Case Study

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>
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LC-MS/MS Detection of Melamine

National Center for Food Safety and Technology develops new method for the detection of melamine in food

Introduction

When the presence of melamine was discovered in animal food sources in early 2007, the National Center for Food Safety and Technology (NCFST) needed to quickly learn more about the effects of food processing on the chemical to learn how to accurately detect the presence of melamine in food.

The NCFST in Chicago, Illinois, is a research consortium involving the U.S. Food and Drug Administration's (FDA) Center for Food Safety and Applied Nutrition (CFSAN), the Illinois Institute of Technology (IIT) and the food industry. The NCFST incorporates the FDA Division of Food Processing Science and Technology and was established in 1988 by the FDA to form a link with industry to share expertise in food technology. The NCFST enables industry representatives to work collaboratively with FDA scientists on food safety and technology research projects.

The Thermo Scientific Accela high-speed LC and TSQ Quantum Ultra triple stage quadrupole mass spectrometer were used to develop an LC-MS/MS method to monitor melamine.

This cooperation enables early insight into emerging food safety issues and the safety of new technologies that may be important for innovation.

The NCFST enlisted the advanced analytical technology of the Thermo Scientific TSQ Quantum Ultra™ triple stage quadrupole mass spectrometer to develop a new LC-MS/MS method for the detection of melamine in food sources. The sensitivity and robustness of the TSQ Quantum Ultra were essential in developing this high-priority methodology to help ensure the safety of the nation's food supply.

Background

News coverage about the presence of melamine in pet food, animal feed, wheat gluten and other protein-based food commodities raised public concern about the chemical in the food supply chain for both animals and humans. Melamine—an industrial chemical used in the manufacturing of plastics, flame retardants and other products—is not approved for use in food or animal feed. Contaminated vegetable proteins imported into the United States from China were found in pet food, which sparked a nationwide pet food recall that began March 15, 2007. Additionally, a portion of the tainted pet food was used to produce farm animal feed and fish feed. The FDA and the U.S. Department of Agriculture discovered that some animals that ate the tainted feed were processed into human food.

Because the NCFST evaluates the effects of food processing on particular components of food, Dr. Peter Varelis, a research professor with the IIT's School of Biological, Chemical and Physical Sciences, knew the NCFST needed to understand what happens to melamine and cyanuric acid—a melamine-related compound—when they are processed in foods. This knowledge is essential to determine which degradation products to monitor that would indicate the presence of melamine or cyanuric acid in foods.

Implementation

In July 2007 the NCFST began using the TSQ Quantum Ultra triple stage quadrupole system coupled with the Thermo Scientific Accela™ high-speed chromatography system to develop its LC-MS/MS method to monitor melamine and its hydrolytic products in processed foods. The TSQ Quantum triple stage quadrupole system is the only instrument that enables Highly Selective Reaction Monitoring (H-SRM) performance, which facilitates the quick and efficient analysis of complex samples such as animal tissue. The Thermo Scientific LC-MS/MS solution has yielded accuracy and precision values for this method that were well within the guidelines of the FDA for analytical method development and validation.

Selecting Instrumentation

Prior to the melamine study, the NCFST did not have an LC-MS system installed in their laboratory. Dr. Varelis had previous experience with Thermo Scientific instrumentation in a different laboratory, and he was impressed with his prior encounters with the company's customer support services. These two factors lead to the NCFST's decision to select Thermo Scientific LC-MS instrumentation.

"Thermo Fisher has assisted us by providing invaluable advice about the analytical column, chromatographic conditions and mass spectrometer settings to achieve optimum sensitivity," Dr. Varelis said. "It's been a tremendous help. The system has come into its own for the analysis of melamine. We can readily achieve a signal-to-noise ratio of better than 3800 to 10 ppb of melamine in catfish," Dr. Varelis commented.

An existing method (Laboratory Information Bulletin No. 4396) for determination of melamine residues in catfish was developed by the FDA, Animal Drugs Research Center, Denver, CO. The method uses the TSQ Quantum mass spectrometer coupled with a Thermo Scientific Surveyor™ LC-MS pump and autosampler. The method showed that the Thermo Scientific instrumentation detected melamine at levels of 10 parts per billion in catfish extract, exceeding the agency's requirements.

Benefits

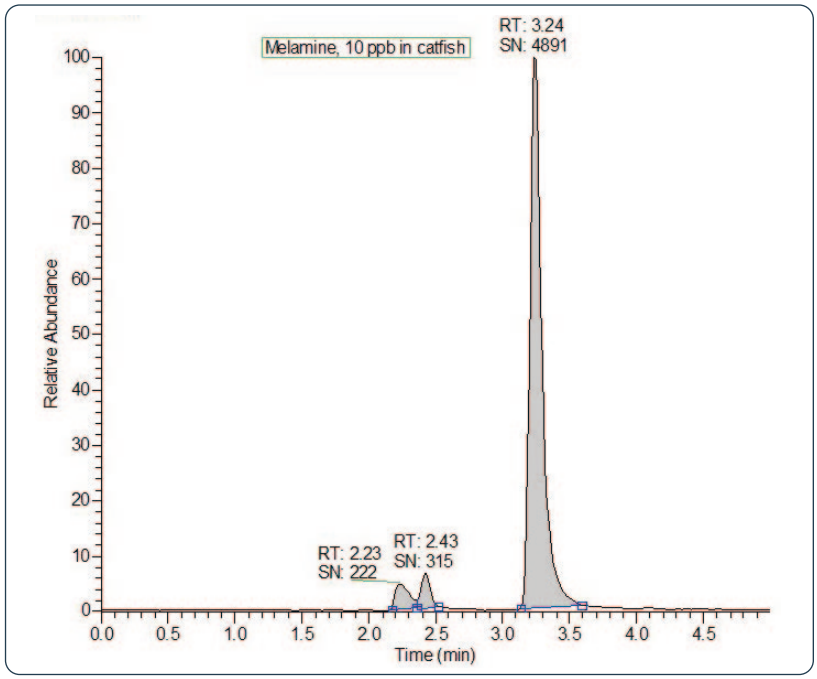
The NCFST has found both its LC-MS instrumentation and its collaboration with the Thermo Fisher support services team to be key components in its high-priority work on melamine. "The TSQ Quantum Ultra gives us great signal-to-noise advantage using H-SRM, which allows us to work at very low levels of detection," Dr. Varelis explained. "It has fantastic, unsurpassed sensitivity. And it's robust, so it gives us reliable and consistent results from day to day."

Next Steps

The NCFST is in the process of validating its LC-MS/MS method and will soon begin its study to understand how food processing techniques affect melamine.

Conclusion

The sensitivity and robustness of the TSQ Quantum Ultra solution proved to be essential factors in developing this high-priority methodology. As a result, the NCFST was able to respond quickly to this emerging issue and help make the nation's food supply safe.



ESI+ total ion chromatogram obtained from processing catfish that was fortified with 10 ppb of melamine. The chromatogram was obtained by analysis of the extract using a Thermo Scientific 150x2.1 mm BioBasic™ AX column and an Accela HPLC. Detection of the analyte was achieved using a TSQ Quantum Ultra by monitoring the MS/MS transitions *m/z* 128 → 85 and *m/z* 128 → 68.

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