

Powder coating resins based on Eastman NPG™ glycol and Eastman™ PIA (purified isophthalic acid) for superior outdoor durability

- Hydroxyl polyester resin and polyester/blocked isocyanate formulation
- Carboxyl polyester resin and polyester/Primid™ XL 552 formulation

Hydroxyl polyester resin and polyester/blocked isocyanate formulation

Synthesis of resin PC-17-8N from Eastman NPG™ glycol and 95/5 Eastman™ PIA/TMA

Reactants	Equivalents	Moles	Grams
Eastman NPG™ glycol	13.48	6.739	702
Trimellitic anhydride (TMA)	0.91	0.304	58
Eastman™ purified isophthalic acid (PIA)	11.54	5.77	958
Fascat™ 4100 (butylstanoic acid)			1.5
Total reactant charge			1,719.5
			Water loss - 219.5
			Resin yield 1,500.0

- Excess glycol of 1%–3% in addition to the charge indicated may be needed depending on the setup of your reactor. We use no excess.
- Eastman™ purified isophthalic acid reacts faster and cleaner than terephthalic acid. There is less popping of the Eastman™ PIA up to the dome of the reactor.

Procedure

1. Setup: A 3-liter reactor equipped with mechanical stirrer, heated packed column, distillation condenser, thermocouples, nitrogen stream, and accessories needed for a computer controlled reactor.
2. Charge reactants, catalyst, and excess glycol to the reactor purged with nitrogen. Increase heat gradually to melt glycol and begin stirring when possible. Maintain a nitrogen stream and stirrer speed at 300 rpm.
3. Set heat control as follows; remove water from the reaction through the heated column and distillation condenser.

25°–180°C	over	1.3 hours
180°–235°C	over	5 hours
235°C	hold	for 5 hours
4. Process to acid number 4–8 (mg KOH/g resin) and cone and plate melt viscosity at 200°C of 18–25 poise.

Resin properties

Hydroxyl number, mg KOH/g resin	30–36
Acid number, mg KOH/g resin	4–8
Gel permeation chromatography	
Weight average molecular weight, Mw	12,000–15,000
Number average molecular weight, Mn	3,600–3,800
T _g , glass transition temperature (DSC, 2nd cycle, midpoint)	55°–60°C
Melt viscosity, cone and plate, at 200°C, poise (Pa·s)	18–25 (1.8–2.5)

Powder coating resins based on Eastman NPG™ glycol and Eastman™ PIA (purified isophthalic acid) for superior outdoor durability *(Continued)*

Formulation of polyester/polyurethane powder coatings based on resin PC-17-8N

Ingredients	Grams
Polyester resin PC-17-8N	574
Blocked isophorone diisocyanate (Vestagon™ B1530)	126
Dibutyltindilaurate, 60% active on powder substrate	11.7
Benzoin	7
Flow control agent (Modaflow™ 2000)	10.5
TiO ₂ , TiPure™ R960	350
Total weight	1,079.2

- If a self-blocked isophorone diisocyanate (e.g., Vestagon BF 1540) is used, a higher extrusion temperature and cure at 190°C (375°F) for 20 minutes are necessary.
- A 30-mm twin-screw extruder is used with zone 1 and zone 2 set at 90°C and 100°C respectively with screw speed of 300 rpm.
- Powder particle size average is 25 microns; maximum is 74 microns.
- Powder caking stability test at 40°C for 3 days is acceptable.

Physical properties of coating enamel^{a,b}

Cure temperature/time, °C/min (°F/min)	176–204/20 (350–400/20)
Thickness, micron (mil)	48.3–53.3 (1.9–2.1)
Initial gloss, Gardner 20°/60°	75/93
Impact resistance, front/reverse, N•m (in•lb)	4.7/0.7 (42/6)
Pencil hardness	H-2H
Solvent resistance, MEK double rubs	200+
Conical mandrel flexibility, 3 mm (1/8 in.), % pass	0

^aUnprimed, zinc phosphated cold roll steel panels are used.

^bImpact test results are determined using a 10X magnifier critically. Your results may be better than those reported here.

Carboxyl polyester resin and polyester/Primid™ XL 552 formulation

Synthesis of carboxyl resin PC-17-9N

Reactants	Equivalent	Moles	Grams
First stage			
Eastman NPG™ glycol	11.630	5.815	605.6
Trimethylolpropane (TMP)	0.540	0.180	24.1
Eastman™ purified isophthalic acid (PIA)	10.945	5.473	909.2
Triphenyl phosphite			0.75
Fascat™ 4100 (butylstannic acid)			1.5
Second stage			
Eastman™ purified isophthalic acid (PIA)	2.107	1.053	175
Total reactant charge			1,716.15
Water loss			-216.15
Resin yield			1,500.0

- Excess glycol of 1% is used in this cook. An excess of 1%–3% may be needed depending on reactor setup.
- Eastman™ purified isophthalic acid reacts faster and cleaner than terephthalic acid. There is less popping of the Eastman™ PIA up to the dome of the reactor.

