

Simultaneous Analysis of Cyanoguanidine and Melamine in Food by LC/MS/MS

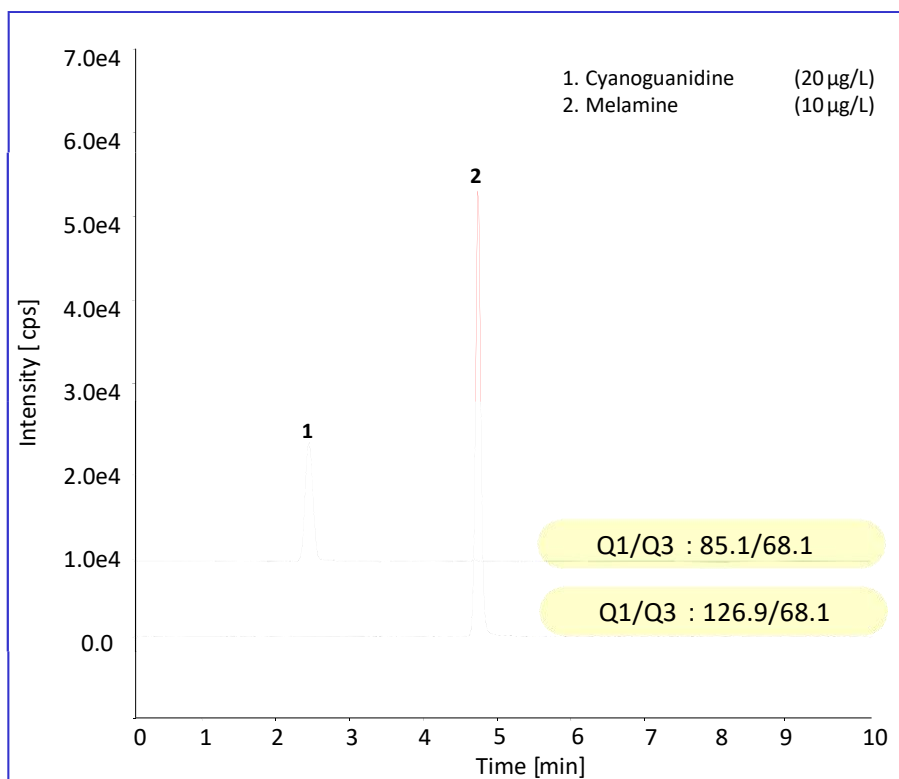
It became a serious problem in China and Taiwan in 2013 that cyanoguanidine, or dicyandiamide, was detected in powdered milk made in New Zealand. Since cyanoguanidine is contained in some chemical fertilizer as nitrification inhibitor, it is considered as a possible cause of the problem that such fertilizer was used in ranch.

In 2008, melamine was detected in milk and dairy products, which also became a big issue. HILIC

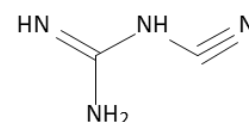
(hydrophilic interaction chromatography) mode was used for the determination of melamine content because melamine is highly hydrophilic. Chemical structure of cyanoguanidine is similar to that of melamine. In this note, cyanoguanidine was analyzed with HILIC mode. Cyanoguanidine and melamine could be determined simultaneously using Inertsil HILIC as a separation column.

(K.Kanno)

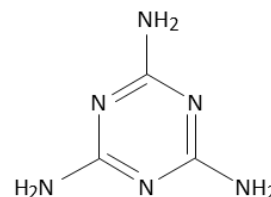
Chromatogram Obtained from Standard Sample



Chemical Structure



Cyanoguanidine
(Dicyandiamide)



Melamine

Structures are created using Chemistry 4-D Draw which is provided by ChemInnovation Software, Inc.

