

固相微萃取-气相色谱法测定白洋淀水样中的邻苯二甲酸酯类化合物

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Determination of phthalate esters in Baiyangdian lake by solid phase microextraction and gas chromatography

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摘要

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摘要 建立了固相微萃取(SPME)-气相色谱法(GC)分析环境水样中痕量邻苯二甲酸酯类化合物(PAEs)的方法。选用100 μm 聚二甲基硅氧烷(PDMS)萃取纤维,在磁力搅拌条件下,对水样中的PAEs萃取富集60 min,然后直接注入GC进样口,在250 $^{\circ}\text{C}$ 温度下解吸4 min后进行PAEs的分离。方法的重现性(以相对标准偏差(RSD)计)为0.2%~9.7%,检出限为0.02~0.83 $\mu\text{g/L}$ 。将本方法应用于白洋淀水样中PAEs的分析检测发现,样品中邻苯二甲酸二异丁酯(DIBP)、邻苯二甲酸二丁酯(DBP)、邻苯二甲酸二(2-乙基己基)酯(DEHP)含量较高。对水样进行两个浓度水平(2.5 $\mu\text{g/L}$ 和5.0 $\mu\text{g/L}$)的加标试验,加标回收率为75.3%~111.0%,RSD为2.1%~8.0%(n=3),满足痕量PAEs的测定要求。

关键词: 固相微萃取 气相色谱 邻苯二甲酸酯类化合物 白洋淀

Abstract: A simple method based on solid-phase microextraction (SPME) coupled with gas chromatography-flame ionization detection (GC-FID) was developed for the determination of trace amounts of phthalate esters (PAEs) in environmental water samples. In this method, polydimethylsilane (PDMS) fiber was chosen to enrich the PAEs. PAEs were extracted for 60 min using the PDMS fiber under stirring with a magnetic stir bar, after that the fiber was introduced into the GC injector port and the extract was desorbed at 250 $^{\circ}\text{C}$ for 4 min. Under the optimized conditions, 13 PAEs can be extracted completely and separated well. The limits of detection (LOD) were from 0.02 to 0.83 $\mu\text{g/L}$ (S/N=3). The water samples collected from Baiyangdian lake were successfully analyzed using the proposed method. Phthalic acid, bis-iso-butyl ester (DIBP), phthalic acid, bis-butyl ester (DBP) and phthalic acid, bis-2-ethylhexyl ester (DEHP) were detected in all the samples. The spiked (2.5 $\mu\text{g/L}$ and 5.0 $\mu\text{g/L}$) recoveries were in the range of 75.3%~111.0% and the relative standard deviations (RSDs) were between 2.1% and 8.0% (n=3). The results show that this would be a valuable method for PAEs analysis in environmental water samples.

Keywords: solid-phase microextraction (SPME) gas chromatography (GC) phthalate esters (PAEs) Baiyangdian

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