[PD4-22] [04/18/2003 (Fri) 13:30 - 16:30 / Hall P]

Therapeutic Monitoring on Urinary Nucleoside and Polyamine Levels of Cancer Patients by CE and GC under Acupuncture Treatment

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Modified nucleosides and polyamines excreted in urine are well-known as biochemical markers for cancer. The metabolomics on the urinary nucleosides and polyamines is thus gaining interest in the cancer study. In this study, the levels of nucleosides and polyamines in urine samples from cancer patients under acupuncture treatment were determined by high resolution capillary electrophoresis and gas chromatography, respectively. The usefulness of the metabolic profiling analyses of urinary nucleosides and polyamines together for the therapeutic monitoring of cancer patients under acupuncture treatment will be demonstrated.

[PD4-23] [04/18/2003 (Fri) 13:30 - 16:30 / Hall P]

Simple and Sensitive Determination of Baclofen in Human Plasma by Column-Switching and Semi-Micro High-Performance Liquid Chromatography

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Purpose : Using a column-switching technique, highly sensitive and selective semi-micro high-performance liquid chromatographic (HPLC) method has been developed for the determination of baclofen in human plasma.
Method : Following precipitation of plasma sample containing baclofen with zinc sulfate-acetonitrile, samples were directly injected on to the system. The analyte was retained in an enrichment column while endogeneous plasma components were eluted to waste. Baclofen was then back-flushed to the semi-micro C18 analytical column for separation and quantification with ultraviolet detector at 220 nm. Used mobile phases for pretreatment and separation were 20 mM potassium phosphate (pH 2.0) and 20 mM potassium phosphate (pH 3.6)-methanol (82:18, v/v), respectively.
Results : The analysis time of one sample was approximately 23 min. The calibration curves were linear in the concentration range of 25-800 ng/ml. The limit of quantitation for the baclofen was 25 ng/ml. The inter- and intra-day reproducibility (CV%) are less than 12%, even at the limit of quantification of the method. The method showed good speed, sensitivity, and reproducibility. After an oral dose of 25 mg of baclofen, blood sample was collected at several time points and plasma was analyzed using the method developed in this study.
Conclusions : This method presented is a simple column switching HPLC UV detection method for the determination of baclofen in plasma. The method showed speed, sensitivity, and reproducibility. The method has been applied to a pharmacokinetic study with great success.

[PD4-24] [04/18/2003 (Fri) 13:30 - 16:30 / Hall P]

Simultaneous determination of talniflumate and its metabolite in human plasma by high-performance liquid chromatography

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Talniflumate is a potent analgesic and anti-inflammatory drug widely prescribed in rheumatoid diseases. The purpose of this work was to develop and validate a specific and robust method for the simultaneous determination of talniflumate and its metabolite, niflumic acid, in human plasma. Indomethacin was used as an internal standard (IS). To simultaneously determine the plasma concentration of talniflumate and niflumic acid, IS solution and methanol were added to plasma samples and the mixture were centrifuged at 3000 g for 30 min. Then, 60 μl of supernatant was injected onto the HPLC reversed-phase column (C18). The signals were monitored by UV detector at 288 nm. The run time was 20 min per sample and analyte was quantified by linear regression of peak area ratio. This assay was validated at a nominal range of 0.1 to 5 μg/ml. Linear over the calibration range was > 0.9972. The interday accuracy ranged from 92.3 to 99.09 % with precision ranging from 91.44 to 94.62 %. The intraday accuracy ranged from 90.65 to 99.29 % with precision ranging from 91.36 to 94.19 %. The retention times of the IS, niflumic acid and talniflumate were 6.5, 7.5 and 13.5 min, respetively. This analytical method was shown to be accurate and reproducible. This method could be suitable for the simultaneous determination of talniflumate and its metabolite in human plasma.

[PD4-25] [04/18/2003 (Fri) 13:30 - 16:30 / Hall P]

Comparison of liquid chromatographic enantiomer resolution of racemic amino compounds on chiral stationary phases of crown ether type

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ChiroSil RCA(+) and SCA(-) HPLC chiral stationary phases (CSPs) developed by covalently bonding (+)- and (-)-(18-crown-6)-2,3,11,12-tetracarboxylic acid (18-C-6-TA) to silica gel were employed for enantioresolution of racemic amino compounds, respectively. Also, these 18-C-6-TA covalently bonded CSPs were compared to a commercially available Crownpak CR CSP prepared by coating chiral crown ether as a chiral selector on ODS column. It was shown that these ChiroSil RCA(+) and SCA(-) columns have the advantage of the reversal of elution order. For the resolution of diphenylalanine enantiomers, especially, it was observed that the chromatographic parameters such as separation factors and retention times are greatly influenced by mobile phase conditions.

[PD4-26] [04/18/2003 (Fri) 13:30 - 16:30 / Hall P]

Identification of Impurities in a Sample of Illicitly Synthesized Methamphetamine

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Analysis of illicit methamphetamine samples seized in Korea is discussed. The samples are extracted with the small portion of ethyl acetate under neutral conditions and the extracts are analyzed by GC-MS. Several impuritiy peaks are found in each chromatrogram. Eight