UNIVERSITEIT VAN AMSTERDAM

Method Development for Comprehensive Two-Dimensional Gas Chromatography

J.H.M. Mommers

Method Development for Comprehensive Two-Dimensional Gas Chromatography

Summary

Comprehensive two-dimensional gas chromatography (GC×GC) is a powerful analytical technique capable of proving high separation power, enhanced sensitivity, and structured chromatograms. Although, GC×GC technology is becoming more mature, more accessible, and increasingly adopted by analytical chemists, GC×GC method development is significantly more difficult than for 1D-GC. For GC×GC more method development choices need to be made and optimization is difficult due to a complex interplay of the many ^{1}D and ^{2}D parameters. Method development is also restricted by the modulation criterion and column-set temperature limitations. Also, the usual difference in the primary and secondary internal diameters prevent that both columns can be operated at their optimal linear gas velocities (flow mismatch issue) and the usually smaller secondary internal diameter may often lead to mass loadability issues. If that's not all, peaks in GC×GC may shift in two directions which causes alignment issues which are less straightforward to solve compared to 1D-GC retention time shifts.

In order to make optimal use of GC×GC, a systematic approach of method development is essential. The choices should be based on a thorough understanding of the main analytical question (and its requirements) and knowledge of the sample analytes and matrix compounds. Based on this, the GC×GC set-up, the initial column-set and all other parameters could be chosen, tested and optimized. In chapter 2, a review is given in which several papers concerning GC×GC method development are discussed and guidelines for GC×GC method development are proposed. The most important and also most difficult task in GC×GC method development is the choice of the column-set. In chapter 3, a procedure is described for the global prediction of best GC×GC column-sets, based on the adapted Abraham solvation parameter model as published by Seeley et al. This prediction procedure may assist in roughly selecting best column-sets as a starting point. In chapter 4 the issue of GC×GC retention time shifts is addressed. A fast and easy to perform, two-step retention time locking procedure for GC×GC is proposed and its feasibility is demonstrated. This 2D-RTL procure is routinely used for already 5 years in our Lab at DSM. In

chapter 5, a retention time locking procedure for locking primary and secondary retention times of detector signals in GC×GC dual-detection is proposed and its practical advantages are demonstrated and discussed. Chapter 6 and 7, both deal with optimizing GC×GC selectivity by tuning selectivity in the first and/or the second dimension. These approaches could be used to further optimize or fine-tune certain (difficult) target or group-type separations especially for truly complex chromatograms. These set-ups also offer enhanced possibilities for qualitative analysis.

The actual analytical power of GC×GC for the analysis of a particular complex sample (application) strongly depends on the method development choices and optimization, but also on good chromatography practice and the data processing approach; all of this to be able to answer the analytical question.