

BAKELITE®

VINYL *Product Data*

PLASTICS

UNION CARBIDE CORPORATION • PLASTICS DIVISION

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BAKELITE® Vinyl Butyral Resins

INTRODUCTION

Vinyl butyral resins were developed in the laboratories of Union Carbide Corporation to meet the need for a better safety glass laminating adhesive film. Excellent impact strength and tenacious adhesion to glass are required and these properties must be retained irrespective of the temperature. No color nor opacity must develop after long exposure to intense sunlight.

Moreover, the bond to the glass must hold, wet or dry, and after many cycles of wetting and drying. Vinyl butyral resins meet these requirements so well that they have been the standard safety glass interlayer since their development in 1936.

The characteristics that have made vinyl butyral resins useful as a safety glass interlayer are also of value in other adhesive applications and in the specialty coatings field.

GENERAL PROPERTIES

The commercially important vinyl butyral resins are actually partial butyrals of polyvinyl alcohol, and retain some unreacted polyvinyl alcohol groups which contribute to the many desirable properties of these resins. At the present time, two types of BAKELITE vinyl butyral resins are marketed by Union Carbide's Plastics

Division under the designations XYHL and XYSG. These are quite similar to each other chemically, but differ somewhat in average molecular weight.

Type XYHL is of medium molecular weight; type XYSG, of somewhat higher molecular weight, yields more viscous solutions, but shows maximum film strength. Some of the general properties of these two resins are given in Table I.

For convenience in handling, XYHL is also available in solution form as XYLS-2. This is a 20 per cent solids solution of XYHL resin in ethanol and can be used in any formulation requiring XYHL vinyl butyral resin. Typical properties of the solution are shown in Table II.

TABLE I

Properties of BAKELITE Vinyl Butyral Resins

	XYHL	XYSG
Form	white powder	white powder
Intrinsic Viscosity	0.81	1.16
Specific Gravity	1.12	1.12
Composition: (approx.)		
Vinyl Butyral Resin	80.7 per cent	80.7 per cent
Vinyl Alcohol Resin	19 per cent	19 per cent
Vinyl Acetate Resin	0.3 per cent	0.3 per cent
Tensile Strength, lb. per sq. in.		8,000-8,500
Modulus of Elasticity, lb. per sq. in.		350,000-400,000
Modulus of Rupture, lb. per sq. in.		11,400
Izod Impact Strength, ft.-lb. (notched specimen)		0.4-0.6
Softening Point, deg. F.		135-140

TABLE II

Properties of BAKELITE XYLS-2 Vinyl Butyral Resin Solution

Appearance	Colorless solution
Total Solids by Weight	20 per cent
Solvent	Ethanol
Brookfield Viscosity @ 25°C.	6,000 cps

Vinyl butyral resins are characterized by their excellent adhesion to a wide variety of non-porous surfaces such as glass, metal, phenolic resins, and cellulosic materials. These resins, when immersed, readily absorb 5 to 8 per cent water and become softer. Prolonged immersion does not increase the water absorption nor is the resin degraded. A unique property of this class of

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resins is the ability to undergo many alternate cycles of sorption and desorption of moisture without disrupting the bond of the resin to a great number of surfaces.

Thus, coatings based on vinyl butyral resins maintain better film integrity in water than many other resins which have higher initial impermeability but which tend to disintegrate on continual immersion.

Vinyl butyral resins are tough, flexible, and shock-resistant and they retain these characteristics over a wide temperature range. These resins are thermoplastic, softening at around 135 deg. F., but they may be reacted with many resins and reagents to reduce or eliminate this thermoplasticity.

The light stability of vinyl butyral resins is excellent. However, coatings based on unmodified vinyl butyral resins are seldom suitable for exterior exposure, usually failing by chalking as a result of water absorption. The vinyl butyral resins do not discolor on heating for several hours at temperatures of 200 deg. F. or below. At higher temperatures, the resins become amber in color, and, on prolonged heating, may lose their alcohol solubility and become more hydrocarbon soluble.

The vinyl butyral resins exhibit the following resistance properties at room temperature:

Weak acid	swells
Strong mineral acid	dissolves
Weak alkali	no effect
Strong alkali	slight effect
Alcohols	dissolves
Ketones	swells
Esters	swells
Aromatic hydrocarbons	swells
Aliphatic hydrocarbons	no effect
Animal oils	no effect
Vegetable oils	no effect
Mineral oils	no effect

The retention of the above properties in films based on vinyl butyral resins is dependent upon the plasticizers and other modifiers present.

Vinyl butyral resins permit unusually high pigment volume loadings or blending with large amounts of low molecular weight resins such as rosin and ester gum without undue loss of film strength.

TABLE III
Solubility¹ of BAKELITE Vinyl Butyral Resin XYSG

	Resin XYSG, 4 per cent in solvent			Resin XYSG, 4 per cent in solvent	
	25 deg. C.	95 deg. C. ²		25 deg. C.	95 deg. C. ²
Acetic Acid	S	S	FLEXOL Plasticizer 3GH ³	I	SW
Acetone	SW	SW	FLEXOL Plasticizer 3GO ³	I	I
Acetylacetone	G	S	Formic Acid	S	S
n-Butyl Alcohol	S	S	Hydrogenated Naphtha (boiling range 94-139 deg. C.)	SO	SW
n-Butyl Acetate (98 per cent)	G	S	Isophorone	S	S
Butyl Phthalyl Butyl Glycolate ³	I	PS	Isopropanol (anhydrous)	S	S
CARBITOL Acetate	G	S	Isopropanol (technical)	S	S
Carbon Disulphide	SO	SW	Isopropyl Acetate (98 per cent)	SW	PS
Carbon Tetrachloride	SO	SO	Mesityl Oxide	G	S
CELLOSOLVE Solvent	S	S	Methanol	S	S
CELLOSOLVE Acetate	G	S	Methyl Acetate (82 per cent)	S	S
Chloroform	G	S	Methyl n-Amyl Ketone	G	S
Cresol	S	S	Methyl CELLOSOLVE	S	S
Cyclohexanone	S	S	Methyl CELLOSOLVE Acetate	G	S
Diacetone Alcohol	S	S	Methyl Isobutyl Ketone	PS	S
Dibutoxyethyl Phthalate ³	I	S	Morpholine	S	S
Dibutyl Phthalate ³	PS	S	2-Nitropropane	I	S-CL
Dichlorethyl Ether	G	S	Nitroethane	G	PS
Diisobutyl Ketone	SO	SO	Petroleum Naphtha	I	SO
Dimethyl Phthalate ³	G	S	Petroleum Ether	I	I
Dioxane	S	S	Propylene Dichloride	G	S
Ethanol (95 per cent)	S	S	Propylene Oxide	S	S
Ethyl Acetate	S	S	Pyridine	S	S
Ethylene Dichloride	G	S	Toluene	PS	S
Ethylene Glycol	SW	SW	Trichloroethylene	SW	G
Ethyl Ether	I	SW	Tricresyl Phosphate ³	I	SW
Fenchone	PS	S	Xylene	SW	SW
FLEXOL Plasticizer DOP ³	I	SW			

Legend: S — Soluble, SO — Softens, G — Gels, CL — Cloudy Solution, I — Insoluble, SW — Swells, P — Partially.

¹Liquids in which the resin is soluble are not necessarily sufficiently active solvents to permit their use in BAKELITE vinyl resin coating formulations.

²Tests were made at temperatures slightly below the boiling points on those chemicals which boil below 95 deg. C.

³These liquids are plasticizers which may be compatible with, but are not necessarily solvents for, the resin.

SOLUBILITY OF VINYL BUTYRAL RESINS

Alcohols are the best solvents for BAKELITE vinyl butyral resins XYHL and XYSG. CELLOSOLVE solvents, cyclic ethers, and some ketones and esters are also solvents, as shown in Table III. While these data were obtained with vinyl butyral resin XYSG, they apply with minor adjustment to vinyl butyral resin XYHL. The latter resin, being of lower molecular weight, is slightly more soluble.

