

## 1 Diclofenac Sodium Suppositories

2 ジクロフェナクナトリウム坐剤

3

4 Diclofenac Sodium Suppositories contain not less  
5 than 93.0% and not more than 107.0% of the labeled  
6 amount of diclofenac sodium ( $C_{14}H_{10}Cl_2NNaO_2$ :  
7 318.13).

8 **Method of preparation** Prepare as directed under Sup-  
9 positories, with Diclofenac Sodium.

10 **Identification** To an amount of Diclofenac Sodium  
11 Suppositories, equivalent to 25 mg of Diclofenac Sodium,  
12 add 200 mL of a mixture of methanol and 0.01 mol/L  
13 sodium hydroxide TS (99:1), and dissolve by warming.  
14 Cool while shaking, add a mixture of methanol and 0.01  
15 mol/L sodium hydroxide TS (99:1) to make 250 mL, and  
16 filter through a pledget of absorbent cotton if necessary.  
17 To 10 mL of this solution add a mixture of methanol and  
18 0.01 mol/L sodium hydroxide TS (99:1) to make 100 mL.  
19 Determine the absorption spectrum of this solution as  
20 directed under Ultraviolet-visible Spectrophotometry  
21 <2.24>: it exhibits a maximum between 280 nm and 284  
22 nm.

23 **Uniformity of dosage unit** <6.02> Perform the test ac-  
24 cording to the following method: it meets the requirement  
25 of the Content uniformity test.

26 To 1 suppository of Diclofenac Sodium Suppositories  
27 add 5 mL of tetrahydrofuran, and sonicate to dissolve.  
28 Add a mixture of methanol and water (3:2) to make ex-  
29 actly 100 mL, shake, and filter through a membrane filter  
30 with a pore size not exceeding 0.5  $\mu\text{m}$ . Discard the first 5  
31 mL of the filtrate, pipet  $V$  mL of the subsequent filtrate,  
32 add a mixture of methanol and water (3:2) to make exact-  
33 ly  $V'$  mL so that each mL contains about 125  $\mu\text{g}$  of diclo-  
34 fenac sodium ( $C_{14}H_{10}Cl_2NNaO_2$ ), and use this solution as  
35 the sample solution. Then, proceed as directed in the As-  
36 say.

37 Amount (mg) of diclofenac sodium ( $C_{14}H_{10}Cl_2NNaO_2$ )  
38  $= M_S \times A_T / A_S \times V' / V \times 1 / 4$

39  $M_S$ : Amount (mg) of diclofenac sodium for assay taken

40 **Melting behavior of suppositories** <2.60> 33 – 36°C

41 **Assay** Weigh accurately the mass of not less than 20  
42 Diclofenac Sodium Suppositories, cut into small pieces  
43 carefully, and mix uniformly. Weigh accurately a portion  
44 of the pieces, equivalent to about 25 mg of diclofenac  
45 sodium ( $C_{14}H_{10}Cl_2NNaO_2$ ), add 5 mL of tetrahydrofuran,  
46 and sonicate to dissolve. Add a mixture of methanol and  
47 water (3:2) to make exactly 100 mL, shake, and filter with

48 a pore size not exceeding 0.5  $\mu\text{m}$ . Discard the first 5 mL  
49 of the filtrate, pipet 10 mL of the subsequent filtrate, add a  
50 mixture of methanol and water (3:2) to make exactly 20  
51 mL, and use this solution as the sample solution. Sepa-  
52 rately, weigh accurately about 50 mg of diclofenac sodi-  
53 um for assay, previously dried at 105°C for 3 hours, and  
54 dissolve in a mixture of methanol and water (3:2) to make  
55 exactly 100 mL. Pipet 5 mL of this solution, add a mixture  
56 of methanol and water (3:2) to make exactly 20 mL, and  
57 use this solution as the standard solution. Perform the test  
58 with exactly 20  $\mu\text{L}$  each of the sample solution and stand-  
59 ard solution as directed under Liquid Chromatography  
60 <2.01> according to the following conditions, and deter-  
61 mine the peak areas,  $A_T$  and  $A_S$ , of diclofenac in each solu-  
62 tion.

63 Amount (mg) of diclofenac sodium ( $C_{14}H_{10}Cl_2NNaO_2$ )  
64  $= M_S \times A_T / A_S \times 1 / 2$

65  $M_S$ : Amount (mg) of diclofenac sodium for assay taken

66 **Operating conditions** –

67 Detector: An ultraviolet absorption photometer  
68 (wavelength: 254 nm).

69 Column: A stainless steel column 4.0 mm in inside di-  
70 ameter and 12.5 cm in length, packed with octadecylsi-  
71 lanized silica gel for liquid chromatography (5  $\mu\text{m}$  in par-  
72 ticle diameter).

73 Column temperature: A constant temperature of about  
74 25°C.

75 Mobile phase: To 13.6 g of sodium acetate trihydrate  
76 add water to make 1000 mL. To 200 mL of this solution  
77 add 300 mL of methanol.

78 Flow rate: Adjust so that the retention time of  
79 diclofenac is about 3.5 minutes.

80 **System suitability** –

81 System performance: When the procedure is run with  
82 20  $\mu\text{L}$  of the standard solution under the above operating  
83 conditions, the number of theoretical plates and the  
84 symmetry factor of the peak of diclofenac are not less  
85 than 2000, and 0.7 to 1.5, respectively.

86 System repeatability: When the test is repeated 6 times  
87 with 20  $\mu\text{L}$  of the standard solution under the above  
88 operating conditions, the relative standard deviation of the  
89 peak area of diclofenac is not more than 1.0%.

90 **Containers and storage** Containers – Tight containers.

91 Storage – In a cold place.

92 **Add the following to 9.41 Reagents, Test**  
93 **Solutions:**

94 **Diclofenac sodium for assay**  $C_{14}H_{10}Cl_2NNaO_2$   
95 [Same as the monograph Diclofenac Sodium. When dried,

96 it contains not less than 99.0% of Diclofenac sodium  
97 (C<sub>14</sub>H<sub>10</sub>Cl<sub>2</sub>NNaO<sub>2</sub>.)  
98