Simultaneous Gas Chromatographic Analysis of Amino Acids and Organic Acids

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N(O,S)-ethoxycarbonylation combined with tert.-butyldimethyl- silylation was optimized and validated for the simultaneous gas chromatographic (GC) analysis of amino acids and organic acids. Ethoxycarbonlyation of amino, phenolic and sufhydry groups with ethyl chlorofomate in aqueous solution was followed by tert.-butyldimethylsilylation of carboxyl and remaining polar groups for the direct GC analysis after solvent extraction. The present method was found to be potentially useful for the biochemical diagnosis of inherited metabolic disorders.

[PD4-20] [10/19/2001 (Fri) 09:00 - 12:00 / Hall D]

Simultaneous Quantitative Analysis of Sphingoid Base 1-Phosphates in Biological Samples by o-Phthalaldehyde Precolumn Derivatization after Dephosphorylation with Alkaline Phosphatase

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This paper describes a simultaneous analytical method for the measurement of sphingoid base 1-phosphates and sphingoid bases from a variety of biological samples. This method consists of two steps of sample pretreatment: the enzymatic dephosphorylation of sphingoid base 1-phosphates by alkaline phosphatase and the subsequent analysis of OPA derivatives of the liberated sphingoid bases by HPLC. By introducing C_{17}-sphingosine 1-phosphate and C_{17}-sphingosine as internal standards, not only phytosphingosine 1-phosphate, sphingosine 1-phosphate, and sphinganine 1-phosphate but also phytosphingosine, sphingosine, and sphinganine present in a sample could be quantified in 12 min on a C_{18} reversed-phase column with a simple mobile phase of acetonitrile : water (90 : 10, v/v). With this HPLC method, we could reproducibly analyze the levels of sphingoid base 1-phosphates over a broad range of concentrations from 0.5 to 100.0 pmol from various biological samples including serum, cultured cells and rat tissue homogenates. The conversion of sphingoid base 1-phosphates into sphingoid bases increased the stability of the OPA adducts. Thus, this indirect measurement of sphingoid base 1-phosphates increased the sensitivity and reproducibility of the method. This HPLC method was also used to measure the changes in the levels of sphingoid base 1-phosphates in cultured cells
after treatment with 1,25-$(OH)_2D_3$, a sphingosine kinase activator, or with fumonisin B_1, a sphinganine N-acyltranferase inhibitor.

[PD4-21] [10/19/2001 (Fri) 09:00 - 12:00 / Hall D]

Diagnostic Patterns for Capillary Electrophoretic Urinary Nucleoside Profiles from Patients with Liver Diseases

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An efficient capillary electrophoretic profiling method in micellar electrokinetic capillary chromatography (MEKC) mode was combined with simple pattern recognition methods for the correlation between urinary

nucleosides and liver disease. For the urinary nucleoside profiles, free nucleosides were extracted by SPE in affinity mode with the subsequent analysis by MEKC. A total of 15 nucleosides were positively identified in urine samples from patients with liver diseases and normal subjects studied. Multivariate statistical analyses appropriate for the correlation between urinary nucleosides profiles and liver diseases was investigated. Canonical discrimination analysis applied to the 15 nucleosides correctly classified each urine specimen into each separate cluster according to the disease type. Star patterns were very informative in comparing the abnormal group with normal control group. The present nucleoside profiling and simple pattern recognition methods appear to be useful for the comparative analysis of urinary nucleosides among groups of normal subjects and patients with liver disease.

[PD4-22] [10/19/2001 (Fri) 09:00 - 12:00 / Hall D]

GC-SIM-MS and GC-Dual ECD Profiling and Screening Analysis of Trace Acidic and Neutral Pollutants from Water Samples

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34 trace pollutants including phenols, organic acids, polyaromatic hydrocarbons, alcohol and esters in water samples were recovered by solid-phase extraction in static adsorption mode using XAD-4 resin as the adsorbent with subsequent modified Soxhlet extraction using dichloromethane containing acetonitrile as the solvent. The extracts were evaporated with subsequent tert.-butyldimethylsilylation for the direct analysis by gas chromatography-mass spectrometry (GC-MS) in selected ion monitoring (SIM) mode and GC-dual electron capture detector (ECD) system on dual-columns of different polarity. Validation of the GC-SIM-MS and GC-ECD methods for the simultaneous profiling analysis of pollutants will be compared.

[PD4-23] [10/19/2001 (Fri) 09:00 - 12:00 / Hall D]

Residue and risk assessment of polychlorinated dibenzo-p-dioxins and dibenzofurans in the Korean population.

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Polychlorinated dibenzo-p-dioxins and dibenzofurans(PCDDs/DFs) are ubiquitous contaminants in the global environment. These compounds are detected in high concentrations in human tissues from developed and industrialized countries. However, the status of PCDDs/DFs contamination is not so clear in the general Korean population. In this study, we examined the contamination levels and characterization of these toxic compounds in human adipose tissue and human milk collected from Korea.

The human adipose and milk samples analysed in this study were collected at hospitals in Seoul, Masan and Jinju. The mean values of International Dioxin Toxic Equivalents (I-TEQ) of PCDDs and PCDFs in human adipose samples from the three regions were calculated as 9 pg TEQ/g(0.2 ~ 30 pg TEQ/g, lipid weight basis) and 8 pg TEQ/g (0.8 ~ 25 pg TEQ/g), respectively. The residue levels of PCDDs-TEQ and PCDFs-TEQ in human milk collected from Masan were 13 pg TEQ/g (lipid wt.) and 4.8 pg TEQ/g, respectively. On the whole, the contamination levels of these compounds in the Korean population were lower than those in the other countries.
Based on the analytical data and assuming a daily intake consumption of 800mL milk with 3% fat, the