Residue Findings of Melamine and Cyanuric Acid in Food

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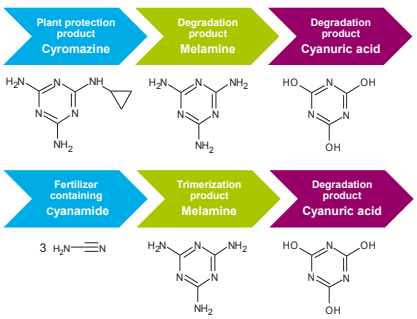
Introduction

Melamine residues were on everyone's lips a few years ago (2008). Extremely high quantities of up to 2500 mg/kg were found in infant milk, milk products and pet food from China [1]. In the latter case melamine was illegally used to give the false impression of a higher quantity of protein. This caused kidney damage and even death to infants and pets [2].

Residues of melamine and its hydrolysis product **cyanuric acid (CA)** in crops can also occur following the **application of PPPs** containing **cyromazine.**

Another possible source of contamination in the fields is the **use of fertilizers** containing cyanamide (known to trimerize to melamine, which further degrades to CA) or fertilizers containing melamine and CA as by-products.

Further entry paths may include irrigating or washing with chlorinated water containing **CA as a chlorine stabilizer** and/or **algicide,** and contact with melamine-based resins.



Analytical method

Analysis was accomplished using the **QuPPe method,** developed by the EURL-SRM. This involves methanol extraction, filtration, dilution and LC-MS/MS analysis. The Hypercarb™ column (CA) is used in the ESI neg. mode and the Acquity® BEH Amide column (melamine, cyromazine) is used in the ESI pos. mode.

Results

From July 2013 to May 2016 more than 5,400 **plant products of different commodity types** were analyzed for residues of cyromazine, melamine and CA. Residues of melamine and CA were found in a wide variety of fruits and vegetables, as well as in processed food.

Overall, 15 % of all the analyzed samples contained CA residues ≥ 0.01 mg/kg. Melamine residues ≥ 0.01 mg/kg were found in 12 % of the samples, whereas cyromazine exceeded this level in only 0.4 %.

In most cases residues of CA and melamine did not correlate with residues of cyromazine. There was also no visible correlation between high levels of melamine or CA and high levels of chlorate residues.

Residue situation

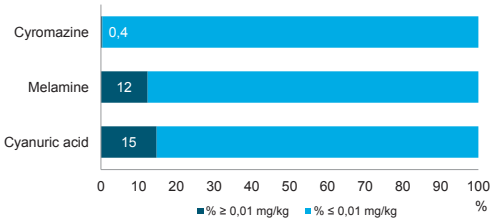


Figure 1: Residues in analyzed Plant Commodities (2013 – 2016)

Highest residue levels and toxicology:

- Melamine: **17 mg/kg in potatoes**; high levels in vegetables, potatoes and mushrooms
- Cyanuric acid: **14 mg/kg in green asparagus**; high levels in vegetables and mushrooms
- Cyromazine: **0.093 mg/kg in ginger**

Melamine strongly interacts with CA or uric acid, forming highly insoluble crystals. Their formation in the urinary tract can lead to severe kidney damage. There is a higher toxicity if melamine and CA are consumed together [1]. A risk assessment of the highest levels found is currently being carried out by the German BfR.

- Conventional vs. organic

All findings of cyromazine residues were detected in conventionally grown products. Melamine and CA were detected in both conventional and organic products. In conventional products the percentage of melamine residues ≥ 0.01 mg/kg was higher whereas CA showed similar frequencies of residue levels ≥ 0.01 mg/kg.

	All samples			Conventional			Organic		
Residues in plant commodities (2013 - 2016)	n	≥ 0.01 mg/kg	Average level of positives [mg/kg]	n	≥ 0.01 mg/kg	Average level of positives [mg/kg]	n	≥ 0.01 mg/kg	Average level of positives [mg/kg]
Cyromazine	5418	0.5 %	0.022	4668	0.5 %	0.022	750	0 %	0
Melamine	5375	12 %	0.11	4631	14 %	0.11	744	7 %	0.14
Cyanuric acid	5376	15 %	0.085	4635	15 %	0.094	741	19 %	0.034

n = number of samples

- Findings in fertilizers

The analysis of 19 different fertilizers revealed in some cases high amounts of both substances up to 7.3 mg/kg (melamine) and 41 mg/kg (CA). In the case of cyanamide fertilizers additional melamine and subsequent cyanuric acid may be generated in treated fields. Cyanamide fertilizers are not permitted in organic agriculture but the reported high persistency of melamine in soil and its transformation to CA could be an explanation for the frequent findings of melamine and CA in organic products.

Summary

Melamine and CA are frequently found both in conventional and organic products. Among the many possible paths of contamination the use of fertilizers seems to be the most relevant.
Risk assessment of the highest levels found appears to be inevitable.

EPRW 2016

Baden-Württemberg

References: [1] EFSA Statement on risks to public health due to the presence of melamine in infant milk and other milk products in China, EFSA Journal (2008) 807; [2] Scientific Opinion on Melamine in Food and Feed, EFSA Journal (2010); 8(4):1573