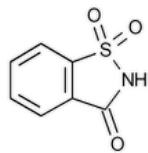


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Saccharin



$C_7H_5NO_3S$ 183.18
1,2-Benzisothiazol-3(2H)-one, 1,1-dioxide;
1,2-Benzisothiazolin-3-one 1,1-dioxide CAS RN®: 81-07-2.

DEFINITION

Saccharin contains NLT 98.0% and NMT 102.0% of saccharin ($C_7H_5NO_3S$), calculated on the dried basis.

IDENTIFICATION

• A. [SPECTROSCOPIC IDENTIFICATION TESTS \(197\)](#), [Infrared Spectroscopy: 197K](#)

ASSAY

• PROCEDURE

Solution A: 50 mM [dibasic potassium phosphate](#) (K_2HPO_4) buffer in 0.1% (v/v) [phosphoric acid](#) solution

Solution B: Methanol

Mobile phase: See [Table 1](#).

Table 1

Time (min)	Solution A (%)	Solution B (%)
0	90	10
7.0	90	10
8.0	5	95
10.0	5	95
10.1	90	10
15.0	90	10

Diluent: Methanol and water (50:50 v/v)
System suitability solution: 0.1 mg/mL of [phthalic anhydride](#) and 0.1 mg/mL of [USP Saccharin RS](#) in Diluent
Standard solution: 0.1 mg/mL of [USP Saccharin RS](#) in Diluent
Sample solution: 0.1 mg/mL of Saccharin in Diluent
Chromatographic system
(See [Chromatography \(621\)](#), [System Suitability](#).)
Mode: LC

Detector: UV 230 nm

Column: 4.6-mm × 15-cm; 3.5-μm packing [L1](#)

Column temperature: 20 ± 5°

Flow rate: 1.0 mL/min

Injection volume: 10 μL

Run time: 15 min

System suitability

Samples: *System suitability solution* and *Standard solution*

[NOTE—The retention times for phthalic anhydride and saccharin are about 6.3 and 7.3 min, respectively. Phthalic anhydride is a potential impurity.]

Suitability requirements

Resolution: NLT 1.5 between the phthalic anhydride and saccharin peaks, *System suitability solution*

Tailing factor: NMT 1.5, *Standard solution*

Relative standard deviation: NMT 0.73% for five replicate injections, *Standard solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of saccharin in the portion of Saccharin taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times 100$$

r_U = peak area of saccharin from the *Sample solution*

r_S = peak area of saccharin from the *Standard solution*

C_S = concentration of [USP Saccharin RS](#) in the *Standard solution* (mg/mL)

C_U = concentration of Saccharin in the *Sample solution* (mg/mL)

Acceptance criteria: 98.0%–102.0% on the dried basis

IMPURITIES

• **RESIDUE ON IGNITION (281):** NMT 0.2%, using an ignition temperature of 600 ± 50°

• **LIMIT OF TOLUENESULFONAMIDES**

Internal standard solution: 0.25 mg/mL of caffeine in [methylene chloride](#)

Standard stock solution: 20.0 μg/mL of [USP o-Toluenesulfonamide RS](#) and 20.0 μg/mL of [USP p-Toluenesulfonamide RS](#) in [methylene chloride](#)

Standard solution: Evaporate 5.0 mL of the *Standard stock solution* to dryness in a stream of nitrogen. Dissolve the residue in 1 mL of the *Internal standard solution*.

Sample solution: Suspend 10 g of Saccharin in 20 mL of water, and dissolve using 5–6 mL of [10 N sodium hydroxide](#). If necessary, adjust the solution with [1 N sodium hydroxide](#) or [1 N hydrochloric acid](#) to a pH of 7–8, and dilute with water to 50 mL. Shake the solution with four quantities each of 50 mL of [methylene chloride](#). Combine the lower layers, dry over [anhydrous sodium sulfate](#), and filter. Wash the filter and the sodium sulfate with 10 mL of [methylene chloride](#). Combine the solution and the washings, and evaporate almost to dryness in a water bath at a temperature not exceeding 40°. Using a small quantity of [methylene chloride](#), quantitatively transfer the residue into a suitable 10-mL tube, evaporate to dryness in a stream of nitrogen, and dissolve the residue in 1.0 mL of the *Internal standard solution*.

Blank solution: Evaporate 200 mL of [methylene chloride](#) to dryness in a water bath at a temperature not exceeding 40°. Dissolve the residue in 1 mL of [methylene chloride](#).

Chromatographic system

(See [Chromatography \(621\), System Suitability](#).)

Mode: GC

Detector: Flame ionization

Column: 0.53-mm × 10-m fused silica; coated with a 2-μm film of phase G3

Temperatures

Injector: 250°

Column: 180°

Detector: 250°

Carrier gas: Nitrogen

Flow rate: 10 mL/min