

Simultaneous determination of talniflumate and its metabolite in human plasma by high-performance liquid chromatography

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Talniflumate is a potent analgesic and anti-inflammatory drug widely prescribed in rheumatoid diseases. The purpose of this work was to develop and validate a specific and robust method for the simultaneous determination of talniflumate and its metabolite, niflumic acid, in human plasma. Indomethacin was used as an internal standard (IS). To simultaneously determine the plasma concentration of talniflumate and niflumic acid, IS solution and methanol were added to plasma samples and the mixture were centrifuged at 3000 g for 30 min. Then, 60 µl of supernatant was injected onto the HPLC reversed-phase column (C18). The signals were monitored by UV detector at 288 nm. The run time was 20 min per sample and analyte was quantified by linear regression of peak area ratio. This assay was validated at a nominal range of 0.1 to 5 µg/ml. Linear over the calibration range was > 0.9972. The interday accuracy ranged from 92.3 to 99.09 % with precision ranging from 91.44 to 94.62 %. The intraday accuracy ranged from 90.65 to 99.29 % with precision ranging from 91.36 to 94.19 %. The retention times of the IS, niflumic acid and talniflumate were 6.5, 7.5 and 13.5 min, respectively. This analytical method was shown to be accurate and reproducible. This method could be suitable for the simultaneous determination of talniflumate and its metabolite in human plasma.

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Comparison of liquid chromatographic enantiomer resolution of racemic amino compounds on chiral stationary phases of crown ether type

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ChiroSil RCA(+) and SCA(-) HPLC chiral stationary phases (CSPs) developed by covalently bonding (+)- and (-)-(18-crown-6)-2,3,11,12-tetracarboxylic acid (18-C-6-TA) to silica gel were employed for enantioresolution of racemic amino compounds, respectively. Also, these 18-C-6-TA covalently bonded CSPs were compared to a commercially available Crownpak CR CSP prepared by coating chiral crown ether as a chiral selector on ODS column. It was shown that these ChiroSil RCA(+) and SCA(-) columns have the advantage of the reversal of elution order. For the resolution of diphenylalanine enantiomers, especially, it was observed that the chromatographic parameters such as separation factors and retention times are greatly influenced by mobile phase conditions.

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Identification of Impurities in a Sample of Illicitly Synthesized Methamphetamine

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Analysis of illicit methamphetamine samples seized in Korea is discussed. The samples are extracted with the small portion of ethyl acetate under neutral conditions and the extracts are analyzed by GC-MS. Several impurity peaks are found in each chromatogram. Eight