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 migraion 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 liqiud 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 ㎛ membrane filter and 5 ㎕ 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 ㎛,150×2.0mm) 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 ㎚. The standard calibration curve was linear over the concentration range 5-500 ng/㎖ 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(%) ≤5.829 , inter-day CV(%) ≤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]

Chromatographic chiral resolution of several racemic drugs containing primary amino moiety on a chiral stationary phase

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A chiral stationary phase (CSP) prepared by bonding (18-crown-6)-2,3,11,12-tetracarboxylic acid (18-C-6-TA) to aminopropyl silica gel by HPLC was used in resolving several racemic drugs containing primary amino moiety. Most compounds used in this study were resolved on the CSP using 80% methanol in water (V/V) containing 10mM sulfuric acid as a mobile phase. These results on the CSP were compared to those on the similar CSP derived from 18-C-6-TA of the same chiral selector by different connecting method.

[PD4-11] [04/18/2003 (Fri) 13:30 - 16:30 / Hall P]

Quantification of intact ambroxol tablet using near-infrared spectroscopy

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NIR reflectance spectroscopy, using a fiber-optic probe was used to determine rapidly and non-destructively the content of ambroxol in intact ambroxol 30 mg (nominal content 12.5% m/m ambroxol) tablets by collecting NIR spectra in range 1100 ~ 1750 nm and using PLSR calibration method. The tablets (10.3 ~ 15.9% m/m ambroxol, i.e., 82 ~ 127% of the nominal label content) were used 7 calibration set and 5 validation set. Unique spectral features of the active constituent (ambroxol) were identified i! n the NIR spectra of the tablet ingredients. The developed NIR method gave results comparable to the values from preparation of tablets, SEC and SEP being 0.49% and 0.49% m/m respectively. The method showed good accuracy and repeatability but bad intermediate precision. NIR spectroscopic determination in intact tablets allowed the potential use of the method on-line for real time monitoring of a running production process.

[PD4-12] [04/18/2003 (Fri) 13:30 - 16:30 / Hall P]

Determination of dextromethorphan and its metabolite dextrorphan in human urine by High-performance liquid chromatography

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A simple and accurate reverse-phase high performance liquid chromatography (HPLC) coupled with photodiode array was developed for the determination of dextromethorphan(DM) and its metabolite dextrorphan(DX) in human urine. Chromatographic separation was accomplished on a cyano analytical column at 220 nm using a mobile phase containing 25 mM triethylammonium phosphate buffer(pH 3.0) in a 0-70% ACN gradient and triazolam(TZ) was used as internal standard (I.S.). There was a linear relationship between peak area ratios of analytes to I.S, and concentration of analytes over the concentration range 10-200 ㎍/㎖ for DM and DX with r value of 0.9962 and 0.9958 respectively. The urinary recovery was 92.69~96.79 % (R.S.D. 2.28~4.03 %) for DM and 81.01~84.19 (R.S.D. 2.30~3.08 %) for DX. The limits of detection(LOD) were