

SIMULTANEOUS DETERMINATION OF PHARMACEUTICAL COMPOUNDS IN ENVIRONMENTAL SAMPLES BY SPE AND GC-MS

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Pharmaceuticals are a new class of widespread environmental pollutants. These compounds and their bioactive metabolites can be continually introduced to the aquatic environment as complex mixtures through industrial discharges, effluents from sewage treatment plants (STEP), aquaculture and livestock farming or leaching of landfills. The sewage system is an important key point to control the environmental contamination, but treatment plants are often unable to remove efficiently a substantial part of the pharmaceuticals. Therefore several pharmaceuticals persist in the treated water and contaminate the environment reaching levels ranged from ng L⁻¹ to g L⁻¹. Nevertheless, their presence in the aquatic environment and their impact on aquatic biota and on human health have not been adequately studied yet, although there is some experimental evidence that pharmaceuticals may cause harmful effects such as morphological and metabolic alterations on aquatic species and induction of antibiotic-resistance in aquatic pathogenic bacteria. The purpose of this study was to present a simple procedure for simultaneous determination of ibuprofen, caffeine, diclofenac, paracetamol and ketoprofen at trace level (ng L⁻¹) in environmental samples using Solid Phase Extraction (SPE) preconcentration, followed by derivatization with N-methyl-N-(trimethylsilyl) trifluoroacetamide (MSTFA) and gas chromatography–mass spectrometry (GC–MS) analysis. SPE was carried out with WCX cartridge while extraction volumes were 500 mL for the Waste Water Treatment Plant (WWTP) and 1000 mL for natural water samples. The LODs of individual compounds, determined by calculating the standard deviation of five replicate analyses, ranged from 0.8 ng L⁻¹ (caffeine) to 2.8 ng L⁻¹ (ketoprofen) for tap water, while for wastewater LODs increased by a factor of 10. In order to evaluate the reliability of the method, it has been applied to the analysis of different water samples: surface water taken from Galeso River, Battentieri River and D’Aiedda Channel (Mar Piccolo basin of Taranto); surface sea waters influenced by urbanization (taken from Mar Grande basin of Taranto); wastewater coming from a WWTP effluent (about 100.000 P.E.); drinking water. Results obtained showed that concentrations varied from not detectable value for surface waters to several hundreds of nanograms per liter for WWTP, confirming the suitability of the method for multi-residue analysis of different environmental matrices.

Keywords: pharmaceutical compounds, SPE extraction