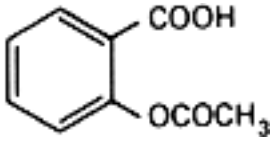
Acetylsalicylic acid (Acidum acetylsalicylicum)

Molecular formula. $C_9H_8O_4$

Relative molecular mass. 180.2

Graphic formula.



Chemical name. 2-(Acetyloxy)benzoic acid; 2-acetoxybenzoic acid; CAS Reg. No. 50-78-2.

Description. Colourless crystals or a white, crystalline powder; odourless or almost odourless.

Solubility. Soluble in about 300 parts of water; freely soluble in ethanol (~750 g/l) TS; soluble in ether R.

Category. Analgesic; antipyretic.

Storage. Acetylsalicylic acid should be kept in a tightly closed container, protected from light.

Additional information. Even in the absence of light, Acetylsalicylic acid is gradually degraded on exposure to a humid atmosphere, the decomposition being faster at higher temperatures.

Requirements

Definition. Acetylsalicylic acid contains not less than 99.0% and not more than 100.5% of $C_9H_8O_4$, calculated with reference to the dried substance.

Identity tests

A. Heat 0.05 g in 2 mL of water for several minutes, cool, and add 1-2 drops of ferric chloride (25 g/l) TS; a violet-red colour is produced, which does not change on the addition of ethanol (~750 g/l) TS.

B. Boil 0.2 g with 4 mL of sodium hydroxide (~80 g/l) TS for about 3 minutes, cool, and add 5 mL of sulfuric acid (~100 g/l) TS; a white, crystalline precipitate is formed. Filter (keep the filtrate for test C), wash the precipitate with water and dry at 105°C. Melting temperature, about 159°C (salicylic acid).

C. Heat the filtrate from test B with 2 mL of ethanol (~750 g/l) TS and 2 mL of sulfuric acid (~1760 g/l) TS; ethyl acetate, perceptible by its odour (proceed with caution), is produced.

Heavy metals. Use 1.0 g and 25 mL of acetone R for the preparation of the test solution as described under 2.2.3 Limit test for heavy metals, procedure 2; determine the heavy metal content according to Method A; not more than 20 µg/g.

Solution in ethanol. A solution of 1.0 g in 10 mL of ethanol (~750 g/l) TS is clear and colourless.

Solution in alkali. A solution of 0.5 g in 10 mL of warm sodium carbonate (50 g/l) TS is clear.

Sulfated ash. Not more than 1.0 mg/g.

Loss on drying. Dry to constant weight at ambient temperature under reduced pressure (not exceeding 0.6 kPa or about 5 mm of mercury) over silica gel, desiccant, R; it loses not more than 5.0 mg/g.

Salicylic acid. Dissolve 0.50 g in sufficient ethanol (~750 g/L) TS to produce 25 mL and transfer 10 mL to a comparison tube. Dissolve separately 0.040 g of salicylic acid R in sufficient water to produce 100 mL. Transfer 1 mL of this solution to a second comparison tube and add to it 10 mL of ethanol (~750 g/L) TS. Add water to both tubes to make 50 mL, followed by 1 mL of ferric ammonium sulfate TS1, mix and allow to stand for 1 minute. The violet colour of the test solution is not more intense than that of the reference solution when compared as described under 1.11.1 Colour of liquids; the salicylic acid content is not more than 2.0 mg/g.

Assay. To about 0.20 g, accurately weighed, add 50 mL of carbonate-free sodium hydroxide (0.1 mol/l) VS, and boil under reflux for 10 minutes. Titrate the excess of alkali with sulfuric acid (0.05 mol/l) TS, using phenolphthalein/ethanol TS as indicator. Repeat the operation without the substance being examined and make any necessary corrections. Each mL of carbonate-free sodium hydroxide (0.1 mol/l) VS is equivalent to 9.008 mg of $C_9H_8O_4$.