamma_{sv} = \text{surface tension of substrate in contact}$ with vapor from the molten powder drop. γ_{sv} tends to decrease the substrate/vapor interface by spreading the liquid over the surface.

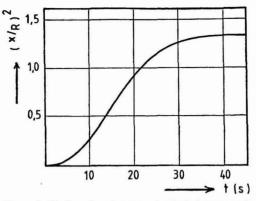


Figure 4—Kinetics of coalescence of spherical epoxy resin particles³

- γ_{pv} = surface tension of molten powder drop (powder coating film) in contact with its vapor. γ_{pv} tends to minimize the liquid/ vapor interface.
- γ_{sp} = interfacial tension between the molten powder coating drop (film) and its substrate. γ_{sp} reduces the substrate/liquid interface.
 - θ = angle between γ_{pv} and the substrate, the so-called *contact angle*.

From Young's equation it is clear that the maximum of wetting of the substrate is achieved when $\theta \to 0$ (Cos. $\theta \to 1$) or when $\gamma_{sv} > \gamma_{pv} \cdot \cos \theta + \gamma_{sp}$.

Consequently, there are two possibilities for improving the wetting characteristics.

- (a) reduce the surface tension of the molten powder γ_{pv}; and
- (b) reduce the interfacial tension between the liquid and the substrate γ_{sp}.

According to Rhodes and Orchard [equation (7)] maximum flow (or film levelling) is achieved with maximum surface tension, but for maximum wetting, a minimum surface tension of the molten powder is needed. How can these two seemingly contradictory requirements be combined to a suitable compromise?

To solve this question it is first necessary to point out that a thermosetting powder system is composed of

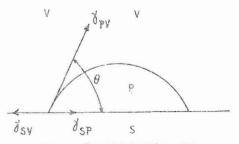


Figure 5—Elements in Young's equation

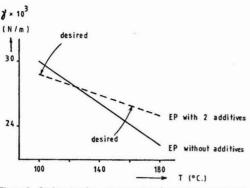


Figure 6—Surface tension vs temperature for an epoxy resin and for an epoxy resin plus two antagonistic additives

different components (resin, curing agent, pigment, flow control agents, etc.) and that, by consequence, its surface tension at a given temperature should be described by

$$\gamma_{p} = f(\gamma_{i}, [i], \mathbf{R}, \mathbf{x}, t)$$
(10)

- in which γ_i = the surface tension of the component i at the given temperature
 - [i] = the concentration of the component i
 - R = the reactivity of the system (by the presence of curing agents)
 - x = the degree of compatibility (c.q. solubility) of the components with each other.
 - t = time of reaction (the surface tension of a powder coating will also change with time, due to changes in [i], R, and x)

To get around this complex of factors, it is necessary for surface tension measurements, to eliminate some factors.

Gabriel⁸ eliminated R and t by measuring surface tensions and contact angles of pure epoxy resins and non-reactive epoxy resin/compatible additive mixtures. He found that γ decreases linearly with increasing temperature (between 100° and 200°C) and that some liquid additives (e.g., ethylacrylate/2-ethylhexylacrylate copolymers with a mean molecular weight \overline{M}_n of nearly 8000) improve wetting by reducing contact angles,

Table 1—Influence of Acrylic Additive On Gloss and Surface Appearance Of an Epoxy Powder Coating

% Additive	Gloss 60°	Surface	
0	52	Craters	
0.25	89	Some craters and orange peel	
0.50	97	Some orange peel	
0.75	98	Very slight orange peel	
0.8	98	Smooth	
1.00	94	Smooth	
2.00	85	Smooth	

whereas others (epoxidized soybean fatty acid, hydroabietyl alcohol) improve flow by raising surface tension.

Relating these facts to Young's equation, one could also interprete the influence of the mentioned additives as follows:

The acrylic copolymers (or Type A-additives) reduce γ_{pv} therefore raising the spreading pressure $\gamma_{sv} - (\gamma_{pv} \cdot \cos \theta + \gamma_{sp})$ and promoting wetting between molten resin and substrate. But, as the effect on surface tension of the epoxy resin caused by the addition of the acrylic additive decreases with increasing temperature, we can say that the improvement of wetting should also be attributed to the reduction of the interfacial tension γ_{vp} .

Epoxidized soybean fatty acid and hydroabietyl alcohol (or type B-additives) raise contact angle θ , which means that Cos θ is reduced, and, with nearly constant values of γ_{sv} and γ_{sp} , γ_{pv} will be increased.

In powder coating technology, the influence of both types of additives can be interpreted as follows:

Epoxy resin without additives leads to "cratering" and "orange peel".

Epoxy resin + Type B-additives leads to "cratering", but no "orange peel".

Epoxy resin + *Type A*-additives leads to "orange peel" but no "cratering."

Epoxy resin + both types of additives leads to good flow (no craters, no "orange peel").

The common expression "flow control agents" is thus misleading and should be substituted by "flow promotors" (e.g., epoxidized soybean fatty acid) and "wetting promotors" (e.g., acrylic copolymers of the type discussed).

Now, after these theoretical aspects of surface tension, it can be shown (*Figure* 6) that the requirements of *maximum* surface tension for good *fiow* and *minimum* surface tension for good substrate- and pigment-wetting are not contradictory, but can be combined to a suitable compromise by *combination* of both types of additives.

Figure 6 illustrates that at 100°C γ_{pc} should be as low as possible for good pigment wetting during extrusion. At temperatures above 150°C, however, γ_{pc} should be as high as possible for good film flow.

It should be emphasized here that the effect of an additive on contact angle and surface tension strongly depends on the type of binder. So it was found in our laboratory, that epoxidized soybean fatty acid reduces the surface tension of acrylic and certain polyester powder coatings. Here the rather poor flow of these powder types will become even worse (more "orange peel") and normal practice in such cases is, therefore, to use only the acrylic additive to prevent cratering.

As a matter of fact, it is also normal practice with most types of epoxy, epoxy/polyester, and pure polyester powder coatings, to use only a type A (acrylic)-additive and not two antagonistic additives.

To explain this, in connection with the previous analysis of the possibilities of improving flow, it is necessary to consider "orange peel" and cratering tendency in more detail.

Looking back to Rhodes' and Orchard's equation (7a) the reader may be puzzled by the total contradiction existing between the well-known reputation of type A (acrylic) additives to give good flow and its total lack of compliance with the definition of a good levelling agent. Equation (7a) is the result of an interval of time showing a tendency to reduce more or less rapidly the amplitude a of the sinusoid, given the time. Therefore, another way to increase the levelling properties will be to increase the levelling time.

This "time" effect can be realized by adding a surplus of type A-additive to a powder coating, which is illustrated by *Tables* 1 and 2 and by *Figure* 7.

From *Tables* 1 and 2 it follows that the optimum level of acrylic additive is comprised between 0.75 and 0.8% for the epoxy-powder and between 0.75 and 1% for the polyester/TGIC-powder. In both cases this interval is a bit higher than the ideal concentration in *Figure* 7 for the *lowest* contact angle (c.q. surface tension).

This can be explained by the fact that the additional quantity of the relatively low molecular weight additive migrates up to the surface of the polymer to equalize its chemical potential at the surface and in the bulk of the resin. When such a medium size polymer with unbranched side chains arrives at the surface, the polar backbone remains dissolved into the polymer, while the alkyl chains try to escape in the surrounding environment, providing better conditions for a stable equilibrium. Consequently, the surface tension tends to equalize across the surface because of the greater concentration of an orientated molecular structure all over the surface. In this way the more even surface tension reinforces the anti-cratering and anti-orange peel effects, and due to its plasticizing effect, the additive provides a longer levelling effect at the surface.

This theory is sustained by Fink-Jensen,⁹ who found that the flow of material from one point to another in a situation like cratering is governed by the equation:

$$q = f(\Delta \gamma) \tag{11}$$

in which q = mass flow of material,

 $\Delta \gamma$ = surface tension gradient across the surface.

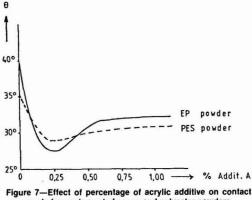
So prevention of cratering involves equalizing the surface tension across the surface.

Feyt and Bauwin¹⁰ arrived at the same conclusion for polyacrylics in solvent-borne coatings. Overdiep¹¹ has made a detailed mathematical analysis of the effect of surface tension gradients on levelling and came, also, to the conclusion that the time dependent decay of sinusoidal brush marks (*Figure* 2) can be written as a function of the surface tension gradient.

VISCOSITY AND PARAMETERS AFFECTING VISCOSITY

In the previous section it was stated that surface tension is the major driving force to cause flow after melting of the powder particles, and that viscosity of the molten coating is the only resistance to flow. Therefore the viscosity of a powder coating should be as low as possible at the curing temperature.

However too low viscosities lead to bad edge coverage and sagging on vertical substrates. According to Walz, et



angle for unpigmented epoxy and polyester powders

al.,¹² the viscosity lies within a favorable range when $\eta = 6000$ to 10,000 m Pa.s.

A main requirement for a good film formation and flow is a sufficiently long time at which the viscosity is low. For thermoplastic powders this depends only on the molecular weight of the polymer and on its curing temperature. For thermosetting powders (containing a curing agent) the reactivity of the system also plays an important role, as well as the heating rate.

The melt viscosity-temperature relationship for polymer melts (without curing agents) can be characterized by the following Arrhenius type equation:

$$\eta = Ae^{E/RT}$$
(12)

or

$$\ln \eta = \frac{E}{RT} + C \tag{12a}$$

where $\eta = \text{melt viscosity}$

A = a constant, characteristic for each type of resin

E = activation energy for viscous flow

R = universal gas constant

T = absolute temperature (° Kelvin).

It appeared that the relation between the logarithmic melt viscosity and the reciprocal temperature is only linear at high temperatures. At lower temperatures the relation is non-linear. This change from non-linear to

Table 2—Influence of	Acrylic Additive
On Gloss and Surface	ce Appearance
Of a Polyester Pov	wder Coating

Gloss 60°	Surface	
54	Craters	
82	Some craters plus orange peel	
90	Orange peel	
92	Some orange peel	
92	Slight orange peel	
87	Slight orange peel	
81	Slight orange peel	

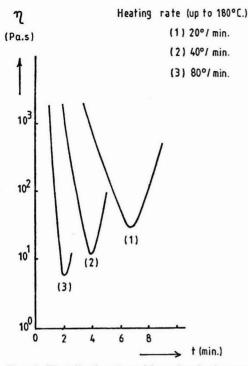


Figure 8—Effect of heating rate on minimum viscosity of an epoxy powder system¹³

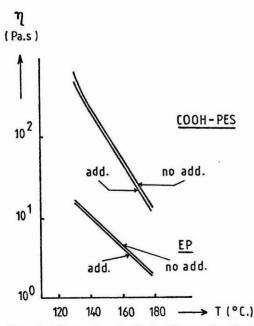


Figure 9—Influence of acrylic "flow" additive on viscosity at various temperatures for an epoxy and a carboxylated polyester resin

linear behavior is determined by the glass transition temperature (Tg) and occurs for a Type 4* epoxy resin at about 125° C, which is about 70 degrees above its Tg. This kind of temperature dependence of melt viscosity is usual for a large number of polymer melts. The linear, high temperature region can therefore be indicated by T > Tg+ 70° C.

Further it has been found, that the activation energy for viscous flow (E) is almost identical for all Type 4 epoxy resins and the average value is 93.4 ± 3.3 kJ/mol. Values for other polymer types lie between 40 and 200 kJ/mol. Some activation energies of often used polymers have been calculated by us from the slopes of their In η -T-curves and listed in *Table* 3.

These values of E, which express the energies necessary to overcome (break) the intermolecular forces at viscous flow, are only valid up to temperatures of about 120°C above Tg. Above these temperatures reliable viscosity measurements are impossible, most probably as a result of crosslinking reactions taking place, e.g., etherification of OH-groups.

Polymer melts, containing curing agents (e.g., thermosetting powder coatings) do not obey the melt viscosity/ temperature relationship as indicated by equation (12) or (12a). Here the viscosity decrease due to temperature increase is apparently opposed by the increase in viscosity due to the crosslinking of the system. As a consequence, the flow time, which is defined by Walz¹² as the time in seconds required for the powder coating at stoving temperature to attain 50,000 mPa.s., will be shorter with increasing reactivity. For proper flow, time should always be > 100 s.

The minimum viscosity attained by a thermosetting powder coating system during stoving is also strongly influenced by the heating rate. Ghijsels and Klaren^B showed with epoxy powders, that they attained lower minimum viscosities, and as a consequence better flow, by the use of higher heating rates. This is illustrated in *Figure* 8. It agrees very well with observations made in practice, that better flow is found with powders applied on thin or preheated substrates.

In a closely related paper, Hannon, et al.¹⁴ used a "time integrated fluidity" parameter F to deal with heating rate effects on levelling of powder coatings. Here $F = \int_{0}^{t} \frac{dt}{\eta}$ in the Orchard/Rhodes equation (7).

The Arrhenius type equation (12) or (12a) should therefore be modified in such a way that the influence of

*Type 4 epoxy resins have an Epoxy Group Content of 1050 to 1175 mmol per kg.

Table 3—Activation Energies of Some Polymers Used in Powder Coatings (in k J/mol.)

Epoxy resin, Type 3 ^a l	37
Carboxylated polyesters for	
EP/PES-powders	22 to 163
Carboxylated polyester for	
PES/TGIC powders	72
PES/TOIC powders	12

(a) Type 3 epoxy resins have an EGC of 1210 to 1380 mmol per kg.

the heating rate is incorporated in this $\ln \eta - 1/T - \text{relation}$. This was done in a theoretical thermodynamic way by Hollands and Kalnin¹⁵ and their equation was used by Kordes¹⁶ to calculate heating rate effects on viscosity of an epoxy/polyester hybrid powder. His calculated minimum viscosities agreed very well with his rheological measurements.

Viscosity measurements of some polymer melts show, that these melts behave as non-Newtonian liquids. It means, that the determined viscosities are dependent on shear rate, in particular at higher temperatures. Melt viscosities should therefore be determined at very low shear rates (less than 1 s^{-1}), in order to be sure that the melt has no yield point and behaves as a Newtonian liquid. Such viscosity measurements were done in our laboratory with a Haake Rotovisko RV3 cone and plate viscometer. With this, we measured the influence of type A (acrylic) additives as well as pigment level (pigment volume concentration) on melt viscosity of various binder types.

From *Figure* 9, it can be seen that the influence of the acrylic additive on viscosity at various temperatures is only very small, and is much less pronounced than its influence on surface tension.

Figure 10 illustrates the effect of TiO₂-pigment content on the melt viscosity of a Type 4 epoxy resin, a carboxylated polyester resin, and a 50/50 mixture of an epoxy resin and a polyester resin (as is used for hybrid powders). This figure demonstrates clearly the increase of melt viscosity with increasing pigment level (pigment volume concentration). A more dramatic increase in viscosity appears at PVC levels of more than 20%.

The effect of pigment level on melt viscosity is also dependent on the extent to which a pigment is dispersed within the resin matrix. Nix and Dodge¹ found that, with equal pigment levels, a powder with finely dispersed pigments showed lower melt viscosity than the same powder with badly dispersed pigments. By improving the degree of dispersion, the melt viscosity may be reduced in some cases by nearly a factor 10.

At the same time interaction between pigment and binder (e.g., adsorption, chemisorption) may play a role. Ghiljadow, et al.¹⁷ showed that, with increasing interaction between pigment and resin particles, improved wetting of the pigment by the binder takes place on the one side, whereas, on the other side, the mentioned rise in viscosity by pigment addition will be smaller.

As is shown by similar slopes in the $\ln \eta / T -$ curves in Figure 11, the activation energy for viscous flow is independent on pigment-concentration.

Often pigmented resins do not behave as Newtonian liquids, but exhibit the so-called plastic flow. That means a plot of shear stress against shear rate is approximately a straight line (as with Newtonian liquids), but one which does not pass through the origin (as shown in *Figure* 12). Therefore "plastic flow" is characterized by a "yield value," that is a certain minimum shear stress, which must be overcome before flow will take place. The presence of such a yield value indicates the existence of rather high interparticle forces. With dispersions of pigments in molten resins these forces are due to attraction between pigment particles. Therefore the magnitude of these

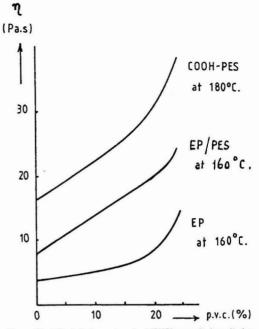


Figure 10—Effect of pigment content (PVC) on melt viscosity for an epoxy resin, an epoxy/polyester-50/50-"hybrid" mixture, and a COOH-polyester resin, which is used in combination with TGIC, (and has a lower acid value and a higher molecular weight than the "hybrid" polyester)

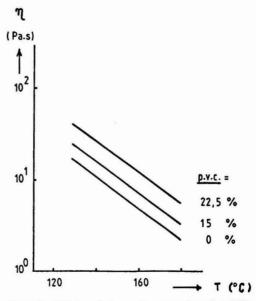


Figure 11—Melt viscosity-temperature curves for various PVCs. Parallel curves indicate equal activation energies for viscous flow

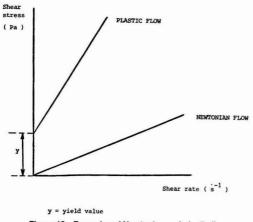


Figure 12—Examples of Newtonian and plastic flow

forces depends on the degree of wetting of the pigment particles and of course on the pigment concentration.

Stachowiak¹⁸ measured the yield values of pigmented epoxy resin based powders with a Weissenberg Rheogoniometer. He came to the conclusion that the yield value of a molten powder plays an important role in determining the extent of flow-out. He found that excellent flow could only be obtained with systems having a yield value of less than 3 Pa. He came to the following correlation between yield values and flow of powder coatings: 0 to 3 Pa—very good to excellent flow; 3 to 7 Pa—moderate flow; 7 to 50 Pa—poor flow; > 50 Pa very poor flow. This classification is not rigorous, but will vary according to the magnitude of the surface tension.

We also measured yield values with a Haake Rotovisko RV 3 cone and plate viscometer. Our investigations were not restricted to epoxy powders, but also measured yield values of epoxy-polyester hybrid powders and outdoordurable polyester-TGIC powders.

The yield value was determined as a function of the pigment (TiO_2)-content at various temperatures. For these measurements we used the following powders:

(1) Epoxy-powders with PVC's of 7.5, 15, and 22.5% (containing 0.5% acrylate additive and 5% accelerated "Dicy" curing agent, both calculated on resin).

(2) Epoxy/Polyester (50/50)—hybrid powders with PVC's of 7.5, 15, and 22.5% (containing 1.0% of the same acrylate additive).

(3) Polyester/TGIC (93/7)—outdoor-durable powder with equal PVC levels (and containing 1.0% of the same acrylate additive).

The influence of pigment content (PVC) and stoving temperature on yield value and observed flow is given in *Table* 4 and is illustrated in *Figure* 13.

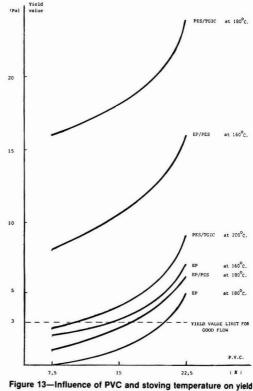
Figure 13 indicates a considerable influence of PVC (and stoving temperature) on yield value. As with the PVC/viscosity curves, there is a rather dramatic increase in the slopes of the curves above PVC levels of approximately 20%. This is because yield value is strongly dependent on viscosity. From Figure 13 and Table 4, it can be concluded, that: (1) For "normal" (usual) PVC levels of 15% good to very good flow is only possible with (a) pure epoxy powders, when stoved at 160°C or higher; (b) epoxy/ polyester (50/50) hybrid powders, when stoved at 180°C or higher; (c) polyester/TGIC powders when stoved at a minimum temperature of 200°C.

(2) For very high PVC levels of 22.5% acceptable flow is only possible with pure epoxy powders, when stoved at 180°C or higher.

When macromolecular materials are adsorbed on the pigment surface, a kind of re-orientation of these macromolecules at the pigment/binder interface can take place, according to Funke and Zorll,¹⁹ which can lead to a higher glass transition point (T_g) of the powder.

Flow time (as previously defined as the time to attain 50,000 mPa.s at stoving temperature) is, for some powder types, increased by pigment additions, but for other types there is no influence of pigment level.

As we have seen now, in all cases pigments (and of course extenders) interfere with film formation and flow. To reduce the negative effects as much as possible, low pigment levels should be chosen (PVC less than 20%). Selection of pigments which give intensive interaction with the binder and/or a high degree of pigment dispersion may also be helpful. In the latter case one could



Igure 13—Influence of PVC and stoving temperature on yield value for three powder types

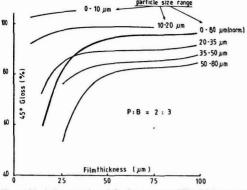


Figure 14—Influence of particle size range and film thickness on gloss of a white epoxy powder.

also think of pigment-wetting agents, which build a "polymerophilic" adsorption layer on the pigment surface.

Particle size has a dramatic effect on film flow (and gloss), as small particles need less time to melt and to coalesce than coarse particles [see also Nix' and Dodge's equation (1)]. When the maximum particle size is reduced, the minimum film thickness required to give good flow and gloss is also reduced. *Figure* 14 illustrates the relationship between gloss and particle size range for an epoxy-powder with a pigment/binding ratio of 2:3 as a function of film thickness.

Figure 14 also illustrates the large positive influence on gloss (and consequently on flow, as gloss measurement values are strongly affected by flow) of the finer powder fractions and the negative contribution of the coarser powder fractions. These five fractions were obtained by "classification" by means of wind sifting and sieving.

For the application of thin film powder coatings with reasonable gloss and flow down to 25 μ m film thickness, we found²⁰ that it is necessary to remove coarse particles (> 50 μ m) from the powder.

CLUSTER FORMATION

An important factor affecting flow is the initial profile of the dry, deposited (unbaked) powder layer on the substrate. Photographic evidence, *Figure* 15, has indicated that "clusters" or large agglomerates of powder particles, if present, can become the "hills" of the orange peel.

The flow mechanism, as proposed by Nix and Dodge in equation (1) still holds, if the important radius of curvature becomes that of the clusters, after individual particles have flown together. That means that a "cluster" can be considered as a very large particle, of which the time of coalescence (or flow time) will be too long.

Causes of clusters may include:

 Electrostatic attraction or repulsion, which may cause clusters to form either before spraying, during transport to the substrate, or upon deposition.

Table 4—Influence of P.V.C. and Stoving Temperature on Yield Value and Flow

Powder Type	PVC (%)	Temperature (°C)	Yield Value (Pa)	Flow (0 to 5) ⁸
ЕР	7.5	160	2	0 to 1
	15		2 3 7	1 to 2
	22.5		7	2 to 3
ЕР	7.5	180	0	0 to 1
15			1	0 to 1
	22.5		5	2
EP/ PES	7.5	160	8	2 to 3
	15		10.5	3
22.5			16	3 to 4
EP/PES	7.5	180	1	0
15 22.5			2.5	1
			6	2 to 3
PES/TGIC	7.5	180	16	3 to 4
	15		18	4
	22.5		24	5
PES/TGIC	7.5	200	2.5	1 to 2
	15		4	2
22.5			9	2 3

(a) Evaluated according to DIN 53230 : 0 = excellent; 3 = moderate; 5 = bad.

- (2) Too many powder particles of less than 5 microns in diameter, which cling to larger particles of 50 to 60 μm diameter.
- (3) Low molecular weight materials at the surface, which may cause sticking of particles to one another.

EFFECT OF ENTRAPPED AIR OR VAPOR ON FILM FORMATION

Entrapped air and vapor bubbles in stoved powder coatings interfere considerably with film formation and should be prevented as far as possible (*Figure* 16).

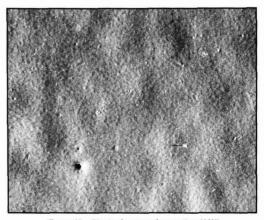


Figure 15—Cluster in a powder coating (10X)



Figure 16—Entrapped air in a powder coating film (SEM, 100X)

Flow must occur simultaneously with at least partial displacement of air (and possibly vapor, produced during stoving) from between individual powder particles. Release of this air will be retarded or partly prevented, as the powder melts first at the air interface to form a continuous layer. Furthermore, air or vapor trapped within powder particles will raise the melt viscosity and interfere with flow.

Sometimes the air bubbles just reach the air interface during stoving, resulting in "micro-pinholing" (in German "Nadelstich").

For a complete removal of air bubbles, Zorll²¹ calculated that the release time may amount to 26 seconds in some cases.

Hoeflaak and Selier²² found a critical film thickness above which air-bubbling occurs. They also showed that there exists a relation between that critical film thickness, stoving temperature, and flow time. Trapped air and vapor bubbles and micro-pinholes can be prevented or reduced by low melt viscosity, long flow time, and reduced film thickness.

The critical film thickness can be increased by

- (1) Increasing stoving temperature; and
- (2) Pretreatment (phosphating) of the metal substrate.

SUMMARY

It has been shown that the flow of a thermosetting powder coating depends on both the rheology and surface tension of the system.

Surface tension here is the major driving force to cause flow after melting and viscosity is the main resistance to flow. A too low surface tension and/or too high melt viscosity will preclude good film flow, as is illustrated in *Figure* 17.

The rheological contribution of flow is not only a matter of resin melt viscosity, but also of such interrelated factors as reactivity, minimum viscosity attained by the rate of heating during stoving, and, last but not least, pigment concentration and degree of pigment dispersion (interaction between pigment and binder).

Special attention has been paid to the effect of pigment volume concentration (PVC) on viscosity and yield value. By means of a rotating cone and plate viscometer it has been found that PVC's of more than 20% strongly increase viscosity and should therefore be prevented.

Good flow is only possible (with all types of the investigated powders), when yield value is lower than 3 Pa, which agrees very well with Stachowiak's results.

Measurements on pure non-reactive resins (without curing agents) have shown that surface tension is strongly influenced by so-called "flow" additives. Some additives improve flow by raising surface tension (and contact angles), whereas others improve wetting by reducing contact angles. Furthermore, it has been indicated that the lowering effect of well-known acrylic additives on melt viscosity is much less pronounced than is generally expected.

Finally, it has been shown that finer powder particles improve flow and gloss, whereas coarser particles have a negative effect on flow and gloss. The best combination of flow and sprayability will be given by the so-called "classified" powders.

At the end of this article attention has been paid to cluster formation and entrapped air, which both interfere with film formation. Ways are indicated to prevent these typical phenomena.

