

液相色谱-串联质谱法快速测定蜂蜜中3种硝基咪唑类药物残留

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Rapid determination of 3 nitromidazole residues in honey using tandem mass spectrometry

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摘要

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摘要 建立了液相色谱-串联质谱(LC-MS/MS)快速测定蜂蜜中甲硝唑、洛硝哒唑和二甲硝咪唑3种硝基咪唑类药物残留的分析方法。溶解后,直接上样至Oasis HLB固相萃取柱净化,依次用水和甲醇-水溶液(1:9, v/v)淋洗,用乙酸乙酯洗脱。洗脱液经浓缩、溶解、过MS/MS检测,外标法定量。在添加水平为0.05~2.0 µg/kg时,平均添加回收率为76.6%~89.7%,相对标准偏差(n=8)为5.2%~9.9%。甲硝唑的检出限为0.1 µg/kg,洛硝哒唑、二甲硝咪唑的检出限均为0.2 µg/kg。应用所建立的方法对出口蜂蜜样品进行了测定,结果表明简单、快速,结果准确、可靠,灵敏度和准确度满足现在日本和欧盟对蜂蜜中3种硝基咪唑类药物残留量的检测要求。

关键词: 液相色谱-串联质谱法 甲硝唑 洛硝哒唑 二甲硝咪唑 蜂蜜

Abstract: A method was developed for the rapid determination of 3 nitromidazole residues in honey, including metronidazole, ronidazole and dimetridazole, using liquid chromatography-tandem mass spectrometry (LC-MS/MS). The honey sample was dissolved in water, and the solution was cleaned up with an Oasis HLB cartridge. The cartridge was washed with water and methanol-water solution (1:9, v/v) in turn, and eluted with ethyl acetate. The solution was concentrated and then analyzed by LC-MS/MS. External calibration was used for quantitative determination. The recoveries and relative standard deviations (n=8) were from 76.6% to 89.7% and 5.2% to 9.9%, at the spike level of 0.05-2.0 µg/kg, respectively. The limits of detection were 0.1 µg/kg for metronidazole, and 0.2 µg/kg for ronidazole and dimetridazole. The method was successfully applied for the inspection of exported honey. With simple and fast sample preparation, the method is sensitive and specific for the determination. The sensitivity and accuracy of the method meet the requirements of the inspection for the 3 nitromidazole residues in honey in Japan and European Union.

Keywords: liquid chromatography-tandem mass spectrometry (LC-MS/MS) metronidazole ronidazole dimetridazole honey

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