0.0012±0.0005 % (n=13). In addition, total ash content was 4.96±2.72 %, and loss on drying was 11.87±1.26 %.

[PD4-31] [2003-10-10 14:00 - 17:30 / Grand Ballroom Pre-function]

Quantitative Determination of Amygdalin Epimers from Armeniacae Semen by High Performance Liquid Chromatography.

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D-Amygdalin and its conversion product, neoamygdalin, were clearly separated on reverse-phase column chromatography by an optimized eluent of 10 mM sodium phosphate buffer (pH 3.5) containing 8.5% acetonitrile. Linearity for analyzing D-amygdalin and neoamygdalin was observed in the range from 0.05 to 0.5 mM. The detection limits for D-amygdalin and neoamygdalin were ca. 5 uM per injected amount. When extracting amygdalin from a whole piece of Armeniacae Semen in the boiling aqueous solution, there was almost no influence of emulsin; it resulted in higher extraction yield. However, a defect, converting D-amygdalin into neoamygdalin by heating, was found. The problem was solved when 4% citric acid was used as an extractant, and the 4% citric acid also prevented from being affected by emulsin. In addition, the extraction yield remained the same with when methanol is used as an extractant regardless of cutting size. HPLC condition as follows Column : Synergi 4μ Hydro-RP 80Å(4.6㎜×250㎜) Mobile Phase : 10mM Sodium Phosphate buffer(pH 3.5) containing 8.5% Acetonitrile Column Temperature : 10℃ Wavelength : 214㎚

[PD4-32] [2003-10-10 14:00 - 17:30 / Grand Ballroom Pre-function]

Determination of Eupatilin in Human Plasma by Liquid Chromatography/Electrospray Ionization Tandem Mass Spectrometry

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A rapid, sensitive and selective liquid chromatography-tandem mass spectrometric (LC/MS/MS) method for the determination of eupatilin in human plasma was developed. Eupatilin and internal standard, (S)-[N-3-(4-(2-(1-methyl-5-tetrazolyl)-pyridine-5-yl)-3-fluorophenyl)-2-oxo-5-oxazolidinyl]methyl acetamide (DA-7867) were extracted from human plasma by liquid-liquid extraction and analyzed on a phenyl-hexyl column with the mobile phase of acetonitrile-ammonium formate (10 mM, pH 3.0) (60:40, v/v). The analytes were detected using an electrospray ionization tandem mass spectrometry in the multiple-reaction-monitoring mode. The calibration curve was linear (r = 0.999) over the concentration range of 1.00-500 ng mL^{-1} with the lower limits of quantification of 1.0 ng mL^{-1} using 100 mL plasma sample. The coefficient of variation and relative error of this assay ranged from 2.4 to 7.0 % and from –7.0 to -2.0 %, respectively. The recoveries of eupatilin ranged from 64.3 to 65.0 %, with that of DA-7867 (internal standard) being 87.0 ± 5.3 %.

[PD4-33] [2003-10-10 14:00 - 17:30 / Grand Ballroom Pre-function]

^{1}H-NMR Studies of Chiral Solvating Agent Induced - Chemical Shift Differences of Ibuprofen Enantiomers

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Chiral discrimination of ibuprofen by ^{1}H-NMR using several chiral solvating agents such as (-)-brucine, (-)-cinchonidine, (1R, 2S)-(-)-ephedrine, (S)-(-)-α-methylbenzylamine, (-)-strychnine and L-(-)-tryptophane was investigated. Racemic ibuprofen treated with one equivalent of chiral solvating agent was preferentially crystallized. Chiral purity of each precipitates was measured by chiral HPLC and chemical shift differences(ΔΔδ)

was calculated. Eventhough (S)-(-)-α-methylbenzylamine was most effective for the preferential recrystalization of (S)-(+)-ibuprofen, chemical shift differentiation ability was weak. (-)-Cinchonidine discriminated the methyl, methylene and benzyl protons of (S)-(+)-ibuprofen and (R)-(-)-ibuprofen effectively.

[PD4-34] [2003-10-10 14:00 - 17:30 / Grand Ballroom Pre-function]

High throughput automated 96-well solid-phase extraction and liquid chromatography-tandem mass spectrometric analysis of beraprost in human plasma

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A sensitive and selective liquid chromatographic method coupled with tandem mass spectrometry (LC-MS/MS) was developed for the determination of beraprost in human plasma. Plasma samples were transferred into 96-well OASIS HLB extraction plate using an automated sample handling system and the drugs were eluted with methanol. The eluents were then evaporated and reconstituted with water. All sample transfer and solid-phase extraction (SPE) was automated through the application of both the PerkinElmer MultiPROBE II HT and TOMTEC Quadra 96 workstation. The reconstituted samples were analyzed by a reversed-phase LC-MS/MS using an electrospray ionization (ESI) interface. The mobile phase was composed of 1 mM ammonium acetate and acetonitrile (50:50, pH 5.0), with a flow rate at 0.2 mL/min. The limit of quantification (LOQ) was 20 pg/mL, using a sample volume of 1.5 mL for the analysis. Beraprost produced a protonated precursor ion ($[M-H]^-$) at m/z 397, and a corresponding product ion at m/z 269. Internal standard produced a protonated precursor ion ($[M-H]^-$) at m/z 367 and a corresponding product ion at m/z 249. Based on a signal-to-noise level (S/N) of 10, the limit of quantification for beraprost was found to be 20 pg/mL. This simple, rapid and robust assay will enable the complete processing of large sample for pharmacokinetic studies of beraprost in human plasma.

[PD4-35] [2003-10-10 14:00 - 17:30 / Grand Ballroom Pre-function]

Liquid Chromatographic Resolution of Pyrethroic Acids and Their Esters on Chiral Stationary Phases

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Pyrethroic acids are essential chiral intermediates of the pyrethroids, which account for about 25% of the world insecticide market and are found to be some of the most effective commercially available pesticides. It was demonstrated that polysaccharide-derived chiral stationary phases (CSPs) are very efficient for the separation of the enantiomers of pyrethroid acids. It was observed that the enantioseparation of pyrethroic methyl ester and ethyl derivatives was well accomplished on brush-type WhelkO-1. The observed enantioselectivity is suitable for the determination of enantiomeric purity of pyrethroid acids and their ester derivatives on these CSPs.

[PE1-1] [2003-10-11 09:00 - 12:30 / Grand Ballroom Pre-function]

Skin penetration enhancement of prostaglandin E1 and its ethyl ester for topical formulations

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Purpose. To investigate the effect of different terpene enhancers on skin penetrations of prostaglandin E1 (PGE1) and its ethyl ester (PGE1-EE), a therapeutic agent for erectile dysfunction, external gel systems were formulated with the specific enhancers having different values in their lipophilicity (log P was ranged in 2.23-