

Implementation of Several FDA Methods for Melamine in a High Volume Laboratory

The detection of melamine and cyanuric acid has evolved over the last several years to encompass many raw materials and foods. In early 2007 Covance Inc. began analysis using the precursor to the FDA's Laboratory Information Bulletin Number 4423 with the intent of using the method for the analysis of pet foods. The use of a labeled internal standard was optional, the chromatography was not always clean and the resulting recoveries were variable. By late 2008 the melamine scare had made its way to infant formula and foods, and lower limits of quantification were needed. The FDA provided two methods nearly simultaneously for the analysis of both melamine and cyanuric acid in infant formula and food for general consumption. In addition, Health Canada had a method for the analysis of melamine only, but at a much lower level of quantification. We chose the one method of the three that best met our clients' needs. The method chosen was the FDA's Laboratory Information Bulletin Number 4422. It is the most time consuming of the three methods, both for analyst time and for analysis time. We made very few changes, instead incorporating time-saving measures during sample preparation and analysis in order to reduce turn around time and increase throughput. A partial validation was performed. In addition, nearly every matrix submitted by clients for analysis was spiked with a known amount of melamine and cyanuric acid. Recoveries were generally good and the inclusion of an internal standard for each of the analytes of interest was crucial. Precision and accuracy data will be presented in a number of different sample types.

John Schmitz, Covance Laboratories, Madison, WI