



Optimization and application of an extraction procedure to determine drugs of abuse in solid environmental matrices of Turia River Basin

Maria Jesus Andres (1), Rodrigo Alvarez (1), Vicente Andreu (2), and Yolanda Pico (1)

(1) University of València, Spain (yolanda.pico@uv.es), (2) Centro de Investigaciones sobre Desertificación (CIDE), CSIC-UV-GV, Moncada, Spain

After their consumption, drug of abuse are excreted through urine or faeces, as parent compound or as secondary metabolites that arrive to wastewater treatment plants. Accordingly, the incomplete removal of these compounds in the treatment plants could release them into environmental compartments [1]. This scenario needs attention from an ecotoxicological perspective because their possible negative effects [2].

The aim of this study is to optimize and apply a solvent extraction and solid phase clean-up methodology to obtain a valid procedure for the extraction of these compounds in different solid matrices. Amphetamine, methamphetamine, ethylamphetamine, ecstasy, ethylone, bk-MMBDB and MBDB belong to phenylethylamine group; codeine and ketamine belong to opioid and phencyclidine group, respectively, and benzoylecgonine is the major excreted metabolite of the alkaloid cocaine.

To optimize the method to determinate drugs of abuse in environmental solid matrices, two replicates and one blank were prepared for each sample of sediment. They were prepared by adding 1 g of sediment sample, 5 mL of buffer (methanol-Mc Ilvaine 50:50) and internal standard to obtain a final concentration in the extract of 25 ng/g. Also standards of drugs of abuse were added to the replicates to obtain a final concentration of 100 ng/g. Then all samples were shaken, sonicated and centrifuged and the supernatant was separated and placed in a 250 mL volumetric flask, which was filled the rest with distilled water. SPE was carried out with Strata-X cartridges and 250 mL of sample were passed through them. The extracts were eluted with 6 mL of methanol, evaporated to dryness and reconstituted in 1 mL of methanol-water 1:9. One of the replicates was filtrated through 0.22 μm pore size and the others were not. The samples were determined by liquid chromatography triple quadrupole mass spectrometry (LC-QqQ-MS/MS) using an electrospray ionization source (ESI) in positive ionization mode.

The results show that extraction recoveries of phenylethylamine group were from 39.3 to 92.4%. For codeine and ketamine, the recoveries ranged from 44.4 to 90.6% and from 61.3 to 79.5%, respectively. Benzoylecgonine presented recoveries ranged from 72 to 77.5%. The precision of the method was below 20% for all the compounds. The method was applied to determine these drugs of abuse in sediments of the Turia River Basin. Ecstasy, codeine, ketamine and benzoylecgonine were found at concentrations ranging from 0.22 ng/g to 25 ng/g in 6 sampling points.

Very limited information exists on the presence of drugs of abuse in environmental matrices. Nothing can be concluded about the differences between the recoveries obtained in unfiltered and filtered samples because they do not follow any trend. These results confirm the reliability of the method as well as its suitability to be applied in environmental studies.

Acknowledgements

This work has been supported by the Spanish Ministry of Economy and Competitiveness trough the project SCARCE-CDS 2009-0065, CGL 2011-29703-C02-01 and CGL 2011-29703-C02-02. MJ Andrés Costa also acknowledges to this Ministry the FPI grant to perform her PhD.

References

- [1] T.H. Boles, M.J.M. Wells, Analysis of amphetamine and methamphetamine as emerging pollutants in wastewater and wastewater-impacted streams, *Journal of Chromatography A* 1217 (2010) 2561.
- [2] C. Postigo, M.J. López de Alda, D. Barceló, Drugs of abuse and their metabolites in the Ebro River basin: Occurrence in sewage and surface water, sewage treatment plants removal efficiency, and collective drug usage estimation *Environment International* 36 (2010) 75.