

[PD4-3] [04/19/2002 (Fri) 10:00 – 13:00 / Hall E]

Determination of Water in Alcohol by Portable Near Infrared (NIR) System

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In this study, water content in the mixture of ethanol and methanol was nondestructively measured by near infrared (NIR) spectroscopy. Two types of NIR instruments, portable NIR system with a photo-diode array and scanning type NIR spectrometer, were used and the calibration results were compared. Partial least squares regression (PLSR) was applied for the calibration and validation for quantitative analysis of water content. The calibration result from both instruments showed good correlation with actual values. The calibration with the use of PLS model predicted water concentration for validation set with a standard error of prediction (SEP) of 0.097% for photo diode array type-NIR, 0.11% for scanning type-NIR, respectively. Also, during 6 days, routine analyses for 3%, 5% and 7% water in alcohol solution were performed to validate the robustness of the developed calibration model. The routine analyses showed good results with standard deviation of within 0.9% for both types of NIR spectrometers. This study showed that the rapid determination of water in the mixture of ethanol and methanol was successfully performed by NIR spectroscopy and the performance of the portable NIR system with a photo diode array detector was comparable to that of the scanning type-NIR spectrometer.

[PD4-4] [04/19/2002 (Fri) 10:00 – 13:00 / Hall E]

Chiral discrimination of β -blockers by proton nuclear magnetic resonance spectroscopy using (S)-2-tert-butyl-2-methyl-1,3-benzodioxole-4-carboxylic acid

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The use of (S)-2-tert-butyl-2-methyl-1,3-benzodioxole-4-carboxylic acid ((S)-TBMB-COOH) as NMR chiral derivatizing agent to resolve the enantiomers of β -blockers was investigated. Optically pure (S)-TBMB-COCl was synthesized and its CH₃CN solution was successfully used for the chiral discrimination of β -blockers as their diastereomeric (S)-TBMB derivatives. Diastereotopic nonequivalence ¹H-NMR examination of the resultant amides without any racemization and kinetic resolution has proved (S)-TBMB-COOH to be useful, efficient and reliable chiral derivatizing agent for the enantiomeric excess determination of β -blockers.

[PD4-5] [04/19/2002 (Fri) 10:00 – 13:00 / Hall E]

Determination of Ambroxol in Human Plasma by Liquid Chromatography Tandem Mass Spectrometry

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A sensitive and selective method for the determination of ambroxol in human plasma have been developed After the addition of the domperidone (internal standard), ambroxol in human plasma were extracted with diethyl ether under basic condition. Centrifuged upper phase was evaporated and dissolved in methanol. The supernatant was directly introduced into LC/MS/MS. Chromatography was carried out on a C18 Xtera column (2.1X30mm) with a particle size of 3.5 μ m. The mobile phase was 20mM ammonium acetate in 90% acetonitrile(pH 9.0) and the flow rate was 250 μ L/min. The mass spectrometer was operated in positive ion