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Celecoxib

$C_{17}H_{14}F_3N_3O_2S$ 381.4

4-[5-(4-Methylphenyl)-3-(trifluoromethyl)-1*H*-pyrazol-1-yl]benzenesulfonamide;

p-[5-*p*-Tolyl-3-(trifluoromethyl)pyrazol-1-yl]benzenesulfonamide CAS RN®: 169590-42-5; UNII: JCX84Q7J1L.

DEFINITION

Celecoxib contains NLT 98.0% and NMT 102.0% of $C_{17}H_{14}F_3N_3O_2S$, calculated on the anhydrous basis.

IDENTIFICATION

Change to read:

• **A.** ▲**Spectroscopic Identification Tests ⟨197⟩, *Infrared Spectroscopy*:** 197A, 197K or 197M▲ (CN 1-May-2020)

[Note—If the spectra obtained show differences, dissolve the substance to be examined and the Reference Standard separately in isopropyl alcohol, evaporate to dryness, and record the new spectra.]

• **B.** The retention time of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Assay*.

ASSAY

• **Procedure**

Buffer: 2.7 g/L of monobasic potassium phosphate adjusted with phosphoric acid to a pH of 3.0 ± 0.2

Mobile phase: Methanol, acetonitrile, and *Buffer* (3:1:6)

Diluent: Methanol and water (3:1)

System suitability solution: 0.5 mg/mL of USP Celecoxib RS and 2.4 µg/mL each of USP Celecoxib Related Compound A RS and USP Celecoxib Related Compound B RS in *Diluent*

Standard solution: 0.5 mg/mL of USP Celecoxib RS in *Diluent*

Sample solution: 0.5 mg/mL of Celecoxib in *Diluent*

Chromatographic system

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

Mode: LC

Detector: UV 215 nm

Column: 4.6-mm × 25-cm; 5-µm packing L11

Column temperature: 60°

Flow rate: 1.5 mL/min

Injection size: 25 µL

Run time: About 1.5 times the celecoxib peak elution

System suitability

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

Suitability requirements

Resolution: NLT 1.8 between celecoxib related compound A and celecoxib and NLT 1.8 between celecoxib and celecoxib related compound B, *System suitability solution*

Relative standard deviation: NMT 0.73%, *Standard solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of $C_{17}H_{14}F_3N_3O_2S$ in the portion of Celecoxib taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times 100$$

r_U = peak response from the *Sample solution*

r_S = peak response from the *Standard solution*

C_S = concentration of the *Standard solution* (mg/mL)

C_U = concentration of the *Sample solution* (mg/mL)

Acceptance criteria: 98.0%–102.0% on the anhydrous basis

IMPURITIES

Inorganic Impurities

• **Residue on Ignition ⟨281⟩:** NMT 0.2%, using a platinum crucible

Organic Impurities

• **Procedure**

Buffer, Mobile phase, Diluent, System suitability solution, Sample solution, and **Chromatographic system:** Proceed as directed in the *Assay*.

Standard solution: 0.5 µg/mL of USP Celecoxib RS in *Diluent*

System suitability

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

Suitability requirements

Resolution: NLT 1.8 between celecoxib related compound A and celecoxib and NLT 1.8 between celecoxib and celecoxib related compound B, *System suitability solution*

Signal-to-noise ratio: NLT 20, *Standard solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of each impurity in the portion of Celecoxib taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times 100$$

r_U = peak response for each impurity in the *Sample solution*

r_S = peak response of celecoxib in the *Standard solution*

C_S = concentration of celecoxib in the *Standard solution* (mg/mL)

C_U = concentration of Celecoxib in the *Sample solution* (mg/mL)

Acceptance criteria

Individual impurities: See *Table 1*. [Note—Disregard any impurity peak less than 0.05%.]

Table 1

Name	Relative Retention Time	Acceptance Criteria, NMT (%)
Celecoxib related compound A[a]	0.9	0.4
Celecoxib	1.0	—
Celecoxib related compound B[b]	1.1	0.10
Individual unspecified impurity	—	0.10
Total impurities	—	0.5

[a] 4-[5-(3-Methylphenyl)-3-(trifluoromethyl)-1*H*-pyrazol-1-yl]benzenesulfonamide.

[b] 4-[3-(4-Methylphenyl)-5-(trifluoromethyl)-1*H*-pyrazol-1-yl]benzenesulfonamide.

SPECIFIC TESTS

• **Water Determination, *Method I*⟨921⟩:** NMT 0.5%, using a 400-mg sample

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in tight containers, protected from light and moisture. Store at room temperature.

• **USP Reference Standards ⟨11⟩**

USP Celecoxib RS

p-[5-*p*-Tolyl-3-(trifluoromethyl)pyrazol-1-yl]benzenesulfonamide.

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USP Celecoxib Related Compound A RS

4-[5-(3-Methylphenyl)-3-(trifluoromethyl)-1*H*-pyrazol-1-yl]benzenesulfonamide.

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USP Celecoxib Related Compound B RS

4-[3-(4-Methylphenyl)-5-(trifluoromethyl)-1*H*-pyrazol-1-yl]benzenesulfonamide.

$C_{17}H_{14}F_3N_3O_2S$ 381.4

Auxiliary Information - Please check for your question in the FAQs before contacting USP.

Topic/Question	Contact	Expert Committee
CELECOXIB	Documentary Standards Support	SM22020 Small Molecules 2

Chromatographic Database Information: Chromatographic Database

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