be integrated together to determine conformance. (Des-ethoxy impurity is 2-methoxy-5-[(2*R*)-2-[(2-phenoxyethyl)amino]propyl]benzenesulfonamide, and methoxy impurity is 2-methoxy-5-[(2*R*)-2-[[2-(2-methoxyphenoxy)ethyl]amino]propyl]benzenesulfonamide.) NMT 0.15% of the sum of des-ethoxy and methoxy impurities is found.] [NOTE—Reporting level for impurities is 0.05%.]

- **PROCEDURE 2**

[NOTE—For impurities eluting after tamsulosin.]

**Buffer, Standard solution,** and **Sample solution:** Proceed as directed for *Procedure 1*.

[NOTE—Use the *Mobile phase* in *Procedure 1* to prepare the *Standard solution* and *Sample solution*.]

**Mobile phase:** Acetonitrile and *Buffer* (1:1)

**Chromatographic system**

(See *Chromatography* ⟨621⟩, *System Suitability*.)

**Mode:** LC

**Detector:** UV 225 nm

**Column:** 4.6-mm x 15-cm; 5-µm packing L1

**Temperature:** 40°

**Flow rate:** 1.0 mL/min

**Injection size:** 10 µL

[NOTE—Record the chromatogram for NLT 5 times the retention time of tamsulosin.]

**System suitability**

**Sample:** *Standard solution*

**Suitability requirements**

**Resolution:** Use a column that meets the resolution requirements of *Procedure 1*.

**Relative standard deviation:** NMT 4% for six replicate injections

**Analysis**

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

Calculate the percentage of any individual impurity, eluting after the tamsulosin peak, in the portion of Tamsulosin Hydrochloride taken:

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

$r_U$ = peak response of each impurity eluting after tamsulosin from the *Sample solution*

$r_S$ = peak response of tamsulosin from the *Standard solution*

$C_S$ = concentration of the *Standard solution* (mg/mL)

$C_U$ = concentration of the *Sample solution* (mg/mL)

**Acceptance criteria**

**Individual impurities:** NMT 0.10% for any individual impurity. [NOTE—Reporting level for impurities is 0.05%.]

- **TOTAL IMPURITIES:** NMT 0.2%, including all impurities in *Procedure 1* and *Procedure 2*

**SPECIFIC TESTS**

- **ENANTIOMERIC PURITY**

**Mobile phase:** Hexane, dehydrated alcohol, methanol, and diethylamine (650:200:150:1)

**System suitability solution:** 40 µg/mL of USP Racemic Tamsulosin Hydrochloride RS in methanol

**Sample solution:** 2.0 mg/mL of Tamsulosin Hydrochloride in methanol

**Standard solution:** 2 µg/mL of Tamsulosin Hydrochloride, from the *Sample solution*, in methanol

**Chromatographic system**

(See *Chromatography* ⟨621⟩, *System Suitability*.)

**Mode:** LC

**Detector:** UV 225 nm

**Column:** 4.6-mm × 25-cm; packing L51

**Temperature:** 40°

**Flow rate:** 0.5 mL/min

**Injection size:** 10 µL

**System suitability**

**Sample:** *System suitability solution*

[NOTE—The relative retention times for the optical isomer and tamsulosin are 0.8 and 1.0, respectively.]

**Suitability requirements**

**Resolution:** NLT 2 between the optical isomer and tamsulosin

**Analysis**

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

Calculate the percentage of the optical isomer in the portion of Tamsulosin Hydrochloride taken:

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

$r_U$ = peak response of the optical isomer from the *Sample solution*

$r_S$ = peak response of tamsulosin from the *Standard solution*

$C_S$ = concentration of the *Standard solution* (mg/mL)

$C_U$ = concentration of the *Sample solution* (mg/mL)

**Acceptance criteria**

**Individual impurities:** NMT 0.3% of the optical isomer

- **OPTICAL ROTATION,** *Specific Rotation* ⟨**781S**⟩**:** –17.5° to –20.5° at 20°

**Sample solution:** 7.5 mg/mL in water. [NOTE—Sample should be previously dried at 105° for 2 h; heat at 60°–70° to dissolve, and allow to cool before using.]

- **LOSS ON DRYING** ⟨**731**⟩**:** Dry a sample at 105° for 2 h: it loses NMT 0.5% of its weight.

**ADDITIONAL REQUIREMENTS**

- **PACKAGING AND STORAGE:** Preserve in tight containers, and store at controlled room temperature.
- **USP REFERENCE STANDARDS** ⟨**11**⟩

USP Racemic Tamsulosin Hydrochloride RS

USP Tamsulosin Hydrochloride RS

## Tamsulosin Hydrochloride Capsules

**DEFINITION**

Tamsulosin Hydrochloride Capsules contain NLT 90.0% and NMT 110.0% of the labeled amount of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$).

**IDENTIFICATION**

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

**ASSAY**

- **PROCEDURE 1**

**Solution A:** Dilute 20 mL of hydrochloric acid with water to 1000 mL.

**Solution B:** Dissolve 8.7 mL of perchloric acid and 3.0 g of sodium hydroxide in 1900 mL of water. Adjust with 1 N sodium hydroxide to a pH of 2.0, and add sufficient water to make 2000 mL.

**Mobile phase:** Acetonitrile and *Solution B* (3:7)

**Internal standard solution:** 0.4 mg/mL of propylparaben in a mixture of acetonitrile and water (3:7)

**Standard stock solution:** 0.5 mg/mL of USP Tamsulosin Hydrochloride RS in a mixture of acetonitrile and water (3:7)

**Standard solution:** Transfer 2.0 mL of the *Standard stock solution* to a suitable container, add 5.0 mL of *Internal standard solution*, and add *Mobile phase* to make 40 mL.

**Sample solution:** Weigh the contents of NLT 20 Capsules. Mix the contents, and transfer a weighed portion of the powder, equivalent to about 1 mg of tamsulosin hydrochloride based on the label claim, into a Teflon-lined, screw-capped centrifuge tube. Place approximately 100 glass balls with a diameter of about 5 mm into the tube, add 20 mL of 0.05 N sodium hydroxide, heat at 50° for 10 min, and shake well for 30 min. Add 15 mL of a mixture of acetonitrile and *Solution A* (2:1) to the solution, and shake well. Add 5.0 mL of the *Internal standard solution*, and shake well. Centrifuge at 1500 rpm for 10 min, and use the superna-

tant, passing it if necessary through a membrane filter of pore size 0.5 µm or smaller.

