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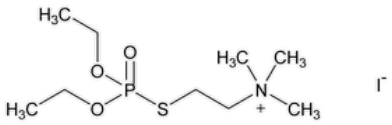
Echothiophate Iodide



$C_9H_{23}INO_3PS$ 383.23

Ethanaminium, 2-[(diethoxyphosphinyl)thio]-*N*,*N*,*N*-trimethyl-, iodide;

(2-Mercaptoethyl)trimethylammonium iodide *S*-ester with *O*,*O*-diethyl phosphorothioate CAS RN®: 513-10-0; UNII: BA9QH3P00T.

DEFINITION

Echothiophate Iodide contains NLT 95.0% and NMT 100.5% of echothiophate iodide ($C_9H_{23}INO_3PS$), calculated on the dried basis.

IDENTIFICATION

- **A.**

  Sample solution: 50 mg/mL

  Analysis: To 2 mL of the *Sample solution* in a test tube add 1 mL of nitric acid. Transfer 1 drop of this mixture to another test tube, add 1 mL of solvent hexane, and shake.

  Acceptance criteria: (1) A brown precipitate of iodine is formed after addition of nitric acid. (2) The solvent hexane acquires a pink color.

- **B.** **Identification Tests—General,** *Phosphate*⟨191⟩

  Analysis: Heat the remainder of the brown reaction mixture from *Identification* test *A* over a flame until a colorless solution remains (about 3 min). Cool, and dilute with water to 10 mL.

  Acceptance criteria: A 2-mL portion of this solution meets the requirements.

- **C.** **Identification Tests—General,** *Sulfate*⟨191⟩**:** A 2-mL portion of the solution obtained in *Identification* test *B* meets the requirements.

ASSAY

- **Procedure**

In the preparation of all reagents, and throughout this procedure, wherever water is specified, use only water that has been distilled, boiled for 10 min, and cooled while protected from the atmosphere.

Solution A: Transfer 5.44 g of anhydrous dibasic sodium phosphate to a 100-mL volumetric flask, and add a volume of 1 N sodium hydroxide VS that contains 36.5 mEq of sodium hydroxide. Add 40 mL of water, shake to dissolve the sodium phosphate, and dilute with water to volume.

Solution B: Dilute 0.1 N iodine with water to 0.004 N, and standardize the solution on the day of use as follows. Weigh 150 mg of arsenic trioxide, and dissolve in 20 mL of 1 N sodium hydroxide, by warming if necessary, in a 500-mL volumetric flask. Dilute with 40 mL of water, add 2 drops of methyl orange TS, then add 3 N hydrochloric acid until the yellow color is changed to pink. Add 2 g of sodium bicarbonate, then add water to volume. Transfer 5.0 mL of this solution to a titration vessel, and add 50 mL of *Solution A* (see *Reagents, Indicators, and Solutions—Solutions*). Titrate with 0.004 N iodine, determining the endpoint potentiometrically, using platinum and silver–silver chloride electrodes. Calculate the normality. Each 197.8 µg of arsenic trioxide is equivalent to 1 mL of 0.004 N iodine.

Sample solution: 1.25 mg/mL of Echothiophate Iodide in water

Analysis: Transfer 10.0 mL of the *Sample solution* to a titration vessel containing 30 mL of water. Add 10.0 mL of *Solution A,* cover, and allow to stand for 20 min at 25 ± 3°. Add 2 mL of glacial acetic acid rapidly and with mixing. Titrate with *Solution B*, determining the endpoint potentiometrically, using platinum and silver–silver chloride electrodes. Correct for the amount of free thiol sulfur by repeating the procedure but adding the glacial acetic acid first, then *Solution A,* and titrating immediately. Subtract the volume of *Solution B* used in the second titration from that used in the first. Each mL of 0.004 N iodine is equivalent to 1.533 mg of echothiophate iodide ($C_9H_{23}INO_3PS$).

Acceptance criteria: 95.0%–100.5% on the dried basis

SPECIFIC TESTS

• **Loss on Drying** ⟨731⟩

Analysis: Dry under vacuum over phosphorus pentoxide at 50° for 3 h.

Acceptance criteria: NMT 1.0%

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in tight, light-resistant containers, preferably at a temperature below 0°.

Auxiliary Information - Please check for your question in the FAQs before contacting USP.

Topic/Question	Contact	Expert Committee
ECHOTHIOPHATE IODIDE	Documentary Standards Support	SM32020 Small Molecules 3

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. Information currently unavailable

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