

技术与应用

## 气相色谱-质谱法快速测定牛奶中的三聚氰胺和三聚氰酸

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**摘要** 建立了气相色谱-质谱法测定牛奶中三聚氰胺和三聚氰酸含量的分析方法。样品经二乙胺-乙腈-水溶液超声提取, 离心, 滤液用氮气吹干后加入硅烷化衍生试剂衍生, 气相色谱-质谱测定, 选择离子模式监测, 外标法定量。在0.025~2 mg/kg范围内, 目标物的峰面积与其质量浓度的线性关系良好( $r > 0.98$ ); 在0.5, 1.0和2.5 mg/kg的添加水平, 三聚氰胺和三聚氰酸的平均回收率分别为84%~87%和75%~102%, 相对标准偏差(RSD)分别为5.7%~11.7%和4.9%~7.8%; 三聚氰胺和三聚氰酸的检出限(LOD)分别为0.05 mg/kg和0.10 mg/kg。结果表明: 该方法简便、快速、灵敏、准确, 适合牛奶中三聚氰胺和三聚氰酸的确证和定量测定。

**关键词** [气相色谱-质谱](#) [三聚氰胺](#) [三聚氰酸](#) [牛奶](#)

## Determination of melamine and cyanuric acid in milk by gas chromatography-mass spectrometry

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### Abstract

A gas chromatography-mass spectrometry method was developed for the quantitative determination and confirmation of melamine and cyanuric acid in milk. The milk sample was extracted with diethylamine-acetonitrile-water solution. The extract was evaporated to dryness and derivatized with N,O-bis(trimethylsilyl)trifluoroacetamide (BSTFA) and chlorotrimethylsilane (TMCS), then analyzed using gas chromatography-mass spectrometry (GC-MS) in selected ion monitoring (SIM) mode. The external standards were used for the quantitative determination. The linear range was from 0.025 to 2 mg/kg. The average recoveries were 84%-87% for melamine and 75%-102% for cyanuric acid, and the relative standard deviations were 5.7%-11.7% for melamine and 4.9%-7.8% for cyanuric acid in the spiked levels at 0.5, 1.0 and 2.5 mg/kg. The limits of detection of melamine and cyanuric acid were 0.05 mg/kg and 0.10 mg/kg, respectively. The method is suitable for the quantitative determination and confirmation of melamine and cyanuric acid residues in milk.

**Key words** [gas chromatography-mass spectrometry \(GC-MS\)](#) [melamine](#) [cyanuric acid](#) [milk](#)

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