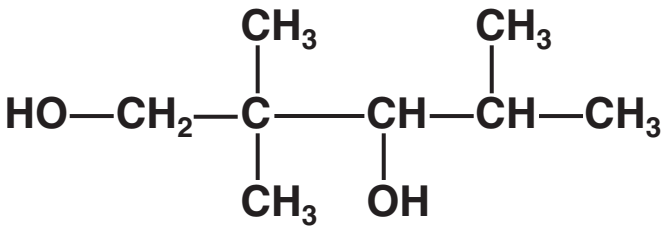
TMPD Glycol
Polyester-Coating Resin Synthesis and Processing Procedures



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

CAS: 144-19-4

Saturated polyester resins based on *Eastman TMPD* glycol exhibit excellent performance in high-solids and waterborne coatings. This glycol imparts a lower solution viscosity than other commonly used diols, which is desirable for high-solids coatings. It also improves such properties as corrosion resistance, stain resistance, detergent resistance, and hydrolytic stability. Polyester polyols based on *TMPD* glycol provide desirable properties to urethane coatings.

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Introduction

The branched, asymmetrical structure of *TMPD* glycol permits the preparation of noncrystalline, low-density polymers for use in coatings applications. The high molecular weight and branched structure of *TMPD* glycol result in bulky, asymmetrical polymers with a minimum amount of hydrogen bonding between chains. This characteristic allows for the formulation of high-solids coatings with lower viscosities and excellent flow properties.

The sterically hindered secondary hydroxyl group and the hydrophobic nature of this glycol combine to contribute outstanding aqueous, alkali, corrosion, and detergent resistance to polyester enamels and improve the package stability of waterborne enamels. In addition, *TMPD* glycol has a high boiling point (215°C).

General Synthesis Techniques

Structural characteristics of the *TMPD* glycol molecule, which impart improved performance to enamel coatings, also require specialized synthesis procedures. Each *TMPD* glycol molecule contains one secondary hydroxyl group that is sterically hindered. Attempts to esterify this secondary hydroxyl group without proper precautions may result in long reaction times, highly colored resins, and excessive glycol loss into the distillate stream.

The following general guidelines should be observed in the preparation of all polyester resins based on *TMPD* glycol. The synthesis techniques for waterborne and high-solids polyester coating resins, as well as polyester polyol preparation techniques, will be discussed separately.

Avoid rapid heating of the reactants

The reaction temperature should be held between 180° and 200°C during the first 30% to 60% of the reaction. After that, the temperature can be increased to a maximum of 215°C.

Limit maximum reaction temperature to 215°C

A balance between short reaction time and low glycol loss is generally attained by limiting the reaction temperature to 215°C.

Reduce reaction time by use of a catalyst

The reaction time for the preparation of polyester resins can be shortened by using organometallic catalysts. Organic compounds of zinc, titanium, and tin have proved effective. The most versatile of these are the organotin catalysts, which are generally used in an amount equal to 0.1% to 0.5% (approximately 0.06% to 0.28% based on the active metal) of the total reactor charge. *Fascat* 4100 butylstannoic acid (Atochem) has proved to be very efficient in this application. Compared with the reaction time required to prepare a typical uncatalyzed polyester resin, the use of organotin catalysts in proper amounts can reduce the reaction time of the resin by as much as 50%.

Glycol rearrangement

The rearrangement of *TMPD* glycol to form volatile ketones and aldehydes is influenced by the type of dibasic acid used to esterify the glycol, the reaction temperature, and the time required for the reaction. Some glycol rearrangement will occur during resin synthesis but can be minimized by following the suggestions listed previously. Typical levels of *TMPD* lost

through this arrangement mechanism are in the order of 1% to 3% of the initial charge of *TMPD*. Direct reaction of *TMPD* with anhydrides can cause significant glycol losses from rearrangement and is not suggested.

React under an inert atmosphere

Low-color polyester resins must be prepared under an inert atmosphere such as nitrogen.

Solvent–Based Resin Synthesis Techniques

High-solids resins using *TMPD* glycol for reaction with polymeric melamines

Polyester resins based on *TMPD* glycol for high-solids, melamine cross-linked coatings are prepared using the one- or two-stage fusion process. The reaction is catalyzed with 0.1% to 0.5% (based on reactor charge) organotin catalyst; a one- to three-hour up-heat time is used to a maximum cook temperature of 215°C. The polyester resins are processed to an acid number range of 5 to 15 and an average molecular weight between 600 and 1,500. Resin HS-3-6T (Eastman Publication N-306) is a typical high-solids resin based on *TMPD* glycol for melamine cross-linking.

Resins based on *TMPD* glycol for reaction with polyisocyanates

Polyester-polyol resins based on *TMPD* glycol for high-solids and conventional urethane coatings are prepared using the one-stage organotin-catalyzed reaction. However, in the preparation of resins for reaction with isocyanates, the level of catalyst is held to a minimum, usually 0.03% to 0.05%. The polyester-polyol resins are reacted to an acid number of less than 2.

Waterborne Resin Synthesis Techniques

TMPD glycol/isophthalic acid resins

These resins are prepared by a two-stage organotin-catalyzed reaction. The *TMPD* glycol, triol, and IPA are melted and reacted at a maximum temperature of 215°C until no unreacted IPA particles are visible in the resin, usually below an acid number of 20. The resin is then cooled to 150°C; the aliphatic acid (often adipic acid) added; and the resin reacted at 215°C to a resin acid number between 50 and 60. This two-stage process eliminates the need for filtering unreacted IPA from the resin.

TMPD glycol/trimellitic anhydride (TMA) resins

These are prepared by a two-stage organotin-catalyzed reaction. *TMPD* glycol and IPA are reacted at 195° to 215°C until an acid number of <10 is reached. The resin is cooled to 150°C, and the TMA and adipic acid are added. The resin is processed at a maximum of 195°C to an acid number between 50 and 60. Processing time is usually 5–7 hours.

Polyester Synthesis Equipment

Because of individual preferences and the availability of equipment, there are numerous variations among laboratories in the methods used to prepare saturated polyesters. Eastman Publication N-345 describes the equipment used in Eastman laboratories to prepare saturated polyester resins based on *TMPD* glycol.

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