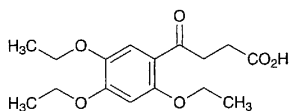


Trepibutone

トレピブトン



$C_{16}H_{22}O_6$: 310.34

4-Oxo-4-(2,4,5-triethoxyphenyl)butanoic acid
[41826-92-0]

Trepibutone, when dried, contains not less than 98.5% of $C_{16}H_{22}O_6$.

Description Trepibutone occurs as white to yellowish white crystals or crystalline powder. It is odorless, and is tasteless or has a slight, characteristic aftertaste.

It is soluble in acetone, sparingly soluble in ethanol (95), slightly soluble in diethyl ether, and practically insoluble in water.

It dissolves in sodium hydroxide TS.

Identification (1) Determine the absorption spectrum of a solution of Trepibutone in diluted dilute sodium hydroxide TS (1 in 10) (1 in 100,000) as directed under the Ultraviolet-visible Spectrophotometry, and compare the spectrum with the Reference Spectrum: both spectra exhibit similar intensities of absorption at the same wavelengths.

(2) Determine the spectrum of a solution of Trepibutone in deuterated chloroform for the nuclear magnetic resonance spectroscopy (1 in 10), using tetramethylsilane for the nuclear magnetic resonance spectroscopy as an internal reference compound, as directed under the Nuclear Magnetic Resonance Spectroscopy (1H): it exhibits a sharp multiple signal A at around δ 1.5 ppm, a triplet signal B at around δ 2.7 ppm, a triplet signal C at around δ 3.3 ppm, a multiple signal D at around δ 4.2 ppm, a sharp single signal E at around δ 6.4 ppm, a sharp single signal F at around δ 7.4 ppm, and a single signal G at around δ 10.5 ppm. The ratio of integrated intensity of each signal, A:B:C:D:E:F:G, is about 9:2:2:6:1:1:1.

Melting point 146 – 150°C

Purity (1) Chloride—Dissolve 0.5 g of Trepibutone in 30 mL of acetone, and add 6 mL of dilute nitric acid and water to make 50 mL. Perform the test using this solution as the test solution. Prepare the control solution as follows: to 0.30 mL of 0.01 mol/L hydrochloric acid VS add 30 mL of acetone, 6 mL of dilute nitric acid and water to make 50 mL (not more than 0.021%).

(2) Heavy metals—Proceed with 1.0 g of Trepibutone according to Method 2, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 20 ppm).

(3) Related substances—Dissolve 0.10 g of Trepibutone in 10 mL of acetone, and use this solution as the sample solution. Pipet 2 mL of the sample solution, add acetone to make exactly 100 mL. To exactly 10 mL of this solution add acetone to make exactly 100 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 10 μ L

each of the sample solution and the standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of isopropylether, acetone, water and formic acid (100:30:3:3) to a distance of about 10 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm): the spots other than the principal spot from the sample solution are not more intense than the spot from the standard solution.

Loss on drying Not more than 0.5% (1 g, 105°C, 4 hours).

Residue on ignition Not more than 0.10% (1 g).

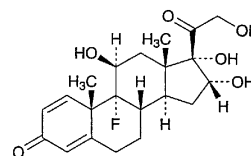
Assay Weigh accurately about 0.5 g of Trepibutone, previously dried, dissolve in 50 mL of ethanol (95), add 50 mL of water, and titrate with 0.1 mol/L sodium hydroxide VS (indicator: 5 drops of phenolphthalein TS). Perform a blank determination, and make any necessary correction.

Each mL of 0.1 mol/L sodium hydroxide VS
= 31.035 mg of $C_{16}H_{22}O_6$

Containers and storage Containers—Tight containers.
Storage—Light-resistant.

Triamcinolone

トリアムシロン



$C_{21}H_{27}FO_6$: 394.43

9-Fluoro-11 β ,16 α ,17,21-tetrahydroxypregna-1,4-diene-3,20-dione [124-94-7]

Triamcinolone, when dried, contains not less than 97.0% and not more than 103.0% of $C_{21}H_{27}FO_6$.

Description Triamcinolone occurs as a white, crystalline powder. It is odorless.

It is freely soluble in *N,N*-dimethylformamide, slightly soluble in methanol, in ethanol (95) and in acetone, and practically insoluble in water, in 2-propanol and in diethyl ether.

Melting point: about 264°C (with decomposition).

Identification (1) Dissolve 1 mg of Triamcinolone in 6 mL of ethanol (95), add 5 mL of 2,6-di-*tert*-butylcresol TS and 5 mL of sodium hydroxide TS, and heat on a water bath for 30 minutes under a reflux condenser: a red-purple color develops.

(2) Add 5 mL of water and 1 mL of Fehling's TS to 0.01 g of Triamcinolone, and heat: a red precipitate is produced.

(3) Proceed with 0.01 g of Triamcinolone as directed under Oxygen Flask Combustion Method, using a mixture of 0.5 mL of 0.01 mol/L sodium hydroxide TS and 20 mL of water as the absorbing liquid. When combustion is completed, shake vigorously so as to absorb the gas evolved: the solution responds to the Qualitative Tests for fluoride.