



Fully automated preparative HPLC using recycling technology

Application : separation of enantiomers

Introduction

Single enantiomeric drugs represent 50 % of the top 500 drugs worldwide. Therefore, preparative scale separation of pure enantiomeric products from racemic drugs is a significant problem to solve in pharmaceutical process research.

Purpose

Isomer purification in the chemical and pharmaceutical industries can be achieved by the use of recycling technology (see 'Hydraulics Schematic'). This application makes use of the advanced specifications of Gilson's preparative pumps.

Materials

Pump: Gilson 333 Pump (prep-scale HPLC) with tubing adapted to recycling.

Detector: Gilson 155 Detector (UV/Vis) with high pressure flow cell.

Sample Injector: Gilson 231 XL.

Dilutor-Dispenser: Gilson 402 with 25 ml syringe and 25 ml transfer tubing.

Valve-actuators and Valves

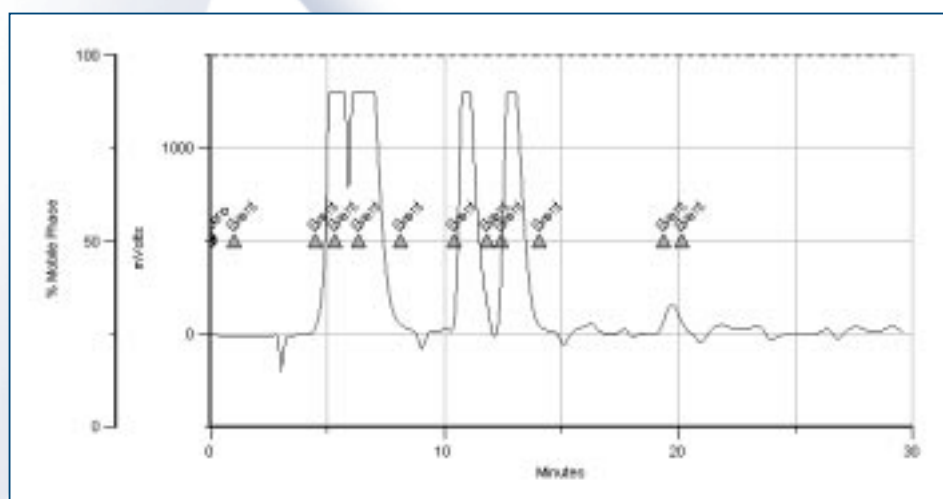
Gilson VALVEMATE equipped with the following valves:

injection + recycling valve: Rheodyne 7000L

collection valve: Rheodyne 7060L.

Method

After injecting the sample into the system, a closed recycling phase is performed. A programmed collection, adapted to the particular shape of the peaks, allows the collection of the pure enantiomeric products. Then, another programmed injection can be executed.



Chromatographic conditions

Column

NOVASEP dynamic axial compression,
ID 50 mm; maximum length: 500 mm

Stationary phase:

SIRIUS 2: reticulated polymer from CHIRALSEP (dp 10-20 μm)

Mobile Phase

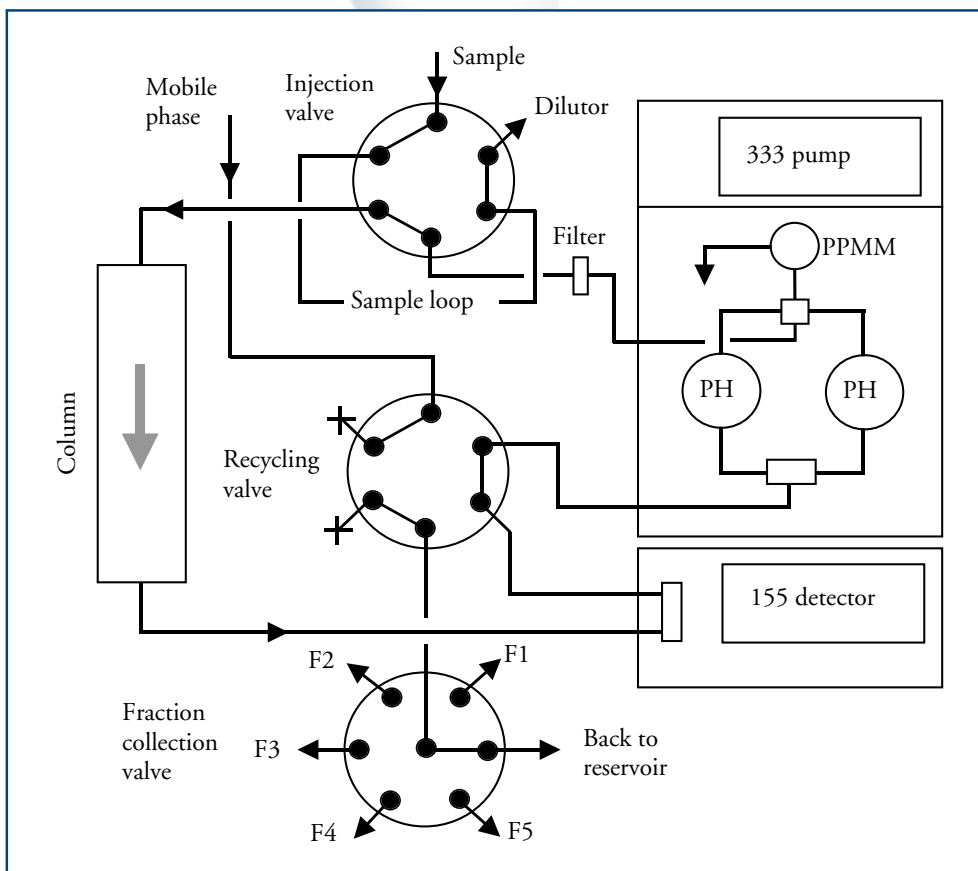
- 0.5 % TFA in Toluene,
- flow rate: 150 ml/min at 21 °C.

Injected Sample

22.4 ml of 1,1'-bi-2-naphtol racemate in CH_2Cl_2 (27.9 mg/ml).

Advantages of recycling technologies

- High purity and recovery yield in the separation of two closely eluted compounds, such as enantiomeric isomers.
- Reliable technique: with limited changes to the outlet tubings, the 333 Pump, with its dual piston technology, presents good features for preparative recycling applications, and the system can easily revert to simple chromatography.
- High increase of column efficiency : this system allows the use of a shorter column without losing resolution, which is of interest when using expensive chiral phases.
- Low eluent consumption and reduced working pressure.



Hydraulics schematic

ISO 9001 Certified

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