ANALYTICAL APPLICATIONS BY USING NEW DEVELOPED PORTABLE NEAR INFRARED (NIR) SYSTEM

Ahn JW, Kang NR, Lim HL, Woo YA, Kim HJ

College of pharmacy, Dongduk Women's University, Seoul 136-714, Korea

A compact and handheld near infrared (NIR) system using microspectrometer was developed. This system was suitable not only in the laboratory, but also in the field or in the process. This system was first applied for classification of geographical origin of herbal medicine such as ginseng and sesame. To identify the origin of ginseng on site, the portable NIR system is more suitable for real field application. For this study, using the compact NIR system, soft independent modeling of class analogies (SIMCA) with 1100-1750 nm NIR spectra was utilized for classification of geographical origin (Korea and China) of both ginseng and sesame. The accuracy of results is more than 90%. Quantitative analysis for petroleum such as toluene, benzene, tri-methyl benzene, and ethyl benzene was performed with partial least squares (PLS) regression with NIR 1100-1750 nm spectra. This study showed that the NIR method and gas chromatography (GC), which is a standard method, have good correlations. Furthermore, the sweetness of fruit was analyzed and the accuracy was confirmed by the developed compact NIR system.

[PD4-6] [04/19/2001 (Thr) 13:30 - 14:40 / Hall 4]

Studies on the Quality Evaluation of Pharmaceuticals (III) - Method Validation of Endotoxin Test in Amino acid injections

Kim HS°, Lee SD, Ze KR, Kim HS°, Kim MJ, Jang SH, Jung KS, Lee YH, Jung HY and Jang SJ

Division of Drug Chemistry, Department of Drug Evaluation, KFDA

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. We applied the LAL test to the detection of bacterial endotoxins in 5 pharmaceutical amino acid injections using gel-clot method and kinetic turbidimetric method and validated the methods by investigating LAL reagent sensitivity, interferences, calibration curve, reproducibility and recovery.
The determined LAL reagent sensitivity was 0.060 EU/mL and the calibration curve of endotoxin standard solutions by kinetic turbidimetric method was linear over the entire range from 0.0078125 to 50 EU/mL. The linear regression coefficient of determination was 0.998 and the limit of detection was 0.005 EU/mL. In all 5 injections, the amount of endotoxin estimated by the LAL test (gel-clot method and kinetic turbidimetric method) was well recovered and there are no significant inteference (both enhancement and inhibition) factors. These results suggest that the LAL test was useful method for quantitative estimation of endotoxin, the probable major cause of pyrogenicity and expected for the substitutive method for pyrogen test in examined amino acid injections by applying criteria of not more than 0.2 EU/mg amino acid.

[PD4-7] [04/19/2001 (Thr) 13:30 - 14:40 / Hall 4]

Enantiomeric Resolution of β₂-agonists on several Chiral Stationary Phases

Choe HK, Lee BC, Kang DI, Kim SG

Central Research Laboratory, R&D Division, Choongwae Pharma Co., PO Box 61, Suwon, South Korea

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

Choi JK, Yang YI, Lee JK, Yoo GS

College of Pharmacy, Chonnam National University, Kwangju 500-757, South Korea

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

Park KL, Kim KH, Jung SH, Lim HM°, Hong JH, Kang JS

College of Pharmacy, Chungnam National University and Kangwon National University

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),