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.