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Development of an LC-MS-MS ESI-QTOF Method for Melamine Determination in Milk

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Abstract. At 2007 in USA, after removal from market of the pet food and the death around fifteen animals, the traceability of inputs used for feed as wheat gluten, rice protein and corn gluten, all imported from China, increased for the melamine contamination. In 2008, the deaths of six infants and the hospitalization of 295.000 people, according to the Chinese government, showed the enormous extent of fraudulent actions for different types of food, mainly for infant formulas produced in China. Melamine (2,4,6-triamino-1,3,5-triazine) is used for resins production, adhesives and plastics. In this fraud, melamine was used as a source of non proteic nitrogen because its composition presents 67% of nitrogen. The Kjeldahl method determines only the total nitrogen content, so the adulteration can mask the big addition of water into milk. The objective of this study was to develop a LC-MS-MS ESI-qTOF method for melamine determination in milk. The extraction was done as described by FDA (2008). The melamine quantification was done in a Phenomenex Kinetex[®] HILIC (50x2.1mm, 1.7µm) core-shell column. A gradient elution consisting of ammonium formate (mobile phase A), as strong solvent, and acetonitrile in formic acid X% (mobile phase B), as weak solvent, was carried out in ten minutes in an ultra performance liquid chromatographic system Waters Acquity[®] equipped with high resolution mass spectrometer Waters Synapt[®] ESI-qTOF. Three characteristic ions for melamine ($m/z = X$, $m/z = Y$ and $m/z = Z$) corresponding, respectively, to the protonated molecule, loss of a cyanamide molecule and loss of ammonia from previous ion were detected in ESI positive mode (capillary = 3.5kV). The method showed good suitability and accuracy to determine the presence of melamine in this matrix due to the presence of these three ions with good intensity. In that paper the conditions and limitation of implementation food quality and safety systems into processing of ready meals for small local catering objects and an agrotouristic farms will be described.