Overview

Purpose: To develop and validate a turn-key system for automated detection of organic contaminants in food matrices and economic adulteration.

Methods: A benchtop, high resolution Orbitrap (Exactive) mass spectrometer system (Figure 2) was used for LC-MS analysis. The system features high mass resolution (180,000 at m/z 200), time-of-flight (TOF) capability for accurate mass measurements, and fast analysis times with 10-20 min analysis time. This system was coupled with a CTC Analytics PAL™ autosampler and a Thermo Scientific Accela UHPLC system to further increase the speed and efficiency of the analysis.

Introduction

The study of organic contaminants in food matrices includes the analysis of known knowns, and “non-targeted” screening of known unknowns and unknown unknowns. The large number of residues is not a limiting factor since the resolving power is high enough to resolve the majority of compounds within 12 minutes. Fast turn-key systems with high sensitivity and selectivity are thus needed to fully meet regulatory demand and satisfy high mass resolution and high speed analysis needs. This turn-key system was first validated by using a known spiked sample in a spinach matrix to demonstrate the effectiveness of the SIEVE™ software analysis. The system was then validated against the ChemSpider database (Royal Society of Chemistry) using accurate mass data generated by the Exactive system. The future trend in food safety analysis and in the detection of economically adulterated foods is the implementation of high mass resolution and accurate mass analysis.

Methods

Sample Preparation

The raw samples were prepared using a modified QuEChERS (Quick, Easy, Cheap, Effective, and Safe) method as described by the European Food Safety Authority (EFSA) for the analysis of pesticide residues in food matrices. The method involves the extraction of pesticides from the matrix using organic solvents and cleanup steps to remove matrix interferences. The samples were then analyzed by LC-MS using the Exactive system.

Data Analysis

Data acquisition and analysis were performed using Thermo Scientific SIEVE software. The software uses accurate mass analysis and performs principal component analysis (PCA) of experimental and control samples.

Results

The turn-key system was demonstrated to be able to distinguish signature differences between the spiked and control samples analyzed at four levels of concentration: 0.5 ppm, 1 ppm, 5 ppm, and 10 ppm. The turn-key system achieved a better than 95% accuracy and remained stable for the entire experiment. The results showed a distinct difference between the spiked and control samples analyzed at these four levels of concentration. Figure 8 shows results obtained from the 0.5 ppm level with 0.5 ppm mass accuracy. Additional samples analyzed showed the LOD of the SIEVE differential was 10 to 50 ppb, analytes pending.

Conclusion

This turn-key system is capable of detecting organic contaminants in food matrices and economic adulteration with high sensitivity and selectivity. It demonstrates the ability to differentiate between spiked and control samples at various levels of concentration. The future trend in food safety analysis and in the detection of economically adulterated foods is the implementation of high mass resolution and accurate mass analysis.

References