

研究论文

超高效液相色谱-串联质谱法测定牛奶和奶粉中残留的左旋咪唑

薄海波^{1*}, 庞国芳², 雒丽丽³, 曹彦忠²

1.青海出入境检验检疫局, 青海 西宁 810000; 2.秦皇岛出入境检验检疫局, 河北 秦皇岛 066002;
3.甘肃农业大学, 甘肃 兰州 730070

收稿日期 2008-8-26 修回日期 2008-10-27 网络版发布日期 2009-3-27 接受日期 2008-10-30

摘要 建立了一种专属、灵敏的超高效液相色谱-串联质谱(UPLC-MS/MS)测定牛奶和奶粉中左旋咪唑残留量的方法。在碱性环境下用乙酸乙酯超声波提取试样中的左旋咪唑,再经稀盐酸反提取、强阳离子交换(SCX)固相萃取柱净化,采用BEHC18超高效液相色谱柱、乙腈-0.1%甲酸(体积比为15:85)流动相分离,以电喷雾离子源正离子检测方式进行质谱分析。实验结果表明,在2.0~100.0 μg/L浓度范围内左旋咪唑呈良好的线性关系,其相关系数 $r=0.997$ 。在低、中、高3个浓度添加水平下,左旋咪唑的回收率为64.5%~102.0%,相对标准偏差小于13.1%。牛奶中左旋咪唑检出限为0.4 μg/kg,奶粉中左旋咪唑检出限为2.0 μg/kg。

关键词 [超高效液相色谱-串联质谱法](#) [强阳离子交换固相萃取](#) [左旋咪唑](#) [牛奶](#) [奶粉](#)

Determination of levamisole residue in milk and milk powder by ultra performance liquid chromatography-tandem mass spectrometry

BO Haibo^{1*}, PANG Guofang², LUO Lili³, CAO Yanzhong²

1.Qinghai Entry and Exit Inspection and Quarantine Bureau, Xining 810000, China; 2.Qinhuangdao Entry and Exit Inspection and Quarantine Bureau, Qinhuangdao 066002, China; 3.Gansu Agricultural University, Lanzhou 730070, China

Abstract

A sensitive and selective method is presented for the determination of levamisole residues in milk and milk powder by ultra performance liquid chromatography-tandem mass spectrometry (UPLC-MS/MS). Levamisole in the test samples was extracted with ethyl acetate under alkaline condition, then transferred into dilute hydrochloric acid, and cleaned-up with a strong cation exchange (SCX) column to obtain an extract suitable for the analysis by UPLC-MS/MS. Levamisole was separated by a BEHC18 UPLC column and acetonitrile-1.0% formic acid (15:85, v/v) as mobile phase, and electrospray ionization was applied and operated in the positive ion mode. The calibration curve was good linear between the peak area and the mass concentration of levamisole from 2.0 to 100.0 μg/L with the correlation coefficient of 0.997. The average recoveries of levamisole spiked in milk and milk powder samples at the three concentrations ranged from 64.5% to 102.0% with the relative standard deviations less than 13.1%. The detection limits of levamisole were 0.4 μg/kg in milk and 2 μg/kg in milk powder.

Key words [ultra performance liquid chromatography-tandem mass spectrometry \(UPLC-MS/MS\)](#) [strong cation exchange solid-phase extraction](#) [levamisole](#) [milk](#) [milk powder](#)

DOI:

通讯作者 薄海波 boh1212@163.com

扩展功能

本文信息

- ▶ [Supporting info](#)
- ▶ [PDF\(292KB\)](#)
- ▶ [\[HTML全文\]\(0KB\)](#)
- ▶ [参考文献](#)

服务与反馈

- ▶ [把本文推荐给朋友](#)
- ▶ [加入我的书架](#)
- ▶ [加入引用管理器](#)
- ▶ [复制索引](#)
- ▶ [Email Alert](#)

相关信息

- ▶ [本刊中包含“超高效液相色谱-串联质谱法”的相关文章](#)
- ▶ [本文作者相关文章](#)

- [薄海波](#)
- [庞国芳](#)
- [雒丽丽](#)
- [曹彦忠](#)