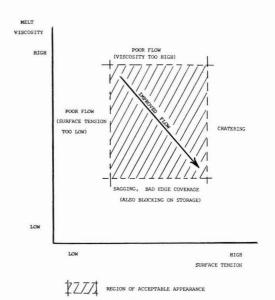


Figure 17—Illustration of the dependence of film flow on surface tension and melt viscosity

It is hoped, that this article has demonstrated that basic knowledge of film formation and flow of powder coatings may be helpful in formulating powders, which can be applied profitably.

ACKNOWLEDGMENT

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Flammability Of Chlorinated Hydrocarbons And Hydrocarbon Admixtures

Harry A. Wray Harry A. Wray Associates*

Some of the misconceptions concerning the flammability properties of chlorinated hydrocarbons and their admixtures with aliphatic or aromatic hydrocarbons were investigated. Reported misunderstandings of the operation of the Setaflash Tester and misinterpretations of the results obtained from it were studied. Although some chlorinated hydrocarbons do not have a flash point in a standard flash point apparatus, they can burn under certain conditions. A flash point occurs only when a flame is propagated over the surface of a liquid. The presence of a blue halo or a greenish-yellow flame enlargement around the ignition flame should not be interpreted as a flash point. Admixtures of hydrocarbons and chlorinated hydrocarbons may have no flash point, or a lower or higher flash point than the hydrocarbon alone, depending on the composition of the admixture.

Furthermore, the need to follow the standard method for operating the Setaflash is emphasized to prevent the quenching effect of combustion or decomposition products developed during a flash within the tester.

INTRODUCTION

With the advent of Clean Air Act regulations and the government's regulation of flammable solvents in transportation and storage, various ways were tried to overcome the restrictions on normal solvent-based coatings, adhesives, polishes, etc. These included the development of water-based, water-reducible, high-solids and solventless coatings. Other products were formulated with certain chlorinated hydrocarbons to take advantage of their apparent nonflammability. These hydrocarbons have also been blended with flammable solvents with the intent of raising the flash point of the latter solvents.

While some chlorinated hydrocarbons may have no flash point in standard, closed-cup flash point testers, such as the Tag and Setaflash Testers, they may have lower and upper limits of flammability (LFL and UFL). Furthermore, a lower temperature limit of flammability (LTL) can be determined for these substances using larger sized vessels than the flash point cup. This essentially eliminates the quenching effect of the walls of the smaller flash point cup. Thus, while no flash point can be obtained in a standard cup, chlorinated hydrocarbons may be shown to be flammable in a larger vessel.

Definitions

The terms used in describing the fundamental flammability properties of liquids are defined as follows:

(1) Propagation of a Flame:¹ The upward and outward movement of a flame front from an ignition source to the vessel's walls.

(2) Lower Limit of Flammability (LFL):¹ The lowest concentration of a combustible substance (gas or vapor) that is capable of propagating a flame through a homogeneous mixture of the combustible substance and a gaseous oxidizer under specified test conditions.

(3) Upper Limit of Flammability (UFL):¹ The maximum concentration of a combustible substance (gas or vapor) that is capable of propagating a flame through a homogeneous mixture of the combustible substance and gaseous oxidizer under specified conditions of test.

(4) Autoignition Temperature (AIT): The minimum temperature of the reaction chamber which leads to the spontaneous ignition of a combustible substance within the chamber under specified conditions of test.

^{*}P.O. Box 94, Swarthmore, PA 19081.

(5) Flash Point (FP):¹ The lowest temperature corrected to a pressure of 760 mm of Hg (101.3 kPa or 1013 mbar) of a substance at which application of an ignition source causes the vapor of a specimen to ignite under specified conditions of test.

(6) Lower Temperature Limit of Flammability (LTL): The lowest temperature corrected to a pressure of 760 mm of Hg (101.3 kPa or 1013 mbar) at which application of an ignition source causes the vapors *in equilibrium* with a liquid (or solid) specimen to ignite and propagate a flame under specified conditions of test.

While the results of flash point and LTL tests are similar, they differ slightly due to differences in direction of the propagation of the flame. In the flash point apparatus, the flame propagates downward from the small flame ignition source, but in the limit of flammability and temperature limit of flammability tests the flame is propagated upward and outward from the ignition source.

The absence of a flash point does not necessarily indicate the nonflammability of a material such as a chlorinated hydrocarbon. These substances may be flammable in vessels larger than a flash point cup as the walls of the cup have a quenching effect on the flame, preventing the formation of a flame front. These liquids may also require a higher energy ignition source than that of the flash point apparatus to produce a flash.

FLAMMABILITY PROPERTIES OF CHLORINATED HYDROCARBON SOLVENTS

Examination of *Table* 1 indicates that there are a few chlorinated hydrocarbons which are truly nonflammable. Of those listed, there are only two which are completely nonflammable, namely, carbon tetrachloride and perchloroethylene. These are the ones which are completely chlorinated.

If the LFL of chlorinated hydrocarbons is compared with aromatic or aliphatic hydrocarbons, it is noted that chlorinated hydrocarbons have a higher LFL than the aromatic or aliphatic hydrocarbons. For example, toluene has an LFL of 1.4% compared to an LFL of 8.0% for 1,1,1-trichloroethane; mineral spirits has an LFL of 0.8%while methylene chloride has an LFL of 12.0%. Thus, a greater amount of vapor must be produced in either a flash point tester or a lower flammable limit apparatus to ignite a chlorinated hydrocarbon than is necessary for nonchlorinated hydrocarbons.

In 1976, M. B. Harve-Bazin⁵ reported that when halogenated hydrocarbons were added to petroleum solvents to produce dry-cleaning mixtures with a higher flash point than the petroleum solvent alone, the reverse occurred in some cases. The effect was reported to be quite evident in the case of dichloromethane (methylene chloride). He further stated that low boiling chlorinated hydrocarbon solvents tend to reduce the flash point while high boiling chlorinated solvents tend to raise the flash point of the mixtures. Chlorinated solvents with an intermediate boiling point were reported to have little effect on the flash point of mixtures with hydrocarbon solvents.

In addition to these results, the article reported a methylene chloride flash point of -23° C. This is contrary to results reported in the literature which show methylene chloride as having no flash point in regular flash point apparatus. However, Coffee, et al.,⁶ reports that methylene chloride in a five liter flask had a LTL of 217° F at 760 mm of Hg.

To check Harve-Bazin's results, we ran several experiments in a Setaflash Closed-Cup Flash Point Tester with methylene chloride.* We were unable to obtain a flash point (propagation of flame across the surface of a liquid) at -23° C (-9° F). However, at this low temperature there appeared a greenish-yellow flame enlargement of the test flame, i.e., as the test flame of the flash point tester is inserted into the methylene chloride vapor, the test flame gets larger and turns from a blue flame to a greenishyellow color. As the methylene chloride is heated, its

*Obtained from Dow Chemical Co., U.S.A.

Name	Formula	`F.Р. °F	AIT °F	LFL %	UFL %	Boiling Point, °F	Ref.
Dichloromethane							
(Methylene chlori	de) CH_2Cl_2	None	1033	12.0	19.0	104	2,3
Chloroform	CHCl ₃	None	1155	-	-	142.2	2,4
Carbon tetrachlorid	eCCl4	None	None	None	None	172.4	2,4
1,1,-Dichloroethane	CH ₃ CHCl ₂	6	856	5.6	11.4	136	4
1,2-Dichloroethane	CH ₂ Cl—CH ₂ Cl	56	775	6.2	16.0	187	2,3
1,1-Dichloroethene	CH ₂ =CCl ₂	0	1058	7.3	16.0	98.6	2,3
1,2-Dichloroethene	CHCl=CHCl	36/39	860	9.7	12.8	118	2,4
1,1,1-Trichloroethan	eCH ₃ CCl ₃	None	1000	8.0	10.5	165	2,3
1,1,2-Trichloroethan	eCH ₂ Cl—CHCl ₂	None		12.0		236	2
	e)CHCl=CCl ₂	None	770	8.0	10.5	189	2,4
	CH3CHClCH2Cl	60	1035	3.4	14.5	190	4
	eCHCl=CHCH ₂ Cl	95		5.3	14.5	217-230	2,4
	e)Cl ₂ C=CCl ₂	None	None	None	None	250	2,4

Millimetres of Hg	Toluene, °C	Trichloroethylene, °C	Methylene Chloride, °C	1,1,1-Trichloroethane, °C
1	26.7	-43.8	-70.0	-52.0
5	9.4	-22.8	-52.1	-32.0
10	+6.4	-12.4	-48.3	-21.9
20	18.4	-10.0	-33.4	-10.8
10	31.8	+11.9	-22.3	+1.6
60	40.3	20.0	-18.7	9.5
00	51.9	31.4	-6.3	20.0
0	69.5	48.0	+8.0	36.2
0	89.6	67.0	24.1	54.6
50	110.6	86.7	40.7	74.1
apor Density	3.1	4.8	2.94	4.55

Table 2—Boiling Point Temperature/Vapor Pressure Relationship of Test Solvents^{8,9}

vapor concentration increases in the flash point cup and the test flame increases in size and maintains its greenishyellow color. At no time was a true flash point observed.

The flame enlargement phenomenon is probably related to thermal decomposition of the test material. Chlorinated hydrocarbons are readily decomposed by heat.⁷ The temperature of the test flame of a Setaflash Tester using Philadelphia city gas was determined to be approximately 1100°F. It is reported that methylene chloride decomposes at 1300° F. However, at the temperature of the Setaflash test flame, there is some decomposition of methylene chloride in the test cup. Decomposition products are hydrogen chloride, carbon dioxide, carbon monoxide, chlorine, and possibly, carbon.⁷ The enlarged flame developed in chlorinated hydrocarbon vapor indicates the presence of unburned carbon and chlorine. The chlorine reacts with copper in the test flame taper to produce a Beilstein test result for copper, i.e., a greenish-colored flame. The odors which are detectable are those of chlorine and hydrogen chloride. (By a secondary reaction, carbon monoxide and chlorine may combine to form low concentrations of phosgene, an extremely toxic gas.)

The greenish-yellow flame should not be confused with the "blue halo" sometimes observed in testing flammable liquids. The halo is evident just prior to the true flash point and the flash point of the liquid can be observed by slightly increasing the temperature. However, raising the temperature of a chlorinated hydrocarbon above the first observation of the greenish-yellow flame does not lead to a flash point and in some cases may lead to the extinguishing of the test flame. The flame enlargement seen when testing methylene chloride does not represent a true flash point.

It appears that Harve-Bazin confused the temperature of the flame enlargement for the flash point. He concluded that:

(1) Chlorinated hydrocarbons with low boiling points (methylene chloride—b.p. 104° F), and thus high vapor pressure, (see *Table 2*) lower the flash point of chlorinated hydrocarbon/hydrocarbon mixtures;

(2) A chlorinated hydrocarbon with relatively high boiling point (perchloroethylene—b.p. 250°F) tends to raise the flash point in a mixture with a hydrocarbon; and (3) A chlorinated hydrocarbon with an intermediate boiling point between the above types $(1,1,1-tri-chloroethane-b.p. 165^{\circ}F)$ has little effect on the flash point values of the mixture.

If the development of an enlarged flame is considered the flash point, his conclusions could be explained as follows:

(1) Methylene chloride with a high vapor pressure would, at a low liquid temperature, produce a sufficient concentration of vapor to decompose partially at the test flame temperature resulting in flame enlargement.

(2) Perchloroethylene, having a low vapor pressure, would require a higher temperature than methylene chloride to produce sufficient vapor for decomposition to cause a similar flame enlargement.

(3) 1,1,1-trichloroethane with a vapor pressure between the above two solvents would require an intermediate temperature—probably close to that of the flash point of the hydrocarbon.

However, some of his reported flash point results may have been true flash points. A letter to the editor in *Chemical and Engineering News*¹⁰ reported that 1,1,1-trichloroethane, when added to a petroleum solvent,

Table 3—Flash Point o	f Mixtures
Of Mineral Spirits and 1,1,1-	Trichloroethane

Volume Percent in Blend, %			Flack Daint OF	
Mineral Spirits	1,1,1-Tri- chloroethane	1st Day	Flash Point, °F 2nd Day	2nd Lab.
100		109	-	_
_	100	None* to 115	100	
90	10	93	95	96
80	20	84	85	83
70	30	77	77	78.5
65	35	69.5	None to 85	None to 205
63	37	None to 110	None to 202	
61	39	None to 113	None to 141	

* "None" indicates no flash evident at conclusion of test at that temperature.

Of Mineral Spirits, Methylene Chloride And 1,1,1-Trichloroethane						
Mineral	Methylene Chloride Volume Percent in	1,1,1-Tri-	Flash P	oint, °F		
Spirits, %	Blend, %	chloroethane, %	1st Lab	2nd Lab		
95	5	-	None to 145	None to 205		
86	3.0	11	None to 200	None to 205		

	Table 4—Flash Po	int of Mixtures
Of	f Mineral Spirits, M	ethylene Chloride
	And 1,1,1-Tric	hloroethane

considerably lowered the flash point of the mixture. This has also come to our attention through private correspondence. To check this unexpected result, the following experiment was conducted.

EXPERIMENTAL DETAILS

Samples of a mineral spirits* and an inhibited 1.1.1trichloroethane[†] were blended by volume at ratios shown in Table 3. (The 1,1,1-trichloroethane had a purity of 93.5% by volume and the amount of the chlorinated hydrocarbon was adjusted accordingly to produce the stated ratios.) These mixtures were then tested for flash point by method ASTM D 3278,11 Standard Test Method

*145 215°C, obtained from Standard Oil Co. of Indiana.

† Chlorothene® NU, a registered trademark of Dow Chemical Co., U.S.A

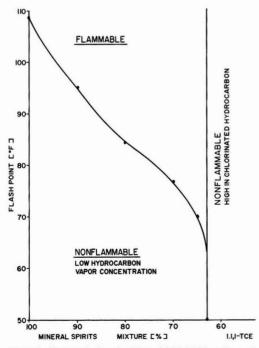


Figure 1-Mixture of mineral spirits and 1,1,1-trichloroethene at various flash points

for Flash Point of Liquids by Setaflash Closed Tester. Table 3 shows the flash point of the mixtures and the individual solvents used in the mixtures. The flash point recorded is the mean of two tests.

Table 3 also shows the results obtained after the storage of the mixtures for nine days and the flash point as determined by another laboratory after three weeks' aging. The results indicate that, as the concentration of 1,1,1-trichloroethane in the mixture increases, the flash point decreases until a concentration of about 35.0% 1.1.1-trichloroethane is reached. At about that concentration, the amount of the chlorinated hydrocarbon present in the mixture produces sufficient vapor to quench the test flame and prevent a flash from occurring.

Figure 1 shows a definite relationship between the concentration of 1,1,1-trichloroethane in a blend with mineral spirits and the flash points. Private information from other studies shows the same trend of a decrease in flash point with an increase in 1,1,1-trichloroethane up to a certain concentration, at which point the blend becomes nonflammable (has no flash point).

While this effect is evident with blends of mineral spirits and 1,1,1-trichloroethane, other blends may show an opposite effect. For example, Table 4 shows the results obtained with a blend of mineral spirits and methylene chloride plus a blend of the same two solvents with 1.1.1-trichloroethane. No flash point was evident with a blend of 95/5 mineral spirits/methylene chloride, nor with a blend of 86% mineral spirits, 11% 1,1,1-trichloroethane, and 3% methylene chloride. In this latter blend, the ratio of the concentration of mineral spirits to 1.1.1-trichloroethane was 88.7% to 11.3%. Using this ratio and interpolating in Figure 1, the blend should have had a flash point of 91°F. However, the addition of methylene chloride to make the three-solvent blend produces a mixture with no flash point.

These results with methylene chloride do not confirm Harve-Bazin's results. Observation of the flash point test with mineral spirits as it progressed to completion showed no flame enlargement but a definite flash point. Starting at 64°F, 1,1,1-trichloroethane exhibited a greenishyellow flame enlargement from that temperature until completion of the test at 115°F.

Rybicky and Stevens¹² have reported their flash point experiences with blends of toluene with various amounts of methylene chloride, 1,1,1-trichloroethane, or trichloroethylene. Table 5 shows their results with toluene/ trichloroethylene mixtures. Note that tests with the 33% blend of trichloroethylene showed that the vapor in the cup flashed at 8°C (46°F) and on continued heating, again flashed at 11°C and 14°C (52°F), but did not flash at 17°C (63°F) or 23°C (73°F).

The results in Table 5 do not show the decrease in flash point demonstrated for the mineral spirits/1,1,1-trichloroethane blend discussed earlier, but they do show an azeotropic effect; flash point did not vary appreciably as the percentage of chlorinated hydrocarbon increased. (This phenomenon also occurs with blends such as butyl alcohol/water.)

Table 6 shows additional Rybicky and Stevens flash point results for mixtures of toluene/methylene chloride and toluene/ chlorothene:

- An 11% methylene chloride blend had a flash point of 7.5°C (45.5°F) and it continued to flash on heating to 10°C (50°F), but it did not flash at 12°C (54°F) or 14°C (57°F).
- A 42% Chlorothene[®] blend had a flash point at 9°C (48°F), but when heated further to 10°C (50°F) and 12°C (50° or 54°F), no flash occurred.
- A 20% Chlorothene blend flashed at 6°C.

None of the results coincide with the mineral spirits/ 1,1,1-trichloroethane phenomenon discussed earlier; at least in the case of the 42% Chlorothene blend, an opposite effect is indicated.

In attempting to duplicate the experience of Rybicky and Stevens, we first determined flash points for the toluene*/chlorinated hydrocarbon blends listed in *Table* 6 using the Setaflash Tester (ASTM D 3278). The blends were prepared on a weight basis using materials "as received" from the supplier. (Chlorothene NU is reported to be 94.5% 1,1,1-trichloroethane by weight.) The last two columns of *Table* 6 show our results, all of which are well within the between-laboratory-reproducibility indicated in D 3278.

The next step was to use the Rybicky and Stevens technique of repeated tests on the same specimen at increasing temperatures. We found that we could not duplicate their results. Using a blend of 11% methylene chloride and 89% toluene and following the D 3278 procedure of allowing one minute for establishing an equilibrium condition between the liquid and its vapor at any one temperature, we obtained a flash at 50°, 54°, 58°, 62°, and 70° F. On repeating the test with a new sample and starting the test at 65° F, a flash occurred. When the test flame was inserted into the cup immediately after the first flash, no flash occurred. After the port was opened several times, a flash was produced at 66°F. Repeating the flash test immediately after the flashing at 66° F, no flash was observed, but a distinct odor of chlorine was noticed. After we blew across the port, there was a flash when the test flame was inserted into the cup.

In a third test with a fresh sample of the same composition, a flash occurred at 46° F (7.8°C) and the mixture kept on flashing as it was tested at 50° , 54° , 58° , and 60° F. Prior to each of the first four tests there was at least a minute hold, but when tested immediately after flashing at 60° F, no flash was observed and again, after an immediate recheck, there was no flash. However, after blowing across or into the port, a flash occurred.

To determine how toluene alone would react when treated similarly, toluene was heated to 42° F, the flash point temperature which had been determined previously. Again at this temperature, a flash occurred. When a flash test was immediately repeated, there was a flash, but when rechecked (second test after the first determination), there was no flash. The liquid was heated to 45.5° F where it flashed, and it flashed again when rechecked immediately. However, after the third recheck, there was no flash.

In a similar test, when a 42/58 blend of 1,1,1-trichloroethane/toluene was checked, a greenish to greenish-

	Table 5—Flash Points	
Df	Toluene/Trichloroethylene Mixtures ¹²	

	Flash Point, °C			
Trichloroethylene, %	Closed Cup	Open Cup		
2.6	5.0			
3.2	6.0	_		
5.8	6.0			
6.9	6.0			
9		8		
10	6.0	1		
14	6.0			
16	6.0	-		
18	6.5			
20	6.5	_		
29		10		
33	8ª	11		
40	11			
60	11			
74	_	30		
90	None			

yellow halo was observed as the temperature was increased to the flash point of 50° F (10° C) (the flash within the cup was greenish-yellow) and a flash was observed each time the temperature was increased by 4° F (2° C). However, if the flash test was repeated *immediately* after each test, there was no flash. When the port was opened sufficiently to remove combustion or decomposition products, a test resulted in a flash. This occurred also after blowing across or into the open port.

A fresh sample of the 42/58 blend was intentionally heated above its known flash point and tested at 74° F (23.3°C). A large flash was observed. An *immediate*, repeat test produced no flash, but one occurred after blowing across the port and again inserting the test flame. This is interpreted to mean that false negatives are not likely to be found when a toluene/chlorinated solvent blend is tested above its true flash point.

DISCUSSION

Rybicky and Stevens suggested the possibility of getting a false negative result if flash point is first checked

		Flash P	oint	
-	Rybi	icky	w	ray
_	°F	°C	°F	°C
100/0/0		_	42	5.5
89/11/0 4		7.5ª	47	8.3
58/0/42 4	8.2	9	50	10.0
80/0/20 4	2.8	6	48	8.9

(a) Flash occurred also at 10°C, but not at 12°C or 14°C

^{*}Redistilled 1°C toluol, obtained from Exxon Co., U.S.A.

at a higher temperature than the actual flash point of the specimen under test; the absence of a flash would indicate that the test material was nonflammable, though just the opposite is true. They suggest using an open cup test to overcome the reported safety deficiency of the closed cup. In an open cup, the more volatile chlorinated hydrocarbons evaporate to a greater extent than toluene and so the mixture has a flash point (see *Table 5*). However, when the U.S. Dept. of Transportation revised their regulations to specify the closed cup, they anticipated the potential problem and included an evaporation test to be run on mixtures of liquids with different volatilities and flash points. Specifically, 49 CFR 173.115 (d) (2) states, in part:

"... a second test shall be made on a sample of the liquid evaporated from an open beaker (or similar container), under ambient pressure and temperature ... conditions, to 90% of its original volume or for a period of four hours, whichever comes first. The lower flash point of the two tests shall be the flash point of the material."

Furthermore, a repeat test of the blend or solvent without use of a new specimen is contrary to the Setaflash Method ASTM D 3278 which states in note 7:

"Never make a repeat test on the same specimen (especially after a flash of the material being tested). Always take a fresh portion for each test."

Our results indicate that when this technique is followed, there is no danger of experiencing a lack of a flash of mixtures of chlorinated hydrocarbons and regular hydrocarbons. The flash of a 42/58 blend of Chlorothene and toluene at 74° F (twenty degrees Fahrenheit above the actual flash point), indicates that the phenomena observed by Rybicky and Stevens would not create a hazard from a false determination of a flash point at a higher temperature than the actual flash point. No difficulty will be experienced if a new sample is used each time a test is made and the method is properly followed.

Apparently, the reason for contrary results when testing for a flash immediately after the first flash is due to the formation of decomposition or combustion products. The decomposition products of chlorinated hydrocarbons (chlorine, hydrochloric acid, etc.) act as quenching agents



HARRY A. WRAY is a consultant on Flammable Liquids. He is a retiree of the Marshall Laboratory of the Finishes and Finished Products Department of E. I. du Pont de Nemours and Company, Inc. Mr. Wray is Chairman of ASTM Subcommittee on Health and Safety of ASTM D-01 on Paint and Related Coatings and Materials, and is past-Chairman of ASTM Coordinating Committee for Flash Point and Related Properties. He received the B.S. Degree from Albright College and the M.A. Degree in Chemistry from Columbia University. to quench the propagation of flame. Likewise, combustion products of hydrocarbons, CO_2 and water, act to quench the flame when hydrocarbons are tested immediately after an initial flash determination.

In addition, when the first flash is experienced there is a reduction in flammable vapors and of oxygen, preventing a subsequent flash. However, if the temperature is increased, or there is sufficient elapsed time between tests (at least a minute), there is developed sufficient flammable vapor and seepage of oxygen into the cup to provide fora flash. Furthermore, opening and closing the port of the test cup vapor space and it also removes most of the decomposition products of the chlorinated hydrocarbons, thus allowing the formation of a flash of the hydrocarbon vapors.

CONCLUSIONS AND PRECAUTIONS

From the experimental work and information reported, the following conclusions can be made:

(1) When testing the flash point of chlorinated hydrocarbons or mixtures containing these hydrocarbons and flammable solvent, the development of a greenishyellow flame enlargement (not to be confused with a "blue halo" sometimes encountered when testing other flammable solvents), should not be considered as the flash point of the specimen. Flame enlargement, the result of the decomposition of the chlorinated hydrocarbon vapor may be large, but, unless the flame spreads over the surface of the liquid in the flash tester, there is no flash point. Care should be exercised when interpreting the observations made during the flash point testing of these materials to be sure that there is actually flame propagation across the surface of the liquid.

(2) Formulators of coatings and other materials with chlorinated hydrocarbons should be aware of the possibility of a decrease in flash point on addition of these solvents to flammable solvents. The decrease may be sufficient to change the classification of a material from a "combustible" to a "flammable" liquid as defined by the U.S. Dept. of Transportation. Thus the flash point of mixtures of chlorinated hydrocarbons and flammable liquids should be tested prior to beginning of production.

(3) If a chlorinated hydrocarbon has no flash point but reduces the flash point of a flammable solvent when added to it, there is a concentration of the chlorinated hydrocarbon above which the mixture will have no flash point. It may, therefore, be necessary for formulators to determine this concentration and its effect upon other properties of their formulations.

(4) Some chlorinated hydrocarbons have high vapor pressures and, therefore, when blended with flammable solvents at ratios which initially are nonflammable may, in use, evaporate rapidly with an increase in the concentration of the flammable solvent and, thus, the mixture becomes flammable. The U.S. Dept. of Transportation's evaporation test should be conducted to determine the effect of evaporation on the flammability of the blend.

(5) Some solvents, when blended with chlorinated hydrocarbons, may produce blends in which the flash point does not appreciably change with change in composition.

(6) Formulators should be aware that some chlorinated hydrocarbons have flash points and thus will burn and others may have no flash point but may have flammable limits within which they may ignite and burn under specific conditions of temperature, pressure and ignition source, especially high energy sources. Thus, care should be observed when using these solvents in processing procedures.

(7) While some chlorinated hydrocarbons may not burn and others have only limited flammability, they are all easily decomposed by heat and, thus, should not come in contact with flames, electric arcs, or hot surfaces. These substances are decomposed into carbon dioxide, carbon monoxide, chlorine, and hydrogen chloride. Subsequent reaction between carbon monoxide and chlorine results in the formation of small amounts of phosgene, a highly toxic gas. Neglecting the very real possibility of an explosion, cutting, welding, and brazing should not be conducted on any container having contained a chlorinated hydrocarbon without adequate precautions to prevent inhalation of the toxic fumes produced.

