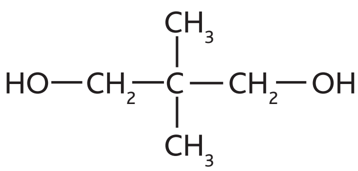
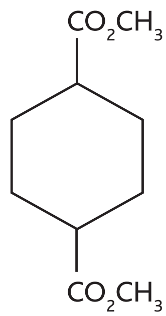
Polyester/polyurethane enamels
Air-dry and baking types
Eastman NPG™ glycol and Eastman™ DMCD (a diester)



Eastman NPG™ glycol



Eastman™ DMCD

Features

- Excellent gloss and depth of finish
- High impact and chip resistance
- Outstanding mechanical properties
- Good corrosion resistance
- Good exterior durability
- Utility in air-dry and baking enamels
- Versatility

Synthesis and properties—Resin UP-17-1ND

Composition

Components[a]	Equivalents	Moles	Grams
Stage 1			
Eastman NPG™ glycol	6.25	3.12	325
Trimethylolpropane	2.08	0.69	93
Eastman™ DMCD (dimethyl 1,4-cyclohexanedicarboxylate)	2.95	1.47	295
Catalyst: Fascat™ 4100 butylstannoic acid			0.3
Glycol stabilizer: *p*-toluenesulfonic acid			1
Stage 2			
Eastman™ IPA	4.42	2.21	367
Trimethylolpropane	2.08	0.69	93
		Total charge	–1174.3
		Calculated methanol loss	–94
		Calculated water loss at acid number = 0	–79
		Theoretical yield	1001.3

[a]*See raw material suppliers list on page 7.*

Synthesis procedure

1. Purge the reaction vessel with nitrogen and regulate the flow to maintain <1% oxygen throughout the cook. Charge the first-stage ingredients plus excess glycol to replace typical losses with the equipment used, catalyst, and stabilizer. Begin up-heat.
2. Increase temperature to a maximum of 190°C (374°F) and hold for 1–2 hours. Increase temperature to 220°C (428°F) and hold until first-stage reaction stops with approximately 95% of the theoretical methanol, 90 grams (112 mL), collected.
3. Cool to 150°C (302°F) and add second-stage ingredients. Increase temperature to 220°C (428°F) and hold to an acid number of approximately 30 (mg KOH/g resin).
4. Begin solvent cook by adding sufficient toluene to maintain reflux while maintaining temperature at 220°C (428°F). Hold until an acid number of <2 (mg KOH/g resin) and a cone and plate viscosity of 8 ± 2 poise at 200°C (392°F) or a Gardner™ viscosity of Z_6-Z_7 at 75 wt% nonvolatiles in toluene is reached.
5. Cool to 130°C (266°F) and add solvent.

Typical resin properties UP-17-1ND

Acid number, mg KOH/g resin	<2
Hydroxyl number, mg KOH/g resin	165–185
Molecular weight (number average), calculated	1,200
Gardner-Holdt™ viscosity	Z_6-Z_7
Gardner™ color	1
Nonvolatiles, wt%	75
Solvent	Toluene
Density kg/L (lb/gal)	1.10 (9.15)
Appearance	Clear

Gardner™ viscosity of resin UP-17-1ND with various solvents

	Nonvolatile content of resin solution		
Solvent	75%	70%	65%
Toluene	Z_{6-7}	Z_3	Y
Xylene	Z_{7+}	Z_4	Z_2
Eastman™ PM acetate	Z_{7-8}	Z_5	Z_{3-}
Eastman™ *n*-butyl acetate	Z_{6-7}	Z_{3-}	X-Y
Eastman™ MIAK	Z_{7-}	Z_{3-4}	Y-Z
Eastman™ MIBK	Z_{6+}	Z_{3-}	X-Y
Eastman™ MAK	Z_{6-7}	Z_{3+}	X-Y

Formulation and physical properties of pigmented air-dry polyester/polyurethane enamel based on resin UP-17-1ND

Components[a]	Weight %
Part A	
UP-17-1ND (75% in toluene)	26.0
Ti-Pure™ R-900 TiO_2 pigment	21.5
Fluorosurfactant flow additive (20% in methyl *n*-propyl ketone [Eastman™ MPK])	0.3
Triethylenediamine (10% in Eastman™ PM acetate)	0.6
Eastman™ CAB-551-0.2 (30% in Eastman™ *n*-butyl acetate)	3.2
Eastman™ PM acetate	4.3
Eastman™ *n*-butyl acetate	18.5
	74.4
Part B	
Desmodur™ N-75 polyisocyanate resin	17.1
Eastman™ *n*-butyl acetate	8.5
	25.6
	Total A + B = 100.0