The unique properties of vinyl butyral resins XYSG and XYHL are the result of a judicious balance of the hydrophilic vinyl alcohol groups and the organophilic vinyl butyral groups in the molecule. As a consequence, these resins are often more soluble in mixed solvents than in any one individual solvent. This is illustrated by the minimum viscosities obtained with mixed solvents as shown in Figures 1, 2, and 3. Water is especially effective in decreasing the viscosity of solvent mixtures, as illustrated in Figure 2. Although the vinyl butyral resins are soluble in commercial alcohols, they are often much less soluble in anhydrous alcohols until a small proportion of water is added. This coupling effect of water may lead to widely divergent viscosities in a vinyl butyral resin solution, depending upon the moisture content of resin, solvent, pigment, or filler in the composition.

PLASTICIZERS

Plasticizers may be added to vinyl butyral resin coatings formulations both to increase their flexibility for some applications and to reduce viscosity of hot melts. Vinyl butyral resins are compatible with FLEXOL plasticizers 3GH, and TOF, dibutyl sebacate, and with many other chemical-type plasticizers. Raw castor oil is compatible, but tends to soften the film without developing elasticity. It may be used advantageously, however, when mixed with solvent-type plasticizers.

COMPATIBILITY OF VINYL BUTYRAL RESINS

Vinyl butyral resins are compatible with many natural resins, a few oils, and certain classes of phenolic resins, as shown in Table IV. Compatibility with nitrocellulose is borderline, differing with the grade of pyroxylin and the solvents used. The same is true with urea-formaldehyde resins. Despite the borderline compatibility, many commercial products are prepared by the addition of plasticizers and mutually compatible resins to increase the formulating latitude.

Vinyl butyral resins are often blended with large amounts of low molecular weight resins, such as rosin, ester gum, or coumarone types, in the formulation of hot melt adhesives.

INSOLUBILIZATION OF BUTYRAL RESINS

The free hydroxyl groups in vinyl butyral resins present a point of chemical reactivity through which the

resin may be insolubilized. Both resinous materials and chemical reagents are effective. Typical of the resinous materials are phenolic and urea-formaldehyde resins. BAKELITE phenolic resin BKR-2620, and phenolic resin-baking solutions BKS-2600 and BKS-2710, have been used to increase the water and solvent resistance and raise the softening point of the vinyl butyral resins. Water resistance is improved with bakes at 275 deg. F., or above, using equal weight of phenolic and vinyl butyral resins. Flexibility is improved with increasing quantities of the vinyl butyral resin. Solvent resistance increases as the proportion of phenolic is increased.

Chemical agents that react with the hydroxyl group may be used to insolubilize vinyl butyral resins. Glyoxal, when added to a vinyl butyral resin solution, acts as a curing agent during the process of air drying to produce a solvent-resistant film. Since the reaction is reversible in the presence of water, the films are not suited for water immersion. Cupric ions, especially in the presence of ammonia, also insolubilize the vinyl butyral resins, as do diisocyanates when the solvents used do not react with the diisocyanates. Precautions must be taken in the handling of diisocyanates because of the toxicity of these materials.

COMPOUNDING

Vinyl butyral resins exhibit excellent pigment wetting properties. Glossy dispersions may be obtained from pebble mill grinds, whereas many other vinyl resins require intensive grinding to develop gloss. Moreover, rather large proportions of pigment may be dispersed in the resin without undue loss of film strength.

When it is desirable to utilize roll mills or other intensive mixers for grinding vinyl butyral resins, it is necessary to take precautions to prevent alteration of the resin. Intensive grinding increases the solubility of the resin in organic solvents, but at the same time increases the softening point of the resin to such an extent that only a short period of milling is feasible. Further milling makes the resin too stiff to handle. Plasticizing the resin with alcohol or even water minimizes the changes that occur during milling.

APPLICATIONS OF VINYL BUTYRAL RESINS

ADHESIVES

The excellent adhesive qualities of vinyl butyral resin, evidenced by its extensive use as a safety glass interlayer, have been instrumental in encouraging evaluation of these resins in strictly adhesive applications.

Vinyl butyral-phenolic resin adhesives may be used for bonding rubber, cork, asbestos board, wood, glass or ceramic parts, cloth, paper, or metals to plastics of the thermosetting type. After the bond has been cured, it will be stable to at least 212 deg. F. and will not be softened readily by the action of water or solvents. In order to bond two metal pieces together, the surfaces,

(Continued on page 9)

