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Development of purification procedures and HPLC-MS/MS method for the determination of glyphosate and AMPA in environmental water

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INTRODUCTION

Glyphosate [*N*-(phosphonomethyl)glycine] is one of the most widely applied herbicides in the world. This compound came on the market in 1970s under the trade name **Roundup**.

It is used for weed control in agriculture, forestry and gardens, and also on railway embankments.

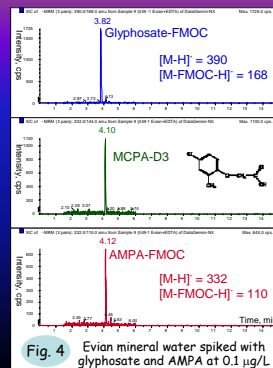
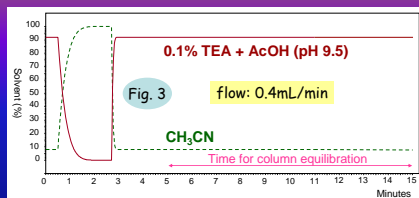
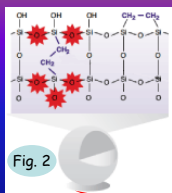
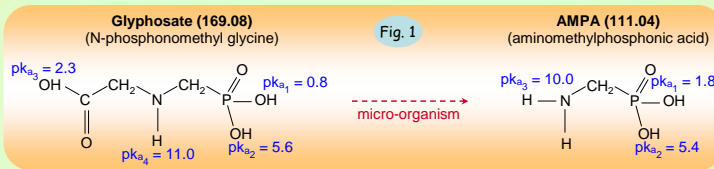
Glyphosate is rapidly adsorbed in soil and **degraded to aminomethylphosphonic acid (AMPA)** (fig. 1) - its main metabolite and then transported to groundwater and surface water.

The high polarity and water-solubility of glyphosate and AMPA has, until recently, made their analysis in water samples problematic, especially at trace levels.

Because of the lack of adequate chemical groups (e.g. chromophores), **glyphosate and AMPA are derivatized with 9-fluorenylmethylchloroformate (FMOC-Cl)** to form fluorescent derivatives. This derivatization reduces also the polar character of the analytes and facilitates the chromatographic retention.

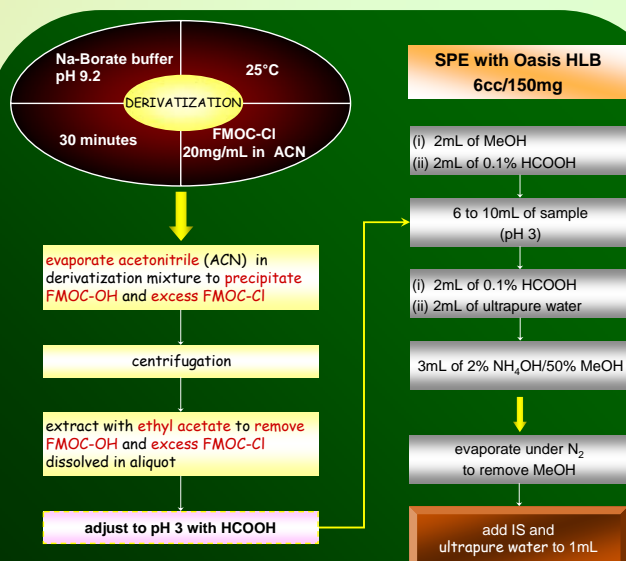
The aim of this study is to improve both extraction and purification protocol, and to develop an efficient and robust HPLC-ESI-MS/MS method for determining glyphosate and AMPA in environmental water.

According to **EU Directive 98/83/EC**, the permissible level for **glyphosate and AMPA in drinking water is 0.1 µg/L**



The novel columns with **Ethylene Bridged Hybrid** particles (fig. 2) - XBridge (2.1x50mm, 3.5µm) or Gemini-NX (2x50mm, 3µm) - were used for the application at a high pH mobile phase. The best separation and sensibility were further obtained with 0.1% triethyl amine (TEA) adjusted to pH 9.5 with acetic acid (fig. 3).

MCPA-D3 was used as **internal standard (IS)** (fig. 4) and quadratic calibration curves were obtained for both analytes.



Recovery of **Glyphosate**: 93%
Recovery of **AMPA**: 70%

CONCLUSION

The new chromatographic method based on the application of **C₁₈ bonded hybrid particle column** and **basic mobile phase** gave **reproducible retention times** and **very sharp peaks**.

The combination of **liquid-liquid extraction** with **ethyl acetate** and **solid-phase extraction (SPE)** on Oasis HLB improved the elimination of the impurities and the pre-concentration of the analytes. We obtained **high recoveries (93% for glyphosate and 70% for AMPA)** with ultrapure and mineral waters spiked at 0.1 µg/L.

In order to improve the recoveries, ongoing research concerns a preliminary sample treatment with **EDTA** for removing **Ca²⁺, Mg²⁺** and others bivalent cations that form strong complexes with analytes.