Diethyltoluamide (Diethyltoluamidum)

C₁₂H₁₇NO

**Relative molecular mass.** 191.3

**Chemical name.** N,N-Diethyl-m-toluamide; N,N-diethyl-3-methylbenzamide; CAS Reg. No. 134-62-3.

**Description.** Colourless or faintly yellow liquid.

**Solubility.** Practically immiscible in water and glycerol R; miscible with ethanol (~750 g/l) TS and ether R.

**Category.** Insect repellent.

**Storage.** Diethyltoluamide should be kept in a tightly closed container.

**Additional information.** CAUTION: Diethyltoluamide is an irritant to eyes and mucous membranes.

**Requirements**

Diethyltoluamide contains not less than 97.0% and not more than 103.0% of C₁₂H₁₇NO, calculated with reference to the anhydrous substance.

**Identity tests**

• Either test A alone or tests B, C, and D may be applied.

A. Carry out the examination as described under 1.7 Spectrophotometry in the infrared region. The infrared absorption spectrum is concordant with the spectrum obtained from diethyltoluamide RS or with the reference spectrum of diethyltoluamide.

B. Refractive index, n_D²₀ = 1.520 - 1.524.

C. To about 2 mL, add 25 mL of hydrochloric acid (~250 g/l) TS and heat under a reflux condenser for 1 hour. Neutralize the solution with sodium hydroxide (~200 g/l) TS, cool, and extract with three quantities, each of 30 mL, of ether R. (Keep the aqueous layer for test D.) Carefully evaporate the ether layer to dryness on a water-bath, and dissolve the residue in 5mL of sodium nitrite (100 g/l) TS. Allow to stand at 5 °C for 10 minutes, add 10 mL of water, and extract with 20 mL of ether R. Evaporate the ether layer and add to the residue 1.0 g of phenol R. Cool and add about 1 mL of sulfuric acid (~1760 g/l) TS; an intense green solution is produced. Pour the mixture into water; the colour turns to red. Add sodium hydroxide (~80 g/l) TS; the colour changes to green.

D. Acidify the aqueous layer obtained in test C with hydrochloric acid (~70 g/l) TS, extract with two quantities, each of 20 mL of ether R, and carefully evaporate the ether layer. Dry the residue at 60 °C; the melting temperature of the residue is about 108 °C.

**Mass density.** ρ₂₀ = 0.996-1.002.

**Sulfated ash.** Not more than 1.0 mg/g.

**Water.** Determine as described under 2.8 Determination of water by the Karl Fischer method. Method A, using about 0.5 g of the substance; the water content is not more than 5.0 mg/g.

**Acidity.** Dissolve 10.0 g in 50 mL of neutralized ethanol TS, titrate with sodium hydroxide (0.01 mol/l) VS using phenolphthalein/ethanol TS as indicator; not more than 4.0 mL of sodium hydroxide (0.01 mol/l) VS is required to obtain the midpoint of the indicator (pink).

**Assay.** Carry out Method A as described under 2.10 Determination of nitrogen, using about 0.3 g, accurately weighed, and 7 mL of nitrogen-free sulfuric acid (~1760 g/l) TS, and proceed with the distillation. Titrate with sulfuric acid (0.05 mol/l) VS using methyl red/ethanol TS as indicator. Repeat the procedure without the Diethyltoluamide being examined and make any necessary corrections.
Each mL of sulfuric acid (0.05 mol/l) VS is equivalent to 19.13mg of $\text{C}_{12}\text{H}_{17}\text{NO}$. 