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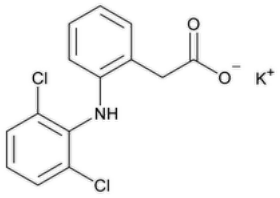
Diclofenac Potassium



$C_{14}H_{10}Cl_2KNO_2$ 334.24

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

Potassium [*o*-(2,6-dichloroanilino)phenyl]acetate CAS RN®: 15307-81-0; UNII: L4D5UA6CB4.

DEFINITION

Diclofenac Potassium contains NLT 99.0% and NMT 101.0% of diclofenac potassium ($C_{14}H_{10}Cl_2KNO_2$), calculated on the dried basis.

IDENTIFICATION

- **A. Spectroscopic Identification Tests ⟨197⟩, *Infrared Spectroscopy*: 197K**
- **B. Spectroscopic Identification Tests ⟨197⟩, *Ultraviolet-Visible Spectroscopy*: 197U**

Solution: 0.01 mg/mL in *Medium*

Medium: Methanol

Acceptance criteria: Meets the requirements

Change to read:

- **C.**

Sample solution: Suspend 0.5 g of Diclofenac Potassium in 10 mL of water. Stir and add water until the substance is dissolved. Add 2 mL of 7 N ▲hydrochloric▲ (ERR 1-Sep-2021) acid, stir for 1 h, and filter with the aid of a vacuum. Neutralize with 5 N sodium hydroxide.

Analysis: To 1 mL of *Sample solution*, add 1 mL of 2 N acetic acid and 1 mL of a freshly prepared 100-g/L solution of sodium cobaltinitrite.

Acceptance criteria: A yellow or orange yellow precipitate is formed immediately.

ASSAY

- **Procedure**

Sample solution: Dissolve about 300 mg of Diclofenac Potassium in 50 mL of glacial acetic acid.

Titrimetric system

(See *Titrimetry ⟨541⟩*.)

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 33.424 mg of diclofenac potassium ($C_{14}H_{10}Cl_2KNO_2$).

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: 40 µg/mL of diethyl phthalate, 0.5 mg/mL of USP Diclofenac Potassium RS, and 22.5 µg/mL of USP Diclofenac Related Compound A RS in *Diluent*

Standard stock solution: 0.25 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: 0.5 mg/mL of Diclofenac Potassium in *Diluent*

Chromatographic system

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

Mode: LC

Detector: UV 254 nm

Column: 4.6-mm × 25-cm; packing L7

Flow rate: 1 mL/min

Injection volume: 30 µL

System suitability

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

[NOTE—The relative retention times for diethyl phthalate, diclofenac related compound A, and diclofenac are about 0.5, 0.7, and 1.0, respectively.]

Suitability requirements

Resolution: NLT 4.0 between diethyl phthalate and diclofenac related compound A, *System suitability solution*

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

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of diclofenac related compound A in the portion of Diclofenac Potassium 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* (µg/mL)

C_U = concentration of Diclofenac Potassium in the *Sample solution* (µg/mL)

Calculate the percentage of each other impurity in the portion of Diclofenac Potassium 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* (µg/mL)

C_U = concentration of Diclofenac Potassium in the *Sample solution* (µg/mL)

Acceptance criteria

Diclofenac related compound A: NMT 0.1%

Each other individual impurity: NMT 0.1%

Total impurities: NMT 0.3%

SPECIFIC TESTS

- **pH** ⟨791⟩

Sample solution: 1% aqueous solution

Acceptance criteria: 7.0–8.5

- **Loss on Drying** ⟨731⟩

Analysis: Dry at 105° under vacuum for 3 h.

Acceptance criteria: NMT 0.5%

ADDITIONAL REQUIREMENTS

- **Packaging and Storage:** Preserve in light-resistant containers, and store at controlled room temperature.
- **USP Reference Standards** ⟨11⟩

USP Diclofenac Potassium 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 POTASSIUM	Documentary Standards Support	SM22020 Small Molecules 2

Chromatographic Database Information: Chromatographic Database

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