Adapting extraction and GC/MS analysis methods to esterified phenoxyacid families

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Introduction

Water is an indispensable part of our nutrition, which has to be analyzed before being consumed. Among the multiple parameters tested, pesticides mainly used by industry and agriculture, are looked for and quantified, in order to know if the limits fixed by the WHO (World Health Organization) are respected. Today, more than 300 pesticides are searched for with a routine method, but new requests concerning three esterified phenoxyacid families have recently been introduced. In order to analyze them routinely by GC/MS, they had to be tested by two types of extractions : Liquid/Liquid (L/L) and Solid Phase Extraction (SPE).

Experimental conditions

Routinely, analyses were realized with Agilent GC/MS (5975C Series), the column used was a HP-5MS J&W (30 m; 0.25 mm; 0.25 µm), and the carrier gas was nitrogen at 1.2 mL/min.

Firstly, the compounds were injected individually at 10 ppm (high concentration) with a SCAN method, allowing us to have the retention times and the fragmentation ions. These informations were collected in order to make a SIM (Single Ion Monitory) method. Then, compounds calibrations were carried out between 10 and 500 µg/L. Finally, water samples were doped with known quantities of the phenoxyacids, and extracted by L/L and SPE extraction. All the extraction yields were calculated with the intention to choose the most adapted method.

Results

Figure 1 shows the interest of the SIM method; indeed, in SCAN, the peaks corresponding to the compounds are not seen because of the low concentration, whereas in SIM, they correspond to all the high peaks. The quantification is made on these latter peaks.

The bar chart presented in figure 2, shows the yields obtained for the doped samples with the two types of extractions. Accepting errors, L/L and SPE extractions seem to be equivalent. Most compounds are between the limits fixed by the lab (in green), showing that they are adapted to the routine method and could be added to all the other pesticides. But for Mecoprop-ethylhexyl ester which is around 40%, to accept its analyses, optimisations will be necessary.

Conclusion

Nowadays, in order to prevent water contaminations, pesticide analyses are growing. Labs are requested to check out more and more substances, that is why, developments presented in this article, are commonly carried out. Today, the esterified phenoxyacids tested are extracted simultaneously with the other residues by the SPE method.



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