JSB is an authorised partner of Agilent Technologies Value Added Reseller

#405

Analysis of Melamine and Cyanuric Acid in Milk by LC-MS/MS

Melamine is an organic base and a trimer of cyanamide, with a 1,3,5-triazine skeleton. Melamine is combined with formaldehyde to produce melamine resin, a very durable thermosetting plastic, and melamine foam, a polymeric cleaning product. The end products include countertops, dry erase boards, fabrics, glues, house wares and flame retardants. Melamine is one of the major components in Pigment Yellow 150, a colorant in inks and plastics.

Melamine is nontoxic in low doses, but when combined with cyanuric acid it can cause fatal kidney stones due to the formation of an insoluble melamine cyanurate.

In 2007 a pet food recall was initiated by some pet food manufacturers who had found their products had been contaminated and caused serious illnesses or deaths in some of the animals that had eaten them. In March 2007, the US Food and Drug Administration reported finding white granular melamine in the pet food, in samples of white granular wheat gluten, imported from a single source in China. Melamine was added to the wheat gluten to artificially increase the apparent protein content. It was found in crystalline form in the kidneys and urine of affected animals as well.

In September 2008, several companies were implicated in a scandal involving milk powder and infant formula, which had been adulterated with melamine, leading to kidney stones and other renal failure, especially among young children. By end of September 2008, nearly 53,000 people had become ill, with more than 12,800 hospitalizations and four infant deaths.

Here, we report the investigation of simultaneous determination of melamine and cyanuric acid by HILIC-MS/MS on a TSKgel Amide-80 3 µm column (2 mm ID x 15 cm L, P/N 21865). Melamine can be detected by electrospray ionization mass spectrometry (ESI-MS) in positive mode (MRM 127/85), cyanuric acid by ESI-MS in negative mode (MRM 128/42) as shown in Figure 2. For milk spiked with melamine and cyanuric acid as a model sample, both melamine and cyanuric acid could be determined with high recovery after a simple deproteination pretreatment (Figure 3). Good linearity from 0.5 to 50 ppb was found for both compounds.

STRUCTURAL FORMULA

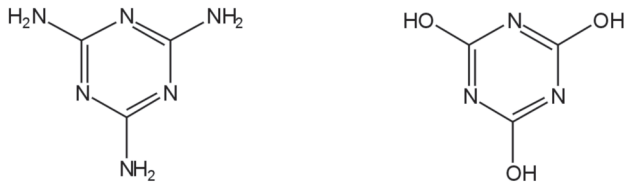

Figure 1

CHROMATOGRAPHIC CONDITIONS

Column:	TSKgel Amide-80 3 µm 2.0 mm ID x 15 cm L
Eluent:	A: 0.05% formic acid in water
	B: 0.05% formic acid in actetonitrile
	75% B
Flow rate:	0.2 ml/min
Injection vol.:	5 µl
Temperature:	40°C
Detection:	LC-ESI-MS/MS, QTrap (MDS SCIEX), 127/85+ (Melamine); 128/42- (Cyanuric acid)

PRETREATMENT OF MILK SAMPLE

Milk + (water / acetonitrile = 20/80) = 10 + 90 (v + v)

↓

Mixture, Ultracentrifugation (5,000 rpm, 5 min)

↓

Filtration (0.5 µm pore size)

TOSOH

SEPARATION OF MELAMINE AND CYANURIC ACID (10 PPB EACH)

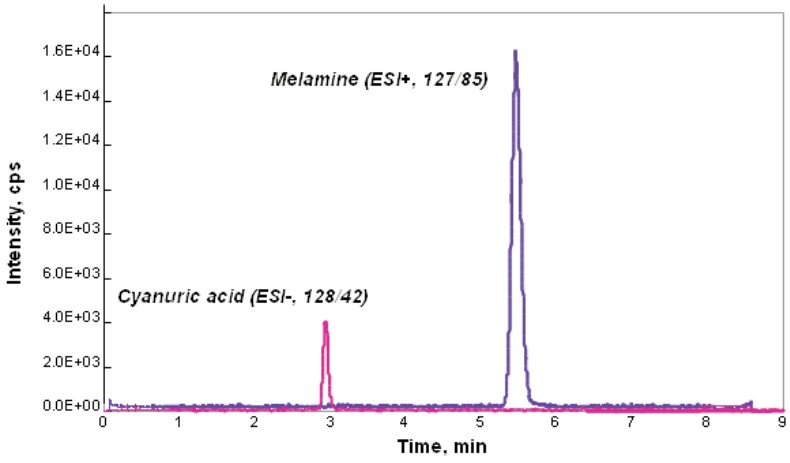

Figure 2

MRM CHROMATOGRAMS OF MILK AND SPIKED MILK SAMPLES (10 PPB EACH)

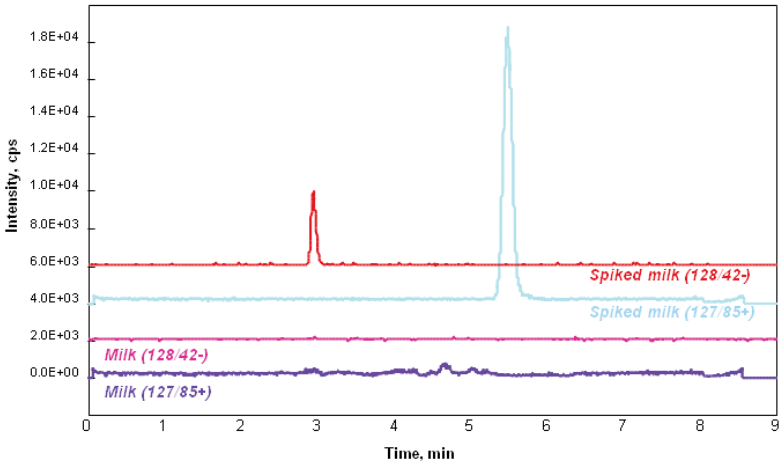

Figure 3

Headquarters
JSB International
Tramstraat 15
5611 CM Eindhoven
T +31 (0) 40 251 47 53
F +31 (0) 40 251 47 58

Zoex Europe
Tramstraat 15
5611 CM Eindhoven
T +31 (0) 40 257 39 72
F +31 (0) 40 251 47 58

Sales and Service

Netherlands
Apolloweg 2B
8239 DA Lelystad
T +31 (0) 320 87 00 18
F +31 (0) 320 87 00 19

Belgium
Grensstraat 7
Box 3 1831 Diegem
T +32 (0) 2 721 92 11
F +32 (0) 2 720 76 22

Germany
Max-Planck-Strasse 4
D-47475 Kamp-Lintfort
T +49 (0) 28 42 9280 799
F +49 (0) 28 42 9732 638

UK & Ireland
Cedar Court,
Grove Park Business Est.
White Waltham, Maidenhead
Berks, SL6 3LW
T +44 (0) 16 288 220 48
F +44 (0) 70 394 006 78

info@go-jsb.com
www.go-jsb.com