CALCIUM SACCHARIN

Prepared at the 24th JECFA (1980), published in FNP 17 (1980) and in FNP 52 (1992). Metals and arsenic specifications revised at the 57th JECFA (2001). An ADI of 0-5 mg/kg bw for saccharin and its calcium, potassium and sodium salts was established at the 41st JECFA (1993).

SYNONYMS

INS No. 954(ii)

DEFINITION

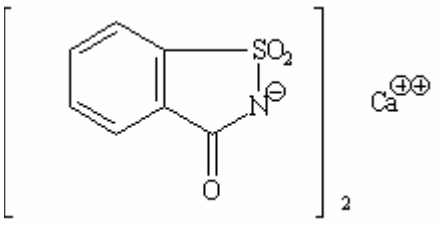
Chemical names

Calcium salt hydrate (2:7) of 1,2-benzisothiazole-3-one-1,1-dioxide, 3-oxo-2,3-dihydrobenzo[d]isothiazol-1,1- dioxide, 2,3-dihydro-3-oxobenziso-sulfonazole; calcium o-benzosulfimide.

Chemical formula

$C_{14}H_8CaN_2O_6S_2 \cdot 3½H_2O$

Structural formula



Formula weight

467.48

Assay

Not less than 99% after drying

DESCRIPTION

White crystals or a white, crystalline powder, odourless or with a faint, aromatic odour

FUNCTIONAL USES

Sweetener

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4)

Freely soluble in water, soluble in ethanol

Melting range of saccharin derived from the sample (Vol. 4)

226 - 230°
To 10 ml of a 1 in 10 solution add 1 ml of hydrochloric acid. A crystalline precipitate of saccharin is formed. Wash the precipitate well with cold water and dry at 105° for 2 h.

Derivation to salicylic acid

Dissolve about 0.1 g of the sample in 5 ml of 5% sodium hydroxide solution. Evaporate to dryness and gently fuse the residue over a small flame until it no longer evolves ammonia. After the residue has cooled, dissolve it in 20 ml of water, neutralize the solution with dilute hydrochloric

acid TS and filter. The addition of a drop of ferric chloride TS to the filtrate produces a violet colour.

Derivation to fluorescent substance — Mix 20 mg of the sample with 40 mg of resorcinol, add 10 drops of sulfuric acid, and heat the mixture in a liquid bath at 200º for 3 min. After cooling, add 10 ml of water and an excess of sodium hydroxide TS. A fluorescent green liquid is produced.

Test for calcium (Vol. 4) — Passes test

PURITY

Loss on drying (Vol. 4) — Not more than 15% (120°, 4 h)

Benzoic and salicylic acid — Add ferric chloride TS dropwise to 10 ml of a hot, saturated solution of the sample. No precipitate or violet colour appears.

Readily carbonizable substances (Vol. 4) — Dissolve 0.2 g of the sample in 5 ml of sulfuric acid TS. Keep at 48° to 50° for 10 min. The colour should not be darker than a very light brownish-yellow (Matching Fluid A).

Toluenesulfonamides (Vol. 4) — Not more than 25 mg/kg

Selenium (Vol. 4) — Not more than 30 mg/kg

Lead (Vol. 4) — Not more than 1 mg/kg
Determine using an atomic absorption technique appropriate to the specified level. The selection of sample size and method of sample preparation may be based on the principles of the method described in Volume 4, “Instrumental Methods.”

METHOD OF ASSAY

Weigh accurately about 0.5 g of the sample and transfer quantitatively to a separator with the aid of 10 ml of water. Add 2 ml of dilute hydrochloric acid TS, and extract the precipitated saccharin first with 30 ml, then with five 20 ml portions, of a liquid composed of 9 volumes of chloroform and 1 volume of ethanol. Filter each extract through a small filter paper moistened with the solvent mixture. Evaporate the combined filtrates on a steam bath to dryness with the aid of a current of air. Dissolve the residue in 75 ml of hot water, cool, add phenolphthalein TS, and titrate with 0.1 N sodium hydroxide. Perform a blank determination, and make any necessary correction. Each ml of 0.1 N sodium hydroxide is equivalent to 20.22 mg of $C_{14}H_8CaN_2O_6S_2$.