Enamel formulation properties		Value
NCO/OH ratio		1.1
Density, kg/L (lb/gal)		1.189 (9.926)
Theoretical % solids		54.8
Pigment/binder ratio		40/60
#4 Ford Cup viscosity, s		19
Pot life, h		~6
Stability, Brookfield™ viscosity, cP,	2 h	60
	4 h	80
	6 h	80

Enamel curing conditions
Cure: 1 week at ambient conditions

Cured film properties[b]	Value
Film thickness, mil	1.8–2.2
Gloss, 60°/20°	92/86
Pencil hardness, mar/cut	H/4H
Impact resistance, in.-lb, direct/reverse	160/140
MEK double rubs, solvent resistance	200+
Salt spray resistance after 2 weeks, creepage in inches	1/32
Conical mandrel flexibility, 3.2 mm (1/8 in.)	Pass
Crosshatch adhesion, % passed	100

[a]*See raw material suppliers list on page 7.*

[b]*Coatings were applied to 2-gauge cold-rolled steel with Bonderite™ 37 pretreatment.*

Formulation and physical properties of clear air-dry polyester/polyurethane enamel based on resin UP-17-1ND

Components[a]	Weight %
Part A	
UP-17-1ND (75% in toluene)	29.3
Fluorosurfactant flow additive (20% in Eastman™ MPK)	0.33
Triethylenediamine (10% in Eastman™ PM acetate)	0.67
Eastman™ CAB-551-0.2 (30% in Eastman™ *n*-butyl acetate)	3.6
Methyl ethyl ketone	16.2
Eastman™ PM acetate	4.8
Eastman™ *n*-butyl acetate	16.1
	71.0
Part B	
Desmodur™ N-75 polyisocyanate resin	19.3
Eastman™ *n*-butyl acetate	9.7
	29.0
	Total A + B = 100.0

Enamel data		Value
NCO/OH ratio		1.1
Density, kg/L (lb/gal)		0.970 (8.095)
Theoretical % solids		37.6
#4 Ford Cup viscosity, s		14
Pot life, h		~6
Stability, Brookfield viscosity, cP initial		25
	2 h	25
	4 h	30
	6 h	38

Enamel curing conditions
Cure: 1 week at ambient conditions

Cured film properties[b]	Value
Film thickness, mil	1.8–2.2
Gloss, 60°/20°	97/80
Pencil hardness, mar/cut	F/H
Impact resistance, in.-lb, direct/reverse	160/160
MEK double rubs, solvent resistance	200+
Conical mandrel flexibility, 3.2 mm (1/8 in.)	Pass
Crosshatch adhesion, % passed	100

[a]*See raw material suppliers list on page 7.*

[b]*Coatings were applied to 20-gauge cold-rolled steel with Bonderite™ 37 pretreatment.*

Automotive refinish or OEM metallic basecoat formulation

Components[a]	Weight %
Part A	
UP-17-1ND (75% in toluene)	9.0
Desmorapid™ PP catalyst (10% in Eastman™ ethyl acetate)	1.0
Eastman™ CAB-551-0.2 (30% in Eastman™ *n*-butyl acetate)	4.0
Epolene™ C-18 dispersion[b] (5% in xylene)	32.0
Sparkle Silver™ flake (32.5%)	6.0
Eastman™ *n*-butyl acetate	19.0
Eastman™ EEP solvent	5.0
Xylene	12.0
	88.0
Part B	
Desmodur™ N-75 resin	6.0
Toluene	3.0
Eastman™ EEP solvent	3.0
	12.0
	Total A + B = 100.0

[a]*See raw material suppliers list on page 7.*

[b]*See procedure for preparing dispersion.*

Procedure for preparing dispersion

1. Add the total amount of Epolene™ C-18 wax to one-half the volume of the desired solvent blend.
2. Heat the mixture under moderate agitation to 70°–80°C using steam coils or a jacketed tank until the Epolene™ C-18 wax is in solution.
3. Cool the clear solution to 3°–5° above its cloud point (65°–68°C) and quickly add the solution to the remaining chilled solvent using vigorous agitation.
4. Continue cooling under moderate agitation to about 30°C by using a cooling coil or by pumping chilled water through the jacketing.
5. Shake or mix the dispersion well before adding it to the final coating composition.
6. Stir in variously sized metal flakes to achieve the desired effects.

