Kirk-Othmer Encyclopedia of Chemical Technology.

GLYCOLS, OTHER GLYCOLS

Glycols such as neopentyl glycol, 2, 2, 4-trimethyl-1, 3-pentanediol, 1, 4-cyclohexanedimethanol, and hydroxypivalyl hydroxypivalate are used in the synthesis of polyesters (qv) and urethane foams (see Foamed plastics). Their physical properties are shown in Table 1 (1–6).

1. Neopentyl Glycol

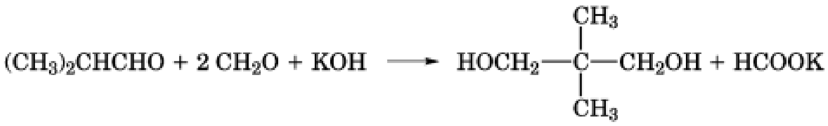
Neopentyl glycol, or 2, 2-dimethyl-1, 3-propanediol [126-30-7] (1) is a white crystalline solid at room temperature, soluble in water, alcohols, ethers, ketones, and toluene but relatively insoluble in alkanes (1). Two primary hydroxyl groups are provided by the 1, 3-diol structure, making this glycol highly reactive as a chemical intermediate. The *gem*-dimethyl configuration is responsible for the exceptional hydrolytic, thermal, and uv stability of neopentyl glycol derivatives.

1.1. Chemical Properties

Neopentyl glycol can undergo typical glycol reactions such as esterification (qv), etherification, condensation, and oxidation. When basic kinetic studies of the esterification rate were carried out for neopentyl glycol, the absolute esterification rate of neopentyl glycol with *n*-butyric acid was approximately 20 times that of ethylene glycol with *n*-butyric acid (7).

1.2. Manufacture

Commercial preparation of neopentyl glycol can be via an alkali-catalyzed condensation of isobutyraldehyde with 2 moles of formaldehyde (crossed Cannizzaro reaction) (2, 8). Yields are ∼70%.



$$(CH_3)_2CHCHO + 2\,CH_2O + KOH \longrightarrow HOCH_2{-}\underset{CH_3}{\overset{CH_3}{\underset{|}{\overset{|}{C}}}}{-}CH_2OH + HCOOK$$

(1)

Neopentyl glycol is manufactured by Eastman Chemical Co., BASF, Perstorp, Hoechst, Mitsubishi Gas, Polioli, and Hüls. In 1993, the bulk U.S. price was $1.61/kg.

1.3. Toxicity

Acute toxicity data for neopentyl glycol **(1)** are reported in Table 2.

Table 1. Physical Properties of Several Glycols

Properties	Neopentyl glycol **(1)**	2, 2, 4-Trimethyl-1, 3-pentanediol **(7)**	1, 4-Cyclohexane-dimethanol[a] **(8)**	Hydroxypivalyl hydroxypivalate **(9)**
CAS Registry Number	[126-30-7]	[144-19-4]	[105-08-5]	[1115-20-4]
molecular formula	$C_5H_{12}O_2$	$C_8H_{18}O_2$	$C_8H_{16}O_2$	$C_{10}H_{20}O_4$
mol wt	104.2	146.2	144.2	204.3
melting range, °C	124–130	46–55	45–50[b]	46–50
sublimation temp, °C	128			
boiling point, °C, at kPa[c]				
at 0.13			118	
at 0.45	93–94			
at 1.33			160	
at 101.3	212	236	286	290
boiling range, °C		215–235		
assay (commercial grade), wt % min	97	96	99	98
density, g/cm^3				
at 20°C	1.06		1.02	1.02
at 15°C		0.937		
pour point, supercooled, °C			10	
crystallization point, °C			35	
viscosity at 50°C, mPa·s(=cP)			675	70
heat of combustion, kJ/mol[d]	−3100	−5050	−4849[e]	
flammability				
fire point, COC, [f] °C	135	118	174	
flash point, COC, [f] °C	129	113	167	161
autoignition temp, [g] °C	388	346	316	404
heat of fusion, [h] kJ/mol[d]	21.77	8.63		
heat of vaporization, kJ/mol[d]				
at 32 kPa, [c] 170°C	67.1			
at 101.3 kPa[c]				95.6
at 101.3 kPa, [c] 204°C	56.5			
hygroscopity, [i] wt % H_2O				
at 50% rh		0.1–0.2		
at 51% rh	0.3			
at 78% rh	11.3			

[a] Mixture of isomers, cis/trans ratio (wt %) = $\sim 32/68$.

