Non-Target and Unknown Screening of Food Samples using LC-High Resolution-MS/MS

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OVERVIEW

Here we present results using a new method to identify unexpected chemical residues and contaminants in food using the SCIEX X500R QTOF system. Samples were extracted using a QExClUSER method and analyzed by LC-HR-MSMS. Unknown compounds were automatically identified by using a non-target peak finding algorithm to detect and compare to separate sample and sample-specific signals from two calibrations. TOF-MS and MSMS data for ions of interest were automatically processed using formula finding and searched against mass spectral databases. The method provides on-scan library searching and empirical formula finding, results are compared with the ChemSpider hit count, and can be used to quickly find the correct match.

INTRODUCTION

Hybrid LC-MSMS systems like quadrupole-time-of-flight (QTOF) provide the ability to perform targeted and non-targeted screening in food samples on a routine basis. The SCIEX X500R QTOF system is a robust, high performance high resolution MSMS system designed for routine use providing:

- Sensitivity to detect compounds at nanolevel readout

- Requiring power to remove interference from complex food matrices

- Linearity to quantify over up to 3 orders of magnitude

- Mass accuracy to identify compounds following regulatory guidelines

- Confident identification using MSMS spectra and ion value

For Research Use Only. Not for use in diagnostic procedures.

EXPERIMENTAL

Sample preparation

Food samples from a Canadian supermarket were extracted using a QExClUSER procedure following guidelines EN 15602/2007. Sample extracts were diluted 1:10 to reduce the matrix load.

LC separation was performed using a SCIEX ExionLC™ AC column with a Phenomenex Kinetex Biphenyl 2.6µm (50x2.1 mm) column at a flow rate of water and methanol at 5mL/min with a 6 heater drone to minimize ion suppression while detecting easily at 10 µg/kg levels. The sensitivity of the X500R QTOF system is maintained benchtop design and a minimized footprint, 6 heater drones are integrated to maintain sample control and dynamic background calibration, introduced in 2010 with the QTrap® HP system designed for routine use providing:

- Sensitivity to detect compounds at nanolevel readout

- Requiring power to remove interference from complex food matrices

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- Confident identification using MSMS spectra and ion value

- Quick data review and reporting utilizing customizable flagging and filtering of results.

RESULTS

X3SR Performance Characteristics

Resolution (~20,000 at m/z half height) and mass accuracy (<5 ppm) are often sufficient to separate the analysis of interest from interfering matrices and thus, are identified as the key parameters for non-targeted identification in routine guidelines. The X3SR QTOF system stabilizes N-optics design of the X500R QTOF system to maximize resolution while maintaining benchtop design and a minimized footprint, 6 heater drones are integrated to maintain sample control and dynamic background calibration, introduced in 2010 with the QTrap® HP system designed for routine use providing:

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Processing Workflow for Unknown Identification in SCIEX OS Software

Extracted ion chromatograms (XIC) are generated using a non-target peak finding algorithm. No masses or retention times are provided to find chromatographic features. Sample-control-comparison is used to separate matrix and sample-specific signals from unknown contaminants.

In addition to more efficient formula finding, MSMS spectra are also used for structural elucidation. Without MSMS spectra it is impossible to conclude a correct structure from a molecular formula alone. The examples shown in Figure 4 illustrate the need of fragment ion detection to confidently differentiate between known Prometon and Terbumeton.