

extraction, the extracts were reconstituted in methanol for GC and GC/MS. Furathiocarb and its main metabolite carbofuran was detected in the gastric contents by TLC, GC and GC/MS, and quantitated in the blood and postmortem tissues (kidney, spleen etc) using GC/NPD.

[PD4-7] [ 10/19/2001 (Fri) 09:00 - 12:00 / Hall D ]

### Identification of Gamma-Hydroxybutyrate(GHB) in Seized materials and Urine samples.

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Gamma Hydroxybutyrate (GHB), known as liquid ecstasy, liquid x or Georgia home boy, is a central nervous system depressant abused for its ability to produce euphoric and hallucinatory states and its alleged ability to release a growth hormone and stimulate muscle growth. GHB can produce drowsiness, dizziness, nausea, unconsciousness, seizures, severe respiratory depression, and coma. It is taken orally and is frequently combined with alcohol and often used as "Date-Rape" drug. In recent days, liquid form of GHB solution and abuser's urine was received to our laboratory for analysis of the drug. The solution was acidified with 10%-HCl and extracted with ethyl acetate and evaporate to dryness at 48°C under the nitrogen. The residue was derivatized with BSTFA and injected into GC/MSD. For the analysis and quantitation of urine sample, GHB-d6 was used for internal standard. Urine sample was solid phase extracted using CLEAN SCREEN ZSGHB020 column and also derivatized and injected into GC/MSD. The full scan mass spectrum of GHB-TMS identifies the following ions in order of abundance m/z 147, 233, 117, 158, 148 and 149. For detection of GHB in urine samples, the mass selective detector was run in selected ion monitoring mode (SIM) and GHB was not detected in urine samples.

[PD4-8] [ 10/19/2001 (Fri) 09:00 - 12:00 / Hall D ]

### High Performance Liquid Chromatography / Electrospray Tandem Mass Spectrometric Method for Quantitation of Lovastatin Acid in Human Plasma

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Lovastatin acid, the major active metabolite of lovastatin in human blood, was analysed by high performance liquid chromatography /electrospray tandem mass spectrometry(LC/MS/MS). This method utilized a solid phase extraction(SPE) procedure for purifying and concentrating the lovastatin acid and internal standard, simvastatin acid. Reversed-phase microbore(15cm X 2.1 mm) column was used for chromatography and the flow rate was 0.2 ml/min. Tandem mass spectrometry was operated in multiple-reaction-monitoring(MRM) mode with a unit mass resolution on both mass analyzers. For quantitation in the MRM mode, the precursor → product ions monitored in the negative-ion mode were m/z 421.3 → 101 (lovastatin acid) and m/z 435.3 → 115 (simvastatin acid). The assay was linear in the concentration range 0.5-50 ng/ml for lovastatin acid when 1 ml aliquots of plasma was extracted. Detection and quantitation limits were 100pg/ml and 500pg/ml in human plasma respectively. The accuracy, intra-day and inter-day precision as determined from QC samples were less than 10%, 4% and 15% respectively. This method was applied to the analysis of clinical samples from a bioequivalence study of lovastatin preparation.

[PD4-9] [ 10/19/2001 (Fri) 09:00 - 12:00 / Hall D ]

### Determination of Acidic Drugs with Metal-Complex based Ion Selective Electrodes.

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A poly(vinylchloride) membrane ion-selective electrode for determining the acidic drugs is described. The sensing membrane of the electrode consists of acidic drug-metal(II)-dipyridylketone oxime as an ion-exchanger site in a PVC matrix plasticized with nitrophenyl ether group. In a borate buffer solutions of pH 8.9, the electrode exhibits a fast, stable and linear response for  $2 \times 10^{-5} \sim 10^{-2}$  mol dm<sup>-3</sup> acidic drug with an anionic slope of near 55 mV decade<sup>-1</sup>. Potentiometric selectivity measurements revealed negligible interferences from several different anions. The electrode displays useful analytical characteristics for the direct determination of acidic drugs such as fenemates, ibuprofen, naproxen and diclofenac in pure form and in pharmaceutical preparations.

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

### **Novel chiral derivatizing agent, (+)-2,4-dimethyl-1,3-benzodioxole-2-acetic acid**

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A carboxylic acid with a 1,3-benzodioxole skeleton, (+)-2,4-dimethyl-1,3-benzodioxole-2-acetic acid ((+)-DMBA) was prepared. The potential as a chiral derivatizing agent was evaluated in terms of the HPLC analysis of (±)-methylbenzylamine. With more than 50 times molar excess of (+)-DMBA chiral derivatization reaction was completed within one hour at 70°C. Diastereomeric derivatives of (±)-methylbenzyl amine were well resolved on the silica column using n-hexane-ethyl acetate as a mobile phase.

[PD4-11] [ 10/19/2001 (Fri) 09:00 - 12:00 / Hall D ]

### **Chiral derivatization of (±)-methyl benzylamine with (+)-2-t-butyl-2-methyl-1,3-benzodioxole-4-carboxylic acid as a chiral derivatizing agent**

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The applicability of (+)-2-t-butyl-2-methyl-1,3-benzodioxole-4-carboxylic acid ((+)-TBMB) as a chiral derivatizing agent for the enantioseparation of a compound containing an amino group such as (±)-methyl benzylamine and some β-blockers was investigated. Diastereomeric derivatives were prepared and confirmed by NMR and Mass. Diastereomeric derivatives were analyzed by normal phase high-performance liquid chromatography. Condition of derivatization (temperature, reagent excess and reaction time) were optimized and compared to each other.

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

### **Chiral Analysis of Baclofen by Capillary Electrophoresis using Highly Sulfated γ-Cyclodextrins as Chiral Selectors**