[b] Mp of cis isomer [3236-47-3] = 41°C; mp of trans isomer [3236-48-4] = 70°C.

[c] To convert kPa to mm Hg, multiply by 7.5.

[d] To convert kJ to kcal, divide by 4.184.

[e] Paar bomb.

[f] Cleveland open cup.

[g] ASTM D286.

[h] Estimated.

[i] At equilibrium; neopentyl glycol at 25–38°C, and 2, 2, 4-trimethyl-1, 3-pentanediol at 25°C.

1.4. Uses

Neopentyl glycol is used extensively as a chemical intermediate in the manufacture of polyester resins (see Alkyd resins), polyurethane polyols (see Urethane polymers), synthetic lubricants, polymeric plasticizers (qv), and other polymers. It imparts a combination of desirable properties to properly formulated esterification products, including low color, good weathering and chemical resistance, and improved thermal and hydrolytic stability.

Table 2. Toxicity Data for Various Glycols

	Structure number[a]			
Parameter	**(1)**	**(7)**	**(8)**	**(9)**
oral LD$_{50}$ (rat), mg/kg	6, 400–12, 800	3, 730	3, 200–6, 400	>3, 200
oral LD$_{50}$ (mouse), mg/kg	3, 200–6, 400	1, 600–3, 200	1, 600–3, 200	1, 600–3, 200
inhalation LC$_{50}$ (rat), [b] mg/L/6 h[b]	168	73.3[c]		>1.18[d]
dermal LD$_{50}$ (guinea pig), g/kg	14[e]	slight	>1	>1
eye irritation (rabbit)	slight[f]	moderate–strong	slight	moderate
Reference	9	10	11	12

[a] See text and Table 1.

[b] Unless otherwise noted.

[c] Also mouse.

[d] 1.18 mg/L/6 h.

[e] No skin sensitization (guinea pig).

[f] Skin irritation (rabbit) is moderate.

The weatherability and hydrolytic stability of unsaturated polyesters based on neopentyl glycol have made it a popular intermediate for use in formulations exposed to severe conditions, eg, in gel coats for cultured marble and marine applications (see Coatings, marine) (13).

Reactive saturated polyester resins (oil-free alkyds) based on neopentyl glycol are produced for use in formulating premium-quality surface coatings (14–16). These coatings exhibit excellent water, detergent, and stain resistance, and excellent weatherability, acid rain resistance, and gloss retention. (17). They may be formulated as conventional solvent-borne coatings, as high solids coatings, or as dry powders for electrostatic coatings applications.

A comparison of coatings formulations based on various glycols to determine the effects of the various glycol structures on the performance properties of the coatings has been made. Properties compared included degree of cure, flexibility, hardness, hydrolytic stability, processibility, chemical and stain resistance, and viscosity (18, 19).

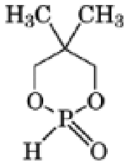
The polyurethane industry provides other uses for neopentyl glycol as an intermediate in the manufacture of hydroxy-terminated polyester polyols. Beginning with basically the same ingredients, products with a wide range of properties, varying from soft to rigid foams to elastomers (qv) and adhesives (qv) may be produced from polyols based on neopentyl glycol. This glycol also is employed to improve thermal, hydrolytic, and uv stability (20–23).

Synthetic lubricants are made with neopentyl glycol in the base-stock polyester (24). Excellent thermal stability and viscosity control are imparted to special high performance aviation lubricants by the inclusion of polyester thickening agents made from neopentyl glycol (25, 26) (see Lubrication and lubricants). Neopentyl glycol is also used to manufacture polymeric plasticizers that exhibit the improved thermal, hydrolytic, and uv stability necessary for use in some exterior applications (27).

Neopentyl glycol can be used for thermal energy storage by virtue of its solid-phase transition, which occurs at 39–41°C, a temperate range useful for solar heating and cooling (28–31).

1.5. Derivatives

A number of derivatives of neopentyl glycol have been prepared; some show promise for commercial applications.



