High-performance liquid chromatography(HPLC) has become the method of choice for the separation of enantiomers on CSP. This is because of the wide applicability of the method and the speed and efficiency at which these separations can be carried out. Enantiomers of racemic drugs often differ in their pharmacokinetic behaviour and/or pharmacololgical action. β_2-agonists, a sympaticomimetic drug-selective β_2-receptor agonists, are used in the treatment of asthma and lung disease. The drugs are usually administered as a racemate, but studies have shown that only one enantiomer has the desired therapeutic pharmacological effect. For that reason it is of great importance that the enantiomers of such molecules can be separated. Enantiomeric Separation of six closely structure related β2-agonists and the other , have not similar structure, was achieved by direct method that using normal phase HPLC on Chirobiotic T, Chiral AGP, Chiralcel OD, (R,R)Whelk-O1, Chiralcel OJ, Chiralpak OT, Chiralpak CR(+), Chirex (D)Phenicillamine and Resolvosil BSA-7.

[PD4-8] [04/19/2001 (Thr) 13:30 - 14:40 / Hall 4]

EZ staining method for proteins in SDS-PAGE

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A rapid and sensitive staining method for protein in polyacrylamide gel electrophoresis (PAGE) using both an acidic dye, zincon (ZC) and a basic dye, ethyl violet (EV) is described. It is based on a counterion-dye staining technique that employs oppositely charged two dyes to form a ion-pair complex. The selective binding of the dye molecules to proteins in an acidic solution produces bluish violet colored bands. It is a rapid procedure, involving only fixing and staining steps that are completed in 45 min. The sensitivity of this method is 5-10 ng of protein which is four-fold better than that of the conventional Coomassie brilliant blue R-250 (CBBR) staining and is comparable to the sensitivity of silver nitrate staining. Due to its sensitivity and rapidity, this stain may be more practical than anyother dye-based stains for routine laboratory purposes. This staining method can be applied to detect for the trace amount of protein in 2D-PAGE.

[PD4-9] [04/19/2001 (Thr) 13:30 - 14:40 / Hall 4]

Enantioselective stabilization of inclusion complexes of metoprolol in carboxymethylated beta-cyclodextrin

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The inclusion complexes of metoprolol (MT) and carboxymethyl-b-cyclodextrin (CMCD) were prepared and the stability constants of the complexes were determined. Binding studies performed using HPLC, UV spectrometry and capillary electrophoresis (CE) indicated that a complex with 1:1 stoichiometry is predominant in the solution. The enantiomers of MT possess relatively high affinity towards CMCD with stability constants of 286 M-1 and 268 M-1 for (R)- and (S)-MT, respectively. Through NMR analysis the structure of MT was predicted to be a bent conformation with the hydrophobic phenyl ring of MT inserted in the shielding cavity of CMCD during complex formation. The NMR data, furthermore, suggested that the chiral side chain and the methoxyethyl moiety of MT are aligned in the deshielding zone, above and below the CMCD torus ring, respectively.

[PD4-10] [04/19/2001 (Thr) 13:30 - 14:40 / Hall 4]

Simultaneous determination of amphetamine (AM), methamphetamine (MA),

methylenedioxyamphetamine (MDA) and methylenedioxymethamphetamine (MDMA) by GC/MS in Hair

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The abuse of amphetamine's methylenedioxy-derivatives such as 3,4-methylenedioxyamphetamine (MDA) and 3,4-dimethylenedioxymethamphetamine (MDMA) has increased recently in Korea. In this study, a method is investigated for the simultaneous determination of amphetamine (AM), methamphetamine (MA), MDA and MDMA in hair. To extraction of drugs, fine cutted hair sample was incubated with MeOH (1% HCl) overnight (18 hrs) while stirring. For GC/MS analysis, the extract was evaporated and derivatized with trifluoroacetic anhydride / EtOAc and applied to Hewlette Packard 5973 MSD with selective ion monitoring (SIM) mode.
Quantification of AM, MA, MDA and MDMA in hair sample were based on peak area ratio to their internal standards, like as AM-d_5, MA-d_5, MDA-d_5 and MDMA-d_5, respectively. Selected ions at m/z 140 for AM, m/z 144 for AM-d_5, m/z 154 for MA and MDMA, m/z 158 for MA-d_5 and MDMA-d_5, m/z 162 for MDA and m/z 167 for MDA-d_5 were used in this analysis.

[PD4-11] [04/19/2001 (Thr) 13:30 - 14:40 / Hall 4]

Studies on the Quality Evaluation of Pharmaceuticals (III) - Comparative Analysis of Pyrogen and Endotoxin Test in Amino acid injections

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Limulus Amebocyte Lysate(LAL) test (endotoxin test) is supposed to be a alternative to the rabbit pyrogen test in that the former is more convenient, specific and inexpensive. To compare the LAL test with the rabbit pyrogen test, we prepared spiked samples of 5 pharmaceutical amino acid injections with concentration of 0.25, 0.5, 1.0 EU/mL and tested those by pyrogen and endotoxin test simultaneously.
The LAL test was accomplished by using 2 different methods, gel-clot method and kinetic turbidimetric method and the pyrogen test was accomplished by using KP official pyrogen test method. In our results, the LAL test was about 15.8 times more sensitive than the rabbit pyrogen test in the case of gel-clot method and about 97.3 times more sensitive than the rabbit pyrogen test in the case of kinetic turbidimetric method. The amounts of endotoxin in 5 amino acid injections estimated by the LAL test was well recovered and correlated with the rise of body temperature in rabbit pyrogen test. These results suggest that the LAL test could be used as an alternative method for the rabbit pyrogen test to examined 5 amino acid injections.

[PD4-12] [04/19/2001 (Thr) 13:30 - 14:40 / Hall 4]

Mass Spectrometric Study of cyclofenil and its metabolites in human urine

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