**Chromatographic system**
(See *Chromatography* ⟨621⟩, *System Suitability*.)
**Mode:** LC
**Detector:** UV 225 nm
**Column:** 4.0-mm × 15-cm or 4.6-mm × 15-cm; 5-µm packing L1
**Column temperature:** 40°
**Flow rate:** 1.0 mL/min for the 4.0-mm column and 1.3 mL/min for the 4.6-mm column. [NOTE—The flow rate can be adjusted as needed to achieve a recommended retention time of approximately 6 min for tamsulosin.]
**Injection size:** 10 µL

**System suitability**
**Sample:** *Standard solution*
**Suitability requirements**
**Resolution:** NLT 12 between tamsulosin and propylparaben. [NOTE—The elution order is tamsulosin hydrochloride followed by propylparaben.]
**Relative standard deviation:** NMT 2.0%, for the ratios of the peak areas for tamsulosin and the internal standard

**Analysis**
**Samples:** *Standard solution* and *Sample solution*
Calculate the percentage of the labeled amount of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) in the portion of Capsules taken:

$$\text{Result} = (R_U/R_S) \times (C_S \times V_S/W) \times 100$$

$R_U$ = ratio of the peak areas for tamsulosin and the internal standard from the *Sample solution*
$R_S$ = ratio of the peak areas for tamsulosin and the internal standard from the *Standard solution*
$C_S$ = concentration of USP Tamsulosin Hydrochloride RS in the *Standard stock solution* (mg/mL)
$V_S$ = volume of the *Standard stock solution* taken to prepare the *Standard solution* (mL)
$W$ = amount of tamsulosin hydrochloride, based on the label claim, taken to prepare the *Sample solution* (mg)

**Acceptance criteria:** 90.0%–110.0%

- **PROCEDURE 2:** Use this *Procedure* for Capsules labeled to meet the requirements of *Dissolution Test 2*.

**Solution B** and **Mobile phase:** Proceed as directed for *Procedure 1*.
**Buffer:** Dissolve 3.4 g of monobasic potassium phosphate in 1 L of water. Adjust with 2 N sodium hydroxide to a pH of 5.80 ± 0.05.
**Standard solution:** Prepare a solution containing 1.2 mg/mL of USP Tamsulosin Hydrochloride RS in methanol. Dilute with *Buffer* to obtain a solution containing 3.2 µg/mL.
**Sample solution:** Weigh the contents of NLT 20 Capsules. Mix the contents, and transfer a weighed portion of the Capsule contents, equivalent to 1.6 mg of tamsulosin hydrochloride, into a 100-mL volumetric flask. Add 20 mL of methanol, stir for 30 min, sonicate for 30 min, and stir again for 30 min. Add 40 mL of methanol, sonicate for another 30 min, and stir for another 60 min. Dilute with methanol to volume, mix well, and allow the solution to stand for 5 min. Dilute 5 mL of this solution with *Buffer* to 25 mL, and allow the solution to stand for 5 min. Pass through a PVDF filter of 0.45-µm pore size, discarding the first 5 mL of the filtrate.
**Chromatographic system:** Proceed as directed for *Procedure 1*, except inject 50 µL.

**System suitability**
**Sample:** *Standard solution*
**Suitability requirements**
**Relative standard deviation:** NMT 2.0%

**Analysis**
**Samples:** *Standard solution* and *Sample solution*
Calculate the percentage of the labeled amount of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) in the portion of Capsules taken:

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

$r_U$ = peak response from the *Sample solution*
$r_S$ = peak response from the *Standard solution*
$C_S$ = concentration of the *Standard solution* (mg/mL)
$C_U$ = nominal concentration of the *Sample solution* (mg/mL)

**Acceptance criteria:** 90.0%–110.0%

- **PROCEDURE 3:** Use this *Procedure* for Capsules labeled to meet the requirements of *Dissolution Test 3*.

**Solution B** and **Mobile phase:** Proceed as directed for *Procedure 1*.
**Buffer:** Dissolve 6.9 g of monobasic sodium phosphate monohydrate in 1 L of water, and adjust with 5 N sodium hydroxide to a pH of 7.2 ± 0.05.
**Standard solution:** Prepare a solution containing 0.5 mg/mL of USP Tamsulosin Hydrochloride RS in a mixture of methanol and water (1:1), and dilute a portion of this solution with methanol to obtain a solution containing 0.03 mg/mL.
**Sample solution:** Weigh the contents of NLT 20 Capsules. Mix the contents, and transfer a weighed portion of the Capsule contents, equivalent to 0.8 mg of tamsulosin hydrochloride, into a 25-mL volumetric flask. Add 5 mL of *Buffer*, shake for 15 min, add 10 mL of methanol, shake for 1 h, and dilute with methanol to volume. Pass through a suitable filter of 0.45-µm pore size.
**Chromatographic system:** Proceed as directed for *Procedure 1*, except inject 20 µL.
**System suitability** and **Analysis:** Proceed as directed for *Procedure 2*.
**Acceptance criteria:** 90.0%–110.0%

- **PROCEDURE 4:** For Capsules labeled to meet the requirements of *Dissolution Test 4*, proceed as directed for *Procedure 1*.
- **PROCEDURE 5:** Use this *Procedure* for Capsules labeled to meet the requirements of *Dissolution Test 5*.

**Solution B, Mobile phase,** and **Chromatographic system:** Proceed as directed for *Procedure 1*.
**Diluent:** Methanol and water (75:25)
**Standard solution:** Prepare a solution containing 0.016 mg/mL of USP Tamsulosin Hydrochloride RS in *Diluent*.
**Sample solution:** Transfer the contents of 10 Capsules (equivalent to 4 mg of tamsulosin hydrochloride) into a 250-mL volumetric flask. Add 200 mL of *Diluent*, stir, and sonicate simultaneously for at least 2 h. Cool, and dilute with *Diluent* to volume. Pass through a suitable filter.
**System suitability** and **Analysis:** Proceed as directed for *Procedure 2*.
**Acceptance criteria:** 90.0%–110.0%

- **PROCEDURE 6:** Use this *Procedure* for Capsules labeled to meet the requirements of *Dissolution Test 6*.

**Solution B** and **Mobile phase:** Proceed as directed for *Procedure 1*.
**Buffer:** Dissolve 6.8 g of monobasic potassium phosphate and 0.9 g of sodium hydroxide in 1 L of water. Adjust with sodium hydroxide solution to a pH of 6.8 ± 0.05.
**Diluent A:** Acetonitrile and *Buffer* (1:1)
**Diluent B:** Acetonitrile and *Solution B* (1:1)
**Standard solution:** Prepare a solution containing 0.4 mg/mL of USP Tamsulosin Hydrochloride RS in *Diluent A*, using sonication as necessary. Dilute 2 mL of this solution with *Diluent B* to 100 mL.
**Sample solution:** Weigh the contents of NLT 20 Capsules. Mix the contents, and transfer a weighed portion of the Capsule contents, equivalent to 4 mg of tamsulosin

hydrochloride, into a 100-mL volumetric flask. Add 60 mL of *Diluent A*, and sonicate with intermittent shaking to disperse the pellets completely. Cool, and dilute with *Diluent A* to volume. Centrifuge, transfer 5 mL of supernatant solution to a 25-mL volumetric flask, and dilute with *Diluent B* to volume. Pass through a nylon membrane filter of 0.45-µm pore size.

**Chromatographic system:** Proceed as directed for *Procedure 1*, except inject 20 µL.

**System suitability** and **Analysis:** Proceed as directed for *Procedure 2*.

**Acceptance criteria:** 90.0%–110.0%

- **PROCEDURE 7:** Use this *Procedure* for Capsules labeled to meet the requirements of *Dissolution Test 7*.

