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Determination of Celecoxib in human plasma using liquid chromatography with high resolution time of flight-mass spectrometry

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ABSTRACT

A sensitive method for the determination of Celecoxib (CXB) in human plasma samples was developed using liquid chromatography coupled to electrospray ionization and time of flight mass spectrometry (LC–ESI–TOF–MS). A full factorial design of experiments (FF-DOE) methodology was applied to optimize the ESI conditions for CXB determination and also to predict the effects of interactions of multiple parameters affecting ionization (i.e., capillary voltage, fragmentor voltage, electrolyte and electrolyte concentration). The optimum ionization voltages were 4500 V and 220 V for capillary and fragmentor, respectively. Even though the highest ESI efficiency was obtained without electrolytes, the addition of 1.0 mM ammonium acetate was shown to be essential to buffer the matrix effect and ensure a consistent response. In contrast to previous studies, deuterated CXB was used as a recovery (surrogate) standard, which enabled the correction of CXB loss during sample preparation. The extraction recovery using solid phase extraction was 87–98%. The instrumental limit of detection of CXB (LOD), 0.33 ng/mL, and matrix affected LOD, 0.55 ng/mL, were similar and comparable to the previously reported LC–MS/MS LODs. This method was employed to determine CXB concentrations in human plasma samples. Upon administration of 400 mg CXB to the healthy women, the concentrations found in the plasma were 440–3300 ng/mL. The inter-day repeatability was less than 4% RSD.

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1. Introduction

Celecoxib (CXB), 4-[5-(4-methylphenyl)-3-(trifluoromethyl) 1H-pyrazole-1-yl] benzene sulfonamide (Fig. 1), which was introduced to clinical practice and marketed under the brand name Celebrex, is one of the COX-2 inhibitors primarily designed to minimize the adverse effects associated with classical nonspecific non-steroidal anti-inflammatory drugs (NSAIDs) [1]. Clinical studies have demonstrated that CXB leads to a significant reduction in joint pain, tenderness and swelling with a statistically significantly lower incidence of gastric ulceration [2]. Moreover, recent studies have also indicated that COX-2 inhibitors decrease colon polyp formation and, thereby, colorectal cancer risk in patients at high risk, such as those with familial adenomatous polyposis [3–8].

A number of analytical approaches were previously employed for CXB determination in pharmaceutical formulations, human plasma and serum samples. These include UV spectrophotometric, fluorimetric, and voltametric methods; however, most often chromatographic methods were used (e.g., HPLC, TLC, and MIKEC, etc.) [9]. The results of LC–MS studies for the quantitation of CXB in pharmaceuticals [10], human plasma, serum, synovial fluid and urine [11–16], animal plasma and urine [16–21] and other matrices [22,23] are summarized in Table 1. The majority of these methods employed either isocratic or gradient LC analysis using electrospray ionization (ESI) with tandem mass spectrometry. Alternatively, an atmospheric pressure chemical ionization (APCI) was employed; however, the reported limits of detection (LODs) of CXB were higher (4–20 ng/mL) for APCI than for ESI methods. For ESI, the lowest LOD reported was 0.02 ng/mL, when using 17 mM acetic acid as the

Abbreviations: APCI, Atmospheric Pressure Chemical Ionization; CXB, Celecoxib; EIC, Extracted Ion Chromatogram; ESI, Electrospray Ionization; FIA, Flow Injection Analysis; FF-DOE, Full Factorial Design of Experiment; HPLC, High Performance Liquid Chromatography; HR, TOF MS High Resolution Time of Flight Mass Spectrometry; I.S., Internal Standard; LOD, Limit of Detection; R.S., Recovery Standard.

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Table 1
LC–MS methods employed for CXB analysis in biological fluids.

