

快速溶剂萃取-气相色谱-三重四极杆质谱法测定沉积物中的酞酸酯

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Determination of phthalate esters in sediment by accelerated gas chromatography-triple quadrupole mass spectrometry

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摘要 建立了快速溶剂萃取(ASE)-气相色谱-三重四极杆质谱(GC-MS/MS)测定沉积物中酞酸酯的方法。样品用二氯甲烷-丙酮(体积比1:1)在100 ℃、103.4 MPa (1500 psi)条件下经快速溶剂萃取、以5 mL/min的速率经凝胶渗透色谱(GPC)净化去除大分子干扰物,并用GC-MS/MS分析测定。采用内标法定量,17种酞酸酯的检出限为0.05~0.40 μg/kg;回收率为50.5%~107.9%,相对标准偏差为3.5%~13.9%。采用替代物基体加入法对方法的性能进行了验证,3种替代物的回收率为65.3%~95.8%。该方法快速、灵敏度高,能同时准确测定沉积物中多种酞酸酯。

关键词: 快速溶剂萃取 气相色谱-三重四极杆质谱 酞酸酯 沉积物

Abstract: A method for the determination of seventeen phthalate esters in sediment by accelerated solvent extraction (ASE), gel-permeation chromatography (GPC) and gas chromatography-triple quadrupole mass spectrometry (GC-MS/MS) has been developed. The target compounds were extracted at 100 °C and 103.4 MPa (1500 psi) by ASE using mixtures of dichloromethane and acetone (1:1, v/v) as solvent. In order to eliminate the interferences from large molecular sizes, the extract was purified at a flow rate of 5.0 mL/min by GPC. Following that, the extract was concentrated to a final volume of 1 mL exactly. The GC-MS/MS was applied to quantitative and qualitative analysis. Internal standard calibration approach was adopted, and the detection limits of seventeen phthalate esters ranged from 0.05 to 0.40 μg/kg were obtained. The correlation coefficients were beyond 0.996, the recoveries were from 50.5% to 107.9%, and the relative standard deviations were from 3.5% to 13.9%. Besides, the surrogate compound were used to monitor the performance of the method, and the recoveries were from 65.3% to 95.8% for the 3 surrogate compounds. The method is fast, sensitive and exact for analyzing seventeen phthalate esters simultaneously.

Keywords: accelerated solvent extraction (ASE) gas chromatography-triple quadrupole mass spectrometry (GC-MS/MS) phthalate esters sediments

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