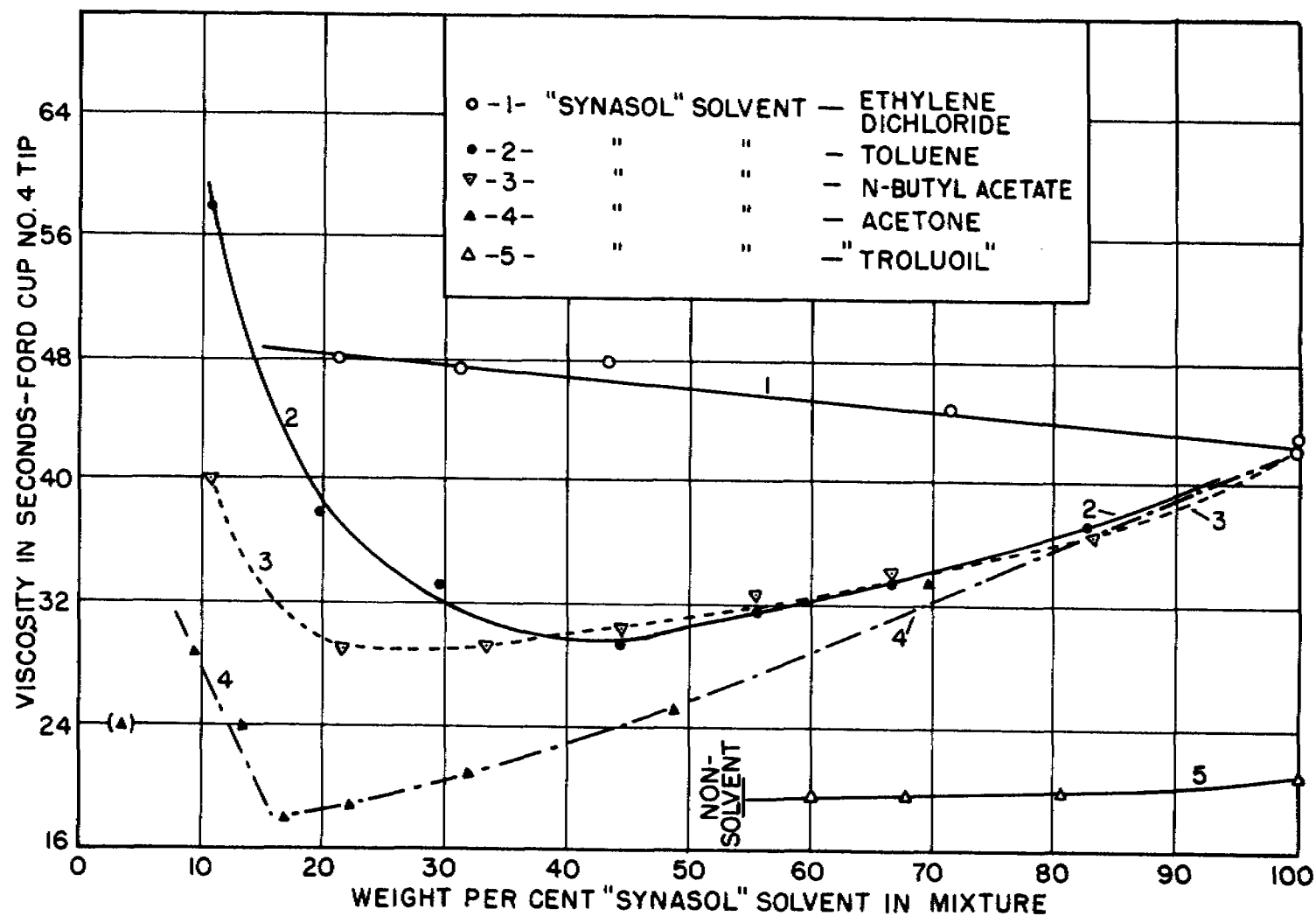


FIGURE 1. VISCOSITY OF 10 PER CENT* SOLUTIONS OF "BAKELITE"
VINYL BUTYRAL RESIN XYHL IN MIXED SOLVENTS

* "SYNASOL" Solvent "Troluol"-XYHL solution run at 7 per cent solids

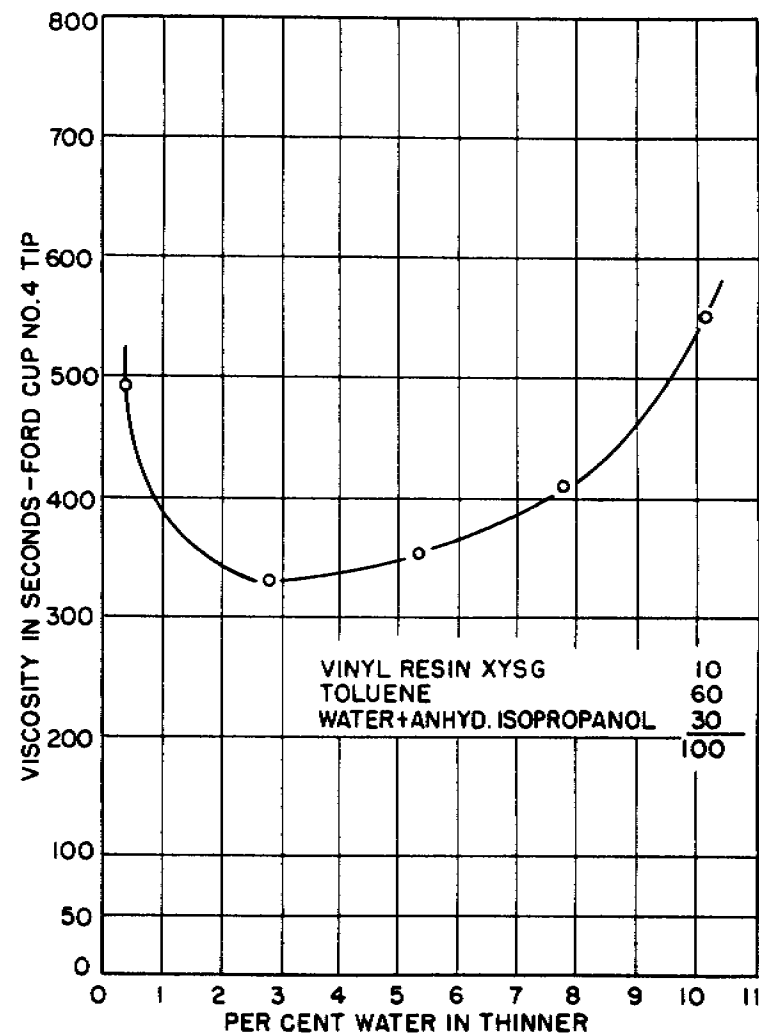
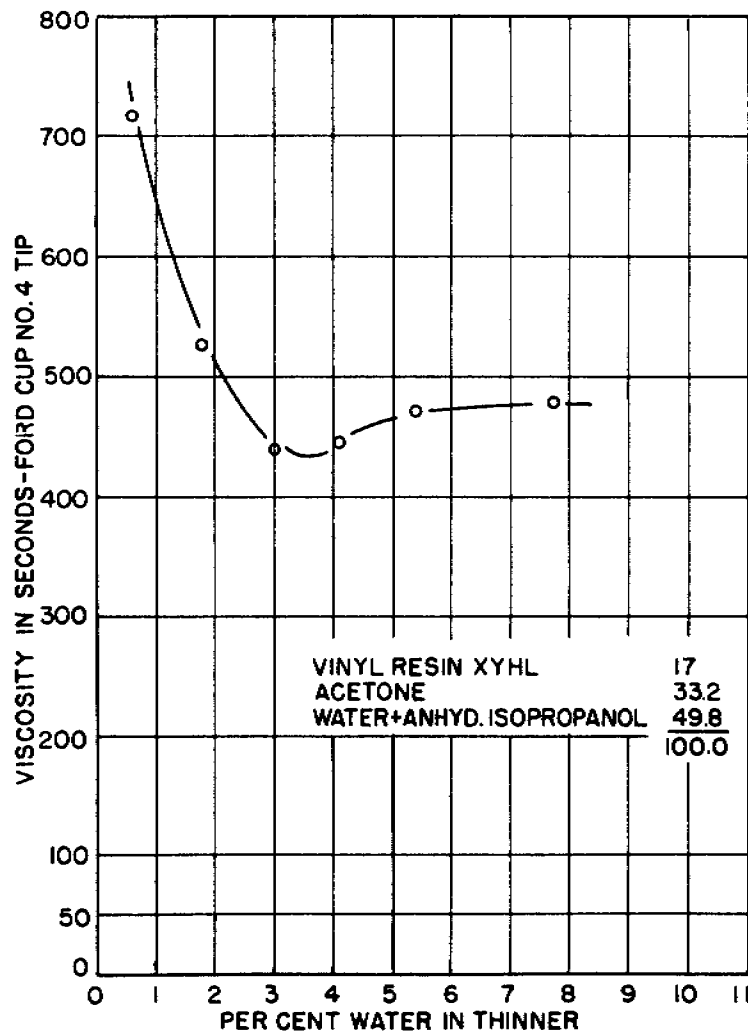


