

Add the following:

Phentolamine Mesylate Injection

DEFINITION
Phentolamine Mesylate Injection is a clear, colorless, sterile solution. It contains NLT 90.0% and NMT 105.0% of the labeled amount of phentolamine mesylate ($C_{17}H_{19}N_3O \cdot CH_4O_3S$). It contains no preservatives.

- IDENTIFICATION**
- A.** The retention time of the major peak of the *Sample solution* corresponds to that of *Standard solution A*, as obtained in the Assay.
 - B.** The UV spectrum of the major peak of the *Sample solution* corresponds to that of *Standard solution A*, as obtained in the Assay.

ASSAY

- 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
28.0	0	100
44.0	0	100
45.0	100	0
55.0	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](#) in 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: Nominally 0.24 mg/mL of phentolamine mesylate prepared from a suitable volume of the pooled Injection (NLT 4 cartridges)

Chromatographic system(See [Chromatography \(621\)](#), [System Suitability](#).)**Mode:** LC**Detector:** UV 232 nm. For *Identification B*, use a diode array detector in the range of 200–400 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 1.0%, *Standard solution A***Analysis****Samples:** *Standard solution A* and *Sample solution*Calculate the percentage of the labeled amount of phentolamine mesylate ($C_{17}H_{19}N_3O \cdot CH_4O_3S$) in the portion of Injection 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 = nominal concentration of phentolamine mesylate in the *Sample solution* (mg/mL)**Acceptance criteria:** 90.0%–105.0%**IMPURITIES**• **ORGANIC IMPURITIES****Buffer, Solution A, Solution B, Mobile phase, Diluent, Standard solution B, Standard solution C, System suitability solution, Sample solution, and Chromatographic system:** Proceed as directed in the Assay.**Sensitivity solution:** 0.11 μg/mL of [USP Phentolamine Mesylate RS](#) in *Diluent***Standard solution:** 1.2 μg/mL of [USP Phentolamine Mesylate RS](#), 2.8 μg/mL of [USP Phentolamine Keto Analog RS](#) from *Standard solution C*, and 5.2 μg/mL of [USP Phentolamine Related Compound A RS](#) from *Standard solution B* in *Diluent***System suitability****Samples:** *System suitability solution*, *Sensitivity solution*, and *Standard solution***Suitability requirements****Resolution:** NLT 2.0 between phentolamine and phentolamine related compound A, *System suitability solution***Relative standard deviation:** NMT 5.0% for phentolamine keto analog, phentolamine, and phentolamine related compound A, *Standard solution***Signal-to-noise ratio:** NLT 10, *Sensitivity solution***Analysis****Samples:** *Standard solution* and *Sample solution*

Calculate the percentage of phentolamine related compound A and phentolamine keto analog in the portion of Injection taken:

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

 r_U = peak response of phentolamine related compound A or phentolamine keto analog from the *Sample solution* r_S = peak response of phentolamine related compound A or phentolamine keto analog from the *Standard solution* C_S = concentration of [USP Phentolamine Related Compound A RS](#) or [USP Phentolamine Keto Analog RS](#) in the *Standard solution* (μg/mL) C_U = nominal concentration of phentolamine mesylate in the *Sample solution* (μg/mL)