(8) In using any closed cup tester, especially the Setaflash Tester, repeat tests should always be made with a fresh specimen.

(9) While an open cup flash point tester may indicate the hazard of a mixture when exposed to air in situations like a spill, the closed cup flash point approximates the temperature at which the vapor pressure of the liquid produces a concentration of flammable liquid equal to the LFL of the material and, therefore, is capable of producing ignition when an ignition source comes in contact with the vapor/air mixture. Thus the closed cup flash point more nearly describes the true fire hazard of a liquid enclosed in a container in transit, in storage, or in a process vessel.

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Effect of Pigmentation on Internal Stress In Latex Coatings

Dan Y. Perera and D. Vanden Eynde Coatings Research Institute*

The internal stress (S) in filled latex coatings was studied. The formulations investigated consisted of single filler systems (one binder/one filler) and mixtures of fillers. The experimental results show that the development of S depends on pigmentation (pigment volume concentration (PVC), type of filler).

With increasing PVC, for PVC < CPVC (critical pigment volume concentration) S increases, and for PVC > CPVC, S decreases. The CPVC-values obtained from S-measurements are in good agreement with those obtained by other methods. The shape of the curve describing S versus PVC was attributed to relaxation processes (important at low PVC's but restricted at higher PVC's). Close to or above the CPVC, relief processes can occur and affect the coating characteristics. The magnitude of S-values is mainly determined by the way the filler/binder interaction (reinforcing) affects the elastic modulus, one of the S-components.

Mathematical relationships were established to predict approximately the S and the CPVC for paints containing a mixture of fillers.

INTRODUCTION

Latex coatings in general are highly pigmented and one can expect that the level of pigmentation and the type of filler (i.e., pigment(s) and extender(s)) influence the coating properties, among them the development of internal stress. This is the case for thermoplastic coatings in solution.¹ For these coatings it was found, as a function of pigment volume concentration (PVC), that below the critical pigment volume concentration (CPVC) the internal stress increases, while above the CPVC the internal stress decreases, indicating the possibility of determining the CPVC from internal stress measurements. This dependence is mainly due to the variation of the elastic modulus (one of the internal stress components) on PVC.

From the literature it appears that the internal stress can affect the coating integrity by inducing cracking and/or detachment of the film.²⁻⁴ In latex coatings, mudcracking, a phenomenon to be avoided, was also attributed to the development of internal stress.^{5,6}

Certain aspects of internal stress development in latex coatings were discussed in a previous paper.⁷ It was shown that the development of internal stress (in one or two stages) is directly correlated with the film formation process. This study also indicated that, while the internal stress is dependent on pigmentation (PVC, type of filler), as in the case of thermoplastic coatings in solution, for latex coatings the relationship between S and PVC is different. In consequence, a more thorough study was undertaken in which single filler systems (one filler/one binder) and mixtures of fillers were considered.

EXPERIMENTAL

Materials

Some characteristics of the binders and the fillers investigated are given in *Tables* 1 and 2. In all paints the coalescing solvent used was Texanol[®].

Measurements

The following types of measurements were carried out: internal stress, evaporation kinetics, mechanical proper-

^{*}Avenue Pierre Holoffe, B-1342, Limelette, Belgium.

A	в	с	D
Specific gravity (g.cm ⁻¹)1.11	1.08	1.15	1.09
Particle size (average) µm0.25	0.10	0.50	0.10
MFT (°C) 13	22	14	8

(a) data quoted by manufacturers. A and B are two different styrene acrylic copolymers and C and D are, respectively, a vinyl acetate/vinyl versatate copolymer and a pure acrylic copolymer.

ties (MP), and density. The first two types of measurements are discussed in more detail in a previous paper.⁷

Mechanical (stress-strain) and density (determined from values of weight and volume) measurements were used to evaluate the CPVC-values. For the CPVCdetermination, it is only valid to use completely formed and dry films.⁸ Since after four months most of the coatings still contained substantial amount of volatiles (especially for PVC < CPVC), the films were first dried in a stove at 105°C until a constant weight was obtained and then reconditioned 30 days in a climate room.

Each measurement of internal stress and evaporation kinetics was made at least three times and that of mechanical properties at least ten times. All the experiments were made at 21 ± 0.8 (°C) and a relative humidity of 52 ± 1 (%).

RESULTS

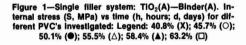
The experiments were made with four binders: copolymers of two styrene acrylics, of a pure acrylic, and of a vinyl acetate/vinyl versatate. The development of internal stress (S) as a function of time for two single filler systems is represented in *Figures* 1 and 2 and for a binary

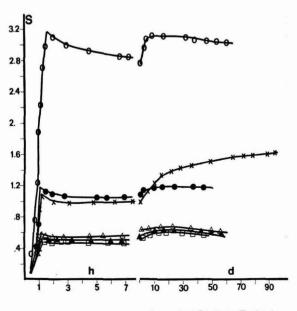
Table 2-Characteristics of Fillers^a

	TIO2		CaCO ₃	
A	В	A	в	Talc
Specific gravity				
Specific gravity (g.cm ⁻¹) 4.05	4	2.71	2.75	2.75
Oil absorption (g linseed				
oil/100 g) ~18	~20	~16.09	~15	~27
Specific surface area				
$(m^2.g^{-1})$ ~13.4	~10	~2.35	~2.5	_
Surface treatment Al.Si	Al.Si.Zn	-		-

filler system (a mixture of the two fillers) in *Figure* 3. Ternary filler systems and the coatings made from the pure acrylic copolymer and the vinyl acetate/vinyl versatate copolymer show similar S vs time-dependences and consequently they are not considered in this paper.⁹

An examination of the results obtained indicate that two stages characterize the development of S. These stages are discussed in detail in a previous paper.⁷ Briefly, in Stage 1 the internal stress increases relatively rapidly. This stage corresponds to the transition phase (Tr) of the evaporation kinetics when the greatest part of coalescence occurs (see also *Figure 4*). In Stage 2, which corresponds to Phase 2 of evaporation kinetics, one can see that, depending on the PVC and the type of filler, S can decrease or first decrease and then increase. These behaviors are due to the fact that in Stage 2(more so than in Stage 1) the relaxation and/or the relief processes induced by the development of internal stress and which diminish the values of S now play a significant role. As will be discussed later, the relaxation processes occur





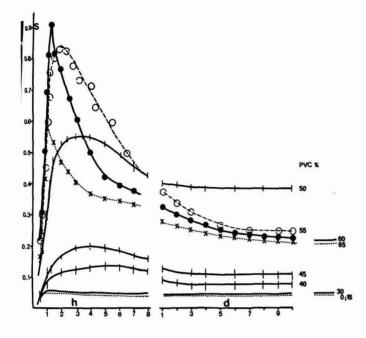


Figure 2—Single filler system: $CaCO_3(A)$ — Binder(A). Internal stress (S, MPa) vs time (h, hours; d, days). The numbers in the figure indicate the different PVC's (%) investigated.

mainly in paints of low PVC while relief processes (breakage of filler/binder bonds, appearance of microfissures) occur mainly in paints of higher PVC.

The dependence of the maximum internal stress (Sm) on PVC and $\Lambda^{10,11}$ (reduced PVC, PVC/CPVC) is given in *Figure* 5 (see also *Figure* 6 for other single filler/binder systems). These figures show that:

(1) Sm is a function of PVC. Sm increases with increasing PVC until a certain PVC level is attained and then decreases. This PVC level is the CPVC. A comparison of CPVC-values obtained from internal stress and other measurements for some of the coatings investigated is given in *Table 3*.

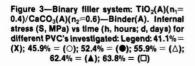
(2) the magnitude of Sm-values are dependent on the type of filler. There are fillers which induce relatively high Sm-values while there are other fillers which induce low ones.

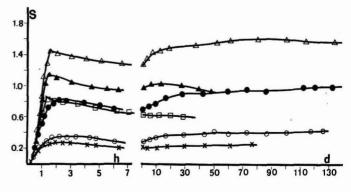
(3) the above results are independent of the type of binder investigated. The values of Sm are affected by the type of binder but the conclusions concerning the Smdependence on PVC are identical.

Mixture of Fillers

Since commercial paints usually contain a mixture of fillers, as already mentioned, experiments were also carried out with such systems. An analysis of the results obtained with binary and ternary filler systems (see *Figure 5* and *Tables 4* and 5) indicates that for a paint formulated at a $\Lambda \leq 1$, the maximum internal stress (S₄) can be predicted reasonably well by means of an additivity rule:

$$S_t = n_1 S_1 + n_2 S_2 + \dots + n_i S_i = \sum_{x=1}^{x=1} n_x S_x$$
 (1)





S	MPa	đ	Equation (2)
CaCO ₃ (A)61.5 ± 1.5		61 ± 1.5	-
$TiO_2(A)$	47.0 ± 1.5	47.5 ± 1.5	—
TiO ₂ (A)			
$(n_1 = 0.4)/CaCO_3 (A)(n_2 = 0.6)57.0 \pm 1.5$	56.0 ± 1.5	56.5 ± 1.5	55.3 ± 1.5
(a) MP = elastic moduli and ultimate properties			

Table 3—Values of CPVC (%) Determined by internal stress (S), mechanical properties (MP), density, and calculated from equation (2). Binder = Styrene-Acrylic (A)

where: n_1 , n_2 , n_i = volume fraction of different fillers present in the mixture

S_1 , S_2 , S_i = maximum internal stress of different single filler systems at a given Λ (below or equal to 1)

Equation (1) indicates that the contribution of each component to S_t is independent of the presence of other components.

From the above experiments (see Figure 5 and Table 3), it was also found that the approximate CPVC of coatings containing a mixture of fillers (Ψ_T) can be calculated from:

$$\frac{1}{\Psi_{\rm T}} = \frac{n_1}{\Psi_1} + \frac{n_2}{\Psi_2} + \cdots + \frac{n_i}{\Psi_i} = \sum_{{\rm X}=1}^{{\rm X}=i} - \frac{n_{\rm X}}{\Psi_{\rm X}}$$
(2)

where n_1 , n_2 , n_i and Ψ_1 , Ψ_2 , Ψ_i are, respectively, the filler volume fractions and the CPVC of different components (single filler systems). It can be shown that equation (2) is similar to the equation defining PVC for a coating containing a mixture of fillers. Such a relationship, however, may not take into account the possibility (1) that particles of one type of filler might fill the voids between the larger particles of another type of filler, a situation which increases the packing density, nor (2) that agglomerates can form during mixing, a situation which decreases the packing density.

It is probably worth mentioning that equations (1) and (2) are also valid for the thermoplastic coatings in solution containing a mixture of fillers.¹²

DISCUSSION AND CONCLUSIONS

The dependence of the maximum internal stress (Sm) on PVC and/or Λ (see *Figures* 5 and 6) below the CPVC is interesting enough to necessitate a comment. One can see that, independent of the type of filler, the dependence of Sm on PVC is different at a low PVC from that at a higher PVC. At low PVC's, the increase of Sm is small and gradual, whereas at higher PVC's (especially close to the CPVC) the increase is marked and abrupt. If one compares this type of dependence with that of other types of coatings (e.g., thermoplastics in solution¹), where the dependence of Sm on PVC is much closer to linearity (see *Figure* 7), the shape of the curve for latex coatings might appear somewhat unusual. In the author's opinion, this behavior can be explained qualitatively if one considers that:

a) the glass transition temperatures (Tg) of the binders used in latex paints investigated are much smaller than the temperature (T = $21 \pm 0.8(^{\circ}C)$) at which the films form (Tg < T). This means that the mobility of binder segments is great and, consequently, the relaxation process occurring during the film formation as a result of the internal stress developed is important.

b) there is a large difference in mobility between that of the binder segments adsorbed to the filler (which are partially or totally immobilized) and that of the segments of the free (bulk) binder (which are very mobile).

c) an increase in filler concentration increases the immobility and the number of adsorbed binder segments.

At low PVC's the free, mobile (non-adsorbed) binder segments are sufficient to overcome the effect of the immobilized segments and to enable the greatest part of the internal stress arising during film formation to relax. As a result, the Sm-values are relatively small and increase steadily with the increase of PVC.

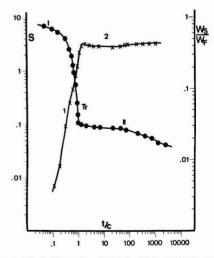


Figure 4—Single filler system: TiO₂(A)—Binder(A); PVC = 45.7%. Internal Stress (S, MPa-(X)) and evaporation kinetics (W_s/W_F-(\bullet)) measurements; t = time (minute); c = film thickness (μ m); W_s = weight of volatiles present in the film; W_F = weight of the dry film

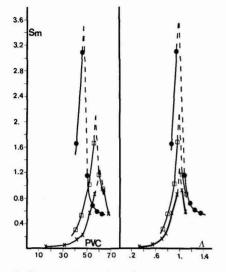


Figure 5—Maximum internal stress (Sm, MPa) dependence on PVC (%) and Λ [reduced PVC (PVC/CPVC)] for the single filler systems: Legend: TiO₂(A)—Binder(A) = (\bullet) and CaCO₃(A)—Binder(A) = (X); and the binary filler system TiO₂(A)(n₁ = 0.4)/CaCO₃ (n₂ = 0.6)—Binder(A) = (D)

d) there is a PVC-range within which the majority of binder segments are immobilized.* Under these conditions the internal stress developing during the film formation cannot relax and the internal stress values will be relatively high.

The above explanations are endorsed by the similarity of Tg and Sm-dependences on PVC^{13,14} [the increase of Tg with increasing PVC, especially at high PVC, is interpreted as a proof that binder segments are immobilized (adsorbed) by the filler¹⁵].

On the basis of the above model one can also explain the dependence of Sm on PVC (see *Figure* 7) for other types of coatings mentioned above (e.g., thermoplastics in solution). Since for most of these binders the Tg > T, the relaxation process is reduced as a result of the restricted mobility of binder segments, and this is independent of the presence of a filler in the paint. For such cases the

 Such a PVC-range probably corresponds to the situation in which the distance between the filler particles is small enough to enable most of the binder sites to reach the filler particles and, consequently, to become immobilized.

Table 4—Maximum internal stress values obtained experimentally (Sm) and those calculated (St) from equation (1) at different values of reduced PVC (Λ) for the binary filler system: TiO₂ (A) (n₁ = 0.4)/CaCO₃ (A)(n₂ = 0.6); Binder = Styrene-Acrylic (A)

Λ	Sm(MPa) (exp)	St(MPa)
0.86		0.97 ± 0.09
0.9	$\dots \dots 1.15 \pm 0.10$	1.20 ± 0.11
1		2.16 ± 0.16

Table 5-Maximum internal stress values obtained experimentally
(Sm) and those calculated (St) from equation (1) for three
formulations. Binder: Styrene-Acrylic (B)

Λª	Sm(MPa)	St(MPa)
$TiO_2 B(n_1 = 0.5) / CaCO_3$		
$B(n_2 = 0.5) \dots \dots$	0.74 ± 0.08	0.80 ± 0.080
$TiO_2 B(n_1 = 0.5) / Talc$		
$B(n_2 = 0.5) \dots 0.845$	0.65 ± 0.06	0.60 ± 0.058
TiO_2B (n ₁ = 0.333) /		
$CaCO_3 B(n_2 = 0.333) /$		
Talc $B(n_3 = 0.333) \dots 0.840$	0.48 ± 0.04	0.53 ± 0.050
(a) Λ = reduced pigment volume concentration	on (PVC/CPVC)	
(a) A = reduced pigment volume concentrati	on (1 (C/C1 (C)	

internal stress increases with PVC mainly because the elastic modulus (one of the S-components) increases with the PVC as a result of stiffening of the coating (an inorganic filler is usually much stiffer than any organic binder).

For the filled latex coatings, as in the case of thermoplastic coatings in solution,¹ by combining the internal stress and evaporation kinetics measurements it can be demonstrated that among the internal stress components (i.e., elastic modulus, internal strain and Poisson's ratio) the elastic modulus is the one which has the greatest effect on the internal stress.

This also explains the reason why at PVC > CPVC, and independent of type of binder and filler, Sm decreases by increasing the PVC. This effect is due to the decrease of the elastic modulus as a result of the continuous increase of the film discontinuity.

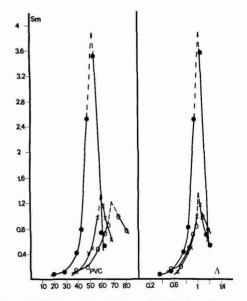


Figure 6—Maximum internal stress (Sm, Pa) dependence on PVC (%) and Λ (reduced PVC) for three single filler systems: TiO₂(B)—Binder(B) = (\bullet); CaCO₃(B)—Binder(B) = (X); Talc-Binder(B) = (O)

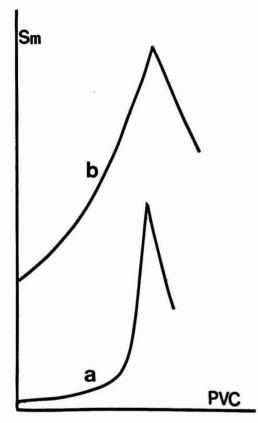


Figure 7—Schematic description of the maximum internal stress (Sm) dependence on PVC for coatings possessing: (a) Tg < T (most latex coatings); (b) Tg > T (most thermoplastic coatings in solution)

As mentioned previously, the magnitude of the maximum internal stress values are dependent on the type of filler. Figures 5 and 6 show that the values of Sm obtained with titanium dioxides, especially near the CPVC, are more than three times higher than those obtained with the extenders calcium carbonate and talc. This behavior is due to the filler/binder interaction which is determined by the nature, and especially by the number, of the interacting sites (acid/base) present on the filler surface and the acid/base character of the binder.¹⁶ Since (a) the specific surface area of titanium dioxides (see Table 2) is much higher than that of the extenders, and (b) both the binder and the extenders possess basic sites, one can expect that the extenders induce a much weaker filler/binder interaction (reinforcing effect) than a titanium dioxide which in addition possesses acid sites.

These considerations also indicate that the process inducing the internal stress relief in certain coatings investigated (see Figures 1-3, especially Figure 2) is dependent on the filler/binder interaction. When the internal stress attains a value higher than that of the adhesion strength between binder and filler and/or that of the coating cohesion, filler/binder dislocations (dewetting) and/or (micro)fissures, all processes relieving the stress in the coating, will occur. This implies that the first type of relief process predominates in coatings containing the extenders calcium carbonate and talc while the second type predominates in coatings containing titanium dioxide. This is confirmed experimentally, because during the period in which S decreases with time: (a) no damage is observed visually or with an optical microscope in the coatings containing only the extenders, and (b) microfissures are present around agglomerates (for PVC \simeq CPVC) and through the whole film (for PVC > CPVC) in the coatings containing only titanium dioxide.

These facts also imply that for the coatings in which relief processes are observed, the internal stress measurements can be used to determine accurately the CPVC only if they occur after the Sm-values are reached, a situation which seems to prevail for the coatings investigated (see *Table* 3).

The occurrence of relief processes (dewetting, microfissures) in certain coatings (especially above or close to the CPVC) supports the arguments presented in reference (8) on the difficulties of determining accurately the CPVC by experimental methods.

SUMMARY

The development of internal stress in latex coatings is dependent on pigmentation (PVC, type of filler). As in the case of other coatings (e.g., thermoplastic in solution) below the CPVC the maximum internal stress (Sm) increases with increasing PVC, while above the CPVC the maximum internal stress decreases. This enables one to use internal stress measurements to determine the CPVC

Below the CPVC, the slope of Sm vs PVC is different at a low PVC (i.e., small and gradual) from that at a higher PVC (i.e., marked and abrupt). This was explained on the basis that the relaxation processes are important at low PVC's while they are restricted at high PVC's.

The magnitude of Sm-values is affected by the type of filler. This is due to the reinforcing effect of each filler, an effect which depends mainly on the filler particle size but also on the nature of the interacting sites present on the filler surface and the acid/base character of the binder.

Among the internal stress components (i.e., elastic modulus, internal strain and Poisson's ratio) the elastic modulus is the one which mainly affects the development of internal stress.

Mathematical relationships were established to calculate approximately the Sm and the CPVC of a coating containing a mixture of fillers. They require only a knowledge of the filler volume fractions (n_i) and the internal stress or the CPVC of single filler systems.

ACKNOWLEDGMENT

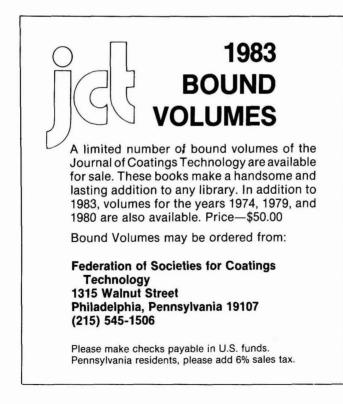
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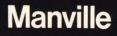
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Environmental Control Or Coatings Composition As a Means for Reducing Cathodic Delamination Of Organic Coatings

Henry Leidheiser, Jr., Wendy Wang, Richard D. Granata, Hyacinth Vedage, and Malcolm L. White Lehigh University*

Described are observations which indicate that environmental variables change the rate of cathodic delamination. The rate of delamination was observed for both a polybutadiene coating and a fluorocarbon coating on abraded steel substrate, The conclusion was reached that since the coating that adjoins the metal is the environment in which the delamination occurs, it is this environment that the coatings formulator can control.

INTRODUCTION

One of the methods by which painted steels deteriorate by an electrolytic process is through the accumulation of alkali as a consequence of the cathodic, oxygen reduction reaction occurring under the coating. This effect is known as cathodic delamination and it shows up during the salt spray testing of scribed steel, during cathodic protection of coated objects such as pipelines and ships, and during the electroless deposition of copper at openings in a photoresist. The phenomena associated with cathodic delamination are moderately well understood,^{1,2} although there is disagreement among workers in the field as to the exact mechanism by which the alkaline environment breaks the bond between the coating and the metal.³⁻⁵

The ideal method for protecting against cathodic delamination would be the development of a system in which the interfacial region is very resistant to alkali. Dickie and co-workers⁶ have observed that certain epoxy systems on steel have good resistance to alkali and perform better in tests which measure the consequences of cathodic delamination. Another method, admittedly impractical in most instances, is to control the environment such that cathodic delamination does not occur. It is the purpose of this paper to describe two older observa-

tions and two recent observations which indicate that environmental variables do indeed change the rate of cathodic delamination.

OBSERVATIONS

It has previously been shown that cathodic delamination does not occur when oxygen is excluded from the solution and the potential is maintained in the range where the oxygen reduction reaction is the predominant cathodic reaction.² Elimination of oxygen provides one method of control that may be applicable in certain circumstances. It has also previously been shown that at

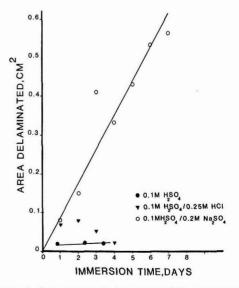
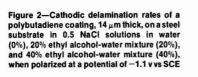


Figure 1—Data showing the importance of the presence of alkali metal ions in the electrolyte in the cathodic delamination phenomenon. Experimental conditions: ground-finish steel substrate; fluorocarbon coating, 125 µm thick; room temperature, cathode potential of -1.2 v vs SCE

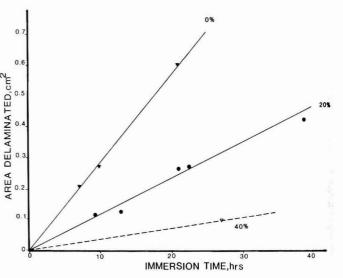
^{*}Dept. of Chemistry and Center for Surface and Coatings Research, Sinclair Laboratory #7, Bethlehem, PA 18015.



equal molar concentrations the rate of delamination in solutions containing alkali metal ions increased in the order Li⁺, Na⁺, K⁺, Cs⁺.² This relative order has been since confirmed in many other coating/metal substrate systems. Our present interpretation of this alkali ion effect is based on the fact that the relative rates of diffusion of these ions in aqueous solution increase in the order Li⁺, Na⁺, K⁺, and Cs⁺ and that the rate of development and the concentration profile of the alkaline environment at or near the delaminating front are determined by the rate at which the cation counterion can reach the site where the OH⁻ ion is being formed. The use of lithium salts instead of sodium or potassium salts may be possible in some closed systems.

Two recent examples of the effect of environment will be cited. Figure 1 shows the rate of delamination of a fluorocarbon coating from abraded steel when polarized at a potential of -1.2 v vs SCE in a 0.1M H₂SO₄ electrolyte. The addition of chloride as HC1 had no significant effect in increasing the rate. However, when the solution was made 0.2M in Na2SO4, the delamination rate became appreciable as shown by the top curve in Figure 1. The insignificant delamination rate in the pure acid is a consequence of the fact that the H⁺ ions are the major current carriers to the cathodic sites on the surface. These ions neutralize the OH⁻ ions that are formed and the pH never becomes high enough to lead to deterioration of the interfacial region. When high concentrations of Na⁺ ions are present, these ions become charge carriers and they provide the necessary counterions under the coating and permit the pH to become high enough to attack the interfacial region.

The second example is shown in *Figure* 2 for the cathodic delamination of a polybutadiene coating from an abraded steel in 0.5M NaC1 containing 0, 20, and 40% by volume concentrations of C₂H₅OH. The rate of



delamination decreased markedly as the alcohol concentration was increased to 20 and 40%. The conductivities of these three solutions are approximately the same and the cathodic polarization curves are approximately the same in all three solutions. The differences in behavior cannot be accounted for by either different conductivities or different cathodic polarization behavior.

CONCLUSION

The nature of the environment has an important effect on the rate of cathodic delamination. Since the coating that adjoins the metal is the environment in which the delamination occurs, it is this environment which is under the control of the coatings formulator. We present the above results as a challenge to coatings formulators to select additives for coatings that will make them less sensitive to cathodic delamination.

ACKNOWLEDGMENT

Appreciation is expressed to the Office of Naval Research and the Electric Power Research Institute for the support they provided which resulted in this article.

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DIVERSIFIED PRODUCTS GROUP

Gadgets and Gimmicks . . .

Presented at the Southern Society Annual Meeting*

PRIZE-WINNING ENTRY

Benham-Gachette Field Test Viscosity Can Tester

JACK E. BENHAM and JULES GACHETTE

Commercial Coatings Corp. 3501 N.W. 74th St. Miami, Florida 33147

Many times, when on technical service field trips, the occasion arises when a viscometer would be useful for conducting comparative viscosity checks, and none are handy. However, on most construction sites or in factories soft drink cans are readily available.

