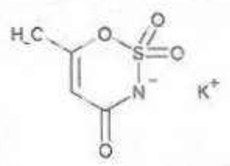
Acesulfame Potassium



$C_4H_4NO_4SK$ 201.24

6-Methyl-1,2,3-oxathiazine-4(3*H*)-one-2,2-dioxide potassium salt;

3,4-Dihydro-6-methyl-1,2,3-oxathiazine-4-one-2,2-dioxide potassium salt [55589-62-3].

DEFINITION

Acesulfame Potassium contains NLT 99.0% and NMT 101.0% of $C_4H_4NO_4SK$, calculated on the dried basis.

IDENTIFICATION

- **A. Infrared Absorption** ⟨197K⟩
- **B. Identification Tests—General,** *Potassium* ⟨191⟩

Sample solution: 100 mg/mL

Acceptance criteria: Meets the requirements

ASSAY

- **Procedure**

Sample: 150 mg

Titrimetric system

(See *Titrimetry* ⟨541⟩.)

Mode: Direct titration

Titrant: 0.1 N perchloric acid VS

Blank: 50 mL of glacial acetic acid

Endpoint detection: Potentiometric

Analysis: Dissolve the *Sample* in 50 mL of glacial acetic acid. Titrate with 0.1 N perchloric acid VS. Perform a blank determination.

Calculate the percentage of acesulfame potassium ($C_4H_4NO_4SK$) in the *Sample:*

$$\text{Result} = [(V - B) \times N \times F \times 100]/W$$

V = titrant volume consumed by the *Sample* (mL)
B = titrant volume consumed by the *Blank* (mL)
N = titrant actual normality (mEq/mL)
F = equivalency factor, 201.2 mg/mEq
W = weight of *Sample* (mg)

Acceptance criteria: 99.0%–101.0% on the dried basis

IMPURITIES

- **Limit of Fluoride**

[Note—Use plasticware throughout this test.]

Solution A: Dissolve 210 g of citric acid monohydrate in 400 mL of water. Adjust with concentrated ammonia to a pH of 7.0, and dilute with water to 1000 mL.

Solution B: 132 mg/mL of dibasic ammonium phosphate

Solution C: To a suspension of 292 g of edetic acid in 500 mL of water, add 200 mL of ammonium hydroxide, adjust with ammonium hydroxide to a pH between 6 and 7, and dilute with water to make 1000 mL.

Buffer solution: Mix equal volumes of *Solution A, Solution B,* and *Solution C,* and adjust with ammonium hydroxide to a pH of 7.5.

Standard stock solution: Weigh 0.442 g of sodium fluoride, previously dried at 300° for 12 h, into a 1-L volumetric flask, and dilute with water to volume. Store the solution in a closed plastic container. Immediately before use, pipet 5 mL of this solution into a 100-mL volumetric flask, and dilute with water to volume. Each mL of this solution contains 10 µg of fluoride ion.

Standard solution A: Mix 0.5 mL of *Standard stock solution* and 15.0 mL of *Buffer solution,* and dilute with water to 50 mL.

Standard solution B: Mix 1.0 mL of *Standard stock solution* and 15.0 mL of *Buffer solution,* and dilute with water to 50 mL.

Standard solution C: Mix 1.5 mL of *Standard stock solution* and 15.0 mL of *Buffer solution,* and dilute with water to 50 mL.

Standard solution D: Mix 3.0 mL of *Standard stock solution* and 15.0 mL of *Buffer solution,* and dilute with water to 50 mL.

Sample solution: To a 50-mL volumetric flask add 3 g of Acesulfame Potassium. Dissolve in water, add 15.0 mL of *Buffer solution,* and dilute with water to volume.

Analysis

Samples: *Standard solution A, Standard solution B, Standard solution C, Standard solution D,* and *Sample solution*

Concomitantly measure the potential (see *Titrimetry* ⟨541⟩), in mV, of the *Standard solutions* and the *Sample solution,* with a suitable pH meter equipped with a fluoride-specific ion electrode and a silver–silver chloride reference electrode. When taking the measurements, transfer the solution to a 25-mL beaker, and immerse the electrodes. Insert a polytef-coated stirring bar into the beaker, place the beaker on a magnetic stirrer having an insulated top, and allow to stir until equilibrium is attained (1–2 min). Rinse, and dry the electrodes between measurements, taking care not to scratch the crystal in the fluoride-specific ion electrode. Measure the potential of each *Standard solution,* and plot the fluoride concentration, in µg/mL, versus the potential, in mV, on semilogarithmic paper. Measure the potential of the *Sample solution,* and determine the fluoride concentration from the standard curve, in µg/mL.

Calculate the content, in ppm, of fluoride in the portion of Acesulfame Potassium taken:

$$\text{Result} = (V \times C/W)$$

V = volume of the *Sample solution* (mL)
C = concentration of fluoride in the *Sample solution,* from the standard curve (µg/mL)
W = weight of Acesulfame Potassium taken to prepare the *Sample solution* (g)

Acceptance criteria: NMT 3 ppm

Delete the following:

- **Heavy Metals,** *Method I* ⟨231⟩**:** NMT 10 ppm (Official 1-Jan-2018)
- **Chromatographic Purity**

Solution A: 3.3 mg/mL of tetrabutylammonium hydrogen sulfate

Mobile phase: Acetonitrile and *Solution A* (2:3)

System suitability solution: 2 µg/mL each of USP Acesulfame Potassium RS and ethylparaben

Standard solution: 0.2 µg/mL of USP Acesulfame Potassium RS

Sample solution: 10 mg/mL

Chromatographic system

(See *Chromatography* ⟨621⟩, *System Suitability.*)

Mode: LC

Detector: UV 227 nm

Column: 4.6-mm × 25-cm; 5-µm packing L1

Flow rate: 1 mL/min

Injection size: 20 µL

System suitability

Sample: *System suitability solution*

Suitability requirements

Resolution: NLT 2 between acesulfame potassium and ethylparaben

NF Monographs