

rapid liquid chromatography/ electrospray ion trap tandem mass spectrometry (LC/MS/MS) method combined with high-throughput solid phase extraction (SPE) has been developed and validated for the simultaneous quantitative determination of ENP and ENPT in human plasma. After addition of internal standard, samples were simultaneously extracted by 96-well C18-SPE cartridge. The organic extract was evaporated to dryness, with subsequent analyzed by LC/MS/MS using the selective reaction monitoring (SRM) mode and time segment. The product ions of ENP and ENPT were characterized by m/z 234, m/z 303 and m/z 206, m/z 303, respectively. These product ions were monitored for the quantitation of ENP and ENPT. As a results, the present method for the simultaneous determination of ENP and ENPT was accurate and reproducible.

[PD4-23] [ 10/18/2002 (Fri) 13:30 - 16:30 / Hall C ]

Simultaneous determination of sildenafil and its active metabolite in human plasma using LC/MS/MS

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The LC/MS/MS method for the simultaneous determination of sildenafil and its active metabolite N-demethylsildenafil in human plasma was developed. Sildenafil, its active metabolite and the internal standard, DA-8159 were extracted from human plasma by liquid-liquid partitioning. A reverse-phase HPLC separation was performed on Luna phenylhexyl column with the mixture of acetonitrile-5 mM ammonium formate (55:45, v/v) as mobile phase. The detection was conducted by electrospray ionization tandem mass spectrometry in the multiple reaction monitoring mode. The lower limits of quantification for sildenafil and N-demethylsildenafil were 2.0 ng/ml. The method showed a satisfactory sensitivity, precision, accuracy, recovery and selectivity.

[PD4-24] [ 10/18/2002 (Fri) 13:30 - 16:30 / Hall C ]

Algorithm for finding the best regression models using NIR spectra

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An algorithm for finding the best regression models has been developed using NIR spectral data. In cases of regression analysis for quantitation with NIR spectral data, it is very critical to find essential features from the spectral data. This task was accessed in two ways. The first one was to use all-possible combinations of variables (wavelengths). Correlation coefficients at each spectral points were calculated to get initial set of variables and all of the possible combinations of variable sets were tested with SEC, SEP and/or R<sup>2</sup>. The second one was to use principal component(PC) analysis with PC selection by all-possible combinations. The initial set of PCs was obtained using Malinowski's IND function or PRESS and all-possible combinations of the PCs were evaluated in terms of SEC and/or SEP. These algorithms were tested with NIR spectral data of synthetic biological fluid to find concentrations of minor component.

[PD4-25] [ 10/18/2002 (Fri) 13:30 - 16:30 / Hall C ]

Chromatographic Analysis of Cilostazol in Human Plasma

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Cilostazol, a quinolinone derivative that inhibits phosphodiesterase, is used for the treatment of intermittent claudication resulting from peripheral arterial disease. In order to perform pharmacological and pharmacokinetic studies of cilostazol, specific, sensitive and reproducible analysis methods are demanded. Therefore, in the present study, an analytical method of cilostazol in human plasma was developed using semi-microbore HPLC equipped with automated column switching system. After direct injection of human plasma, deproteinization and fraction of analyte occurred on a Capcell Pak MF Ph-1 column (20 x 4 mm I.D.). The cilostazol fraction was transferred from the MF Ph-1 column to an intermediate C18 column (35 x 2 mm I.D.) using 10% acetonitrile in