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)