To construct the Benham-Gachette Field Test Viscosity Can Tester, just pick up an empty 12 fluid ounce aluminum soda can and, using a sharp knife, a sharp pair of scissors, or preferably, a metal hand saw, cut off the top of the can one inch from the top. Drill a $\frac{1}{4}$ to $\frac{1}{4}$ in. hole in the side of the can $\frac{2}{2}$ in. from the bottom. Then bevel the bottom with a hammer or some other blunt, heavy instrument using a hard piece of wood so that the bevel is $\frac{1}{4}$ in. from the bottom side of the can to the center of the bottom of the bevel. Next, drill a $\frac{1}{8}$, $\frac{1}{4}$, $\frac{1}{8}$, or $\frac{1}{2}$ in. hole in the center of the beveled bottom (*Figure* 1).

After heating or cooling the paint to be tested to normal room temperature (70-85° F), fill the can by placing the finger over the bottom hole and pouring the paint until it runs out of the side hole (the can will hold about 9½ ounces of paint). Remove your finger from the bottom hole, allowing the paint to run freely and, using the second hand on your watch or a stop watch, if available, time the total seconds it takes for the stream of paint coming through the bottom center hole to break at a point less than one inch from the can.

Figure 2 gives the comparative data for both Krebs units and #4 Ford Cup seconds so you may interpolate the field results and to also know which model should be used.

MODEL	CENTER HOLE	RANGE (KU)	
412-18	1/8"	50	
412-28	1/4 "	50-90	
412-38	3/8"	90-120	
412-48	1/2 "	120-140	

The accuracy of your B-G Can Tester is directly proportional to the accuracy of the size of the can holes (within 5%). We suggest that you calibrate each product to be tested in the lab with one of the B-G Can Testers and your lab viscometer. Use at least two different viscosities 10 units apart.

Continuous Drum Filling From a Vorti-Siv

JAMES E. TISHEUAR

The Gilman Co., Inc. P.O. Box 1257 Chattanooga, Tennessee 37401

Our goal was to reduce the time required to fill drums and clean up. Materials used to achieve this were: (1) set of drum rollers; (2) plastic sheet; (3) piece of four inch box guttering; and (4) air wrench.

A 330 gallon portable tank of solvent based enamel was placed on our filling dock and an air agitator was placed in the tank for constant agitation during the filling cycle. A Vorti-Siv was placed under the filling spigot, and a plastic sheet with an eight-inch diameter hole through which the paint flowed to the pan, was placed over the pan to catch paint splashes. A set of drum rollers was placed in front of the Vorti-Siv, and a 55 gallon open head drum was placed on the rollers. Grounding was done according to plant procedures, and the filling operation began.

As the first drum fills, the second drum is placed beside it so a quick change of drums can be made when the paint reaches a marked level in the first drum. The operator then uses the guttering to divert the flow of paint to the second

^{*} Introduced at the Annual Meeting of the Southern Society for Coatings Technology, March 7–9, 1984, in Clearwater, FL.

SOUTHERN SOCIETY

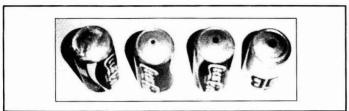


Figure 1

drum without shutting down the Vorti-Siv and using the other container to catch or stop the paint flow. The operator then moves the full drum away and closes the lid with an air wrench to save time.

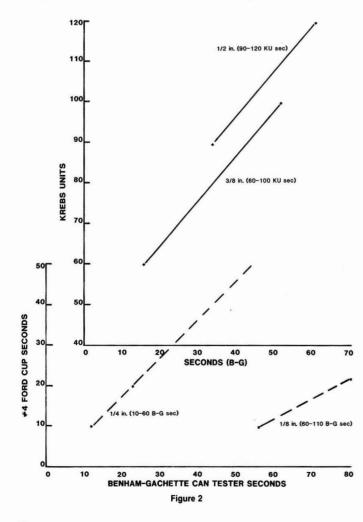
Drum filling time is about 2½ minutes. The operation is repeated until all six drums are filled. Total time to fill all drums and clean the Vorti-Siv is 45 minutes.

"Mud-Cracking" Test Procedure

DAN DIXON

Freeport Kaolin Co. P.O. Box 337 Gordon, Georgia 31031

"Mud-Cracking" is a general term applied to the phenomena of intersecting cracks which penetrate at least one coat in.



a dried paint film. Some latex paint systems are prone to mud-crack, especially if applied to porous substrates and when dried at low temperature.

Currently, ASTM methods for mudcracking of interior latex systems do not exist for guidance, recommendations or simplicity. In the past, paints were poured onto porous substrates such as sheetrock or wallboard, then tilted at a 45° angle for testing. These paints had different viscosities and film thicknesses, depending on the pigmentation.

A method for elimination and testing of vast numbers of pigments was developed for speed and accuracy. A quick test procedure as simple as pouring the paint into a small cylinder and pulling the film down onto a substrate was developed. A simple device was constructed to allow films of 40-50 mils, thus eliminating the variable of thickness. A $2\frac{1}{2}$ " stainless steel stock, $1\frac{1}{4}$ " high was constructed and notched out to cast a 45 mil thickness film (See *Figure* 3). Cracks will appear as the film dries.

Other than the drawdown device, this procedure requires no special equipment. Any porous substrate will do – sheetrock, wallboard, cardboard, etc.

High Speed Laboratory Mill

S. G. SANFILIPPO

Reichhold Chemicals, Inc. P.O. Box 1610 Tuscaloosa, Alabama 35403

A low cost laboratory high speed dispersant will duplicate plant batches which are made on larger and more sophisticated equipment.

EQUIPMENT

- (1) Veriac
- (2) Sears Router (1-1/2 horse power)
- (3) ¹/₈" stainless steel shaft, approx. 12" in length with one end threader
- (4) High speed blade approximately 11/2" in diameter
- (5) Laboratory equipment stand
- (6) Tackometer

ASSEMBL.Y

Attach the high speed disperser blade onto the shaft by screwing a nut onto the stainless steel shaft followed by a washer, then followed by the high speed disperser blade; finishing up with another washer and another bolt to hold it together. The blade must be centered properly to minimize and/or avoid whimping. Insert shaft into router, check and tighten as per instructions. Attach electrical cord or router for source of power and determine power versus rpm. by use of a tackometer. By getting a reading on the tackometer of the rpm. of the shaft, one can determine on a chart power versus rpm. This would

Journal of Coatings Technology

correspond with the rpm, which are being utilized in plant production to make the various batches of paint.

CONCLUSION:

As most equipment is readily available in the majority of coatings and paint laboratories, coming up with a laboratory high speed disperser utilizing the above equipment will be fairly cheap. More important than cost is that laboratory procedures can now readily be transposed into actual plant procedure with little or no guesswork.

A System to Minimize Time **On Retrieving Resins** And Pigments

JAMES W. LEE

Wyandotte Paint Products Co. 6369 Old Peachtree Rd. Norcross, Georgia 30071

Resin and pigment cans always seem to get mixed up, and one can spend a lot of time sorting through them to find the pigment or resin one needs.

To simplify retrieving the cans, paint them the color that will be put in them. The pigment cans are easily painted their respective color; the resins cans can be color coded with pastel colors and then stenciled with the name of the resin. At a glance, then, one can locate the color code of a particular resin or pigment.

Below are listed some examples of what we have used:

RESINS	COLOR
Alkyd	Buff
Acrylic	Avocade
Hard Gum	Wheat
Cellulose	Lt. Mar
Silicones	Lt. Orai
Urea &	
Melamine	Lt. Gray
Vinyls	Lt. Pink
Water Base	Lt. Gree
Epoxy	Lt. Blue

PIGMENT

White Yellow Red Black Orange Brown Green Blue Metallics Extenders CODE 0 oon nge en

COLOR CODE White Yellow Oxide Red Black Orange Brown Green Blue Aluminum Dk. Gray



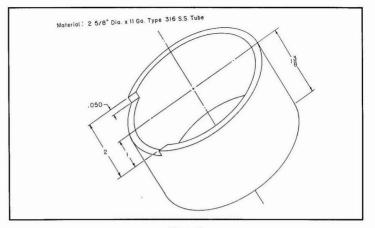


Figure 3

Single Pigment System of Pigment Evaluation

PAM WYCHE

Freeport Kaolin Co. P.O. Box 337 Gordon, Georgia 31031

The Single Pigment System (SPS) is a fast, easy method used to compare two or more pigments or slurries side by side in an emulsion system, on either a weight or volume basis. Optical differences in pigments are quickly observed using this method. Physical differences are not tested here due to the small volume and character of the "paint".

Virtually a lab batch "mini-paint", the SPS incorporates the most common materials that usually go into a paint (see Procedure) and the pigment or slurry chosen. The advantages of this test are: (1) the small amount of pigment needed; (2) the short grinding time; (3) the lack of need for specialized equipment; and (4) the short time span from make down to visual inspection of the dry paints on the opacity panel. The equipment needed for SPS comparison should be on hand in most paint laboratories.

THE SINGLE PIGMENT SYSTEM

Scope:	To determine optical differences
	between two or more pigments in
	an emulsion system.
r .	

- Equipment: (1) Balance (weighs to nearest tenth or better)
 - (2) Dispersator
 - (3) Opacity-Penetration panels and Bird bar (0.003'')
 - (4) Reflectometer with optional sheen attachment
 - (5) Beaker, 400 ml, or plastic 16 oz. cup

Materials:	Basic Formulation (gra	ms)
	Thickener, 2% sol	75
	Water	50
	Ethylene glycol	
	Dispersant (liquid)	
	Defoamer	2
	Preservative	0.3
	Pigment	100
	Grind above 10 min the	n add:
	Water	25
	Latex emulsion	50

Procedure: Make paint as outlined in formulation, (may put through paint screen) then pull down onto opacity panels using Bird bar. Allow to dry, then read with reflectometer or visually inspect if no readings needed.

Open Forum is an experiment in communications designed to give readers the opportunity to share creative concepts in all aspects of coatings. Suggested topics include color, formulation and manufacture, testing, and selection of raw materials. These "tricks of the trade" need not be prepared formally such as research papers, but should, however, be thorough in their preparation and presentation. Submissions should be sent to "Open Forum" Editor, Journal of Coatings Technology, 1315 Walnut St., Philadelphia, PA 19107.

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235 EAST 42ND STREET NEW YORK. N.Y. 10017 Dept. 4C-2 These subcommittee reports are for the use of the membership of ASTM Committee D-1 in continuing its work and for the interest of readers in the activities of ASTM Committee D-1. The reports are not official actions of ASTM and may not be quoted as representing any action or policy of the American Society for Testing and Materials.

May 1984 Subcommittee Reports Of ASTM Committee D-1

The May meeting of ASTM Committee D-1 on Paint and Related Coatings and Materials was held on May 20–23, 1984 at the Queen Elizabeth Hotel, Montreal, Canada. In the three and one-half days preceding the final session and general meeting of Committee D-1, 130 members and guests met in 138 scheduled meetings of D-1 subcommittees and working task groups. The present membership of Committee D-1 is 507.

Memorial Resolution was presented on the passing of Carlye M. Lochboehler.

Special Highlight of the meeting was the presentation of the following awards: W. T. Pearce Award to Peter Kamarchik, Henry A. Gardner Award to Harry A. Wray, and Certificates of Appreciation to H. Fujimoto, C. W. Fuller, J. Peters, D. L. Campbell, and A. Beitelman. Another highlight was a talk by H. E. Ashton on the subject of "Canadian/U.S. Standards Relationships."

Highlights

The following items of major interest to the coatings industry warrant special emphasis:

EMMA Tests on Automotive Coatings—Sub. D01.27.10 has initiated new interlaboratory test to compare automotive coatings in the present EMMA night-time-wetting cycle and in a modified cycle in which higher panel temperature will be used. Concurrent tests on the same coatings will be run at several sites in Florida (open rack, black box,, and 5° plywood-backed racks).

Microbial Spoilage Prevention—Sub. D01.28.01 is seeking participants in a round-robin to evaluate the Presumptive Challenge Test Method for determining the capacity of preservatives to prevent microbial spoilage of paint.

Water-Miscibility of Water-Soluble Solvents-Sub. D01.35.10 discussed the problems associated with the method, D 1722. Shippers have expressed concern over nebulous results allegedly causing excessive demurrage. Test results or literature is solicited which may document usefulness of this test.

Bridge Condition Assessment—Sub. D01.46 agreed that a need exists for a guide for surveying bridges to assess the coating

deterioration. A new group will meet next January to consider a scope.

Prefinished Hardboard Siding—Sub. D01.52.13, a new task group, will review test needs in specifying prefinished hardboard siding. Tests presently being used are solicited.

Discharge Characteristics of Paint Brushes and Rollers— Sub. D01.61 has initiated two task groups to test methods for determining what test methods will provide satisfactory results on the subject.

Future Meetings

January 20-24, 1985—Orlando, FL (Marriott) July 7-11, 1985—San Diego, CA (Town & Country Hotel) January 12-16, 1986—New Orleans, LA (Sheraton New Orleans) June 15-19, 1986—Philadelphia, PA (Franklin Plaza) January, 1987—Orlando or Tampa, FL area June, 1987—Dearborn, MI (Hyatt Regency)

New Standards

New standards since the May, 1984 meetings of D-1 approved by the ASTM Committee on Standards in the months shown: (subcommittee jurisdiction)

New D-1 standards approved by the ASTM Committee on Standards in the month: (Subcommittee jurisdiction)

January 1984

D 4301-84, Test Method for Total Chlorine in Epoxy Resins and Compounds (Sub .33)

April 1984

D 4358-84, Test Method for Lead and Chromium in Air Particulate Filter Samples of Lead Chromate Type Pigment Ducts by Atomic Absorption Spectroscopy (Sub .21)

D 4359-84, Test Method for Determining Whether a Material is a Liquid or a Solid (Sub .22)

D 4360-84, Specification for Metal in a Small n-Amyl Ketone (Sub .35)

D 4361-84, Test Method for Parent Apparent Tack of Printing Inks by the Inkometer (Sub .56)

D 4368-84, Guide for Testing Polyvinyl Chloride Resins (Sub .33)

June 1984

D 4366-84, Method for Hardness of Organic Coatings by Pendulum Damping Task (Sub .23)

D 4367-84, Test Method for Benzene in Hydrocarbon Solvents by Gas Chromatography (Sub .35)

D 4370-84, Test Method for Acid and Base Millequivalent Content of Electrocoat Bath Samples (Sub .21)

New Standards in D-1 related committees approved by ASTM Committee on Standards in the months:

D-33 Committee on Protective Coating and Lining Work for Power Generating Facilities

January 1984

D 4286-84, Practice for Determining Coating Contractor Qualifications for Nuclear Powered Electric Generating Facilities (Sub .07)

E-12 Appearance of Materials

April 1984

E 991-84, Practice for Color Measurements of Fluorescent Specimens (Sub .02)

DIVISION 1 ADMINISTRATIVE

SUBCOMMITTEE D01.05 INTERCOMMITTEE RELATIONS

J.C. Weaver, Chairman

Liaisons between D-1 and other ASTM committees and with organizations outside of ASTM grow more complex. Some of these are urgent, as with the compliance procedures imposed by EPA on limits on VOC, volatile organic content, of automotive, coil, container, ink, and other factory-applied coatings. D-1's new vice-chairman-liaison, Hiro Fujimoto, will be aided if the chairman of each of the 28 technical subcommittees in D-1 will assign a task group number to "Liaison," list thereunder the relevant ASTM or other bodies and be responsible directly for liaison results and possible problems thereon.

SUBCOMMITTEE D01.06 USA COMMITTEE FOR ISO/TC 35 ON PAINT AND PAINT MATERIALS AND TC 50 ON LAC

J. C. Weaver, Chairman

TC 35's next plenary meeting will be hosted by the Canadian government, May 19-26, 1985, in Hull, Quebec, across the river from Ottawa. Meeting with the main committee will be its subcommittees: 1—Terminology, 9—General Test Methods for Paints and Varnishes, 10—Test Methods for Binders for Paints and Varnishes, and 13—Drying Oils.

TC 35's approximately 150 standards are mostly parallel to some of D-1's 500 standards and are listed in the D-1 Table of Contents order in the backs of ASTM Standards, Volumes 06.01, 06.02, and 06.03. Details may vary in relevant pairs. D-1 subcommittee chairmen are urged to review relevant TC 35 standards toward improvement of their own works.

Toxicity regulation of paints allegedly due to the heavy metals, lead, etc., at very low levels, obviously depends on careful precisions of analyses. Canadian insistence has caused ISO/TC35/SC9/WG14, with its secretariat in DIN, Berlin, to collaborate with ISO/TC181 on Safety of Toys, to develop ISO Draft Proposal 8124.1, November, 1983 and its supporting analytical methods. DP 6714, "Preparation of Acid Extracts from Dried Paint Films," details the dry paint sample procurement, limited comminution thereof, and digestion in 0.07 mol/L of hydrochloric acid to simulate stomach digestion, followed by atomic absorption determination in the extract of lead, antimony, arsenic, barium, cadmium, chromium, mercury, and selenium, all in the 0.01 to 0.06 % range.

ASTM as a whole is reassessing its roles and relations with ISO (Geneva), BSI (United Kingdom), DIN (Berlin), and NNI (Netherlands), as well as its funding problems with ANSI (New York). This may chart new international directions for D-1 in 1985.

SUBCOMMITTEE D01.15 LECTURES & SYMPOSIA

H. A. Wray, Chairman

The subcommittee decided to recommend to the D01.90 Executive Subcommittee that a one day seminar be held in January 1985 in Orlando with representatives from four government regulatory agencies: DOT, OSHA, EPA, and CSPC. They would present talks and answer questions. The help of Sid Andrews, former chairman of the board of ASTM and a former member of the government of the State of Florida, will be solicited to obtain the cooperation of the Orlando and Florida State Chambers of Commerce, the regulatory agencies of the State of Florida, NPCA, through its Florida section, and the Southern Society of the Federation, and possibly, representatives of CMA. These groups will be invited to attend the seminar and thus provide a larger forum for the presentation.

SUBCOMMITTEE D01.18 EDITORIAL

H. E. Ashton, Chairman

D01.18.02, Indexing, S. LeSota, Chairman, discussed the scope of the group and its approach to indexing. However, it was realized that it was premature to establish the rules of the game. It was obvious that this is too big a task for just four members. More members are solicited.

DIVISION 20 RESEARCH AND GENERAL METHODS

SUBCOMMITTEE D01.20 SAMPLING, STATISTICS, ETC.

H. E. Ashton, Chairman

The chairman reported that D 3980 was printed without the errors having been corrected. A marked copy will be Wacker HDK® Fumed Silica

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In connection with setting up a computer program based on D 3980, C. J. Sherman said he had compared E 180 and D 3980 and believes there is no reason why such a program cannot be prepared. He asked for guidance on which computer the program should be designed for. It was the consensus that, if possible, it should be for the IBM. The question was raised as to how a user could acquire the program. The answer was that disks would be available through ASTM at a minimum cost to cover handling.

The chairman reported that he had been unable to review any of the sampling procedures. During discussion it was suggested that if the sampling practice, D 3925, was revised to include raw materials, it should recommend keeping samples for some time in case difficulties should arise with the manufactured coating. The chairman said that he would endeavor to carry out the review before the next meeting.

The chairman stated that due to pressure of work he would have to resign, but would like to be custodian of D 3980 until it is finally completed. Mr. Sherman said he would be prepared to consider becoming chairman after he finishes the computer program, so the present chairman agreed to remain until then.

SUBCOMMITTEE D01.21 CHEMICAL ANALYSIS OF PAINTS AND PAINT MATERIALS

R. W. Scott, Chairman

D01.21.13, Coordination of VOC Standards and Information, J. C. Weaver, Chairman, opened the meeting by quickly summarizing the purpose of this task group and emphasized the "information" aspect of its activity.

What is the status of the test data and information requested back in June 1982, by Dennis Crumpler of the US-EPA? All test data has been collected and the report is on Jim Barry's desk for review. Nothing more is known about this report or how it will be expedited. There is interest in the data gathered by the EPA and how it correlates with and affects Method 24.

There is an effort to try to simplify VOC compliance enforcement by having EPA bless voluntarily issued paint suppliers VOC data sheet which would be issued to the end user and which would convey the necessary information, such as density, solvent content, etc., needed to calculate VOC. Indications are EPA will allow calculated VOC, from the formula of the product, or the use of Method 24 to report VOC content (purely voluntary) with a new EPA form to be used by the end user to calculate addition of dilution solvent, and to report VOC compliance. If this concept is approved, we are told, it will go out as a letter from EPA's Joe Cannon to regional administrators and SIP bodies as acceptable to be used unless the reported VOC values are questioned and the Referee Method 24 needed.

The question of how to get the EPA personnel to participate in the ASTM and especially Sub. D01.21's activities was discussed at length. Does it involve the lack of travel funds or a lack of activities related to their area of interest? The possibility of holding a symposium related to VOC measurements, test methods and closely related subjects was suggested.

The concise and straightforward D 3960 "Practice for Determining VOC of Paints and Related Coatings," is under revision in TG 56. In addition to the incorporation of the methods for determining exempted halohydrocarbon (TG 55) and the revised D 4017 for water (TG 54), should VOC calculations to meet government regulations be incorporated as an addendum. This will be addressed at the next meeting.

Currently, EPA is not considering revising Method 24, but the consensus is the method should be reviewed. Approaches on how to handle this matter were discussed, including a proposed meeting with the US-EPA prior to the next January 1985 D-1 meeting.

California regulatory agencies and California ARB are focusing on the control of hazardous waste materials which includes solvents, oils and other volatile organic materials mixed with water and sludges generated by a number of different industries. If the hazardous material contains more than 1.0% VOC, it must be treated before disposal. The test methods to measure the VOC are: (1) vacuum distillation; (2) gravimetric purge and trap (GP&T); (3) head space analysis; (4) direct injection into a gas chromatograph. The GP&T method is favored; claimed requires 40 minutes test time/sample.

In recent weeks ASTM headquarters has organized, under Chairman C. Mathews, and ASTM staffer E. Sullivan, an ASTM Environmental Coordinating Committee. Initially, the water, air, and earth committees are involved.

Chairman Weaver has recommended formation of Sub. D01.12 on Environmental Concern to the D-1 Executive Committee. This group will work outside of Sub. D01.21 on government environmental issues, and it will be a liaison and coordinating committee.

VOC regulations are made and in place. The new phase is in "enforcement." Whereas, Federal agencies have been concerned with the impact of their regulations on industry, the enforcers on the State level are only concerned with following the letter of the law. This has created a problem with such large paint users as General Motors, Ford, and Chrysler in St. Louis, California, and recently in Michigan.

Transfer efficiency of paint as a laboratory test method has been developed by the EPA and its Centec contractor. However, the EPA has not taken any formal action on this laboratory method which was developed by paint users, equipment manufacturers and paint producers. Presently, the EPA is looking at the maintenance of equipment as it affects transfer efficiency.

It was reported the California's South Coast Air Quality Management District is asking the wood furniture industry to raise their conventional spray gun transfer efficiency from 30% to 54% by the best technology available as stated in Rule 1136. This regulation is based on no known new technology.

D01.21.22, Analysis of Electrocoat Bath Samples, W. B. Van Der Linde, Chairman, reviewed the results of the ballot D01.01 (84-1) items 1, 2, and 3, which covered three "Electrocoat Bath Sample Test Methods" (pH, conductivity, and solids/pigment content).

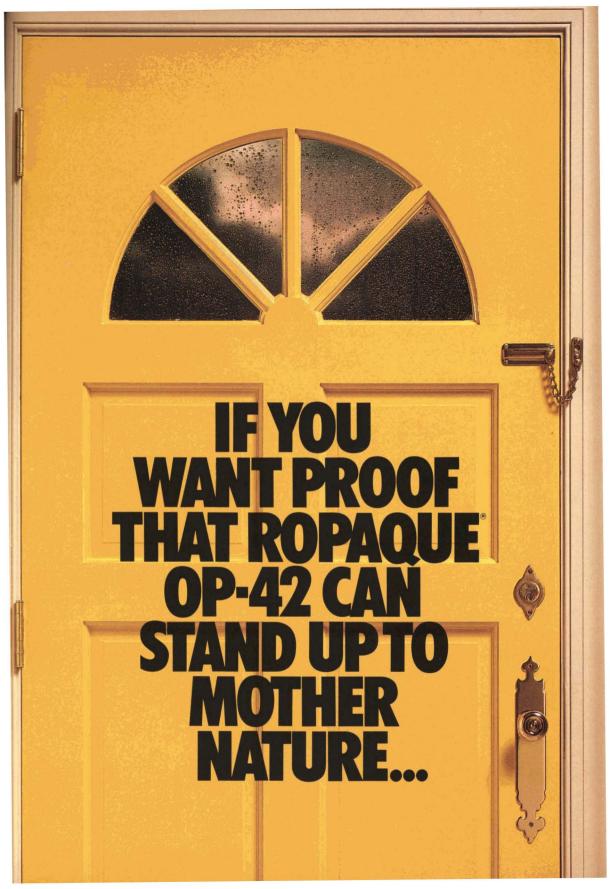
No negative votes were received prior to this meeting. Editorial comments were received from L. Schaeffer, A. H. Landrock, W. V. Moseley, E. R. Lewis, and J. R. S. Reid. Most of these comments were incorporated into the test methods. The comments by Reid and Lewis to change weight to mass in Item 3 were found to be non-persuasive; E380 allows the use of weight instead of "mass."

Just prior to this meeting, negative votes and editorial comments on all three items were received from H. E. Ashton. After a private discussion with him on this matter, appropriate changes were made which satisfied Mr. Ashton.

The omission of the inter-laboratory coefficient of variation, Item 3, 10.1 will be corrected after the meeting. After all necessary changes are made, the three test methods will be ready for Society balloting.

Although five active members are willing to participate in the proposed round robin to evaluate the new "Solvent weight determination" and "Cationic contamination determination" test methods, we have had no success in securing a supplier for the required electrocoat bath samples. There is great reluctance on the part of paint suppliers to provide commercial samples, particularly with the solvent content spelled out.

The chairman will try to circumvent this problem by writing to members of



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D01.21.23, Analysis of Thermoplastic Traffic Markings, J. M. O'Brien, Sr., Chairman. Round robins will be instituted immediately with the supplying of thermoplastic formulations, methods and evaluation forms necessary for the proper reporting of data.

Four laboratory participants have agreed to participate in the next round robin. All participants will be asked to report their results prior to the next meeting. The previous work done by R. Price on the AAS methods for the analysis of lead chromate will be reviewed by this reactivated group.

D01.21.27, Test for Pigment Content of Paints by Low Temperature Furnace Ashing, W. V. Moseley, Jr., Chairman. This method has been revised to reflect technical and editorial comments from the Sub. 21 letter ballot and resubmitted to ASTM staff manager Brent Backus for D01 letter ballot.