**Solution B** and **Mobile phase:** Proceed as directed for *Procedure 1*.

**Buffer:** Dissolve 76 g of tribasic sodium phosphate in 1 L of water.

**Diluent:** 0.1 N hydrochloric acid and *Buffer* (3:1), adjusted with diluted hydrochloric acid or sodium hydroxide solution to a pH of 7.0

**Standard solution:** 0.0016 mg/mL of USP Tamsulosin Hydrochloride RS in *Diluent*

**Sample solution:** Weigh the contents of NLT 20 Capsules. Mix the contents, and transfer a weighed portion of the Capsule contents, equivalent to 0.4 mg of tamsulosin hydrochloride, into a 250-mL volumetric flask, and dilute with *Diluent* to volume. Stir for 24 h by mechanical means at 40° protected from light. Pass through a suitable filter of 0.45-µm pore size.

**Chromatographic system:** Proceed as directed for *Procedure 1*, except inject 50 µL.

**System suitability** and **Analysis:** Proceed as directed for *Procedure 2*.

**Acceptance criteria:** 90.0%–110.0%

- **PROCEDURE 8:** Use this *Procedure* for Capsules labeled to meet the requirements of *Dissolution Test 8*.

**Solution B, Mobile phase,** and **Chromatographic system:** Proceed as directed for *Procedure 1*.

**Standard solution:** Prepare a solution containing 1.0 mg/mL of USP Tamsulosin Hydrochloride RS in methanol. Dilute 5 mL of this solution with *Mobile phase* to 20 mL.

**Sample solution:** Weigh the contents of NLT 20 Capsules. Mix the contents, and transfer a weighed portion of the Capsule contents, equivalent to 2.5 mg of tamsulosin hydrochloride, into a 100-mL volumetric flask. Add 25 mL of 0.1 N sodium hydroxide, and sonicate for 30 min. Add 30 mL of *Mobile phase*, and shake by mechanical means for 30 min. Centrifuge, and pass through a PVDF membrane filter of 0.45-µm pore size, discarding the first few mL of the filtrate.

**System suitability** and **Analysis:** Proceed as directed for *Procedure 2*.

**Acceptance criteria:** 90.0%–110.0%

- **PROCEDURE 9:** For Capsules labeled to meet the requirements of *Dissolution Test 9*, proceed as directed for *Procedure 1*.

## PERFORMANCE TESTS

- **DISSOLUTION** ⟨**711**⟩

**Test 1**

**Acid stage medium:** Dissolve 2.0 g of sodium chloride in 5.7 mL of hydrochloric acid, and add water to make up to 1000 mL. To 500 mL of this fluid add 1 mL of polysorbate 80 aqueous solution (3 g in 200 mL of water) just before the test; 500 mL.

**Buffer stage medium:** Phosphate buffer, pH 7.2 (dissolve 6.8 g of monobasic potassium phosphate in 250 mL of water, add 90 mL of 0.2 N sodium hydroxide and 500 mL of water, adjust with 0.2 N sodium hydroxide or 0.2 N hydrochloric acid to a pH of 7.2 ± 0.05, and dilute with water to volume); 500 mL

**Apparatus 2:** 100 rpm, with sinker (see *Figure 2a* in *Dissolution* ⟨711⟩)

**Times:** 2, 3, and 8 h

**Analysis:** Perform the test using *Acid stage medium*. At 2 h after the start of the test, withdraw 10.0 mL of the solution under test (T1). Drain the *Acid stage medium* immediately by suction through a tube capped with a 60-mesh stainless wire screen. Rinse the drain tube while adding the *Buffer stage medium* previously warmed, and continue the test. At 3 h after the start of the test (1 h after replacement of the *Medium*), withdraw 10.0 mL of the solution under test (T2), replace the same volume with warmed *Buffer stage medium*, and continue the test. At 8 h after the start of the test (6 h after the replacement of the *Medium*), withdraw 10.0 mL of the solution under test (T3).

**Internal standard solution:** 0.008 mg/mL of propylparaben in acetonitrile and water (3:7)

**Standard solution:** Prepare a solution containing 0.5 mg/mL of USP Tamsulosin Hydrochloride RS in acetonitrile and water (3:7). Transfer 4.0 mL of this solution to a 100-mL volumetric flask, and dilute with *Acid stage medium* to volume. Transfer 4.0 mL of this dilution to another 100-mL volumetric flask, and dilute with *Acid stage medium* to volume. This solution has a known concentration ($C_S$) of about 0.8 µg/mL of tamsulosin hydrochloride. Transfer 10.0 mL of this last dilution to a test tube, and add 2.0 mL of the *Internal standard solution*.

**Sample solutions:** Add 2.0 mL of the *Internal standard solution* to T1, mix well, and pass through a suitable filter of 0.5-µm pore size, discarding the first 5 mL. Add 1.0 mL of 0.5 N hydrochloric acid and 2.0 mL of *Internal standard solution* to T2, mix well, and pass through a suitable filter of 0.5-µm pore size, discarding the first 5 mL. Add 1.0 mL of 0.5 N hydrochloric acid and 2.0 mL of *Internal standard solution* to T3. Mix well, and pass through a suitable filter of 0.5-µm pore size, discarding the first 5 mL.

**Chromatographic system:** Proceed as directed in the *Assay, Procedure 1*, but using the *Standard solution* described for *Dissolution*, and inject 250 µL instead of 10 µL.

Calculate the ratios ($R_{T1}$, $R_{T2}$, $R_{T3}$, and $R_S$) of the peak area of tamsulosin to that of the internal standard for all *Sample solutions* and the *Standard solution*. Calculate the percentage of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) dissolved at each of the following time points.

At 2 h:

$$D_1 = [(C_S \times V)/L] \times (R_{T1}/R_S) \times 100$$

At 3 h:

$$D_2 = [(C_S \times V)/L] \times [(R_{T2}/R_S) + R_{T1}/R_S)] \times 100$$

At 8 h:

$$D_3 = [(C_S \times V)/L] \times [(R_{T3}/R_S) + (R_{T2}/R_S \times V_{T2}/V) + (R_{T1}/R_S)] \times 100$$

$C_S$ = concentration of the *Standard solution*
$V$ = volume of *Medium*, 500 mL
$L$ = label claim (mg/Capsule)
$V_{T2}$ = volume of the withdrawn aliquot of T2, 10 mL

**Tolerances:** See *Table 1*.

**Table 1**

| Time (h) | Amount Dissolved |
|---|---|
| 2 | 13%–34% |
| 3 | 47%–68% |
| 8 | NLT 80% |

The percentages of the labeled amount of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) dissolved at the times specified conform to *Acceptance Table 2* in *Dissolution* ⟨711⟩.

