Chiral Purifications with the ACCQ*Prep* HP125

Abstract

When there is a need to purify chiral compounds, the ACCQ*Prep* HP 125 can be equipped with a chiral column and can easily perform this type of purification. In this application *trans*-stilbene oxide is used as a model for other chiral compounds since it is a standard for chiral purification.
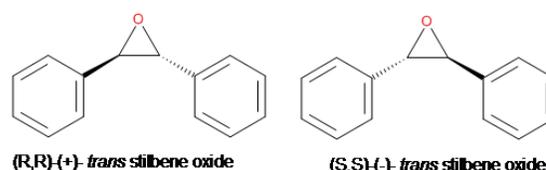
Preparing the ACCQ*Prep* HP125

To avoid possible damage to the chiral columns from inappropriate solvents, follow the steps below before attaching your column. Read the instructions for your column to determine which solvents are compatible with your column:

❏ When configuring the chiral column, set the maximum pressure limit in the ACCQ*Prep* configuration as per the instructions that came with the column. Setting this pressure limit prevents possible damage to the column.

❏ Since the ACCQ*Prep* is generally used for reverse phase separations, it is a good idea to prime the system with an intermediate solvent such as 2-propanol first. This solvent is typically miscible with both reverse phase solvents and the solvents most often used for chiral.

❏ Remove the reverse phase column and replace the column with a simple union so the column position can be bypassed and the entire fluid path can be flushed with 2-propanol.

❏ Use manual control to flush the injection loop and column tubing.

❏ Remove and dry the solvent lines and then place the lines into the solvents to be used for the chiral purification.

❏ Prime the ACCQ*Prep* with the solvents used for the purification.

❏ Use manual control to flush the injection loop and tubing leading to the column with the solvents used for the chiral purification.

❏ If the system is equipped with an AutoSampler, flush the wash station with a solvent compatible with the chiral column. Again, you must take into account the miscibility of the solvent previously used and the new wash solvent that is to be used.

❏ Verify the Bracketed Injection Tube Station, if used, contains a solvent compatible with the chiral column used (system equipped with the AutoSampler only).

Experimental and Results

All solvents were HPLC grade. The column was a ChiralPak AS column, 21x250 mm, 20 µ particle size. The column was run at 18 mL/min with a mobile phase of 10% 2-propanol in hexanes. The trans-stilbene oxide was dissolved in hexanes containing 10% 2-propanol. For the first run, 3.9 mg was injected while the second run used 52.0 mg sample. Detection was 230 and 210 nm. All runs used an ACCQ*Prep* HP 125 system and AutoSampler (PN 68-5230-097).

(R,R)-(+)- *trans* stilbene oxide   (S,S)-(-)- *trans* stilbene oxide

Run 1: 3.9 mg injection
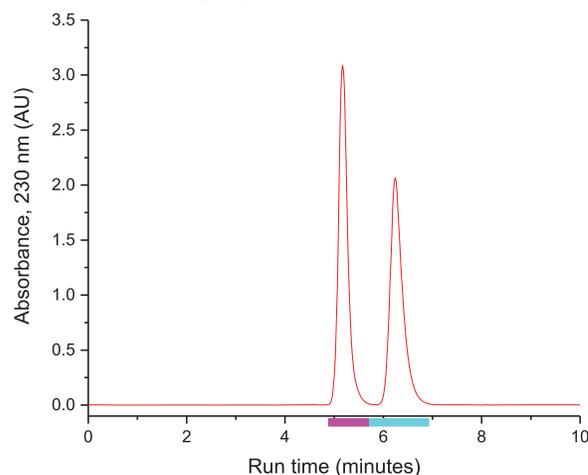
Run 2: 52.0 mg injection
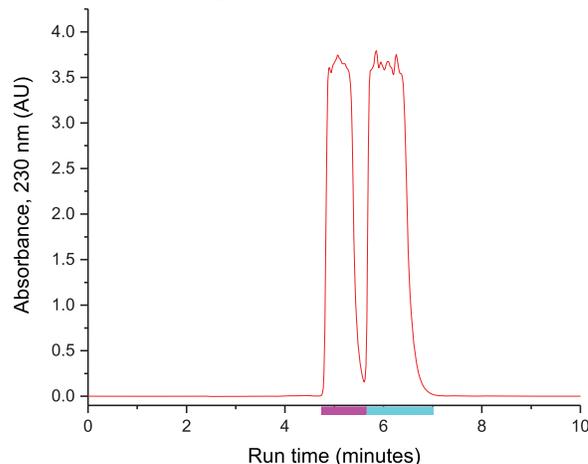
Both isomers were easily resolved with the ACCQ*Prep*HP 125 system. Resolution between compounds determines the load capacity for a column. Columns, solvent systems and compounds will determine the actual load for any experiment.

Conclusion

The ACCQ*Prep* HP125 can accurately deliver solvent, even at the low back-pressures generated by many chiral columns, to generate accurate isocratic purifications. The ability to set a pressure limit for different columns allows a mix of columns to run on the system, but prevents damage to the column in the case of a clog or other over-pressure event. The AutoSampler option allows unattended automated injection of samples, allowing many runs to be made to compensate for the relatively low loading allowed by chiral columns. The active solvent and waste management also allows for unattended operations since the system will automatically pause the run if a solvent is low or the waste bottle is full. Active solvent management prevents loss of compound or damage to the column, while active waste management prevents spills from over-filled waste containers.

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