[PD4-1] [ 10/19/2001 (Fri) 09:00 - 12:00 / Hall D ]

## Analysis of Amino acids in Multiamino acid Infusion by HPLC

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This study was analyzed amino acids in multiamino acid infusions by high performance liquid chromatography. A rapid and simultaneous determination of amino acid was performed by two method. One was the dabsyl derivtives method using μ-Bondapak C18 with visible detector (440nm) and the other was the o-phthaldialdehyde(OPA) derivatives method using Novapack C18 with fluoresence detector(EX 324, EM 425). Amino acids were successfully separated within 30 minutes.
The result was as follows, In calibration curve of Dabsyl derivatives of amino acid, linearity was greater than 0.995. Their recovery rates were greater than 85%. In case of OPA derivatives of amino acids, linearity was greater than 0.997. Their Recovery rates were greater than 85%.

[PD4-2] [ 10/19/2001 (Fri) 09:00 - 12:00 / Hall D ]

## Comparision of enantiomeric resolution on chiral stationary phases derived from crown ethers

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A new HPLC chiral stationary phase (CSP) prepared by bonding (+)-(18-crown-6)-2,3,11,12-tetracarboxylic acid to silica gel has been developed and employed for enantioresolution of various racemic compounds containing a primary amino group. In this study, this CSP developed in our group is compared to a commercially available Crownpak CR CSP derived from chiral crown ether.

[PD4-3] [ 10/19/2001 (Fri) 09:00 - 12:00 / Hall D ]

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[PD4-4] [ 10/19/2001 (Fri) 09:00 - 12:00 / Hall D ]

## (+)-(18-Crown-6)-2,3,11,12-tetracarboxylic acid: A new chiral solvating agent for NMR spectroscopy

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We recently reported that a new HPLC chiral stationary phase prepared by bonding (+)-(18-crown-6)-2,3,11,12-tetracarboxylic acid (18-C-6-TA) has been successfully employed in resolving various racemic compounds containing a primary amino functional group. Related to these results, in this study it is shown that the chiral selector 18-C-6-TA could be used as a chiral solvating agent for resolution of amino acids and their methyl esters by NMR spectroscopy. It was observed that the chemical shift changes of the alpha protons of these analytes were sufficiently large enough to determine the ratio of the enantiomers (0.2-0.3ppm).

[PD4-5] [ 10/19/2001 (Fri) 09:00 - 12:00 / Hall D ]

## Determination of Silibinin in human plasma by high performance liquid chromatography: validation and application in pharmacokinetic study

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A reversed phase high performance liquid chromatographic method for the determination of silibinin diastereomers in human plasma is described. The method was applied to the assay of total (free and conjugated) silibinin levels in healthy volunteers after oral administration. Total silibinin diastereomers were hydrolysed enzymatically using b-glucuronidase/sulfatase at pH 5.0 followed by extraction with diethyl ether of the pH 8.5 alkalinized solution. The diastereomers and the internal standard naringenin were seperated on a Lichrospher 100 C18 column (250mm´4mm I.D.) with ultraviolet detection at 288 nm. As the mobile phase, 40~60% acetonitrile in 2% formic acid by gradient elution was employed for the separation. Inter-day and intra-day precision were less than 6% and 4%(CV), respectively. The accuracy were 96~112% and the recovery were 60~70% for silibinin The calibration curve was linear over a concentration range of 20~2000 ng/ml. The method is relatively simple, sensitive, and satisfactory for the analysis of silibinin in plasma samples from pharmacokinetic study in humans.

[PD4-6] [ 10/19/2001 (Fri) 09:00 - 12:00 / Hall D ]

## Analysis of Furathiocarb and its Main Metabolite Carbofuran of Postmortem Specimens in a Fatal Case

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A fatal poisoning(suicide) by the insecticide furathiocarb is described. A 35-year-old man was found dead lying flat on the floor of the boiler room in a deserted house. A farmer found him dead and reported to the police. The public prosecutor ordered to examine the cause of death closely. The corpse was sended to Western District Office, National Institute of Scientific Investigation and the autopsy was performed. Besides the color of the gastric contents was milk-white, the autopsy findings were unremarkable. The following postmortem samples were taken for toxicological investigations: blood, gastric contents, spleen, kidney etc. The analytes in postmortem specimens obtained at autopsy were extracted by liquid-liquid extraction with ethyl acetate. After the ethyl acetate layer was evaporated, the residue was partitioned into hexane and acetonitrile, and the hexane layer was discarded to remove lipophyllic impurities. The acetonitrile layer was analyzed. Tissue specimens were homogenized with distilled water, deproteined with 10% trichloroacetic acid and applied for liquid-liquid extraction. After