

## 同位素稀释-气相色谱-串联质谱法测定土壤中的指示性毒杀芬

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中国科学院知识创新工程重要方向项目(No. KZCX2-YW-JS406)和环保公益性行业科研专项项目(No.200909096).

## Determination of indicator toxaphene in soil by isotope dilution tandem mass spectrometry

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摘要	参考文献	相关文章
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**摘要** 建立了土壤样品中指示性毒杀芬Parlar No.26 (P26)、Parlar No.50 (P50)和Parlar No.62 (P62)的同位素稀释-气相色谱(ID-GC-MS/MS)的分析方法。土壤样品使用压力溶剂萃取装置(PLE)提取,以丙酮-正己烷(1:1, v/v)混合溶液为提取溶剂;提取液依性硅胶柱和活化硅胶柱净化;洗脱液经氮吹浓缩至20  $\mu$ L后,利用GC-MS/MS的多反应监测(MRM)模式进行定性和定量。结果表明本方法中的P26、P50和P62进行分析,相对标准偏差(RSD)小于11%,回收率可以达到55%~110%;P26、P50和P62的仪器检出限分别为6.0  $\mu$ g。将该方法用于某地区农田表层土壤中3种指示性毒杀芬的检测,其中P26的含量为0.17 ng/g、P50为0.08 ng/g、P62为0.17 ng/g。此方法适用于土壤样品中指示性毒杀芬的分析。

**关键词:** 同位素稀释法 气相色谱 串联质谱 指示性毒杀芬 土壤

**Abstract:** Although toxaphene is now banned in use, the analysis of toxaphene has attracted increasing interest due to its persistence and widespread atmospheric transport in the environment. A new method based on isotope dilution gas chromatography-tandem mass spectrometry (ID-GC-MS/MS) has been developed for the determination of toxaphene specific congeners comprised of Parlar No. 26 (P26), Parlar No. 50 (P50) and Parlar No. 62 (P62) in soil. A  $^{13}\text{C}_{10}$ -labeled indicator toxaphene solution was added to the sample prior to pretreatment. Then the sample was extracted using pressurized liquid extraction (PLE) followed by purification on multilayer acidic silica column and silica column. The eluent was concentrated under gentle nitrogen gas flow and spiked with the injection of internal standard of  $^{13}\text{C}_{10}$ -chlordane. Identification and quantification of the analytes were carried out in the multiple reaction monitoring (MRM) mode after the GC separation. The linear range was 20~800  $\mu\text{g/L}$  for three congeners, limit of detection (LOD) ranged from 3.0 to 6.0  $\mu\text{g}$ . The five point calibration curves showed a good linearity for all the congeners ( $R^2 > 0.99$ ). The relative standard deviations (RSDs) were below 11% for and the spiked recoveries were in the range of 55%~110%. The developed analytical method is suitable for the determination of toxaphene specific congeners in soil.

**Keywords:** isotope dilution gas chromatography (GC) tandem mass spectrometry (MS/MS) indicator toxaphene soil

Received 2010-01-14; published 2010-05-28

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引用本文: