

柱后氨化-超高效液相色谱-串联质谱法测定牛奶和奶粉中的三聚氰酸

云环*, 张朝晖, 高洋洋, 何悦

北京出入境检验检疫局技术中心, 北京 100026

Determination of cyanuric acid in milk and milk powder by ultra performance liquid chromatography-tandem mass spectrometry with post-column ammoniation

YUN Huan*, ZHANG Zhaohui, GAO Yangyang, HE Yue

Technology Center of Beijing Entry-Exit Inspection and Quarantine Bureau, Beijing 100026, China

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摘要 建立了一种柱后氨化-超高效液相色谱-串联质谱法(UPLC-MS/MS)测定牛奶和奶粉中三聚氰酸的分析方法。样品用5%三氯乙酸溶液提取, 无需净化和浓缩,用Acquity UPLC HSS T3色谱柱(50 mm×2.1 mm, 1.8 μm)进行分离,以乙腈和水作为流动相进行梯度洗脱,柱后氨化,电喷雾负离子(ESI⁻)模式电离,多反应监测(MRM)模式进行检测。结果表明: 三聚氰酸在10.0~160.0 μg/L范围内线性关系良好($r \geq 0.99$)。牛奶在0.05、0.10、0.20 mg/kg的添加浓度的回收率为60%~118.5%,相对标准偏差(RSD, n=6)为8.2%~12.6%;奶粉在0.25、0.50、1.00 mg/kg的添加浓度的回收率为88%~108%, RSD(n=6)为2.5%~5.7%。牛奶中方法的定量限(信噪比为10)为0.05 mg/kg,奶粉中为0.25 mg/kg。该方法快速、灵敏、准确,适合于牛奶、奶粉等乳及乳制品中三聚氰酸的快速、高灵敏度的分析检测。

关键词: 柱后氨化 超高效液相色谱-串联质谱 三聚氰酸 牛奶 奶粉

Abstract: A method for the determination of cyanuric acid in milk and milk powder has been developed by ultra performance liquid chromatography-tandem mass spectrometry (UPLC-MS/MS) with post-column ammoniation. After extracted by 5% trichloroacetic acid, the samples were loaded onto an Acquity UPLC HSS T3 column (50 mm×2.1 mm, 1.8 μm) and separated with a gradient elution. The electrospray was operated in the negative mode and monitored by the multiple reaction monitoring (MRM) mode. The calibration curves showed a good linearity in the range of 10.0~160.0 μg/L, and the correlation coefficients (r) were higher than 0.99. When the spiked levels were 0.05, 0.10, and 0.20 mg/kg, the recoveries of cyanuric acid in milk ranged from 60% to 118.5%, with the relative standard deviations (RSDs) of 8.2%~12.6%; while the spiked levels were 0.25, 0.50, and 1.00 mg/kg in the milk powder, the recoveries ranged from 88% to 108% and the RSDs ranged from 2.5%~5.7%. The limits of quantification (LOQs, S/N=10) of cyanuric acid were 0.05 mg/kg in milk sample and 0.25 mg/kg in milk powder. The results indicate that the method is simple, rapid, sensitive and suitable for the qualitative and quantitative analyses of cyanuric acid in milk and milk powder samples.

Keywords: post-column ammoniation ultra performance liquid chromatography-tandem mass spectrometry (UPLC-MS/MS) cyanuric acid milk milk powder

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Corresponding Authors: 云环,工程师,主要研究方向为食品中药物残留的分析. Email: yunh@bjciq.gov.cn

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