Status: Currently Official on 16-Feb-2025
Official Date: Official Prior to 2013
Document Type: USP Monographs
DocId: GUID-2A56DBA4-4AF5-4F67-9E00-36C47914AA37_1_en-US
DOI: https://doi.org/10.31003/USPNF_M58780_01_01
DOI Ref: 1oyp3

Orbifloxacin Tablets

» Orbifloxacin Tablets contain not less than 90.0 percent and not more than 110.0 percent of the labeled amount of orbifloxacin ($C_{19}H_{20}F_3N_3O_3$).

Packaging and storage—Preserve in tight containers, and store between 2° and 30°.

Labeling—Label to indicate that it is for veterinary use only.

USP Reference standards ⟨11⟩—

USP Orbifloxacin RS

Identification—

A: *Thin-Layer Chromatographic Identification Test* ⟨201⟩.

Absorbent: silica gel.

Diluent: a mixture of chloroform, methanol, and glacial acetic acid (8:1:1).

Test solution—Crush 1 Tablet and transfer into a centrifuge tube. Add *Diluent* quantitatively, and mix to obtain a final concentration of about 0.56 mg per mL of orbifloxacin. Centrifuge the solution.

Standard solution—Prepare a solution of USP Orbifloxacin RS in *Diluent* having a concentration of about 0.56 mg per mL.

Application volume: 5 µL

Developing solvent system: a mixture of chloroform, methanol, water, and ammonium hydroxide (18:7:1:0.02).

B: The retention time of the major peak in the chromatogram of the *Assay preparation* corresponds to that in the chromatogram of the *Standard preparation,* as obtained in the *Assay.*

Dissolution ⟨711⟩—

Medium: 0.1 N hydrochloric acid; 1000 mL.

Apparatus 2: 50 rpm.

Time: 30 minutes.

Determine the amount of $C_{19}H_{20}F_3N_3O_3$ dissolved by employing the following procedure.

Standard solution—Prepare a solution of USP Orbifloxacin RS in *Medium* with a final concentration of about *L*/100, where *L* is the Tablet label claim in mg. Transfer 10.0 mL of this solution to a 100-mL volumetric flask, dilute with *Medium* to volume, and mix.

Test solution—Pass a portion of the solution under test through a suitable 0.8-µm filter, discarding the first 3 mL.

Procedure—Determine the amount of $C_{19}H_{20}F_3N_3O_3$ dissolved by employing UV absorption at the wavelength of maximum absorbance at about 291 nm on portions of the *Test solution* in comparison with the *Standard solution* using *Medium* as blank. Calculate the amount of orbifloxacin dissolved by the formula:

$$100{,}000(A_U/A_S)(C_S/L)$$

in which A_U and A_S are the absorbances obtained with the *Test solution* and the *Standard solution,* respectively; C_S is the concentration, in mg per mL, of orbifloxacin in the *Standard solution;* and *L* is the Tablet label claim in mg.

Tolerances—Not less than 80% (*Q*) of the labeled amount of $C_{19}H_{20}F_3N_3O_3$ is dissolved in 30 minutes.

Uniformity of dosage units ⟨905⟩**:** meet the requirements.

Water Determination, *Method Ic* ⟨921⟩**:** between 3.5% and 7.0%.

Test preparation—Accurately weigh 5 Tablets, and transfer into a 50-mL centrifuge tube. Add 25 mL of anhydrous methanol, and cap.

Blank: 25 mL of anhydrous methanol in a 50-mL centrifuge tube.

Procedure—Rotate the *Test preparation* and the *Blank* for 16 hours. Centrifuge. Titrate an equal volume of the *Test preparation* and the *Blank* so that the amount of water titrated will be approximately 1000 µg to 1500 µg.

Chromatographic purity—

Buffer, Mobile phase, Standard preparation, System suitability preparation, and *Chromatographic system*—Prepare as directed in the *Assay.*

Standard solution—Dilute quantitatively with *Buffer* the *Standard preparation* to obtain a solution having a known concentration of about 0.00004 mg per mL.

Test solution—Transfer 10 Tablets into a volumetric flask. Add *Buffer* to fill the flask about 70%, shake for 2 hours, and sonicate for 5 minutes. Dilute quantitatively, and stepwise if necessary, with *Buffer* to obtain a solution having a concentration of about 0.22 mg per mL. Pass a portion of the solution through a 0.8-µm filter.

Chromatographic system (see *Chromatography ⟨621⟩*)—Inject the *Buffer* as directed for *Procedure* to verify that there are no interfering peaks.