**Test 2:** If the product complies with this test, the labeling indicates that it meets USP *Dissolution Test 2*.
**Acid stage medium:** 0.003% polysorbate 80, pH 1.2 (dilute 8.5 mL of hydrochloric acid with water to 900 mL, add 0.03 mL of polysorbate 80, adjust with 0.2 N sodium hydroxide or 0.2 N hydrochloric acid to a pH of 1.2 ± 0.05, and dilute with water to 1000 mL); 500 mL
**Buffer stage medium:** Phosphate buffer, pH 7.2 (proceed as directed for *Test 1*); 500 mL
**Apparatus 2:** 100 rpm, with sinkers[1]
**Times:** 2 h for the *Acid stage medium* and 6 h for the *Buffer stage medium*; 8 h total test time
**Analysis:** Perform the test using *Acid stage medium*. At 2 h after the start of the test, withdraw a sample of the solution under test. Carefully discard the *Acid stage medium* and replace it with the *Buffer stage medium* previously warmed, and continue the test. At 6 h after the replacement of the *Medium*, withdraw a sample of the solution under test.
**Standard stock solution:** Transfer about 25.0 mg of USP Tamsulosin Hydrochloride RS to a 50-mL volumetric flask. Add 25 mL of methanol. Dilute with *Buffer stage medium* to volume. Sonicate until dissolved.
**Standard solution:** Dilute the *Standard stock solution* with *Buffer stage medium* to obtain a final concentration of 0.8 µg/mL.
**Sample solution:** Pass a portion of the solution under test through a suitable filter.
**Buffer solution:** 3.4 g/L of monobasic potassium phosphate in water. Adjust with 2 N sodium hydroxide to a pH of 5.80 ± 0.05.
**Mobile phase:** Acetonitrile and *Buffer solution* (1:3)
**Chromatographic system**
(See *Chromatography* ⟨621⟩*, System Suitability*.)
**Mode:** LC
**Detector:** UV 225 nm
**Column:** 4.6-mm × 15-cm; 5-µm packing L1
**Flow rate:** 1.0 mL/min
**Injection size:** 50 µL
**System suitability**
**Sample:** *Standard solution*
**Suitability requirements**
**Column efficiency:** NLT 2000
**Tailing factor:** NMT 2.0
**Relative standard deviation:** NMT 2.0%
Calculate the concentration of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) in the *Medium* at each time point ($C_i$).
At 2 h:

$$C_1 = (r_U/r_S) \times C_S$$

At 6 h:

$$C_2 = (r_U/r_S) \times C_S$$

$r_U$ = peak response from the *Sample solution*
$r_S$ = peak response from the *Standard solution*
$C_S$ = concentration of the *Standard solution* (mg/mL)
Calculate the percentage of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) dissolved at each time point, ($Q_i$).
At 2 h:

$$Q_1 = (C_1 \times V) \times 100/L$$

At 6 h:

$$Q_2 = (C_1 + C_2) \times (V/L) \times 100$$

$V$ = volume of *Medium* (mL)
$L$ = label claim (mg/Capsule)
**Tolerances:** NMT 25% of the labeled amount of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) is dissolved in 2 h. NLT 85% of the labeled amount of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) is dissolved in 8 h.

[1] A suitable sinker is available as catalog number CAPWHT-2S from www.qla-llc.com.

**Test 3:** If the product complies with this test, the labeling indicates that it meets USP *Dissolution Test 3*.
**Acid stage**
**Acid stage medium:** 0.003% polysorbate 80, pH 1.2 (proceed as directed for *Test 2*); 500 mL
**Apparatus 2:** 100 rpm, with sinkers
**Time:** 2 h
**Analysis:** Pass a portion of the solution under test through a suitable filter. Leave the remaining *Acid stage medium* in the vessel and proceed with the *Buffer stage*.
**Tolerances:** NMT 10% ($Q$) of the labeled amount of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) is dissolved.
**Buffer stage**
**Buffer stage concentrate:** 0.1 M phosphate buffer, pH 11.6 (138 g of sodium phosphate monohydrate and 320 mL of 5 N sodium hydroxide in 10 L of water. Adjust with 5 N sodium hydroxide to a pH of 11.6 ± 0.05.)
**Buffer stage medium:** Add 500 mL of the *Buffer stage concentrate* to the remaining *Acid stage medium* in each vessel. The final pH is about 7.2.
**Apparatus 2:** 100 rpm, with sinkers
**Time:** 3 and 8 h, including the 2 h in the *Acid stage medium*
**Standard stock solution:** 0.5 mg/mL of USP Tamsulosin Hydrochloride RS in water and methanol (1:1)
**Standard solution:** Dilute the *Standard stock solution* with *Buffer stage medium* to obtain a final concentration of 0.3 µg/mL.
**Sample solution:** Pass a portion of the solution under test through a suitable filter.
**pH 5.5 buffer solution:** 6.8 g/L of monobasic potassium phosphate in water. Adjust with 2 N sodium hydroxide to a pH of 5.5 ± 0.05.
**Mobile phase:** Acetonitrile and *pH 5.5 buffer solution* (2:3)
**Chromatographic system**
(See *Chromatography* ⟨621⟩*, System Suitability*.)
**Mode:** LC
**Detector:** UV 275 nm
**Column:** 4.6-mm × 15-cm; 5-µm packing L1
**Column temperature:** 30°
**Flow rate:** 1 mL/min
**Injection size:** 100 µL
**System suitability**
**Sample:** *Standard solution*
**Suitability requirements**
**Tailing factor:** NMT 2.0
**Relative standard deviation:** NMT 2.5%
Calculate the concentration of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) dissolved at each time point ($C_i$).
At 3 h:

$$C_1 = (r_U/r_S) \times C_S$$

At 8 h:

$$C_2 = (r_U/r_S) \times C_S$$

$r_U$ = peak response from the *Sample solution*
$r_S$ = peak response from the *Standard solution*
$C_S$ = concentration of the *Standard solution* (mg/mL)
Calculate the cumulative percentage of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) dissolved at each time point ($Q_i$):

$$Q_i = (C_1V_1 + \sum_{i=2}^{n-1} C_iV_S + C_nV) \times (100/L)$$

$V_1$ = volume of *Acid stage medium*, 500 mL
$V_S$ = volume of sample taken (mL)
$C_n$ = concentration of tamsulosin hydrochloride at each time point
$V$ = volume of *Buffer stage medium*, 1000 mL

$L$ = label claim (mg/Capsule)
**Tolerances:** See *Table 2*.

**Table 2**

| Time (h) | Amount Dissolved |
|---|---|
| 3 | 65%–85% |
| 8 | NLT 80% |

The percentages of the labeled amount of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) dissolved at the times specified conform to *Acceptance Table 2* in *Dissolution* ⟨711⟩.

