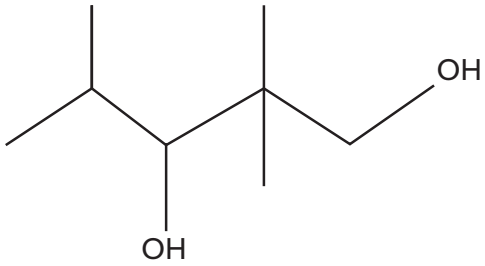
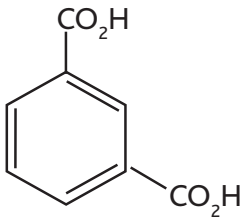
High-solids polyester liquid coating resin formulations HS-3-5T and HS-3-6T

Based on Eastman TMPD™ glycol and Eastman™ purified isophthalic acid (PIA)



2,2,4-Trimethyl-1,3-pentanediol
CAS: 144-19-4



Purified isophthalic acid
CAS: 121-91-5

Features

HS-3-5T

- 2.72 lb/gal (326 g/L) determined VOC
- Excellent chemical and stain resistance
- Excellent corrosion resistance
- Good hardness

HS-3-6T

- 2.45 lb/gal (294 g/L) determined VOC
- Excellent chemical resistance
- Excellent corrosion resistance

The resin composition and processing procedure presented in this publication demonstrate the utility of Eastman TMPD™ glycol in a polyester resin for coating applications. These formulations have not been optimized for any particular property or end use. Alternative raw materials and process conditions should be considered to optimize for a particular application.

HS-3-5T resin formulation

Reactants	Equivalents	Moles	Wt, g
Eastman TMPD™ glycol	12.00	6.00	877
Trimethylolpropane (TMP)	1.65	0.55	74
Eastman™ purified isophthalic acid (PIA)	4.54	2.27	377
Adipic acid (AD)	4.54	2.27	332
		Charge	1,660
		H_2O	**–160**
		Yield	1,500

Catalyst: 3.3 g Fascat™ 4100 (0.2% based on total charge)

Processing procedure

- Charge all the reactants and catalyst to a 2-L reaction kettle equipped with a heating mantle, agitator, nitrogen purge, thermocouple, partial condenser, water trap, and total condenser. Reference Eastman publications N-345 and N-206 on glassware assembly and synthesis tips on Eastman TMPD™ glycol, respectively.
- Set the purge rate to 0.4 standard cubic feet per hour (SCFH) and increase the temperature to 200°C (392°F). Hold at 200°C for about 3 hours.
- Increase the temperature to a maximum of 210°C (410°F) and hold for a final acid number of 6 ± 2 (mg KOH/g resin) and a cone and plate viscosity of 0.35 to 0.50 Pa·s (3.5 to 5.0 P) at 100°C (212°F) or a Gardner-Holdt™ viscosity of Z–Z_2 at 85 wt % theoretical nonvolatiles in xylene.
- Allow the resin to cool to 140°C (284°F) and add 265 g xylene.

HS-3-6T resin formulation

Reactants	Equivalents	Moles	Wt, g
Stage 1			
Eastman TMPD™ glycol	12.24	6.12	895
Trimethylolpropane (TMP)	0.88	0.29	39
Eastman™ purified isophthalic acid (PIA)	4.38	2.19	364
Adipic acid (AD)	4.38	2.19	320
Stage 2			
Trimethylolpropane	0.88	0.29	39
		Charge	1,657
		H_20	**–157**
		Yield	1,500

Catalyst: 1.6 g Fascat™ 4100 (0.1% based on total charge)

Processing procedure

- Charge all of the Stage 1 reactants and all of the catalyst to a 2-L reaction kettle equipped with a heating mantle, agitator, nitrogen purge, thermocouple, partial condenser, water trap, and total condenser. Reference Eastman publications N-345 and N-206 on glassware assembly and Eastman TMPD™ glycol synthesis tips, respectively.
- Set the purge rate at 0.4 standard cubic feet per hour (SCFH) and increase the temperature to 200°C (392°F). Hold at 200°C until approximately 78 mL (half of theoretical total) of distillate has been collected.
- Add the Stage 2 TMP and increase the reaction temperature to a maximum of 215°C (419°F). Hold at 215°C for a final acid number of 6 ± 2 (mg KOH/g resin) and a cone and plate viscosity of 0.05–0.20 Pa·s (0.5–2.0 P) at 125°C (257°F). Total time from start of up-heat to completion is about 6 hours.
- Allow the resin to cool to 140°C (284°F) and add solvent.

