Chromatographic chiral resolution of several racemic drugs containing primary amino moiety on a chiral stationary phase

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A chiral stationary phase (CSP) prepared by bonding (18-crown-6)-2,3,11,12-tetracarboxylic acid (18-C-6-TA) to aminopropyl silica gel by HPLC was used in resolving several racemic drugs containing primary amino moiety. Most compounds used in this study were resolved on the CSP using 80% methanol in water (V/V) containing 10mM sulfuric acid as a mobile phase. These results on the CSP were compared to those on the similar CSP derived from 18-C-6-TA of the same chiral selector by different connecting method.

[PD4-11] [04/18/2003 (Fri) 13:30 - 16:30 / Hall P]

Quantification of intact ambroxol tablet using near-infrared spectroscopy

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NIR reflectance spectroscopy, using a fiber-optic probe was used to determine rapidly and non-destructively the content of ambroxol in intact ambroxol 30 mg (nominal content 12.5% m/m ambroxol) tablets by collecting NIR spectra in range 1100 ~ 1750 nm and using PLSR calibration method. The tablets (10.3 ~ 15.9% m/m ambroxol, i.e., 82 ~ 127% of the nominal label content) were used 7 calibration set and 5 validation set. Unique spectral features of the active constituent (ambroxol) were identified i! n the NIR spectra of the tablet ingredients. The developed NIR method gave results comparable to the values from preparation of tablets, SEC and SEP being 0.49% and 0.49% m/m respectively. The method showed good accuracy and repeatability but bad intermediate precision. NIR spectroscopic determination in intact tablets allowed the potential use of the method on-line for real time monitoring of a running production process.

[PD4-12] [04/18/2003 (Fri) 13:30 - 16:30 / Hall P]

Determination of dextromethorphan and its metabolite dextrorphan in human urine by High-performance liquid chromatography

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A simple and accurate reverse-phase high performance liquid chromatography (HPLC) coupled with photodiode array was developed for the determination of dextromethorphan(DM) and its metabolite dextrorphan(DX) in human urine. Chromatographic separation was accomplished on a cyano analytical column at 220 nm using a mobile phase containing 25 mM triethylammonium phosphate buffer(pH 3.0) in a 0-70% ACN gradient and triazolam(TZ) was used as internal standard (I.S.). There was a linear relationship between peak area ratios of analytes to I.S, and concentration of analytes over the concentration range 10-200 ㎍/㎖ for DM and DX with r value of 0.9962 and 0.9958 respectively. The urinary recovery was 92.69~96.79 % (R.S.D. 2.28~4.03 %) for DM and 81.01~84.19 (R.S.D. 2.30~3.08 %) for DX. The limits of detection(LOD) were

600 ng for DM and 300 ng for DX. Dextromethorphan and dextrorphan concentrations in human urine were quantified after hydrolysis. To compare the effectiveness of hydrolysis by enzyme and acid, specimens were hydrolyzed by two method and quantification was performed. As a result, the yield of dextrorphan by enzyme hydrolysis was higher than acidic hydrolysis.

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

A study of test method for impurities(related compounds) in pharmaceutical products

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The high-performance liquid chromatography method was performed for test method development of related compounds in pharmaceuticals. Using reverse-phase column and gradient elution of 1%acetonitrile-acetonitrile: H2O:triethylamine (70:30:0.5), lansoprazole, 2-hydroxybenzimidazole
, 2-mercaptobenzimidazole, lansoprazole sulfone, lansoprazole sulfide could be individually identified and quantitated. The correction factor by sensitivity was calculated, this test method showed a good repeatability and recovery with the range of 93.2 ~104.7%. Another test method, thin-layer chromatography method has been developed for measurement of lansoprazole and related compounds. Identification and quantitation were performed with silicagel F254 HPTLC plate, using development solvents of ethylacetate-chloroform-methano(12:5:1) & chloroform-methanol(10:1). The absorbance was monitered at 285nm. This HPLC & TLC method can be applied to test related compounds of lansoprazole.

[PD4-14] [04/18/2003 (Fri) 13:30 - 16:30 / Hall P]

Stability of 13C-urea/PEG capsules by LC-APCI-MS

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The applicability of liquid chromatography-atmospheric-pressure chemical-ionization mass spectrometry (LC-APCI-MS) for the determination of 13C-urea in 13C-urea/PEG capsules has been studied. It is essential to assess the stability of a newly developed low-dose (38 mg) 13C-urea/PEG capsule, which will be used for 13C-urea breath test (13C-UBT) to detect Helicobacter pylori infection. Standard curve was linear over the concentration range 10-1000 mg/ml. Intra- and inter-day variations were less than 2.75 % in APCI-MS. The detection limit was 10 pg when selected ion monitoring (SIM) was employed. The content of 13C-urea in capsules was within the acceptable range between 95 and 105 %. Therefore, it was established that 13C-urea/PEG capsules were stable under an accelerated stability condition that was set at 40 ±2℃with relative humidity of 75 ±5 % during 6 months by using LC-APCI-MS.

[PD4-15] [04/18/2003 (Fri) 13:30 - 16:30 / Hall P]

Development of analytical method of DMDM hydantoin, Sorbic acid, Phenoxy ethanol in Cosmetics