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Biochem Pharmacol (Los Angel) 2018, Volume 7
DOI: 10.4172/2167-0501-C1-010

10th World Congress on **Pharmacology**
&
6th International Conference and Exhibition on
Advances in Chromatography & HPLC Techniques
August 02-03, 2018 | Barcelona, Spain

Enantiomeric separation of fluoxetine on C18 column using DFDNB based chiral derivatizing reagent having L-amino acids as chiral auxiliaries

Hariom Nagar[1] and **Ravi Bhushan**[2]
[1]Suresh Gyan Vihar University, India
[2]Indian Institute of Technology Roorkee, India

Optically pure amino acids L-Val, L-Phe, L-Leu and S-methyl-L-Cysteine were used to synthesize chiral derivatizing reagents FDNP-L-Val, FDNP-L-Phe, FDNP-L-Leu and FDNP-SMLC. These reagents were characterized using UV, IR, CHN, and 1H NMR. These all are containing fluoro dinitro benzene as the chromophore and hence are of good molar absorptivity. The synthesis of diastereomers of fluoxetine was carried out by the reaction of these chiral derivatizing reagents with fluoxetine under microwave irradiation for 60s at 75% (of 800W) and also by stirring for 45 min at 50˚C. The diastereomers were enantioseparated by reversed-phase high-performance liquid chromatography on a C18 column with detection at 340 nm using gradient elution with mobile phases containing aq TFA (0.1%)-MeCN in different compositions. The conditions of derivatization and chromatographic separation were optimized. The method was validated for accuracy, precision, limit of detection and limit of quantification.

hariomnagariitr@gmail.com