Celecoxib Capsules

Published on: 01.08.2024 **Last date for comments:** 14.09.2024

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Manufacturers, regulatory authorities, health authorities, researchers, and other stakeholders are invited to provide their feedback and comments on this draft proposal. Manufacturers are also invited to submit samples of their products to the IPC to ensure that the proposed monograph adequately controls the quality of the product(s) they manufacture. Comments and samples received after the last date will not be considered by the IPC before finalizing the monograph.

Please send any comments you may have on this draft document to lab.ipc@gov.in, with a copy to Dr. Gaurav Pratap Singh (email: gpsingh.ipc@gov.in) before the last date for comments.

Document History and Schedule for the Adoption Process

Description	Details
Document version	2.0
Monograph proposed for inclusion	IP 2026
Tentative effective date of monograph	July, 2026
First draft published on IPC website for public comments	06.06.2024
Draft revision published on IPC website for public comments	01.08.2024
Further follow-up action as required.	

Celecoxib Capsules

Celecoxib Capsules contain not less than 95.0 per cent and not more than 105.0 per cent of the stated amount of celecoxib, $C_{17}H_{14}F_3N_3O_2S$.

Usual strengths. 100 mg; 200 mg.

Identification

A. Determine by infrared absorption spectrophotometry (2.4.6). Shake a quantity of the mixed contents of the capsules containing 0.1 g of celecoxib with 25 ml of *ether*, filter and evaporate the filtrate to dryness. The residue comply with the following test. Compare the spectrum with that obtained with *celecoxib IPRS* or with the reference spectrum of celecoxib.

B. In the Assay, the principal peak in the chromatogram obtained with the test solution corresponds to the peak in the chromatogram obtained with reference solution (a).

Tests

Dissolution (2.5.2).

Apparatus No. 2 (Paddle),

Medium. 1000 ml of a solution containing 1.0 per cent w/v of *sodium dodecyl sulphate* in *0.04 M anhydrous trisodium orthophosphate*, adjusted to pH 12 with *orthophosphoric acid* or *sodium hydroxide solution, strong*.

Speed and time. 75 rpm and 45 minutes.

Withdraw a suitable volume of the medium and filter.

Determine by liquid chromatography (2.4.14)

Test solution. Use the filtrate, dilute if necessary, with the dissolution medium.

Reference solution (a). A 0.005 per cent w/v solution of *celecoxib IPRS* in the dissolution medium.

Reference solution (b). A solution containing 0.0024 per cent w/v, each of, *celecoxib impurity A IPRS* and *celecoxib impurity B IPRS* and 0.48 per cent w/v of *celecoxib IPRS* in a mixture of 75 volumes of *methanol* and 25 volumes of *water*. Dilute 1.0 ml of the solution to 10.0 ml with the dissolution medium.

Chromatographic system

– a stainless steel column 25 cm x 4.6 mm, packed with end-capped phenylsilane bonded to porous silica (5 µm) (Such as Supelcosil LC-DP),
– column temperature: 60°,
– mobile phase: a mixture of 60 volumes of a buffer solution prepared by dissolving 2.7 g of *sodium dihydrogen orthophosphate* in 1000 ml of *water*, adjusted to pH 3.0 with *orthophosphoric acid*, 30 volumes of *methanol* and 10 volumes of *acetonitrile*,
– flow rate: 1.5 ml per minute,
– spectrophotometer set at 215 nm,
–injection volume: 25 µl.

Inject reference solution (b). The test is not valid unless the resolution between the peaks due to celecoxib impurity A and celecoxib is not less than 1.5 and between the peaks due to celecoxib and celecoxib impurity B is not less than 1.8.

Inject reference solution (a) and the test solution.

Calculate the content of $C_{17}H_{14}F_3N_3O_2S$ in the medium.

Q. Not less than 75 per cent of the stated amount of $C_{17}H_{14}F_3N_3O_2S$.

Related substances. Determine by liquid chromatography (2.4.14).

Solvent mixture. 75 volumes of *methanol* and 25 volumes of *water*.

Test solution. Disperse a quantity of the mixed content of the capsules containing 50 mg of Celecoxib in the solvent mixture and dilute to 100.0 ml with the solvent mixture, filter.

Reference solution (a). A 0.0001 per cent w/v solution of celecoxib *IPRS* in the solvent mixture.

Reference solution (b). A solution containing 0.00024 per cent w/v, each of, *celecoxib impurity A IPRS* and *celecoxib impurity B IPRS* and 0.048 per cent w/v of *celecoxib IPRS* in the solvent mixture.

Use the chromatographic system as described under Dissolution.

Name	Relative retention time
Celecoxib impurity A[1]	0.9
Celecoxib (Retention time:about 27 minutes)	1.0
Celecoxib impurity B[2]	1.1

[1]4-[5-(3-methylphenyl)-3-(trifluoromethyl)-1H-pyrazol-1-yl] benzenesulphonamide,
[2]4-[3-(4-methylphenyl)-5-(trifluoromethyl)-1H-pyrazol-1-yl] benzenesulphonamide.

Inject reference solution (b). The test is not valid unless the resolution between the peaks due to celecoxib impurity A and celecoxib is not less than 1.5 and between the peaks due to celecoxib and celecoxib impurity B is not less than 1.8.

Inject reference solution (a) and the test solution. In the chromatogram obtained with the test solution, the area of any peak corresponding to celecoxib impurity A is not more than twice the area of the principal peak in the chromatogram obtained with reference solution (a) (0.4 per cent), the area of any other secondary peak is not more than the area of the principal peak in the chromatogram obtained with reference solution (a) (0.2 per cent) and the sum of areas of all the secondary peaks is not more than 5 times the area of the principal peak in the chromatogram obtained with reference solution (a) (1.0 per cent). Ignore any peak with an area less than 0.5 times the area of the principal peak in the chromatogram obtained with reference solution (a) (0.1 per cent).

Uniformity of dosage units (2.5.4). Comply with the tests stated under Capsules.

Other tests. Comply with the tests stated under Capsules.

Assay. Determine by liquid chromatography (2.4.14).

Solvent mixture. 75 volumes of *methanol* and 25 volumes of *water*.

Test solution. Weigh a quantity of the mixed contents of 20 capsules containing about 200 mg of Celecoxib in the solvent mixture, with the aid of ultrasound for 10 minutes, shake for 30 minutes, dilute to 100 ml with the solvent mixture and filter. Dilute 5.0 ml of the filtrate to 20.0 ml with the solvent mixture.

Reference solution (a). A 0.05 per cent w/v solution of *celecoxib IPRS* in the solvent mixture.

Reference solution (b). A solution containing 0.00024 per cent w/v, each of, *celecoxib impurity A IPRS* and *celecoxib impurity B IPRS* and 0.048 per cent w/v of *celecoxib IPRS* in the solvent mixture.

Use the chromatographic system as described under Dissolution test.

Inject reference solution (a) and (b). The test is not valid unless the resolution between the peaks due to celecoxib impurity A and celecoxib is not less than 1.5 and between the peaks due to celecoxib and celecoxib impurity B is not less than 1.8 in the chromatogram obtained with reference solution (b) and the relative standard deviation for replicate injections is not more than 2.0 per cent in the chromatogram obtained with reference solution (a).

Inject reference solution (a) and the test solution.

Calculate the content of celecoxib, $C_{17}H_{14}F_3N_3O_2S$ in the capsules.

Storage. Store protected from light and moisture, at a temperature not exceeding 30°.