D01.21.42, AAS Analysis of Titanium Dioxide Content in Pigments Recovered from Whole Paints, W. V. Moseley, Jr., Chairman. This method has been revised since the last D-1 letter ballot to reflect numerous changes suggested by Messrs. Ashton, Dewilde, Wilson, Wray, and Landrock and sent to ASTM staff manager Backus for Society letter ballot.

D01.21.46, X-ray Analysis of Pigments, A. Monroe Snider, Jr., Chairman. Discussions focused on two methods that had been subjected to round robin investigation in 1982, but which had not been acted upon in 1983. One method is entitled "Standard Method for Titanium Dioxide in Paint by X-ray Fluorescence." The other method is entitled "Standard Method for Pigment Identification and Quantitative Determination in Paint Films by X-ray Diffraction."

Results from three laboratories that participated in the round-robin XRF analysis of Ti 0_2 are available. A fourth laboratory may also have received samples for round-robin analysis. M. Snider will determine whether the fourth set of data is available. Statistical analysis of the round-robin results will be performed by J. H. Smith using an E180 program. The round-robin results and statistics will be sent to committee members for review. If the results are satisfactory, the method will be presented for Sub. D01.21.46 approval. Otherwise, additional refinements and another roundrobin will be necessary.

Results of four laboratories that participated in the round-robin of the general XRD method show an undesirable spread. Comments on the method by round-robin participants and committee members will be reviewed by R. Scott and M. Snider to determine how the method can be further refined. The inclusion of greater procedural detail and the use of an internal standard will be considered.

Members who expressed an interest in participating in future round-robins were W. Moseley (Virginia Highway Dept.), R. Scott (Sherwin-Williams), M. Snider (PPG), and H. Swafford (Glidden). The chairman solicited suggestions on other laboratories that may be willing to participate in round-robins.

D01.21.47, Analysis of Calcium Borosilicate, J. Austin, Chairman. The present agenda included discussions on the completed editorial revisions and precision statement incorporated in Draft #3 of the test method. The group reviewed the round-robin results which included both the high and low B_2O_3 versions of the pigment. These results were used as the basis for the method's precision statement.

The TG felt the precision statement developed from the statistical analysis of the round-robin results was more than adequate for the proposed test method. The group unanimously recommended the test method (Revised Draft #3) be submitted for a simultaneous letter ballot of both D-1 and Sub. D01.21. The results of this letter ballot will be reviewed at the next scheduled TG meeting (January 1985), with the goal of incorporating the method into the 1986 ASTM Book of Standards.

D01.21.48, Analysis of Zinc Hydroxy Phosphate, L. DiCarlo, Chairman. Thirty affirmatives, no negatives, and twentytwo absentions were received on the April 1984 twenty-one letter ballot for the proposed analysis of Zinc Hydroxy Phosphate. A second draft incorporating comments and a "Significance and Use" statement will be presented for a D-1 letter ballot.

D01.21.53, Trace Levels of Monomers in Paints, T. Benga, Chairman, discussed the results of the latest round-robin. Samples were sent out to five laboratories and results received from three. The monomers determined in four latex samples were: methyl methacrylate, n-butyl acrylate, n-butyl methacrylate, and styrene. The results indicate that the method is applicable to in-house quality control, but the results generated are highly time dependent; free monomers react and polymerize. A revision of the method will be made to incorporate, in the scope, this time dependency. The precision statement will also contain an explanation for the poor reproducibility, which is due to a variation in the samples used for the roundrobin and not a fault of the method.

D01.21.54, Revision of D 4017 Water in Paint by the Karl Fischer Method, K. Leavell, Chairman. The task or goal of this group is the improvement of D 4017, "Karl Fischer Water Determination by Use of Hydranal Reagent."

The results of the last round-robin experiments were discussed. It was discovered that the initial condition using 30 mL methanol/10mL of Hydranal buffer gave poor solvency for two of these samples. The best results were from using the 1/1 pyridine/buffer. The data showed: (1) 1/1 pyridine/buffer gave good results; (2) 30/10 methanol/buffer increased deviation; (3) One of the three samples (electrocoat) kicked out in shipping and data on it might not be valid; and (4) The hydranal reagent gave slightly better data than the Karl Fischer method from a previous round-robin which used different type samples.

TG54 proposed to look at three samples again, i.e., a trade sales paint, a can coating, and an automotive waterreduced topcoat to be provided by H. Swafford and H. Fujimoto. The method to be used (solvent/buffer) will be defined by the chairman.

As in previous VOC calculations involving high water content and low VOC numbers, even small changes in water content led to large errors. For example: $46.93 \text{ H}_2\text{O} = 47.66 \text{ g/L}$ vol, $46.44 \text{ H}_2\text{O} = 63.26 \text{ g/L}$ vol.

D01.21.55, Halohydrocarbons in Coatings, C. Niemi, Chairperson. After a review of the statistics done by G. Cunningham on the last round-robin results and completion of the research report, the proposed method for the "Analysis of Dichloromethane and 1,1,1-Trichloroethane in Paints & Coatings by Direct Injection into a Gas Chromatograph" has been submitted to ASTM headquarters for balloting. It was requested that balloting be expedited at least to the point that an ASTM number be assigned to the draft. This is because the procedure is already being used in industry but cannot easily be referenced. (US Army Mil-C-46168c partially incorporated the ASTM draft because it could not be referenced).

R. W. Scott and J. C. Weaver have indicated they will endorse concurrent balloting of Sub. D01.21 and D-1. If the July balloting is completed without receiving any negatives, the proposed method could go through the December Society balloting with a February printing in the Vol. 6.01 ASTM Book of Standards.

D01.21.56, Revision of D 3960 Standard Practice for Determining VOC of Paints and Related Coatings, M. E. Sites, Chairperson, decided to conduct roundrobin #3 in conjunction with TG54 Revision of D 4017. An initial roundrobin conducted by this task group appeared to show some improvement in precision obtained when doing water determination using Hydranal water reagent. Three water-reducible coatings will be sent to collaborators for determination of VOC and % H2O. These samples will include a can coating, a household paint, and a water-reducible automotive topcoat. A precision statement for VOC's on these water-reducible coatings will be presented at the January 1985 meeting. It is hoped we may be able to improve our precision using this method for water determination.

A revised version of D 3960 including a precision statement for solvent reducible coatings generated by round-robin #1 will be submitted for balloting by Sub. D01.21 and D-1 by July 15th. The method being developed by TG55, "Test Method for Determination of Halohydrocarbons in Coatings," will be included in D 3960 after it has been balloted and received a "B" method number.

A report on progress of TG25 Revision of Volume Non-Volatile was given. This method is included in Standard Practice for Determination of VOC. The method will be submitted for simultaneous D-1 and Sub. D01.21 letter ballots by July 15. A precision statement generated by H. Ashton after a statistical analysis of round-robin data will be included. A note will be included on use of Quantachrome's gas pycnometer to determine the density of the dried paint film. Initial work done by H. Fujimoto on this "quick and clean" method showed promise. Work will be done by this task group to develop a method and initiate a round-robin using this procedure. This will be noted in D 2697, "Volume Non-Volatiles in Coatings".

D01.21.71, Trace Metals in Coatings, H. D. Swafford, Chairman, was reactivated at the request of J. C. Weaver to consider the desirability of drafting a test method which would essentially copy and translate into ASTM editorial format the ISO/TC35/SC9/WG4 document "Paints and Varnishes—Preparation of Acid Extracts from Dried Paint Films." This document is an international study which specifies methods for the preparation of acid extracts for the determination of the "soluble" metal contents of dried paint films.

After more than a two-year effort (January 1974-June 1976) experimental evidence accumulated by TG 21.03A concluded that neither a simulated digestive extraction technique nor a simple dilute hydrochloric acid extraction procedure are generally satisfactory for determining the extractability of heavy metals from cured paint films.

In view of the above, it was the consensus of the task group members that it would be inappropriate to recommend or support the inclusion of the ISO document in the ASTM Book of Standards.

Task Group 71 will now return to an inactive status.

D01.21.80, Exploratory Analytical Research, K. H. Leavell, Chairman. Quantitative FTIR was suggested as a potential area for committee action. R. W. Scott agreed to write up a general method for FTIR usage which could be applied towards a current ASTM standard, such as hydroxyl number.

H. Fujimoto reported on the use of a Quantachrome's Gas Pycnometer for measurement of VNV in coatings. The Pycnometer compares favorably with the disk method and is very rapid (1-2 minutes). None of the current committee members has an instrument. More evaluation of this technique will be done before our next meeting.

Evaluation of Hydranal Titrant for Karl Fischer Water Determination was discussed briefly. More information will be provided in TG54's minutes. Current SEC methods for coating vehicles are considered out-of-date. There is a need to update our previous methods or generate a method using the latest and best technology.

SUBCOMMITTEE D01.22 HEALTH AND SAFETY

H. A. Wray, Chairman

The chairman reported that, at the request of H. E. Ashton, the Coordinating Committee for Flash Point and Related Properties recommended changes in the titles of all the ASTM test methods to coordinate the titles for easy reference. Sub. D01.22 approved the revision of five standards as follows:

D 3278, Flash Point by Setaflash Closed-Cup Apparatus;

D 3934, Determination of Whether or Not a Liquid will Flash at a Given Test Temperature Under Equilibrium Conditions by a Closed Cup Apparatus;

D 3941, Flash Point by Equilibrium Methods with Closed-Cup Apparatus;

D 1310, Flash Point and Fire Point by Tag Open-Cup Apparatus; D 4206, Sustained Burning of Liquid Mixtures by the Setaflash Open-Cup Apparatus;

D01.22.01, Precautionary Statements, J. J. Brezinski, Chairman, decided to develop a proposed policy on precautionary statements for D-1 and to circulate it as a letter ballot to Sub. D01.22, D01.35 and the D01.90 Executive Subcommittee.

A letter will be sent to the ASTM Committee on Standards outlining the group's activity relating to use of precautionary statements and its response to Committee D-2's recent letter.

D 1310, "Flash and Fire Point by Tag Open-Cup Apparatus," received extensive comments as the result of the recent letter ballot. These comments were addressed and, for the most part, accepted. One of the most important improvements made in the method was the revision of some of the specifications for calibrating liquids along with a correction in the source of these liquids.

It was reported that OSHA has referenced D 3278 in their proposed regulations and that CPSC has proposed a change in the flash point methods in their regulations from an open-cup method to a closed-cup method. A number of petitions have been sent to U.S. Regulatory Agencies to utilize D 4206 in their regulations to control mixtures which will not sustain burning, but will have a flash point in the flammable range.

A study will be made to determine what, if any, work should be undertaken by Sub. D01.22 on the subject of labeling, as the result of OSHA's Hazard Communication Rule for the Workplace and in labeling of packages for both industrial and consumer use.

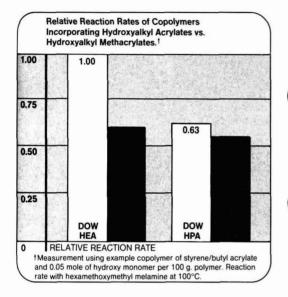
SUBCOMMITTEE D01.23 PHYSICAL PROPERTIES OF APPLIED PAINT FILMS

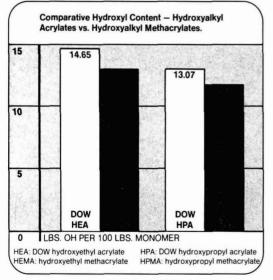
M. P. Morse, Chairman

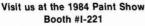
D01.23.10, Adhesion, H. E. Ashton, Chairman, is developing procedures for adapting the Adhesion By Tape Test method to accommodate thick films. Work is continuing on determining the precision of the new adhesion pull-off test. A round-robin test has been initiated.

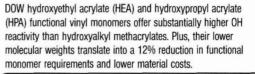
D01.23.12, Dry Film Thickness, K. A. Trimber, Chairman, has made an extensive revision of D 1005, "Measurement of Dry Film Thickness by Micrometer," to cover the use of hand and mounted micrometers in measuring the thickness of free films and films on substrates. This method has been submitted for Society ballot.

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D01.23.11, Wet Film Thickness, H. A. Ball, Chairman, has developed a practice for performing wet film thickness measurements with notched gauges. D1212, covering the use of the Interchemical and Pfund gauges, has been revised to conform to the latest required format. Both drafts will be submitted to simultaneous Sub. D01.23 and D-1 letter ballots.

D01.23.14, Hardness and Abrasion Resistance, has drafted a method for hardness by pendulum damping test. A round-robin test to establish the precision of both Koenig and Parsoz pendulum testers is underway.

D01.23.15, Static Friction of Coating Surfaces, has drafted a method that provides for the determination of static friction by two procedures: (1) inclined plane, and (2) horizontal pull. Extensive revisions have been made in the method draft to accommodate the suggested changes received as a result of the recent D-1 letter ballot. This latest draft will be resubmitted to D-1 ballot.

D01.23.16, Water Vapor Permeability of Paint Films, has drafted an extensive revision of D1653 to provide for the use of alternate permeability cups and for more than one set of temperature and humidity conditions. A draft will be submitted to Sub. D01.23 letter ballot.

SUBCOMMITTEE D01.24 PHYSICAL PROPERTIES OF LIQUID PAINTS & PAINT MATERIALS

C. K. Schoff, Chairman

D01.24.19, Viscosity by Efflux Cups, C. K. Schoff, Chairman, discussed D 4212, "Viscosity by Dip-Type Cups." The chairman had broken the method into drafts of two separate methods-Shell Cup and Zahn Cup. These were reviewed and discussed. The consensus was that D 4212 should be withdrawn and replaced by the two methods. The Shell Cup method would be written as a standard method, but the Zahn Cup method would be downgraded to a practice, if that action is possible. If this cannot be done then it would be made very clear in the scope, significance and use, and precision statements, that precision is very poor, etc. It was suggested that a new round-robin be run with cooperators using their own cups (rather than a special matched set of cups) to show the poor precision. Rather than go to that effort, it was felt that the precision statement should explain that interlaboratory testing had been carried out under ideal conditions and, therefore, gave a rosy idea of precision.

D01.24.20, Rotational Viscometers, was chaired by C. K. Schoff in the absence of the chairman, D. Howard. New drafts were presented of the several precision statements for D 2196, "Viscosity by Brookfield Viscometers," These were mainly for information as not all the statements were completed. The final versions will be submitted at the January 1985 meeting along with a revision of D 2196, which will be up for reapproval or revision in 1985.

The main topic of discussion was the new proposed method for determination of viscosity at low shear rates by the relaxation technique. No one was willing to become chairman of the task group, so this method will move slowly. There was interest in the method, however, and in the very rough draft that was presented.

The draft includes two separate techniques for handling data, computer, and through the use of templates. There was a question as to whether the computer program would, or should, be included in the method or whether it should be on file at ASTM headquarters. It must be determined how this type situation is handled with other methods. A more complete draft will be presented at the next meeting.

D01.24.22, Density, discussed D 1475, "Density of Paint and Related Products," which is up for reapproval. The method needs a significance and use statement and there are serious doubts about the accuracy of the precision statement. Because many people consider the precision impossible to achieve and the original round-robin data are unavailable, the chairman was going to ask for a new round-robin. However, he discovered that Sub. D01.21.56 already had carried out such testing. Their data and statement were reviewed and discussed. The consensus was that both were quite acceptable, so their precision statement will be included in the revision of D 1475, which will be submitted for concurrent Sub. D01.24 and D-1 balloting.

D01.24.26, Electrical Properties of Liquid Coatings, R. Huddleston, Chairman, reviewed the first rough draft of a new method for Measurement of the Electrical Resitivity of Liquid Paint. The method contains both D.C. (Ransburg) and A.C. techniques. The chairman requested comments on the draft from members to help in the preparation of a second draft due January 1985.

D01.24.27, Flocculation, is a new and, at this point, tentative task group. There were several attendees, but only one was from a paint producer. The others were all from pigment suppliers. Most floccul tion problems occur during the man facture and application of the pair. Therefore, flocculation is more of a pai problem than a pigment one. Unless the is more input from paint producers, the will be little point in continuing.

An outline for a proposed practice fc testing for degree of flocculation wa discussed. There was a question whethe a method or practice was needed. would be difficult, if not impossible, t develop a method that gave numerici results and had a meaningful precisio statement. Therefore, some form of pratice would be the probable result if th group continues.

Because of the question of interest, c lack of it, in this area, the membership c D-I will be surveyed for the need for practice on degree of flocculation. If appears that such a practice is unneces sary, the project will be dropped. If ther is a reasonable indication of need, it wi continue. A meeting of the task group wi be held in Orlando in January 1985 to tes further interest in this area. It sometime takes several meetings to get thing started.

SUBCOMMITTEE D01.26 OPTICAL PROPERTIES

C. J. Sherman, Chairman

D01.26.02, Color Measurement, C. S McCamy, Chairman, is still revising D 1535, "Specification of Color by the Munsell System." A draft will be submitted for Sub. D01.26 ballot. D 2244 "Instrumental Evaluation of Color Difference of Opaque Materials," was revised and simplified and will also be submitted for Sub. D01.26 ballot. A draft of a proposed standard practice on Color Standards will be submitted to the subcommittee for comments before the next meeting.

D01.26.06, Hiding Power, L. Schaeffer, Chairman, reviewed an extensive revision of D 2805, "Hiding Power of Paints," and voted to submit it to Sub. D01.26 ballot.

D01.26.11, Gloss and Goniophotometry, R. S. Hunter, Chairman, has revised D 523, "Test for Specular Gloss," and found it ready for D-1 ballot. The draft for Visual Evaluation of Gloss will be submitted for Society ballot. A task group of four members was formed to work on a new Distinctness of Image test method.

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SUBCOMMITTEE D01.27 ACCELERATED TESTS FOR PROTECTIVE COATINGS

E. A. Praschan, Chairman

D01.27.04, Light and Water Exposure Apparatus, S. Totty, Chairman, agreed to find the negatives persuasive on the proposed Standard Practice for Conducting Tests on Coatings Using a Fluorescent UV-Condensation Light and Water Exposure Apparatus. A redraft will be reballotted by D-1.

D01.27.09, Corrosion, R. Williamson, Chairman, discussed upcoming work to verify the reproducibility of two different test cycles for producing "Scab" corrosion. After some preliminary testing to establish details of testing a round-robin will be conducted and should be completed in time for a presentation of results at the next meeting.

D01.27.10, Outdoor Accelerated Weathering, M. Morse, Chairman, is conducting an extensive evaluation of the new EMMA night-time-wetting (n.t.w.) cycle at DSET Laboratories in Arizona. A recently completed study involving 19 coil coatings shows that after 33 weeks of exposure on EMMA n.t.w. the gloss loss and color change is in very good agreement with values obtained from 36 months 5° S open rack exposure in Florida. In a previous study involving 14 automotive coatings, 15 weeks exposure on the EMMA n.t.w. cycle produced color change values in good agreement with the values obtained after 24 months exposure on 5° S open rack in Florida. A lower acceleration rate for gloss loss was produced by this EMMA n.t.w. cycle with these same automotive coatings.

A new interlaboratory test has been initiated to compare automotive coatings in the present EMMA n.t.w. cycle and in a modified cycle in which higher panel temperature will be utilized. The same coatings will be exposed at several sites in Florida (open rack, black box, and 5° plywood-backed racks).

D01.27.14, Filiform Corrosion, R. Williamson, Acting Chairman, is attempting to accelerate the formation of filiform corrosion over the rate produced by the current method (D 2803). Earlier work shows that this can probably be accomplished by using higher humidity cabinet temperatures. The exact temperature to be used and the acceleration still must be determined. It is planned to begin roundrobin testing prior to the January meeting.

D01.27.17, Evaluation of Weathering Effects, A. Allen, Chairman, continued a discussion started last meeting on a draft of a revision of D 660, "Evaluation of Checking." Based on the inputs from these discussions, the chairman will rewrite the draft for discussion at the next meeting.

SUBCOMMITTEE D01.28 BIODETERIORATION

D. L. Campbell, Chairman

D01.28.01, Package Stability, W. B. Woods, Chairman, made a request for participants in a round-robin to evaluate the Presumptive Challenge Test Method for determining the capacity of preservatives to prevent microbial spoilage of paint. There is a possibility that the Hercules biology lab will cooperate.

D01.28.02, Rapid Determination of Enzyme Presence, D. P. Leipold, Chairman, reported that prior to the meeting the degradation of various cellulosic thickener solutions was tested. Of those tested sodium carboxymethyl cellulose (CMC) 4H1 retained the least viscosity after 24 hours at the four enzyme levels tested. Since it was most sensitive to enzyme attack it was used for further testing. In further tests the CMC 4H1 solution was combined with paint at 50:50 weight % ratios. These solutions were inoculated with 0.01, 0.10, 0.50, and 1.00 ppm cellulose enzyme. All solutions showed viscosity loss after 24 hours. These results were distributed to the group.

A question was raised whether the enzyme levels tested were representative of contamination levels in paint. There was no satisfactory answer to this question.

Representatives from Nuodex and R.T. Vanderbilt agreed to act as cooperators to reproduce this data and to test the method with actual spoiled paint. SCM Pigments and Air Products may cooperate after reviewing the method. Hercules will supply the CMC 4H1 and cellulose enzyme to the cooperators prior to the January meeting.

D01.28.04, Resistance of Paint Films to Attack by Algae, W. B. Woods, Chairman, discussed the addition of a paragraph on terrestrial algae to Chapter 6.4, Biological Deterioration of Paints and Paint Films. A request was made for participants in round-robin testing of a tentative algae resistance procedure. D01.25.05, Recoating Mildewed Surfaces, D. L. Campbell, Chairman, discussed a New Standard Guide for Determining the Presence of and Removing Microbial (Fungal or Algal) Growth on Paint and Related Coatings. Comments and articles submitted by G. A. Stearns, Oscar Meyer Foods, were reviewed and discussed. Separating the sodium hypochlorite solution and the trisodium phosphite solution was recommended to avoid pH problems.

DIVISION 30 PAINT MATERIALS

SUBCOMMITTEE D01.31 PIGMENT SPECIFICATIONS

C. W. Fuller, Chairman

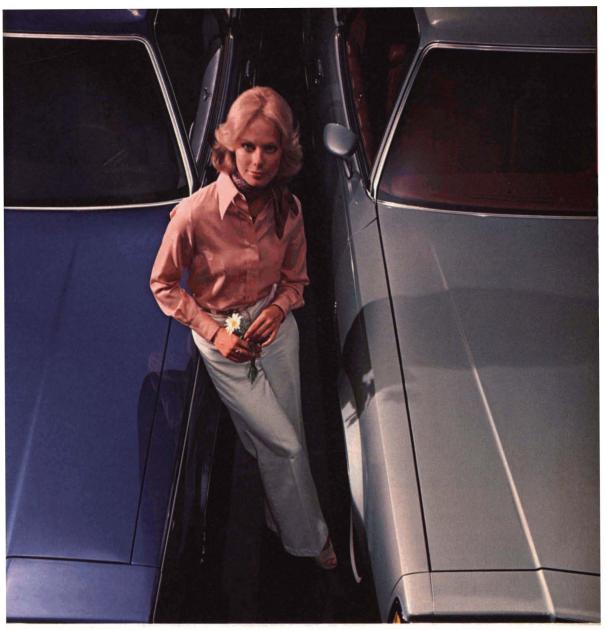
Comments and negatives on eight items from the latest ballot were discussed and action taken where necessary. However, in reference to comments and negatives received from H. E. Ashton, it was moved that "The closing date on the three methods D 3619, D 480, and D 603 being April 24, 1984, and his correspondence having been received on May 26, 1984, the committee did not have time to consider the comments prior to the meeting or to refer his comments to the task group for appropriate comments. D-1 was to be asked for an automatic extension of time on these three methods so that the comments and negatives can be reviewed properly."

Prior to the next meeting the following methods will be reviewed: D 79, Zinc Oxide, D 81, Basic Carbonate of Lead, D 212, Chrome Green, D 765, Raw and Burnt Sienna, D 3360, Particle Size Distribution and D 1199, Calcium Carbonate Pigments.

SUBCOMMITTEE D01.32 DRYING OILS

P. C. Stievater, Chairman

Sub. D01.32 has 30 methods due for reapproval in 1984. Fifteen were listed on the D 0103 main and subcommittee ballots (83-3) issued August 10, 1983 and 15 were listed on the D 0104 (83-4) main and subcommittee ballots issued October 19, 1983. No negatives were received on



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any items, but comments were received on many of them.

Since no Sub. D01.32 meeting was held in December 1983, the changes could not be discussed at the meeting. However all 30 methods were placed on the Society ballots, the first 15 on the January 1984 Society ballot and the other 15 on the April 1984 Society ballot. Replacing on these Society ballots was done prior to the editorially changed methods being submitted by Sub. D01.32.

The principal reason for holding up the methods was that many lacked sections on Significance and Use and on Precision and Accuracy (Bias) and the committee does not have the necessary background material in its possession to write some or most of these sections. In addition, adding these sections would be considered technical changes and would require reballoting at main and/or subcommittee levels.

An attempt will be made to pull the Significance and Use section from the present Scope section, in which case the changes would only be considered editorial. A Precision and Bias section will be added, but in most cases it will have to state that the information is not available or is under development.

A request is being made, via this report, for present or former committee members to supply information on any of the methods under Sub. D01.32 jurisdiction which will help in the development of these sections.

SUBCOMMITTEE D01.33 VARNISH & RESINS, INCLUDING SHELLAC

A. C. Abbott, Chairman

Accepting a negative ballot by H. E. Ashton, on D 1395, "Test for Abrasion Resistance of Clear Floor Coatings," the subcommittee approved submission for concurrent ballot of Sub. D01.33 and D-I for withdrawal. The procedures in D 1395 are essentially covered by the methods referenced in D 1395.

Comments on the D-1 ballot of the revision of D 3132, "Test Method for Solubility Parameter Range of Resins and Polymers," were incorporated. D 3132 as revised will be advanced to Society ballot.

Comments on the D-1 ballot of the revision of D 3733, "Test Method for Silicone Content of Silicone Polymers and Silane-Modified Alkyds by Atomic Absorption Spectroscopy," were incorporated. D 3733, as revised, will be advanced to Society ballot. A summary of method was written for inclusion in the revision of D 1398, "Test Method for Fatty Acid Content of Alkyd Resins and Alkyd Resin Solutions," and the updated revision will be submitted for concurrent ballot of Sub. D01.33 and D-1.

Task group chairmen are needed for the urethane, alkyd, and emulsion groups.

D01.33.01, Varnish, H. A. Ball, Chairman reported D 1643, "Test Method for Gas Checking," had been approved for publication. Comments by A. H. Landrock, L. R. Thurman, H. M. Werner, J. C. Weaver, and H. Fujimoto, and a negative by E. R. Lewis were approved for incorporation in a fourth draft of the major revision of D 154, "Guide to Testing Varnishes." This will be submitted for a concurrent ballot of Sub. D01.33 and D-1.

