

Obtaining new pesticide limits of quantification using gas chromatography coupled with ion trap mass spectrometry

David PAGES

Introduction

There are over five hundred pesticides worldwide used in agricultural regions and new agrochemicals are continuously introduced to the market. A new legislation published in October 27, 2011, imposes new pesticide and hydrocarbon limits in water. The subject of this study was to achieve these limits for thirteen pesticides using gas chromatography (GC) coupled with ion trap mass spectrometry (MS). To reach these limits, two choices are possible, on one hand, the noise could be reduced, and on the other hand, the intensity of the signal could be increased.

Experimental methods

The pesticides analysed were in a pesticide mixture in ethyl acetate. The machines used were a Varian CP 3800 and a Varian Saturn 2000 MS. The injection system was composed in two parts. First, the solvent was evaporated while the split was opened. After, the split was closed and the pesticides were evaporated and injected into the column. So, the injection conserved the pesticide quantity. The column used into the GC was a DB 5 MS (30m x 0.25mm x 0.25 μ m). The temperature method applied on the column was 50 to 320°C. The ion trap scanned the mass from 50 to 600 Thomson. Chrysen D-12 was used as an internal standard.

Results

The best choice was to increase the signal with the large volume injection system (LVI): the ATAS GL - Optic 3.

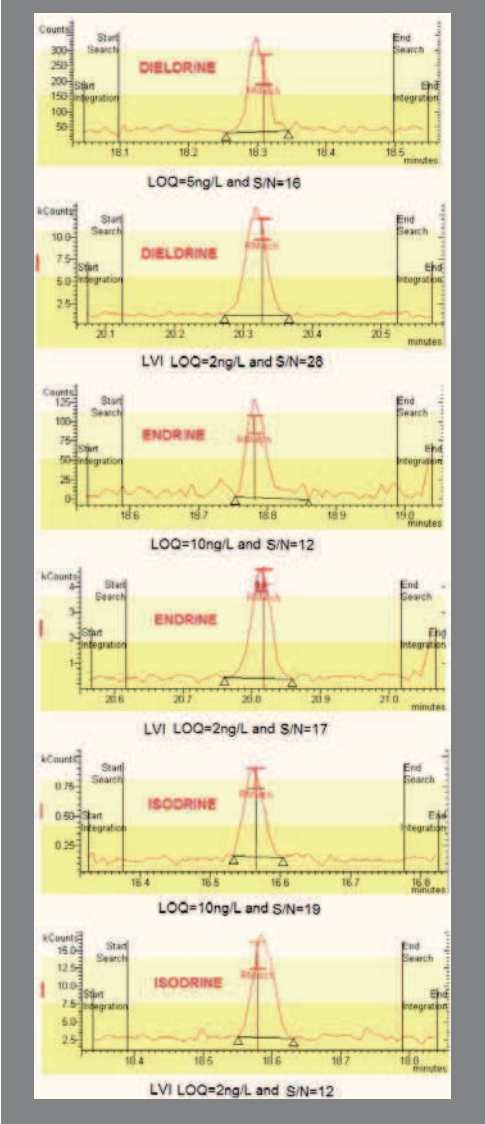
The Optic3 injected 100 μ L and the injection system usually used injected 5 to 8 μ L. So the LVI injector could boost the signal. This injector did not increase the dispersion, because it had a large volume liner and an injection program which was adapted.

The figure 1 shows the chromatograms for three pesticides for which the limits were more difficult to comply with than the other analysed pesticides. The other pesticides had the same evolution using LVI. Figure 1 was a comparison between the usual injection system and the LVI. The new limits were accepted if the signal to noise ratio (S/N) was greater than ten. The results with the references were very interesting with the LVI (Figure 1). The signal intensities were increased more than the noise for these three compounds. So the S/N was sufficient to obtain the limits imposed by the law. In fact, the GC peaks had good intensities. Moreover, they were defined with a correct resolution. This conclusion has to be validated with a real sample which could contain interfering molecules.

Conclusion

To conclude, a new formulation of methacrylate monolith was optimized in both capillary and microchip format. Even if a complete study of its electrochromatographic behavior needs to be carried out, the first results showed very promising performance, allowing the electrochromatography separation of a range of neutral solutes in a short time (10 min) with a low solvent consumption.

Figure 1: Comparisons GC peaks obtained using LVI or usual injector and the DB5MS column, from 60 to 320°C and scanning 50 to 600 Thomson



LA
DRO
ME -
laboratoires

Laboratoire départemental d'analyse de la Drôme
37 Avenue de Lautagne
26000 Valence