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Determination of melamine in feeds by LC-MS/MS

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Abstract In recent years, there were some reports of the healthy damage in the person and the animal taking in the food and feed which melamine was mixed intentionally. Therefore, in Japan, the maximum limit of 2.5 mg/kg for melamine in feeds had been established by Ministry of Agriculture, Forestry and Fisheries of Japan (MAFF) in April, 2012. In this study, we developed the quantitative methods of melamine in feeds based on the method of FDA, and evaluated the application to Japanese official method. The sample solution extracted from feed with acetonitrile-water (1:1) was subjected to liquid chromatograph-tandem mass spectrometer (LC-MS/MS) for determination of melamine. The LC separation was carried out on an hydrophilic interaction chromatography column (MERCK, SeQuant ZIC-HILIC, 2.1 mm i.d.×150 mm, 5 μm) using 10 mmol/L ammonium acetate solution-acetonitrile (17:3) as a mobile phase. The determination was performed in a selected reaction monitoring (SRM) mode. A spiked test was conducted with five kinds of samples spiked with 0.2 and 1 mg/kg of melamine. The spike test resulted in recoveries ranging from 93.0 % to 117 % (in relative standard deviations (RSD) within 3.6 %) of melamine. The method limit of quantitation and the method limit of detection for melamine were 0.2 mg/kg and 0.06 mg/kg, respectively. A collaborative study was conducted in nine laboratories using three kinds of samples spiked with melamine at 0.4 and 1 mg/kg. The mean recovery of melamine ranged from 97.1 to 99.3 % (the reproducibility standard deviation (RSDR) were within 7.0 %) and HorRat values were 0.34 to 0.44. This method has been accepted the application as a Japanese official method by MAFF in August, 2011. We have established a simple and sensitive quantitative method for melamine in feeds and feed ingredients.

Keywords melamine;liquid chromatograph-tandem mass spectrometer(LC-MS/MS);electrospray ionization (ESI);feed;fish meal;soybean meal;dried skim milk;collaborative study