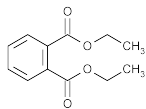


Diethyl Phthalate



$C_{12}H_{14}O_4$ 222.24
1,2-Benzenedicarboxylic acid, diethyl ester.
Diethyl phthalate [84-66-2].

» Diethyl Phthalate contains not less than 98.0 percent and not more than 102.0 percent of $C_{12}H_{14}O_4$, calculated on the anhydrous basis.

Caution—Avoid contact.

Packaging and storage—Preserve in tight containers.

USP Reference standards (11)—

USP Diethyl Phthalate RS

Identification, Infrared Absorption (197F), on undried specimen.

Specific gravity (841): between 1.118 and 1.122, at 20°.

Refractive index (831): between 1.500 and 1.505, at 20°.

Acidity—Mix 20.0 g with 50 mL of alcohol that previously has been neutralized to a phenolphthalein endpoint, and titrate with 0.10 N sodium hydroxide to a phenolphthalein endpoint: not more than 0.50 mL is required for neutralization.

Water, Method I (921): not more than 0.2%.

Residue on ignition (281)—Heat gently about 10 g, accurately weighed, until the liquid has evaporated, and ignite the residue to constant weight. Not more than 0.02% is found.

Assay—Transfer about 1.5 g of Diethyl Phthalate, accurately weighed, to a flask, add 50.0 mL of 0.5 N alcoholic potassium hydroxide VS, attach a reflux condenser to the flask, and boil on a water bath for 1 hour. Add 20 mL of water, then add phenolphthalein TS, and titrate the excess potassium hydroxide with 0.5 N hydrochloric acid VS. Perform a blank determination (see *Residual Titrations* under *Titrimetry* (541)). Each mL of 0.5 N potassium hydroxide is equivalent to 55.56 mg of $C_{12}H_{14}O_4$.

Diethylene Glycol Monoethyl Ether



$C_6H_{14}O_3$ 134.17

DEFINITION

Diethylene Glycol Monoethyl Ether contains NLT 99.0% and NMT 101.0% of $C_6H_{14}O_3$. It is produced by condensation of ethylene oxide and alcohol, followed by distillation.

IDENTIFICATION

- **A. INFRARED ABSORPTION** (197F): Potassium bromide plates being used
- **B.** The retention time of the major peak of the *Sample solution* corresponds to that of the *System suitability solution*, as obtained in the *Assay*.

ASSAY

Change to read:

PROCEDURE

▲**Sample:** Diethylene Glycol Monoethyl Ether (neat)▲NF30

System suitability solution: 1 mg/mL each of 2-methoxyethanol, 2-ethoxyethanol, ethylene glycol, diethylene glycol, and USP Diethylene Glycol Monoethyl Ether RS in methanol

Chromatographic system

(See *Chromatography* (621), *System Suitability*.)

Mode: GC

Detector: Flame ionization

Column: 0.32-mm × 30-m fused-silica bonded with a 1.0-μm layer of phase G46

Temperature

Injector: 250°

Detector: 275°

Column: See the temperature program table below.

Initial Temperature (°)	Temperature Ramp (°/min)	Final Temperature (°)	Hold Time at Final Temperature (min)
120	—	120	1
120	12	225	2

Carrier gas: Helium

Flow rate: 2.2 mL/min

Injection size: 0.5 μL

System suitability

Sample: *System suitability solution*

[NOTE—The relative retention times for 2-methoxyethanol, 2-ethoxyethanol, ethylene glycol, diethylene glycol monoethyl ether, and diethylene glycol are about 0.40, 0.43, 0.50, 0.93, and 1.0, respectively.]

Suitability requirements

Resolution: NLT 2.0 between 2-ethoxyethanol and ethylene glycol

Relative standard deviation: NMT 2.0%, determined from diethylene glycol monoethyl ether

Analysis

Sample: Diethylene Glycol Monoethyl Ether (neat)
Calculate the percentage of diethylene glycol monoethyl ether ($C_6H_{14}O_3$) in the portion of Diethylene Glycol Monoethyl Ether taken:

$$\text{Result} = (r_U/r_T) \times 100$$

r_U = peak response for diethylene glycol monoethyl ether

r_T = sum of the responses of all the peaks

Acceptance criteria: 99.0%–101.0%

IMPURITIES

Organic Impurities

PROCEDURE 1: LIMIT OF FREE ETHYLENE OXIDE

Acetaldehyde solution: 10 μg/mL of acetaldehyde.

[NOTE—Prepare fresh just before use.]

Ethylene oxide stock solution

[CAUTION—Ethylene oxide is toxic and flammable.

Prepare these solutions in a well-ventilated fume hood, using great care. Protect both hands and face by wearing polyethylene protective gloves and an appropriate face mask.]

[NOTE—Before using the polyethylene glycol 200 in this test, remove any volatile components from it by placing 500 mL of the polyethylene glycol 200 in a 1000-mL round-bottom flask, attaching the flask to a rotary evaporator, and evaporating at a temperature of 60° at a pressure of 1.5–2.5 kPa for 6 h.]

Fill a chilled pressure bottle with liquid ethylene oxide, and store in a freezer when not in use. Use a small piece of