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.

We could reduce the total analysis time to half by using chiral column, which has a capacity to separate chiral mixture without pre-column derivatization. The convenience and simplicity of this method will sufficiently compensate the high cost of chiral column.

[PD4-16] [04/19/2002 (Fri) 10:00 - 13:00 / Hall E]

Homogeneity test for proficiency testing samples, Water Soluble Multi-vitamin Preparations

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Quality Assurance is essential to ensure the same quality of products and one important means to assure Quality Assurance would be for laboratories to participate in interlaboratory proficiency testing schemes. Proficiency testing provides a means of comparing the Quality Assurance performance of the analyst and method of choice and also provides a chance to find a cause of error and to improve an accuracy and precision of quality analysis within a strictly confidential framework. To improve the analytical confidence of six regional agencies of KFDA, we organized proficiency testing program of water soluble multi-vitamin preparation and test samples were prepared as 6 multi-vitamin solutions containing 3 unknown species of water soluble vitamins. Each test sample was randomly selected and analysed to verify the homogenicity of sample preparations prior to distribution of them to test agencies because homogenicity of sample preparations was essential to ensure proper evaluation of proficiency test results of participating laboratories. Homogenicity test was performed by analysing content of each vitamin contained in 15 random sample of each prepared multi-vitamin solution and analysis of vitamin was accomplished by using high performance liquid chromatographic method. We statistically analysed the assay results of vitamins in 6 test samples by one-way ANOVA and calculated Ss/σ based on 「The International harmonized protocol for the proficiency testing of analytical laboratories (IUPAC, ISO & AOAC 1991)」 to estimate homogeneity of samples. The calcurated mean Ss/σ values of 3 vitamins in 6 multi-vitamin solutions were 0.148, 0.153, 0.291, 0.273, 0.128 and 0.234, respectively, and all values were not more than 0.3, the critical value of confirming satisfactory homogeneity. As a result, we assumed that homogenicity of all 6 multi-vitamin solutions was established and those solutions were appropriate as proficiency testing samples.

[PD4-17] [04/19/2002 (Fri) 10:00 - 13:00 / Hall E]

Rapid Analysis of Vancomycin in Human Plasma by Liquid Chromatography Tandem Mass Spectrometry

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A novel liquid chromatography-tandem mass spectrometric (LC/MS/MS) method is described for the determination of vancomycin in human plasma. After the addition of the caffeine (internal standard), the sample preparation involved only protein precipitation and centrifugation. The supernatant was directly introduced into LC/MS/MS. Chromatography was carried out on a C18 Xterra column(2.1X30mm) with a particle size of 3.5μm. The mobile phase was 0.25% formic acid in 10% acetonitrile and the flow rate was 250μL/min. The mass spectrometer was operated in positive ion mode using the electrospray ionization source maintained at 400℃. Nitrogen was used as the nebulizer, curtain, collision and auxiliary gas. Vancomycin and caffeine were detected by MS/MS using multiple reaction monitoring(MRM). Vancomycin gave a predominant doubly protonated parent molecule([M+2H]2+) at m/z 725 and a corresponding product ion of m/z 100. Detection of vancomycin was accurate and precise, with a limit of detection of 1nM in plasma. The calibration curve for vancomycin in human plasma was linear in a concentration range of 10nM~100μM for plasma. This method has been successfully applied to determined the concentration of vancomycin in human plasma from pharmacokinetic and relative studies.