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A simple, accurate GC analytical technique for the determination of phthalates, commonly used as plasticizers during the manufacturing process of PVC bags, in blood component preparations was developed and validated. The blood component preparations were extracted with n-hexane. The n-hexane layer was evaporated to dryness and the residue was dissolved in 1 mL of n-hexane and analyzed by GC and GC/MS. A linear response was found for a variety of phthalates tested within the range 0.5~99.5 ㎍/L with correlation coefficient (r) greater than 0.99. These results suggested that this method could be applied to determine the phthalates released from the blood component preparations.

[PD4-6] [04/18/2003 (Fri) 13:30 - 16:30 / Hall P]

Determination of L-FMAUS, a new L-FMAU derivative, in rat plasma and urine by high-performance liquid chromatography

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A high-performance liquid chromatographic method using the liquid extraction procedure was developed for the determination of L-FMAUS, a new L-FMAU derivative, in rat plasma and urine using 3-aminophenyl sulfone as an internal standard. A 100-μl aliquot of distilled water containing the L-cysteine (100 mg/ml) was added to a 100-μl aliquot of biological sample. L-Cysteine was employed to protect binding between 5'-thiol of I and protein in the biological sample. After vortex-mixing for 30 s, a 50-μl aliquot of the mobile phase containing the internal standard (10 μg/ml of 3-aminophenyl sulfone) and a 1-ml aliquot of ethyl acetate were added. After vortex-mixing and centrifugation at 9000 g for 4 min, the ethyl acetate layer was collected and dried under nitrogen gas. The residue was reconstituted with a 100-μl aliquot of the mobile phase, centrifuged, and a 50-μl aliquot of the supernatant was injected directly onto a C_{18} reversed-phase column. The mobile phases, 50 mM KH_2PO_4 (pH = 2.5) : acetonitrile (85:15, v/v) for rat plasma and 50 mM KH_2PO_4 (pH 2.5) : acetonitrile : methanol (85:10:5, v/v/v) for urine sample, were run at a flow-rate of 1.2 ml/min. The column effluent was monitored by an ultraviolet detector set at 265 nm. The retention times for I and the internal standard were approximately 9.7 and 12.5 min, respectively, in plasma samples and the corresponding values in urine samples were 16.8 and 14.9 min. The detection limits for I in rat plasma and urine were 0.1 and 0.5 μg/ml, respectively. The coefficients of variation of the assay (within-day and between-day) were generally low; below 8.60% for plasma and 8.86% for urine. No interferences from endogenous substances were found.

[PD4-7] [04/18/2003 (Fri) 13:30 - 16:30 / Hall P]

Determination of tiropramide in human plasma by LC/MS/MS

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A liquid chromatography-tandem mass spectrometric (LC/MS/MS) method for the determination

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]