ENVIRONMENTAL ANALYSIS

ANALYSIS OF HERBICIDES IN WATER USING AUTOMATED ONLINE SPE ENRICHMENT

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Solution Note

Environmental

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Abstract

This application brief demonstrates the use of automated online enrichment for the sensitive LC-MS/MS analysis of herbicides in water samples.

Introduction

Herbicides are widely used in agriculture to protect crops from unwanted weeds. They can enter the aquatic system through runoff, during production or while being stored improperly. Because of their potential adverse effects on human health, both acute and long term, their presence and concentration needs to be monitored in water intended for human use (EU Council Directive 98/83/EC). The current limit is 500 ng/L for the sum of all pesticides and 100 µg/L for individual pesticides. The Limit of Detection (LOD) of the analytical method is required to be 25% of this value, which results in an LOD of 25 ng/L.

In water analysis, LC-MS/MS has become the method of choice, but at the required low concentration levels, several analytes are accessible to mid-range triple quadrupole mass spectrometers only after preconcentration steps. This is not only time consuming, but also requires additional equipment and space. The Agilent 1200 Infinity Series Online SPE system, which is based on a 1290 Infinity Flexible Cube and reusable SPE cartridges, offers a fully integrated solution for automated analyte enrichment. In this application the Flexible Cube based setup was used in combination with an Agilent 6460 triple quadrupole LC-MS/MS to analyze herbicides spiked into tap water at concentrations ranging down to 1 ng/L.

UHPLC-QQQ Method:

- 1260 UHPLC with Online-SPE solution
- 6460 QQQ, fast polarity switching
- SPE Cartridge: PLRP-S 2.1 x 12.5 mm, 15-20 µm
- Column: Agilent PLRP-s 100A, 50 x 2.1 mm, 3 um
- Acetic Acid, Ammonium fluoride, Water, Methanol

Summary:

Analysis of herbicides in water with automated online enrichment in combination with an Agilent 6460 triple quadrupole mass spectrometer meets and exceeds sensitivity requirements of current regulation.

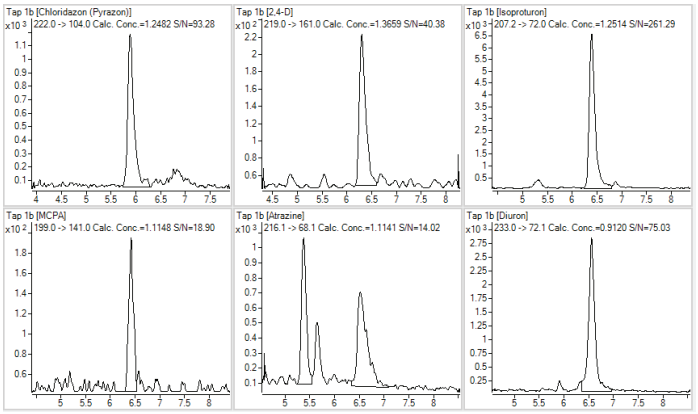

Figure 1: A tap water sample spiked with herbicides at 1 ng/L, Compounds-at-a-Glance view for fast review of large numbers of analytes/samples.

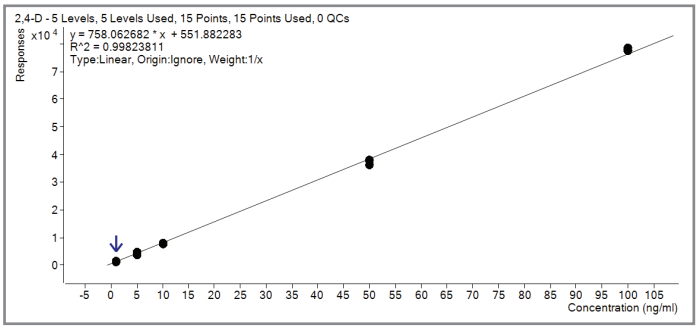

Figure 2: Linear Calibration of 2,4-D between 1 and 100 ng/mL.

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