concentrated to dry residue and reconstituted in ethyl acetate for direct analyses by capillary column gas chromatography. Chiral amidation was optimized and validated for the simultaneous assay of multi NSAID enantiomers in a single run. The present method will be useful for the simultaneous chiral separation of commercial products such as ibuprofen, naproxen and fenoprofen.

[PD4-20] [04/18/2003 (Fri) 13:30 - 16:30 / Hall P]

Studies on the quantification of sobrerol by high-performance liquid chromatography

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This study was designed to develop and validate an isocratic reversed phase high performance liquid chromatographic(HPLC) method for the quantitation of sobrerol in drug preparations, and obtain the data pool that can be used in the revision of pharmacopoeia.
The separation of sobrerol and the other compounds (S-carboxymethylcysteine, acetaminophen, methyl paraben, propyl paraben, and sobrerol degradants) was achieved in a C18 column with an acetonitrile-methanol-water (24.5:10.5:65.0) mobile phase. The detection was performed at 200nm. The linearity of peak area responses versus concentrations was demonstrated from 20 ~ 60 ㎍/㎖ of sobrerol by a correlation coefficient of 0.9995. The limit of detection (LOD) and quantitation(LOQ) was 0.6㎍/㎖ and 2㎍/㎖, respectively. Recovery from excipients was 100.8% (drug substance), 101.1%(capsule), and 99.5%(syrup), respectively. The precision of this method showed RSD of 0.9%(drug substance), 1.9% (capsule), and 1.6% (syrup), respectively. According to a recovery and precision study, this method was shown to be quantitative, and sobrerol in analytical solutions was stable for 1 day. A comparison study of HPLC and GC showed that validation parameters were similar to each other except the range of linearity.

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Sensitive Determination of Felodipine in Human Plasma by LC-MS/MS

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This study established a highly sensitive novel quantification method for detecting felodipine in human plasma using LC-MS/MS. The mobile phase used after degassing was composed of 1 mM ammonium acetate and acetonitrile (20:80, pH 6.0), with flow rate of 200 uL/min. One mL plasma were pipetted into glass tubes and spiked with 0.1 mL of internal standard solution. After adding 6 mL of diethyl ether, the plasma sample was then shacked for 15 min. A centrifuged upper layer was then evaporated and reconstituted with 120 uL mobile phase and 20 uL of sample were injected into LC-MS/MS. Felodipine produced a protonated precursor ion ([M+H]+) at m/z 384 with a major product ion at m/z 338. On the other hand, internal standard produced a protonated precursor ion ([M+H]+) at m/z 347 with a major product ion at m/z 315. Based on a signal-to-noise level (S/N) of 9-10, the limit of quantification for felodipine was found to be 0.05 ng/mL. Validation experiments have shown that the assay has good precision and accuracy over a wide concentration range. This method is not only selective and reliable also faster and simpler compared to other recently reported methods. Likewise, this method has been successfully applied to preliminary pharmacokinetic studies.