

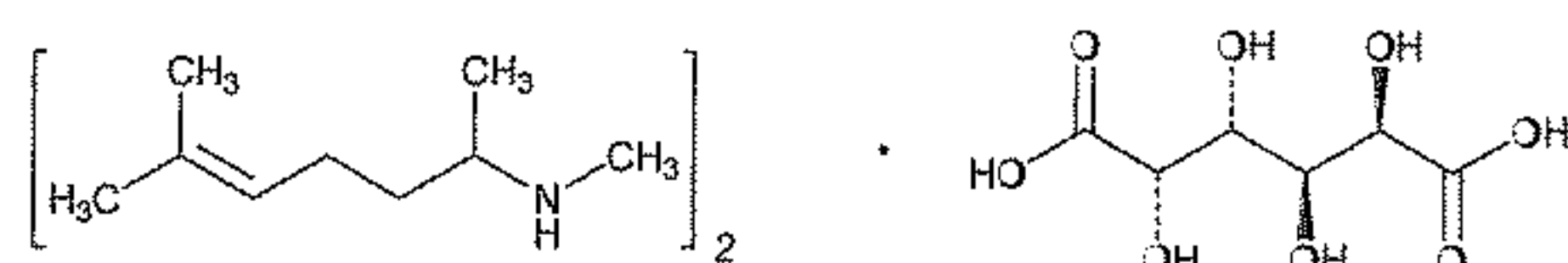
Individual impurities: NMT 0.5%  
Total impurities: NMT 2.0%

**SPECIFIC TESTS**

- **OPTICAL ROTATION (781S)**, *Procedures, Specific Rotation*  
Sample solution: 40 mg/mL in 6 N hydrochloric acid  
Acceptance criteria: +38.9° to +41.8°
- **PH (791)**  
Sample solution: 10 mg/mL in water  
Acceptance criteria: 5.5–7.0
- **LOSS ON DRYING (731)**  
Analysis: Dry at 105° for 3 h.  
Acceptance criteria: NMT 0.3%

**ADDITIONAL REQUIREMENTS**

- **PACKAGING AND STORAGE:** Preserve in well-closed containers.
- **USP REFERENCE STANDARDS (11)**  
USP L-Isoleucine RS  
USP L-Leucine RS  
USP L-Valine RS

**Isometheptene Mucate**

$(C_9H_{19}N)_2 \cdot C_6H_{10}O_8$  492.65  
Isometheptene, galactarate (2:1) (salt);  
6-Methylamino-2-methylheptene, tetrahydroxyadipic acid  
(2:1) (salt) [7492-31-1].

**DEFINITION**

Isometheptene Mucate contains NLT 99.0% and NMT 103.0% of isometheptene mucate  $[(C_9H_{19}N)_2 \cdot C_6H_{10}O_8]$ , calculated on the dried basis.

**IDENTIFICATION**

- **A. INFRARED ABSORPTION (197K)**

**ASSAY**• **PROCEDURE**

**Sample:** 500 mg of Isometheptene Mucate

**Titrimetric system**

(See *Titrimetry (541)*.)

**Mode:** Residual titration

**Titrant:** 0.1 N bromine VS

**Back-titrant:** 0.1 N sodium thiosulfate VS

**Endpoint detection:** Visual

**Analysis:**

**Sample:** *Sample*

Transfer the *Sample* to a 125-mL conical flask. Add 25 mL of water, and swirl to dissolve. Add 45.0 mL of *Titrant*. Attach a 30-mL separator to the conical flask, and evacuate the system. Stir with a magnetic stirrer for 1 h. Equilibrate the system to atmospheric pressure. Add 5 mL of hydrochloric acid to the separator, and allow 4 mL of it to enter the conical flask. Add 10 mL of potassium iodide TS to the separator, and allow the contents to pass into the conical flask. Immediately titrate the liberated iodine with *Back-titrant*, adding 3 mL of starch TS as the endpoint is approached. Perform a blank titration. Each mL of 0.1 N sodium thiosulfate is equivalent to 12.32 mg of isometheptene mucate  $[(C_9H_{19}N)_2 \cdot C_6H_{10}O_8]$ .

Acceptance criteria: 99.0%–103.0% on the dried basis

**IMPURITIES**

- **RESIDUE ON IGNITION (281):** NMT 0.1%

**SPECIFIC TESTS**

- **PH (791)**  
Sample solution: A solution of Isometheptene Mucate in water (1 in 20)  
Acceptance criteria: 6.0–7.5
- **LOSS ON DRYING (731)**  
Analysis: Dry at 60° for 18 h.  
Acceptance criteria: NMT 1.0%

**ADDITIONAL REQUIREMENTS**

- **PACKAGING AND STORAGE:** Preserve in well-closed containers.
- **USP REFERENCE STANDARDS (11)**  
USP Isometheptene Mucate RS

**Isometheptene Mucate, Dichloralphenazone, and Acetaminophen Capsules****DEFINITION**

Isometheptene Mucate, Dichloralphenazone, and Acetaminophen Capsules contain NLT 85.0% and NMT 110.0% of the labeled amounts of isometheptene mucate  $[(C_9H_{19}N)_2 \cdot C_6H_{10}O_8]$  and dichloralphenazone  $(C_{15}H_{13}Cl_2N_2O_5)$ , and NLT 90.0% and NMT 110.0% of the labeled amount of acetaminophen  $(C_9H_9NO_2)$ .

**IDENTIFICATION**

- **A.** The retention times of the major peaks of the *Sample solution* correspond to those of the *Standard solution*, as obtained in the *Assay*.

**ASSAY**• **PROCEDURE**

**Mobile phase:** Acetonitrile, 0.07 M monobasic potassium phosphate, 0.007 M sodium 1-decanesulfonate, and diethylamine (250:750:25:15). Adjust with phosphoric acid to a pH of 3.5.

**Sensitivity solution:** Empty the contents of 1 Capsule into a 100-mL volumetric flask. Add 80 mL of water, and swirl to dissolve. Dilute with water to volume. Pass a portion of this solution through a glass-fiber filter, discarding the first 5 mL of the filtrate. [NOTE—Prepare this solution, chromatograph it, and evaluate it as directed for *System suitability* before preparing the *Standard solution* and the *Sample solution*.]

**Standard solution:** USP Reference Standards in water as listed below

**Acetaminophen:** 3.25 mg/mL of USP Acetaminophen RS

**Dichloralphenazone:** 3.25/ mg/mL of USP Dichloralphenazone RS, where *J* is the ratio of the labeled amount, in mg, of dichloralphenazone to the labeled amount of acetaminophen in mg/Capsule

**Isometheptene mucate:** 3.25/*J'* mg/mL of USP Isometheptene Mucate RS, where *J'* is the ratio of the labeled amount, in mg, of isometheptene mucate to the labeled amount of acetaminophen in mg/Capsule

**Sample solution:** Transfer 20 Capsules to a 2000-mL volumetric flask. Add 1900 mL of water, and heat on a steam bath until the Capsules disintegrate. While still warm, shake by mechanical means for 15 min, sonicate for 15 min, and allow to cool to ambient temperature. Dilute with water to volume. Pass a portion of this mixture through a glass-fiber filter, discarding the first 5 mL of the filtrate.