Home ▶ All Journals ▶ Physical Sciences ▶ International Journal of Environmental Analytical Chemistry ▶ List of Issues ▶ Volume 91, Issue 14 ▶ Analytical study on ethephon residue det ....

International Journal of Environmental Analytical Chemistry ›

Volume 91, 2011 - Issue 14

Original Articles

Analytical study on ethephon residue determination in water by ion-pairing liquid chromatography/tandem mass spectrometry

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Pages 1380-1391 | Received 25 May 2010, Accepted 27 Aug 2010, Published online: 21 Oct 2011

Cite this article https://doi.org/10.1080/03067319.2010.520131

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Abstract

A detailed analytical study on ethephon residue determination in water, making use of ion-pairing liquid chromatography coupled to electrospray tandem mass spectrometry (LC/MS/MS), has been carried out. Ethephon is a plant growth regulator, highly polar, which is typically present in aqueous solution in anionic form due to its acid character. Both its extraction and pre-concentration from water samples and its chromatographic retention are difficult. Several approaches for sample pretreatment have been tested including direct injection into the chromatographic system, on-line solid phase extraction (SPE) and off-line SPE, with the best results being obtained after off-line SPE, using Oasis MAX cartridges (mixed-mode strong anion-exchange). After testing several ion-pairing reagents, tetrabuthylammonium acetate (TBA) was selected. This was added to the samples before LC/MS/MS analysis to facilitate ethephon chromatographic retention. The acquisition of several specific MS/MS transitions together with the evaluation of their relative intensity ratios allowed the reliable confirmation of the analyte in samples. The optimised approach was tested in low-salinity water spiked at 0.1 µg L^{-1} level with satisfactory recovery, and a limit of detection of 0.02 µg L^{-1}. To this purpose, the water sample was partially de-ionised in an initial stage, in order to remove major ions that would have interfered in analyses. The application of this methodology to more saline/complex water samples, as surface or wastewater, was problematic and a thorough optimisation of the de-ionisation conditions would be required.

Keywords:

ethephon

ion-pairing liquid chromatography

tandem mass spectrometry

tetrabuthylammonium, water analysis

Acknowledgements

This work has been developed under financial support of the Ministry of Education and Science, Spain (CTM2006-06417). The authors acknowledge the financial support of Generalitat Valenciana, as research group of excellence PROMETEO/2009/054. C. Ripollés is very grateful to the Ministry of Education and Science for her pre-doctoral grant.

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