U.S. PHARMACOPEIA

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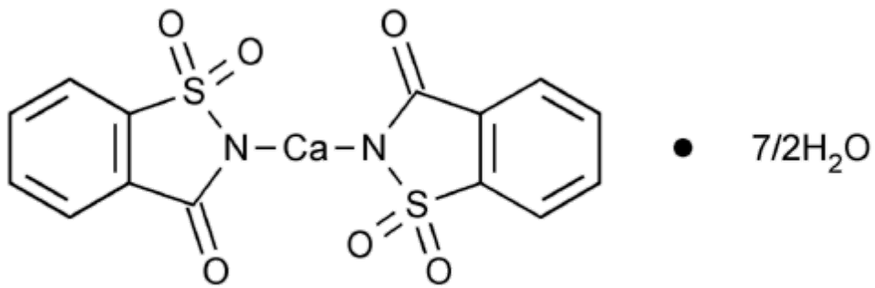
Saccharin Calcium



$C_{14}H_8CaN_2O_6S_2 \cdot 3½H_2O$ 467.49

1,2-Benzisothiazol-3(2*H*)-one, 1,1-dioxide, calcium salt, hydrate (2:7).
1,2-Benzisothiazolin-3-one 1,1-dioxide calcium salt hydrate (2:7) [*6381-91-5*].

Anhydrous 404.44 [*6485-34-3*].

» Saccharin Calcium contains not less than 98.0 percent and not more than 101.0 percent of $C_{14}H_8CaN_2O_6S_2$, calculated on the anhydrous basis.

Packaging and storage— Preserve in well-closed containers.

Labeling— Where the quantity of saccharin calcium is indicated in the labeling of any preparation containing Saccharin Calcium, this shall be expressed in terms of saccharin ($C_7H_5NO_3S$).

USP Reference standards ⟨ 11 ⟩ **—** *USP o-Toluenesulfonamide RS*. *USP p-Toluenesulfonamide RS*.

Identification—

A: Dissolve about 100 mg in 5 mL of sodium hydroxide solution (1 in 20), evaporate to dryness, and gently fuse the residue over a small flame until it no longer evolves ammonia. Allow the residue to cool, dissolve in 20 mL of water, neutralize with 3 N hydrochloric acid, and filter: the addition of a drop of ferric chloride TS to the filtrate produces a violet color.

B: Mix 20 mg with 40 mg of resorcinol, add 10 drops of sulfuric acid, and heat the mixture in a suitable liquid bath at 200° for 3 minutes. Allow it to cool, and add 10 mL of water and an excess of 1 N sodium hydroxide: a fluorescent green liquid results.

C: A solution (1 in 10) meets the requirements of the tests for *Calcium* ⟨ 191 ⟩.

D: To 10 mL of a solution (1 in 10) add 1 mL of hydrochloric acid: a crystalline precipitate of saccharin is formed. Wash the precipitate with cold water, and dry at 105° for 2 hours: it melts between 226° and 230°, the procedure for *Class I* being used (see *Melting Range or Temperature* ⟨ 741 ⟩).

Water, *Method I* ⟨ 921 ⟩**:** not more than 15.0%.

Readily carbonizable substances ⟨ 271 ⟩**—** Dissolve 200 mg in 5 mL of sulfuric acid TS, and maintain at a temperature of 48° to 50° for 10 minutes: the solution has no more color than *Matching Fluid A*.

Selenium ⟨ 291 ⟩**:** 0.003%.

Toluenesulfonamides—

Internal standard solution, Standard stock solution, and *Standard preparations*— Prepare as directed for *Internal standard solution*, *Standard stock solution*, and *Standard preparations* in the test for *Toluenesulfonamides* under *Saccharin* (see NF monograph).

Test preparation— Prepare as directed under *Column Partition Chromatography* (see *Chromatography* ⟨ 621 ⟩), employing a chromatographic tube fitted with a porous glass disk in its base, a plastic stopcock on the delivery tube, and a reservoir on the top. Add a mixture consisting of 10 g of *Solid Support* and a solution of 2.0 g, accurately weighed, of Saccharin Calcium in 8.0 mL of sodium carbonate solution (1 in 20), and proceed as directed for *Test preparation* in the test for *Toluenesulfonamides* under *Saccharin* (see NF monograph), beginning with “Pack the contents.”

Chromatographic system and *Procedure*— Proceed as directed for *Chromatographic system* and *Procedure* in the test for *Toluenesulfonamides* under *Saccharin* (see NF monograph).

Heavy metals, *Method I* ⟨ 231 ⟩**—** Dissolve 4 g in 46 mL of water, add 4 mL of dilute hydrochloric acid (1 in 12), mix, and rub the inner wall of the vessel with a glass rod until crystallization begins. Allow the solution to stand for 1 hour, then pass through a dry filter, discarding the first 10 mL of the filtrate, and use 25 mL of the subsequent filtrate for the *Test Preparation:* the limit is 0.001%.

Limit of benzoate and salicylate— To 10 mL of a solution (1 in 20), previously acidified with 5 drops of 6 N acetic acid, add 3 drops of ferric chloride TS: no precipitate or violet color appears.

Organic volatile impurities, *Method I* ⟨ 467 ⟩**:** meets the requirements.

Residual solvents ⟨ 467 ⟩**:** meets the requirements.
(Official January 1, 2007)

Assay— Accurately weigh about 500 mg of Saccharin Calcium, and transfer completely to a separator with the aid of 10 mL of water. Add 2 mL of 3 N hydrochloric acid, and extract the precipitated saccharin first with 30 mL, then with five 20-mL portions, of a mixture of chloroform and alcohol (9:1). Evaporate the combined extracts on a steam bath to dryness, with the aid of a current of air, then dissolve the residue in 40 mL of alcohol, add 40 mL of water, mix, add phenolphthalein TS, and titrate with 0.1 N sodium hydroxide VS. Perform a blank determination on a mixture of 40 mL of alcohol and 40 mL of water, and make any necessary correction. Each mL of 0.1 N sodium hydroxide is equivalent to 20.22 mg of $C_{14}H_8CaN_2O_6S_2$.

Auxiliary Information— *Staff Liaison* : Catherine Sheehan, B.Sc., Scientist
Expert Committee : (EM105) Excipient Monographs 1
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