

## 同位素稀释高分辨气相色谱-高分辨质谱法测定土壤中痕量有机氯农药残留

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### Determination of trace organochlorine pesticides in soil using resolution gas chromatography-high resolution mass spectror

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摘要

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**摘要** 建立了同位素稀释高分辨气相色谱-高分辨质谱测定土壤中痕量有机氯农药残留的分析方法。采用加速溶剂法萃取土壤样品,以(体积比为1:1)为提取溶剂,提取液经过Florisil硅土固相萃取小柱净化后,采用高分辨DB-5MS毛细管色谱柱(30 m×0.25 mm×0.25 μm)以保留时间和同位素特征离子丰度比定性,同位素峰面积比定量。实验结果表明,方法的回收率为77.3%~114.5%,相对标准偏差(RSD)≤10.81% (n=5),检出限均小于0.04 pg/g。应用该方法检测某地区表层土壤中的有机氯农药,结果表明该方法适合测定环境土壤有机氯残留。

**关键词:** 高分辨气相色谱-高分辨质谱 同位素稀释 有机氯农药 土壤

**Abstract:** A method for the determination of trace organochlorine pesticides (OCPs) in soil using isotope dilution high resolution gas chromatography-high resolution mass spectrometry (ID-HRGC-HRMS) was developed. The sample was extracted by accelerated solvent extractor (ASE) and cleaned-up by a Florisil solid phase extraction (SPE). The analytes were separated by HRGC on a DB-5MS column (30 m×0.25 mm×0.25 μm) and determined by HRMS. The identifications of OCPs were based on the retention time of <sup>13</sup>C-labelled standard and the abundance ratio of exact mass-to-charge ratios. The quantitative analysis was performed using the ratios of the integrated area of <sup>13</sup>C-labelled standards. This method has the recoveries ranging from 77.3% to 114.5% and the relative standard deviations (RSD) less than 10.81% (n=5). The limits of detection (LODs) of this method for all OCPs were lower than 0.04 pg/g. The results indicated that the method is rapid, selective and sensitive for precise determination of organochlorine pesticides at trace level in soil.

**Keywords:** high resolution gas chromatography-high resolution mass spectrometry (HRGC-HRMS) isotope dilution organochlorine pesticide soil

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