

# **Melamine Analysis**



**Products** 

## **Complete Analytical Solution**

# **Melamine Analysis Kit**

Includes column, standards, derivatization reagent, & accessories

*PLUS* easy-to-follow instructions with procedural checklist to simplify laboratory documentation

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**Individual Kit Components Available for Purchase** 

**Melamine Analysis Supplies** 

GC Columns

Rxi®-5Sil MS column with 5m Integra-Guard™

## Analytical

## Reference Materials

1mL Melamine Stock Standard (1,000µg/mL)

1mL Cyanuric Acid Stock Standard (1,000µg/mL)

1mL Ammelide Stock Standard (1,000µg/mL)

1mL Ammeline Stock Standard (1,000µg/mL)

1mL Benzoguanamine Internal Std (1,000µg/mL)

1mL Melamine Mix Standard (1,000µg/mL)

## **Derivatization Reagents**

25g BSTFA w/ 1% TMCS

### GC Accessories

50mL centrifuge tubes

13mm, 0.45µm nylon syringe filter

### Frequently Asked Questions

Q: What is melamine?

A: Melamine is an organic compound that is rich in nitrogen. When combined with formaldehyde it produces melamine-resin. Melamine-resin is widely used in several textiles, plastics, adhesives, flame-resistant products, and some cleaning agents.

Q: Why is melamine added to food?

A: Melamine is illegally added to food to artificially elevate the protein content values of products. The Kjeldahl method, which is used to determine protein content, works by testing for nitrogen content. When protein is digested, nitrogen is released and converted to ammonia. The amount of ammonia is determined by titration and is correlated to the amount of protein present. This method yields falsely high numbers when nonprotein nitrogen is in the sample.

Q: Has there ever been another case of melamine contamination?

A: The first melamine scare dates back to 1987 when contamination occurred in several beverages such as coffee, orange juice, lemon juice and fermented milk. This contamination was caused by leaching of melamine from melamine-resin cups. In 2004 and again in 2007 melamine contamination in rice and wheat gluten made thousands of animals sick, sometimes resulting in death. The most recent melamine scare was in 2008 when contaminated milk powder resulted in recalls of infant formula, biscuits, candies and several other products containing milk powder.

www.melaminepoisoning.info

Q: What level of melamine is safe?

A: The Food and Drug Administration (FDA) has set tolerable daily intakes (TDI) for melamine for infant and noninfant food stuffs. The TDI for noninfant food is 0.63mg of melamine per kg of body weight per day (0.63mg/kg bw/d). This equates to about 2.5 part per million (ppm) or µg/g in a given food commodity. For infant formula the FDA has set the TDI at 0.063mg/kg bw/d. This is a 10-fold safety factor to account for the fact that the majority of infants total caloric intake is from infant formula. This TDI equates to about 1ppm or 1µg/g in a given food commodity. The World Health Organization (WHO) has set their TDI for all food at 0.2mg/kg bw/d. Although none of these TDIs are reported as "safe" these are the amounts that have been determined, based on rat studies, that should yield no increased human health risk. | www.fda.gov & www.who.int/en/

Q: What is the solubility of melamine?

A: The solubility of melamine alone is 3.1g/L in water. However, when the combination of melamine and cyanuric acid form the crystal structure melamine cyanurate, the solubility significantly decreases to 0.01g/L in water, due to the strong hydrogen bonding that takes place in the melamine cyanurate crystals.

Q: What is backflash and do I need to worry about it with pyridine?

A: Backflash occurs when the vapor expansion volume of a solvent is larger than the internal volume of the injection port liner. This can cause sample carryover, gas line contamination, or decreased analyte response. Use Restek's backflash calculator to determine if you will have any issues. If you determine that you do have backflash, there are several ways to try and alleviate this problem. Decreasing injection volume, decreasing injection port temperature, and slowing down the total flow are all ways to help avoid backflash. | Use the backflash calculator

Q: Why do I need to derivatize my sample?

A: Derivatization is a chemical process where a chemical compound is transformed into a product with a similar structure but often different functionalities. Melamine and related analogs are not volatile compounds. By derivatizing these compounds they are converted to a volatile derivative of melamine therefore allowing these compounds to be analyzed by gas chromatography.

Q: Why is it necessary to dry the sample completely before derivatization?

A: The derivatization reagent BSTFA is a trimethylsilyl donor that reacts with a wide range of polar compounds to replace noncarbon bonded hydrogens with a –Si(CH3)3 group. Therefore, if any water is present, it will compete for the derivatization reagent and may result in the incomplete derivatization of the analytes of interest.

Q: What is the difference between solvent-only and matrix-matched standards?

A: When making a reference standard there are two different types of standards that can be utilized. Solvent-only standards are standards that are made in the extracting solvent alone. This type of standard is the most commonly used. Matrix-matched standards are standards that are spiked into the blank matrix extract. Matrix-matched standards are useful when any type of matrix effect is taking place. A matrix effect can alter the intensity of a given analyte, yielding false positives and sometimes false negatives.

Q: What is a matrix protection effect?

A: Matrix protection effects are matrix-based effects that enhance (or protect) the signal of an analyte, compared to what would be observed without the matrix being present. When a sample is injected into a GC, some matrix components may interact with active sites in the injection port, thereby reducing the potential for interactions between those active sites and the target analytes. These matrix components are termed analyte protectants and their interactions with active sites prevent target analyte absorption and minimize degradation, ultimately increasing the overall signal of an analyte in a commodity, in comparison to the response that would be seen for the analyte in solvent alone.1,2,3

- 1. C.F. Poole, J. Chromatogr. A 1158 (2007) 241.
- 2. T. Čajka, K. Maštovská, S.J. Lehotay, J. Hajšlová, J. Sep. Sci. 28 (2005) 1048.
- 3. K. Maštovská, S.J. Lehotay, M. Anastassiades, Anal. Chem. 77 (2005) 8129.

### Literature

GC/MS Analysis of Melamine and Cyanuric Acid Below 1µg/g in Infant Formula



The recent establishment of a  $1\mu g/g$  safety threshold for melamine in infant foods has led to an immediate need for more sensitive methods. Here we established GC/MS conditions for highly reproducible analyses and evaluated the effectiveness of both solvent-based and matrix-matched standards. Using this method, melamine and cyanuric acid were reliably detected at and below  $1\mu g/g$  in infant formula.

Find out more (453k PDF)



Melamine contamination was implicated in a large pet food recall that occurred in 2007 when animals died after eating contaminated pet food. Here, a modified GC/MS method, based on an FDA method, was used to analyze for melamine & related compounds cyanuric acid, ammelide, and ammeline in dry cat food. Analytes were easily identified by retention time matching and mass spectra. | Find out more

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