Procedure—Separately inject equal volumes (about 10 µL) of the *Standard solution* and the *Test solution* into the chromatograph, record the chromatograms, and measure the area responses for the major peaks. Calculate the percentage of related compounds in the portion of Tablets taken by the formula:

$$100(C_S/C_T)(r_i/r_S)(1/F)$$

in which C_S is the concentration, in mg per mL, of USP Orbifloxacin RS in the *Standard solution;* C_T is the concentration, in mg per mL, of the *Test solution;* r_i is the peak area response for each impurity obtained from the *Test solution;* r_S is the peak area response for the orbifloxacin peak obtained from the *Standard solution;* and F is the relative response factor for each impurity, as presented in *Table 1*.

Table 1

Component	Relative Retention Time	Relative Response Factor (*F*)	Limit (%)
cis-1-Cyclopropyl-7-(3,5-dimethyl-1-piperazinyl)-5,6,8-trifluoro-4(1*H*)-quinolinone	0.57	0.29	NMT 0.5
Orbifloxacin	1.0	1.00	—
cis-1-Cyclopropyl-7-(3,5-dimethyl-1-piperazinyl)-6,8-difluoro-1,4-dihydro-5-hydroxy-4-oxo-3-quinolinecarboxylic acid	2.9	0.71	NMT 0.5
All other related compounds and impurities	—	0.11	NMT 0.5
Total known and unknown	—	—	NMT 1

Assay—

Buffer—In a 2-L flask, dissolve about 11.8 g of sodium citrate in 1600 mL of water, and mix. Add 180 mL of glacial acetic acid, and mix. Adjust with 6 N sodium hydroxide to a pH of 3.5, dilute with water to volume, and mix.

Mobile phase—Prepare a filtered and degassed mixture of *Buffer,* methanol, and dioxane (91:6:4). Make adjustments if necessary (see *System Suitability* under *Chromatography ⟨621⟩*).

Standard stock preparation—Dissolve an accurately weighed quantity of USP Orbifloxacin RS in *Buffer,* and dilute quantitatively, and stepwise if necessary, with *Buffer* to obtain a solution having a known concentration of about 0.2 mg per mL.

Standard preparation—Accurately transfer a quantity of *Standard stock preparation* and dilute quantitatively, and stepwise if necessary, with *Buffer* to obtain a solution having a known concentration of about 0.02 mg per mL.

System suitability preparation—Dissolve about 40 mg of methyl 4-aminobenzoate in 2 mL of methanol, and dilute with *Buffer* to 200 mL. Pipet 10.0 mL of this solution and 10.0 mL of the *Standard stock preparation* into a 100-mL volumetric flask. Dilute with *Buffer* to volume, and mix.

Assay preparation—Transfer 10 Tablets into a volumetric flask. Add *Buffer* to fill the flask about 70%, shake for 2 hours, and sonicate for 5 minutes. Dilute quantitatively, and stepwise if necessary, with *Buffer* to obtain a solution having a known concentration of about 0.02 mg per mL. Pass a portion of the solution through a 0.8-µm filter.

Chromatographic system (see *Chromatography ⟨621⟩*)—The liquid chromatograph is equipped with a 290-nm detector and 4.6-mm × 3-cm column that contains 3-µm packing L1. The flow rate is about 0.8 mL per minute. Chromatograph the *System suitability preparation,* and record the peak response as directed for *Procedure:* the relative retention times are about 1.3 for methyl 4-aminobenzoate and 1.0 for orbifloxacin; the resolution, *R,* between methyl 4-aminobenzoate and orbifloxacin is not less than 2; the tailing factor is not more than 1.8; and the relative standard deviation for replicate injections is not more than 2.0%.

Procedure—Separately inject equal volumes (about 10 µL) of the *Standard preparation* and the *Assay preparation* into the chromatograph, record the chromatograms, and measure the area responses for the major peaks. Calculate the quantity, in mg, of orbifloxacin ($C_{19}H_{20}F_3N_3O_3$)

in the portion of Tablets taken by the formula:

$$C(D_U)(r_U/r_S)$$

in which C is the concentration, in mg per mL, of USP Orbifloxacin RS in the *Standard preparation;* D_U is the dilution factor of the *Assay preparation,* in mL; and r_U and r_S are the peak area responses obtained from the *Assay preparation* and the *Standard preparation,* respectively.

Auxiliary Information - Please check for your question in the FAQs before contacting USP.

Topic/Question	Contact	Expert Committee
ORBIFLOXACIN TABLETS	Documentary Standards Support	SM32020 Small Molecules 3
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	SM32020 Small Molecules 3

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. PF 34(2)

Current DocID: GUID-2A56DBA4-4AF5-4F67-9E00-36C47914AA37_1_en-US
DOI: https://doi.org/10.31003/USPNF_M58780_01_01
DOI ref: 1oyp3