and injected for GCMS including achiral stationary phase. In the derivatization procedure, Both racemates were silylated and formed diastereomeric cerivatives.
As a result, It was possible to simultaneous enantioseparate both racemates by GCMS even with achiral capillary column. Compared with that of chiral HPLC, This method was found to give a better resolution and sensitivity. Furthermore, The GCMS system allows us to determinate the trace amount of it by using a SIM (single ion monitoring) mode.

[PD4-11] [04/19/2002 (Fri) 10:00 - 13:00 / Hall E]

Rapid analysis of tizanidine in human plasma by gas chromatography/mass spectrometry

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An efficient gas chromatography-mass spectrometry (GC-MS) method has been developed and validated for the quantitative determination of tizanidine in human plasma. Plasma samples were simply extracted with ethyl acetate at basic pH and the extracts were converted into trimethylsilyl (TMS) derivatives for the direct separation by GC-MS with the selected ion monitoring (SIM) mode. Reaction of tizanidine with N-methyl-N-(trimethylsilyl)trifluoroacetamide (MSTFA) caused di-trimethylsilylation in imidazoline moiety and this silylation significantly improved the chromatographic properties of the compound. The determination of tizanidine was accurate and reproducible, with a limit of quantitation of 0.5 ng/ml in plasma. The standard calibration curve for tizanidine was linear (r2 = 0.999) over the concentration range 0.5-10.0 ng/ml in human plasma. The intra- and inter-day precision over the concentration range of tizanidine was well within the 6.9% (relative standard deviation, RSD) and accuracy was between 99.2 and 110.5%.

[PD4-12] [04/19/2002 (Fri) 10:00 - 13:00 / Hall E]

Determination of some Acidic Drugs with Ion-Selective Membrane Electrode

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A sensitive poly(vinyl chloride) membrane electrode for determining the acidic drugs is described. The sensing membrane of the electrode consists of acidic drug-metal ion(II)-di-2-pyridyl ketone ternary complex as an ion-exchanger and o-nitrophenyl ether group as a plasticizer.
It shows a linear response towards mefenamate ion and ibuprofen anion over the concentration range 1× 10-2 ~ 5×10-5 mol L-1 with an anion slope of -56.3 and 54.2 ㎷ decade-1 in pH 8.9 buffer and pH 5 buffer solution respectively.
The behavior of the electrode is considerably influenced by the plasticizer employed and the optimum response appears to result when benzyl-2-nitrophenyl ether is present.
The electrode was applied to the determination of mefenamic acid and ibuprofen in pure form and in pharmaceutical preparations.

[PD4-13] [04/19/2002 (Fri) 10:00 - 13:00 / Hall E]

Postmortem Blood Concentration of Levomepromazine, Chlorpromazine, Flurazepam, Tramadol, Benztropine and Caroverine in a Case

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This paper presents a case related to levomepromazine, chlorpromazine, flurazepam, tramadol, benztropine

caroverine etc. A 33-year-old female who was in the hospital treatment was found lying in the rest room of a neurological hospital. The public prosecutor ordered to examine the cause of death closely. The corpse was sent to Western District Office, National Institute of Scientific Investigation and the autopsy was performed. We received the postmortem blood and gastric contents obtained at autopsy for toxicological investigations, together with the medical prescription and her prescribed drugs. The analytes were extracted by back extraction with ethyl acetate. After extraction, the extracts were reconstituted 50㎕ dextromethorphan (IS, 100㎍/㎖ in methanol). Levomepromazine, chlorpromazine, flurazepam, tramadol, benztropine and caroverine were detected in gastric contents and blood by GC/MS and quantitated in the blood using GC. These drugs were consistent with the medical prescription, and also detected in her prescribed drugs. The quantitative contents in postmortem blood were levomepromazine 0.92㎍/㎖, chlorpromazine 0.38㎍/㎖, flurazepam 0.23㎍/㎖, tramadol 0.30㎍/㎖ and benztropine 0.26㎍/㎖, caroverine 0.29㎍/㎖, respectively.

[PD4-14] [04/19/2002 (Fri) 10:00 - 13:00 / Hall E]

Determination of Hydrogen Peroxide Concentration by Portable Near-Infrared (NIR) System

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This experiment was carried out to determine the hydrogen peroxide concentration of 3% antiseptic hydrogen peroxide solutions. Hydrogen peroxide standards were prepared over the range of 0 to 25% concentration and the near infrared (NIR) spectra for hydrogen peroxide standard solutions were collected through a quartz cell in 1mm pathlength. Partial least square(PLS) regression was explored to develop a calibration model over the spectral range 1100-1750nm. We found the variation of absorbance band due to OH vibration of hydrogen peroxide depending on the concentration change around 1400nm. The calibration showed good results with a standard error of prediction(SEP) of 0.18%. In order to validate the developed calibration model, routine analyses were performed using newly prepared standard samples and commercial antiseptic hydrogen peroxide solutions. The hydrogen peroxide values from the NIR calibration model were compared with the values for a redox titration method. Results of the NIR routine analyses showed good correlation with those of the reference method, the redox titration. This study showed that the determination of hydrogen peroxide in the antiseptic solution was successfully performed by portable NIR system without very harmful solvents.

[PD4-15] [04/19/2002 (Fri) 10:00 - 13:00 / Hall E]

Enantiospecific Determination of Ibuprofen by HPLC

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Achiral-chiral HPLC method has been developed for the stereo-specific analysis of ibuprofen. Achiral analysis was carried out using a Novapak C18 (4.6mmx 250mm, 5㎛) column with acetonitrile/water/acetic acid/triethylamine(55:45:0.1:0.02) at a flow-rate of 1.0ml/min. Diastereomers of ibuprofen were detected at 232nm. Separation is based on the resolution of the diastereomeric amides formed on reaction of the ibuprofen enantiomers with (S)-(-)-and (R)-(+)-1-(1-naphthyl)ethylamine(NEA) in the presence of ethylchloroformate. The standard calibration curve of each ibuprofen diastereomers showed good linearity from 0 upto 50.2㎍/㎖ (R=0.9976, S-form, R=0.9981, R-form). R/S ratio of standard solutions was 1.01± 0.01.
Chiral analysis was carried out using a (R,R)-Welk-O1 (4.6mmx 250mm, 5㎛) column with hexane/isopropanol/acetic acid (98:2:0.5) at a flow-rate of 0.9ml/min. Detection of the enantiomers was successful at 220nm without pre-column derivatization. Same method was applied to the determination of ibuprofen tablets containing racemic mixtures. Standard ibuprofen racemic mixture showed linear correlation up to 51.0㎍/㎖, and the R value was 0.9993 for S-ibuprofen, 0.9993 for R-ibuprofen, respectively. And the R/S ratio of commercially available ibuprofen tablets determinated by chiral column method was 0.972~ 1.049.