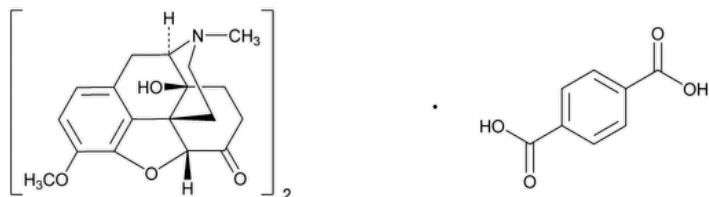


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Oxycodone Terephthalate



$(C_{18}H_{21}NO_4)_2 \cdot C_8H_6O_4$ 796.86

Morphinan-6-one, 4,5-epoxy-14-hydroxy-3-methoxy-17-methyl-, 1,4-benzenedicarboxylate (2:1 salt), (5 α);

4,5 α -Epoxy-14-hydroxy-3-methoxy-17-methylmorphinan-6-one 1,4-benzenedicarboxylate (2:1 salt) CAS RN[®]: 64336-55-6.

DEFINITION

Oxycodone Terephthalate contains NLT 97.0% and NMT 103.0% of oxycodone terephthalate $(C_{18}H_{21}NO_4)_2 \cdot C_8H_6O_4$, calculated on the dried basis.

IDENTIFICATION

• A. [MELTING RANGE OR TEMPERATURE \(741\)](#)

Sample solution: Transfer 50 mL of the filtrate retained from the test for *Content of Terephthalate Acid* to a 125-mL conical flask. Render the solution alkaline with 6 N ammonium hydroxide. Allow the mixture to stand until a precipitate is formed. Filter, wash the precipitate with 50 mL of cold water, and dry for 2 h at 105°.

Acceptance criteria: The precipitate melts between 218° and 223°, but the range between the beginning and end of the melting does not exceed 2°.

Change to read:

• B. [▲ SPECTROSCOPIC IDENTIFICATION TESTS \(197\)](#), [Infrared Spectroscopy: 197K](#) ▲ (CN 1-MAY-2020)

Sample: Use a portion of the dried precipitate obtained in *Identification* test A.

Acceptance criteria: Meets the requirements

Change to read:

• C. [▲ SPECTROSCOPIC IDENTIFICATION TESTS \(197\)](#), [Ultraviolet-Visible Spectroscopy: 197U](#) ▲ (CN 1-MAY-2020)

Sample solution: 150 μ g/mL in 0.1 N hydrochloric acid

Acceptance criteria: Exhibits a maxima at 280 nm

ASSAY

• PROCEDURE

Mobile phase: To 2.2 g of sodium 1-octanesulfonate in 740 mL of water add 260 mL of methanol, 10 mL of glacial acetic acid, and 0.1 mL of triethylamine. Mix, and adjust with 5 N sodium hydroxide to a pH of 6.5 ± 0.1 . Pass through a filter of 0.5- μ m or finer pore size.

Diluent: 0.1 N hydrochloric acid

Internal standard solution: 0.1 mg/mL of ethylparaben prepared by dissolving in 2% of the flask volume of methanol and diluting with *Diluent* to volume

Standard stock solution: 0.75 mg/mL of [USP Oxycodone RS](#) in *Diluent*

Standard solution: 0.11 mg/mL of [USP Oxycodone RS](#) prepared as follows. Transfer 15.0 mL of *Standard stock solution* to a 100-mL volumetric flask, add 20.0 mL of *Internal standard solution*, and dilute with *Diluent* to volume.

Sample stock solution: 0.71 mg/mL of Oxycodone Terephthalate in *Diluent*. Filter, discarding the first 5 mL.

Sample solution: 0.14 mg/mL of Oxycodone Terephthalate prepared as follows. Transfer 10.0 mL of the *Sample stock solution* to a 50-mL volumetric flask, add 10.0 mL of *Internal standard solution*, and dilute with *Diluent* to volume.

Chromatographic system

(See [Chromatography \(621\)](#), [System Suitability](#).)

Mode: LC

Detector: UV 280 nm

Column: 3.9-mm × 15-cm; packing L1

Column temperature: 50 ± 1.0°

Flow rate: 1 mL/min

Run time: Twice the retention time of the main oxycodone peak

Injection size: 30 µL

System suitability

Sample: *Standard solution*

Suitability requirements

Resolution: NLT 6 between oxycodone and ethylparaben

Column efficiency: NLT 1800 theoretical plates

Relative standard deviation: NMT 2.0%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of oxycodone terephthalate ($C_{18}H_{21}NO_4)_2 \cdot C_8H_6O_4$ in the portion of Oxycodone Terephthalate taken:

$$\text{Result} = (R_U/R_S) \times (C_S/C_U) \times (M_{r1}/M_{r2}) \times 100$$

R_U = peak response ratio of oxycodone to ethylparaben from the *Sample solution*

R_S = peak response ratio of oxycodone to ethylparaben from the *Standard solution*

C_S = concentration of [USP Oxycodone RS](#) in the *Standard solution* (mg/mL)

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

M_{r1} = one-half of the molecular weight of oxycodone terephthalate, 398.43

M_{r2} = molecular weight of oxycodone, 315.37

Acceptance criteria: 97.0%–103.0% on the dried basis

IMPURITIES

• [RESIDUE ON IGNITION \(281\)](#): NMT 1%

• ORGANIC IMPURITIES

Solution A: 2.2 g of sodium 1-octanesulfonate in 850 mL of water. Add 150 mL of methanol, 20 mL of glacial acetic acid, and 1.0 mL of triethylamine. Pass through a filter of 0.5-µm or finer pore size.

Solution B: 2.2 g of sodium 1-octanesulfonate in 500 mL of water. Add 500 mL of methanol, 20 mL of glacial acetic acid, and 1.0 mL of triethylamine. Pass through a filter of 0.5-µm or finer pore size.

Mobile phase: See [Table 1](#).

Table 1

Time (min)	Solution A (%)	Solution B (%)
0	90	10
30	80	20
50	0	100
55	0	100

Diluent: 0.1 N hydrochloric acid

Standard stock solution: 0.9 mg/mL of [USP Oxycodone RS](#) in *Diluent*