

Chiral separation method of the enantiomers of several β_2 -agonists was investigated by capillary electrophoresis employing β -cyclodextrin, carboxymethyl- β -cyclodextrin, hydropropyl- β -cyclodextrin as chiral selectors. The effect of concentration of the β -cyclodextrin derivatives added to the background electrolyte, of the pH of the buffer and of the temperature on the effective mobility and resolution of the studied compounds were examined. On the basis of the result, the effects of the addition of polyethylene glycol on enantiomeric resolution could be explained. Optimum conditions of capillary electrophoretic enantioseparations with cyclodextrin additives are given.

[PD4-7] [04/21/2000 (Fri) 14:50 - 15:50 / [1st Fl, Bldg 3]]

Chiral separation of β -blockers after derivatization with (-)-MTPACI by Gas Chromatography/Mass Spectrometry

Kim KH1, Kim JH1, Kim HJ1, Ko MY⁰1, Mar WC2, Kang JS3, Hong SP4, Lee JH1

1College of Pharmacy, Kangwon National University, 2Natural Product Research Institute, Seoul National University, 3College of Pharmacy, Chungnam National University, 4College of Pharmacy, Kyung Hee University

Gas chromatography/mass spectrometric chiral separation method was investigated. Several β -blocking drugs were reacted with the chiral acylating reagent (-)- α -methoxy- α -(trifluoromethyl) phenylacetyl chloride to obtain, in each case, two diastereomeric amide derivatives. Prior to N-acylation, hydroxyl groups of drugs were derivatized with N-methyl-N-(trimethylsilyl) trifluoroacetamide. The effect of reaction time, reaction temperature and concentration of chiral reagent, (-)-MTPACI on the chiral derivatization reaction were investigated. Elution order of the diastereomeric amides was observed for four compounds, atenolol, metoprolol, betaxolol and bisoprolol, by chromatographing the individual enantiomers separately under the same chromatographic condition. Each enantiomer was prepared by semi-preparative HPLC using a chiralcel OD chiral column.

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Determination of optical purity in levofloxacin preparations

Choi SO⁰, Kong HS, Kim EJ, Jang JY, Jung HY, Park HY*, Jang SJ

Division of Antibiotics, Department of Drug Evaluation, Korea Food and Drug Administration, *Ewha Womans University

Levofloxacin is the (-)-isomer of ofloxacin. Ofloxacin was originally marketed as a racemate; i.e., a mixture of the two optical isomers in equal ratios. And (-)-isomer of ofloxacin was more potent than the (+)-isomer in antibacterial activity. Therefore, levofloxacin has been developed as a single isomeric preparation by racemic switch. In this study, we separated the ofloxacin enantiomers using mobile phase additives, derivatization and a chiral stationary phase in order to control the quality of levofloxacin preparations.

Firstly was the separation carried out on a novapak C18 column using 1 mmol/L cupric sulfate-methanol (90 : 9) as a mobile phase with UV detection. Secondly was it carried out on a novapak C18 column using 0.2 mmol/L phosphoric acid (pH 1.85)-acetonitrile (80 : 20) as a mobile phase with fluorescence detection, having been derivatized with diphenylphosphorylchloride and L-leucinamide. Thirdly was it carried out on a bovineserumalbumin column using 0.2 mmol/L phosphate buffer (pH 8.0)-methanol (97 : 3) as a mobile phase with UV detection. With the result of purity determination in levofloxacin preparations, we have concluded that optical purity was quite satisfactory in levofloxacin preparations.