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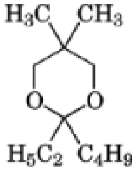

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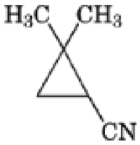

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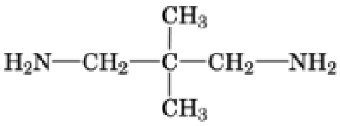
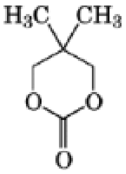


(5)

$$H_2N{-}CH_2{-}C(CH_3)_2{-}CH_2{-}NH_2$$



(6)

1.5.1. Organophosphorus Derivatives

Neopentyl glycol treated with pyridine and phosphorus trichloride in anhydrous dioxane yields the cyclic hydrogen phosphite, 5, 5-dimethyl-1, 3-dioxaphosphorinane 2-oxide **(2)** (32, 33). Compounds of this type may be useful as flameproofing plasticizers, stabilizers, synthetic lubricants, oil additives, pesticides, or intermediates for the preparation of other organophosphorus compounds (see Flame retardants; Phosphorus compounds).

1.5.2. Acetals and Ketals

Acetals of 1, 3-diols are prepared by refluxing the diol with the aldehyde in the presence of an acid catalyst, even in an aqueous medium. The corresponding ketals are more difficult to prepare in aqueous solution, but cyclic ketals of neopentyl glycol, eg, 2-butyl-2-ethyl-5, 5-dimethyl-1, 3-dioxane **(3)**, can be prepared if the water of reaction is removed azeotropically (34).

1.5.3. Cyclopropane Derivatives

2, 2-Dimethylcyclopropanenitrile [5722-11-2] **(4)** has been made by preparing the di-*p*-toluenesulfonate of neopentyl glycol and treating the diester with potassium cyanide (35).

1.5.4. Diamine

2, 2-Dimethyl-1, 3-propanediamine [7328-91-8] **(5)** has been prepared by amination of neopentyl glycol by treating the glycol with ammonia and hydrogen at 150–250°C at 10–31 MPa (1500–4500 psig) over a Ni catalyst. The diamine is useful for preparation of crystalline polyureas by reaction with diisocyanates (36).

1.5.5. Esters

Neopentyl glycol diesters are usually liquids or low melting solids. Polyesters of neopentyl glycol, and in particular unsaturated polyesters, are prepared by reaction with polybasic acids at atmospheric pressure. High molecular weight linear polyesters (qv) are prepared by the reaction of neopentyl glycol and the ester (usually the methyl ester) of a dibasic acid through transesterification (37, 38). The reaction is usually performed at elevated temperatures, *in vacuo,* in the presence of a metallic catalyst.

Cyclic carbonates are prepared in satisfactory quality for anionic polymerization by catalyzed transesterification of neopentyl glycol with diaryl carbonates, followed by tempering and depolymerization. Neopentyl carbonate (5, 5-dimethyl-1, 3-dioxan-2-one) **(6)** prepared in this manner has high purity (99.5%) and can be anionically polymerized to polycarbonates with mol wt of 35, 000 (39).

2. 2, 2, 4-Trimethyl-1, 3-Pentanediol

2, 2, 4-Trimethyl-1, 3-pentanediol **(7)** is a white, crystalline solid. It is used in surface coating and unsaturated polyester resins. It also appears promising as an intermediate for synthetic lubricants and polyurethane elastomers and foams.

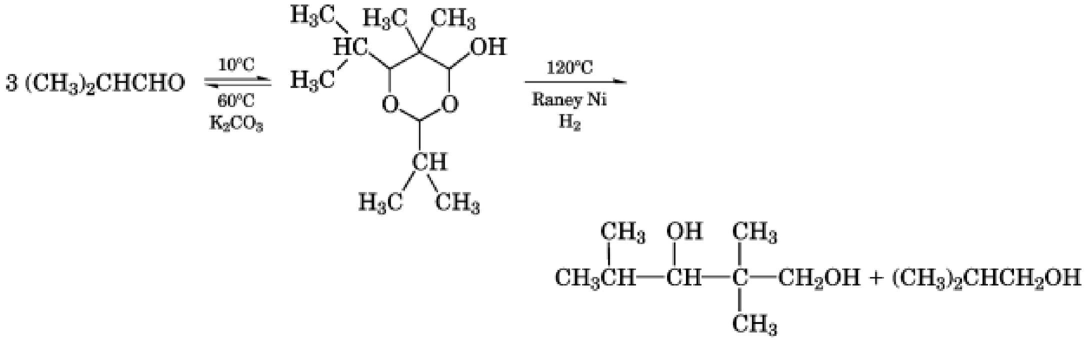
Trimethylpentanediol is soluble in most alcohols, other glycols, aromatic hydrocarbons, and ketones, but it has only negligible solubility in water and aliphatic hydrocarbons (4).