HPLC Condition

System : LC800
Column : Inertsil HILIC
(5 µm, 150 x 3.0 mm I.D.)
Eluent : A) CH₃CN
B) 10 mM Ammonium acetate
A/B = 90/10 — 0.5 min — 90/10 — 5.5 min —
50/50 (Equilibration for 5 min), v/v
Flow Rate : 0.5 mL/min
Col. Temp. : 40 °C
Detection : MRM (SRM)
Inj. Vol. : 5.0 µL

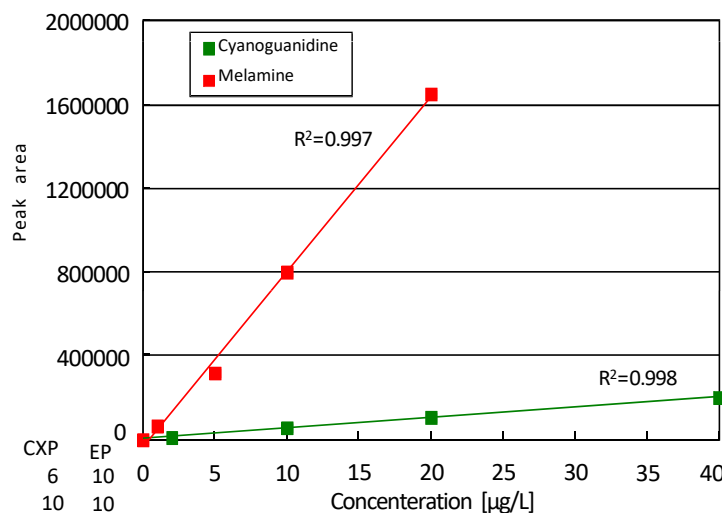
MS/MS (MRM) Condition

System : 4000 QTRAP (ABSciex)
Detection : Compound

	Q1	Q3	DP	CE	CXP	EP
Cyanoguanidine	85.1	68.1	41	41	6	10
Melamine	126.9	68.1	41	41	10	10

Ion Source : ESI (Posi)

	CUR	CAD	IS	TEM	GS1	GS2	ihe
	10	8	5500	700	60	30	on



Calibration Curve

Chromatograms Obtained from Powdered Milk Sample

Example of
Sample Pretreatment

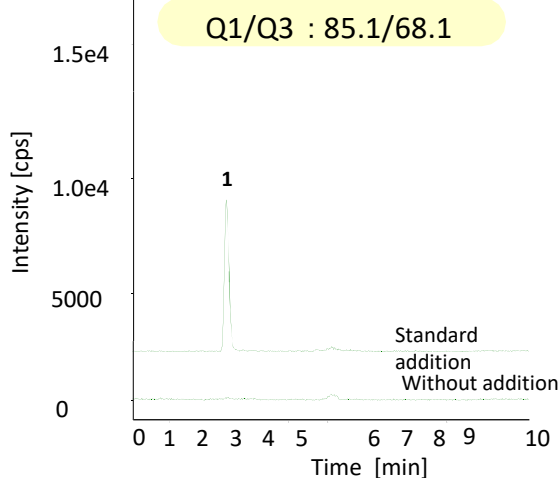
Sample

1.0 g

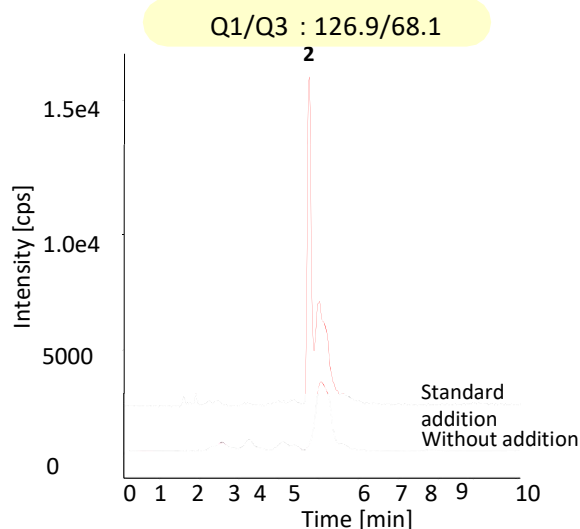
Extraction

- 20 mL of water/acetonitrile = 50/50 (v/v)
- Sonication extraction for 30 min
- Filtration (0.45 µm membrane filter)

HPLC



1. Cyanoguanidine
2. Melamine



Cyanoguanidine and melamine was added to commercially available powdered milk. Each concentration was adjusted to 10 µg/L (= 0.2 mg/kg powdered milk).

< HPLC Column used in This Note >

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

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