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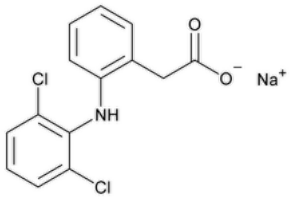
Diclofenac Sodium



$C_{14}H_{10}Cl_2NNaO_2$ 318.13

Benzeneacetic acid, 2-[(2,6-dichlorophenyl)amino]-, monosodium salt;

Sodium [*o*-(2,6-dichloroanilino)phenyl]acetate CAS RN®: 15307-79-6; UNII: QTG126297Q.

DEFINITION

Diclofenac Sodium contains NLT 99.0% and NMT 101.0% of diclofenac sodium ($C_{14}H_{10}Cl_2NNaO_2$), calculated on the dried basis.

IDENTIFICATION

Change to read:

• **A.** ▲**Spectroscopic Identification Tests ⟨197⟩, *Infrared Spectroscopy*: 197K**▲ (CN 1-May-2020)

• **B.** The retention time of the diclofenac peak of the *Sample solution* corresponds to that of the *System suitability solution*, as obtained in the test for *Organic Impurities*.

• **C.** The residue obtained by igniting it imparts an intense yellow color to a nonluminous flame.

ASSAY

• **Procedure**

Sample solution: Dissolve about 450 mg of Diclofenac Sodium in 25 mL of glacial acetic acid.

Titrimetric system

Titrant: 0.1 N perchloric acid VS

Analysis: Titrate with *Titrant*, determining the endpoint potentiometrically. Perform a blank determination, and make any necessary correction. Each mL of 0.1 N perchloric acid is equivalent to 31.81 mg of diclofenac sodium ($C_{14}H_{10}Cl_2NNaO_2$).

Acceptance criteria: 99.0%–101.0% on the dried basis

IMPURITIES

• **Organic Impurities**

Solution A: 0.01 M phosphoric acid and 0.01 M monobasic sodium phosphate (1:1). If necessary, adjust with additional portions of the appropriate components to a pH of 2.5 ± 0.2.

Mobile phase: Methanol and *Solution A* (70:30)

Diluent: Methanol and water (70:30)

System suitability solution: 20 µg/mL of diethyl phthalate, 7.5 µg/mL of USP Diclofenac Related Compound A RS, and 0.75 mg/mL of USP Diclofenac Sodium RS in *Diluent*

Standard stock solution: 0.75 mg/mL of USP Diclofenac Related Compound A RS in methanol

Standard solution: 1.5 µg/mL of USP Diclofenac Related Compound A RS in *Diluent* from *Standard stock solution*

Sample solution: 0.75 mg/mL of Diclofenac Sodium in *Diluent*

Chromatographic system

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

Mode: LC

Detector: UV 254 nm

Column: 4.6-mm × 25-cm; packing L7 (end-capped)

Flow rate: 1 mL/min

Injection volume: 10 µL

Run time: 2.5 times the retention time of diclofenac

System suitability

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

[NOTE—The relative retention times for diethyl phthalate, diclofenac related compound A, and diclofenac are 0.5, 0.6, and 1.0, respectively.]

Suitability requirements

Resolution: NLT 2.2 between diethyl phthalate and diclofenac related compound A; NLT 6.5 between diclofenac related compound A and diclofenac, *System suitability solution*

Relative standard deviation: NMT 5%, *Standard solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of diclofenac related compound A in the portion of Diclofenac Sodium taken:

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

r_U = peak response of diclofenac related compound A from the *Sample solution*

r_S = peak response of diclofenac related compound A from the *Standard solution*

C_S = concentration of USP Diclofenac Related Compound A RS in the *Standard solution* (mg/mL)

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

Calculate the percentage of each other impurity in the portion of Diclofenac Sodium taken:

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

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

r_S = peak response of diclofenac related compound A from the *Standard solution*

C_S = concentration of USP Diclofenac Related Compound A RS in the *Standard solution* (mg/mL)

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

Acceptance criteria

Diclofenac related compound A: NMT 0.2%

Each other individual impurity: NMT 0.2%

Total impurities: NMT 0.5%

SPECIFIC TESTS

• **COLOR OF SOLUTION:** A solution (1 in 20) of Diclofenac Sodium in methanol is colorless to faintly yellow, and the absorbance of the solution, determined in a 1-cm cell at 440 nm, is NMT 0.050, methanol being used as the blank.

• **CLARITY OF SOLUTION:** The solution prepared as directed under *Color of Solution* is not significantly less clear than an equal volume of methanol contained in a similar vessel and examined similarly.

• **pH** ⟨791⟩

Sample solution: A solution (1 in 100)

Acceptance criteria: 7.0–8.5

• **LOSS ON DRYING** ⟨731⟩

Analysis: Dry at 105°–110° for 3 h.

Acceptance criteria: NMT 0.5%

ADDITIONAL REQUIREMENTS

• **PACKAGING AND STORAGE:** Preserve in tight, light-resistant containers.

• **USP REFERENCE STANDARDS** ⟨11⟩

USP Diclofenac Sodium RS

USP Diclofenac Related Compound A RS

N-(2,6-Dichlorophenyl)indolin-2-one.

$C_{14}H_9Cl_2NO$ 278.14

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

Topic/Question	Contact	Expert Committee
DICLOFENAC SODIUM	Documentary Standards Support	SM22020 Small Molecules 2

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
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