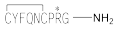
Vasopressin



$C_{46}H_{65}N_{15}O_{12}S_2$ 1084.24
Vasopressin, 8-L-arginine [113-79-1].

DEFINITION

Change to read:

Vasopressin is a polypeptide hormone having the properties of causing the contraction of vascular and other smooth muscles, and of antidiuresis. It is prepared by chemical synthesis. It contains NLT 95.0% and NMT •105.0%• (RB 1-Jul-2011) of vasopressin ($C_{46}H_{65}N_{15}O_{12}S_2$), calculated on the anhydrous, acetic acid-free basis. •• (RB 1-Jan-2011)

IDENTIFICATION

- **A.** The retention time of the vasopressin peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Assay*.
- **B. MASS SPECTRAL ANALYSIS**
  Infusion solution: Acetonitrile, water, and trifluoroacetic acid (80:20:0.08)
  Standard solution: 1 mg/mL of USP Vasopressin RS in water
  Sample solution: 1 mg/mL of Vasopressin in water. [NOTE—The final concentrations of the *Standard solution* and the *Sample solution* can be adjusted, depending on the sensitivity of the mass spectrometer used in the testing.]
  Instrumental conditions
  (See *Mass Spectrometry* ⟨736⟩.)
  Mode: LC/MS spectrometer
  Interface/detection: Infusion system connected to an electrospray interface (positive ion)
  Flow rate: 0.3 mL/min
  Injection size: 10 µL
  Analysis
  Samples: *Standard solution* and *Sample solution*
  Acceptance criteria: Should contain peaks with mass-to-charge ratios of 1084 and 543.

ASSAY

Change to read:

- **PROCEDURE**
  Mobile phase: Dissolve 6.6 g of dibasic ammonium phosphate in 950 mL of water. Adjust with concentrated phosphoric acid to a pH of 3.0. Dilute with water to 1000 mL. To 870 mL of this solution add 130 mL of acetonitrile, and mix. Filter under vacuum through a nylon membrane of 0.45-µm pore size. [NOTE—The retention time of the vasopressin peak is very sensitive to small changes in acetonitrile concentration in the *Mobile phase*.]
  System suitability solution: Dissolve suitable quantities of USP Lypressin RS and USP Vasopressin RS in 0.25% glacial acetic acid to obtain a solution having a known concentration of about 25 µg/mL of each substance.
  Standard solution: Dissolve the entire contents of a vial of USP Vasopressin RS in a known volume of 0.25% glacial acetic acid. [NOTE—The solution may be diluted as necessary to a working concentration range for the assay.]
  Sample solution: Transfer about 10 mg of Vasopressin to a 25-mL volumetric flask. Dissolve in 0.25% glacial acetic acid, and dilute with the same solvent to volume.
  Chromatographic system
  (See *Chromatography* ⟨621⟩, *System Suitability*.)
  Mode: LC
  Detector: UV 220 nm
  Column: 4.6-mm × 25-cm; packing L1
  Column temperature: 40 ± 1°
  Flow rate: 1.0 mL/min
  Injection size: 20 µL. [NOTE—The column is allowed to equilibrate for 1 h before making the first injection.]
  System suitability
  Samples: *System suitability solution* and *Standard solution*
  [NOTE—Inject into an equilibrated liquid chromatograph, allowing about 60 min for complete elution.]
  [NOTE—The retention time of the vasopressin peak is between 6 and 9 min.]
  Suitability requirements
  Resolution: NLT 1.1 between the vasopressin and lypressin peaks
  Relative standard deviation: NMT 2.0% for the vasopressin peak
  Analysis
  Samples: *Standard solution* and *Sample solution*
  Calculate the •percentage of vasopressin ($C_{46}H_{65}N_{15}O_{12}S_2$) in the portion of Vasopressin taken:• (RB 1-Jul-2011)

$$\text{Result} = \bullet(r_U/r_S \times C_S/C_U) \times 100\bullet \text{ (RB 1-Jul-2011)}$$

r_U = peak response from the *Sample solution*
r_S = peak response from the *Standard solution*
•C_S = concentration of USP Vasopressin RS in the *Standard solution* (mg/mL)
C_U = concentration of Vasopressin in the *Sample solution* (mg/mL)• (RB 1-Jul-2011)

Acceptance criteria: 95.0%–•105.0%• (RB 1-Jul-2011) on the anhydrous, acetic acid-free basis

IMPURITIES

- **ORDINARY IMPURITIES:** The sum of the responses of impurities from the *Sample solution* in the *Assay* is NMT 5% of the area of the vasopressin peak.

SPECIFIC TESTS

- **MICROBIAL ENUMERATION TESTS** ⟨**61**⟩ and **TESTS FOR SPECIFIED MICROORGANISMS** ⟨**62**⟩**:** The total bacterial count is NMT 200 cfu/g. For products of animal origin, it also meets the requirements of the tests for absence of *Salmonella* species and *Escherichia coli*.
- **WATER DETERMINATION,** *Method Ic* ⟨**921**⟩**:** NMT 8.0%
- **ACETIC ACID IN PEPTIDES** ⟨**503**⟩**:** NMT 15.0%

ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in tight containers, preferably of Type I glass, in a refrigerator.
- **USP REFERENCE STANDARDS** ⟨**11**⟩
  USP Lypressin RS
  USP Vasopressin RS