**Test 4:** If the product complies with this test, the labeling indicates that it meets USP *Dissolution Test 4*.
**Acid stage medium:** 0.003% polysorbate 80, pH 1.2 (proceed as directed for *Test 2*); 500 mL
**Buffer stage medium:** Phosphate buffer, pH 7.2 (proceed as directed for *Test 1*); 500 mL
**Apparatus 2:** 100 rpm, with sinkers
**Times:** 2 h for the *Acid stage medium*, and 3 and 8 h for the *Buffer stage medium* (including the 2 h in the *Acid stage medium*)
**Analysis:** Perform the test using the *Acid stage medium*. At 2 h after the start of the test, withdraw a sample of the solution under test. Carefully discard the *Acid stage medium*, replace it with the *Buffer stage medium* previously warmed, and continue the test. At 1 h and 6 h after the replacement of the *Medium*, withdraw a sample of the solution under test. Replace the volume of *Medium* withdrawn with the same volume of *Buffer stage medium*, previously warmed.
**50 mM sodium perchlorate solution:** Dissolve 7.0 g of monohydrate sodium perchlorate in 1 L of water, and add 5 mL of phosphoric acid.
**Mobile phase:** *50 mM sodium perchlorate solution* and acetonitrile (3:2)
**Acid stage standard stock solution:** Transfer 20 mg of USP Tamsulosin Hydrochloride RS to a 500-mL volumetric flask, add 25 mL of methanol, and sonicate until dissolved. Dilute with *Acid stage medium* to volume.
**Acid stage standard solution:** Dilute the *Acid stage standard stock solution* with *Acid stage medium* to obtain a final concentration of about 0.08 µg/mL.
**Buffer stage standard stock solution:** Transfer 20 mg of USP Tamsulosin Hydrochloride RS to a 250-mL volumetric flask, add 25 mL of methanol, and sonicate until dissolved. Dilute with *Buffer stage medium* to volume.
**Buffer stage standard solution:** Dilute the *Buffer stage standard stock solution* with *Buffer stage medium* to obtain a final concentration of about 0.8 µg/mL.
**Sample solution:** Pass a portion of the solution under test through a suitable filter.
**Chromatographic system**
(See *Chromatography* ⟨621⟩, *System Suitability*.)
**Mode:** LC
**Detector:** UV 230 nm
**Analytical column:** 3.9-mm × 15-cm; 5-µm packing L1
**Guard column:** 3-mm × 4-cm; packing L1
**Column temperature:** 35°
**Flow rate:** 1.5 mL/min
**Injection size:** 200 µL
**System suitability**
**Samples:** *Acid stage standard solution* and *Buffer stage standard solution*
**Suitability requirements**
**Column efficiency:** NLT 1600, *Acid stage standard solution*; NLT 1300, *Buffer stage standard solution*
**Relative standard deviation:** NMT 3.0%, *Acid stage standard solution*; NMT 1.5%, *Buffer stage standard solution*
Calculate the concentration of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) dissolved at each time point ($C_i$).

At 2 h:

$$C_1 = (r_U/r_S) \times C_S$$

At 3 h:

$$C_2 = (r_U/r_S) \times C_S$$

At 8 h:

$$C_3 = (r_U/r_S) \times C_S$$

$r_U$ = peak response from the *Sample solution*
$r_S$ = peak response from the *Standard solution*
$C_S$ = concentration of the *Standard solution* (mg/mL)
Calculate the cumulative percentage of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) dissolved at each time point ($Q_i$):

$$Q_i = (C_1V_1 + \sum_{i=2}^{n-1} C_iV_S + C_nV) \times (100/L)$$

$V_1$ = volume of *Acid stage medium*, 500 mL
$V_S$ = volume of sample taken (mL)
$C_n$ = concentration of tamsulosin hydrochloride at each time point
$V$ = volume of *Buffer stage medium*, 1000 mL
$L$ = label claim (mg/Capsule)
**Tolerances:** See *Table 3*.

**Table 3**

| Time (h) | Amount Dissolved |
|---|---|
| 2 | 0%–10% |
| 3 | 45%–68% |
| 8 | NLT 80% |

The percentages of the labeled amount of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) dissolved at the times specified in the *Buffer stage* conform to *Acceptance Table 2* in *Dissolution* ⟨711⟩.

**Test 5:** If the product complies with this test, the labeling indicates that it meets USP *Dissolution Test 5*.
**Acid stage medium:** 0.003% polysorbate 80, pH 1.2 (proceed as directed for *Test 2*); 500 mL
**Buffer stage medium:** Phosphate buffer, pH 7.2 (proceed as directed for *Test 1*); 500 mL
**Apparatus 2:** 50 rpm, with sinkers
**Times:** 2 h for the *Acid stage medium*, and 3 and 5 h for the *Buffer stage medium* (including the 2 h in the *Acid stage medium*)
**Buffer solution:** Dissolve 1.0 g of octanesulfonic acid sodium salt and 1.4 g of monobasic potassium phosphate in 1 L of water. Adjust with potassium hydroxide to a pH of 6.5 ± 0.05.
**Mobile phase:** *Buffer solution* and acetonitrile (3:2)
**Standard stock solution:** 0.04 mg/mL of USP Tamsulosin Hydrochloride RS in *Buffer stage medium*
**Standard solution:** Dilute the *Standard stock solution* with *Buffer stage medium* to obtain a final concentration of 0.8 µg/mL.
**Sample solution:** Centrifuge a portion of the solution under test at NMT 3000 rpm for NLT 20 min.
**Analysis:** Perform the test using *Acid stage medium*. At 2 h after the start of the test, withdraw a sample of the solution under test. Carefully discard the *Acid stage medium*, replace it with the *Buffer stage medium* previously warmed, and continue the test. At 1 and 3 h after the replacement of the *Medium*, withdraw a sample of the solution under test. Replace the volume of *Medium* withdrawn with the same volume of *Buffer stage medium*, previously warmed.
**Chromatographic system**
(See *Chromatography* ⟨621⟩, *System Suitability*.)

**Mode:** LC
**Detector:** UV 225 nm
**Column:** 3.9-mm × 7.5-cm; 5-µm packing L7
**Column temperature:** 30°
**Flow rate:** 1.5 mL/min
**Injection size:** 100 µL

**System suitability**
**Sample:** *Standard solution*
**Suitability requirements**
**Relative standard deviation:** NMT 4.0%

Calculate the concentration of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) dissolved at each time point ($C_i$).

At 2 h:

$$C_1 = (r_U/r_S) \times C_S$$

At 3 h:

$$C_2 = (r_U/r_S) \times C_S$$

At 5 h:

$$C_3 = (r_U/r_S) \times C_S$$

$r_U$ = peak response from the *Sample solution*
$r_S$ = peak response from the *Standard solution*
$C_S$ = concentration of the *Standard solution* (mg/mL)

Calculate the cumulative percentage of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) dissolved at each time point ($Q_i$):

$$Q_i = (C_1V_1 + \sum_{i=2}^{n-1} C_iV_S + C_nV) \times (100/L)$$

$V_1$ = volume of *Acid stage medium*, 500 mL
$V_S$ = volume of sample taken (mL)
$C_n$ = concentration of tamsulosin hydrochloride at each time point
$V$ = volume of *Buffer stage medium*, 500 mL
$L$ = label claim (mg/Capsule)

**Tolerances:** See *Table 4*.

**Table 4**

| Time (h) | Amount Dissolved |
|---|---|
| 2 | 15%–35% |
| 3 | 60%–80% |
| 5 | NLT 80% |

The percentages of the labeled amount of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) dissolved at the times specified conform to *Acceptance Table 2* in *Dissolution* ⟨711⟩.