D01.33.23, Epoxy and Phenolics Resins, H. D. Marshall, Chairman, reported he is working with D-11 (Rubber) on new proposed specifications for thermoplastic and thermosetting phenolic resins used in rubber.

Many of the test procedures required for this specification are already ASTM methods. The following proposed new test procedures are being reviewed editorially by H. E. Ashton: (1) Stroke cure on Phenolic Resins; (2) Percent Methylol Content in Heat Reactive Phenolics; (3) Capillary Method for Shrink and Melting Point; (4) Test Method for Determining Presence of o-Methylol; and (5) Method for Determining pH on Solid Phenolics.

The chairman is preparing nine other new methods for consideration at the January Meeting.

D01.33.24, Nitrogen Resins, J. Smith, Chairperson, reported both the procedure and the equipment to be used need refinement and testing before a roundrobin can be conducted on a proposed method for determining free formaldehyde in amino resins.

D01.33.25, PVC and PVB Resins, J. Brezinski, Chairman, reported the "Standard Guide for Testing Poly (Vinyl Chloride) Resins" passed Society ballot.

One of the methods cited in the guide method, D 1303, "Standard Test Method for Total Chlorine in Vinyl Chloride Resins," is being balloted (through Committee D-2) at the Society level for withdrawal. The D-1 Guide already has incorporated a suitable alternate method, E 442, should D 1303 become obsolete.

Committee D-20 (Section 15.08 on Vinyl Chloride Monomer and Resins) is also conducting round-robin studies to improve the precision of several methods cited in the D-1 Guide, which includes Method D 2222, "Methanol Extract of Vinyl Chloride Resins." Methods D 1823 and D 1824 (Viscosity Methods for Vinyl Plastisols and Organosols, respectively) are being modified with new precision statements.

SUBCOMMITTEE D01.35 SOLVENTS, PLASTICIZERS AND CHEMICAL INTERMEDIATES

L. R. Thurman, Chairman

D01.35.10, Solvents—Hydrocarbons & Ketones, S. A. Yuhas, Jr., Chairman. W. J. Frost chaired the meeting in the absence of the chairman. Documentation of test data for precision statements have not been found for six of the 15 test methods. A request was made for members to search their files for these test data.

An item of new business was the discussion of D 1722, "Test for Water Miscibility of Water-Soluble Solvents." It is often used as a quick method of analysis for trace quantities of non-specific impurities. Shippers have expressed concern over nebulous results which allegedly cause excessive demurrage and other inconveniences. Users and producers believe this is a useful test method. Copies of any test results or literature which may document the usefulness of this method are solicited for discussion at the next meeting.

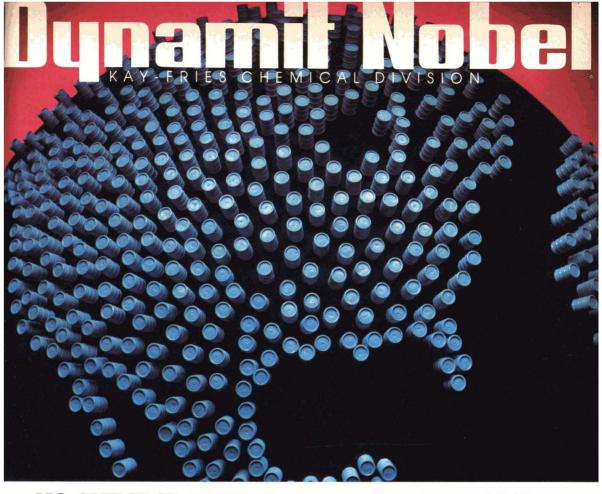
D01.35.20, Reactive Monomers-Vinyl Monomers and Aldehydes, T. H. Golson, Chairman, heard a request to investigate two new items: (1) A method for measuring polymers in monomers, which could help to decide shelf-life of monomers, and (2) A practice to remove inhibitors from monomers. Inhibitors can interfere with kinetic studies. Literature and suggested methods are requested for review at the next meeting.

D01.35.30, Chemical Intermediates, J. R. Morrison, Chairman discussed the proposed specification for 2-ethyl-hexanol. Gas chromatographic assay methods were reviewed. Information and literature will be searched for a more appropriate column for the assay method.

D 4052 is being evaluated as an appropriate method for determining apparent specific gravity of glycerine.

D01.35.40, Plasticizers and Ester Solvents, reviewed the combining of six ethyl acetate and butyl acetate specifications. These will be submitted to Sub. D01.35 ballot.

Under new business, the terms '% acid as acetic' and 'acid number' were discussed.



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'Acid number' has gained some acceptance, however, the consensus of the group was that '% acid as acetic' should be continued in use.

Specifications for propylene derived glycol ethers will be developed for review at the next meeting.

D01.35 New Business—A fifth task group for Sub. D01.35 was proposed with a chairman to be appointed later. The new task group will be Sub. D01.35.50, entitled 'Liaison'. Its responsibility will be to interface with all other ASTM committees and various other standards organizations and to inform Sub. D01.35 members of actions affecting Sub. D01.35 affairs.

Comments attached to recent ballots have indicated preference for English units over SI units. Discussion led to a motion that a recommendation be made to the D01.90 Executive Subcommittee that, pursuant to paragraphs H-3 and H-4 of the "Blue Book," preference be given to acceptable metric units in D-I Standards.

Based on comments received during the recent ballot of D 1258, the new paragraph 6.4, "Sulfate Ash Procedure," will be reviewed by the task group Sub. D01.35.30 to consider recommended detailed procedures to run sulfate ash.

DIVISION 40 PAINT PRODUCTS APPLIED ON SITE

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

J. C. Weaver, Chairman pro tem

Resignations of Sub. D01.41 Chairman, R. A. Brown, and Vice-Chairman, W. V. Moseley, Jr., in January leaves Sub. D01.41 in need of reorganization and of reassessment of its goals.

An April 16 poll and questionnaire to the nearly 50 members brought 24 responses of which 19 favored and three disfavored continuance of Sub. D01.41 within its current scope.

A proposed Standard Specification for Flat Interior Latex Paint, Draft no. 1, March 21, 1984, by Irving Bloom of the U. S. Naval Facilities Engineering Command, was issued on April 13 to Sub. D01.41 members. Mr. Bloom reported 26 responses comprising about one third each of affirmatives, negatives, and abstentions. He was authorized to use all the comments with these responses to prepare a revised draft thereon.

The general mission of Sub. D01.41 drew diverse viewpoints from the 28 attendees. A reading of the Sub. D01.41 scope from its 1978 inception heightened the disagreements. Ad hoc task forces will be appointed toward the tandem targets of the October meeting of D01.90 Executive Subcommittee and the January 20-24, 1985 general meeting of D-1 to attempt to resolve these differences.

Some of the controversy is over paint producer preference for their branded paints over allegedly minimal performance being deceptively assured by paint specifications which are mainly on composition rather than on performance. Paint specifications which are limited to performance are recognized as more difficult, especially with several degrees of freedom of choice. ASTM C920 Standard Specification for Elastomeric Joint Sealants was suggested as a strictly performance model. It omits any reference to composition and is mainly a classification of two types, two grades, two classes, and six uses, i.e., allowing possibly 48 choices. A critic asked if this specification is specific or specious.

SUBCOMMITTEE D01.42 ARCHITECTURAL PAINTS

R. H. Rowland, Chairman

D01.42.02, Practical Soil and Stain Removal, C. Tatman, Chairman, reviewed the purpose of the task group and its future direction as well as Draft #2 of the Practical Soil and Stain Removal method. The new draft incorporated the suggestions received in the last ballot. It will be resubmitted to Sub. D01.42 ballot.

D01.42.04, Wet Adhesion, F. Winkelman, Chairman, discussed the first draft of a potential round-robin. The chairman agreed to evaluate the following three parameters prior to sending out materials for the round-robin: (1) Negative control paints which will demonstrate poor wet adhesion; (2) The effect of utilizing a draw-down application in place of the brush application called for in the first draft; and (3) Use of one or two different substrates, most notably an alkyd coating applied to wood, in place of the 7-mil polyester panel described in the draft.

Pending the outcome of this work the draft will be revised and sent out for round-robin testing.

D01.42.06, Standard Practice for Testing Latex Semi-Gloss and Gloss Paints, S. LeSota, Chairman, reviewed the draft of this recommended practice. At the suggestion of H. E. Ashton, several changes will be made in the document prior to submittal for Sub. D01.42 letter ballot.

D01.42.07, Sag Resistance, L. Schaeffer, Chairman, reviewed the results of the recent ballot on the new proposed sag resistance test method. One comment received from H. E. Ashton concerning variations given in the precision statement will be reviewed and suitably resolved. Several other comments received will also be incorporated prior to its submittal for Society ballot.

D01.42.09, Color Acceptance (new title), R. Rowland, Chairman, discussed the results of the recent round-robin. The data were developed using an acrylic latex paint employing either red iron oxide, phthalo green, or violet tinting colors. In general the results from five cooperators were in excellent agreement as far as the 'delta' E (Hunter Units) determinations. The results were below 1 'delta' E unit and as such, it was decided to test a "problem" paint with a 'delta' E in the 3-5 range so that the method can be evaluated for such a colorant acceptance situation. A round-robin will be undertaken before the next meeting.

D01.42.13, Brushability, D. Leipold, Chairperson, presented the results of the November 1983 round-robin. It was concluded that interlab variation existed in both the ICI viscosity and "ease of brushability" tests. Considerable discussion concerning brushability and what quality it actually refers to took place. It was decided that for the purposes of this task group brushability refers to brush drag. A new round-robin will be conducted prior to the next meeting to correlate brush drag with high shear (10,000 sec⁻¹) viscosity. This round-robin will utilize laboratory sample paints with identical Stormer viscosities, but differing high shear viscosities. The high shear viscosities will vary by approximately 0.5 poise, paint to paint. A subjective evaluation of brush drag will also be requested from the cooperators.

D01.42.16, Opacity (Practical Method), R. Armstrong, Chairman, discussed results of the round-robin held prior to the meeting. This consisted of application of the three test paints to the special three gray-striped paper panels. The paints were applied to the entire panel by roller (3-inch wide) at a spreading rate of 400 sq ft/gal. After the first coat was dry, half of the panel was recoated with the same paint. The cooperators were asked to take instrumental reflectance readings of the paints over the various gray-striped areas

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where one coat had been applied and were asked to take similar readings of the two coats of paint applied over the lightest gray stripe (complete hiding). Cooperators ranked the hiding of the paints over the three gray stripes in the same order. There was not very good reproducibility between cooperators, probably due to the use of various instruments in the reflectance measurements. The members suggested that the method be written up and submitted for Sub. D01.42 ballot since it would be useful for ranking the hiding power of different paints or comparing the hiding of a test paint to that of a control paint.

D01.42.17. Roller Spatter. J. Price. Chairman, reviewed the results of the fourth round-robin which showed excellent agreement among the cooperators concerning the ranking of the five test paints. Interlab correlation concerning the amount of spatter generated by each test paint was poor. Possible causes of these interlab differences were discussed. It was agreed that the group chairman would rewrite the method to clear up some of the procedural misunderstandings encountered during this round-robin. It was also agreed to allot two hours to this task group at the next meeting at which time a working session would be set up wherein those attending the meeting will have the opportunity to conduct the Roller Spatter test. A discussion will follow this "laboratory" session which should help further in promulgating an eventual method for ballot. It was agreed that pending the outcome of the next meeting "laboratory" work, a set of standards be created to grade Roller Spatter results.

D01.42.18, Block Resistance, U. Jackson, Chairman, held its first meeting. There was an open discussion on the objective of the group which led to the establishment of some basic test conditions. These conditions will be evaluated in a round-robin to be conducted prior to the next meeting. Five cooperators have agreed to participate.

SUBCOMMITTEE D01.44 TRAFFIC COATINGS

R. L. Davidson, Chairman

D01.44.01, Thermoplastics, J. O'Brien, Chairman. A review of the recently run bond strength test uncovered a number of problems that need to be resolved, at which point the test will be re-run. After a (1) The finished specification will need to address freeze-thaw criteria depending on the geographical location of the user. The country may need to be zoned in order to address this problem.

(2) The specification will need to address the substrate, i.e., Portland Cement Concrete or Bituminous Concrete and the condition of the substrate, i.e., new or worn. W.V. Moseley will attempt to prepare bituminous substrates for such testing and J. O'Brien, Portland Cement Concrete substrates.

(3) W.V. Moseley of the State of Virginia D.O.T. will conduct a study in an attempt to determine the effect of temperature on the proposed abrasion resistance test. The round-robin on abrasion resistance will not be conducted until his work is completed.

(4) It was noted that there must be temperature restraints on sample preparation for the reflectance study.

(5) It was suggested that the AASHTO test for yellowness index needs to be incorporated in this specification.

D01.44.02, Traffic Paint, J. Atkins, Chairman, reviewed all test methods Sub. D01.44 is responsible for and it was noted that only two are overdue. In both cases, submittals have been made for editorial review before subcommittee and committee balloting.

A preliminary report was made on the durability round-robin conducted at the Pennsylvania test site by nine cooperators. Results will be submitted to the proper group so that a precision statement can be generated.

A question was raised why, with the strict criteria set up by ASTM on the conducting of a round-robin, a simple cookbook set of directions could not be developed in order for each group to generate its own precision statements. All too many have conducted the tests, submitted the data, and waited an extremely long time for the results.

D01.44.03, Night Visibility, reported that the Casey letter to GSA concerning night visibility of airport markings was withdrawn from GSA and the issue is closed. Due to the lack of statistical data there was nothing that could be done on the test procedures D-1214 and D-1155 on the sieve analysis and rounds of glass beads. A suggested testing procedure using portable retroreflectometers to determine the reflectivity of horizontal pavement markings was submitted to the committee for comments.

SUBCOMMITTEE D01.45 MARINE COATINGS

L. S. Birnbaum, Chairman

D01.45.05, Algae Control, C. D. Stevens, Chairman, reviewed Draft #2 of the proposed Standard Test Method for Evaluation of Anti-Fouling Coating Algal Control Efficacy. Based on the discussion, C. D. Stevens will run some tests to determine effect of preconditioning of the test panels, and choice of test panel substrates. Revisions to be incorporated into the next draft will include additional clarification on spacing, frequency of inspection, cleaning, and modification of rating scale to conform with ASTM rating method in Section 9.4 (p. 85) of the Handbook. Battelle, Ameron, and Miami Marine agreed to participate in a round-robin. details of which the chairman would generate after reviewing Section 9 in the Handbook. The subcommittee expressed its appreciation to Chairman Stevens for his successful efforts in expediting the development of this method.

D01.45.06, Dynamic Testing, D. Laster, Chairman, reviewed a draft dated 2/17/84 of the Standard Method for Marine Anti-Fouling Coating Systems under Hydrodynamic Flow, prepared by the chairman. Revisions to be incorporated into the next draft will include editorial changes to Sections 4.4 and 5.1.1. Several members agreed to send in proposed changes to Section 5.1.2 concerning static exposure on use of a drum as an alternate to panels mounted in a rack for incorporation into the new draft to be prepared by the chairman.

After the above changes have been implemented, it was agreed to initiate a round-robin. The chairman will preparea proposed plan for this effort.

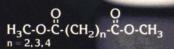
The incorporation of a non-consensus dynamic test method into F-25.02 proposed draft of an antifouling specification was brought to the attention of the attendees. Sub. D01.45 chairman, L. S. Birnbaum, was requested to prepare a letter to F-25.02 to withdraw this test from their specification until the completion of the effort of this task group. There was no objection to F-25.02 incorporating their method into an appendix or in a note as an interim non-standard test method.

D01.45.07, Anti-Fouling Rating, C. Perez, Chairman, voted to end the static and dynamic tests which have been underway for two years. Both Miami Marine and Battelle will report their results using their rating schemes. Simul-

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taneously, contacts will be made to obtain rating schemes used overseas. Five color slides will be selected from each test site showing a range in performance. The ten selected slides will be converted to 1:1 photographs to be prepared by C. D. Stevens. These photographs will then be circulated to at least 10 raters for rating by the two schemes. After detailed rating instructions have been generated by Perez and Herlihy, a protocol for circulation of photographs, and evaluation of results will be developed. It is proposed to determine feasibility of this technique to serve as a round-robin for evaluation of rating schemes.

D01.45 Business-Chairman Birnbaum reported on the status of progress of F-25.02 (Coatings) and F-25.02.07 (Task Group on conversion of Federal and MIL Specs. to ASTM methods). F-25.02, through ASTM staff members, agreed to provide Sub. D01.45 members with drafts of proposed standards and specifications for review and comment, and with copies of subcommittee minutes. F-25.02.07 is currently working on a draft of an ASTM specification for antifouling paint. Similarly, Sub. D01.45 will furnish F-25.02 members with copies of pertinent Sub. D01.45 documentation for their review and comments.

Chairman Birnbaum reported that although the Navy had extended the deadline for receipt of comments to permit transmittal of coordinated Sub. D01.45 member comments, he had received no comments and, therefore, did not forward an ASTM Sub. D01.45 response.

Additional biocide leaching methods were provided by C. D. Stevens and circulated to attendees for information. Drafts of proposed methods received after the December meeting were circulated to members with the December minutes. No further action was taken on this item. A task group chairman is still being sought for this effort.

SUBCOMMITTEE D01.46 INDUSTRIAL PROTECTIVE PAINTING

K. A. Trimber, Chairman

D01.46.02, Surface Preparation, K. A. Trimber, Chairman, reported that the last D-1 letter ballot on the new standard, "Standard Methods for Field Measurement of Surface Profile of Blast Cleaned Steel," resulted in two negatives and six comments. One negative was withdrawn at the meeting and the other found not persuasive. The comments involved minor editorial changes. The standard will be submitted for Society ballot. A few minor editorial changes have been made in D 2000, "Pictorial Surface Preparation Standards for Painting Steel Surfaces," which is due for reapproval in 1984. It will be submitted to concurrent Sub. D01.46 and D-1 ballot.

D01.46.03, Repainting, D. Noxon, Chairman. R. Martell chaired the meeting in the absence of the chairman. A subcommittee ballot of "New Test Method for Field Identification of Coatings" resulted in two negatives and comments. The negatives and comments were all found to be persuasive. The changes will be made and it will be resubmitted to Sub. D01.46 ballot.

D01.46.04, Pull-Off Adhesion Test, A. Cunningham, Chairman, discussed the results of the subcommittee ballot of "Proposed Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers." Five negatives and comments were received. Four negatives and the comments were found to be persuasive and were handled with editorial changes. One negative was found to be not persuasive. The revised standard will be submitted to concurrent Sub. D01.46 and D-1 ballot.

D01.46.07, Inspection, R. Martell, Chairman, has requested a one-year extension on the reapproval of D 3276, "Standard Guide for Painting Inspectors," which was due for reapproval in 1984. A new draft of the standard had been prepared and circulated to the task group prior to the meeting. The draft contains a comprehensive checklist of quality items that an inspector must examine when inspecting the preparation of concrete and metal substrates, and the application of coatings. The checklist was expanded during the meeting. A brief description of the various tests will be added to the next draft which will be circulated to the task group members in August.

New Task Group, Condition Assessment, A. Schwartz, Chairman, held a meeting to discuss the formation of the new group for the specifying of coatings for bridges and elevated structures. The attendees ultimately decided that such a group would be developing nothing different than what is currently being done by others (ASHTO, SSPC, etc.), and also questioned whether DOT's would even use the information. After lengthy discussion it was agreed that a need did exist for making a condition assessment of bridges. This would involve the development of a guide for surveying bridges to assess the degree of coating deterioration. A specific scope will be circulated to D-1 members in August.

New Business—Mary McKnight offered to arrange for one or two lectures to be presented at the next meeting to all interested parties. The presentations will involve current work being done to predict coating life. When the specific details are available, they will be circulated to D-1 members and published in *ASTM Standardization News*.

SUBCOMMITTEE D01.48 ZINC RICH COATINGS

D. C. Kinder, Chairman

The subcommittee discussed the results of the ballot on the proposed Standard Test Methods for Evaluation of Cathodic Protective Capability (Throwing Power) of Zinc Rich Coatings. Six negative ballots were received. A number of the negatives were concerned with the validity of the test method with respect to reproducibility and correlation with other test methods (specifically salt spray and test fence) and with real world performance. It was the consensus of members present that the ballot be withdrawn until precision data can be collected by round-robin testing. R. Wakefield, the task group chairman, will prepare the round-robin test procedure for distribution.

DIVISION 50 PAINTS FOR FACTORY APPLICATION

SUBCOMMITTEE D01.51 POWDER COATINGS

C. Grenko, Chairman

D01.51.01, Editorial and Definitions, T. Toplisek, Chairman. E. Oswald chaired the meeting in the absence of the chairman. A list of definitions was presented and will be distributed to Sub. D01.51 members for consideration and review for possible inclusion in paragraph 4 of D 3451.

D01.51.02, Physical Properties of Powder Coating Materials, E. Marx, Chairman. The meeting was chaired by K. Kipp in the absence of the chairman. Three practices have been prepared for subcommittee ballot. They are: (1) Color Measurement of Powder Coating Resins using a Tristimulus Colorimeter; (2) Color of Powder Coating Resin Solutions (Platinum Cobalt Scale); and (3) Determination of Melt Viscosity of Coating Resins by Plate and Cone Viscometer.

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D01.51.03, Applications and Film Formation, R. Huddleston, Chairman, was presented with a recommended practice for the determination of compatibility of powder coatings which will be submitted to Sub. D01.51 ballot. A transfer efficiency test was attempted, but not considered feasible at this time because of difficulty in determining, (1) a suitable target and, (2) flow rate measurements. Impact fusion was discussed and it was determined that no practical test has yet been devised. Search for a suitable test will continue.

D01.51.04, Properties of Powder Coating Films, C. Danick, Chairman, discussed a proposed method for determining hiding power of powder coatings, edge coverage, and cure end point and their possible incorporation in paragraphs 20-36 of D 3451.

D01.51.05, Safety and Hazards, K. Kipp, Chairman, had a discussion and reached a conclusion that this group should go on inactive status since the group has neither the expertise or the facilities to develop standards pertaining to safety. However, it was proposed to keep abreast of developments by other organizations such as the National Fire Protection Association, Bureau of Mines and the Powder Coatings Institute.

General-Sub. D01.51 will communicate with the Powder Coatings Institute, Society of Manufacturing Engineers and ASTM Committee D-9. D-9 has already approached Sub. D01.51 on assuming some of their coating methods.

SUBCOMMITTEE D01.52 FACTORY-COATED WOOD PRODUCTS

S. B. Schroeder, Chairman

Editorial revisions to D 3259, "Standard Recommended Practice for Infrared Determination of the Temperature of Applied Coatings on Wood Products During the Curing Cycle," which were suggested by A. H. Landrock, H. A. Wray, and H. E. Ashton, were discussed and approved. All of the other methods under the jurisdiction of Sub. D01.52 were discussed briefly with emphasis on the following: D 2793, "Standard Test Method for Block Resistance of Organic Coatings on Wood Substrates"-The chairman presented a tentative significance and use statement which was accepted for incorporation in this method. It will now be submitted to a concurrent Sub. D01.52 and D-1 ballot as a practice instead of a method; D 2921, "Standard Method for Qualitative Tests for the Presence of Water Repellents and Preservatives in Wood Products"-This method will fall under the jurisdiction of the new Task Group 14 on Coated Mill Work. A study of this method and other mill work related methods will commence at the next meeting, when the task group will be organized; D 3719, "Standard Test Method for Dirt Collection on Coated Exterior Panels"-It was determined that the chairman will produce a tentative significance and use statement and mail it along with the minutes of this meeting to the Sub. D01.52 members prior to the next meeting. This, and other revisions, will be acted on then.

D01.52 B, Hardboard, S. B. Schroeder, Chairman, continued the dialogue of previous meetings regarding development of new methods to measure wetting or swelling of hardboard due to paint surfactants. Additional data from determinations made with the AHA developed "protection and interaction" test was disclosed by several of those present. This data and possible correlating data from related tests (Cobb Ring) will be submitted to the chairman for summation or analysis prior to the next meeting. Concerns were expressed over the reproducibility of the test as presently configured and a possible need to specify standard drying conditions for the paint film. Other AHA tests published in voluntary standard, A-135.6, also were discussed briefly.

D01.52.13, Prefinished Hardboard, K. Kruse, Chairman, is a new task group formed in concept at the last meeting. The chairman presented a brief scope or objective for the group to review and to develop test methods to be used in specifying prefinished hardboard siding. A survey form was provided to those present. The results will be the basis for discussion at the next meeting.

D01.52.14, Coated Mill Work, Chairman is still to be determined. This group was approved in concept unanimously. R. Panchot volunteered to survey people in the Mill Work Industry for test method needs. This will be reported at the next meeting.

Other Groups—An effort has begun in Sub. D01.52 to broaden the base of activities. Various volunteers have promised either to provide names and phone numbers, or to contact directly, the key individuals in other wood building product industry segments. A progress report will be made at the next meeting.

Other Business—A discussion of the Sub. D01.52 scope resulted in a suggested change in the subcommittee title from the present one, which is believed to be unreasonably broad, to the new title of "Factory-Coated Wood Building Products." This change, which was approved unanimously, will be presented to the D01.90 Executive Subcommittee for consideration.

A number of comments indicated that the many years of service of R. C. Marck, the previous Sub. D01.52 chairman, will not soon be forgotten by his many friends and fellow ASTM members.

SUBCOMMITTEE D01.53 COIL COATED METAL

R. A. Cassel, Chairman

Sub. D01.53 met with three members and three guests in attendance. The main topic was the lack of working participants in the subcommittee. The chairman commented that within the next two months, he and J. C. Weaver were planning to visit with R. Schuck, Vice-President— Technical of the National Coil Coaters Association, in an attempt to gather support. Another suggestion made at the meeting was that a news release in the *Standardization News* cover the problem and request help.

Should additional support not be forthcoming the chairman can see no other recourse than to complete the existing document work and revert to an inactive subcommittee status until one of the existing documents needs review.

SUBCOMMITTEE D01.55 FACTORY-APPLIED COATINGS ON PREFORMED PRODUCTS

F. Zurlo, Chairman

D01.55.01, Coatings For Wood Products, M. Mull, Chairman. R. F. Wint chaired the meeting in the absence of the chairman. D 1211, "Temperature Change Resistance of Clear Nitrocellulose Lacquer Films Applied to Wood," was reviewed by a task group and circulated to the Sub. D01.55 members. The scope was modified to eliminate redundancy between it and the 'Significance and Use' section. More important, the construction procedure for test panels in paragraph 6.1 has been specified in more detail than previously. The method is ready for D-1 ballot.

