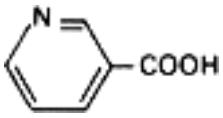
Nicotinic acid (Acidum nicotinicum)

Molecular formula. $C_6H_5NO_2$

Relative molecular mass. 123.1

Graphic formula.



Chemical name. 3-Pyridinecarboxylic acid; CAS Reg. No. 59-67-6.

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

Solubility. Sparingly soluble in water; freely soluble in boiling water; soluble in 100 parts of ethanol (~750 g/l) TS; practically insoluble in ether R.

Category. Component of vitamin B complex; vasodilator.

Storage. Nicotinic acid should be kept in a well-closed container, protected from light.

Requirements

Definition. Nicotinic acid contains not less than 99.0% and not more than 101.0% of $C_6H_5NO_2$, calculated with reference to the dried substance.

Identity tests

• Either test A alone or all 3 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 nicotinic acid RS or with the *reference spectrum* of nicotinic acid.

B. Heat 0.1 g with 0.4 g of anhydrous sodium carbonate R; pyridine, perceptible by its odour, is produced.

C. Dissolve 10 mg in 10 mL of water. To 2 mL add 2 mL of thiocyanate reagent, obtained by adding, drop by drop, ammonium thiocyanate (0.1 mol/l) VS to bromine TS1 until the yellow coloration disappears. Then add 3 mL of aniline (25 g/l) TS and shake; a yellow colour is produced.

D. Melting temperature, about 235°C.

Heavy metals. Use 1.0 g for the preparation of the test solution as described under 2.2.3 Limit test for heavy metals, Procedure 3; determine the heavy metals content according to Method A; not more than 20 µg/g.

Chlorides. Dissolve 1.25 g in a mixture of 2 mL of nitric acid (~130 g/l) TS and 20 mL of water, filter if necessary, and proceed as described under 2.2.1 Limit test for chlorides; the chloride content is not more than 0.2 mg/g.

Sulfated ash. Not more than 1.0 mg/g.

Loss on drying. Dry to constant weight at 105°C; it loses not more than 10 mg/g.

pH value. pH of a 13 mg/mL solution, 3.0-3.5.

Related substances. Carry out the test as described under *1.14.1 Chromatography*, Thin-layer chromatography, using silica gel R2 as the coating substance and a mixture of 85 volumes of 1-propanol R, 10 volumes of anhydrous formic acid R, and 5 volumes of water as the mobile phase. For the test solution, dissolve 75 mg in 5.0 mL of water with gentle heating; this constitutes solution A. Prepare a reference solution containing 0.12 mg/mL of nicotinic acid RS; this constitutes solution B. Apply to the plate 10 µl of solution A using two 5-µl aliquots, allowing the plate to dry in a current of cold air after the first application; then apply separately 5 µl of solution B. After removing the plate from the chromatographic chamber, allow it to dry in a current of warm air, and examine the chromatogram in ultraviolet light (254 nm). Beside the principal spot, not more than 3 spots are obtained with solution A, and they are not more intense than the spot obtained with solution B.

Assay. Dissolve about 0.25 g, accurately weighed, in 50 mL of carbon-dioxide-free water R, and titrate with carbonate-free sodium hydroxide (0.1 mol/l) VS, using phenolphthalein/ethanol TS as indicator. Each mL of carbonate-free sodium hydroxide (0.1 mol/l) VS is equivalent to 12.31 mg of $C_6H_5NO_2$.