Determination of Enantiomeric Purity of (S)-(+)-Ibuprofen by ^{1}H-NMR using (-)-Cinchonidine as a Chiral Solvating Agent

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^{1}H-NMR method for the determination of enantiomeric purity of (S)-(+)-ibuprofen was developed using (-)-cinchonidine as a chiral solvating agent. (S)-(+)-ibuprofen was prepared by optical resolution of racemic ibuprofen using preferential recrystallization method with (S)-(-)-α-methylbenzylamine and (R)-(-)-ibuprofen by semi-preparative chiral HPLC using chiral OD column and n-hexane/2-propanol/trifluoroacetic acid as a mobile phase. Several concentrations of synthetic mixture of (S)-(+)-ibuprofen and (R)-(-)-ibuprofen were added to the (-)-cinchonidine disolved in $CDCl_3$. All ^{1}H-NMR spectra were referenced to TMS taken as 0.00ppm on the scale. The relative intensities of the resonance for the enantiomerc aromatic protons at 6.822~6.803ppm for (R)-(-)-ibuprofen and 6.934~6.914ppm for (S)-(+)-ibuprofen were measured. The recovery of (R)-(-)-ibuprofen present in (S)-(+)-ibuprofen was 101.5 3.2%. C.V.(%) was 9.61% and detection limit was 0.8%.

[PD4-20] [2003-10-10 14:00 - 17:30 / Grand Ballroom Pre-function]

Development of simultaneous analysis of Pheniramine Maleate and Naphazoline Hydrochloride Ophthalmic Solution

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We want to actively cope with technological developments by improving our drug specification in Korean Pharmaceutical Codex. Pheniramine maleate and naphazoline HCl of Pheniramine Maleate and Naphazoline Hydrochloride Ophthalmic Solution were ordained as UV spectroscopy and HPLC, respectively. Simultaneous analysis of those using HPLC has been developed. The assays of two drug products were 97.2%, 102.2% (pheniramine maleate), 96.8%, 101.3% (naphazoline HCl) respectively ; the specification range is 90.0~110.0%. The assay of pheniramine maleate was quite different from those using established UV spectroscopy method (262nm) as 111.7%, 115.5%, which were over the spectification. To elucidate the cause of this difference, we checked UV absorbances of another component (naphazoline maleate) and excipients ; mixture ratio followed specification. Absorbance of pheniramine maleate at 262nm was 0.4266, while that of naphazoline HCl was 0.0359, and excipients did not absorbed. The corrected results were 103.1%, 106.9%. Therefore it is urgent to change the specification of pheniramine maleate of Pheniramine Maleate and Naphazoline Hydrochloride Ophthalmic Solution to HPLC method. Accuracy, detection limit, quantitation limit, linearity, precision, system suitability of our suggested HPLC method were examined.

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Analysis of β-Blockers in Whole Blood by GC/MS-SIM

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We describe here solid-extraction and derivatisation methods of β-adrenoceptor blocking drugs used for the treatment of various cardiovascular disorders such as hypertension, angina pectoris and cardiac arrhythmia: propranolol, metoprolol, sotalol, timolol, oxprenolol, alpranolol, atenolol, pindolol. Solid-extraction and derivatisation methods are described involving the use of Bond Elut Certify cartridges, MSTFA and MBTFA. Gas chromatographic-mass spectrometry analysis(GC/MS) was carried out select-ion monitroing mode. Recovery, detection limit and calibration curves were carried out the extraction of their spiked whole blood using Bond Elut Certify cartridges. The application of this method on some forensic cases is also presented for the simultaneous screening for β-blockers in postmortem blood.