Interim Revision Announcement
Official: May 1, 2021

Gemfibrozil Tablets

DEFINITION
Gemfibrozil Tablets contain NLT 90.0% and NMT 110.0% of the labeled amount of gemfibrozil ($C_{15}H_{22}O_3$).

IDENTIFICATION
Change to read:

- **A.**

Sample: ▲Nominally▲ (IRA 1-May-2021) 100 mg of gemfibrozil from a quantity of finely ground Tablets

▲**Standard:** 100 mg of USP Gemfibrozil RS▲ (IRA 1-May-2021)

Analysis: Shake the *Sample* with 10 mL of 0.1 N sodium hydroxide. Filter the mixture into a 50-mL centrifuge tube, and acidify the filtrate with 3 N sulfuric acid to obtain a copious precipitate. Centrifuge, and discard the clear solution. Wash the precipitate with small portions of water, and allow it to air-dry. ▲Prepare a potassium bromide dispersion of the precipitate and the *Standard*, each previously dried over silica gel for 4 h.▲ (IRA 1-May-2021)

Acceptance criteria: The IR absorption spectrum of ▲the *Sample*▲ (IRA 1-May-2021) exhibits maxima only at the same wavelengths as ▲the *Standard*.▲ (IRA 1-May-2021)

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

ASSAY
Change to read:

- **Procedure**

Mobile phase: Add 10 mL of acetic acid, glacial to 800 mL of methanol in a 1000-mL volumetric flask, and dilute with water to volume.

System suitability solution: 0.2 mg/mL of USP Gemfibrozil RS and 0.05 mg/mL of ▲2,5-dimethylphenol▲ (IRA 1-May-2021) in *Mobile phase*

Standard stock solution: 1 mg/mL of USP Gemfibrozil RS in methanol

Standard solution: 0.2 mg/mL of USP Gemfibrozil RS from the *Standard stock solution* in *Mobile phase*

Sample stock solution: Nominally 1 mg/mL of gemfibrozil prepared as follows. Transfer the equivalent of 100 mg of gemfibrozil from NLT 20 finely powdered Tablets to a 100-mL volumetric flask. Add about 80 mL of methanol and shake to dissolve. Dilute with methanol to volume and pass through a suitable filter.

Sample solution: Nominally 0.2 mg/mL of gemfibrozil from the *Sample stock solution* in *Mobile phase*

Chromatographic system

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

Mode: LC

Detector: UV 276 nm

Column: 3.9-mm × 30-cm; ▲10-µm▲ (IRA 1-May-2021) packing L1

Flow rate: 0.8 mL/min

Injection volume: 10 µL

System suitability

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

Suitability requirements

Resolution: NLT 8.0 between gemfibrozil and ▲2,5-dimethylphenol,▲ (IRA 1-May-2021) *System suitability solution*

Relative standard deviation: NMT 2.0%, *Standard solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of gemfibrozil ($C_{15}H_{22}O_3$) in the portion of Tablets taken:

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

r_U = peak response ▲of gemfibrozil▲ (IRA 1-May-2021) from the *Sample solution*

r_S = peak response ▲of gemfibrozil▲ (IRA 1-May-2021) from the *Standard solution*

C_S = concentration of USP Gemfibrozil RS in the *Standard solution* (mg/mL)

C_U = nominal concentration of gemfibrozil in the *Sample solution* (mg/mL)

Acceptance criteria: 90.0%–110.0%

PERFORMANCE TESTS

Change to read:

- **Dissolution** ⟨711⟩

Medium: 0.2 M phosphate buffer prepared as follows. Dissolve 545 g of potassium phosphate, monobasic in 5 L of water, add 131 g of sodium hydroxide, dilute with water to about 19.5 L, and mix well. Adjust with either 1 N phosphoric acid or 1 N sodium hydroxide to a pH of 7.5. Dilute with water to 20 L; 900 mL.

