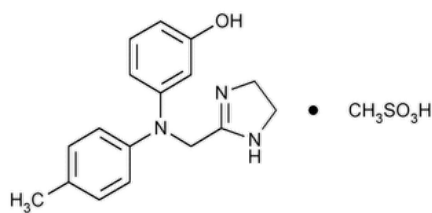


Status: Currently Official on 16-Feb-2025
Official Date: Official as of 01-May-2022
Document Type: USP Monographs
DocId: GUID-042B66BD-9CA4-4E13-8085-3689F6EEAB14_3_en-US
DOI: https://doi.org/10.31003/USPNF_M63960_03_01
DOI Ref: d4qio

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Phentolamine Mesylate

Change to read:



$C_{17}H_{19}N_3O \cdot CH_4O_3S$ 377.46
Phenol, 3-[[[(4,5-dihydro-1H-imidazol-2-yl)methyl](4-methylphenyl)amino]-, monomethanesulfonate (salt);
m-[N-(2-Imidazolin-2-ylmethyl)-*p*-toluidino]phenol monomethanesulfonate (salt);
▲3-[[[(4,5-Dihydro-1H-imidazol-2-yl)methyl](4-tolyl)amino]phenol methanesulfonate▲ (USP 1-May-2022) CAS RN[®]: 65-28-1; UNII: Y7543E5K9T.

DEFINITION
Phentolamine Mesylate contains NLT 98.0% and NMT 102.0% of phentolamine mesylate ($C_{17}H_{19}N_3O \cdot CH_4O_3S$), calculated on the dried basis.

IDENTIFICATION

Change to read:

- **A. [SPECTROSCOPIC IDENTIFICATION TESTS \(197\)](#), [Infrared Spectroscopy](#):** 197M▲ or 197A▲ (USP 1-May-2022)

Change to read:

- **B. [SPECTROSCOPIC IDENTIFICATION TESTS \(197\)](#), [Ultraviolet-Visible Spectroscopy](#):** 197U

Sample solution: 20 µg/mL▲ of Phentolamine Mesylate▲ (USP 1-May-2022) in [water](#)

Acceptance criteria: Meets the requirements

Change to read:

- **C. ▲**The retention time of the major peak of the *Sample solution* corresponds to that of *Standard solution A*, as obtained in the *Assay*.▲ (USP 1-May-2022)

ASSAY

Change to read:

• **PROCEDURE**

▲**Buffer:** Transfer 10.2 g of [monobasic potassium phosphate](#) to a suitable container. Add 3000 mL of [water](#) to dissolve. Adjust with [phosphoric acid](#) to a pH of 2.5.

Solution A: [Methanol](#) and *Buffer* (20:80)

Solution B: [Methanol](#) and *Buffer* (70:30)

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

Table 1

Time (min)	Solution A (%)	Solution B (%)
0	100	0
5.0	100	0

Time (min)	Solution A (%)	Solution B (%)
28.0	0	100
44.0	0	100
45	100	0
55	100	0

Diluent: Transfer 1.36 g of [sodium acetate](#) and 0.5 g of [ethylenediamine tetraacetate disodium dihydrate](#) to a 1000-mL volumetric flask. Add 1000 mL of [water](#) to dissolve. Adjust with [glacial acetic acid](#) to a pH of 3.8.

Standard solution A: 0.22 mg/mL of [USP Phentolamine Mesylate RS](#) in *Diluent*. [NOTE—Sonication may be needed to aid the dissolution.]

Standard solution B: 0.22 mg/mL of [USP Phentolamine Related Compound A RS](#) in *Diluent*. [NOTE—Sonication may be needed to aid the dissolution.]

Standard solution C: 0.22 mg/mL of [USP Phentolamine Keto Analog RS](#) prepared as follows. Transfer a suitable amount of [USP Phentolamine Keto Analog RS](#) to a suitable volumetric flask. Add [methanol](#) to 15% of the flask volume, and sonicate. Dilute with *Diluent* to volume.

System suitability solution: 0.22 mg/mL of [USP Phentolamine Mesylate RS](#) and 1.1 µg/mL each of [USP Phentolamine Related Compound A RS](#) from *Standard solution B* and [USP Phentolamine Keto Analog RS](#) from *Standard solution C* in *Diluent*

Sample solution: 0.22 mg/mL of Phentolamine Mesylate in *Diluent*

Chromatographic system

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

Mode: LC

Detector: UV 232 nm

Column: 4.6-mm × 15-cm; 4-µm packing [L87](#)

Flow rate: 1 mL/min

Injection volume: 10 µL

System suitability

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

[NOTE—The relative retention times for phentolamine keto analog, phentolamine, and phentolamine related compound A are about 0.84, 1.00, and 1.09, respectively.]

Suitability requirements

Resolution: NLT 2.0 between phentolamine and phentolamine related compound A, *System suitability solution*

Tailing factor: NMT 2.0, *Standard solution A*

Relative standard deviation: NMT 0.73%, *Standard solution A*

Analysis

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

Calculate the percentage of phentolamine mesylate ($C_{17}H_{19}N_3O \cdot CH_4O_3S$) in the portion of Phentolamine Mesylate taken:

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

r_U = peak response of phentolamine from the *Sample solution*

r_S = peak response of phentolamine from *Standard solution A*

C_S = concentration of [USP Phentolamine Mesylate RS](#) in *Standard solution A* (mg/mL)

C_U = concentration of Phentolamine Mesylate in the *Sample solution* (mg/mL) ▲ (USP 1-May-2022)

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

IMPURITIES

- [RESIDUE ON IGNITION \(281\)](#): NMT 0.1%
- [CHLORIDE AND SULFATE \(221\)](#), *Sulfate*

Sample: 0.10 g of Phentolamine Mesylate

Acceptance criteria: The *Sample* shows no more sulfate than corresponds to 0.20 mL of 0.020 N sulfuric acid (0.2%)