FIGURE 2. EFFECT OF WATER ON "BAKELITE" VINYL BUTYRAL RESIN SOLUTIONS

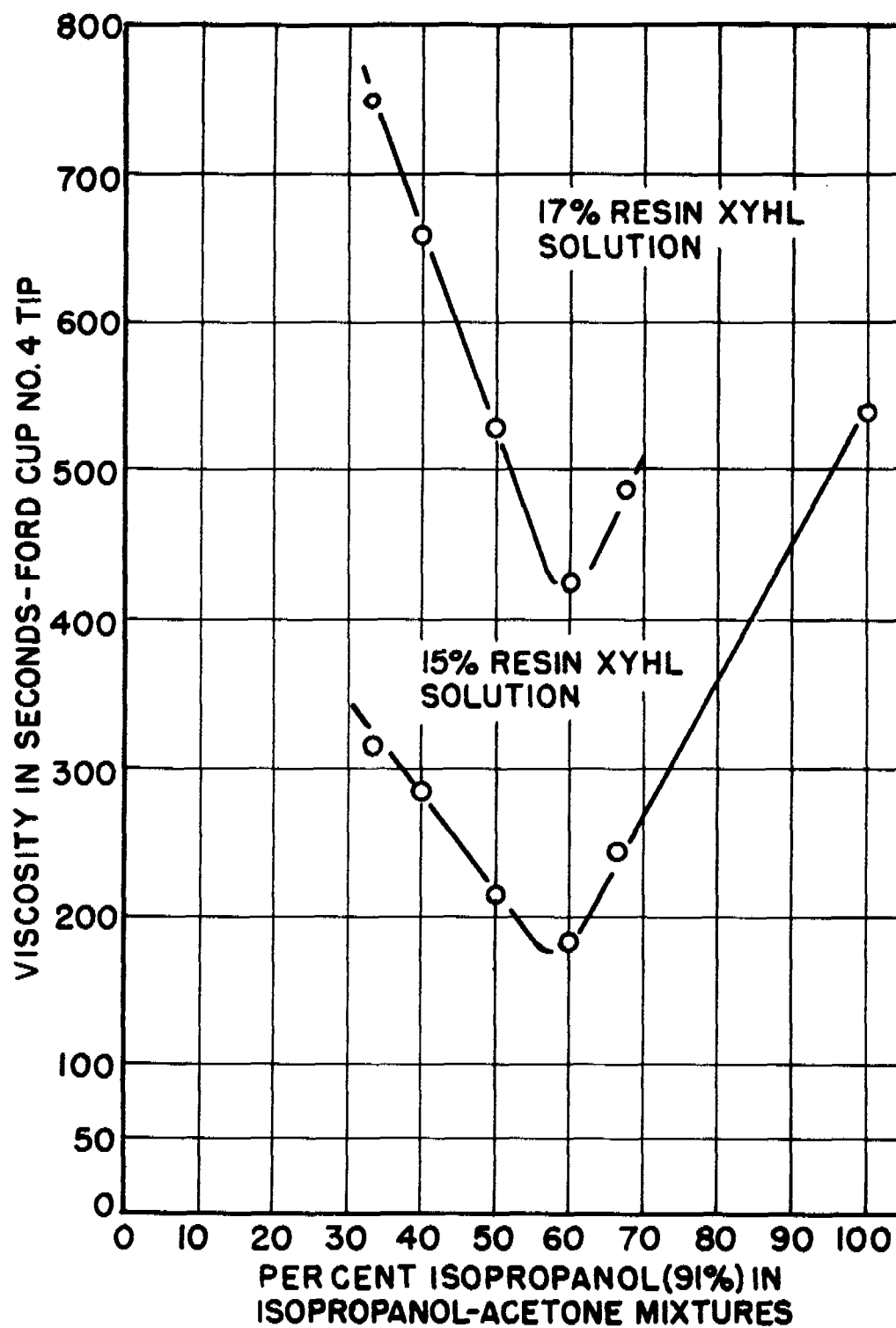


FIGURE 3. VISCOSITY MEASUREMENTS OF "BAKELITE" VINYL BUTYRAL RESIN XYHL IN ISOPROPANOL-ACETONE MIXTURES

TABLE IV
Compatibility of BAKELITE Vinyl Butyral Resin XYSG*

	Supplier (See Key)	Solvents	9/1	Weight Ratio, Resin XYSG To Test Substance				1/9	Remarks
				4/1	1/1	1/4			
COMMERCIAL SYNTHETIC RESINS									
"Acryloid" B-7 ¹	12	Isopropanol	SIC	SIC	SIC	SIC	SIC	Poor adhesion; soft	
"Acryloid" B-7 ²	12	Isopropanol	SIC	SIC	SIC	SIC	SIC		
"Acryloid" B-82	12	Isopropanol	C	C	C	C	C		
"Acryloid" C-10 ³	12	Isopropanol				IC	SIC	Short; adhesion fair	
"Acryloid" F-10 ⁴	12	Isopropanol	IC	IC					
"Amberol" No. 226	12	n-Butyl Alcohol	C	C	C	C	C		
BAKELITE Phenolic Resin CKR-2432	3	Isopropanol-Toluene	C	C	C	C	C	Films cheesy	
BAKELITE Phenolic Resin BKS-2710	3				C	C	C		
BAKELITE Phenolic Resin Baking Solution BKS-2600	3	Isopropanol	IC	IC	IC	IC	IC		
"Beckacite" 1001	11	Isopropanol-Toluene	C	C	SIC	SIC	SIC	1/1 to 1/9 hard, good adhesion; 9/1 to 4/1, hard and brittle	
"Beckacite" 1003	11	Isopropanol-Toluene	C	C	C	SIC	SIC	9/1 to 1/1 slightly tacky	
"Beckacite" 1110	11	Isopropanol-Toluene-Hexone	SIC	SIC	SIC	C	C		
"Beckacite" 1111	11	Isopropanol-Toluene	IC	IC	SIC	C	C		
"Beckosol" 1320	11	Isopropanol-Xylene	IC	IC	IC	IC	IC		
"Beetle" 212-9	1	Isopropanol	IC	IC	IC				
"Beetle" 216-8	1	Isopropanol	IC	IC	IC				
"Beetle" 227-8 ⁵	1		SIC	IC	IC	IC	IC		
Cellulose Acetate	—	Esters, Ketones — Alcohols	IC	IC	IC	IC	IC		
Cellulose Acetopropionate	—	Isopropanol-Acetone-Ethyl Dichloride	IC	IC	IC	IC	SIC		
Cellulose Acetobutyrate	—	Isopropanol-Acetone-Ethyl Dichloride	IC	IC	IC	IC	IC		
"Parlon" Chlorinated Rubber	8		IC	IC	IC	IC	IC		
"Cumar" Resin P-25	4	Isopropanol-Toluene	IC	IC	IC	IC	IC		
"Duraplex" C-45 LV	12	n-Butyl Alcohol	IC	IC	IC	IC	IC		
"Durez" No. 550	5	n-Butyl Alcohol	C	C	C	C	C		
"Synthe Copal" Ester Gum	11	Isopropanol-Toluene	SIC	IC	IC	IC	IC		
General Electric R-108	7	Isopropanol	C	C	C	C	C		
"Melmac" 245-8	1	Isopropanol	IC	IC	IC				
"Nevillac" Resin Hard	10	Isopropanol-Toluene	C	C	C	C	C	Little film strength; softness increased by resin, good adhesion	
"Neville" Resin R-17	10	Isopropanol-Toluene	IC	IC	IC	IC	IC		
"Nevindene" R-1	10	Isopropanol-Toluene	IC	IC	IC	IC	IC		
½" SS Nitrocellulose		n-Butyl acetate + MIBK + Ethanol	C	C	C	VSH	C		
½" RS Nitrocellulose		n-Butyl acetate + MIBK + Ethanol	IC	IC	IC	VSH	VSH		
"Lucrite" 44 n-Butyl Methacrylate Polymer	6	CELLOSOLVE Solvent	IC	IC	IC	IC	IC		
"Rezyl" No. 330-5	1	Isopropanol-Toulene-Xylene	IC	IC	IC	IC	IC		
"Rezyl" X-315	1	Isopropanol	SIC	SIC	SIC	SIC	SIC		
"Santolite" MS-80%	9	Isopropanol-Toluene	C	C	C	SIC	IC	9/1 to 1/1 hard, adherent, 1/4 soft	
"Teglac" No. 152	1	Isopropanol-Toluene	IC	IC	IC	IC	IC		
"Teglac" No. 161	1	CELLOSOLVE Solvent	IC	IC	IC	IC	IC		
"Uformite" F-240	12	Isopropanol	C	C	C	C	C		
"Varcum" No. 250	11	n-Butyl Alcohol	C	C	C	C	C		
¹ Solution in ethylene dichloride									
² Solution in toluene									
³ Solution in ethyl acetate									
⁴ Solution in mineral spirits									
⁵ Solution in xylene									

