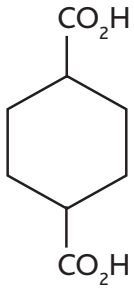
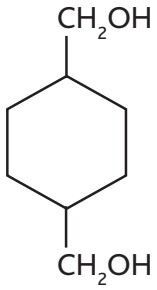
Appliance coil coatings

Based on Eastman™ 1,4-CHDA and Eastman™ CHDM



1,4-CHDA



CHDM

- Excellent flexibility with stain resistance
- Good hardness and humidity resistance
- Low color

Starting point formulations
PA-1-1CNCp and PA-1-2CNCp

Table 1 **Reactor charge**[a]

	PA-1-1CNCp			PA-1-2CNCp		
	Equivalents	Moles	Grams	Equivalents	Moles	Grams
First stage						
Eastman NPG™ glycol	7.38	3.69	385	6.58	3.29	343
Eastman™ CHDM glycol	4.92	2.46	355	4.39	2.19	316
1,6-Hexanediol	—	—	—	1.22	0.61	72
Adipic acid	1.16	0.58	85	—	—	—
Eastman™ purified isophthalic acid (PIA)	2.32	1.16	193	2.30	1.15	191
Eastman™ purified terephthalic acid (PTA)	2.32	1.16	193	2.30	1.15	191
Second stage						
Eastman™ 1,4-CHDA	5.80	2.90	499	6.90	3.45	594
Total charge			1,710			1,707
Theoretical distillate			210			207
Theoretical yield			1,500			1,500

$$R = \frac{\text{Equivalents of OH}}{\text{Equivalents of } CO_2H} = 1.06$$

Eastman™ CHDM:Eastman NPG™ glycol molar ratio	40:60
Eastman™ PIA:Eastman™ PTA molar ratio	50:50
1,6-HD or AD molar content based on glycol or acid component	10%

Catalyst: 1.7 g Fascat™ 4100, charged in 2 parts
Nitrogen flow: 0.8 standard cubic ft/h (SCFH)
Azeotrope solvent: 45 g Aromatic™ 150

[a] *See raw material suppliers table on page 4.*

Synthesis procedure

First stage

1. Charge first stage reactants (glycols first) and half of the catalyst to a 2-liter reaction kettle equipped with a heating mantle, agitator, nitrogen supply, temperature probe, steam-heated packed partial condenser, water trap, and total condenser.
2. Heat to 220°C (428°F) over 2 hours. Maintain at 220°C until an acid number of 1 (mg KOH/g resin) or less is reached, typically an additional 1 hour.
3. Cool to 140°C (284°F) for second stage charge.

Second stage

4. After cooling to 140°C (284°F), charge the Eastman™ 1,4-CHDA.
5. Heat to 230°C (446°F) over 2 hours. Maintain at 230°C until an acid number of 25 (mg KOH/g resin) or less is reached, typically an additional 2 hours.
6. Cool to 140°C (284°F).
7. After cooling to 140°C (284°F), remove steam-heated packed partial condenser or switch to an open column. Charge the remaining catalyst and 45 grams (plus the amount required to fill the water trap) of Aromatic™ 150.
8. Heat to 230°C (446°F) over 1 hour. Maintain at 230°C until an acid number of 4 ± 1 (mg KOH/g resin) is reached, typically an additional 2 hours.
9. Cool to 140°C (284°F) and adjust to 60 wt% solids with Aromatic™ 150. Total processing time may range from 9 to 12 hours.

Table 2 **Resin properties**

Resin properties	PA-1-1CNCp	PA-1-2CNCp
Acid number, mg KOH/g resin	4	4
Hydroxyl number, mg KOH/g resin	36	31
Molecular weight, M_n[a]	4,270	4,630
Molecular weight, M_w[a]	10,720	12,980
ICI viscosity @ 200°C, poise (Pa·s)	33 (3.3)	29 (2.9)
Gardner-Holdt™ viscosity	Z_5	Z_4
APHA color	80	80
T_g,[b] °C	23	23
Solvent	Aromatic™ 150	Aromatic™ 150
Calculated nonvolatiles, wt%	60	60
Determined density, lb/gal (kg/L)	8.77 (1.05)	8.70 (1.04)
Days to hazing @ room temperature	210	270

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

[b] *Determined by DSC (Midpoint of 2nd heat reported; upheat rate of 20°C/min).*

Table 3 **Enamel formulation**

Ingredients	Wt%
Polyester resin (60 wt% nonvolatiles)	56.3
Cymel™ 301 melamine resin	3.6
Ti-Pure™ R-960 TiO_2 pigment	25.0
Nacure™ 1419 catalyst	1.9
Acrylic flow control agent	0.6
Solvent blend[a]	12.6
	100.0
Pigment:binder ratio	40:60
Polyester:melamine ratio	90:10

[a] *64/12/24 wt % blend of Eastman™ C-11 ketone/Eastman™ EEP/Eastman™ EB*

Adjust the enamels to 50% by volume solids with the solvent blend. Using an automated drawdown device, apply coating to Q-Panel™ AL-39 aluminum substrates with chromium pretreatment and cure for 30 seconds at 313°C (595°F) to obtain a peak metal temperature of 216°C (420°F).

