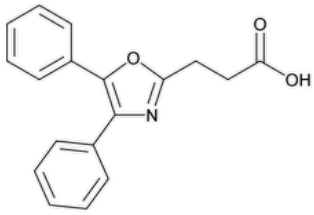
Status: Currently Official on 16-Feb-2025
Official Date: Official as of 01-May-2022
Document Type: USP Monographs
DocId: GUID-7A06F042-60E1-49C5-B738-83F3D6C85130_6_en-US
DOI: https://doi.org/10.31003/USPNF_M59160_06_01
DOI Ref: 1o1z6

Oxaprozin

Change to read:



$C_{18}H_{15}NO_3$ 293.32

2-Oxazolepropanoic acid, 4,5-diphenyl-;

4,5-Diphenyl-2-oxazolepropionic acid CAS RN®: 21256-18-8; ▲UNII: MHJ80W9LRB.▲ (USP 1-May-2022)

DEFINITION

Oxaprozin contains NLT 98.0% and NMT 102.0% of oxaprozin ($C_{18}H_{15}NO_3$), calculated on the dried basis.

[NOTE—Because of light sensitivity, protect all oxaprozin samples and *Standard solutions* from light.]

IDENTIFICATION

- **A. SPECTROSCOPIC IDENTIFICATION TESTS ⟨197⟩, *Infrared Spectroscopy*:** 197K or 197A
- **B.** The retention time of the oxaprozin peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Assay*.

ASSAY

- **PROCEDURE**

Solution A: 0.1% phosphoric acid, pH 2.0 ± 0.1

Mobile phase: Acetonitrile and *Solution A* (45:55)

Standard solution: 0.2 mg/mL of USP Oxaprozin RS in acetonitrile

Sample solution: 0.2 mg/mL of Oxaprozin in acetonitrile

Chromatographic system

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

Mode: LC

Detector: UV 285 nm

Column: 4.6-mm × 15-cm; 5-µm packing L7

Column temperature: 25°

Flow rate: 1.0 mL/min

Injection volume: 20 µL

System suitability

Sample: *Standard solution*

Suitability requirements

Tailing factor: NMT 2.0

Relative standard deviation: NMT 0.73%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of oxaprozin ($C_{18}H_{15}NO_3$) in the portion of Oxaprozin taken:

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

r_U = peak response of oxaprozin from the *Sample solution*

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

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

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

Acceptance criteria: 98.0%–102.0% on the dried basis

IMPURITIES

• **Residue on Ignition ⟨281⟩:** NMT 0.3%

Delete the following:

▲• **Arsenic ⟨211⟩, *Procedures, Method II*:** NMT 1 ppm▲ (USP 1-May-2022)

• **Organic Impurities**

Solution A: 0.1% phosphoric acid, pH 2.0 ± 0.1

Solution B: Acetonitrile

Mobile phase: See *Table 1*.

Table 1

Time (min)	Solution A (%)	Solution B (%)
0	70	30
20	70	30
21	70	30
60	0	100
61	70	30
70	70	30

Diluent A: Acetonitrile, methylene chloride, and water (48:1:1)

Diluent B: Acetonitrile and water (1:1)

Standard stock solution: 200 µg/mL of USP Oxaprozin RS in acetonitrile

Standard solution: 5 µg/mL of USP Oxaprozin RS in *Diluent A* from the *Standard stock solution*

Sensitivity solution: 0.3 µg/mL of USP Oxaprozin RS in *Diluent A* from the *Standard solution*

System suitability stock solution: 200 µg/mL of benzil in acetonitrile

System suitability solution: 10 µg/mL each of benzil and USP Oxaprozin RS in *Diluent A* from the *System suitability stock solution* and *Standard stock solution*, respectively

Sample solution A: 1 mg/mL of Oxaprozin prepared as follows. Transfer about 100 mg of Oxaprozin to a 100-mL volumetric flask. Add 2 mL of methylene chloride, 2 mL of water, and 75 mL of acetonitrile. Sonicate after each solvent is added. Dilute with acetonitrile to volume. [Note—This is used to monitor all known and unknown impurities, except oxaprozin imidazole analog and oxaprozin amide.]

Sample solution B: 1 mg/mL of Oxaprozin in *Diluent B*. [Note—This is used to monitor only oxaprozin imidazole analog and oxaprozin amide.]

Chromatographic system

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

Mode: LC

Detector: UV 238 nm

Column: 4.6-mm × 15-cm; 5-µm packing L7

Flow rate: 1.0 mL/min

Injection volume: 20 µL

System suitability

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

Suitability requirements

Resolution: NLT 3.0 between oxaprozin and benzil, *System suitability solution*

Tailing factor: NMT 2.0, *Standard solution*

Relative standard deviation: NMT 5.0%, *Standard solution*

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

Analysis

Samples: *Sample solution A* and *Sample solution B*

Calculate the percentage of oxaprozin imidazole analog and oxaprozin amide in the portion of Oxaprozin taken:

$$\text{Result} = (r_U/r_T) \times (1/F) \times 100$$

r_U = peak area of oxaprozin imidazole analog or oxaprozin amide from *Sample solution B*

r_T = sum of the peak areas from *Sample solution B*

F = relative response factor (see *Table 2*)

Calculate the percentage of any other impurity in the portion of Oxaprozin taken:

$$\text{Result} = (r_U/r_T) \times (1/F) \times 100$$

r_U = peak area of each individual impurity from *Sample solution A*

r_T = sum of the peak areas from *Sample solution A*

F = relative response factor (see *Table 2*)

Acceptance criteria: See *Table 2*. The reporting threshold is 0.03%.

Table 2

Name	Relative Retention Time	Relative Response Factor	Acceptance Criteria, NMT (%)
Oxaprozin imidazole analog[a]	0.14	0.87	0.1
Benzoin[b]	0.42	0.83	0.1
Oxaprozin amide[c]	0.73	1.10	0.1
Benzoin succinate[d]	0.84	1.18	0.1
Oxaprozin	1.00	1.00	—
Benzil	1.08	0.78	0.1
Oxaprozin bisoxazole analog[e]	1.50	0.68	0.1
Tetraphenylpyrazine[f]	1.57	0.48	0.1
Individual unspecified impurity	—	1.00	0.06
Total impurities	—	—	0.5

[a] 3-(4,5-Diphenyl-1*H*-imidazol-2-yl)propanoic acid.

[b] 2-Hydroxy-1,2-diphenylethan-1-one.

[c] 3-(4,5-Diphenyloxazol-2-yl)propanamide.

[d] 4-Oxo-4-(2-oxo-1,2-diphenylethoxy)butanoic acid.

[e] 1,2-Bis(4,5-diphenyloxazol-2-yl)ethane.

[f] 2,3,5,6-Tetraphenylpyrazine.

SPECIFIC TESTS

• **Loss on Drying** ⟨731⟩

Analysis: Dry at 105° for 2 h.

Acceptance criteria: NMT 0.3%

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in tight, light-resistant containers, and store at controlled room temperature.

• **USP Reference Standards** ⟨11⟩

USP Oxaprozin RS

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

Topic/Question	Contact	Expert Committee
OXAPROZIN	Documentary Standards Support	SM22020 Small Molecules 2
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	SM22020 Small Molecules 2

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. 46(5)

Current DocID: GUID-7A06F042-60E1-49C5-B738-83F3D6C85130_6_en-US

DOI: https://doi.org/10.31003/USPNF_M59160_06_01

DOI ref: 1o1z6