Automotive refinish topcoat formulation

Components[a]	Weight %
Part A	
UP-17-1ND (75% in toluene)	30.0
Eastman™ CAB-551-0.2 (30% in Eastman™ *n*-butyl acetate)	5.0
Eastman™ *n*-butyl acetate	23.0
Xylene	11.9
Eastman™ EEP	5.5
Desmorapid™ PP catalyst (10% in Eastman™ ethyl acetate)	2.3
Fluorosurfactant flow additive (20% in Eastman™ MPK)	0.3
	78.0
Part B	
Desmodur™ N-75 resin	16.0
Toluene	3.0
Eastman™ EEP	3.0
	22.0
	Total A + B = 100.0

[a]*See raw material suppliers list on page 7.*

Clear urethane baking enamel formulation

Components[a]	Weight %
UP-17-1ND resin (75% in toluene)	31.44
Aliphatic polyisocyanate resin	38.42
Dibutyltin dilaurate catalyst	0.26
Fluorosurfactant flow additive (20% in Eastman™ MPK)	0.52
Eastman™ *n*-butyl acetate	14.68
Xylene	14.68
	100.00

Enamel curing conditions	Value
NCO/OH ratio	1/1
Cure time at 138°C, min	30
Viscosity, cP	122
Nonvolatiles, wt%	58

Enamel was reduced to spray viscosity with solvent to obtain 53% nonvolatiles.

Cured film properties	Value
Impact resistance in.-lb, direct/reverse	160/160
Conical mandrel flexibility, 3.2 mm (1⁄8 in.)	Pass
Pencil hardness, mar	H

[a]*See raw material suppliers list on page 7.*

Weathering

A clear polyester urethane coating formulation was prepared, and various stabilizers were added to separate portions. Weatherability of this coating system was determined using Florida 5° south, Black Box exposure. The formulation, stabilizer systems, and gloss retention data are summarized in the following:

Clear polyester urethane coating formulation

Components[a]	Parts by weight
Part A	
UP-17-1ND (75% in toluene)	29.2
Eastman™ *n*-butyl acetate	16.2
Fluorosurfactant flow aid (10% in Eastman™ methyl *n*-propyl ketone)	0.3
Triethylenediamine (10% in Eastman™ PM acetate)	0.7
Eastman™ CAB-551-0.2 (30% in Eastman™ *n*-butyl acetate)	3.6
Methyl ethyl ketone	16.2
Eastman™ PM acetate	4.8
Tinuvin™ stabilizer package (shown in the page 7 table)	
Part B	
Desmodur™ N-75 polyisocyanate resin (75%)	19.3
Eastman™ *n*-butyl acetate	9.7
	Total A + B = 100.0

[a]*See raw material suppliers list on page 7.*

Stabilizer packages and gloss retention values after 5° south, Black Box Florida weathering of cured test panels[1]

	% Gloss retention			
	60° Gloss		20° Gloss	
Stabilizer system, %[a]	1 Year	18 Months	1 Year	18 Months
A. None	54	0	26	0
B. Tinuvin™ 292 HALS[b]	99	91	92	85
C. Tinuvin 328 UVA[c]	85	27	74	16
D. Tinuvin 292 HALS, 1% Tinuvin 328 UVA, 1%	98	93	91	87
E. Tinuvin 292 HALS, 1% Tinuvin 328 UVA, 1% Irganox™ 1010 antioxidant, 0.5%	96	94	91	86
F. Tinuvin 292 HALS, 1% Irganox 1010 antioxidant, 0.5%	99	93	86	85

[a]*Percent based on binder solids.*

[b]*HALS—Hindered Amine Light Stabilizer*

[c]*UVA—Ultraviolet Absorber*

The results of this study demonstrate that both ultraviolet stabilizers (UVAs) and hindered amine light stabilizers (HALS) are effective in providing increased exterior durability for this coating formulation; however, the HALS is most effective. Only a slight additional improvement can be gained by combining a HALS with either a UVA or an antioxidant.

Raw material suppliers

Eastman™ *n*-butyl acetate	Eastman
Eastman™ CAB-551-0.2	Eastman
Desmodur™ N-75 polyisocyanate resin	Bayer Material Science
Desmorapid™ PP catalyst	Bayer Material Science
Eastman™ DMCD	Eastman
Eastman™ EEP[a]	Eastman
Epolene™ C-18 wax	Westlake Chemical
Fascat™ 4100 butyl stannoic acid	Arkema
Fluorad™ FC 430 flow additive	3M
Irganox™ 1010 antioxidant	Ciba Specialty Chemicals
Eastman™ MIAK (methyl isoamyl ketone)	Eastman
Eastman™ MIBK (methyl isobutyl ketone)	Eastman
Eastman™ MAK (methyl *n*-amyl ketone)	Eastman
Eastman NPG™ glycol	Eastman
Eastman™ PM acetate[b]	Eastman
Sparkle Silver™ flake	Silberline
Ti-Pure™ R-900 TiO_2	DuPont
Tinuvin™ stabilizers	Ciba Specialty Chemicals

[a]*Ethyl 3-ethoxypropionate*

[b]*Propylene glycol monomethyl ether acetate*

[1]*Coatings were applied to zinc-phosphate treated steel panels and cured for 1 week at ambient conditions prior to initiation of the testing.*

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