

Free Access*: [Preparation of \[¹³C₃\]-melamine and \[¹³C₃\]-cyanuric acid and their application to the analysis of melamine and cyanuric acid in meat and pet food using liquid chromatography-tandem mass spectrometry](#)

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Volume 25, 2008, Issue 10

For the determination of melamine and cyanuric acid the labelled internal standards [¹³C₃]-melamine and [¹³C₃]-cyanuric acid were synthesized using the common substrate [¹³C₃]-cyanuric chloride by reaction with ammonia and acidified water, respectively. Standards with excellent isotopic and chemical purities were obtained in acceptable yields. These compounds were used to develop an isotope dilution liquid chromatography/mass spectrometry (LC/MS) method to determine melamine and cyanuric acid in catfish, pork, chicken, and pet food. The method involved extraction into aqueous methanol, liquid-liquid extraction and ion exchange solid phase clean-up, with normal phase high-performance liquid chromatography (HPLC) in the so-called hydrophilic interaction mode. The method had a limit of detection (LOD) of 10 µg kg⁻¹ for both melamine and cyanuric acid in the four foods with a percentage coefficient of variation (CV) of less than 10%. The recovery of the method at this level was in the range of 87-110% and 96-110% for melamine and cyanuric acid, respectively.

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