One negative from the recent ballot on D 365 was found not persuasive. The safety caveat had been included in paragraph 1.2 and other precautions for specific hazardous solutions in paragraphs 1.2.2.1, 12.3.1, and 12.5.1. Other

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The negative by H. E. Ashton relating to paragraphs 8.1 and 10.1 of D 2337, "Freeze-Thaw Stability of Multicolor Lacquers," was found to be persuasive. The sections involved will be modified to accommodate the negative. The negative relating to paragraph 9.1 was voted not persuasive. The internal phase of these aqueous emulsions may agglomerate changing both particle size and shape. Therefore, the procedure must be retained as written.

D01.55.07, Industrial Water-Reducible Coatings, by F. Zurlo, Acting Chairman, discussed numerous negatives and comments by H. E. Ashton on the proposed "Guide for Testing Industrial Water-Reducible Coatings." Nearly all were acceptable and appropriate changes were made. A section on block resistance will be added. Several points were not persuasive. Other sections were rewritten to accommodate those points found persuasive. Minor differences in text were noted, but the group preferred the existing form. With the negatives resolted by these changes, the method will be submitted for Society ballot.

SUBCOMMITTEE D01.56 PRINTING INKS

J. M. Fetsko, Chairman

D01.56.01, Ink Viscosity—This task force, which had previously developed D4040 on the falling rod viscometer, was reactivated in order to investigate test methods for liquid inks and other lower viscosity systems. C. Shepard was appointed chairman.

D01.56.02, Lightfastness (of Printed Matter)—A. Mulvihill, new Chairman, succeeding J. Benson, reported that, through the courtesy of Pantone, Inc., the committee has received over 300 replicates of 16 different prints representing four pigments, two print coverages, and two types of paper. On the basis of exploratory experiments, fading studies will be conducted so as to determine rate of degradation in available artificial light devices and to sunlight under various types of glass, seasons of the year and locations around the country.

D01.56.06, Ink Tack—C. Shepard reported that the new test method, "Apparent Tack of Printing Inks by the Inkometer," passed the ASTM Society ballot and will appear as D 4361 in Part 06.01 of the 1985 Book of Standards.

D01.56.09, Tinting Strength (of Dispersions)—W. Riedel, Chairman, reported that 12 laboratories participated in a round robin in which diarylick yellow flushes were let down in a blue base for visual evaluation and in a white base for instrumental evaluation of relative strength. The required precision statement for D 3425 can now be calculated; however, the subcommittee and the ink industry will be surveyed for experiences with various types of bases and other key points in the procedure.

D01.56.10, Water Uptake by Litho Inks—G. Bien, Chairman, reported that 10 laboratories participated in a final round robin in which water uptake of six inks was measured at four times, both gravimetrically and volumetrically. After the results are analyzed statistically, it can be decided whether the volumetric approach can supercede gravimetric determination.

D01.56.11, Nonvolatile Matter (of Heat-set Inks)—B. Blom, Chairman, reported that 10 laboratories participated in the final round robin in which six heatset inks were tested for nonvolatile matter by heating a relatively thin film of sample at 110°C for three hours. There was excellent agreement between the experimental and theoretical NVM's and the method can now be written up for submission to ASTM. Next, D 2369 will be examined for applicability to various types of liquid inks.

D01.56.12, Fineness-of-Grind-J. Cichon, Chairman, reported that D 1316, "Test for Fineness of Grind of Printing Inks by the Production Grindometer," will be modified to specify that the average of scratch endpoints on four drawdowns constitutes a single determination. Early returns from a round robin in progress not only confirmed the validity of the new instructions but also appeared to show a good correlation between speckle readings and gloss measurements taken directly on the drawdowns.

D01.56.13, Ink Mileage—B. Blom, Chairman, will distribute pairs of case history inks to four laboratories in order to determine the validity of relative ink mileage tests run on bench-type presses. Consideration will also be given to full scale round robins to determine the precision and accuracy of automated bench presses as well as common routine control tests.

D01.56.14, Setting of Heat-Set Inks— A. Urdea, Chairman, reviewed test procedures received from committee members, most of which use the Sinvatrol Tester for drying heat-set inks. A method specifying belt speed, temperature increments, and end-point determination will be prepared and checked out in a forthcoming round robin.

D01.56.15, Specific Gravity (of Printing Inks)—G. Bien was appointed chairman of a new task force to study the applicability of existing ASTM test methods for measuring specific gravity of various types of inks.

D01.56.16, Abrasion (of Gravure Inks)—J. Hart was appointed chairman of a new task force established to investigate test methods for abrasion of rotogravure inks. A limited round robin will be conducted to compare a new high speed tester with testers commonly used in the USA.

SUBCOMMITTEE D01.57 ARTISTS' PAINTS

J. T. Luke, Chairman

A meeting was held on April 8 at the Michigan Inn, Detroit, with 15 members and guests present. Chairwoman Luke received approval of the minutes from the December meeting.

The Chairwoman made reference to the three Standards prepared by the subcommittee which have been published in the 1984 Annual Book of ASTM Standards, Vol. 06.01, Section 6. The subcommittee did not have the opportunity to proofread the final copy and some errors were found and brought to the attention of ASTM. An errata sheet will be prepared.

The revisions to D 4302 and D 4303 contained in the October ballot #10-83 have all been approved. Proposed additional revisions were presented and discussed as follows:

(1) D 4302, Section 5.6—Add some alternate wordings that may be used for conformance such as "Conforms to the quality requirements of ASTM D 4302" or "Conforms to the quality and health requirements of ASTM Standards D 4302 and D 4236" or "Conforms to ASTM D 4302 and D 4236." Any of the above statements could be used to claim conformance as well as the phrases given in the current revision to D 4302.

A motion was passed to present this new section to a subcommittee ballot.

(2) D 4303, add new Section 10.2– Visually check the exposed specimen against the unexposed specimen which has been kept in subdued light to verify that the color difference stated on ΔE^*ab agrees with visual assessment. Make this check following the second and any further exposures.

A motion was passed to present the new section to a subcommittee ballot.



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(3) D 4303, present Section 10.2.1-Adiscussion was held on a recent subcommittee ballot as to the requirements for the first two lightfastness tests which will place pigments in appropriate lightfastness categories. The five possible combinations were:

- (a) Any two tests, including tests at the same site at different times.
- (b) Any two tests, provided the second test is made at a different location having significantly different exposure conditions.
- (c) One sun test and one indoor test (fluorescent or Xenon-arc apparatus).
- (d) One south Florida sun test and one indoor test (fluorescent or Xenonarc apparatus).
- (e) One south Florida sun test (or equivalent location in another country) and one fluorescent test.

Option 4 received the most first choice votes. A motion was passed in favor of revising Section 10.2.1 to include option 4. It was also suggested that a reference pigment be included every time a new test is started. This could be used to determine if any aberrations occurred during the test.

(4) D 4303, Section 10.2.3—Discussion was held as to what test should be made in cases where one test is within $\pm 0.5 \Delta E^*$ ab of the dividing line between categories. It was agreed that it was best to repeat the test that gave the lower lightfastness rating being sure to exclude data caused by any anomalies. Exact wording will have to be worked out.

(5) D 4236, Insert section of conformance—new Section 5.9 a statement of conformance is needed. This statement will be similar to the conformance statement worked out for D 4302. A motion was passed to prepare a revision incorporating similar language as contained in D 4302 Section 5.6 and incorporate it into a new Section 5.9.

(6) D 4236, Section 1.3—An editorial change is recommended changing "should" to "shall".

D01.57.01, Preparation of Samples for Colorimetric Determination, T. Pamer, Chairman. Information was circulated showing the colorimetric data from acrylic samples prepared by two participants using 10 mil draw-down equipment. The data looked favorable enough to conduct a round robin using 10 mil clearance templates to be supplied by Irv Shack. D01.57.03, Tinting Strength, T. Pamer, Chairman. A round-robin was conducted on tinting strength using the method of Ruth Johnston-Feller. Good agreement was achieved by most participants. T. Pamer will collect data to set levels of dilution with white for each pigment and then see if some standard curves for minimum tinting strength can be established.

D01.57.07. Physical Properties, H. Levison, Chairman. A report "The Adhesion and Cohesion of Normal Films of Artists' Acrylic Emulsion and Oil Tube Paints," prepared by H. Levison, was distributed. The report describes testing done using a "Semimicro System 2000 Adhesion Testing Apparatus." The force necessary to determine the adhesion between paint coats or the inner cohesion of a paint film that may be the weakest point in multiple paint coats is specified in the report. Subcommittee members were asked to read the report and consider whether it would be useful to have an ASTM practice based on the method.

D01.57.09, Lightfastness of Water Color (new Task Group), T. Vonderbrink, Chairman. A brief discussion was held on possible ways to test the lightfastness of artists' water colors. Some work is currently being done preparing water color washes and masking one half of the sample and noting color change after exposure.

A "Proposed Procedure for Testing Lightfastness of Artists' Water Colors," prepared by T. Vonderbrink, was circulated for comments. The proposed method involves air brushing water colors on water color paper until 40% relative reflectance at the wavelength of maximum absorption is reached. Written comments on the proposed method were requested.

Information on the HPUV Indoor Actinic Exposure System was distributed. The producer of the unit said if there is sufficient interest this unit could possibly be modified to simulate normal indoor exposure in accelerated lightfastness tests.

D01.57.10, Tests for Evaluation by Consumers, Z. Pinney, Chairman. This is a new task group which is looking into simple ASTM methods which could be used by consumers to do product testing. A discussion was held as to some of the possible projects that the task group should include in their work. D01.57.11, Alkyd Artists' Paints (new Task Group), P. Staples, Chairman. A report from P. Staples was circulated covering how ASTM D 4302 could be revised to include alkyd paints and subcommittee members were asked to comment before the January Sub. D01.57 meeting.

Also requested was an opinion of the subcommittee as to whether all pigments had to be retested in the alkyd vehicle or whether the lightfastness ratings developed for linseed oil paints could be applied for the alkyds.

After much discussion, the following motion was passed: "The subcommittee will formulate a list of pigments which should be tested as follows: Lightfastness I pigments in both oils and acrylics do not have to be tested unless they are suspect as to color changes by the Review Committee. The Review Committee will also use the 1959 test data developed by Max Saltzman. All Lightfastness II pigments in either oil or acrylic vehicle must be tested."

DIVISION 60 PAINT APPLICATION

SUBCOMMITTEE D01.61 PAINT APPLICATION TOOLS

F. B. Burns, Chairman

D01.61.01, Paint Brushes, T. J. Sliva, Chairman. Two paint brush characterization tests were presented for discussion as candidates for round-robin testing. One of these methods is to determine the paint discharge characteristics of a brush. Eight cooperators agreed to participate, and the chairman plans to complete a roundrobin before the next meeting.

D01.61.02, Paint Rollers, J. F. Price, Chairman, announced that the first round-robin test on Discharge Characteristics of a Roller Cover was in progress, and to date three of the seven cooperators had returned the results. Discussion of the proposed test method at this meeting centered on the variability of the test substrate. The chairman agreed to examine the effects of a variety of substrates prior to the next meeting, when the complete round-robin results will be discussed.

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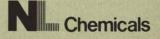
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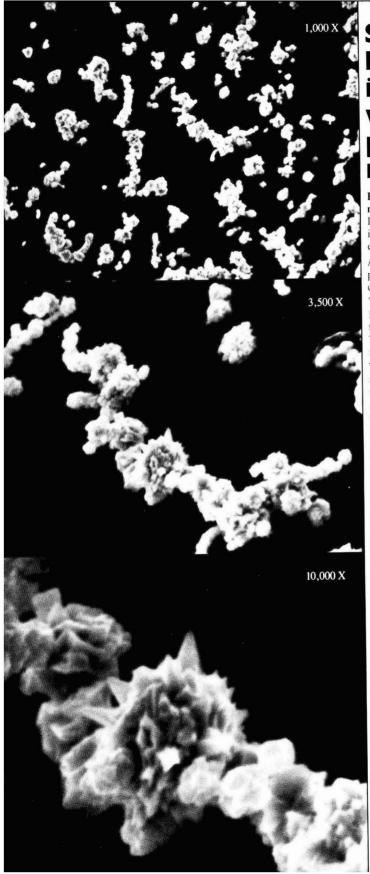
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New Courses Are Added To Kent State Fall Program

Two new short courses will be presented this fall at Kent State University, Kent, OH. "Paint Application for Paint Manufacturers" will be held November 7-8, and "Introduction to Coatings Technology" will be held November 13-16.

"Paint Application for Paint Manufacturers" will describe the industrial coating processes with emphasis on how they are applied. Safety, economy, ecology, and energy requirements for various modes of paint application and cure will also be discussed. The instructor will be Prof. Norman R. Roobol, of GMI Engineering and Management Institute and NR Consulting.

"Introduction to Coatings Technology" will discuss the materials used in coatings manufacture, formulating techniques, testing, and an introduction to new trends in coatings. The instructor will be Prof. John A. Gordon, Jr., of the University of Missouri-Rolla and Pacific Technical Consulting. Attention will be given to the needs of newcomers to the coatings industry.

For additional information, please contact Dr. Carl Knauss, Kent State University, Chemistry Dept., Kent, OH 44242. and lunch; there is no charge for the second day.

As in the past, attendees are urged to bring color samples of any materials with which they are having problems, for discussion with the technical experts who will be on hand to provide solutions. In order for Macbeth to tailor each seminar to the attendees, registrants are asked to fill out a questionnaire before the seminar. This will provide sufficient information to enable the instructor to "zero in" on the particular requirements of the attendees.

The locations and dates for the new 1984 schedule are as follows: Cherry Hill, NJ, November 1-2; Vancouver, BC,

Canada, November 8-9; Grand Rapids, MI, November 15-16.

The following seminars are scheduled for 1985: Boston, MA, December 6-7; Charlotte, NC, January 10-11; Dallas, TX, January 24-25; Atlanta, GA, February 7-8; Detroit, MI, April 12-13; Seattle, WA, April 18-19; Cherry Hill, NJ, May 2-3; San Francisco, CA, May 16-17; and Chicago, IL June 6-7.

In addition to this schedule, arrangements can be made for an in-house presentation of "The Fundamentals of Color." Additional information and application forms can be obtained from Jeanne M. Dolan, Macbeth, Little Britain Rd., P.O. Box 230, Newburgh, NY 12550.



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As a result of the company's growth in Southern Florida, U.S. Paint, a division of Grow Group, Inc., has named Jeffrey Holland to the position of Acting Sales Representative for that area. Mr. Holland is a member of the St. Louis Society.

Paul D. Roeder has been promoted to Chemist at U.S. Paint. He formerly served the company for eight years as a Laboratory Technician.

Dr. Bruce C. Henshaw has been promoted to Business Manager for Acrylic/ PVA Products in the Specialty Polymers Div. of McCloskey Varnish Co., Philadelphia, PA. A member of the Pacific Northwest Society, Dr. Henshaw will be responsible for both the technical development and marketing of the firm's acrylic/PVA products.

McCloskey Varnish Co. has announced the promotion of Gary A. Welp to Business Manager in the Specialty Polymers Div./Pacific Northwest Region. Mr. Welp is also a member of the Pacific Northwest Society.

Fred R. Spohrer has been appointed Technical Director for Red Devil Paints & Chemicals, Mount Vernon, NY. A member of the New York Society, Mr. Spohrer most recently held the position of Senior Chemist for the firm.

Greg Mains has joined Mooney Chemicals, Inc., Cleveland, OH, as a Sales Representative for the southwestern territory. He will be based in Houston, TX. Mr. Mains succeeds Timothy D. McLaughlin, who has been promoted to Sales Manager and has relocated to the company's corporate headquarters.

Larry liams has been appointed Vice-President—Technical for United Coatings, Inc., Chicago, IL. In this position, he will direct the firm's activities for research, development, and quality assurance.

Universal Color Dispersions, Lansing, IL, has named **Chuck Fritze** to the position of National Sales Manager. Mr. Fritze is a member of the Chicago Society.





J. Holland

B. Henshaw



G. Robertson

J.

R. Orsulak

Gregg A. Robertson has been appointed Area Supervisor, Sales and Service, for the Metals Coatings Div. of Diamond Shamrock Chemicals Co., Chardon, OH. He will be responsible for east coast sales and customer service activities for the division's proprietary, non-aqueous degreasing and phosphating solution.

Timothy T. Walsh has been appointed President of Walsh & Associates, Inc., St. Louis, MO. He succeeds **Robert F.** Walsh, who will serve as Chairman of the Board. Both men are members of the St. Louis Society.

Bryan L. Kinnamon, Project Manager of Goodyear Research and Development projects for the past two years, has been named Director of Technical Center Manufacturing. He succeeds Edward W. Kissel, who was recently named Production Director for the Western Hemisphere, Goodyear International.

The Coatings Division of Ferro Corporation, Cleveland, OH, has named **Thomas J. Case** to the position of Sales/Service Representative for organic coatings. He will be responsible for sales and technical service of Vedoc[®] powder coatings and Fiberglass Reinforced Plastic gel coats in the northwest region of the U.S.

Thomas E. Rayman has joined the research staff of PRA Laboratories, Inc., Chicago, IL. A member of the C-D-I-C Society, he will specialize in industrial and trade sales resins.

Silberline Manufacturing Co., Lansford, PA, has promoted **Richard P. Orsulak** to the position of Quality Assurance Director. He will direct the activities of the Quality Control Labs in all of the company's U.S. plants.

John E. Stanko, Jr., a member of Silberline for 14 years, has been promoted to Product Assurance Supervisor. He will be responsible for all activities of the Lansford-based Quality Control Labs.

Justin B. Arnold, of Paul Uhlich & Co., Inc., has been elected President of the Dry Color Manufacturers' Association. He succeeds Allan Weissglass, of Magruder Color Co., Inc., who will serve on the Board of Directors as Immediate Past-President.

Also elected were: First Vice-President—S. Paul Malchick, of BASF Wyandotte Corp. and Second Vice-President—John Boehle, of CIBA-GEIGY Corp. Re-elected as Executive Vice-President and Secretary/Treasurer was J. Lawrence Robinson. Pamela Lehr was appointed Assistant Secretary.

Elected to the Board of Governors were: Peter Birrell, of Dominion Colour Co.; Jon Fisher, of Harshaw/Filtrol Partnership; Heinz Geiss, of American Hoechst Corp.; Paul Klein, of Sun Chemical Corp.; and Norman Rumpf, of Apollo Colors, Inc.

Layton "Rick" Kinney, Past-President of the Chicago Society, has been appointed head of the recently created New Ventures Group at the Sherwin-Williams Co., Chicago, IL. Mr. Kinney and the group will be responsible for seeking new markets for existing Sherwin-Williams technology and new technologies for coatings.

As part of its organizational restructuring, Macbeth, division of Kollmorgen Corp., has established five product groups to provide a series of specific color communication capabilities. Newly appointed managers of the restructured product groups are: Harold Marcus-Munsell Color Group of color standards and companion products; Michael Mc-Govern-Daylighting Group of controlled lighting systems; Peter Tutini-Off-Line Group of color measurement instruments; David Atkinson-Systems Group of computerized color matching systems; and George Lorditch-On-Line Group of computerized color control systems for continuous production operations. Dr. James Davidson will serve as President.

The election of national officers for 1984-85 was recently announced by the Chemical Coaters Association: President-Gary Frazier, of Hentzen Coatings, Inc.; First Vice-President-Thomas McCardle, of Kolene Corp.; Second Vice-President-Bruce Reinhardt, of Nordic Ware, Inc.; Secretary-Garry Vandekieft, of Broan Manufacturing Co.; and Treasurer-Philip Vadeboncoeur, of Heatbath Corp.

The following were elected to threeyear terms of the Board of Directors: Wilbur Chinery, of Mack Trucks, Inc.; Merle Kvaal, of Graco, Inc.; L.G. Myers, of LubeCon Maintenance Systems, Inc.; and Charles Sass, of Deere & Company.

Thomas France has been appointed Sales Representative for the Midwest region of Day-Glo Color Corp., Cleveland, OH. Mr. France will be based in Chicago, IL.

Richard G. Light has been named President of Whittaker, Clark & Daniels, Inc., South Plainfield, NJ, F.F. Roesch will continue in the position of Chairman of the Board and Chief Executive Officer

United Technologies-Inmont, Clifton. NJ, has named Matthew Guagliardo to the position of Group Leader, Automotive Coatings R&D, in the central research laboratories of the company.

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Obituary

Richard Van Deutekom, one of the founders of the Kansas City Society, died May 10 at the age of 94. He retired from the Tnemec Co., Inc., Kansas City, in 1959

Mr. Van Deutekom immigrated to the United States from Holland in 1890, and began his long career in the paint industry with Sherwin-Williams Co. in 1909 in Chicago, where his responsibilities included house paint and furniture finish production. He left Sherwin-Williams in the 1920's and became a consultant to two paint companies in St. Louis. In 1930, he came to the Tnemec Co. as Vice-President in charge of production, formulation, and purchasing.

Mr. Van Deutekom's interests extended beyond the paint industry. He was an accomplished athelete and long distance runner, being chosen for the 1912 U.S. Olympic team (he did not compete because of business pressures). He and his wife, Florence, who died earlier this year, travelled extensively.

Marvin Kress, Technical Director of Red Devil Paints & Chemicals, died on July 13. A 39-year veteran of the paint and coatings industry, Mr. Kress was responsible for chemical purchasing, product formulation, quality control, and research and development. He was a member of the New York Society.

Evelyn Young Roemer, co-founder of the Jesse S. Young Co., died on July 1. Mrs. Roemer was President of the Young organization until 1982, when illness caused her to retire.

Literature

PVC Filter Cartridge Housings

A new line of PVC filter cartridge housings designed to provide low cost housings with broad chemical compatibility has been introduced in literature. These housings are intended for industries which require the filtration and purification of water, seawater, chemicals, plating solutions, and other corrosive fluids. For additional information, write AMF Cuno, Public Relations Dept., 400 Research Pky., Meriden, CT 06450.

Tanks

Flat bottom tanks which are constructed of heavy steel with welded seams to withstand maximum loads and to prevent ruptures are described in recently released literature. For more information, contact Certified Equipment & Mfg. Co., P.O. Box 298, Springfield, 1L 62705.

Adhesion Measurement

A precision instrument for measuring adhesion by the tape test is the focus of technical literature. Adhesion evaluations in accordance with ASTM D-3359 can be performed in the field or laboratory. For further information, contact KTA-Tator, Inc., 115 Technology Drive, Pittsburgh, PA 15275.

Pigment

Literature is available on a new phthalocyanine blue designed for paints and coatings. Phthalocyanine blue exhibits low rheology and a high degree of transparency. Data on Sunfast® Blue 248-0615 is available from Sun Chemical Corp., Pigments Div., Technical Service Dept., 4526 Chickering Ave., Cincinnati, OH 45232.

Color Chart

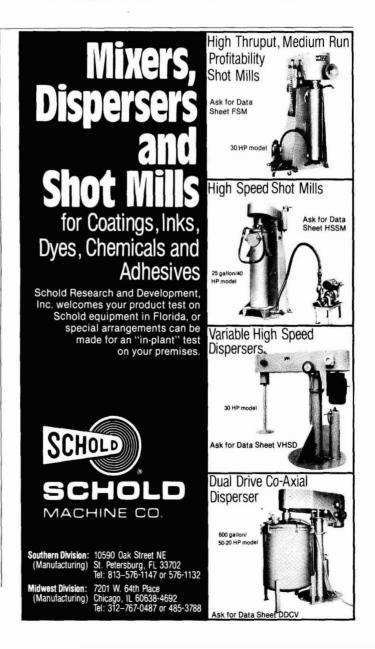
A new 32-color, powder coatings chart can now be obtained. The color chart includes 15 polyester/polyurethane and 17 epoxy powder coatings standard colors. For a copy of the color chart, or more information, contact Ferro Coatings Div., Ferro Corp., Edward J. Bickrest, Manager of Communications, 4150 E. 56th St., Cleveland, OH 44105.

Propylene Glycol Ethers

The cost/performance effectiveness of proplylene glycol ethers and acetate used as substitutes for ethylene-based solvents is described in a new folder. Copies are available from ARCO Chemical Co., Marketing Communications Dept., 1500 Market St., Philadelphia, PA 19101.

Spectrophotometers

Performance specifications of a new generation of spectrophotometers is included in a recently published product bulletin. For details on MS-2020+, write Macbeth, a division of Kollmorgen Corp., Color Communications Products, P.O. Box 230, Newburgh, NY 12550.



Color and Appearance Measurement

A new 265-page guide for the instrumental testing of products for appearance quality or for conformity to appearance specifications has recently been published by the American Society for Testing and Materials. The contents consist of appearance measurements for scientists, engineers, and technologists with quantitative permanent data about the important attributes of products. Included in the book are 65 standards. For information or a copy of the book, contact ASTM, 1916 Race St., Philadelphia, PA 19103.

"A Special Chemistry"

A 36-page, four-color publication which details major markets of products including agricultural chemicals, pigments, dyestuffs, urethane chemicals, expandable polystyrene, vitamins, and intermediate, specialty and industrial organic chemicals, has recently been published. Requests for copies of "A Special Chemistry," should be sent to BASF Wyandotte Corp., Public Affairs Dept., 100 Cherry Hill Rd., Parsippany, NJ 07054.

Interlaboratory Organic Coatings

Results of an interlaboratory study comparing the precision, sensitivity, and correlation provided by three ASTM methods for measuring abrasion resistance of organic coatings have been summarized in a recently published ASTM report. The three methods compared are Test Method for Abrasion Resistance of Organic Coatings by the Air Blast Abrasion Test (D 658), Test Method for Abrasion Resistance of Organic Coatings by the Falling Abrasive Tester (D 968), and Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser (D 4060). The report (D01-1037) is available by contacting Dolores Collyer, ASTM Information Center, 1916 Race St., Philadelphia, PA 19103.

Polymers

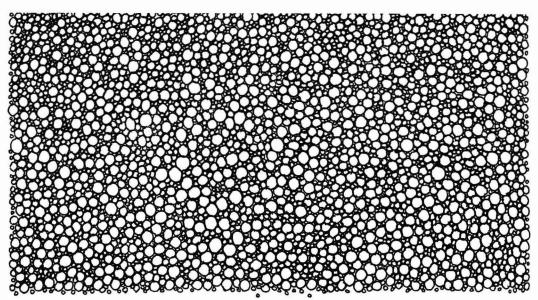
A new pamphlet describes the composition, characteristics, and benefits of a full line of water-soluble polymers for industrial use. For copies, write to the Water-Soluble Polymers Div., Henkel Corp., 7900 W. 78th St., Minneapolis, MN 55435.