¹Solution in ethylene dichloride

²Solution in toluene

³Solution in ethyl acetate

⁴Solution in mineral spirits

⁵Solution in xylene

TABLE IV (Continued)
Compatibility of BAKELITE Vinyl Butyral Resin XYSG*

	Supplier (See Key)	Solvents	9/1	Weight Ratio, Resin XYSG To Test Substance				1/9	Remarks
NATURAL GUMS & RESINS†									
Accroides	—	Isopropanol	SIC	SIC	C	C	C	C	9/1 to 4/1 adhesion fair, 1/1 to 1/9 hard, brittle
Batavia Damar	—	Isopropanol-Toluene	IC	IC	IC	IC	IC	IC	
Boea-Bold	—	Isopropanol-Toluene	C	C	C	C	C	C	9/1 to 1/1 hard, short, good adhesion; 1/4 to 1/9 soft
Congo Hard	—	Isopropanol Hexone	C	C	C	C	C	C	9/1 to 4/1 hard, adhesion poor. 1/1 fair adhesion. 1/4 to 1/9 short
Damar (dewaxes)	—	Ethanol-Isopropanol-Toluene	C	C	C	C	C	C	9/1 to 4/1 good film strength; 1/1 to 1/9 brittle
Damar (Singapore)	—	Isopropanol-Toluene	SIC	SIC	SIC	SIC	SIC	SIC	9/1 to 1/1 hard, adhesion fair; 1/4 to 1/9 soft
Damar (P.E.I. Macassar)	—	Isopropanol-Toluene	IC	IC	IC	IC	IC	IC	
Elemi	—	Isopropanol-Toluene	C	C	C	C	C	C	9/1 strong film, poor adhesion. 4/1 to 1/9 short
Manila	—	Isopropanol-CELLOSOLVE	C	C	C	C	C	C	Hard, brittle, adhesion fair
Manila Lobe	—	Isopropanol CELLOSOLVE	C	C	C	C	C	C	Hard, brittle, adhesion fair
Mastic	—	Isopropanol	C	C	C	C	C	C	1/1 to 1/9 short, good adhesion 9/1 to 4/1 strong, hard, poor adhesion
Pontianak	—	Isopropanol-Hexone	C	C	C	C	C	C	9/1 to 4/1 hard, poor adhesion 1/1 to 1/9 short
Sandarac	—	Isopropanol	C	C	C	C	C	C	9/1 to 1/4 hard, good adhesion. 1/9 very short

Resin XYSG with Waxes and Oils

No. 100 Linseed Oil	2	Isopropanol-Toluene	C	C	IC				
OKO-M-7 Linseed Oil	2	Isopropanol-Toluene	IC	IC	IC	IC	IC		
Pale Blown Castor Oil		Isopropanol-Toluene	C	C	C	C	C	C	Addition of oil causes softness
Raw Castor Oil AA	—	Isopropanol-Toluene	C	C	C	C	C	C	Oil causes softness
Refined Perilla Oil	—	Isopropanol-Toluene	IC	IC	IC	IC	IC	IC	
Tung Oil	—	Isopropanol-Toluene-Hexone	IC	IC	IC	IC	IC	IC	
White Heavy Bodied Oil	2	Isopropanol-Toluene-Hexone	SIC	C	SIC	IC	C	C	1/9 soft, greasy

†Natural gums and resins are available through members of the American Gum Importers Association, Inc., 360 Furman Street, Brooklyn, N.Y. Names and addresses of members as well as complete technical data on these products may be obtained from the Association.

Legend: C — Compatible, SIC — Slightly Incompatible, IC — Incompatible, VSH — Film hazy in Tyndall beam.

*While these data were obtained with vinyl butyral resin XYSG, they apply with minor adjustment to vinyl butyral resin XYHL.

which should be grease-free, are coated and dried to remove solvent. Drying may be accomplished at room temperature or at a low bake not above a temperature of 250 deg. F. for about 5 minutes. Temperatures higher than 250 deg. F. cause excessive hardening of the adhesive and make subsequent bonding difficult. After the solvent has been completely removed, the surfaces are pressed together and heated. The time and temperature required will depend on the materials being bonded; from 15 minutes at 275 deg. F. to a few seconds at 400 deg. F. will suffice.

Adhesives based on vinyl butyral-phenolic resin combinations are also used in the lamination of plywood. The vinyl butyral resin acts as a "plasticizer" or fortifier for the phenolic resins, serving to increase the shock resistance, and to improve the adhesion of the phenolic to wood, especially under conditions of high humidity.

While the ratio of vinyl butyral resin to phenolic resin will vary for different applications, depending on the materials being bonded and the nature of the application, increasing the ratio of vinyl butyral resin will have the following general effects:

1. Increase the tensile strength.
2. Increase the impact strength.
3. Decrease creep resistance.
4. Decrease boiling water resistance.

Vinyl butyral resins can be used in hot melt form if mixed with compatible plasticizers and resins. Hydroabietyl alcohol and vinyl butyral resin XYHL in equal parts will make a melt that is pourable at 350 deg. F., yet remains tough and shock-resistant at room temperature. Hot melts based on vinyl butyral resins are used commercially for high-speed bookbinding.

WOOD FINISHES

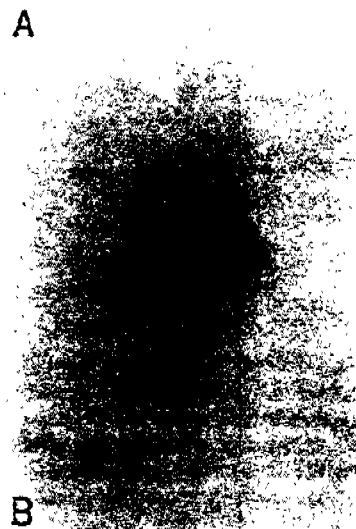
BAKELITE vinyl butyral resin XYHL has proven to be a valuable component of wood sealers and finishes. It imparts toughness to the film and enables the coating to maintain adhesion under a wide variety of conditions. When a vinyl butyral resin is used in a sealer under pyroxylin lacquers, the coatings are more resistant to marring and pressure marking as a consequence of the better adhesion to the wood.

Normally, the vinyl butyral resin is modified with phenolic resins, shellac, or nitrocellulose for wood coatings in order to improve the water resistance. Compositions with shellac have better durability than either resin alone on exterior exposure. Seemingly, the vinyl butyral resin reinforces the shellac, minimizing cracking and loss of adhesion. These same compositions are useful as sanding-sealers under pyroxylin and BAKELITE vinyl resin VAGH or VAGD lacquers.

The excellent adhesion and sealing qualities of the resin are illustrated by its use in knot sealers. Knots, even though they do not loosen, often exude pitch and volatile materials which destroy the adhesion of paint



Since economy grade lumber often contains knots, it is necessary to cover them to seal out their excessive pitch bleed which helps hasten failure of the finished paint surface. A formulation of phenolic-vinyl resins effectively seals a split knotted surface as shown by the lower half of the photo above which compares a coated and uncoated section of a wood panel. When used as prime coat, phenolic-vinyl based formulations effectively seal knotted surfaces thereby eliminating the need of any special preparations in order to protect the finished paint surface. (See below.)



The upper half of this panel was coated with a standard primer leaving the knot clearly visible. Two coats of house paint were then applied and the panel was exposed to a mercury vapor ultraviolet light source for 450 hours. Lower half of panel was coated with a primer based on a formulation of BAKELITE phenolic-vinyl resins which successfully prevented the knot from bleeding through the finished paint surface.

films, discolor them and make them brittle. This leads to cracking and peeling over the knot area long before the clear portion of the wood needs repainting.

After conducting extensive research to solve this problem and evaluating over 600 formulations, the Western Pine Association of Portland, Oregon, developed a knot sealer based on BAKELITE phenolic resin BKS-2710 and BAKELITE vinyl butyral resin XYHL. This coating makes it possible to use "economy" lumber for exterior use without sacrifice in appearance and durability.

The sealer is brushed over the unprimed knots and the surrounding area, after which regular outdoor house paint can be applied in the usual manner. Several years of weathering tests have shown this treatment to have outstanding merit.

The following formulation for knot sealer WP-578 (Union Carbide formulation VP-2756) meets the MIL-S-12935 (CE) specification:

WP-578	Parts by Weight	Per Cent by Weight (Approx.)
BAKELITE Phenolic Resin Varnish BKS-2710 (60 per cent N. V.)	5.0	33.3
BAKELITE Vinyl Butyral Resin XYHL	0.5	3.3
95 per cent Alcohol (denatured)	9.5	63.4
	15.0	100.0

Vinyl butyral resin XYHL is dissolved by adding it to the alcohol under agitation. The BKS-2710 resin is then added to the XYHL-alcohol solution with thorough agitation.

