



Sumpfstr. 3, CH-6300 Zug; Fax: 041 748 50 65, Tel: 041 748 50 60
e-mail: info@infochroma.ch, www.infochroma.ch

Zwitterionic HILIC/MS/MS for the Analysis of Melamine and Cyanuric Acid in Milk Powder

Since the discovery of pet food scandal in United States of American in March 2007, SeQuant has been in discussions with institutes and authorities world-wide regarding the analysis of melamine and cyanuric acid using zwitterionic HILIC (Hydrophilic interaction liquid chromatography) technology, which is a straightforward HPLC method for analyzing polar and hydrophilic compounds.

RPLC-MS and GC-MS are the most common analytical analysis methods for determination of melamine and cyanuric acid. However, they have the drawbacks of using ion-pairing reagent or requiring sample derivation in or before analysis, which reduce the detection sensitivity for these two compounds. The pilot study¹ carried by US Food and Drug Administration (FDA) showed that using HILIC and mass spectrometry can detect them at very low regulator level. That work has also been followed by additional studies²

The Chinese scientists, Prof. Biying Chang, Dr. Qingsheng Liu and Linyun Li in the Feed Institute and Vegetable Institute, The Chinese Academy of Agriculture Science, followed the information from FDA in October, 2007, and try to set up a similar zwitterionic HILIC/MS/MS method for the Ministry of Agriculture, China. Their aim was to develop a HILIC method to analyze both melamine and cyanuric acid in animal feeds and grains, but without using isotope-labeled internal standards³. By using a SeQuant ZIC®-HILIC column and a gradient elution, their newly developed method is able to determine, in separate runs, both compounds in animal feeds at 1 ng/mL and 50 ng/mL, respectively. After the recent milk powder scandal in China, this method was applied to analyze cyanuric acid in the milk powder with slight modification on the sample preparation.

Experimental

Column:

SeQuant™ ZIC®-HILIC column
150 x 2.1 mm, 5 μm or 3.5 μm, 200 Å

Injection sample:

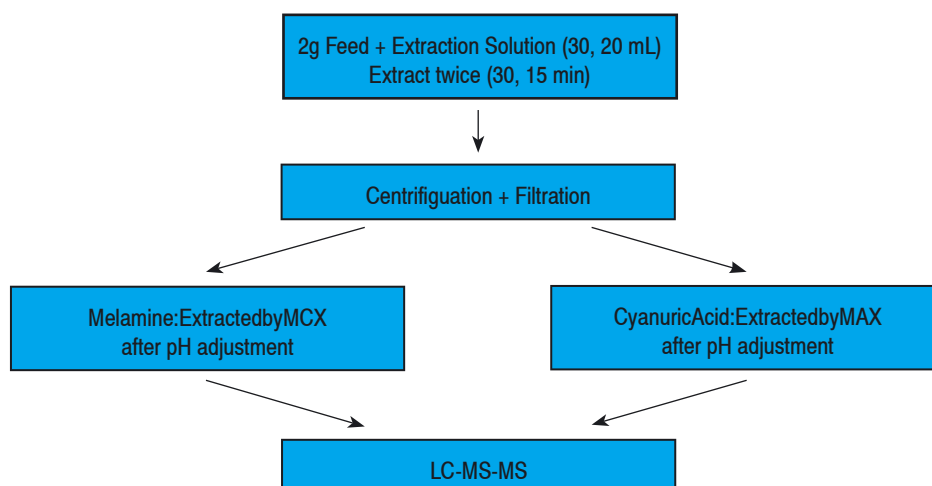
The sample extracts were diluted by acetonitrile

Mobile:

Eluent A: 10 mM Ammonium Acetate
Eluent B: Acetonitrile

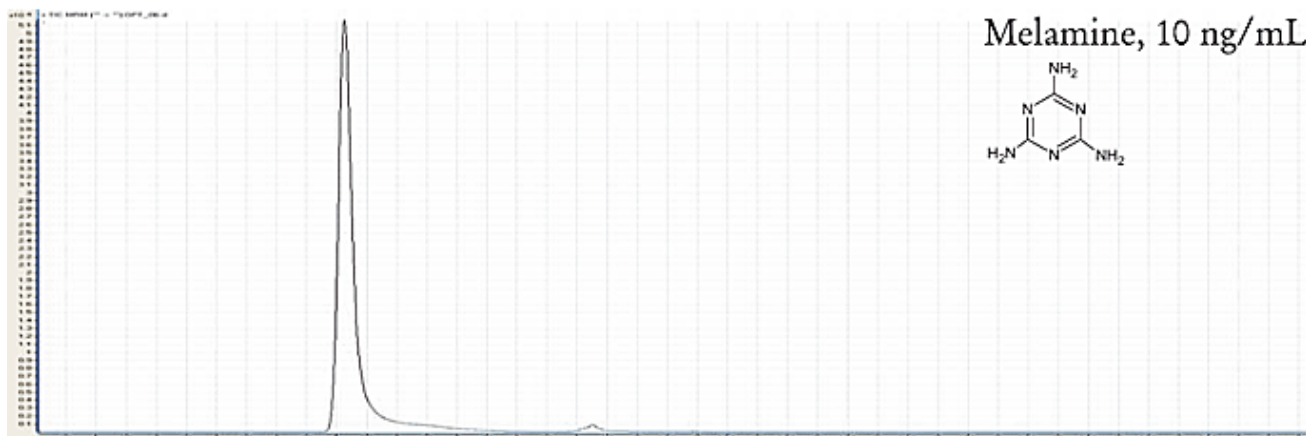
Gradient Profile:

Time (min)	A %	B %	Flow (mL/min)
0	10	90	0.3
1.0	10	90	0.3
4.0	30	70	0.3
4.1	50	50	0.3
7.0	50	50	0.3
7.1	10	90	0.3
20	10	90	0.3



Results:

1) Melamine and cyanuric acid in animal feeds



2) Cyanuric acid in milk powder



Reference

- 1) Presentation by Alex Krynitsky at Florida Pesticide Residue Workshop, 2007.
- 2) Simultaneous Determination and Confirmation of Melamine and Cyanuric Acid in Animal Feed by Zwitterionic HILIC Chromatography and Tandem Mass Spectrometry. Rapid Communications in Mass Spectrometry, in press. Corresponding author: david.heller@fda.hhs.gov
- 3) Personal communication with Prof. Biying Chang, Feed Institute, The Chinese Academy of Agriculture Science., Email: chbiying@126.com