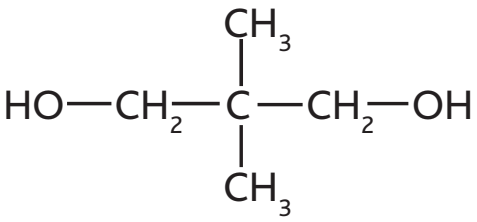
Binder resin for onyx

Based on Eastman NPG™ glycol



Eastman NPG™ glycol

One of the most popular forms of cultured marble is onyx. In addition to the freedom of design and choice of colors available in cultured marble and granite, onyx offers an appealing depth of color. Because consumer acceptance of onyx for sanitary ware and bathroom vanity tops is very well established, many manufacturers place onyx at the top of these types of product lines.

The International Cast Polymer Alliance (ICPA) has adopted a set of standards for the performance of cultured marble, cultured granite, and onyx vanity tops. Those manufacturers who can demonstrate that their products meet these performance requirements are allowed to place a certification label on their vanity tops. The certification labels have been found by many to be an aid in marketing the products. One of the most critical ICPA standards is the resistance of vanity tops to thermal shock cracking.

There are many factors that affect the ability of a vanity top to meet the ICPA thermal shock standards, but it is generally agreed that the binder resin is one of the more important considerations. Evaluations have been conducted at Eastman to develop a binder resin formulation that can be used to produce onyx vanity tops with the performance features required for ICPA certification. As a result, a resin based on Eastman NPG™ glycol has been formulated and shown to possess many desirable properties (Table 1), including excellent resistance to thermal shock cracking, low color, and good cure characteristics.

Table 1 **Onyx binder resin based on Eastman NPG™ glycol[a] BR-11-4N**

Reactants (molar composition)	
Eastman NPG™ glycol	3.48
Propylene glycol (PG)	0.87
Phthalic anhydride (PA)	1.00
Adipic acid (AD)	1.00
Maleic anhydride (MA)	2.14
Reactant ratios	
NPG glycol:PG (molar ratio)	80:20
PA:AD (molar ratio)	1:1
MA, wt%, based on 100% resin solids	25
Styrene, wt%	32
Synthesis	
Final acid number, mg KOH/g resin	12
Number average molecular weight (by gel permeation chromatography)	2,400
Gardner™ viscosity in 40% Eastman™ PM solvent (propylene glycol monomethyl ether)	U
Brookfield™ viscosity in 32% styrene, cP	1,250

[a]*See Table 5 for cook log.*

Physical properties

Resin BR-11-4N is a resilient formulation, yet it maintains a 90°C heat deflection temperature and high strength values. This combination of good physical properties along with some resiliency makes this an attractive binder resin (Table 2).

Table 2 Physical properties of formulation BR-11-4N (1/8-in. unfilled castings)

Heat distortion temperature, °C (°F)	90 (194)
Tensile strength, MPa (psi)	55 (8,000)
Tensile elongation, %	3
Flexural strength, MPa (psi)	103 (15,000)
Flexural modulus, MPa (psi X10^5)	2,896 (4.2)

Improved resistance to thermal shock cracking

The ICPA requires vanity tops pass 500 hot/cold cycles to meet its performance standards for resistance to thermal shock cracking (ANSI Z-124.3). Results far exceeding this minimum requirement have been obtained with vanity tops cast from formulation BR-11-4N. As many as 1,200 cycles have been successfully completed.

Low resin color

It is generally accepted in the polyester industry that low-color resins can be formulated from NPG glycol. Resin BR-11-4N has consistently been synthesized on a pilot plant scale with a platinum cobalt (Pt-Co) color of 30. This low color is quite important in the production of quality onyx parts.

Good cure characteristics

In trial runs conducted at customers' plants, resin BR-11-4N has been shown to cure quite rapidly with low promoter levels. Vanity tops cast from this resin typically were ready for demolding one hour and forty minutes after addition of the catalyst. Naturally, the rate of cure will depend on the cure system used in the resin. Formulation BR-11-4N contains 25 wt% maleic anhydride for good reactivity with low amounts of promoters.

Cure system

One requirement for a good onyx binder resin is that it must cure with very low color. The cure system used in the test vanity tops cast from resin BR-11-4N is given in Table 3. Although this system provides a low color after cure, other cure systems may provide even less color.

Table 3 Cure system

	Parts per hundred resin (Phr)
Inhibitor Eastman™ hydroquinone[a]	0.015
Promoters Cobalt octoate, 6%	0.1
Aliquat™ 336 fatty quaternary ammonium chloride[b]	0.025
Catalyst Methyl ethyl ketone peroxide, 60%[c]	1.0

[a] *Manufactured by Eastman Chemical Company*

[b] *Manufactured by Cognis Corporation*

[c] *Manufactured by Arkema Inc.*

Onyx formulation

The depth of color that distinguishes onyx from the more traditional cultured marble is obtained by matching the refractive indexes of the binder resin and filler. This results in an improvement in translucency or depth of color. Although the refractive index of the binder resin can be changed slightly through blends of monomers, the selection of the filler is the key point. The most commonly used fillers for onyx are aluminum trihydrate and frit glass. Two onyx formulations are given in Table 4 using resin BR-11-4N in combination with each of these fillers.

Table 4 Onyx formulation

	Weight %
Formula 1	
Resin BR-11-4N	35
Alcoa™ C-331 aluminum trihydrate[a]	65
	100
Formula 2	
Resin BR-11-4N	27
Frit glass[b]	73
	100

[a]*Manufactured by Aluminum Company of America*

[b]*Manufactured by Ferro Corporation*

Table 5 Cook log for formulation BR-11-4N Eastman NPG™ glycol/PG/PA/AD/MA

Reactants	Mole ratio	Moles	Weight units
Eastman NPG™ glycol	3.48	0.414	43.1
Propylene glycol	0.87	0.104	7.9
Phthalic anhydride	1.00	0.119	17.6
Adipic acid	1.00	0.119	17.4
Maleic anhydride	2.14	0.255	25.0
			111.0
		Calculated water loss	–11.0
		Yield	100.0

Catalyst: None
Nitrogen flow: 0.4 liter/min
Up-heat time: 2.5 hours to 180°C (356°F)

Processing time, h	Reaction temp., °C (°F)	Distillate, grams	Acid no.	Gardner™[a] viscosity	Remarks
0.00	180 (356)				Heat to 200°C (392°F).
2.00	200 (392)	8.6			
5.00	200 (392)	9.7			
10.00	200 (392)	10.2			
12.00	200 (392)	10.4	22	J	
15.00	200 (392)	10.7	20	M	N_2 up to 1.0 L/min
18.00	200 (392)	11.0	13	T	
19.00	200 (392)	11.0	12	U	Heat off. Cool to 140°C (284°F). Add 50 ppm HQ and 32% styrene.

Procedure: Charge the Eastman NPG™ glycol, propylene glycol, phthalic anhydride, adipic acid, and maleic anhydride. Purge with nitrogen and begin heat up. Proceed according to "Remarks" given with cook log.

[a]*Sixty percent solids in Eastman™ PM solvent*

EASTMAN

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.
Fascinatio 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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