Apparatus 2: 50 rpm

Time: 30 min

Standard stock solution: ▲0.33 mg/mL of▲ (IRA 1-May-2021)USP Gemfibrozil RS in *Medium* prepared as follows. Dissolve USP Gemfibrozil RS in an amount of methanol not to exceed 1% of the total volume of the *Standard stock solution*. Dilute with *Medium* to volume.

Standard solution: ▲A known concentration of USP Gemfibrozil RS, similar to that of *Sample solution*, prepared by diluting the *Standard stock solution* with 1 N sodium hydroxide▲ (IRA 1-May-2021)

Sample solution: Pass a portion of the solution under test through a suitable filter. Dilute with 1 N sodium hydroxide to a ▲suitable nominal concentration of gemfibrozil.▲ (IRA 1-May-2021)

Instrumental conditions

Mode: UV

Analytical wavelength: 276 nm

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of gemfibrozil ($C_{15}H_{22}O_3$) dissolved:

$$\text{▲Result} = (A_U/A_S) \times C_S \times D \times V \times (1/L) \times 100$$

A_U = absorbance of the *Sample solution*

A_S = absorbance of the *Standard solution*

C_S = concentration of USP Gemfibrozil RS in the *Standard solution* (mg/mL)

D = dilution factor of the *Sample solution*

V = volume of *Medium*, 900 mL

L = label claim (mg/Tablet) ▲ (IRA 1-May-2021)

Tolerances: NLT 80% (Q) of the labeled amount of gemfibrozil ($C_{15}H_{22}O_3$) is dissolved.

- **Uniformity of Dosage Units** ⟨905⟩**:** Meet the requirements

IMPURITIES

Change to read:

- **Organic Impurities**

Mobile phase, System suitability solution, and **Standard stock solution:** Prepare as directed in the *Assay*.

Standard solution: 0.05 mg/mL of USP Gemfibrozil RS from the *Standard stock solution* in *Mobile phase*

Sensitivity solution: 0.005 mg/mL of USP Gemfibrozil RS from the *Standard solution* in *Mobile phase*

Sample solution: Nominally 10 mg/mL of gemfibrozil prepared as follows. Transfer 500 mg of gemfibrozil from NLT 20 finely powdered Tablets to a 50-mL volumetric flask, and add about 40 mL of *Mobile phase*. Sonicate and shake for 20 min. Dilute with *Mobile phase* to volume and pass through a suitable filter.

Chromatographic system

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

Mode: LC

Detector: UV 276 nm

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

Flow rate: 1 mL/min

Injection volume: 10 µL

Run time: NLT 3 times the retention time of gemfibrozil

System suitability

Samples: *System suitability solution* and *Sensitivity solution*

Suitability requirements

Resolution: NLT 8.0 between gemfibrozil and ▲2,5-dimethylphenol,▲ (IRA 1-May-2021) *System suitability solution*

Relative standard deviation: NMT 2.0% for the gemfibrozil peak, *System suitability solution*

Signal-to-noise ratio: NLT 10, *Sensitivity solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of each impurity in the portion of Tablets taken:

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

r_U = peak response of each impurity from the *Sample solution*

r_S = peak response of gemfibrozil from the *Standard solution*

C_S = concentration of USP Gemfibrozil RS in the *Standard solution* (mg/mL)

C_U = nominal concentration of gemfibrozil in the *Sample solution* (mg/mL)

Acceptance criteria: ▲The reporting threshold is 0.05%.▲ (IRA 1-May-2021)

Individual impurities: NMT 0.17%

Total impurities: NMT 1.0%

ADDITIONAL REQUIREMENTS

Change to read:

- **Packaging and Storage:** Preserve in tight containers. ▲Store at controlled room temperature. Protect from light and humidity.▲ (IRA 1-May-2021)
- **USP Reference Standards** ⟨11⟩
  USP Gemfibrozil RS

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Not Applicable

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