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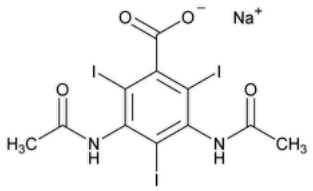
# Diatrizoate Sodium



$C_{11}H_8I_3N_2NaO_4$ 635.90

Benzoic acid, 3,5-bis(acetylamino)-2,4,6-triiodo-, monosodium salt;

Monosodium 3,5-diacetamido-2,4,6-triiodobenzoate CAS RN®: 737-31-5; UNII: V5403H8VG7.

## DEFINITION

Diatrizoate Sodium contains NLT 98.0% and NMT 102.0% of diatrizoate sodium ($C_{11}H_8I_3N_2NaO_4$), calculated on the anhydrous basis.

## IDENTIFICATION

• **A. Thin-Layer Chromatographic Identification Test ⟨201⟩**

**Solution A:** Sodium hydroxide in methanol (0.8 in 1000)

**Standard solution:** 1 mg/mL of USP Diatrizoic Acid RS in *Solution A*

**Sample solution:** 1 mg/mL of Diatrizoate Sodium in *Solution A*

**Developing solvent system:** Methanol, chloroform, and ammonium hydroxide (10:20:2)

**Analysis**

**Samples:** *Standard solution* and *Sample solution*

Locate the spots using short-wavelength UV light.

**Acceptance criteria:** Meets the requirements

• **B.** A 5-mg/mL solution in water imparts an intense yellow color to a nonluminous flame confirming the presence of sodium.

## ASSAY

• **Procedure**

**Sample solution:** Transfer 300 mg of Diatrizoate Sodium to a glass-stoppered, 125-mL conical flask, add 30 mL of 1.25 N sodium hydroxide and 500 mg of powdered zinc, connect the flask to a reflux condenser, and reflux the mixture for 1 h. Cool the flask to room temperature, rinse the condenser with 20 mL of water, disconnect the flask from the condenser, and filter the mixture. Rinse the flask and filter thoroughly, adding the rinsings to the filtrate. Add 5 mL of glacial acetic acid and 1 mL of tetrabromophenolphthalein ethyl ester TS.

**Titrimetric system**

(See *Titrimetry ⟨541⟩*.)

**Mode:** Direct titration

**Titrant:** 0.05 N silver nitrate VS

**Endpoint detection:** Visual

**Analysis:** Titrate the *Sample solution* with the *Titrant* until the yellow precipitate just turns green.

Calculate the percentage of diatrizoate sodium ($C_{11}H_8I_3N_2NaO_4$) in the portion of Diatrizoate Sodium taken:

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

$V$ = sample *Titrant* volume (mL)

$N$ = *Titrant* normality (mEq/mL)

$F$ = equivalent weight of diatrizoate sodium, 212 mg/mEq

$W$ = weight of Diatrizoate Sodium (mg)

**Acceptance criteria:** 98.0%–102.0% on the anhydrous basis

## IMPURITIES

***Change to read:***

• **Limit of Free Aromatic Amine**

**Solution A:** 1 mg/mL of *N*-(1-naphthyl)ethylenediamine dihydrochloride in a mixture of propylene glycol and water (70:30)

**Standard stock solution:** 0.5 mg/mL of USP Diatrizoic Acid Related Compound A RS▲prepared as follows. Transfer 5 mg of USP Diatrizoic Acid Related Compound A RS to a 10-mL volumetric flask. Add 0.2 mL of 0.1 N sodium hydroxide. Dilute with water to volume.▲ (USP 1-Dec-2019)

**Standard solution:** Transfer 4 mL of water, 10 mL of 0.1 N sodium hydroxide, and 1.0 mL of *Standard stock solution* to a 50-mL volumetric flask.

**Sample solution:** Transfer 1.0 g of Diatrizoate Sodium to a 50-mL volumetric flask, and add 5 mL of water and 10 mL of 0.1 N sodium hydroxide.

**Blank solution:** Transfer 5 mL of water and 10 mL of 0.1 N sodium hydroxide to a 50-mL volumetric flask.

**Instrumental conditions**

**Mode:** Vis

**Analytical wavelength:** 465 nm

**Cell:** 1 cm

**Analysis**

**Samples:** *Standard solution*, *Sample solution*, and *Blank solution*

Treat the *Samples* as follows. Add 25 mL of dimethylsulfoxide. Place the flask in an ice bath for 5 min. Add 2 mL of hydrochloric acid, and allow to stand in the ice bath for 5 min. Add 2.0 mL of sodium nitrite solution (20 mg/mL), and allow to stand in the ice bath for 5 min. Remove the flask from the ice bath, add 1.0 mL of sulfamic acid solution (80 mg/mL), and allow to stand in the ice bath for 5 min. [**Caution—**Considerable pressure is produced.] Add 2.0 mL of *Solution A*. Remove the flasks from the ice bath and allow to stand in a water bath at room temperature. Shake occasionally, releasing the pressure by loosening the stopper. Dilute with water to volume. Within 5 min, measure the absorbances of the *Standard solution* and *Sample solution* against the blank.

**Acceptance criteria:** The absorbance of the *Sample solution* is NMT that of the *Standard solution* (NMT 0.05% of free aromatic amine).

## SPECIFIC TESTS

- **Water Determination ⟨921⟩, *Method I*:** NMT 10.0%
- **Limit of Iodine and Iodide**

**Standard solution:** Dissolve 0.5 mg of potassium iodide in 24 mL of water in a 50-mL stoppered centrifuge tube.

**Sample solution:** Transfer 2.0 g to a 50-mL stoppered centrifuge tube, dilute with water to 24 mL, and shake to dissolve.

**Analysis**

**Samples:** *Standard solution* and *Sample solution*

Treat the *Samples* as follows. Add 5 mL of toluene and 5 mL of 2 N sulfuric acid, shake, and centrifuge. The toluene layer shows no red color. Add 1 mL of sodium nitrite solution (1 in 50), shake, and centrifuge.

**Acceptance criteria**

1. The *Sample solution* does not show any red color in the toluene layer prior to addition of the sodium nitrite solution.
2. The red color of the toluene layer in the *Sample solution* after the addition of sodium nitrite solution is not darker than the *Standard solution* (NMT 0.02% of iodide).

## ADDITIONAL REQUIREMENTS

- **Packaging and Storage:** Preserve in well-closed containers. Store at room temperature.
- **USP Reference Standards ⟨11⟩**

USP Diatrizoic Acid RS

USP Diatrizoic Acid Related Compound A RS

5-Acetamido-3-amino-2,4,6-triiodobenzoic acid.

$C_9H_7I_3N_2O_3$ 571.88



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

| Topic/Question | Contact | Expert Committee |
|---|---|---|
| DIATRIZOATE SODIUM | Documentary Standards Support | SM42020 Small Molecules 4 |

**Chromatographic Database Information:** Chromatographic Database

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