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研究论文

同位素内标稀释液相色谱-串联质谱法测定鱼贝类组织中残留的环丙氟哌酸

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摘要 建立了不同鱼贝类肌肉组织中以氘代同位素为内标测定环丙氟哌酸残留量的液相色谱-串联质谱(LC-MS/MS)方法。样品加入内标环丙氟哌酸-D8和磷酸盐缓冲溶液(pH 7.0)后进行匀质并用乙腈超声提取, 经正己烷脱脂后采用Waters Oasis MAX小柱净化, 在Cloversil-C18柱上, 以乙腈-0.05%三氟醋酸(体积比为25∶75)为流动相, 采用多反应监测(MRM)模式, 液相色谱-电喷雾质谱法测定。根据环丙氟哌酸和氘代内标物的定量离子质量色谱图的峰面积比值, 采用内标法定量。结果表明, 环丙氟哌酸和内标的定量离子峰面积比值与环丙氟哌酸浓度在0.1～50.0 μg/kg范围内呈现良好的线性关系, 相关系数为0.9991, 方法的定量检测限为0.1 μg/kg, 回收率为92.5%～98.1%, 相对标准偏差(RSD)小于4.3%。将该方法用于市场上10种鱼和贝类样品的检测, 结果表明该法具有灵敏、准确的优点, 完全满足残留分析的确证检测要求。

关键词 固相萃取 液相色谱-串联质谱法 同位素内标 环丙氟哌酸 鱼贝类组织

Determination of ciprofloxacin residue in fish/shellfish tissues using liquid chromatography-tandem mass spectrometry with isotope internal standard dilution technique

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Abstract

Using isotope internal standard dilution technique, a high performance liquid chromatography-tandem mass spectrometry (HPLC-MS/MS) method has been developed for the identification and quantitative determination of ciprofloxacin residue in the tissues of various fishes/shellfishes. The homogenized tissue sample added with ciprofloxacin-D8 and phosphate buffer solution (pH 7.0) was extracted with acetonitrile under ultrasonication, and degreased with hexane. After solid-phase extraction (SPE) was performed on an Oasis MAX cartridge, the sample was separated on a Cloversil-C18 column (150 mm×4.6 mm, 5 μm) by using the mobile phase consisting of CH3CN-0.05%CF3COOH (25∶75, v/v). The detection was carried out by LC-MS/MS using an electrospray ionization interface in multiple reaction monitoring (MRM) mode. The quantification using isotope-labelled internal standard was based on the peak area ratio of ciprofloxacin and deuterated internal standard in the MRM mode. The calibration curve was linear within the range of 0.1-50.0 μg/kg and the limit of quantification was 0.1 μg/kg (S/N≥10). The recovery was between 92.5% and 98.1%, and the relative standard deviation was less than 4.3%. The application of this method was further demonstrated by analyzing ten various real samples from local markets. The results show that this method is sensitive, accurate and suitable for the confirmative determination of ciprofloxacin residues.

Key words solid-phase extraction （SPE） liquid chromatography-tandem mass spectrometry (LC-MS/MS) isotope internal standard ciprofloxacin fish/shellfish tissues

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