

of tiropramide in human plasma was developed. Tiropramide and internal standard, cisapride were extracted from human plasma with MTBE at basic pH. A reverse-phase LC separation was performed on Luna C8 column with the mixture of acetonitrile-ammonium formate (10 mM, pH 4.5) (5:5, v/v) as mobile phase. The detection of analytes was performed using an electrospray ionization tandem mass spectrometry with positive ion mode in the multiple-reaction-monitoring mode. The assay run-time was less than 3 min. The single liquid-liquid extraction quantitatively recovered tiropramide and the internal standard from plasma samples. The lower limits of quantification for tiropramide was 2.0 ng/ml. The data confirmed that the plasma samples of tiropramide were stable at room temperature and for up-to three freeze-thaw cycles. The method showed a satisfactory sensitivity, precision, accuracy and selectivity.

[PD4-8] [04/18/2003 (Fri) 13:30 - 16:30 / Hall P]

Chiral Separation of Aromatic Amino Acids by Capillary Electrophoresis using (+)-18-crown-6 tetracarboxylic acid and (-)-18-crown-6 tetracarboxylic acid as Chiral Selectors

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Recently, particular attention has been paid to the chiral separation of amino acid enantiomers because of their different biological activities. Hence, the high optical purity of aromatic amino acids is critical because of their important functions in the central nervous system. For the accurate chiral discrimination, we attempted to exploit the crosschecking each enantiomeric migration orders of aromatic amino acids measured using (+)-18C6H4TA and (-)-18C6H4TA as the chiral selectors under pH 2.0, tris/citric acid buffer.

[PD4-9] [04/18/2003 (Fri) 13:30 - 16:30 / Hall P]

Determination of rebamipide in human plasma by column-switching high-performance liquid chromatography.

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A column-switching semi-micro HPLC method with fluorescence detection was developed for the direct analysis of rebamipide in human plasma. Plasma was filtered through a 0.45 μm membrane filter and 5 μl of the filtrate was directly injected onto the pre-column. After elution of the plasma proteins to waste, the retained rebamipide and internal standard (ofloxacin) were transferred to a C18 semi-microcolumn (5 μm , 150 \times 2.0mm) where they were separated using acetonitrile-1.4% acetic acid (40:60, v/v) as mobile phase. The column effluent was monitored by fluorescence detection at an excitation wavelength of 330 nm and an emission wavelength of 375 nm. The standard calibration curve was linear over the concentration range 5-500 ng/ml with correlation coefficient of 0.999. The lower limit of quantification (at signal-to-noise ratio S/N=10) was 5 ng/mL. This method showed good precision (intra-day CV(%) \leq 5.829, inter-day CV(%) \leq 8.447) and accuracy (100.0-105.3%) with the total analysis time of 11min. The present method was successfully applied to the pharmacokinetic study of rebamipide in man.

[PD4-10] [04/18/2003 (Fri) 13:30 - 16:30 / Hall P]