



Ellutia
Chromatography Solutions



Melamine in Milk Powder



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Some manufacturers in China have been discovered adding water to their raw milk in order to increase the volume of production. As a result of this dilution the milk has a lower protein concentration. In cow and sheep milk the percentage of protein can range from 3.3% to 5.8%. Companies using the milk for further manufactured products normally check the protein level through Kjeldahl analysis to evaluate the total nitrogen content. The addition of melamine increases the nitrogen content of the milk and therefore its apparent protein content.

Melamine is an organic base chemical most commonly found in the form of white crystals. Rich in nitrogen, Melamine is widely used in plastics, adhesives, countertops, dishware and whiteboards. The addition of melamine into food is not approved by the FAO/WHO Codex Alimentarius (food standard commission), or by any national authorities.

When combined with cyanuric acid, which may also be present in melamine powder, melamine can form crystals that can give rise to kidney stones. These small crystals can also block the small tubes in the kidney potentially stopping the production of urine, causing kidney failure and

even death. Melamine has also been shown to have carcinogenic effects in animals.

In 2007, melamine was found in wheat gluten and rice protein concentrate exported from China and used in the manufacture of pet food in the United States, causing the death of a large number of animals due to kidney failure. Melamine contamination has been found in a number of different brands of powdered infant formula, in one brand of a frozen yogurt dessert and in one brand of a canned coffee drink, all of which were most probably manufactured using the contaminated powdered milk.

In 2008, melamine was found in the products of one in every five milk suppliers in China. The scandal caused outrage among consumers and distraught parents, leading to international outcries regarding the standards of food safety in China. More than 20 people were convicted for their roles in the scandal with two people being executed. Despite a crackdown on melamine-laced milk products, more tainted supplies of raw materials were found in 2010. It is internationally recognized that milk solids manufacturers need strict monitoring within China as well as globally.

GC Conditions

Injector Temperature	250°C
Carrier Gas Type	Helium
Carrier Gas Control Method	Constant Flow
Split Time	1 min
Column Flow	1.3 ml min ⁻¹
Injection Volume	3 µl
Column 1	Guard Column 0.25 mm x 0.25 µm
Column 2	EL-5 30 m x 0.32 mm x 0.5 µm
Column Temperature Program	
Initial Temperature	115°C/3 min Hold Time
Temperature Ramp 1	200°C (10°C min ⁻¹)
Column Temperature	290°C/7 min Hold Time
TEA Conditions	
Pyrolyser Temp	850°C
Interface Temp	250°C
Sensitivity	230
Pump Type	Edwards nXDS10i
Mode	Nitrogen

Method

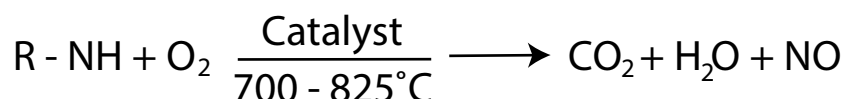
Standards of Melamine and Casein (the most abundant protein found in milk) were analysed on the Ellutia 820 TEA, operating in Nitrogen mode. The TEA is the most sensitive system on the market for detecting nitro, nitroso and nitrogen groups. When paired with the Ellutia 200 Series GC it makes a great low powered, space saving combination but the TEA can also be paired with other branded GCs.

It should be noted that even though both standards were derivatised using BSTFA + TMCS, Casein is a comparably large unvolatile protein that remained within the GC injector liner after injection. This results in no interference from Casein towards any response seen for Melamine.

