

[PD4-11] [10/18/2002 (Fri) 13:30 - 16:30 / Hall C]

Chiral separation of β -blockers after derivatization with a new chiral derivatization agent, GATC and Comparison with GITC on derivatizing conditions and chromatographic parameters

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A new chiral derivatization agent with sugar moiety, 2,3,4,6-tetra-O-acetyl-D-galactopyranosyl isothiocyanate (GATC) was synthesized. Several β -blockers were investigated for the possible separation of the enantiomers by reversed-phase HPLC after derivatization with this new chiral derivatization agent (GATC). GATC was reacted readily with β -blockers at room temperature and the reaction mixture could directly be injected into the HPLC system. The corresponding diastereomers were well resolved on an ODS column with acetonitrile-ammonium acetate buffer as a mobile phase and monitored at UV 254nm. Conditions of derivatization (agent excess, temperature and reaction time) were optimized and compared with GITC.

[PD4-12] [10/18/2002 (Fri) 13:30 - 16:30 / Hall C]

Narrowbore high-performance liquid chromatographic method for the determination of cetirizine in plasma using column switching

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A column switching HPLC assay was developed to allow the separation and quantitation of cetirizine in human plasma by ultraviolet (UV) detection. Plasma samples were prepared by liquid-liquid extraction. After drying, the residue was reconstituted in 20 mM phosphate buffer (pH 2.8) containing 15% acetonitrile. The samples were initially injected onto a clean-up Capcell Pak MF C18 column, (50 mm x 4.6 mm I.D.), and the chromatographic region containing the peaks of interest was followed in an analytical C18 microcolumn (250 mm x 1.5 mm I.D.) via column switching device. The separation of analyte was performed using 20 mM phosphate buffer (pH 2.8) containing acetonitrile (65 : 35) and quantified by monitoring absorbance at 230 nm. The limit of detection of cetirizine in plasma was 2 ng/ml, and the assay was validated in the concentration range of 5-400 ng/ml. It has been applied to determine the concentration of cetirizine in plasma from healthy volunteers dosed with cetirizine dihydrochloride tablets.

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

Proficiency Test for Pharmaceutical Companies in Analyzing Drug Products (I) - Comparison of Criteria for Satisfactory Test Results

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Quality control by analytical ability with a certain level of precision and accuracy is important. This is true especially in pharmaceutical industries, for a failure in quality control can result in a failure in drug medication, in turn, sometimes a heavy damage to patient's health condition and/or the worst damage to company's reputation. On this background, Kyungki KFDA prepared test pharmaceutical products, which were distributed to 114 pharmaceutical companies in Kyungin Province in year 2000. The formulations of ibuprofen and sobrerol were prepared in two different levels of concentration, respectively. Only the mode of analysis was specified with the samples. Ibuprofen samples were instructed to be analyzed by HPLC and sobrerol by GC. Any other details in analyzing samples were selected by analytical personnel of each company. The results of test were analyzed according to FAPAS protocol based on ISO/IEC Guide 43-1:1997 E "Proficiency testing by interlaboratory

comparisons" and, in comparison, by Huber's elimination rule based on median absolute deviation (MAD). As a conclusion, FAPAS protocol was influenced by the existence of outliers in data set, for outliers may move mean value toward them and inflate standard deviation resulting in obscure z-scores, while Huber's elimination rule pointed out all of the outlying test results.

[PD4-14] [10/18/2002 (Fri) 13:30 - 16:30 / Hall C]

Chiral discrimination studies of (+)-(18-crown-6)-2,3,11,12-tetracarboxylic acid by NMR spectroscopy

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The chiral stationary phase derived from (+)-(18-crown-6)-2,3,11,12-tetracarboxylic acid (18-C-6-TA) as a chiral selector has been employed for resolution of several α -amino acids in HPLC. In a quest for the origin of chiral recognition of α -amino acids in the presence of 18-C-6-TA as a chiral selector, these interactions responsible for the differential affinities shown toward enantiomers were investigated by NMR spectroscopy. In this study, we have performed detailed NMR studies for each enantiomer of alanine or alanine methyl ester with 18-C-6-TA to investigate the chiral recognition mechanism of these diastereomeric complexes.

[PD4-15] [10/18/2002 (Fri) 13:30 - 16:30 / Hall C]

Chiral Separation of Aromatic Acids by Capillary Electrophoresis Using HP β -Cyclodextrin as the Chiral Selector

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Capillary electrophoretic direct chiral separation method is described for the determination of the absolute configuration of chiral aromatic acids. The enantiomeric separation was achieved by capillary electrophoresis using HP β -cyclodextrin (CD) as the chiral selector. The effect of CD concentration was investigated to optimize the chiral separation and resolution. When applied to microbial culture fluid, the present method allowed positive identification of chiral aromatic acids and their chirality as well.

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

Dissolution Test for Indomethacin by the Portable Near-Infrared(NIR) System

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Near-infrared (NIR) system was used to determine rapidly and simply indomethacin in buffer solution for a dissolution test for tablets and capsules. Indomethacin standards were prepared ranging from 10 to 50ppm using mixture of phosphate buffer(pH 7.2) and water(1:4). The near infrared(NIR) transmittance spectra of indomethacin standard solutions were collected by using a quartz cell in 1mm and 2mm pathlength. Partial least-square regression(PLSR) was explored to develop calibration models over the spectral range 1100-1700nm. The model using 1mm quartz cell was better than that using 2mm quartz cell. The PLSR models developed gave standard errors of prediction(SEP) of 0.858(ppm). In order to validate the developed calibration model, routine analysis were performed using another standard solutions. The NIR routine analyses showed good correlation with actual values. Standard Error of Prediction(SEP) is 1.614(ppm) for 7 indomethacin samples in routine analysis and its error was permeable in the regulation of Korean Pharmacopoeia(VII). These results show the potential use of the real time monitoring for indomethacin during a dissolution test.