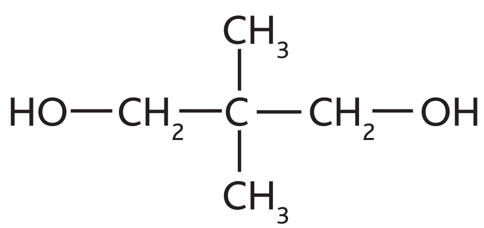
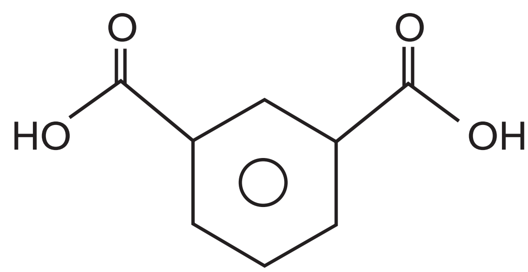
High-solids polyester resins for appliance and general metal coatings

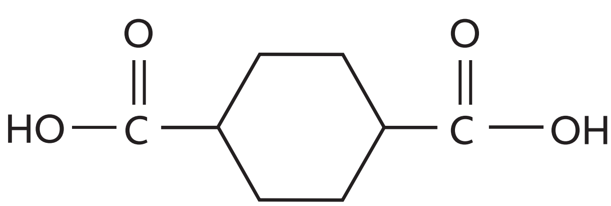
Based on Eastman NPG™ glycol; Eastman™ 1,4-CHDA; Eastman™ purified isophthalic acid (PIA); and Eastman™ purified terephthalic acid (PTA)



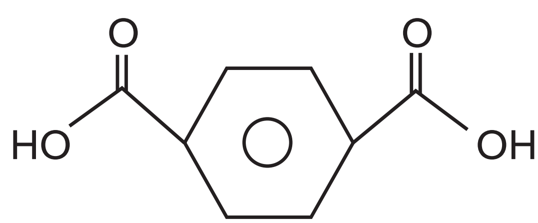
Eastman NPG glycol
2,2-dimethyl-1,3-propanediol
CAS 126-30-7



Eastman PIA
Purified isophthalic acid
CAS 121-91-5



Eastman 1,4-CHDA
1,4-cyclohexanedicarboxylic acid
CAS 1076-97-7



Eastman PTA
Purified terephthalic acid
CAS 100-21-0

Starting point formulations
HS-3-1NCp and HS-3-2NCp

Resin formulation

			HS-3-1NCp	HS-3-2NCp
Component	Equivalents	Moles	Grams	
Stage 1				
Eastman NPG™ glycol	14.85	7.42	773	773
Phthalic anhydride	4.03	2.02	299	–
Eastman™ purified isophthalic acid (PIA)	3.02	1.51	–	2.51
Eastman™ purified terephthalic acid (PTA)	1.01	0.50	–	84
Stage 2				
Trimethylolpropane	0.59	0.20	26	26
Eastman™ 1,4-CHDA	6.05	3.02	521	521
Stage 3				
Trimethylolpropane	0.59	0.20	26	26
		Total charge	1,645	1,681
		Theoretical distillate	–145	–181
		Theoretical yield	1,500	1,500

No catalyst or stabilizer
Nitrogen flow: adequate to maintain <1% oxygen

Processing procedure

Stage 1

- Charge all stage 1 reactants to a 2-L reaction kettle equipped with a heating mantle, agitator, nitrogen blanket, thermocouple, partial condenser, water trap, and total condenser.[1]
- Heat to 220°C (428°F) over 3 hours. Maintain at 220°C (428°F) until an acid number of 5 (mg KOH/g resin) or less is obtained, typically an additional 2 hours. The distillate at the end of this step will be approximately 34 mL for HS-3-1NCp and 68 mL for HS-3-2NCp.

Stage 2

- After cooling to 140°C (284°F) or less, add stage 2 reactants.
- Heat to 220°C (428°F) and maintain until one-half of the theoretical distillate is collected for this stage. The total distillate at the end of this step will be approximately 73 mL for HS-3-1NCp and 90 mL for HS-3-2NCp. This step normally requires 30 to 60 minutes.

Stage 3[2]

- After cooling to 140°C (284°F) or less, add third-stage TMP charge.
- Heat to 220°C (428°F) and maintain until an acid number of 10 ± 1 (mg KOH/g resin) is obtained. This step requires 2 hours. The total distillate at this step will be approximately 120 mL for HS-3-1NCp and 154 mL for HS-3-2NCp.
- After cooling to 140°C (284°F) or less, add solvent.

