1 Celecoxib Tablets

2 セレコキシブ錠

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4 Celecoxib Tablets contain not less than 95.0% and 5 not more than 105.0% of the labeled amount of 6 celecoxib ($C_{17}H_{14}F_{3}N_{3}O_{2}S$: 381.37).

7 Method of preparation Prepare as directed under Tablets,8 with Celecoxib.

9 Identification To a quantity of powdered Celecoxib Tab10 lets, equivalent to 50 mg of Celecoxib, add 70 mL of metha11 nol, shake, add methanol to make 100 mL, and centrifuge. To

12 1 mL of the supernatant liquid add methanol to make 50 mL,

13 and determine the absorption spectrum of this solution as di-

14 rected under Ultraviolet-visible Spectrophotometry <2.24>: it

15 exhibits a maximum between 250 nm and 254 nm.

16 Uniformity of dosage units <6.02> Perform the Mass var-

17 iation test, or the Content uniformity test according to the fol-18 lowing method: it meets the requirement.

To 1 tablet of Celecoxib Tablets, add 7*V*/10 mL of a mixture of acetonitrile and water (3:1), shake thoroughly until the tablet is disintegrated, and add a mixture of acetonitrile and water (3:1) to make exactly *V* mL so that each mL contains about 0.4 mg of celecoxib ($C_{17}H_{14}F_3N_3O_2S$). Centrifuge this solution, pipet 5 mL of the supernatant liquid, add exactly 5 mL of the internal standard solution, add a mixture of ace-

tonitrile and water (3:1) to make 50 mL, and use this solutionas the sample solution. Then, proceed as directed in the Assay.

 $=M_{\rm S} \times Q_{\rm T}/Q_{\rm S} \times V/50$

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30 $M_{\rm S}$: Amount (mg) of Celecoxib RS taken

31 *Internal standard solution*—A solution of benzyl parahy32 droxybenzoate in a mixture of acetonitrile and water (3:1) (1
33 in 2000).

Dissolution *<6.10>* When the test is performed at 50 revolutions per minute according to the Paddle method, using 900 mL of a solution, prepared by dissolving 5 g of polysorbate 80 in 2nd fluid for dissolution test to make 1000 mL, as the dissolution medium, the dissolution rates in 45 minutes of a 100-mg tablet and a 200-mg tablet are not less than 80% and not less than 75%, respectively.

Start the test with 1 tablet of Celecoxib Tablets, withdraw not less than 20 mL of the medium at the specified minute after starting the test, and filter through a membrane filter with a pore size not exceeding $0.45 \ \mu\text{m}$. Discard not less than 10 mL of the first filtrate, pipet *V* mL of the subsequent filtrate, add the dissolution medium to make exactly *V'* mL so that each mL contains about 11 μ g of celecoxib 48 ($C_{17}H_{14}F_3N_3O_2S$), and use this solution as the sample solution.

49 Separately, weigh accurately about 14 mg of Celecoxib RS,

50 dissolve in 2 mL of acetonitrile, and add the dissolution me-

51 dium to make exactly 250 mL. Pipet 10 mL of this solution, 52 add the dissolution medium to make exactly 50 mL, and use 53 this solution as the standard solution. Determine the absorb-54 ances, A_T and A_S , at 260 nm of the sample solution and stand-

ard solution as directed under Ultraviolet-visible Spectrophotometry <2.24>, using the dissolution medium as the blank.

57 Dissolution rate (%) with respect to the labeled amount of 58 celecoxib ($C_{17}H_{14}F_3N_3O_2S$)

 $=M_{\rm S} \times A_{\rm T}/A_{\rm S} \times V'/V \times 1/C \times 72$

 $M_{\rm S}$: Amount (mg) of of Celecoxib RS taken

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61 C: Labeled amount (mg) of celecoxib (C₁₇H₁₄F₃N₃O₂S) in
62 1 tablet

63 Assav Weigh accurately the mass of not less than 20 64 Celecoxib Tablets, and powder. Weigh accurately a portion 65 of the powder, equivalent to about 0.1 g of celecoxib (C17H14F3N3O2S), add 140 mL of a mixture of acetonitrile 66 and water (3:1), shake thoroughly, add a mixture of acetoni-67 68 trile and water (3:1) to make exactly 200 mL, and centrifuge. 69 Pipet 5 mL of the supernatant liquid, add exactly 5 mL of the 70 internal standard solution, add a mixture of acetonitrile and 71 water (3:1) to make 50 mL, and use this solution as the sam-72 ple solution. Separately, weigh accurately about 25 mg of 73 Celecoxib RS, dissolve in a mixture of acetonitrile and water 74 (3:1) to make exactly 50 mL. Pipet 5 mL of this solution, add 75 exactly 5 mL of the internal standard solution, add a mixture 76 of acetonitrile and water (3:1) to make 50 mL, and use this 77 solution as the standard solution. Perform the test with 25 μ L each of the sample solution and standard solution as directed 78 79 under Liquid Chromatography <2.01> according to the fol-80 lowing conditions, and calculate the ratios, $Q_{\rm T}$ and $Q_{\rm S}$, of the 81 peak area of celecoxib to that of the internal standard.

Amount (mg) of celecoxib (C₁₇H₁₄F₃N₃O₂S)
=
$$M_{\rm S} \times Q_{\rm T} / Q_{\rm S} \times 4$$

84 $M_{\rm S}$: Amount (mg) of Celecoxib RS taken

85 *Internal standard solution*—A solution of benzyl parahy86 droxybenzoate in a mixture of acetonitrile and water (3:1) (1
87 in 2000).

88 Operating conditions—

89 Detector: An ultraviolet absorption photometer (wave-90 length: 215 nm).

Column: A stainless steel column 4.6 mm in inside diameter and 25 cm in length, packed with phenylated silica gel for
liquid chromatography (5 µm in particle diameter).

94 Column temperature: A constant temperature of about95 60°C.

Mobile phase: Adjust 0.02 mol/L potassium dihydrogen
phosphate TS to pH 3.0 with phosphoric acid. To 600 mL of

98 this solution add 300 mL of methanol for liquid chromatog-

99 raphy and 100 mL of acetonitrile for liquid chromatography.

100 Flow rate: Adjust so that the retention time of celecoxib is

101 about 22 minutes.

102 System suitability—

103System performance: When the procedure is run with 25104 μ L of the standard solution under the above operating condi-105tions, the internal standard and celecoxib are eluted in this106order with the resolution between these peaks being not less107than 10.

108 System repeatability: When the test is repeated 6 times

109 with 25 μ L of the standard solution under the above operating

conditions, the relative standard deviation of the ratio of thepeak area of celecoxib to that of the internal standard is not

112 more than 1.0%.

113 Containers and storage Containers—Tight containers.