

# DETERMINATION OF MULTI-CLASS PESTICIDE RESIDUES AND METABOLITES IN DRINKING AND SURFACE WATER BY LC-ESI-MS/MS

Adriana Demoliner<sup>1</sup>, Sergiane S. Caldas<sup>1</sup>, Fabio F. Gonçalves<sup>1</sup>, Ednei G. Primel<sup>1</sup>,  
Osmar D. Prestes<sup>2</sup>, Martha B. Adaime<sup>2</sup>, Renato Zanella<sup>2,\*</sup>

<sup>1</sup> LACOM, Universidade Federal do Rio Grande (FURG), Depto. de Química, Rio Grande-RS, Brasil

<sup>2</sup> LARP, Universidade Federal de Santa Maria (UFSM), Depto. de Química, Santa Maria-RS, Brasil

\*renatozanella@pq.cnpq.br

In the management of water resources, regulations have been created that in general establish concentrations of few nanograms per liter for pesticide residues. The different physicochemical characteristics and wide polarity range of the pesticides and their transformation products make their simultaneous preconcentration and determination difficult. Thus, to make the determination at this magnitude possible, the laboratories frequently choose the LC-MS/MS together with a SPE preconcentration step to provide reliable results [1]. The aim of this work was the development of an efficient, quick, and suitable method for the simultaneous determination of pesticides and metabolites in drinking and surface water. The investigated pesticides were: clomazone, bispyribac-sodium, diuron, atrazine, simazine, imazetapyr, imazapic, metsulfuron-methyl, quinclorac, penoxsulam, 2,4-D, pyrazosulfuron-ethyl, bentazone, propanil, irgarol, tebuconazole, fipronil and carbofuran. The metabolites were: 3,4-DCA and 3-hydroxy carbofuran. These pesticides are nowadays widely used, especially in irrigated rice fields in southern Brazil. In the present study, a single preconcentration step using cartridges of solid phase extraction (SPE), followed by identification and quantification through liquid chromatography coupled to electrospray ionization tandem mass spectrometry (LC-ESI-MS/MS) was employed to perform the analysis.

All compounds presented excellent linearity with correlation coefficients higher than 0.99. Samples were prepared using solid-phase extraction (SPE) in cartridges containing 500 mg of C18ec. The method limits of detection varied between 0.4 and 40.0 ng L<sup>-1</sup> and the limits of quantification varied between 4.0 and 100.0 ng L<sup>-1</sup>. Mean recoveries using SPE varied between 70 to 120% for 95% of the compounds, with relative standard deviations (RSDs) below ≤20% for all. Through multiple reaction monitoring (MRM) two different precursor ion-product ion transitions were selected for each pesticide for quantification and qualification.

The LC-ESI-MS/MS method allowed sensitive and selective quantification and identification at low levels requiring only a simple sample preparation procedure by solid phase extraction that proved to be convenient and effective for the determination of polar pesticide residues and transformation products in surface and drinking water samples without interferences at concentration levels in accordance with regulatory requirements for surface and drinking water.

The method was applied to monitoring for six months surface water samples collected from different regions of the Rio Grande do Sul State located in South of Brazil.

[1] Barceló, D., MC Hennion, M.C. 2003. Trace Determination of pesticides and their degradation products in water, vol. 19, Elsevier Science, Amsterdam.

CNPq, CT-HIDRO, CAPES and FAPERGS