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Title: Innovative Sampling and Extraction Methods for the Determination of Nonsteroidal Anti-inflammatory Drugs in Water

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1 **Innovative sampling and extraction methods for the determination of nonsteroidal anti-**
2 **inflammatory drugs in water**

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10 **Abstract**

11 Two different innovative approaches were used for the determination of nonsteroidal anti-
12 inflammatory drugs (NSAIDs) in water: stir bar sorptive extraction (SBSE) and passive
13 sampling, followed by electrospray ionization liquid chromatography-tandem mass
14 spectrometry.

15 SBSE was developed by comparing EG-Silicone and PDMS stir bars and optimizing main
16 parameters to attain high preconcentration. Quantitative analysis was carried out by mass
17 spectrometry in negative ionization mode and multiple reaction monitoring. The SBSE-LC-
18 MS/MS method provided satisfactory figures of merit with LOD (7.5 to 71 ng L⁻¹) and LOQ
19 (22.5-213 ng L⁻¹). The developed method was successfully applied to real samples collected
20 from river water and wastewater effluents. The obtained results showed the presence of all
21 analytes at trace levels, in a wide range of concentrations. The passive sampling approach was
22 carried out by using Polar Organic Chemical Integrative Sampler (POCIS); samplers were
23 deployed for 15 days in river and tap water, allowing to detect analytes at ultra-trace levels. Time
24 Weighted Average concentration of NSAIDs in river water was estimated in the range 0.33-0.46
25 ng L⁻¹, using the sampling rates previously obtained by means of a simple calibration system.

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