Compositions containing approximately 50 per cent vinyl butyral resin XYHL are useful as sealers over asphaltic materials to prevent bleeding into topcoats. They also serve as excellent sealers on porous woods such as redwood. These same coatings have given excellent service as clear outdoor finishes on wood, although they present some difficulty in applying at adequate film thicknesses because of the viscosity of the vinyl butyral resin solution.

CLOTH COATINGS

By combining vinyl butyral resins with heat-reactive ingredients or vulcanizing agents, extremely tough, resistant coatings for cloth can be formulated. These coatings, which can be applied with standard spreading equipment, have better adhesion to most fabrics than many other plastics. In some cases, the resultant adhesion exceeds the actual fabric strength. Coatings of this type, made with blends of vinyl butyral resin XYSG and phenolic resins such as BKS-2710, can be cured at 275 deg. F. to 300 deg. F. for thirty to sixty minutes to give films which are resistant to water, alcohol, acetone, and dry cleaning fluids. The vinyl butyral resin will comprise 50 to 70 per cent of the resin solids in a typical formulation. Blends of FLEXOL Plasticizer 3GH

and castor oil are suitable plasticizers. These coatings have been used as waterproof cloth coatings to replace rubberized fabrics in such applications as raincoats, ponchos, and inflatable equipment. This type of coating may be cemented and cured after assembly in much the same way as rubber products are vulcanized after fabrication. Vinyl butyral resin base cloth coatings are characterized by excellent resistance to blocking at elevated temperatures, including conditions of steam sterilization, yet they do not crack when bent sharply at 0 deg. F. For coatings subjected to lower temperatures, plasticized vinyl chloride coatings are to be preferred.

Cloth coatings of excellent color and color stability, good washability, and freedom from staining can be prepared from plasticized vinyl butyral resins by curing the resin with various urea or melamine formaldehyde resins.

METAL FINISHES

Vinyl butyral resins find application in a number of specialty finishes. One-half to two per cent of vinyl butyral resins XYHL or XYSG are used to effectively control the "eyeing" of phenolic resin baking solutions applied over imperfectly cleaned surfaces. In larger proportions, vinyl butyral resin XYHL can be used to improve the flexibility of phenolic resin-baking finishes such as BKS-2600 and BKR-2620. Vinyl butyral resin XYHL will range from 10 to 100 per cent of the weight of the phenolic resin. Increasing the butyral resin content increases the haze and decreases the chemical resistance of the films but makes possible the formulation of some excellent specialty coatings. For example, a coating based on one part of BAKELITE vinyl resin XYHL and two parts of BAKELITE phenolic resin-baking solution BKS-2600 will withstand commercial can forming operations at thicknesses up to 0.0003 and has extremely good resistance to steam processing.

Vinyl resin XYHL can be used in clear finishes for exterior exposure when modified with a urea-formaldehyde resin. For example, a 3:1:1 ratio of resin XYHL, FLEXOL 3GH and "Uformite" resin F-240 has given outstanding service as a clear coating on chromium plate. Ethanol/toluene or methyl ethyl ketone/xylene/n-butyl alcohol mixtures have proven satisfactory for thinning. Both air-dry coatings and those baked for ten minutes at 350 deg. F. were satisfactory after 1300 hours in an accelerated weathering unit and after 103 days of roof exposure. At a slight sacrifice in performance, a higher solids coating can be obtained using equal proportions of the two resins.

Air-dry coatings with resistance to a wide variety of solvents can be formulated from vinyl resin XYHL by the addition of 10 per cent glyoxal, based on the resin weight. Phenolic resins such as BKS-2710 may be added to improve water resistance, and rust-inhibitive pigments added to prevent underfilm corrosion.

METAL CONDITIONERS

The most important application of vinyl butyral resins in the coatings field is in the production of metal conditioners, or pretreatments, more widely known as wash primers. These wash primers are solutions of BAKELITE vinyl resin XYHL, including XYLS-2, containing rust inhibitors. They are applied as thin coatings of between .0001 and .0005 inches and merely require air-drying to establish a firm bond to metal surfaces. This inhibitive film serves to prevent corrosion and undercutting of the film by rust as well as providing a firm anchor for subsequent paint coats.

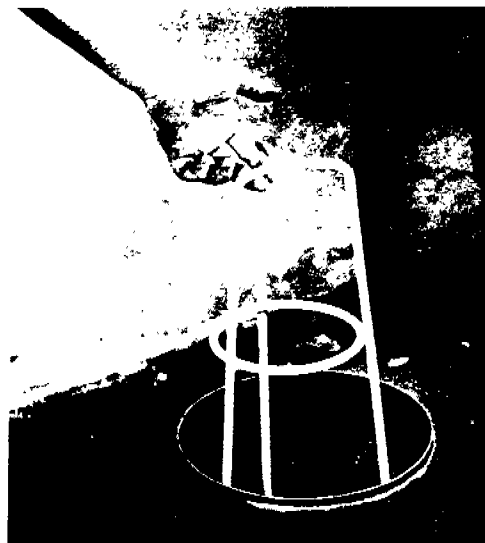
Wash primers do not replace conventional primers and shopcoats due to the thickness customarily applied, but must be classed with industrial phosphate metal treatments. They provide a temporary protection against atmospheric rusting, and must be protected by topcoats before exposure to the weather. These topcoats can be applied within fifteen minutes of the application of the wash primer since the vinyl butyral resin solution dries entirely by solvent evaporation. If desired, this drying can be speeded by heat.

The fact that wash primers can be applied with conventional spray equipment eliminates the need for expensive tanks, ovens, and technical control which are required for conventional metal pretreatments. Moreover, the simplicity of operation permits one to prepare metal surfaces and apply a coating in the field that is at least equivalent to a shop-applied system.

Phenolic, oleoresinous, and alkyd paints adhere to the wash-primed metal. Coatings based on BAKELITE vinyl resin VAGH or VAGD develop excellent adhesion. Most nitrocellulose lacquers adhere to wash primers as a result of modifiers such as alkyds contained in the lacquer. Nitrocellulose, itself, has relatively little adhesion to the wash primer film. In general, coatings based on vinyl chloride copolymers (except vinyl resin VAGH or VAGD), vinylidene chloride, and acrylic and methacrylic resins do not adhere to the wash primers. Just as alkyd resins improve the adhesion of nitrocellulose lacquers to wash primers, suitable blends of vinyl resin VAGH or VAGD with other vinyl resins will develop the required adhesion.

USE OF METAL CONDITIONERS

Metal conditioners, or wash primers, were developed for use in the marine field and a complete vinyl resin system based on these metal conditioners has consistently outperformed all others in laboratory and actual service tests. A steel vessel, treated with the wash primer, overcoated with an anti-corrosive primer based on BAKELITE vinyl resin VAGH or VAGD, and covered with an anti-fouling coat based on BAKELITE vinyl resin VYHH, has been protected for over seven years without repainting. The wash primer holds the paint system firmly to the steel and prevents the spread of underfilm



In mass production systems, time saved can be measured in profit performance. By dipping this stand into a wash primer based on vinyl resins, 2½ to 3½ minutes of labor per stand was saved in production time. As a binding material, wash primers perform double duty by adhering to metals much better than the average coating and providing a better adhesive base for the finished top coat.

corrosion despite occasional mechanical breaks in the film. These areas can be easily cleaned and touched up without damage to the surrounding finish.

Multiple coats of this all-vinyl system can be applied quickly, with resultant economies in dry-dock service. In addition, the protective life of the system leads to greater savings since less frequent painting is necessary.

The outstanding results in the marine field have been duplicated in other fields. Pipe lines, oil storage tanks, and offshore drilling rigs are protected with the wash primer-vinyl paint system. The speed of application and abrasion resistance of this system have led to many applications on bridges, dam gates and barges. Air conditioning equipment, particularly fans where the corrosion on the edge of blades is severe, is often protected by the wash primer system.