2.1. Chemical Properties

Trimethylpentanediol, with a primary and a secondary hydroxyl group, enters into reactions characteristic of other glycols. It reacts readily with various carboxylic acids and diacids to form esters, diesters, and polyesters (40). Some organometallic catalysts have proven satisfactory for these reactions, the most versatile being dibutyltin oxide. Several weak bases such as triethanolamine, potassium acetate, lithium acetate, and borax are effective as stabilizers for the glycol during synthesis (41).

2.2. Manufacture and Processing

2, 2, 4-Trimethyl-1, 3-pentanediol can be produced by hydrogenation of the aldehyde trimer resulting from the aldol condensation of isobutyraldehyde [78-84-2].



(7)

Eastman Chemical Co. is the only manufacturer of this glycol. Prices in 1993 were \$1.20/kg for bulk quantities.

2.3. Toxicity

Acute toxicity data for this glycol **(7)** are reported in Table 2.

2.4. Uses

The versatility of trimethylpentanediol as an intermediate is reflected by the diversity of its commercial applications.

Unsaturated polyesters derived from trimethylpentanediol are characterized by a low exotherm, low shrink curing, and a product that has good electrical properties and excellent hydrolytic stability and chemical resistance (10, 19, 40, 41). These unsaturated resins exhibit low viscosity, low density, and good glass–fiber or filler wetting (10, 41). They are resistant to hydrolytic action, probably because of the protection of the ester linkage by the pendent methyl groups, and by virtue of the lesser number of ester groups in a given weight of material than would be afforded with lower molecular weight glycols (42). Chemically resistant applications where trimethylpentanediol-based unsaturated resins are used include reinforced polyester storage tanks and pipelines for hot, concentrated acids, some solvents, mild bases, hypochlorite solution, and ammonia (see Polyesters, unsaturated).

Saturated polyester resins based on trimethylpentanediol are used in various coating applications, most notably in water-borne (43) and high solids coatings (44, 45). Resins manufactured with this diol are characterized by low viscosities, which permits formulation of enamels with 85% nonvolatiles when sprayed. Such formulations are cross-linked with isocyanates or melamines to give premium coatings useful for industrial applications, appliance coil coatings, and the like (44–46). Other saturated polyester resins based on trimethylpentanediol are useful in high gloss hypoallergenic nail polishes (47) (see Polyesters, thermoplastic).

The monoisobutyrate ester of trimethylpentanediol is especially useful as a coalescing aid in flat and semigloss (48) latex pain formulations (see Paint). This product is commercially available from Eastman as Texanol ester alcohol.

The diisobutyrate ester of trimethylpentanediol is an economical, low color primary plasticizer for use in surface coatings, vinyl flooring, moldings, and other vinyl products. This diester is commercially available from Eastman as Kodaflex TXIB plasticizer (49).

Various other diesters, mixed esters, and polyesters of trimethylpentanediol are useful as monomeric or polymeric plasticizers for coatings and plastic film and sheeting (49). They are compatible with, and useful in, cellulosics, vinyls, polystyrenes, and some other plastics.

Trimethylpentanediol is used in hard-surface cleaners as a coupling agent (50) and in temporary or semipermanent hair dyes (51). Other applications involving trimethylpentanediol, or a derivative, are in urethane elastomers (52), in foams (53), as a reactive diluent in urethane coatings (see Urethane polymers) (54), as a sound-insulating, glass–laminate adhesive (see Adhesives) (55), as a bactericide–fungicide (56), and as a cross-linking agent in poly(vinyl chloride) adhesive (57).

