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Comparative Evaluation of Liquid-Liquid Extraction, Solid-Phase Extraction and Solid-Phase Microextraction for the Gas Chromatography-Mass Spectrometry Determination of Multi-Class Priority Organic Contaminants in Wastewater

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1 **Comparative Evaluation of Liquid-Liquid Extraction, Solid-Phase**
2 **Extraction and Solid-Phase Microextraction for the Gas**
3 **Chromatography-Mass Spectrometry Determination of Multi-**
4 **Class Priority Organic Contaminants in Wastewater**

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8
9 **Abstract**

10 The European Water Framework Directive (WFD) 2000/60/EC establishes guidelines to
11 control the pollution of surface water by sorting out a list of priority substances that
12 involves a significant risk to or via the aquatic systems. In this article, the analytical
13 performance of three different sample preparation methodologies for the GC-MS/MS
14 determination of multi-class organic contaminants -including priority compounds from
15 the WFD- in wastewater samples using gas chromatography-mass spectrometry was
16 evaluated. The methodologies tested were: a) liquid-liquid extraction (LLE) with n-
17 hexane; b) solid-phase extraction (SPE) with C₁₈ cartridges and elution with ethyl
18 acetate:dichloromethane (1:1 (v/v)), and c) headspace solid-phase microextraction (HS-
19 SPME) using two different fibers: polyacrylate and
20 Polydimethylsiloxane/Carboxen/Divinilbenzene. Identification and confirmation of the
21 selected 57 compounds included in the study (comprising polycyclic aromatic
22 hydrocarbons (PAHs), pesticides and other contaminants) was accomplished using gas
23 chromatography tandem mass spectrometry (GC-MS/MS) with a triple quadrupole
24 instrument operated in the multiple reaction monitoring (MRM) mode. Three MS/MS
25 transitions were selected for unambiguous confirmation of the target chemicals. The
26 different advantages and pitfalls of each method were discussed. In the case of both

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