Storage Tanks

A new four-page brochure describes a line of underground storage tanks for hazardous materials. The all-steel tanks provide secondary containment plus continuous monitoring for maximum protection against leaks. The two-color brochure describes and illustrates the tanks, details features and benefits, lists standard and optional features, and provides specifications. For additional details on Haz-Tank[®] underground storage tank, contact Clawson Tank Co., 4701 White Lake Rd., Clarkson, MI 48016.

Cycloaliphatic Epoxide

A 12-page booklet on the use of cycloaliphatic epoxide as an acid scavenger and stabilizer has been issued. Applications in functional fluids, electrical products, high-solids coatings and printing inks, sealants and plastics are discussed. Copies of "Cycloaliphatic Epoxide ERL-4221 Acid Scavenger-Stabilizer," designated F-5005, can be obtained from Union Carbide Corp., Specialty Polymers and Composites Div., Dept. M1553, Danbury, CT 06817.



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	25	"Automotive Finishes"—Ralph Williams (July 1977)	\$	
	26	"Corrosion and the Preparation of Metallic Surfaces for Painting"—Clive H. Hare (Feb. 1978)	\$	1.10.2010.012
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Electromagnetic Sciences Directory

A new source directory for industrial, medical, and scientific users interested in exploring new processes and high productivity through the use of radio frequency and microwave energy has been released. "International Directory of Electromagnetic Heating and Instrumentation," published by the International Microwave Power Institute (IMPI), is the only compilation of source data for systems, components, and services available in this specialized field. Copies may be obtained by sending \$8 to IMPI, Tower Suite 520, 301 Maple Ave. West, Vienna, VA 22180.

Polymer Capabilities

A new four-page brochure, designed to help those involved in the analysis and specification of polymers components for end use markets such as adhesives, flexible packaging, textiles and nonwovens, and paint and paper coatings industries, has recently been published. For a copy of "76 Polymers: A Name to Grow With," contact Gus Cottros, 76 Polymers, Chemicals Div., Union Oil Company of California, 1900 E. Golf Rd., Schaumburg, IL 60195.

Image Analysis System

A newly developed image analysis system for the measurement and evaluation of microscopic and macroscopic images has been introduced in literature. The system is designed for use for direct microscopic observation or on photos, prints, or transparencies, across a broad range of scientific disciplines. For additional details on the Zeiss Interactive Digital Analysis System (ZIDAS), contact Scientific Instruments Div., Carl Zeiss, Inc., One Zeiss Drive, Thornwood, NY 10594.

Vinyl Resins

A 38-page booklet describing vinyl resins for coatings has recently been issued. The booklet discusses 12 vinyl resins, which are produced by a proprietary solution polymerization process, providing advantages in coatings. An applications table matches these resins with their use in packaging, product finishes, building products, marine and maintenance, and magnetic tapes. Copies of the booklet, "UCAR Solution Vinyl Resins for Coatings," are available from Union Carbide Corp., Specialty Chemicals Div., Dept. K3442, Danbury, CT 06817.

Computer Color Control

A new full-color brochure describes a complete family of integrated computer color control systems. Operating in conjunction with each other, the systems are designed to provide fast and accurate creation, specification and communication of color; color formulation and correction, colorant weighing and dispensing; and quality control checking. For details, contact Applied Color Systems, Inc., P.O. Box 5800, Princeton, NJ 08540.

Corrosion Preventive Bases

A group of solvent-type bases that are designed to provide a barrier to water and humidity are described in recently released literature. These wax-modified products exhibit high resistance to water and also stable viscosities at low temperatures which simplify spraying and handling of finished coatings. Further information on SACI 2400 rust preventive concentrates may be obtained from Witco Chemical Corp., Sonneborn Div., 520 Madison Ave., New York, NY 10022.

Packaging Adhesive

Information is available on a new water-resistant vinyl acetate-ethylene copolymer emulsion which is designed for numerous packaging-type applications. The properties include compatability with fully and partially hydrolyzed polyvinyl alcohol, better open tack time, and water resistance. For more information on Airflex 320 emulsion, contact Air Products and Chemicals, Inc., Polymer Chemicals Div., P.O. Box 538, Allentown, PA 18105.

Solvent Dispersions

A new black pigmented solvent dispersion in a universal type vehicle system is the subject of recently released literature. For details, write CDI Dispersions, 27 Haynes Ave., Newark, NJ 07114.

Acrylic Emulsion

A water-borne acrylic emulsion that offers adhesion to plastics and metals combined with resistance to chemicals and corrosion is discussed in new literature. Sample formulations, including a gray texture coating for business machines, and product property data sheets are available from Polyvinyl Chemical Industries, John Fitzwater, Manager, Coatings Market, 730 Main St., Wilmington, MA 01887.



ADHESION ASPECTS OF POLYMERIC COATINGS

Edited by K.L. Mittal

Published by Plenum Press New York, NY 1983, 657 pages

Reviewed by Larry F. Brinkman H.B. Fuller Company St. Paul, MN

This book contains 36 papers, most of which were presented at the Symposium on Adhesion Aspects of Polymeric Coatings held under the auspices of the Electrochemical Society in Minneapolis, MN, May 10-15, 1981.

The use of coupling agents is discussed in ten different papers. Contributions to adhesion include improved surface wetting and physisorption which creates reactive sites at the interface. The bulk of the discussion centers on silanes, but some work covers titanates, zirconates, and beta-diketones. The material presented in these papers will offer insight to the chemist who has formulated with coupling agents for years without a clear understanding of why they work so well.

Surface preparation is examined in several articles. Beneficial changes in surface chemistry may be introduced by varying metal/oxygen ratios with proper substrate etch. Topography may be controlled for better adhesive contact angle and surface pore shapes optimized for mechanical adhesion. Surface oxide microlayers provide the prime entrance for water which leads to corrosion and adhesive failure.

Photoresist adhesion is covered in several papers. Lithographic materials and the processing of silicon dioxide surfaces are discussed in reference to making integrated circuit elements. This is rather specialized chemistry and may only be applicable to a smaller group of industrial scientists. The final section discusses advances in adhesion testing. Papers on state of the art thermography and ultrasonics for non-destructive qualitative testing are interesting, but will never be tested by anyone but the analytical chemist.

This book is a collection of articles describing interface properties, one of the most critical aspects in adhesion. It offers something for the researcher as well as the formulating chemist and should be passed from reader to reader.

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FEDERAL TECHNOLOGY CATALOG, 1982

Summaries of Practical Technology

Published by U.S. Dept. of Commerce National Technical Information Service Springfield, VA 400 pages, \$19.50

Reviewed by Ross Dowbenko PPG Industries, Inc. Allison Park, PA

The large, paperback book is published by NTIS (National Technical Information Service), an agency of the U.S. Dept. of Commerce, which according to the introduction is the central source for the public sale of U.S. Government sponsored research, development, and engineering reports. According to the Introduction, "This catalog is a compilation and index to the more than 1000 Tech Notes announced in the past year. Each Tech Note fact sheet has been summarized to provide enough information to allow judgment whether a particular technology is worth pursuing."

The technology summaries cover the following ten areas: computers, electrotechnology, energy, engineering, life sciences, machinery and tools, manufacturing, materials, physical sciences, and testing and instrumentation. In addition to this, there is a subject index in which each technical note is indexed alphabetically.

Since this review is written for a special kind of readership, people who do research in polymers and paints, it was of interest to examine some of the entries in the index. Thus, there was one entry under Paint, titled, "Wide Temperature Range Torque Stripe Paint: Urethane Based Material Retains Its Useful Properties in Extreme Heat and Cold." The entry of Coatings in the subject index has 18 entries. Except for a few, most of the entries have to do with coatings which are not of the type that the readership of this journal usually deals with, for example, technologies such as carbide electrodeposition, coatings for hybrid micro circuits, corrosion resistant ceramics, plasma spray for difficult to braise alloys, electrodeposition of tantalum chromium alloys, protective coatings and sealants for solar mirrors, and others. A few of the entries deal with coatings as we know them, for example, the phosphating process to improve corrosion resistance, a flame retardant coating based on polyurethanes. and a few others. The entries Polymeriza-

ADHESIVE AND SEALANT COMPOUND FORMULATIONS

Written by Ernest W. Flick

Published by Noyes Publications, Park Ridge, NJ 366 pages, \$48.00

Reviewed by Joseph W. Prane, Industrial Consultant Elkins Park, PA

Papers to Be Featured in the November Issue

Effect of Organic Solvents on Internal Stress in Latex Coatings—Dan Y. Perera and D. Vanden Eynde, of Coatings Research Institute.

Thermosetting Coatings. Analytical and Predictive Capability by Chemorheology—Richard R. Eley, of Glidden Coatings and Resins, Div. of SCM Corporation.

Transformation of Liquid to Amorphous Solid: Effect of Reaction Mechanism on the Time to Vitrify for Linear and Network Polymerization— Marc T. Aronhime and John K. Gillham, of Polymer Materials Program, Princeton University.

Forensic Chemistry of Coatings—Hans K. Raaschou Nielsen, of Scandinavian Paint and Printing Ink Research Institute.

Viscometry: Myth and Reality-Los Angeles Society.

tion and Polymers in the subject index have a total of nine entries. It will be of interest to list all of these entries. These are as follows: neutralizing amine-cured epoxy surfaces, perflorinated imidovl imidoxime polymers, thermal polymerization of n-butyl acrylate, characterization of polymers by NMR, deformationinduced Anisotropy of polymers, hybrid polymer microspheres, materials for plastic solar thermal collectors, polymer alloy blood compatible surface, and super absorbent multi-layer fabric. Other entries in specific polymers such as polyesters, polyethylene, polypropylene, polymethyl methacrylate, polyimide resins, polystyrene, polyurethane resins have similar entries of marginal interest and relevance to those working in polymers and paint.

While this book may have its place in a complete library which is devoted to polymers and coatings research and development, this book is not recommended for individual researchers working in these fields.

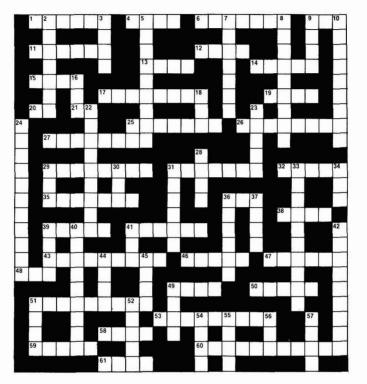
This book is the second edition of a similar work which was published by Noyes in 1978. This later edition follows a similar pattern and compiles 444 adhesive and sealant formulations derived from the brochures and data sheets of a large number of raw material manufacturers. Only water-borne and solvent-free products are treated. Solvent-borne compositions are excluded, despite their importance in the adhesives industry.

The book is divided into 8 sections: Water-Based Industrial Type Adhesives (construction, fabric, flocking, foam, general purpose, industrial); Water-Based Paper and Packaging Adhesives; Water-Based Coatings (industrial, paper, and paperboard); Hot Melt Adhesives (general purpose, paper, and paperboard. pressure sensitive); Hot Melt Coatings for Paper and Plastic; Epoxy Adhesives, Castings, and Coatings (general purpose. castings and laminating, electrical, potting and encapsulation, industrial): Plastisol Compounds; and Miscellaneous Compounds (caulking, masonry, mastics, oilbased, sealants, urethanes, miscellaneous).

The book concludes with lists of trademarked raw materials and addresses of suppliers. This volume is a convenient compilation of starting formulas for water-borne and solvent-free adhesive, sealant (and some coating) compositions. However, many of these formulations can already be out of date as raw materials have been withdrawn, new materials have been withdrawn, new materials introduced, and suppliers enter and leave the market.

CrossLinks

by Earl Hill



Solution to be published in November Issue

ACROSS

- 1. Reaction product with alcohols (Org. Chem.)
- To touch (building construction)
- 6. Color theory pioneer
- 9. Color Committee (Abr.)
- 11. SI temperature unit
- 12. Belongs with 6 across
- 13. Refractometer 14. Think positivel
- 14. Think positively
- 15. Crystalline mineral silicate
- 17. Imported drying oil
- 19. Airplane coating
- 20. Large orders can cause this (Abr.)
- 21. Preposition
- Familiar mineral form
 Term involving gloss measurement
- 27. Compound formed during drying (Chem.)
- 29. Space age coating
- 31. New SI unit

- 32. Useful in baking enamels
 35. Another word for CRT (Computers)
- 36. Useful alkaline reactant (Abr.)
- 38. Well known society (Abr.)
- 39. Artificial sulphate
- 41. Feldspar mineral
- 43. Acetic acid reaction product (Chem.)
- 46. Version of 49 down
- 47. Metallic pigment reactant
- 48. Opposite of beginning 49. ____ mark (obvious junc-
- ture)
- 50. Natural gum extrudate
- 51. Coating for photographic plates
- 53. Gypsum
- 58. A test for yellowness
- 59. Developer of color scale
- 60. Type of lacquer
- 61. Important resin component

DOWN

- 2. Found in the laboratory
- 3. Type of oil (Rheol. term)
- 5. Gaseous emulsion com-
- ponent
- 7. Rheology pioneer
- 8. Substance which changes surface tension of water
- Goes with plate (viscosity)
- 10. To dissipate
- Nitrogen containing (prefix)
- 18. Chemist's society (Abr.)
- 22. Part of pigment mfg. process
- Organic chemistry compound
- 24. Derivative of Castor Oil
- 25. Advertisement
- 28. Good paint characteristic (Rheol.)
- 29. ___ Margin (Abr.)
- 30. Prefix (Org. Chem.)
- 31. Early latex paint ingredient

- 33. Chromophore reactant
- 34. Adverse film defect
- 36. Gum arabic source
- 37. Unit of vapor transmission
- A quantity of caulking mat'l.
- 40. Gaseous hyd. raw material
- 41. ___ Institute (Abr.)
- 42. Silicate mineral
- 44. An ____ solution (Chem.) 45. When we start out for
- work 49. Source of organic thickeners
- 51. What it's all about (Finance)
- 52. Iron ore pigment
- 54. ____ roll (on a mill)
- 55. Drying oil
- 56. Financial yardstick (Abr.)
- 57. Milling ingredient

Coming Events

FEDERATION MEETINGS

(Oct. 24-26)—62nd Annual Meeting and 49th Paint Industries' Show. Conrad Hilton Hotel, Chicago, IL. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, PA 19107).

1985

(Apr. 17-18)—Symposium on Color and Appearance Instrumentation.) Jointly sponsored by FSCT, Manufacturers Council on Color and Appearance, and Inter-Society Color Council. Sheraton Station Square, Pittsburgh, PA. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, PA 19107).

(May 14–17)—Federation "Spring Week." Seminar on 14th and 15th; Society Officers on 16th; and Board of Directors on 17th. Omni International/Baltimore, MD. (FSCT, 1315 Walnut St., Philadelphia, PA. 19107).

(Oct. 7-9)—63rd Annual Meeting and 50th Paint Industries' Show. Convention Center, St. Louis, MO. (FSCT, 1315 Walnut St., Philadelphia, PA 19107).

1986

(May 13-16)—Federation "Spring Week." Seminar on 13th and 14th; Society Officers on 15th; and Board of Directors on 16th. (FSCT, 1315 Walnut St., Philadelphia, PA 19107).

(Nov. 5-7)—64th Annual Meeting and 51st Paint Industries' Show. World Congress Center, Atlanta, GA. (FSCT, 1315 Walnut St., Philadelphia, PA 19107).

SPECIAL SOCIETY MEETINGS

1985

(Feb. 13-15)—"Water-Borne and Higher-Solids Coatings" Symposium sponsored by the Southern Society for Coatings Technology and the University of Southern Mississippi. New Orleans, LA. (Dr. Gordon L. Nelson, Chairman, Department of Polymer Science, University of Southern Mississippi, 'Hattiesburg, MS 39406-0076).

(Feb. 26-Mar. 1)—Western Coatings Societies Symposium and Show. Disneyland Hotel, Anaheim, CA. (11911 S. Woodruff Ave., Downey, CA 90241).

(Mar. 12-13)—Cleveland Society. 27th Annual Technical Conference on "Advances in Coatings Technology." Case Western Reserve University, Cleveland, OH. (Dr. Richard R. Eley, Glidden Coatings & Resins, P.O. Box 8827, Strongsville, OH 44136).

(Mar. 27-29)—Southern Society. Annual Meeting. Atlanta Hilton Hotel, Atlanta, GA. (Salvatore G. Sanfilippo, Reichhold Chemicals, Inc., P.O. Box 1610, Tuscaloosa, AL 35403).

(Apr. 10-12)—Southwestern Paint Convention of Dallas and Houston Societies. Anatole Hotel, Dallas, TX. (T. LaBaw, Sherwin-Williams Co., 2802 W. Miller Rd., Garland, TX 75041).

(Apr. 25-27)—Pacific Northwest Society for Coatings Technology Symposium. Empress Hotel, Victoria, B.C. (Ottwin Schmidt, Helzer Canada Ltd., 8531 Cullen Crescent, Richmond, B.C., V6Y 2W9 Canada).

1986

(Mar. 25-27)—Southern Society. Annual Meeting. Hilton Hotel, Savannah, GA. (Ronald R. Brown, Union Chemicals Div., P.O. Box 26845, Charlotte, NC 28213).

OTHER ORGANIZATIONS

(Oct. 21-26)—Interfinish '84. Jerusalem, Israel. (Interfinish '84 Secretariat, P.O. Box 29313, 61292 Tel Aviv, Israel).

(Oct. 22-24)—National Paint & Coatings Association.97th Annual Meeting. Palmer House, Chicago, IL. (Karen Bradley, NPCA, 1500 Rhode Island Ave., N.W., Washington, DC 20005).

(Oct. 23-24)--11th International Naval Stores Meeting. Fairmont Hotel, San Francisco, CA.

(Oct. 23-26)—Fall Technical Meeting of the National Coil Coaters Association. Hyatt Regency O'Hare Hotel, Chicago, IL. (NCCA, 1900 Arch St., Philadelphia, PA 19103).

(Nov. 13-14)—"Innovations in Finishing and Machining for the Wood Industry" Conference, sponsored by the Association for Finishing Processes of the Society of Manufacturing Engineers. Greensboro-High Point Marriott Hotel, Greensboro, NC. (Donna Theisen, AFP/SME Administrator, Society of Manufacturing Engineers, One SME Drive, P.O. Box 930, Dearborn, MI 48128).

(Nov. 14–15)—Seventh Resins & Pigments Exhibition. Crest Hotel de Boflelaan, Amsterdam, Holland. ("Polymers, Paints & Colour Journal," Queensway House, 2 Queensway, Redhill, Surrey RH1 1QS, England).

(Nov. 16-17)—37th National Decorating Products Show. McCormick Place, Chicago, IL. (NDPA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132).

(Nov. 26-27)—"Job Estimating Workshop for Painting Contractors" Short Course. University of Missouri-Rolla, Rolla, MO. (Cindy Sonewald, Dept. of Chemistry, UMR, Rolla, MO 65401).

(Nov. 28-30)—FINSTRAT '84, Conference and Exposition on Finishing Strategies for the 80's. Marriott Hotel and Anaheim Convention Center, Anaheim, CA. (Association for Finishing Processes of SME, One SME Dr., P.O. Box 930, Dearborn, MI 48128).

(Nov. 28-30)—"Maintenance Painting" Short Course. University of Missouri-Rolla, Rolla, MO. (Cindy Sonewald, Dept. of Chemistry, UMR, Rolla, MO 65401).

(Dec. 3-7)—Spray Painting Seminar. Binks Manufacturing Co., Franklin Park, IL. (Binks Manufacturing Co., Training Div., 9201 W. Belmont Ave., Franklin Park, IL 60131).

(Dec. 4-6)—Plant Engineering and Maintenance Show and Conference/West. Moscone Convention Center, San Francisco, CA. (Show Manager, Plant Engineering & Maintenance Shows, 999 Summer St., Stamford, CT 06905).

(Dec. 16-21)—1984 International Chemical Congress of Pacific Basin Societies. Honolulu, Hawaii. (PAC CHEM '84, Meetings and Divisional Activities, American Chemical Society, 1155 Sixteenth St., N.W., Washington, DC 20036).

1985

(Mar. 5-8)—Painting and Decorating Contractors of America. Annual Convention. Red Lion Inn, Portland, OR. (PDCA, 7223 Lee Highway, Falls Church, VA 22046).

(Mar. 25-27)—"Electrochemical Techniques for Corrosion" Symposium to be held during CORROSION/85, sponsored by the National Association of Corrosion Engineers. Boston, MA. (Symposium Chairman, Robert Baboian, Texas Instruments, Inc., Mail Station 10-13, Attleboro, MA 02703).

(Mar. 25-29)—CORROSION/85. Annual Conference for the National Association of Corrosion Engineers, Boston, MA.

(Meetings Manager, NACE Headquarters, P.O. Box 218340, Houston, TX 77218).

(Apr. 16–18)—PaintCon '85. O'Hare Expo Center, Chicago, IL. (Professional Exposition Management Co., Inc., Ste. 205, 2400 E. Devon Ave., Des Plaines, IL 60018).

(June 10-12)—International Conference on Biologically Influenced Corrosion, sponsored by the National Association of Corrosion Engineers, Washington, DC. (Meetings Manager, NACE Headquarters, P.O. Box 218340, Houston, TX 77218).

(June 11-13)—Eastern Plant Engineering & Maintenance Show and Conference. Georgia World Congress Center, Atlanta, GA. (Show Manager, Plant Engineering & Maintenance Shows, 999 Summer St., Stamford, CT 06905).

(June 16-19)—Dry Color Manufacturers' Association. Annual Meeting. The Greenbriar, White Sulphur Springs, WV. (DCMA, 206 N. Washington St., Ste. 202, P.O. Box 931, Alexandria, VA 22313).

(June 26-29)—Oil & Colour Chemists' Association's Biennial Conference. Edinburgh, Scotland. (R.H. Hamblin, OCCA, Priory House, 967 Harrow Rd., Wembley, Middlesex, HAO 25F).

(Sept. 2-4)—Federation of Scandinavian Paint and Varnish Technologists. 11th Congress. SAS Hotel Scandinavia, Oslo, Norway. (Paal Ivan, Nodest Industries A/S, Boks 500, N-3001 Drammen, Norway).

(Nov. 4-6)—National Paint and Coatings Association. 98th Annual Meeting. Hilton Hotel, New Orleans, LA. (Karen Bradley, NPCA, 1500 Rhode Island Ave. N.W., Washington, DC 20005).

1986

(Apr. 14-15)—ASTM Symposium on "Testing of Metallic and Inorganic Coatings," Chicago, IL. (Teri Carroll, ASTM Standards Development Div., 1916 Race St., Philadelphia, PA 19103).



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omissions.

Humbug' from Hillman

For the past year, Howard Jerome has been feeding the company computer. What has been coming out is not known to me but the Jerome observations and his Laws of Programming should give us reason to question the results.

Laws of Computer Programming

 Any given program, when running, is obsolete.
 Any program will expand to fill any available memory.

- If computers get too powerful, we can organize them into a committee, that will do them in.
- If builders built buildings the way programmers wrote programs, then the first woodpecker that came along would destroy civilization.
- Inside every large program is a small program struggling to get out.
- If a test installation functions perfectly, all subsequent systems will malfunction.
- Not until a program has been in production for at least six months will the most harmful error then be discovered.

And finally-

Machines work, people should think.

. . .

I am indebted, also to Larry Hill, the Hon. Editor of "Surface Coatings, Australia," the Journal of the Oil and Colour Chemists Association, Australia for some very kind comments and a real chuckle of a story.

Elderly gent of limited means saves up and buys paint (latex) for his weatherboard house. Starts at the front, but by the time he moves down one side, he realizes that he has not enough paint, nor funds to buy more. He practices the "extension with water" technique several times over, and produces a job which, when viewed from afar, is barely acceptable (so to speak). Just then a thunderstorm breaks with torrential rain and ghastly consequences for the paint job.

In his dilemma, he cries "Oh God, what do I do now?" The thunder crashes and a voice replies, "Repaint, repaint, and go thou and thin no more."

. . .

And a few more delightful Ahlfisms from Bob Ahlf via Tom Miranda:

Bilingualism is OK between consenting adults.

• Remember—you are one of the people who can be fooled some of the time!

• Going to work every day isn't bad. It's that eight hour wait before you go home that's the problem.

- I agree with you but you're wrong.
- · He, who is lost-hesitates!
- I didn't ask to be born handsome—I wish I had!

Accident Report

Dear Sir:

I am writing in response to your request for additional information about my recent accident. In block number 3 of the accident report form, I put "trying to do the job alone" as the cause of the accident. You said in your letter that I should explain more fully, and I trust that the following details will be sufficient.

I am a bricklayer by trade. On the date of the accident, I was working alone on the roof of a new six-story building. When I completed my work, I discovered I had about 500 pounds of bricks left over. Rather than carry the bricks down by hand, I decided to lower them in a barrel by using a pulley which fortunately was attached to the building at the sixth floor.

Securing the rope at the ground level, I went up to the roof, swung the barrel out and loaded the bricks into it. Then I went back to the ground and untied the rope, holding it tightly to insure a slow descent of the 500 pounds of bricks. You will note in block number 11 of the accident report form that I weigh 135 pounds.

Due to my surprise at being jerked off the ground so suddenly, I lost my presence of mind and forgot to let go of the rope. Needless to say, I proceeded at a rapid rate up the side of the building.

In the vicinity of the third floor, I met the barrel coming down. This explains the fractured skull and broken collarbone.

Slowed only slightly, I continued my rapid ascent, not stopping until the fingers of my right hand were two knuckles deep into the pulley. Fortunately, by this time I had regained my presence of mind, and was able to hold tightly to the rope in spite of my pain.

At approximately the same time, however, the barrel of bricks hit the ground and the bottom fell out of the barrel. Devoid of the weight of the bricks, the barrel now weighed approximately 50 pounds. I refer you again to my weight in block number 11. As you might imagine, I began a rapid descent down the side of the building.

In the vicinity of the third floor I met the barrel coming up. This accounts for the two fractured ankles and lacerations of my legs and lower body. The encounter with the barrel slowed me enough to lessen my injuries when I fell into the pile of bricks and, fortunately, only three vertebrae were cracked. I am sorry to report, however, that as I lay there on the bricks in pain, unable to stand and watching the empty barrel six stories above me, I again lost my presence of mind and let go of the rope. The empty barrel weighed more than the rope, so it came back down on me and broke both of my legs.

I hope I have furnished the information you require on how the accident occurred.

Herb Hillman
 Humbug's Nest
 P.O. Box 135
 Whitingham, VT 05361

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*from the Greek TRIBEIN - 'to rub or wear'

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