2.5. Derivatives

2.5.1. Esters

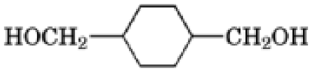
The monoisobutyrate ester of 2, 2, 4-trimethyl-1, 3-pentanediol is prepared from isobutyraldehyde in a Tishchenko reaction (58, 59). Diesters, such as trimethylpentane dipelargonate (2, 2, 4-trimethylpentane 1, 3-dinonanoate), are prepared by the reaction of 2 mol of the monocarboxylic acid with 1 mol of the glycol at 150–200°C (60, 61). The lower aliphatic carboxylic acid diesters of trimethylpentanediol undergo pyrolysis to the corresponding ester of 2, 2, 4-trimethyl-3-penten-1-ol (62). These unsaturated esters reportedly can be epoxidized by peroxyacetic acid (63).

2.5.2. Ketals

Trimethylpentanediol reportedly forms a cyclic ketal by heating it with benzophenone in the presence of sulfonic acid catalysts at reflux temperatures in toluene (64). These are said to be useful as aprotic solvents for ink-jet printing and as inflammation inhibitors for cosmetic preparations (65).

3. 1, 4-Cyclohexanedimethanol

1, 4-Cyclohexanedimethanol, 1, 4-dimethylolcyclohexane, or 1, 4-bis(hydroxymethyl) cyclohexane **(8)**, is a white, waxy solid. The commercial product consists of a mixture of cis and trans isomers (6). This diol is used in the manufacture of polyester fibers (qv) (64), high performance coatings, and unsaturated polyester molding and laminating resins (5).



(8)

1, 4-Cyclohexanedimethanol is miscible with water and low molecular weight alcohols and appreciably soluble in acetone. It has only negligible solubility in hydrocarbons and diethyl ether (6).

3.1. Chemical Properties

The chemistry of 1, 4-cyclohexanedimethanol is characteristic of general glycol reactions; however, its two primary hydroxyl groups give very rapid reaction rates, especially in polyester synthesis.

3.2. Manufacture

The manufacture of 1, 4-cyclohexanedimethanol can be accomplished by the catalytic reduction under pressure of dimethyl terephthalate in a methanol solution (47, 65). This glycol also may be prepared by the depolymerization and catalytic reduction of linear polyesters that have alkylene terephthalates as primary constituents.

Poly(ethylene terephthalate) may be hydrogenated in the presence of methanol under pressure and heat to give good yields of the glycol (see Polyesters) (66, 67).

1, 4-Cyclohexanedimethanol is produced commercially by Eastman Chemical Co. The price within the United States in bulk truckloads as of June 1993 was 99% purity, $2.49/kg.

3.3. Toxicity

Acute toxicity data are reported in Table 2 (11).

3.4. Uses

The most important application for 1, 4-cyclohexanedimethanol is in the manufacture of linear polyesters for use as polyester fibers such as the Kodel polyester fibers (68) (see Fibers, polyester). Compared with fibers made from poly(ethylene terephthalate), fibers made from poly(1, 4-cyclohexanedimethanol terephthalate) have lower densities and higher melting points. Linear polyesters produced from dicarboxylic acids, eg, terephthalic acid, generally have greater hydrolytic stability and better electrical properties than similar polyesters made from other glycols. The high dielectric strength, good dielectric constant, and low dielectric loss make them useful for many electrical applications, eg, in capacitors, wire coatings, and magnetic tape coatings (69, 70).

Unsaturated resins based on 1, 4-cyclohexanedimethanol are useful in gel coats and in laminating and molding resins where advantage is taken of the properties of very low water absorption and resistance to boiling water (6). Thermal stability is imparted to molding resins, both thermoplastic (71, 72) and thermoset (73–76), enabling retention of physical and electrical properties at elevated temperatures (77). Additionally, resistance to chemical and environmental exposure is characteristic of products made from these resins (78).

High performance polyester enamels are manufactured from saturated resins containing 1, 4-cyclohexanedimethanol. Such enamels may be formulated as electrostatically applied powder coatings (79–82), water-borne (83, 84), cationic electrodeposition coatings (85), or solvent-based coatings (see Coating processes). These coatings are characterized by an extremely hard, durable, stain- and detergent-resistant finish (88–88). Other polyester resins based on 1, 4-cyclohexanedimethanol are also used as plasticizers (89), in hot-melt adhesives (90, 91), in elastomers (92, 93), and urethane elastomeric coatings (94) (see Coatings).

