

PEAK DISPERSION EVALUATION AND OPTIMIZATION IN GC×GC

P.-H. Stefanuto^a, J.-M. Dimandja^b, J.-F. Focant^a

^a Organic and Biological Analytical Chemistry Group, CART, University of Liège, Belgium

^b Department of Chemistry and Biochemistry, Spelman College, Atlanta, USA

From the early days of comprehensive two-dimensional gas chromatography (GC×GC), the concept of orthogonality has always been an important topic of discussion and debate. While the theoretical construct of orthogonality is fairly well understood and accepted, the practical evaluation of orthogonality in real GC×GC chromatograms has presented a bigger challenge in terms of finding consensus criteria amongst investigators. In fact, some recent studies have even suggested that orthogonality evaluation may not be as relevant as originally thought for GC×GC optimization. A more practical measure of bidimensional separation efficiency is the evaluation of the true peak capacity. The efficient use of the available peak capacity in a given separation is dependent on the stationary phase coupling, the operating conditions, and the relative dispersion of the peaks in the two-dimensional plane (1).

In this study, we investigated methods for peak dispersion evaluation and chromatographic quality criteria with the aim to contribute to the development of a statistical method for the optimization of GC×GC separation. The occupation of the two-dimensional space was evaluated using different approaches. First, geometrical methods were investigated: percentage of occupation (2), and convex hull ratio calculation (3). Second, the calculation of the dispersion was conducted using average Euclidian distance and standard deviation of this value. In order to evaluate, the quality of the chromatography, other criteria were also taken into account (e.g. the tailing factor).

In order to improve the robustness of this approach, we implemented a Box-Behnken surface response methodology. In this design of experiment, the peak dispersion and the chromatographic quality criteria were used as a response and the temperature ramp, the flow and the secondary oven offset were the variable parameters.

This approach demonstrated that peak dispersion and chromatographic criteria combined with a DOE provide a valuable way to optimize the GC×GC separation.

References

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