

Rapid Detection of Melamine in Dry Milk Using AccuTOF-DART

Introduction

Recent events have led to the recall of both pet food and dairy food products from international consumer markets. In both cases, melamine was added to these products to show a higher chemical signature for proteins, which in turn would increase the reported quality of the food. Unfortunately, the effect of this melamine addition caused the death of both pets and babies that consumed these tainted products. As a result, there is growing government and consumer concern towards the presence of melamine in food products.^{1,2} Because of this concern, there is a need for a rapid and accurate test to quickly determine the presence of melamine in these food products. Previously, the JEOL AccuTOF-DART was shown to be an effective technique for determining the presence of melamine in pet food.³ In this work, we extend the application of AccuTOF-DART to show that melamine can be rapidly detected when it is present in dry nonfat milk.

Experimental

Solid melamine granules were artificially spiked into commercially available dry nonfat milk at levels between 1000ppm and 500 ppb. These samples were then pulverized with a mortar and pestle to homogenize the mixtures. For analysis, the AccuTOF-DART system was set to the following parameters: needle voltage 3500V, discharge

electrode 150V, grid electrode 40V, Helium temperature 150 degrees C, and He flowrate 2.3 L/min. A melting point tube was dipped and swirled through the melamine/milk mixture and then placed in the Helium stream between the DART and the AccuTOF atmospheric pressure interface. The data was collected in a matter of seconds from the moment the samples were introduced into the DART stream. A representative mass spectrum is shown in Figure 1 that shows the high resolution and isotopic data for the melamine [M+H]⁺. Additionally, a semi-quantitative calibration curve was constructed to show the ability of the AccuTOF-DART to measure melamine in dry milk over a dynamic range of concentrations (Figure 2). Furthermore, using this methodology, the AccuTOF-DART was able to detect 1 ppm of melamine in dry milk, which is below the United States Food and Drug Administration's maximum allowable concentration of 2.5 ppm.¹

Conclusion

Unlike other analytical techniques, the AccuTOF-DART methodology described above does not require time consuming extractions or chromatographic methods to detect melamine in dry milk. Additionally, within seconds of sampling the tainted milk, the AccuTOF-DART provides high resolution and isotopic data to identify melamine.

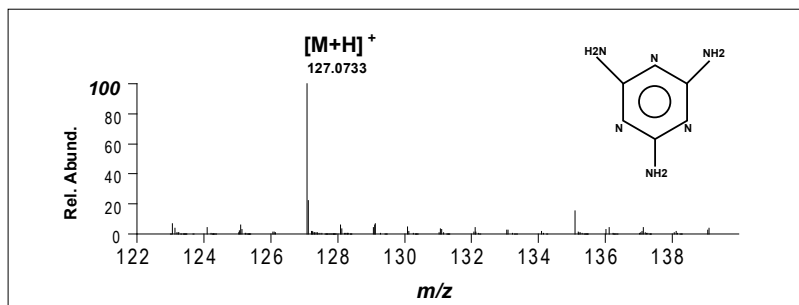


Figure 1. Mass spectrum of melamine in dry nonfat milk.

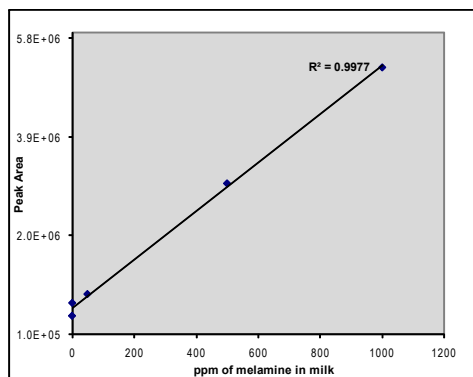


Figure 2. Semi quantitative calibration curve for melamine in dry nonfat milk.

References

- 1 Kwisnek, S. FDA Issues Interim Safety and Risk Assessment of Melamine and Melamine-related Compounds in Food. *FDA News* **2008**, [cited 2008 October 21]; Available from: <http://www.fda.gov/bbs/topics/NEWS/2008/NEW01895.html>.
- 2 Statement of EFSA on risks for public health due to the presences of melamine in infant milk and other milk products in China. *The EFSA Journal* **2008**, 807: p. 1-10.
- 3 Vail, T., P.R. Jones, and O.D. Sparkman. Rapid and unambiguous identification of melamine in contaminated pet food based on mass spectrometry with four degrees of confirmation. *J Anal Toxicol.* **2007**, 31(6): p. 304-12.