色谱 2007 25 (2): 262-266 ISSN: 1000-8713 CN: 42-1226/O

研究论文

气相色谱-负离子化学源-质谱法分析深海鱼油食品中五种多溴联苯醚残留

林竹光[1];涂逢樟[2];马 玉[1];陈美瑜[1];张莉莉[1];孙若男[1];李小波[1];陈招斌[1]

厦门大学化学化工学院化学系分析化学教研室[1]

龙岩学院化学与材料工程系[2]

收稿日期 2006-9-14 修回日期 2006-12-9 网络版发布日期 2007-3-30 接受日期

摘要 摘要 建立了气相色谱-负离子化学源-质谱法(GC-NCI-MS)同时分析深海鱼油食品中5种多溴联苯醚(PBDEs)残留的分析方法。深海鱼油食品用正己烷超声提取、中性和酸性硅胶层析柱净化和正己烷洗脱后，以PCB-103为内标物，采用GC-NCI-MS的选择离子监测方式(SIM)分析；同时探讨了5种PBDEs NCI-MS特征离子的断裂机理。当深海鱼油食品空白的加标质量浓度为20和100 μg/kg时，加标回收率为88.6%~111.3%，相对标准偏差为3.8%~13.5%，方法检测限为0.77~1.34 μg/kg，线性范围为1~500 μg/kg，相关系数皆大于0.9992，此方法成功地应用于深海鱼油食品中5种痕量PBDEs残留的同时分析。

关键词

分类号

Multiresidue Determination of Polybrominated Diphenyl Ethers (PBDEs) in Fish Oil by Gas Chromatography-Mass Spectrometry

Abstract

Abstract: An analytical multiresidue method for the simultaneous determination of 5 polybrominated diphenyl ethers (PBDEs) in fish oil was developed. PBDEs were extracted from fish oil with hexane and cleaned up on an acid silical gel column, then were determined by using a gas chromatography- mass spectrometry operated in negative chemical ionization mode and quantified in selective ion monitoring mode, and with PCB103 as internal standard. Meanwhile, fracture mechanism of some PBDEs negative chemical ionization mass spectrometry were evaluated. Recovery studies were performed at 20 and 100 μg/kg fortification levels for each PBDEs, and the recoveries ranged from 88.6% to 111.3% with a relative standard deviation between 3.8% and 13.5% for the different PBDEs. The developed method was linear over the range assayed, 1~500 μg/L, with determination coefficients >0.9992. Finally, the developed analytical method has been successfully applied to the determination of PBDEs in several fish oil samples.

Key words

DOI:

通讯作者 林竹光 linzg@xmu.edu.cn