

Solid-phase microextraction coupled to liquid chromatography for the analysis of phenolic compounds in water

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Abstract

Solid-phase microextraction (SPME) coupled to high-performance liquid chromatography (HPLC) has been applied to the analysis of priority pollutant phenolic compounds in water samples. Two types of polar fibers [50 μm Carbowax–templated resin (CW–TPR) and 60 μm polydimethylsiloxane–divinylbenzene (PDMS–DVB)] were evaluated. The effects of equilibration time and ionic strength of samples on the adsorption step were studied. The parameters affecting the desorption process, such as desorption mode, solvent composition and desorption time, were optimized. The developed method was used to determine the phenols in spiked river water samples collected in the Douro River, Portugal. Detection limits of 1–10 $\mu\text{g l}^{-1}$ were achieved under the optimized conditions. © 2001 Elsevier Science B.V. All rights reserved.

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1. Introduction

Phenolic compounds are a group of organic pollutants present in the environment as a result of various processes such as industrial, biogeochemical and as pesticide degradation products [1]. Due to their toxicity and persistence, a number of phenolic compounds have been included in the legislation. In this respect, the European Union (EU) has included the phenols cited below in its Directive 76/464/EEC concerning dangerous substances discharged into the aquatic environment: 2-amino-4-chlorophenol, 4-chloro-3-methylphenol, 2-chlorophenol, 3-chlorophenol, 4-chlorophenol, pentachlorophenol and trichlorophenols. The US Environmental Protection

Agency (EPA) list of priority pollutants also includes 11 phenolic compounds. Some of them are included in the EU directive, but others are not, such as 2-nitrophenol, 4-nitrophenol, 2,4-dinitrophenol, 4,6-dinitro-2-methylphenol, 2,4-dichlorophenol, 2,4-dimethylphenol and 2,4,6-trichlorophenol. The Directive 75/440/EEC states that the maximal concentration of phenolic compounds in surface water for drinking purposes should be 1–10 $\mu\text{g l}^{-1}$ [2]. Current official analytical methods, US EPA 604 [3], 625 [4] (acid extractable section) and 8041 [5], are based on liquid–liquid extraction (LLE), followed by gas chromatography (GC) using several detection methods, like electron-capture detection (ECD) and mass spectrometry (MS). However, the direct analysis of phenols by GC is problematic and GC is usually performed after derivatization. Moreover, the need for cleaner procedures led to sample prepara-

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