Sample matrix	HPLC method/Mobile phase (v/v)	Column L × I.D.; particle size	Type of MS ionization	Electrolyte (mM)	Sample preparation method	Sample volume (μL)	Injection volume (μL)	LOD[a] (ng/mL)	LOQ[a] (ng/mL)	Reference
Pharmaceuticals	Isocratic, acetonitrile–1%CH_3COOH (80:20)	Shim-Pack GLC CN C_{18} 150 × 6 mm; 5 μm	APCI-Ion Trap MS +Ve mode, SIM[b]	167 mM CH_3COOH	Extracted with methanol	1 tablet	20	NA[c]	50	[10]
Human plasma	Isocratic, acetonitrile–1%CH_3COOH (80:20)	Shim-Pack GLC CN C_{18} 150 × 6 mm; 5 μm	APCI-Ion Trap MS +Ve mode, SIM	167 mM CH_3COOH	LLE[d] with ethylacetate	1000	20	20	50	[11]
Human plasma	Isocratic, methanol–water–(50:50)–1%CH_3COOH)	Nucleosil C_8 110 × 2 mm; 5 μm	APCI-Ion Trap MS +Ve mode, SRM[e]	167 mM CH_3COOH	LLE with dichloromethane:hexane (50:50, v/v)	1000	100	NA	5	[12]
Human/rat plasma	Isocratic, acetonitrile–water–0.025% NH_4OH (85:15)	Nucleosil C_{18} 30 × 2 mm; 5 μm	ESI–MS/MS –Ve mode, SRM	0.025% NH_4OH	SPE[f] C_{18}, 100 mg, 1 mL	200	10	NA	NA	[16]
Human plasma	Isocratic, methanol–10 mM CH_3COONH_4 (75:25)	Purospher C_{18} 55 × 2 mm; 3 μm	ESI–MS/MS –Ve mode, SRM	10 mM CH_3COONH_4	Precipitation, centrifugation	100	2	NA	7	[15]
Human plasma	Isocratic, methanol–10 mM $COONH_4$ (95:5, pH 3.0)	Luna HILIC, C_{18} 50 × 2 mm; 3 μm	ESI–MS/MS –Ve mode, SRM	10 mM $COONH_4$	LLE with methyl *tert*-butyl ether	200	NA	NA	10	[14]
Equine plasma	Gradient, A: 0.1% CH_3COOH in water, B: acetonitrile	Supelcosil ABZ_Plus, C_{18} 33 × 2.1 mm; 3 μm	ESI–MS/MS +Ve mode, SRM	17 mM CH_3COOH	Protein precipitation trichloroacetic acid, SPE C_8-SCX, 130 mg, 3 mL	3000	20	0.02	NA	[18]
Rat plasma	Isocratic, methanol–10 mM CH_3COONH_4 (80:20, pH 4)	YMC-Pack, C_{18} 50 × 4.6 mm; 3 μm	ESI–MS/MS –Ve mode, SRM	10 mM CH_3COONH_4	Protein precipitation	50	10	NA	1.5	[19]
Equine urine	Gradient, A:0.1% CH_3COOH in water, B: acetonitrile	Supelcosil ABZ Plus, C_{18} 33 mm × 2.1 mm; 3 μm	ESI–MS/MS –Ve mode, SRM	17 mM CH_3COOH	Protein precipitation trichloroacetic acid, SPE C_8-SCX, 130 mg, 3 mL	3000	20	4	NA	[17]
Equine urine	Gradient, A:0.1% CH_3COOH in water, B: acetonitrile	Acquity C_{18} BEH 100 × 2.1 mm; 1.7 μm	ESI–MS/MS -Ve mode, SRM	17 mM CH_3COOH	SPE Abs Elut Nexus	6000	5	0.25	NA	[21]
Rabbit plasma/urine	Gradient, A: acetonitrile/0.025 M CH_3COONH_4 (20:80, pH 4.5),B: acetonitrile/0.025 M CH_3COONH_4 (60:40, pH 4.5)	NovaPak C_{18} 150 mm × 3.9 mm; 4 μm	ESI–MS/MS –Ve mode, SRM	25 mM CH_3COONH_4	SPE C_{18}, 100 mg, 1 mL	1000	100	NA	NA	[20]
Human serum and synovial fluid	Gradient, A: 0.1% HCOOH in water, B: methanol	Acquity C_{18} BEH 50 × 2.1 mm; 1.7 μm	ESI-qTOF–MS +Ve mode, SRM ICP-MS	22 mM HCOOH	LLE with ethylacetate	200 200 tablet	10 10	12 55	NA	[13]
Ayurvedic/herbal products	Gradient, A: 10 mM CH_3COONH_4 in water, B: methanol	C_{18} 250 mm × 4.6 mm; 5 μm	APCI–TOF–MS, +Ve mode	10 mM CH_3COONH_4	Sonication, centrifugation		10	NA	NA	[23]
Milk	Gradient, A:methanol/acetonitrile 8:2, B: 0.010 M $COONH_4$ (pH 5)	Luna C_8 150 × 2.1 mm; 3 μm	ESI–MS/MS -Ve mode, SRM	10 mM $COONH_4$	LLE with acetonitrile, SPE NH_2	10^4	20	NA	NA	[22]

[a] LOD/LOQ reported in sample matrix.
[b] SIM: selected ion monitoring.
[c] NA: data not available.
[d] LLE: liquid–liquid extraction.
[e] SRM: selected reaction monitoring.
[f] SPE: solid phase extraction.

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