[PD4-18] [04/19/2002 (Fri) 10:00 - 13:00 / Hall E]

Enantioselective Determination of Flurbiprofen in Human Urine using column switching-HPLC

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We separated flurbiprofen that is racemic compound using RU-2 column by column - switching HPLC method and improved greatly detection limit (LOD) in human urine. Moreover, it is not pretreatment of sample. We measured specific rotation using polarimeter and confirmed d - form and l - form. This method was showed linearity between 0.23~110.8μg/mL of both isomers. Result, LOD was l-form : 0.023μg/mL, d-form : 0.005μg/mL and C.V value in Inter-day and intraday of precision showed 1.84% low.

[PD4-19] [04/19/2002 (Fri) 10:00 - 13:00 / Hall E]

In vitro metabolism of a novel phosphodiesterase-5 inhibitor DA-8159 in rat liver preparations using LC/MS

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The in vitro metabolism of a new erectogenic, DA-8159 has been studied by LC with UV detection and on-line LC-electrospray mass spectrometry using rat hepatic microsomal incubation and rat liver perfusion. Both rat liver microsomal incubation of DA-8159 in the presence of NADPH and single-pass liver perfusion of DA-8159 resulted in the formation of three metabolites (M1-3). M1 was tentatively identified as hydroxy-DA-8159. M2 and M3 were identified as N-demethyl-DA-8159 and 5-(2-propyloxy-5-aminosulphonyl phenyl)-1-methyl-3-propyl-1,6-dihydro-7H-pyrazolo(4,3-d)pyrimidin-7-one (DA-8164), respectively, on the basis of LC-MS/MS analysis with authentic standards. The pathways of DA-8159 metabolism were hydroxylation, N-hydrolysis and N-demethylation.

[PD4-20] [04/19/2002 (Fri) 10:00 - 13:00 / Hall E]

In vitro metabolic stability of sildenafil derivatives and identification of their metabolites by liquid chromatography/ion trap mass spectrometry

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It was examined that in vitro metabolic stability of sildenafil derivatives and detection of their metabolites by high performance liquid chromatography (HPLC) coupled with ion trap mass spectrometry (MS). The samples from incubation with human and rat liver microsomes were analyzed by HPLC/electrospray (ESI) ion trap MS in full-scan mode. The metabolic stability of the drugs was determined by comparing their signals after incubation for 0 and 30 min, respectively. In addition, the technique allowed simultaneous detection of metabolites formed during the same incubation without having to reanalyze the samples. The metabolites were first characterized by nominal mass measurement of the corresponding protonated molecules. Subsequent multi-stage tandem spectrometry (MSn) on the ion trap instrument allowed confirmation of the detected metabolites.

[PD4-21] [04/19/2002 (Fri) 10:00 - 13:00 / Hall E]

Chiral Separation of Aromatic Amino Acids by Capillary Electrophoresis using Successive