GL Sciences Inc.

Analysis of Melamine in Food by LC/MS/MS

In 2007, pet foods imported from China caused serious illnesses or deaths in a large number of cats and dogs. US Food and Drug Administration reported that melamine was found in the pet foods as well as in urine of affected animals. Moreover, in 2008, milk and infant formula adulterated with melamine caused kidney stones and other renal failure, especially among young children.

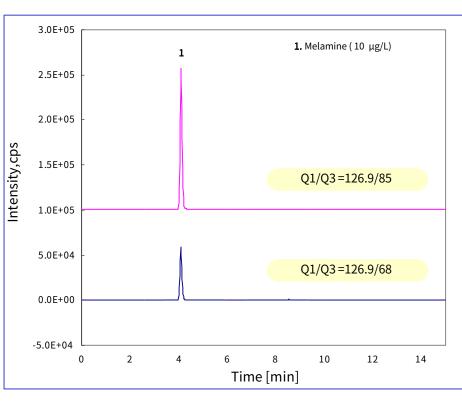
Since melamine is very hydrophilic compound, it is

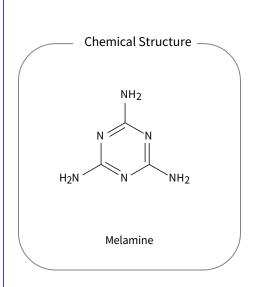
difficult to retain and analyze melamine using ODS columns. It is also undesirable to use ion-pair reagents because the sensitivity is reduced in LC/MS/MS detection.

In this note, Inertsil HILIC (hydrophilic interaction chromatography) was used as a separation column. Melamine was successfully retained and determined with good linearity.

(K.Suzuki)

A chromatogram obtained from standard solution





HPLC Conditions

System

Column : Inertsil HILIC (5 µm, 150 x 3.0 mm I.D.) Cat.No. 5020-07715

: A) CH₃CN

B) 10 mM ammonium acetate **Eluents**

 $A/B = 85/15 - 0.5 \min - 85/15 - 6.5 \min - 50/50$

(equilibration for 8 min), v/v

Flow rate : 0.5 mL/min Col. Temp. : 40 °C Detection : MRM (SRM)

Inj. Vol. : 5 µL

MS/MS (MRM) Conditions

System : API-4000 Q TRAP (Applied Biosystems Japan Ltd.) Detection: Compound Q1 Q3 DP CXP Melamine 126.9 85 41 10 27 14

126.9

68 Ion source : ESI (Posi) CUR CAD IS TEM GS1 GS₂ ihe 10 8 5500 700 30 on

41

10

41

10

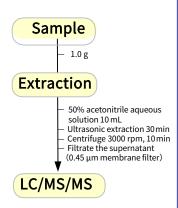
Melamine Q1: 126.9 Q3: 85 y = 90059x - 2498 1800000 Melamine Q1: 126.9 Q3: 68 R2=0.9995 1600000 1400000 1200000 1000000 800000 600000 = 34987x - 3270.3 200000 R2 = 0.9998 Concentration [µg/L]

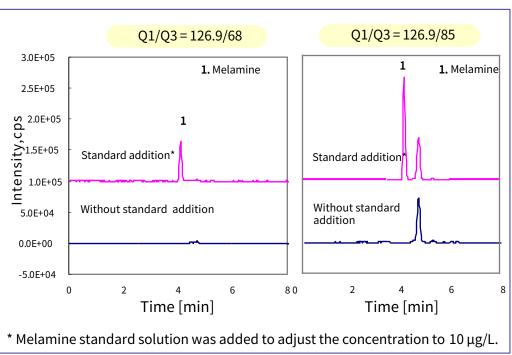
The calibration curves for melamine



2000000

An example of sample pretreatment





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