Procedure

1. Setup: A 3-liter reactor equipped with mechanical stirrer, heated packed column, distillation condenser, thermocouples, nitrogen stream, and accessories needed for a computer-controlled reactor.
2. Charge and heat the first stage glycols to 140°C. Add the first stage acids, stabilizer, and catalyst while maintaining a nitrogen stream and a stirrer speed of 300 rpm.
3. Set heat control as follows; remove water from the reaction through the heated column and distillation condenser.

25°–180°C	over	1.3 hours
180°–235°C	over	5 hours
235°C hold	for	3 hours
4. Process to acid number of 5–10 (mg KOH/g resin) and cone and plate melt viscosity at 200°C of 4–8 poise.
5. Cool to 180°C and add the second stage Eastman™ purified isophthalic acid.
6. Heat to 235°C and hold at 235°C for 2 hours followed by 2 hours of mild vacuum.
7. Process to acid number 32–38 (mg KOH/g resin) and cone and plate melt viscosity at 200°C of 24–30 poise.

Resin properties

Hydroxyl number, mg KOH/g resin	5–10
Acid number, mg KOH/g resin	32–38
Gel permeation chromatography	
Weight average molecular weight, Mw	10,000–11,000
Number average molecular weight, Mn	3,000–3,300
T _g , glass transition temperature (DSC, 2nd cycle, midpoint)	63°–67°C
Melt viscosity, cone and plate, at 200°C, poise (Pa·s)	24–30 (2.4–3.0)

Powder coating resins based on Eastman NPG™ glycol and Eastman™ PIA (purified isophthalic acid) for superior outdoor durability (Continued)

Formulation and physical properties of polyester/Primid™ XL552 powder coating based on resin PC-17-9N

Ingredients	Grams
Polyester resin PC-17-9N	570
Primid™ XL 552	30
Benzoin	3
Flow control agent (Modaflow 2000)	10.5
TiO ₂ , TiPure™ R960	300
Total weight	913.5

- A 30-mm twin-screw extruder is used with zone 1 and zone 2 set at 100°C and 110°C respectively with screw speed of 300 rpm.
- Powder particle size average is 25 microns; maximum is 74 microns.
- Powder-caking stability test at 40°C for 3 days is acceptable.

Physical properties of coating enamel^{a,b}

Cure temperature/time, °C/min (°F/min)	176–204/20 (350–400/20)
Thickness, micron (mil)	48.3–53.3 (1.9–2.1)
Gloss, Gardner 20°/60°	80/94
Impact resistance, front/reverse, N•m (in•lb)	4.7/0.7 (42/6)
Pencil hardness	H
Solvent resistance, MEK double rubs	200+
Conical mandrel flexibility, 3 mm (1/8 in.), % pass	0

^aUnprimed, zinc phosphated cold roll steel panels were used.

^bImpact test results were determined using a 10X magnifier critically. Your results may be better than those reported here.

Raw material suppliers

Eastman NPG™ glycol	Eastman
Trimellitic anhydride	BP Amoco
Trimethylolpropane (TMP)	Celanese, Perstorp
Eastman™ purified isophthalic acid (PIA)	Eastman
Fascat™ 4100	Arkema, Inc.
Primid™ XL552 EMS	EMS Chemie AG
Benzoin	Estron Chemical, Inc.
Modaflow™ 2000	Cytec
Dibutyltindilaurate, 60% active on powder substrate	Aldrich
TiPure™ R960	DuPont



**Eastman Chemical Company
Corporate Headquarters**

P.O. Box 431
Kingsport, TN 37662-5280 U.S.A.
Telephone:
U.S.A. and Canada, 800-EASTMAN (800-327-8626)
Other Locations, (1) 423-229-2000
Fax: (1) 423-229-1193

Eastman Chemical Latin America

9155 South Dadeland Blvd.
Suite 1116
Miami, FL 33156 U.S.A.
Telephone: (1) 305-671-2800
Fax: (1) 305-671-2805

Eastman Chemical B.V.

Fascinatia Boulevard 602-614
2909 VA Capelle aan den IJssel
The Netherlands
Telephone: (31) 10 2402 111
Fax: (31) 10 2402 100

**Eastman (Shanghai) Chemical
Commercial Company, Ltd. Jingan Branch**

1206, CITIC Square
No. 1168 Nanjing Road (W)
Shanghai 200041, P.R. China
Telephone: (86) 21 6120-8700
Fax: (86) 21 5213-5255

Eastman Chemical Japan Ltd.

MetLife Aoyama Building 5F
2-11-16 Minami Aoyama
Minato-ku, Tokyo 107-0062 Japan
Telephone: (81) 3-3475-9510
Fax: (81) 3-3475-9515

Eastman Chemical Asia Pacific Pte. Ltd.

#05-04 Winsland House
3 Killiney Road
Singapore 239519
Telephone: (65) 6831-3100
Fax: (65) 6732-4930

www.eastman.com

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