

同位素稀释气相色谱-质谱法测定土壤/底泥中23种有机氯农药

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Determination of organochlorine pesticide residues in soil/sediment by isotope dilution gas chromatography-mass spectrometry

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摘要

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摘要 建立了同位素稀释气相色谱-质谱法(ID-GC-MS)测定土壤/底泥中23种有机氯农药残留的分析方法。样品经干燥、研磨、筛分、250 mL丙酮-正己烷(体积比为1:1)混合溶剂进行索氏提取24 h,再经铜粉、凝胶渗透色谱和弗罗里硅土固相萃取柱净化,最后用DB-1701柱分离和电子轰击电离源质谱在选择离子监测模式下进行检测,并用¹³C标记同位素稀释内标法定量。实验结果表明,23种化合物的回收率良好,线性范围为50~1600 μg/L,同位素内标的回收率范围为60%~110%,相对标准偏差为1.5%~18%;方法的检出限为10.3 μg/kg。该方法的净化效果较好,定量准确,适用于土壤/底泥这类复杂基质中农药残留的确证分析。

关键词: 同位素稀释气相色谱-质谱 有机氯农药 土壤 底泥

Abstract: A method was developed for the determination of organochlorine pesticide (OCP) residues in soil/sediment using high resolution gas chromatography coupled with low resolution mass spectrometry. The analytical procedure consisted of Soxhlet extraction, sulfur removal with copper powder, clean-up with gel permeation chromatography and a florisil column of solid phase extraction (SPE). The analytes were separated on an HP-5MS capillary column and detected in selected ion monitoring (SIM) mode and quantified using internal standard calibration curves of isotope dilution technique. The linear correlations of calibration standard solutions were good for all the OCPs. The recovery and relative standard deviations of labeled compound solutions ranged from 60% to 110% and from 1.5% to 18%, respectively. The limits of detection ranged from 0.20 to 10.3 μg/kg were established for the 23 OCPs. The method showed satisfactory clean-up effect and precision quantification. It is suitable for the determination and confirmation of pesticides in complex matrices such as soil, sediment.

Keywords: isotope dilution gas chromatography-mass spectrometry (ID-GC-MS) organochlorine pesticides soil sediment

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