Resin properties

	Formula	
	HS-3-5T	HS-3-6T
Equivalents of OH/equivalents of COOH (R value)	1.5	1.6
Target acid number, mg KOH/g resin	4–8	4–8
Calculated hydroxyl number, mg KOH/g resin	170	198
Determined molecular weight (by gel permeation chromatography)	800–1,000	900–1,100
Dilution solvent	Xylene	Xylene
Calculated nonvolatiles, % solids	85	85
Gardner-Holdt™ viscosity	Z—Z_2	Z
Gardner™ color	1	1
Density, g/L (lb/gal)	1,051 (8.74)	1,041 (8.69)

Polyester/melamine enamel composition

	Formula	
	HS-3-5T	HS-3-6T
Ingredients	**Wt%**	
Resin formula (85 wt% in xylene)	39.40	40.3
Hexamethoxymethylmelamine	14.35	14.7
Ti-Pure™ R-960 TiO_2	31.93	32.5
p-Toluenesulfonic acid catalyst (40 wt% in i-propanol)	0.36	0.4
Fluorosurfactant (20 wt% in Eastman™ EEP)	0.48	0.5
Eastman™ MAK (methyl *n*-amyl ketone)	10.00	7.6
Eastman™ EEP	0.96	2.0
Eastman™ PM acetate	0.64	—
Xylene	0.09	—
Eastman™ *n*-butyl alcohol	1.79	2.0
Total	**100.00**	**100.00**

Enamel properties

	Formula	
	HS-3-5T	HS-3-6T
Pigment/binder ratio	40/60	40/60
Polyester/melamine ratio	70/30	70/30
Density,[a] g/L (lb/gal)	1,322 (11.03)	1,316 (10.98)
Calculated nonvolatiles, wt%	76.4	80.9
Determined nonvolatiles,[b] wt%	75.3	79.3
Determined VOC,[c] g/L (lb/gal)	326 (2.72)	294 (2.45)
#4 Ford Cup viscosity, s	37	43

[a]*Determined using a Byk-Gardner™ standard wt/gal cup.*

[b]*Test performed according to ASTM Test Method D2369-90.*

[c]*Calculation performed according to ASTM Test Method D3960-90 (10.1).*

Cured film[a] properties

	Formula	
	HS-3-5T	HS-3-6T
Film thickness, mils (microns)	1.3–1.8 (33–46)	1.3–1.8 (33–46)
Gloss, 60°/20°	89/76	—
Pencil hardness, scratch	2H	2H
Impact resistance, N·m (in.·lb)		
Direct	11 (100)	13.6 (120)
Reverse	2 (20)	5.6 (50)
Solvent resistance, MEK double rubs	200+	200+
1/8 in. Conical mandrel flexibility,[b] % pass	92	90
Cleveland™ humidity,[c] 40 h at 60°C (140°F)		
% gloss retention, 60°/20°	100/99	—
20° gloss retention, %	—	93
Blistering	V.V. few #8	none
Stain resistance		
Iodine for 30 min	Slight effect	Severe effect
Mustard for 24 hr	No effect	Slight effect
Ink for 24 hr	No effect	—
Chemical resistance[d]		
50% NaOH solution, 1 h at room temperature	No effect	No effect
50% H_2SO_4 solution, 1 h at room temperature	No effect	No effect
Salt spray[e] resistance after 500 h	No creepage	Pass, ≤ 1/16 in. creepage

[a]*Coating applied to 20 gauge, cold-rolled, Bonderite™ 37 pretreated steel test panels. Baked 20 minutes at 149°C (300°F).*

[b]*Test performed according to ASTM Test Method D522-88 (A).*

[c]*Test performed according to ASTM Test Method D4585-87.*

[d]*Test performed according to ASTM Test Method D1308-87.*

[e]*Test performed according to ASTM Test Method B117-90*

HS-3-6T Viscosity[a] profile

Solvent	Theoretical % solids	Determined % solids[b]	Gardner-Holdt™ viscosity	Brookfield viscosity cP
Xylene	95	88.3	Z_8	1,420
	90	83.5	Z_5	
	85	79.3	Z	
	80	74.5	U	
	75	69.3	J	
	70	64.8	E	
Aromatic™ 100	95	87.3	Z_8	—
	90	82.3	Z_5	
	85	78.0	Z_1	
	80	73.5	V	
	75	68.2	K	
	70	62.5	E	
Eastman™ EEP	95	91.0	Z_8	1,660
	90	85.0	Z_5	
	85	79.7	Z_1	
	80	75.6	V	
	75	70.6	M	
	70	65.8	G	
Eastman™ MAK (methyl *n*-amyl ketone)	95	89.4	Z_7	808
	90	84.2	Z_4	
	85	79.8	X	
	80	74.7	S	
	75	69.9	H	
	70	65.7	C	
Eastman™ PM acetate	85	77.6	—	1,930

[a]*Determined at 25°C (77°F).*

[b]*After 60 minutes at 110°C (230°F); reference ASTM D2369-90.*

Raw material suppliers

Aromatic™ 100	Exxon
Adipic acid	DuPont
Eastman™ *n*-butyl alcohol	Eastman
Eastman™ EEP	Eastman
Fascat™ 4100 catalyst	Arkema
Hexamethoxymethylmelamine	Cytec
Eastman™ MAK (methyl *n*-amyl ketone)	Eastman
Eastman™ purified isophthalic acid (PIA)	Eastman
Ti-Pure™ R-900 TiO_2	DuPont
Eastman TMPD™ glycol	Eastman
p-Toluenesulfonic acid	Aldrich
Trimethylolpropane	Geo Specialties, Perstorp

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