Table 4 Enamel properties and performance

	PA-1-1CNCp	PA-1-2CNCp
Film thickness, microns (mils)	19 (0.75)	19 (0.75)
Gloss @ 60°/20°	90/78	86/73
MEK double-rub solvent resistance[a]	90	80
Hardness/flexibility		
T-bends[b]	**With grain/Against grain**	
Initial	1T/0T	1T/0T
Overbake, 30 s @ 260°C (500°F)	3T/2T	3T/2T
Wet heat, 30 s in boiling water	2T/2T	2T/1T
Reverse impact resistance @ 40 in.-lb (4.5 N·m), % pass	100	100
Pencil hardness (to mar)		
Initial	2H	2H
30 min boiling water test, min to recovery	15	30
Adhesion		
Crosshatch adhesion, % pass[b]	100	100
Stain resistance[c,d]	**Covered/Uncovered**	
Iodine after 30 min	3/4	2/4
Mustard after 24 h	4/5	3/4
Lipstick after 24 h	4/4	3/3
Ink after 24 h	4/4	4/3
Catsup after 24 h	5/5	5/5
Grape juice after 24 h	5/5	5/5
Etch resistance after 8 h [c,d]		
50% NaOH solution	5/5	5/5
50% H_2SO_4 solution	5/5	5/5
Detergent resistance @ 74°C (165°F)[e]	**5 Days/10 Days**	
Creepage detected	none/none	none/none
% Gloss retention 60°	96/69	98/95
20°	74/24	84/63
Blister size[f]	8/6	8/6
Blister frequency[f]	4/2	4/2
Cracking[b]	5/5	5/5
Cleveland humidity resistance[g] @ 60°C (140°F)		
% Gloss retention, 60°/20° after: 1,000 h	100/99	99/89
1,250 h	99/99	95/78
1,500 h	88/54	11/3
1,750 h	74/36	7/3

[a] *Double rubs with methyl ethyl ketone (MEK) to breakthrough*

[b] *Results were checked using Scotch brand tape No. 610 (3M Company). After 24 hours relaxation, samples showed no cracking under unmagnified visual inspection.*

[c] *The stain- and chemical-resistant panels were washed with Dawn™ dishwashing detergent (Procter & Gamble Company), rinsed with water, and wiped dry before evaluation.*

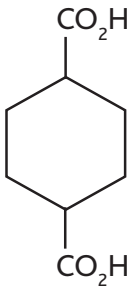
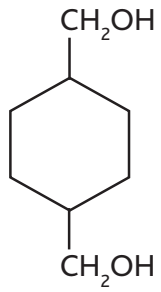
[d] *Scale: 5 = no effect; 1 = severe effect*

[e] *Test environment from ASTM method D2248.*

[f] *ASTM method D714, evaluating degree of blistering of paint. Blister size rating: 10 = no blisters; 2 = large blisters; Blister frequency: 5 = none; 1 = dense*

[g] *ASTM method D4585*

Structure/property benefits

Eastman intermediate	Structure	Benefits
 1,4-CHDA	Saturated ring structure	Excellent hardness and flexibility ratio Better hydrolytic stability, etch, and stain resistance than aliphatic acids Very good solubility in molten glycols for rapid processing Low resin color
	1,4-Substituted saturated ring structure	Excellent thermal stability Moderate T_g
 CHDM	Primary, unhindered hydroxyl groups	Very rapid polymer synthesis Lower temperature or reduced cure time during crosslinking Low resin color
	1,4-Substituted saturated ring structure Symmetrical structure	Very good hardness and flexibility ratio High T_g Excellent thermal stability High T_g

Summary

Both resins contain Eastman™ 1,4-CHDA, Eastman™ purified isophthalic acid (PIA), Eastman™ purified terephthalic acid (PTA), Eastman™ CHDM, and Eastman NPG™ glycol. The cycloaliphatic structures of Eastman™ 1,4-CHDA and Eastman™ CHDM provide **good hardness** and contribute **flexibility**. These appliance formulations offer reproducible 1T-bends from resins containing only 5 mole percent of the typical flexibilizing monomers adipic acid or 1,6-hexanediol.

Raw material suppliers

Eastman™ 1,4-CHDA	Eastman
Eastman™ CHDM glycol	Eastman
Eastman NPG™ glycol	Eastman
Eastman™ EEP	Eastman
Eastman™ C-11 ketone	Eastman
Eastman™ EB	Eastman
Eastman™ purified isophthalic acid	Eastman
Cymel™ 301 resin	Cytec
Fascat™ 4100 catalyst	Arkema
Ti-Pure™ R-960 TiO_2	DuPont
1,6-Hexanediol	BASF
Eastman™ purified terephthalic acid	Eastman
Adipic acid	DuPont
Nacure™ 1419 catalyst	King Industries
Aromatic™ 150 solvent	Exxon

EASTMAN

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