Fast Screening and Quantification of Pesticide Residues in Baby Food Using GC Orbitrap MS Technology

Dominic Roberts,¹ Cristian Cojocaru,¹ Richard Fussell,¹ Mike Hetmanski,² Paul Silcock¹ and Jason Cole²
¹Thermo Fisher Scientific, Runcorn, UK; ²FERA Science Ltd, Sand Hutton, York, UK

Overview

Purpose: To evaluate the utility of Orbitrap-based GC-MS technology for fast pesticides screening and quantification.

Methods: Baby food samples were extracted following QuEChERS protocol. The final extracts were spiked with a mixture of 132 pesticides at concentrations corresponding to 0.5—200 ng/g (ppb). A Thermo Scientific™ Q Exactive™ GC hybrid quadrupole-Orbitrap mass spectrometer was used in all experiments. Data was acquired and processed using the Thermo Scientific™ TraceFinder™ software.

Results: The results of this study show that the Q Exactive GC system is an ideal analytical tool for the analysis of pesticide residue testing in complex matrices, offering high performance full-scan analysis at fast GC separation. Routine mass resolution of 60,000 FWHM and consistent sub-ppm mass accuracy ensures selective and confident compound detection and identification. The Q Exactive GC system provides highly comparable quantitative performance with respect to existing state-of-the-art GC triple quadrupole MS instruments.

Introduction

Pesticides are measured almost exclusively by liquid chromatography (LC) and gas chromatography (GC) analytical methodologies. GC offers good separation efficiency and a choice of MS detectors such as single or triple quadrupoles. However, targeting specific compounds during acquisition limits the scope of analysis. This limitation has led to increased interest in the development of methods using MS analyzers that can operate in full quadrupole. However, targeting specific compounds throughout the acquisition (Table 2).

Methods

Sample Preparation

Baby food samples were extracted using the citrate buffered QuEChERS protocol. The homogenized sample (10 g) was mixed with acetonitrile (10 mL) followed by the addition of MgSO₄ (4 g), NaCl (1.0 g), disodium hydrogen citrate sesquihydrate (0.5 g) and trisodium citrate dehydrate (1.0 g). Dispersive solid phase extraction [MgSO₄ (150 mg), C₁₈ (50 mg), PSA (50 mg) and carbon (7.5 mg) per mL of extract] was used for sample clean-up. The final extracts were spiked with a mixture of 132 pesticides.

Gas Chromatography

1 µL was injected into a PTV injector (cold splitless) and compound separation was achieved using a Thermo Scientific™ TRACE™ 1310 gas chromatograph and a Thermo Scientific™ TraceGOLD™ TG-551MS 30 m length × 0.25 mm inner diameter × 0.25 µm film thickness column (Table 1). A Thermo Scientific™ TSQ™ 8000 Evo triple quadrupole GC-MS, with a broad scope pesticide residue testing, even with fast GC run times requires fast MS acquisition rates through full scan acquisition. Quantitative performance comparable to triple quadrupoles is also presented.
Data Analysis
Data was acquired and processed using the TraceFinder software. TraceFinder allows easy data reviewing and data reporting.

Results

Chromatography
Good chromatographic separation was obtained using the GC conditions (Figure 1). The total ion chromatogram as well as the extracted ion chromatograms (XIC, 2 ppm extraction mass window) of the first (dichlorvos, m/z 184.97650, RT=4.46 min) and the last eluting pesticide (deltamethrin, m/z 252.90451, RT=10.33 min) is shown.

MS Acquisition Speed
Using short GC run times requires fast MS acquisition rates in order to obtain an adequate number of scans/peak. An example of typical number of scans acquired using the Q Exactive GC system operated at 60,000 resolution is shown below (Figure 2). Noticeably, beside the adequate number of scans/peak, excellent mass accuracy was obtained for every scan across the peak (<0.4 ppm RMS).

Instrumental Detection Limit (IDL)
System sensitivity was assessed by calculating the IDL for each individual pesticide. The results of this experiment show that the sensitivity is comparable to that of the Thermo Scientific™ TSQ™ 8000 Evo triple quadrupole GC-MS, with 91% of pesticides having an IDL < 2 ng/g (Figure 4).

Mass Accuracy
Obtaining accurate mass information in a consistent manner is critical for determining the identity of a pesticide. The mass accuracy for all 132 pesticides was assessed at 5 (or 10, depending on compound) ng/g level from a series of n=10 repeat injections. The mass deviation values did not exceed 1 ppm for any of the analytes and the overall mass accuracy average value was 0.4 ppm, providing the highest confidence in accurate and selective detection (Figure 5).

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Linarity of response
Quantitative linearity was assessed using matrix-matched calibration standards injected in triplicate at each level. In all cases the coefficient of determination ($R^2$) was >0.99 with an average value of $R^2 = 0.997$ and with residual values from the regression line of <25%.

FIGURE 6. Coefficient of determination and residuals values (%RSD) for prothiofos and benalaxyl calculated for a linear range of 0.5–100 ng/g.

Conclusion
• The Q Exactive GC system offers the capability to perform high performance quantitative analysis in full scan for broad scope pesticide residue testing, even with fast GC separations.
• The fast scan speed, high resolution, and outstanding mass accuracy, together with full scan sensitivity, allow reproducible and accurate pesticide quantification at very low levels.
• Routine mass resolution of 60,000 FWHM eliminates isobaric interferences, increasing confidence in results when screening pesticides in complex matrices.
• The Q Exactive GC system provides highly comparable quantitative performance with respect to GC triple quadrupole MS instruments.

References