derivatization procedure (concentration of GATC, reaction temperature and time) was investigated.

[PD4-3] [04/18/2003 (Fri) 13:30 - 16:30 / Hall P]

Determination of meloxicam in human plasma by semi-micro high -performance liqiud chromatography.

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This study describes a simple and sensitive semi-micro HPLC method with UV detection and direct deproteinization. The plasma protein was precipitated using perchloric acid (60%) and the supernatant was directly injected onto the semi-micro HPLC system. The separation was achieved on a C18 (25 mm X 2.0 mm I.D) analytical column with a mobile phase of sodium acetate buffer (pH 3.5, 50 mmol) - acetonitrile (60:40, V/V). The retention time observed for meloxicam and internal standard (piroxicam) were 4.6 min and 3 min, respectively. The column effluent was monitored by UV detection at 355 nm. The method was linear over the concentration range 20-1500 ng/㎖ with correlation coefficient of 0.999. The lower limit of quantification (at signal-to-noise ratio S/N=10) was 20 ng/mL. This method showed good precision (intra-day CV (%) ≤3.010 , inter-day CV(%) ≤7.329) and accuracy (101.1-109.4%). The present method was successfully applied to the pharmacokinetic study of meloxicam in man.

[PD4-4] [04/18/2003 (Fri) 13:30 - 16:30 / Hall P]

Simultaneous Chiral Discrimination of Nine Non-Steroidal Antiinflammatory Drugs by Cyclodextrin-Modified Capillary Electrophoresis in Normal and Reversed Polarity Modes

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Simultaneous enantioseparation of nine racemic non-steroidal antiinflammatory drugs (NSAIDs) for their accurate chiral discrimination was achieved by cyclodextrin (CD) modified capillary electrophoresis in the normal polarity (NP) mode and in the reversed polarity (RP) mode. The NP mode employed neutral tri-O-methyl-β-cyclodextrin (TMβCD) as a selector dissolved in MES buffer (pH 6.0). The RP mode used a mixture of neutral TMβCD and slightly charged carboxymethyl-β-CD as the dual selectors dissolved in phosphate buffer (adjusted to pH 3.0 with triethanolamine) containing hexadimethrine bromide. The present NP and RP modes were complements each of the other for the simultaneous enantiomeric purity test of ibuprofen, ketoprofen and flurbiprofen, and also for the chiral separation of ibuprofen and its metabolites in urine.

[PD4-5] [04/18/2003 (Fri) 13:30 - 16:30 / Hall P]

A GC analytical method of phthalates in plasticized blood component preparations

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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