

液相色谱-串联质谱法快速测定婴幼儿配方奶粉中39种激素残留量

祝伟霞, 刘亚凤*, 袁萍, 杨冀州

河南出入境检验检疫局, 河南 郑州 450003

Quick determination of 39 hormones residues in infant formula by liquid chromatography-tandem mass spectrometry

ZHU Weixia, LIU Yafeng*, YUAN Ping, YANG Jizhou

Henan Entry-Exit Inspection and Quarantine Bureau, Zhengzhou 450003, China

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摘要 建立了液相色谱-串联四极杆质谱同时测定婴幼儿配方奶粉中17种糖皮质激素、11种孕激素、3种雄性激素和8种雌激素残留的快速确证方法。采用乙腈提取奶粉中待测组分,提取液经冷冻离心与正己烷除脂、亲水-亲脂平衡固相萃取柱净化、甲醇洗脱。分别在正、负电喷雾离子化多反应监测模式下检测39种激素。正离子模式下的流动相为乙腈-0.1%甲酸,色谱柱为普通硅胶基质的C18柱;负离子模式下的流动相为乙腈-0.1%氨水,色谱柱为能耐受宽pH范围的超高效C18柱。在该优化条件下,39种激素定量限(S/N≥10)为0.02~5 μg/kg,方法回收率为59.5%~117.9%,相对标准偏差(RSD)为6.4%~16.3%。经测定多种市售婴幼儿配方奶粉,表明该方法操作简单、测定结果准确,可用于婴幼儿配方奶粉中多种内源性化学合成类激素残留的快速测定。

关键词: 液相色谱-串联质谱 固相萃取 冷冻除脂 激素 婴幼儿奶粉

Abstract: A quick confirmative method was developed for determining the residues of 17 glucocorticoids, 11 progesterones, 3 androgens and 8 estrogens in infant formula by liquid chromatography-tandem quadrupole mass spectrometry (LC-MS/MS). The sample was extracted with acetonitrile at first. Then the lipid substances were removed by centrifugation under freezing condition and liquid-liquid extraction with hexane of the extract. The purification was carried out on hydrophilic-lipophilic solid-phase extraction columns and methanol was used as the eluted solvent. The detection of 39 analytes was carried out in the positive or negative multi-reaction monitoring (MRM) mode, separately. Acetonitrile-0.1% formic acid was used as the mobile phase and an ordinary silica gel C18 column was selected to separate the analytes in the positive mode. Acetonitrile-0.1% aqueous ammonia as mobile phase and the separation was carried out on an ultra-performance C18 column with a wide pH range in the negative mode. The limits of quantification (LOQ, S/N≥10) were 0.02~5 μg/kg. The overall recoveries varied from 59.5% to 117.9%, and the relative standard deviations (RSD) were between 6.4% and 16.3%. The real sample tests showed that the simple and accurate method can be used for determining the residues of multi-endogenous and chemically synthesized hormones in milk powders.

Keywords: liquid chromatography-tandem mass spectrometry (LC-MS/MS) solid-phase extraction (SPE) frozen delipidation hormones infant formula

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Corresponding Authors: 刘亚凤,高级工程师. Email: wxzhu121@163.com

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