Diclofenac sodium

**DEFINITION**
Sodium 2\{(2,6-dichlorophenyl)amino\}phenyl\{acetate.

**Content:** 99.0 per cent to 101.0 per cent (dried substance).

**CHARACTERS**

- Appearance: white or slightly yellowish, slightly hygroscopic, crystalline powder.
- Solubility: sparingly soluble in water, freely soluble in methanol, soluble in ethanol (96 per cent), slightly soluble in acetone.

**mp:** about 280 °C, with decomposition.

**IDENTIFICATION**

- First identification: A, D.
- Second identification: B, C, D.

**ASSAY**

- **Titration:** Titrate with 0.1 M perchloric acid, determining the end-point potentiometrically (2.2.20).
- **Dissolve 0.250 g in 30 mL of anhydrous acetic acid R.**

**STORAGE**

- In an airtight container, protected from light.

**IMPURITIES**

- Specified impurities: A, B, C, D, E.

**System suitability**

- **reference solution (a):**
- **resolution:** minimum 6.5 between the peaks due to impurity A and diclofenac.

**Limits**

- **impurities A, B, C, D, E:** for each impurity, not more than the area of the principal peak in the chromatogram obtained with reference solution (a) (0.2 per cent);
- **total:** not more than 2.5 times the area of the principal peak in the chromatogram obtained with reference solution (a) (0.5 per cent);
- **disregard limit:** 0.25 times the area of the principal peak in the chromatogram obtained with reference solution (a) (0.05 per cent).

**Heavy metals (2.4.8):** maximum 10 ppm.

- 2.0 g complies with test C. Use a quartz crucible. Prepare the reference solution using 2 mL of lead standard solution (10 ppm Pb) R.

**Loss on drying (2.2.32):** maximum 0.5 per cent, determined on 1.000 g by drying in an oven at 105 °C for 3 h.

**Mobile phase**

- mix 34 volumes of a solution containing 0.5 g/L of phosphoric acid R and 0.8 g/L of sodium dihydrogen phosphate R, adjusted to pH 2.5 with phosphoric acid R, and 66 volumes of methanol R.

**Flow rate:** 1 mL/min.

**Detection:** spectrophotometer at 254 nm.

**Injection:** 20 μL.

- **Run time:** 1.5 times the retention time of diclofenac.

**Retention time**

- impurity A and diclofenac:
  - minimum 6.5 between the peaks due to
  - impurity A and diclofenac.

**System suitability**

- **reference solution (b):**
- **resolution:** minimum 6.5 between the peaks due to
  - impurity A and diclofenac.

**Thin-layer chromatography (2.2.27).**

- **Test solution.** Dissolve 25 mg of the substance to be examined in methanol R and dilute to 5 mL with the same solvent.
- **Reference solution (a).** Dissolve 25 mg of diclofenac sodium CRS in methanol R and dilute to 5 mL with the same solvent.
- **Reference solution (b).** Dissolve 10 mg of indometacin R in reference solution (a) and dilute to 2 mL with the same solvent.

**Plate:** TLC silica gel GF254 plate R.

**Mobile phase:** concentrated ammonia R, methanol R, ethyl acetate R (10:10:80 V/V/V).

**Application:** 5 μL.

**Development:** over a path of 10 cm.

**Drying:** in air.

**Detection:** examine in ultraviolet light at 254 nm.

**System suitability**

- **reference solution (b):**
- the chromatogram shows 2 clearly separated spots.

**Results:** the principal spot in the chromatogram obtained with the test solution is similar in position and size to the principal spot in the chromatogram obtained with reference solution (a).

**C. Dissolve about 10 mg in 10 mL of ethanol (96 per cent) R.** To 1 mL of this solution add 0.2 mL of a mixture, prepared immediately before use, of equal volumes of a 6 g/L solution of potassium ferricyanide R and a 9 g/L solution of ferric chloride R. Allow to stand protected from light for 5 min.

**Heavy metals**

2.0 g complies with test C. Use a quartz crucible. Prepare the reference solution using 2 mL of a 6 g/L solution of ferric chloride R and a 9 g/L solution of potassium ferricyanide R, adjusted to pH 2.5 with phosphoric acid R, and 66 volumes of ethanol R.
Dicloxacillin sodium

DEFINITION
Sodium (2S,5R,6R,6S)-6-[[3-(2,6-dichlorophenyl)-5-methylisoxazol-4-yl][carbonyl]amino]-3,3-dimethyl-7-oxo-4-thia-1-azabicyclo[3.2.0]heptan-2-carboxylate monohydrate. Semi-synthetic product derived from a fermentation product. Content: 95.0 per cent to 102.0 per cent (anhydrous substance).

CHARACTERS
Appearance: white or almost white, hygroscopic, crystalline powder.

Solubility: freely soluble in water, soluble in ethanol (96 per cent) and in methanol.

IDENTIFICATION
First identification: A, D.
Second identification: B, C, D.
A. Infrared absorption spectrophotometry (2.2.24).
Preparation: discs.
Comparison: dicloxacillin sodium CRS.
B. Thin-layer chromatography (2.2.27).
Test solution. Dissolve 25 mg of the substance to be examined in 5 mL of water R.

ASSAY
Dissolve 0.250 g in 30 mL of anhydrous acetic acid R. Titrate with 0.1 M perchloric acid, determining the end-point potentiometrically (2.2.20).
1 mL of 0.1 M perchloric acid is equivalent to 31.81 mg of C19H16Cl2N3NaO5S.H2O.

STORAGE
In an airtight container, protected from light.

Dicloxacillinum natricum

C19H16Cl2N3NaO5S,H2O
M, 510.3
[13412-64-1]

01/2008:0663 corrected 6.0