

## Simultaneous Quantitative Determination of Melamine and Cyanuric Acid in Cow's Milk and Milk-Based Infant Formula by Liquid Chromatography–Electrospray Ionization Tandem Mass Spectrometry

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An isotope dilution liquid chromatography–electrospray ionization tandem mass spectrometry method for the simultaneous determination of melamine and cyanuric acid in cow's milk (range of 0–0.3 mg/kg) and milk-based infant formulas (ranges of 0–0.3 and 0–2.0 mg/kg) is described. This quantitative method entails simple sample preparation, limited to a protein precipitation in acetonitrile/water followed by a centrifugation and direct injection of the supernatant. Selected reaction monitoring of two diagnostic transition reactions for each analyte and each corresponding ( $^{13}\text{C}_3, ^{15}\text{N}_3$ )-labeled compound enables selective and confirmatory detection. Acquisition was performed sequentially in the negative ion mode for cyanuric acid, while in the positive mode for melamine within the same run. Validation of the method was conducted according to European Union criteria (CD 2002/657/EC). Internal standard-corrected recoveries were within the 99–116% range for both analytes in the two matrix types, along with repeatability and intermediate reproducibility values of  $\leq 12.3$  and  $\leq 31.2\%$ , respectively. LODs were 0.025 and 0.050 mg/kg for melamine and cyanuric acid, respectively, whereas LOQs, set arbitrarily at the lowest fortification level, were 0.05 and 0.10 mg/kg for melamine and cyanuric acid, respectively.  $\text{CC}\alpha$  and  $\text{CC}\beta$ , at the 1 mg/kg maximum limit (ML) for infant formula powder endorsed by WHO, were respectively 1.03 and 1.05 mg/kg for melamine and 1.04 and 1.09 mg/kg for cyanuric acid.

**KEYWORDS:** Melamine; cyanuric acid; milk; liquid chromatography tandem mass spectrometry; LC-MS/MS

### INTRODUCTION

contaminated feed (9, 10) and of Chinese infants fed infant formulas and other milk powders tainted with MEL (1).