[1] *Refer to Eastman publication N-345, Processing polyester resins for coatings applications.*

[2] *It may be possible to two-stage these resins (combine the first and second stages), but this has not been investigated. Previous work in Eastman Technical Service and Development laboratories has shown that two-staging the TMP brancher is beneficial. More information on this subject is available in Eastman publication N-305, Polyester resin synthesis techniques for achieving lower VOC and improving coating performance.*

Resin properties

	HS-3-1NCp	HS-3-2NCp
Equivalents of OH/equivalents of COOH (R value)	1.59	1.59
Target acid number, mg KOH/g resin	10	10
Calculated hydroxyl number, mg KOH/g resin	222	222
Molecular weight, M_n[a]	840	960
Cone and plate viscosity @ 100°C, Pa·s (P)	0.57 (5.7)	0.78 (7.8)
Gardner-Holdt viscosity, 85% solids in xylene	Z_4	Z_5
Days to hazing at room temperature	>180	105

[a]*Molecular weight determined using GPC with a refractive index detector.*

Enamel formulation

Ingredient	Wt%
Polyester resin (85 wt% in xylene)	45.0
Cymel™ 303 melamine resin	12.0
Ti-Pure™ R-900 TiO_2	33.0
p-Toluenesulfonic acid catalyst (40 wt% in i-propanol)	0.3
Fluorosurfactant additive (20 wt% in Eastman™ EEP)	0.5
Solvent blend[a]	9.2
	100.0
Pigment:binder ratio	40:60
Polyester:melamine ratio	75:25
Additional solvent blend was added to each enamel to obtain #4 Ford Cup viscosity of 30 seconds.[a]	

[a]*Eastman™ MAK/Eastman™ EEP/*n*-butyl alcohol in a 70:15:15 ratio.*

Enamel properties

	HS-3-1NCp	HS-3-2NCp
Determined density, g/L (lb/gal)	1,356 (11.31)	1,344 (11.21)
Nonvolatiles, wt%		
Calculated	79	78
Determined		
20 min @ 177°C (350°F)	74.0	72.4
90 min @ 110°C (230°F)	76.6	75.2
Determined VOC,[a] wt VOC/vol coating		
20 min @ 177°C (350°F), g/L (lb/gal)	342 (2.85)	357 (2.98)
90 min @ 110°C (230°F), g/L (lb/gal)	316 (2.64)	334 (2.79)

[a]*VOC temperature conditions suggested in the May 25, 1988, issue of Chemical Week as EPA Reference Method 24.*

Cured film properties[a]

	HS-3-1NCp	HS-3-2NCp
Film thickness, mils (microns)	1.8 (45.7)	1.9 (48.3)
Gloss 60°/20°	91/ 80	91/ 80
Solvent resistance, MEK double rub[b]	200+	200+
Flexibility/hardness		
3.2-mm (1/8-in.) Conical mandrel flexibility, % pass	100	100
Impact resistance		
Direct, N·m (in.-lb)	13.1 (116)	13.1 (116)
Reverse, N·m (in.-lb)	5.0 (44)	6.8 (60)
Pencil hardness, mar	4H	4H
Stain resistance, covered/uncovered[c,d]		
Iodine after 30 min	4/4	3/4
Mustard after 24 h	5/5	5/5
Lipstick after 24 h	5/5	5/5
Ink after 24 h	5/5	4/5
Ketchup after 24 h	5/5	5/5
Grape juice after 24 h	5/5	5/5
Chemical resistance, covered/uncovered[c,d]		
50% NaOH solution after 8 h	5/5	5/5
50% Sulfuric acid solution after 8 h	2/2	2/2
Moisture resistance		
Cleveland humidity after 1,000 h @ 60°C (140°F)		
% Gloss retention @ 60°/20°	99/95	100/100
Blistering[d]	5	5
Salt spray after 1,000 h		
Creepage, mm (in.)	1.59 (0.0625)	1.59 (0.0625)
Blistering[d]	5	5
Cracking[d]	5	5
Detergent resistance after 10 d @ 74°C (165°F)[e]		
Creepage, mm (in.)	4.76 (0.1875)	3.18 (0.125)
% Gloss retention @ 60°/20°	99/84	97/84
Blistering[d]	4	3
Cracking[d]	4	5

[a] *Enamels were sprayed on 20-gauge cold-rolled Bonderite™ 37 pretreated steel test panels and cured for 20 minutes at 177°C (350°F).*

[b] *200 double rubs with MEK, quickly wiped dry. Pass = no thumbnail mar.*

[c] *The stain and chemical resistance panels were washed with Dawn™ dishwashing detergent, rinsed with water, and wiped dry before evaluation.*

[d] *Scale: 5 = no effect, 4 = slight effect, 3 = moderate effect, 2 = considerable effect, and 1 = severe effect.*

[e] *The test environment from ASTM Method D2248-73 was used.*

Formulation comparison summary

HS-3-1NCp	HS-3-2NCp
← Overall similar coating performance →	
Lower viscosity	More flexible
Lower VOC	Better moisture resistance
More compatible with xylene	

Raw material suppliers

n-Butyl alcohol	Eastman
Cymel™ 303 melamine resin	Cytec
Eastman™ 1,4-CHDA[a]	Eastman
Eastman™ EEP[b]	Eastman
Eastman™ purified isophthalic acid (PIA)	Eastman
Eastman™ MAK	Eastman
Eastman NPG™ glycol	Eastman
Phthalic anhydride	Stepan
Eastman™ purified terephthalic acid (PTA)	Eastman
Ti-Pure™ R-900 TiO_2	DuPont
Trimethylolpropane	Geo Specialties, Perstorp
p-Toluenesulfonic acid catalyst	Aldrich

[a]*1,4-Cyclohexanedicarboxylic acid*

[b]*Ethyl 3-ethoxypropionate*

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