**Test 6:** If the product complies with this test, the labeling indicates that it meets USP *Dissolution Test 6*.

**Acid stage medium:** 0.003% polysorbate 80, pH 1.2 (proceed as directed for *Test 2*); 500 mL

**Buffer stage medium:** Phosphate buffer, pH 7.2 (proceed as directed for *Test 1*); 500 mL

**Apparatus 2:** 100 rpm using a 40-mesh basket as a sinker, and the paddle height adjusted at 4.5 cm from the bottom of the vessel

**Times:** 2 h for the *Acid stage medium*, and 3 and 8 h for the *Buffer stage medium* (including the 2 h in the *Acid stage medium*)

**Analysis:** Perform the test using *Acid stage medium*. At 2 h after the start of the test, withdraw a sample of the solution under test. Carefully discard the *Acid stage medium*, replace it with the *Buffer stage medium* previously warmed, and continue the test. At 1 h and 6 h after the replacement of the *Medium*, withdraw a sample of the solution under test. Replace the volume of *Medium* withdrawn with the same volume of *Buffer stage medium*, previously warmed.

**Buffer solution:** Dissolve 3 g of sodium hydroxide and 8.7 mL of perchloric acid in 1900 mL of water, adjust with 0.5 M sodium hydroxide to a pH of 2.0 ± 0.05, and dilute with water to 2000 mL.

**Mobile phase:** *Buffer solution* and acetonitrile (7:3)

**Standard stock solution:** 0.5 mg/mL of USP Tamsulosin Hydrochloride RS in methanol

**Acid stage standard solution:** Dilute the *Standard stock solution* with *Acid stage medium* to obtain a final concentration of 0.8 µg/mL.

**Buffer stage standard solution:** Dilute the *Standard stock solution* with *Buffer stage medium* to obtain a final concentration of 0.8 µg/mL.

**Sample solution:** Pass a portion of the solution under test through a suitable filter of 0.45-µm pore size.

**Chromatographic system**
(See *Chromatography* ⟨621⟩, *System Suitability*.)
**Mode:** LC
**Detector:** UV 225 nm
**Column:** 4.6-mm × 15-cm, 3-µm packing L1
**Column temperature:** 40°
**Flow rate:** 1.5 mL/min
**Injection size:** 100 µL

**System suitability**
**Samples:** *Acid stage standard solution* and *Buffer stage standard solution*
**Suitability requirements**
**Column efficiency:** NLT 7000
**Tailing factor:** NMT 2.0
**Relative standard deviation:** NMT 2.0%

Calculate the concentration of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) dissolved at each time point ($C_i$).

At 2 h:

$$C_1 = (r_U/r_S) \times C_S$$

At 3 h:

$$C_2 = (r_U/r_S) \times C_S$$

At 8 h:

$$C_3 = (r_U/r_S) \times C_S$$

$r_U$ = peak response from the *Sample solution*
$r_S$ = peak response from the *Standard solution*
$C_S$ = concentration of the *Standard solution* (mg/mL)

Calculate the cumulative percentage of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) dissolved at each time point ($Q_i$):

$$Q_i = (C_1V_1 + \sum_{i=2}^{n-1} C_iV_S + C_nV) \times (100/L)$$

$V_1$ = volume of *Acid stage medium*, 500 mL
$V_S$ = volume of sample taken (mL)
$C_n$ = concentration of tamsulosin hydrochloride at each time point
$V$ = volume of *Buffer stage medium*, 500 mL
$L$ = label claim (mg/Capsule)

**Tolerances:** See *Table 5*.

**Table 5**

| Time (h) | Amount Dissolved |
|---|---|
| 2 | 0%–20% |
| 3 | 47%–68% |
| 8 | NLT 80% |

The percentages of the labeled amount of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) dissolved at the times specified conform to *Acceptance Table 2* in *Dissolution* ⟨711⟩.

**Test 7:** If the product complies with this test, the labeling indicates that it meets USP *Dissolution Test 7*.
**Acid stage medium:** 0.003% polysorbate 80, pH 1.2 (proceed as directed for *Test 2*); 500 mL
**Buffer stage medium:** Phosphate buffer, pH 7.2 (proceed as directed for *Test 1*); 500 mL, deaerated
**Apparatus 2:** 100 rpm, with wire helix sinkers
**Times:** 2 h in the *Acid stage medium*, and 4 and 12 h in the *Buffer stage medium* (including the 2 h in the *Acid stage medium*)
**Standard stock solution:** 1 mg/mL of USP Tamsulosin Hydrochloride RS in alcohol
**Acid stage standard solution:** Dilute the *Standard stock solution* with *Acid stage medium* to obtain a final concentration of about 0.2 µg/mL.
**Buffer stage standard solution:** Dilute the *Standard stock solution* with *Buffer stage medium* to obtain a final concentration of about 0.8 µg/mL.
**Sample solution:** Pass a portion of the solution under test through a suitable filter of 0.45-µm pore size.
**Analysis:** Perform the test using *Acid stage medium*. At 2 h after the start of the test, withdraw a sample of the solution under test. Carefully discard the *Acid stage medium*, replace it with the *Buffer stage medium* previously warmed, and continue the test. At 1 and 6 h after the replacement of the *Medium*, withdraw a sample of the solution under test. Replace the volume of *Medium* withdrawn with the same volume of *Buffer stage medium*, previously warmed.
**Mobile phase A:** 2.76 g/L of monobasic potassium phosphate in water. Adjust with phosphoric acid to a pH of 2.5 ± 0.05.
**Mobile phase B:** Acetonitrile
**Gradient program:** See *Table 6*.

**Table 6**

| Time (min) | Mobile phase A (%) | Mobile phase B (%) |
|---|---|---|
| 0 | 86 | 14 |
| 3 | 86 | 14 |
| 4.5 | 30 | 70 |
| 5 | 86 | 14 |

**Chromatographic system**
(See *Chromatography* ⟨621⟩, *System Suitability*.)
**Mode:** LC
**Detector:** UV 225 nm
**Column:** 2-mm × 5-cm; packing L1
**Column temperature:** 30°
**Flow rate:** 1.5 mL/min
**Injection size:** 100 µL
**System suitability**
**Samples:** *Acid stage standard solution* and *Buffer stage standard solution*
**Suitability requirements**
**Tailing factor:** NMT 1.5
**Relative standard deviation:** NMT 2.0%
Calculate the concentration of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) dissolved at each time point ($C_i$).
At 2 h:

$$C_1 = (r_U/r_S) \times C_S$$

At 4 h:

$$C_2 = (r_U/r_S) \times C_S$$

At 12 h:

$$C_3 = (r_U/r_S) \times C_S$$

$r_U$ = peak response from the *Sample solution*
$r_S$ = peak response from the *Standard solution*
$C_S$ = concentration of the *Standard solution* (mg/mL)
Calculate the cumulative percentage of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) dissolved at each time point ($Q_i$):

$$Q_i = (C_1V_1 + \sum_{i=2}^{n-1} C_iV_S + C_nV) \times (100/L)$$

$V_1$ = volume of *Acid stage medium*, 500 mL
$V_S$ = volume of sample taken (mL)
$C_n$ = concentration of tamsulosin hydrochloride at each time point
$V$ = volume of *Buffer stage medium*, 500 mL
$L$ = label claim (mg/Capsule)
**Tolerances:** See *Table 7*.