Aluminum sheets are prime-coated in the flat with wash primer and then formed into house siding. The primed surface is much more receptive to paint than untreated aluminum and the paint adheres tenaciously. Aluminum foil treated with wash primers is easier to print, and adhesives bond to it more firmly. The adhesion of coatings to aluminum aircraft is improved by wash primers.

Wash primers are used widely to coat galvanized iron, a surface which is usually difficult to paint. Exterior signs and outdoor movie screens which are wash-primed retain their paint despite cold weather shock instead of having to be repainted each spring as has been customary.

APPLICATION OF METAL CONDITIONERS

The proper application techniques play an important role in the eventual performance of paint systems. This is particularly true of the more resistant systems such as the vinyl types. To establish adhesion to a metal surface and to prevent rusting, the metal pretreatment must first be in contact with the metal. Grease, oils and other contaminants should be removed from the surface before applying the wash primer, as should any mill scale and rust present. Grit or sandblasting has proven more effective than wire brushing or flame descaling in providing an acceptable surface for the wash primer. Acid washes, such as those based on acetic acid which are frequently suggested as a pretreatment for galvanized iron, should not be used since salts left on the surface may be a source of osmotic blisters.

The performance of wash primers over rusty steel is always poorer than over clean steel. Moreover, on a rusty surface, the results are often erratic. When a wash primer is applied to a surface covered with mill scale, it is possible that the galvanic action between the steel and the mill scale can be accelerated leading to more rapid failure. This latter effect is more often observed in underwater exposures. The economies possible by less frequent painting can only be realized by avoiding the

erratic and inferior performance resulting from improperly prepared surfaces.

Wash primers may be applied by conventional methods such as spraying, dipping, brushing, or roller coating. It is essential, however, that they be applied as a wet coat in order to cover the surface and penetrate cavities and corrosion pits. Since a heavy coating of wash primer may detract from the performance, the primer should be diluted with additional alcohol. This achieves the desired fluidity without applying a dry film thicker than the preferred maximum of approximately .0005 inch. Precautions should be taken to avoid conventional paint thinners for this dilution, since even small amounts of high boiling aliphatic hydrocarbons may precipitate the butyral resin during drying and ruin performance.

Although not good practice, the wash primers may be applied over wet metal. In this case it is essential that the thinner contain adequate n-butyl alcohol to ensure against resin precipitation. Usually thirty per cent n-butyl alcohol in the thinner will give good performance. On wet or rough surfaces, brush application is preferred since this ensures that the wash primer penetrates any pits in the surface.

PREPARATION AND STORAGE OF METAL CONDITIONERS

Pigmented wash primers are made in conventional paint equipment, precautions being taken to prevent contamination by incompatible ingredients. The vinyl butyral resin is dissolved in the solvents, care being taken that the resin is added slowly to prevent the formation of large resin clumps. The pigment base should be prepared from the base solution by dispersing the pigment in a pebble mill, grinding to a Hegman gauge reading of 7. Steel mills and equipment should be avoided since the alcohol solvents used often lead to rapid rusting, and the iron contamination which is introduced into the wash primer detracts from the adhesion. For the same reason, the containers used for storage should be lined with a phenolic resin-baking or vinyl resin coating or should be of a non-reactive metal such as terneplate.

The acid diluent should be packaged in glass or polyethylene containers or in containers having a suitable baked phenolic or vinyl resin coating. The same precautions are required if the diluent and base are combined in a one-package wash primer.

Table V lists suggested pigment grinding bases and diluent compositions for several representative wash primers.

CHARACTERISTICS AND SELECTION OF METAL CONDITIONERS

The original wash primer, WP-1, was developed by Union Carbide's Plastics Division laboratories as part of a government contract to improve shipbottom paints.



Photo reveals two sections of a rear view of a ship's rudder. Left side was coated with one of the more efficient systems used during World War II. The right side of the rudder was treated with a vinyl resin base system. Note the difference in the two sides after two years of continuous service. Also consider the maintenance benefits involved for commercial and naval shipping. The side coated with vinyl base resins is virtually free of marine growth and corrosion. It could easily be re-coated with minor effort. However, the left side of the rudder requires a complete and more costly refinishing job.

Three types of WP-1 are now available. Two compositions are formulated with the dry XYHL resin. They differ in their specifications using isopropanol or ethanol as the primary solvent. The third composition is based on XYLS-2 (XYHL resin already in solution). All WP-1 type wash primers are two-component primers which must be used within eight hours after mixing, otherwise the adhesion of the subsequent coat may be impaired. The inconveniences attendant upon these limitations sparked an intensive research program to develop improved formulae. A number of modifications have been developed as shown in Table V. The characteristics of these various wash primers are discussed below.

Basic Zinc Chromate Metal Conditioners, WP-1 Primers

These are the most widely used wash primers, and they have been subjected to the most extensive testing.

WP-1 primers (both MIL-C-15328A and MIL-C-15328B) have been found to give outstanding adhesion to the widest number of surfaces such as:

steel	cadmium	galvanized iron
stainless steel	tin	magnesium
zinc	aluminum	glass

WP-1 formulations are the preferred primers for use where exposure to salt water or salt spray is involved. These metal conditioners give maximum protection where the topcoat is a relatively permeable type such as alkyd, oleoresinous, or emulsion paint.

For outstanding performance, the WP-1 wash primers should be topcoated with a coating based on BAKELITE vinyl resin VAGH or VAGD. This is an especially effective system in sea water and corrosive atmospheres. On fresh water immersion, or under conditions of high humidity, this system may develop osmotic blisters when the temperature is above 100 deg. F. This occurs when

TABLE V
Composition of Wash Primers

	VP-2757 WP-1 (MIL-C-15328A)	VP-2983 WP-1 (MIL-C-15328A)	VP-2980 WP-1 (MIL-C-15328B)	VP-2849 Phenolic Modified	VP-2894 Lead Chromate Modified	UF-2333* (MIL-C-14505)	VP-2979* XE-5298 Chromic Phosphate Modified
	Parts by Weight	Parts by Weight	Parts by Weight	Parts by Weight	Parts by Weight	Parts by Weight	Parts by Weight
Base Grind							
BAKELITE Vinyl Butyral Resin, XYHL	7.2	—	7.35	10.39	9.0	10.30	9.0
BAKELITE Vinyl Butyral Solution, XYLS-2	—	36.0	—	—	—	—	—
BAKELITE Phenolic Resin, BKS-2710	—	—	—	15.58	—	—	—
Basic Zinc Chromate Pigment ¹ (Insoluble)	6.9	6.9	7.08	3.46	—	5.14	—
Lead Chromate Pigment (low in soluble salts)	—	—	—	—	8.6	—	—
Chromic Phosphate Pigment	—	—	—	—	—	—	9.0
Strontium Chromate Pigment	—	—	—	—	—	5.14	—
Precipitated Red Iron Oxide	—	—	—	0.86	—	—	—
Magnesium Silicate (Talc)	1.0	1.0	1.04	2.16	1.4	1.60	1.4
Ethanol (anhydrous SYNASOL solvent)	—	—	—	—	—	54.00	54.5
Lampblack	0.1	0.1	0.08	—	—	—	—
Isopropanol, 99 per cent	—	—	46.25	—	53.0	18.60	—
Ethyl Alcohol, 95 per cent	48.8	19.9	—	34.20	—	—	—
Xylol	—	—	—	7.80	—	—	—
Butanol	16.1	16.1	16.38	7.80	—	—	—
Water	—	—	2.00	—	—	—	—
Toluene	—	—	—	10.39	—	—	—
Methyl Isobutyl Ketone	—	—	—	—	13.0	—	16.1
	80.1	80.0	80.18	92.64	85.0	94.78	90.0
Acid Diluent							
Phosphoric Acid, 85 per cent	3.6	3.6	3.66	1.84	2.9	3.15	1.8
Water	3.2	3.2	3.28	—	2.9	2.07	1.8
Isopropanol, 99 per cent	—	—	12.88	—	9.2	—	—
Ethyl Alcohol, 95 per cent	13.1	13.2	—	5.52	—	—	—
Ethanol (anhydrous SYNASOL solvent)	—	—	—	—	—	—	6.4
	19.9	20.0	19.82	7.36	15.0	5.22	10.0
	100.0	100.0	100.0	100.0	100.0	100.0	100.0

*One package wash primer. The acid diluent can be added to the grind without affecting the shelf life of the primer.