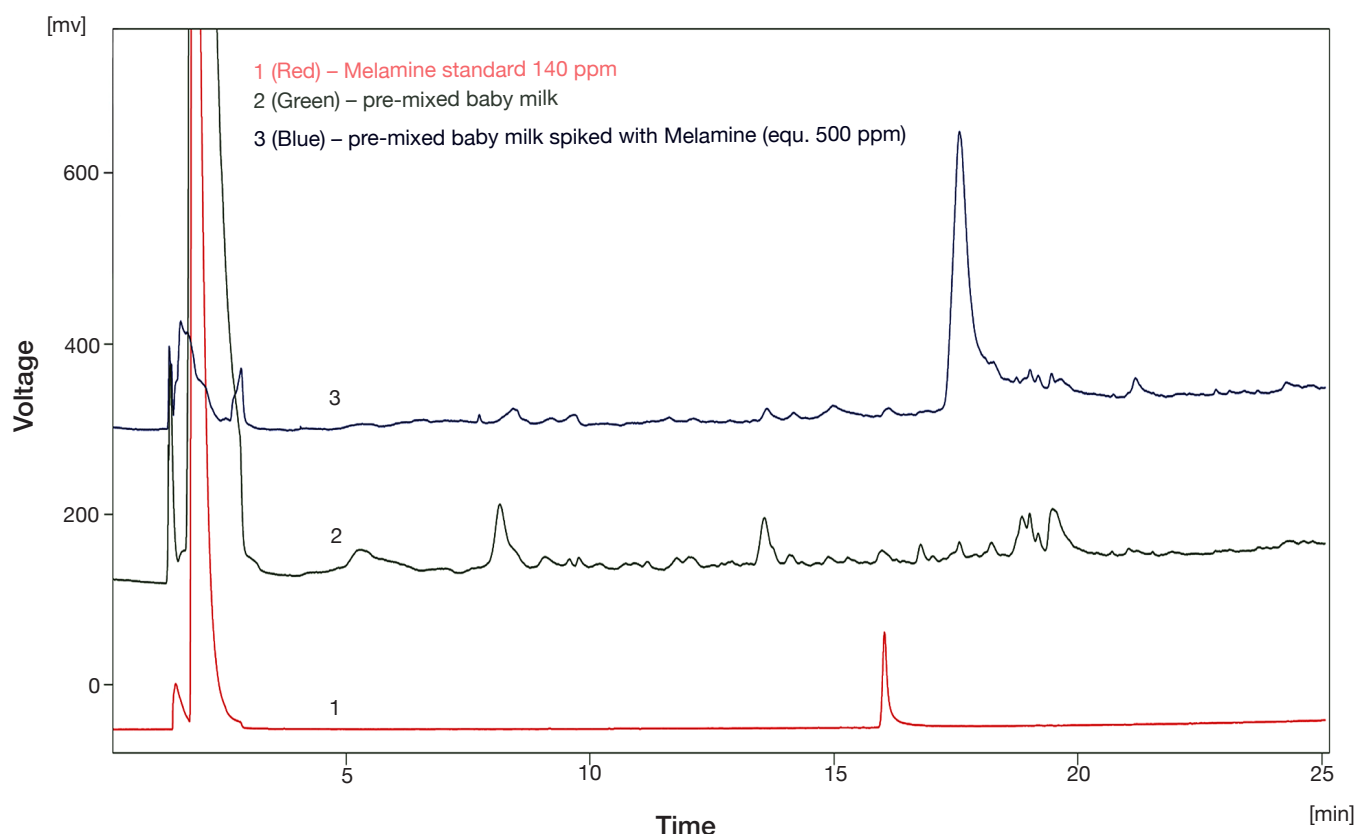
The World Health Organisation (WHO) and European Food Safety Authority (EFSA) have set daily exposure limits to Melamine at 0.2 mg/kg bodyweight per day. An estimation for relevant required detection limits can be calculate – an average 6 month old baby weighs 7.5 kg and consumes 0.75 litre of milk per day. Therefore, a 0.2 mg/kg exposure would equate to solution concentrations of 2 mg/L (2ppm).

Due to product availability, a pre-mixed carton of baby milk was tested. This would also allow for direct evaluation of melamine exposure per recommended serving. The sample showed responses for other nitrogen containing compounds, resulting in a noisy baseline. Samples require further clean-up and pre-concentration to reach relevant detection levels.

What is Nitrogen Mode on a TEA?



Nitrogen mode, compared with Nitroso mode, operates with the pyrolyser heated to 850°C instead of 500°C. This higher temperature allows for complete breakdown of nitrogen containing compounds. This free nitrogen can then be freely reacted with the generated ozone before entering into the reaction chamber for detection, rather than simply breaking away the nitro (N=O) group from a Nitroso compound.



Ordering Guide

TEA

TEA 820 - 230V	(Part no. 32000820)
TEA Pyrolyser Interface Kit	(Part no. 32020300)
NXDSi 10 Dry Pump	(Part no. 32020200)
Ozone Destroyer	(Part no. 32001060)

Gas Chromatograph

200 Series Gas Chromatograph (No Detector)	(Part no. 20500430)
Elution Software, Single Instrument	(Part no. 23001001)
Colibrick - 1 Channel	(Part no. 23001022)
EL - 5 30 m x 0.32 mm x 0.50 µm	(Part no. 51100183)
GC Mounting kit (for EL Autosamplers)	(Part no. 30500018)
Ellutia Autosampler Control Software	(Part no. 23001012)

Ellutia Liquid Autosampler offerings

Ellutia EL3100A Automatic Liquid Autosampler - 15 position	(Part no. 30500011)
Ellutia EL3000A Automatic Liquid Sampler - 121 position	(Part no. 30500010)
Ellutia EL3200A Automatic Liquid Sampler - 209 position	(Part no. 30500012)

Accessories

2 ml Vials	(Part no. 20511101)
2 ml Vials Screw Caps	(Part no. 20511107)
10 µl Liquid Syringe	(Part no. 20511203)
Septa (pk50)	(Part no. 20512101)