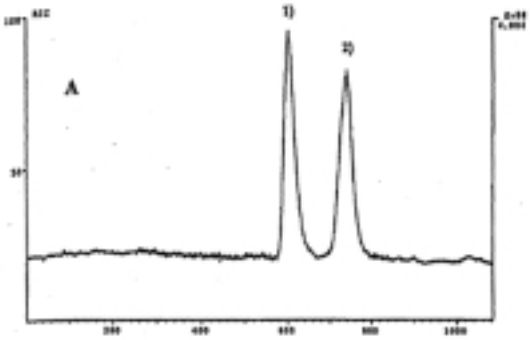
CHIRAL-AGP method optimization for LC/MS

Most methods published on the **CHIRAL-AGP** columns are optimized for UV or fluorescence detection. However, today many analytical chemists use **MS**-detection on a regular basis. They need other buffers in the mobile phase and they also prefer shorter columns, as their **MS**-detector can create a high detection selectivity. In this Application bulletin we will show ways to transfer methods to better suit MS conditions.

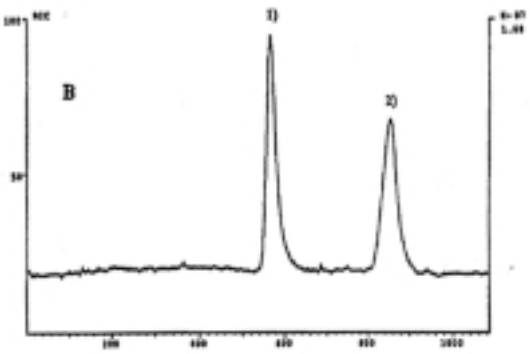
Verapamil and gallopamil

In an article by C. Rustichelli et al in *Chromatographia vol.44 (1997) p.477-483* a method for resolution of the enantiomers of verapamil and gallopamil on **CHIRAL-AGP** using LC/MS is described. The column dimension used is 100x4.0 mm and the mobile phase 11% acetonitrile in 10 mM ammonium acetate pH 6.8. Flow 0.9 ml/min. Below are the reconstructed ion-current chromatograms:

Verapamil



Gallopamil

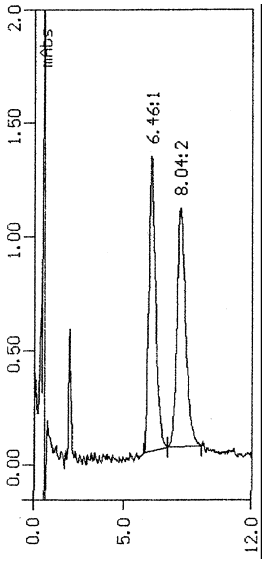


The analysis time for verapamil is 15 min. and for gallopamil 17 min.

To decrease the retention time for the verapamil separation the column dimension was changed to 50x2.0 mm. The buffer concentration was also decreased, as well as the modifier concentration.

Column:
CHIRAL-AGP 50x2.0 mm

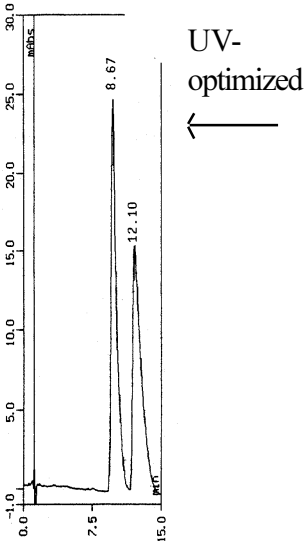
Mobile phase:
10% acetonitrile in 5 mM ammonium acetate (pH 6.6)



As can be seen, the analysis time is now reduced to 9 min for verapamil. The flow rate is 0.22 ml/min.

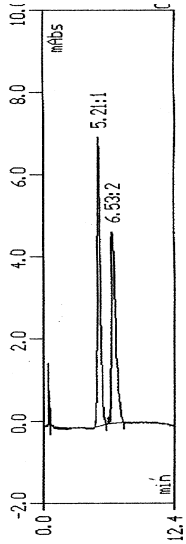
Conversion from UV-method to LC/MS method

Pindolol is used as an example. In the UV-method the mobile phase is 10 % acetonitrile in 10 mM sod.ph.b. pH 7.0 and the column dimension 100x4.0 mm with a flow rate of 0.9 ml/min. To make it suitable for MS the phosphate buffer was replaced with 10 mM ammonium acetate which has a pH of ca. 6.6. To optimize selectivity the acetonitrile concentration was decreased to 7%. Column dimension was 50x2.0 mm and flow rate 0.22 ml/min.



UV-optimized ⟵

MS-optimized ⟶



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