The other wash primers, mentioned in Table V, are two-part primers. The base grind and the acid diluent have to be stored separately. After mixing, they should be used within a short time, since there is a gradual decline in the adhesion of films applied after the mixed primer has aged for several hours.

¹Suppliers: X-2913, Kentucky Color Co.; J-1345, Mineral Pigments Corp.; X-2259, Imperial Paper & Color Department, Hercules Powder Co.

the topcoat has relatively little ionic permeability as is the case with coatings based on vinyl and phenolic resins. Pigmentation of the topcoats with leafing aluminum pigments may minimize the osmotic blisters. In general, however, where higher temperatures and humidity are encountered, it is advisable to use one of the wash primers containing fewer soluble ions.

The rust inhibitive action of WP-1 primers is enhanced as the quality of the pigment dispersion is improved. This improved dispersion is best obtained by extended grinding rather than by the use of grinding aids, since most surface-active agents detract from the protection afforded by the wash primer.

In the mixing of WP-1 primers, add the diluent to the base slowly, with agitation, to prevent local gelation as a result of a momentary unbalance of the thinner. The mixing should be done in clean containers. Contamination by oils or high boiling naphthas, such as mineral spirits, is detrimental to performance.

WP-1 should be used within eight hours after mixing. After this time any remaining primer should be discarded, since there is a gradual decline in the adhesion of films applied after the mixed primer has aged about eight hours. *Note: This is not accompanied by any visible change in the solution.* Eventually the mixed material may gel, but the usefulness of WP-1 is spent long before this occurs.

The proportion of acid diluent is an important factor in the performance of wash primers. While a variation of 10 to 20 per cent in the volume of acid diluent does not bring about noticeable changes in the performance of the wash primer, larger variations should be avoided. *Users therefore should be cautioned not to add extra acid diluent to reduce the viscosity.* If further reduction in viscosity is required, isopropanol or n-butyl alcohol should be used. On certain surfaces, such as clean magnesium, it may be preferable to reduce the proportion of acid diluent by 75 per cent, but the necessity for this should be established by experiment.

Phenolic Modified Wash Primer

The phenolic modified wash primer VP-2849 was developed to improve the durability of the wash primer itself. In this way, the time of recoating of the wash primer is not critical. Exposure tests show that the phenolic wash primer itself will protect steel from corrosion in atmospheric conditions in excess of two years. Three year atmospheric exposures of the phenolic wash primer topcoated with a vinyl chloride based system shows comparable performance to that of the conventional WP-1 types wash primer previously discussed.

This type wash primer is suggested as a replacement for WP-1 where the wash primer system will not be topcoated within three months.

Lead Chromate Metal Conditioner

The lead chromate wash primer VP-2894 was developed specifically for use on steel and may not develop as good adhesion as WP-1 on other metal surfaces. VP-2894 is suggested as a replacement for WP-1 primers where the coating system is subjected to fresh water immersion or condensing moisture. Formula VP-2894 has the additional advantage that the acid diluent may be mixed with the base and stored for a long period without loss of ability to adhere. Storage tests of eighteen months have not revealed any deterioration in the primer. This means that a one-package primer can be supplied and the material can be used in dip tanks. Since the composition is designed for storage, VP-2894 is prepared at a somewhat higher solids content and, as formulated, may be too viscous for spraying. Thinning 20 to 30 per cent with n-butyl alcohol will reduce the composition to spray viscosity.

The easier dispersibility of lead chromate usually results in a smoother primer than can be obtained with basic zinc chromate. This is advantageous when gloss coatings are applied over the primer. On the other hand, when visual control of the thickness is used, there is a tendency to apply too heavy a coating.

Several precautions are suggested in the use of the VP-2894 primer. For best adhesion, vinyl resin VAGH or VAGD coatings applied over the VP-2894 primer should not be pigmented with lead pigments. Topcoats based on red lead, chrome yellow and chrome orange have shown poor adhesion, especially if the wash primer is aged some time before coating. Moreover, the VP-2894 primers have given some indication of poorer adhesion over wet surfaces than WP-1 primers, especially when heavy films are applied. Additional thinning with n-butyl alcohol before application, or warming during drying, usually eliminates these difficulties. Finally, the VP-2894 primers are somewhat slower in developing adhesion; although on steel the final adhesion is equal to that of WP-1 primers.

To sum up, VP-2894 is formulated for use on steel. VP-2894 is definitely poorer than WP-1 primers on aluminum and magnesium and the performance of this wash primer should be checked on other metal surfaces before use.

Chromic Phosphate Metal Conditioner VP-2979 or XE-5298

Union Carbide's Plastics Division laboratories have shown that salt-free chromic phosphate serves as an excellent rust inhibitor in vinyl butyral solutions. Wash primers based on chromic phosphate may be combined with the acid diluent as a one-package system which shows no evidence of loss of ability to adhere after many

months of storage. These chromic phosphate wash primers show less tendency to develop osmotic blisters than the WP-1 primers, basic zinc chromate formulations. For this reason, they are to be preferred in locations involving high humidity or fresh water immersion.

Formula VP-2979 or XE-5298 is shown in Table V (page 13) and is suggested for use on steel. While this formula has not been tested as extensively as the WP-1 formulations, it gives excellent performance in all types of accelerated tests, and is offered as a starting point for the development of improved single-package primers.

Like the VP-2894 formula, this chromic phosphate wash primer is formulated with higher resin and lower phosphoric acid content. Thus the formulation as written is more viscous and may require additional thinning with alcohol for spray applications. Since chromic phosphate pigments are prone to settle in the container, the composition should not be stored at too low a viscosity.

KEY TO SUPPLIERS FOR TABLE IV

1. American Cyanamid Company, Wayne, N.J.
2. Archer Daniels Midland Company, Minneapolis 40, Minn.
3. Union Carbide Corporation, Plastics Division, New York 17, N.Y.
4. Allied Chemical Corporation, Barrett Division, New York 6, N.Y.
5. Durez Plastics Div., Hooker Chemical Corp., North Tonawanda, N.Y.
6. E. I. du Pont de Nemours & Co., Inc., Wilmington 98, Del.
7. General Electric Company, Chemical Materials Dept., Pittsfield, Mass.
8. Hercules Powder Company, Wilmington 99, Del.
9. Monsanto Chemical Company, St. Louis 68, Mo.
10. Neville Chemical Company, Pittsburgh 25, Pa.
11. Reichhold Chemicals, Inc., White Plains, N. Y.
12. Rohm & Hass Company, Philadelphia 5, Pa.

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**UNION CARBIDE CORPORATION
PLASTICS DIVISION**

270 PARK AVENUE, NEW YORK, N.Y. 10017

SALES OFFICES

Atlanta, Georgia 30309	1371 Peachtree St., N.E.	(404) 873-4961
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St. Louis, Missouri 63122	122 North Kirkwood Road	(314) Yorktown 5-2440
San Francisco, California 94106	22 Battery Street	(415) Yukon 2-1360

IN CANADA

Union Carbide Canada Limited

123 Eglinton Avenue, East
Toronto 12, Canada

Outside United States and Canada

Plastics Department

Union Carbide International Company

Division of Union Carbide Corporation
270 Park Avenue, New York, N.Y., U.S.A. 10017

Cable Address: BAKELITE, New York