

研究论文

气相色谱-质谱/质谱法检测蔬菜中的毒死蜱及其代谢物

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摘要 建立了蔬菜中毒死蜱及其代谢物3, 5, 6-三氯-2-羟基吡啶 (3, 5, 6-trichloro-2-pyridinol, TCP)的气相色谱-质谱/质谱分析方法。蔬菜样品采用丙酮提取, 浓缩后TCP用N-甲基-N-叔丁基二甲基硅基三氟乙酰胺 (MTBSTFA) 衍生, 再经氟罗里硅土固相萃取柱净化。采用三重四极杆质谱电子轰击多反应监测 (MRM) 模式测定。采用内标法对毒死蜱定量, 方法的检出限为1 $\mu\text{g}/\text{kg}$, 加标回收率为75.57%~106.41%, 相对标准偏差 (RSD) 为8.33%~17.58%。采用外标法对TCP定量, 方法的检出限为0.5 $\mu\text{g}/\text{kg}$, 加标回收率为69.11%~108.43%, RSD为5.20%~19.42%。在2~100 $\mu\text{g}/\text{L}$ 范围内, 两种被测物的线性关系良好 ($r > 0.99$)。该方法可用于蔬菜中毒死蜱及其代谢物的检测。

关键词 [气相色谱-质谱/质谱法](#); [毒死蜱](#) [代谢物](#) [蔬菜](#)

Determination of chlorpyrifos and its metabolite in vegetables using gas chromatography-tandem mass spectrometry

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Abstract

A method was developed for the determination of chlorpyrifos and its metabolite 3,5,6-trichloro-2-pyridinol (TCP) in vegetables using gas chromatography-tandem mass spectrometry (GC-MS/MS). The sample was extracted with acetone. After the concentration, TCP was derivatized with N-(tert-butyl dimethylsilyl)-N-methyltrifluoroacetamide (MTBSTFA). The purification was performed using a florisil solid-phase extraction cartridge. The analyte was detected by triple quadrupole MS in multi-reaction monitoring mode. An internal standard method was used for the quantification of chlorpyrifos, the limit of quantification was 1 $\mu\text{g}/\text{kg}$, and the recoveries were in the range of 75.57%-106.41% with the relative standard deviations (RSDs) of 8.33%-17.58%. An external standard method was used for the quantification of TCP, the limit of quantification was 0.5 $\mu\text{g}/\text{kg}$, and the recoveries were in the range of 69.11%-108.43% with the RSDs of 5.20%-19.42%. The good linear relationships were obtained in the range of 2-100 $\mu\text{g}/\text{L}$ for the both target analytes. The method can be applied for the determination of chlorpyrifos and its metabolite in vegetables.

Key words [gas chromatography-tandem mass spectrometry \(GC-MS/MS\)](#) [chlorpyrifos](#) [metabolite](#) [vegetables](#)

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