**Table 7**

| Time (h) | Amount Dissolved |
|---|---|
| 2 | 5%–25% |
| 4 | 46%–66% |
| 12 | NLT 80% |

The percentages of the labeled amount of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) dissolved at the times specified conform to *Acceptance Table 2* in *Dissolution* ⟨711⟩.
**Test 8:** If the product complies with this test, the labeling indicates that it meets USP *Dissolution Test 8*.
**Acid stage medium:** 0.003% polysorbate 80, pH 1.2 (proceed as directed for *Test 2*); 500 mL
**Buffer stage medium:** Phosphate buffer, pH 7.2 (proceed as directed for *Test 1*); 500 mL
**Apparatus 2:** 100 rpm, with sinkers
**Times:** 2 h in the *Acid stage medium*, and 3 and 8 h in the *Buffer stage medium* (including the 2 h in the *Acid stage medium*)
**Standard stock solution:** 0.4 mg/mL of USP Tamsulosin Hydrochloride RS in methanol
**Acid stage standard solution:** Dilute the *Standard stock solution* with *Acid stage medium* to obtain a final concentration of 0.8 µg/mL.
**Buffer stage standard solution:** Dilute the *Standard stock solution* with *Buffer stage medium* to obtain a final concentration of 0.8 µg/mL.
**Analysis:** Perform the test using *Acid stage medium*. At 2 h after the start of the test, withdraw a sample of the solution under test. Carefully discard the *Acid stage medium*, replace it with the *Buffer stage medium* previously warmed, and continue the test. At 1 and 6 h after the replacement of the *Medium*, withdraw a sample of the solution under test. Replace the volume of *Medium* withdrawn with the same volume of *Buffer stage medium*, previously warmed.
**Sample solution:** Pass a portion of the solution under test through a suitable filter of 0.45-µm pore size.
**Buffer solution:** Dissolve 6.8 g of dibasic ammonium phosphate in 800 mL of water, and add 2 mL of triethylamine. Adjust with phosphoric acid to a pH of 6.5. Dilute with water to 1000 mL.
**Mobile phase:** *Buffer solution* and acetonitrile (7:3)
**Chromatographic system**
(See *Chromatography* ⟨621⟩, *System Suitability*.)

**Mode:** LC
**Detector:** UV 225 nm
**Column:** 4.6-mm × 15-cm; 5-µm packing L1
**Column temperature:** 30°
**Flow rate:** 1.0 mL/min
**Injection size:** 100 µL

**Suitability requirements**
**Sample:** *Buffer stage standard solution*
**Column efficiency:** NLT 2000 theoretical plates
**Tailing factor:** NMT 2.0
**Relative standard deviation:** NMT 2.0%

Calculate the concentration of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) dissolved at each time point ($C_i$).

At 2 h:

$$C_1 = (r_U/r_S) \times C_S$$

At 3 h:

$$C_2 = (r_U/r_S) \times C_S$$

At 8 h:

$$C_3 = (r_U/r_S) \times C_S$$

$r_U$ = peak response from the *Sample solution*
$r_S$ = peak response from the *Standard solution*
$C_S$ = concentration of the *Standard solution* (mg/mL)

Calculate the cumulative percentage of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) dissolved at each time point ($Q_i$):

$$Q_i = (C_1V_1 + \sum_{i=2}^{n-1} C_iV_S + C_nV) \times (100/L)$$

$V_1$ = volume of *Acid stage medium*, 500 mL
$V_S$ = volume of sample taken (mL)
$C_n$ = concentration of tamsulosin hydrochloride at each time point
$V$ = volume of *Buffer stage medium*, 500 mL
$L$ = label claim (mg/Capsule)

**Tolerances:** See *Table 8*.

**Table 8**

| Time (h) | Amount Dissolved |
|---|---|
| 2 | 0%–10% |
| 3 | 50%–70% |
| 8 | NLT 80% |

The percentages of the labeled amount of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) dissolved at the times specified conform to *Acceptance Table 2* in *Dissolution* ⟨711⟩.

**Test 9** If the product complies with this test, the labeling indicates that it meets USP *Dissolution Test 9*.

**Acid stage medium:** 0.003% polysorbate 80, pH 1.2 (proceed as directed for *Test 2*); 500 mL
**Buffer stage medium:** Phosphate buffer, pH 7.2 (proceed as directed for *Test 1*); 500 mL
**Apparatus 2:** 100 rpm, with helix wire coil sinker
**Times:** 2 h in *Acid stage medium*, and 3 and 8 h in *Buffer stage medium* (including the 2 h in *Acid stage medium*)
**Standard stock solution:** 0.05 mg/mL of USP Tamsulosin Hydrochloride RS in methanol
**Acid stage standard solution:** Dilute the *Standard stock solution* with *Acid stage medium* to obtain a final concentration of 0.8 µg/mL.
**Buffer stage standard solution:** Dilute the *Standard stock solution* with *Buffer stage medium* to obtain a final concentration of 0.8 µg/mL.
**Sample solution:** Pass a portion of the solution under test through a suitable filter of 0.45-µm pore size.
**Analysis:** Perform the test using *Acid stage medium*. At 2 h after the start of the test, withdraw a sample of the solution under test. Carefully discard the *Acid stage medium*, replace it with the *Buffer stage medium* previously warmed, and continue the test. At 1 and 6 h after the replacement of the *Medium*, withdraw a sample of the solution under test. Replace the volume of *Medium* withdrawn with the same volume of *Buffer stage medium*, previously warmed.
**Buffer:** 3.45 g/L of monobasic ammonium phosphate in water. Adjust with triethylamine to a pH of 6.5 ± 0.05.
**Mobile phase:** *Buffer* and acetonitrile (3:2). Add 1 g of sodium 1-pentanesulfonate per L of the mixture.

**Chromatographic system**
(See *Chromatography* ⟨621⟩, *System Suitability*.)
**Mode:** LC
**Detector:** UV 225 nm
**Column:** 4.6-mm × 25-cm; 5-µm packing L7
**Flow rate:** 1.0 mL/min
**Injection size:** 100 µL

**System suitability**
**Sample:** *Acid stage standard solution* or *Buffer stage standard solution*
**Suitability requirements**
**Column efficiency:** NLT 3000 theoretical plates
**Tailing factor:** NMT 2.0
**Relative standard deviation:** NMT 2.0%

Calculate the concentration of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) dissolved at each time point ($C_i$).

At 2 h:

$$C_1 = (r_U/r_S) \times C_S$$

At 3 h:

$$C_2 = (r_U/r_S) \times C_S$$

At 8 h:

$$C_3 = (r_U/r_S) \times C_S$$

$r_U$ = peak response from the *Sample solution*
$r_S$ = peak response from the appropriate *Standard solution*
$C_S$ = concentration of the appropriate *Standard solution* (mg/mL)

Calculate the cumulative percentage of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) dissolved at each time point ($Q_i$):

$$Q_i = (C_1V_1 + \sum_{i=2}^{n-1} C_iV_S + C_nV) \times (100/L)$$

$V_1$ = volume of *Acid stage medium*, 500 mL
$V_S$ = volume of sample taken (mL)
$C_n$ = concentration of tamsulosin hydrochloride at each time point
$V$ = volume of *Buffer stage medium*, 500 mL
$L$ = label claim (mg/Capsule)

**Tolerances:** See *Table 9*.

**Table 9**

| Time (h) | Amount Dissolved |
|---|---|
| 2 | 0%–20% |
| 3 | 45%–65% |
| 8 | NLT 80% |

The percentages of the labeled amount of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) dissolved at the times specified conform to *Acceptance Table 2* in *Dissolution* ⟨711⟩.

