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QuEChERS extraction followed by on-line clean-up and tandem mass spectrometric determination of selected drugs in wastewater and sewage sludge

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The determination of organic micropollutants of high environmental concern (e.g. carcinogenic and/or toxic and/or endocrine disrupting compounds) in effluent wastewater and sewage sludge is of great importance, above all when these wastewater treatment by-products are recycled for land applications. The assessment of micropollutant concentration levels in these matrices is also of paramount importance in order to investigate the wastewater treatment process, so as to differentiate actual degradation processes from simple matrix transfer phenomena.

Among the various organic micropollutants of high environmental concern, pharmaceuticals have a strong ability to interact with the endocrine systems of human beings and animals. Pharmaceutical compounds, after administration to humans and animals, are excreted as such and/or as metabolites, thus reaching wastewater treatment plants (WTPs) and, in the presence of an incomplete removal, surface waters too [1].

Within pharmaceutical compounds, nonsteroidal anti-inflammatory drugs (NSAIDs) are without doubts among the most utilised in Italy, as well as developed countries [2].

In this study, the determination of 7 common NSAIDs and 6 hydroxylated metabolites in wastewater and sewage sludge from GIDA (Prato, Italy) WTPs, was carried out by QuEChERS extraction [3] coupled with online SPE clean-up and liquid chromatographic-tandem mass spectrometric analysis.

After development and optimization of the method, which involved the selection of the stationary phase and gradient elution, followed by online SPE conditions and H₂O/CH₃CN ratio in the QuEChERS extraction, the analytical protocol was investigated for apparent recovery, source-dependent matrix effect, method detection and quantification limits.

The method was finally applied to the analysis of the 13 target analytes highlighting their presence in wastewater at ng/L levels and in sewage sludge in the concentration range from tens to hundreds µg/kg of dried sludge.

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