Another area in which 1, 4-cyclohexanedimethanol is commercially important is in the manufacture of polyurethane foams (see Foamed plastics). The two primary hydroxyl groups provide fast reaction rates with diisocyanates, which makes this diol attractive for use as a curative in foams. It provides latitude in improving physical properties of the foam, in particular the load-bearing properties. Generally, the ability to carry a load increases with the amount of 1, 4-cyclohexanedimethanol used in producing the high resilience foam (95). Other polyurethane derivatives of 1, 4-cyclohexanedimethanol include elastomers useful for synthetic rubber products with a wide range of hardness and elasticity (96).

3.5. Derivatives

3.5.1. Mixed Phosphonate Esters

Unsaturated, mixed phosphonate esters have been prepared from monoesters of 1, 4-cyclohexanedimethanol and unsaturated dicarboxylic acids. For example, maleic anhydride reacts with this diol to form the maleate, which is treated with benzenephosphonic acid to yield an unsaturated product. These esters have been used as flame-retardant additives for thermoplastic and thermosetting resins (97).

3.5.2. Diesters

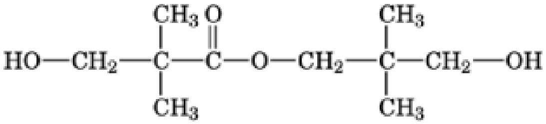
Diesters prepared from the diol and monocarboxylic acids are useful as antioxidants (qv) for polypropylene (98), and as plasticizers (qv).

3.5.3. Polyesters

Polyesters containing carbonate groups have been prepared from this diol (see Polycarbonates) (99). Films of this polymer, formed from an acetone or ethyl acetate solution, exhibit excellent adhesive properties.

4. Hydroxypivalyl Hydroxypivalate

Hydroxypivalyl hydroxypivalate or 3-hydroxy-2, 2-dimethylpropyl 3-hydroxy-2, 2-dimethylpropionate **(9)** is a white crystalline solid at room temperature. It is used to manufacture polyester resins for use in surface coatings where good resistance to weathering and acid rain are of particular importance (6).



$$HO{-}CH_2{-}\overset{\overset{\displaystyle CH_3}{|}}{\underset{\underset{\displaystyle CH_3}{|}}{C}}{-}\overset{\overset{\displaystyle O}{\|}}{C}{-}O{-}CH_2{-}\overset{\overset{\displaystyle CH_3}{|}}{\underset{\underset{\displaystyle CH_3}{|}}{C}}{-}CH_2{-}OH$$

(9)

Hydroxypivalyl hydroxypivalate is soluble in most alcohols, ester solvents, ketones, and aromatic hydrocarbons. It is partially soluble in water (6).

4.1. Chemical Properties

Both hydroxy groups on hydroxypivalyl hydroxypivalate are primary, which results in rapid reactions with acids during esterification. The absence of hydrogens on the carbon atom beta to the hydroxyls is a feature this glycol shares with neopentyl glycol, resulting in excellent weatherability. The relatively high molecular weight of this glycol requires lower levels of aromatic acid to produce polyester resins, thus contributing to the improved weatherability of polyesters made from this glycol.

4.2. Manufacture

Hydroxypivalyl hydroxypivalate may be produced by the esterification of hydroxypivalic acid with neopentyl glycol or by the intermolecular oxidation–reduction (Tishchenko reaction) of hydroxypivaldehyde using an aluminum alkoxide catalyst (100, 101).

Eastman Chemical Co., BASF, Mitsubishi Gas, and Union Carbide are manufacturers of this glycol. The U.S. price in June 1993 was $2.97/kg.

4.3. Toxicity

Acute toxicity data for **(9)** appear in Table 2.

4.4. Uses

Saturated polyesters made from hydroxypivalyl hydroxypivalate are most often used for formulating coatings which have very low initial color and which retain the low color exposure to weathering. The most typical example is in clear topcoat useful in automotive finishes (102). This glycol is often used as a partial replacement for neopentyl glycol in polyester resins to provide better resin solubility, reduced crystallinity, lower glass-transition temperatures, and lower melt viscosity. These characteristics make this glycol particularly useful for resins used in coil coatings, powder coatings, waterborne coatings, and unsaturated polyester gel coats (6, 103).

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