• **Uniformity of Dosage Units ⟨905⟩:** Meet the requirements
**Procedure for content uniformity**
[Note—Use the following *Procedure for content uniformity* if *Procedure 1* is used in the *Assay*. For all other formulations, proceed as directed in the test for *Uniformity of Dosage Units* ⟨905⟩.]
**Solution A, Solution B, Mobile phase,** and **Standard stock solution:** Prepare as directed in the *Assay*, *Procedure 1*.
**Internal standard solution:** 0.16 mg/mL of propylparaben in a mixture of acetonitrile and water (3:7)
**Standard stock solution 1:** Transfer 5.0 mL of the *Standard stock solution* to a 25-mL volumetric flask, and dilute with a mixture of acetonitrile and water (3:7) to volume.
**Standard solution:** Transfer 4.0 mL of *Standard stock solution 1* to a suitable container. Add 5.0 mL of the *Internal standard solution,* and add the *Mobile phase* to make 40 mL.
**Sample solution:** Place the contents of 1 Capsule into a Teflon-lined, screw-capped centrifuge tube. Place approximately 100 glass balls with a diameter of about 5 mm into the tube, add 20 mL of 0.05 N sodium hydroxide, heat at 50° for 10 min, and shake well for 30 min. Add 15 mL of a mixture of acetonitrile and *Solution A* (2:1) to the solution, and shake well. Add 5.0 mL of the *Internal standard solution,* and shake well. Centrifuge at 1500 rpm for 10 min, and use the supernatant, passing it if necessary through a membrane filter of pore size 0.5 µm or smaller.
**Chromatographic system:** Proceed as directed in the *Assay*, *Procedure 1*, except inject 25 µL.
**Analysis**
**Samples:** *Standard solution* and *Sample solution*
Calculate the percentage of the labeled amount of tamsulosin hydrochloride ($C_{20}H_{28}N_2O_5S \cdot HCl$) in the Capsule taken:

$$\text{Result} = (R_U/R_S) \times (C_S \times V_S/L) \times 100$$

$R_U$ = ratio of the peak areas for tamsulosin and the internal standard from the *Sample solution*
$R_S$ = ratio of the peak areas for tamsulosin and the internal standard from the *Standard solution*
$C_S$ = concentration of USP Tamsulosin Hydrochloride RS in *Standard stock solution 1* (mg/mL)
$V_S$ = volume of *Standard stock solution 1* taken to prepare the *Standard solution* (mL)
$L$ = label claim (mg/Capsule)

**ADDITIONAL REQUIREMENTS**
• **Packaging and Storage:** Preserve in tight containers. Store at controlled room temperature.
• **Labeling:** When more than one *Dissolution* test is given, the labeling states the *Dissolution* test used only if *Test 1* is not used.
• **USP Reference Standards ⟨11⟩**
USP Tamsulosin Hydrochloride RS
(–)-(*R*)-5-[2-[[2-(*o*-Ethoxyphenoxy)ethyl]amino]propyl]-2-methoxybenzenesulfonamide monohydrochloride.
$C_{20}H_{28}N_2O_5S \cdot HCl$ 444.97

## Tannic Acid

Tannin.
Tannic acid; Tannin [1401-55-4].

» Tannic Acid is a tannin usually obtained from nutgalls, the excrescences produced on the young twigs of *Quercus infectoria* Oliver, and allied species of *Quercus* Linné (Fam. Fagaceae), from the seed pods of Tara (*Caesalpinia spinosa*), or from the nutgalls or leaves of sumac (any of a genus *Rhus*).

**Packaging and storage—**Preserve in tight, light-resistant containers.
**Identification—**
**A:** To 2 mL of a solution (1 in 10) add 1 drop of ferric chloride TS: a bluish black color or precipitate results.
**B:** To a solution (1 in 10) add an equal volume of gelatin solution (1 in 100): a precipitate is formed.
**Loss on drying** ⟨731⟩**—**Dry it at 105° for 2 hours: it loses not more than 12.0% of its weight.
**Residue on ignition** ⟨281⟩: not more than 1.0%.
**Arsenic,** *Method II* ⟨211⟩: 3 ppm.
**Heavy metals,** *Method II* ⟨231⟩: 0.004%.
**Gum or dextrin—**Dissolve 2 g in 10 mL of hot water: the solution is not more than slightly turbid. Cool, filter, and divide the filtrate into two equal portions. To one portion add 10 mL of alcohol: no turbidity is produced.
**Resinous substances—**To a portion of the filtrate obtained in the test for *Gum or dextrin* add 10 mL of water: no turbidity is produced.

## Adhesive Tape

» Adhesive Tape consists of fabric and/or film evenly coated on one side with a pressure-sensitive, adhesive mixture. Its length is not less than 98.0 percent of that declared on the label, and its average width is not less than 95.0 percent of the declared width. If Adhesive Tape has been rendered sterile, it is protected from contamination by appropriate packaging.

**Packaging and storage—**Preserve in well-closed containers, and prevent exposure to excessive heat and to sunlight. Tape that has been rendered sterile is so packaged that the sterility of the contents of the package is maintained until the package is opened for use.
**Labeling—**The package label of Tape that has been rendered sterile indicates that the contents may not be sterile if the package bears evidence of damage or previously has been opened. The package label indicates the length and width of the Tape, and the name of the manufacturer, packer, or distributor.
**Dimensions—**Measure its length: it is not less than 98.0% of the labeled length. Measure its width at 5 locations evenly spaced along the center line of the Tape: the average of 5 measurements is not less than 95% of the labeled width of the Tape.
**Tensile strength—**Determine the tensile strength of Tape, after previously unrolling and conditioning it for not less than 4 hours in a standard atmosphere of 65 ± 2% relative humidity, at 21 ± 1.1° (70 ± 2°F), with a pendulum-type testing machine, as described under *Tensile Strength* ⟨881⟩. The Tape made from fabric has a tensile strength, determined warpwise, of not less than 20.41 kg (45 pounds) per 2.54 cm of width. The Tape made from film has a tensile strength of not less than 3 kg per 2.54 cm of width.
**Adhesive strength—**Determine the adhesive strength of Tape that is made from fabric by cutting a strip of the Tape 2.54 cm wide and approximately 15 cm long, and applying 12.90 sq cm, 2.54 cm by 5.08 cm, of one end of the strip to a clean plastic or glass surface by means of a rubber roller under a pressure of 850 g, passing the roller twice over the Tape at a rate of 30 cm per minute. Adjust the temperature of the plastic or glass surface and the Tape to 37°, and conduct the test immediately thereafter as directed under *Tensile Strength* ⟨881⟩,