Enhanced sensitivity for the quantitative analysis of pesticides using a GC Orbitrap MS

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Abstract

Purpose: Pesticide analysis in food is critical to ensure the safety of the products that are distributed and consumed worldwide. In this study, the Thermo Scientific™ Orbitrap™ Exploris GC MS was evaluated for quantitative analysis of pesticides in highly-pigmented matrices such spinach.

Methods: Orbitrap technology featuring an advanced EI ion source provides full-scan (FS) high-resolution accurate mass (HRAM) capability with a more efficient ionization enabling enhanced sensitivity and robustness for reliable detection of trace level pesticides in acetonitrile QuEChERS extracts.

Results: The results obtained in this study fully comply with the acceptance criteria defined in current international and European regulations for multiresidue analysis of pesticides in food.

Introduction

Detection of pesticide residues in fruits and vegetables is essential for food safety, but it can be challenging in complex, highly pigmented matrices due to interferences. Orbitrap technology provides high resolution and mass accuracy, enabling confident identification of coeluting and isobaric compounds while reducing false results. Furthermore, the FS-HRAM acquisition enables simultaneous quantitation and screening within a single analysis with the capability of retrospective data analysis, allowing laboratories to reprocess datasets for additional or emerging compounds without re-injecting samples.
The Orbitrap Exploris GC S mass spectrometer features the Thermo Scientific™ NeverVent™ Advanced Electron Ionization Source (NVAEI) which offers an improved design providing enhanced sensitivity and robustness, allowing for increased instrument uptime and maximized sample throughput for rapid return on investment (ROI).

Materials and methods

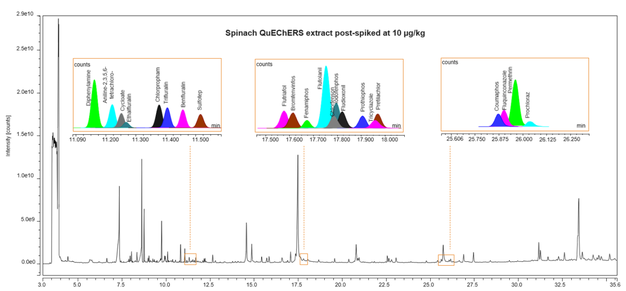
Test method(s)

Purified QuEChERS extracts of spinach vegetables were post-spiked with a mixture of 185 pesticides and used to evaluate linearity, sensitivity and overall quantitative performance.

Data analysis

Data was acquired, processed and reported using the Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) software, which provides integrated instrument control, automated workflows, and an intuitive interface for data analysis, customizable reporting, and compliant data management in accordance with FDA 21 CFR Part 11 and European Commission (EU) Annex 11. Its advanced reprocessing capability streamlines sequence setup, data review, and reporting offering the possibility to easily flag and check compliance with the current regulations.

Figure 1. TIC (full scan: *m/z* 50-600) and XIC (insets) for a QuEChERS extract of spinach post-spiked at 10 µg/kg showing an example of FS-HRAM chromatography.



Results

Compliant analyte identification

The use of HRAM-GCMS reduces the matrix interferences ensuring a confident and selective identification of analytes (Figure 1). All target compounds satisfied the SANTE criteria at the default MRL of 10 µg/kg and more than 90% were confirmed at concentrations of 1 µg/kg following the SANTE/11312/2021v2026 guidance criteria of:

- two ions were detected for each pesticide with mass accuracy ≤ 5 ppm with precursor and product ions in the extracted ion chromatograms fully overlapped,
- due to the added value of accurate mass measurement, matching ion ratios is not necessary, however, an ion ratio (IR) deviation within ± 30% (relative) of the average of calibration standards from the same sequence was considered to provide additional support for identification
- retention times (RT) within a tolerance of ± 0.1 minutes.

Sensitivity performance (IDL) and analytical linearity

Instrument sensitivity was evaluated using calculated IDLs derived from replicate injections of low-level, matrix-matched standards. The Orbitrap Exploris GC S demonstrated detection limits at levels at or below 0.5 µg/kg for most of the investigated pesticides (Figure 2).
Linear dynamic range was evaluated using matrix-matched calibration curves ranging 0.5–200 µg/kg, with each calibration level injected in triplicate. All target analytes demonstrated excellent linearity, with coefficients of determination (R^2) > 0.993, response factor (%RSD) values below 20% across the calibration range, and ion ratios (IRs) within ±30% of the average calibration standard values. System suitability for quantitative analysis was confirmed by calculated concentrations within ±20% of expected values and recoveries ranging from 96% to 104%. An example of calibration curve for Aldrin is shown in Figure 3.

Figure 2. Distribution of calculated IDLs for the investigated compounds

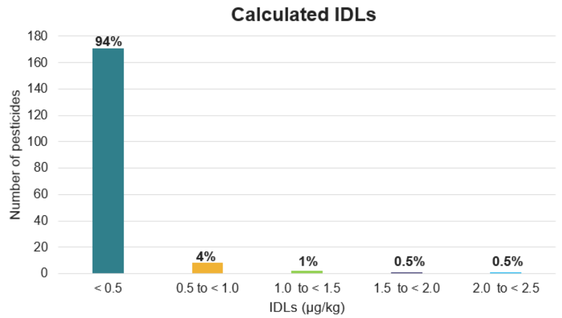
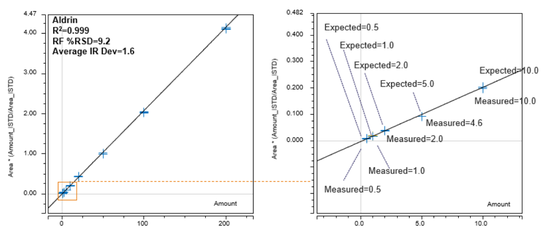


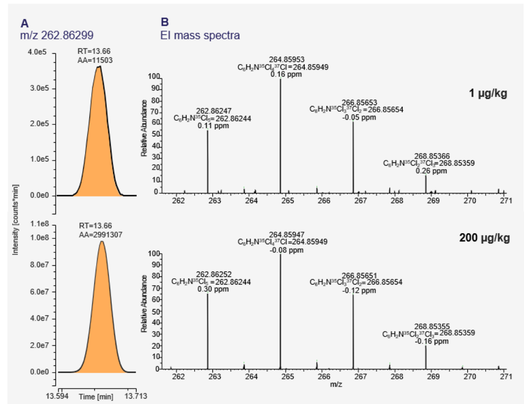
Figure 3. Example of full-range calibration curve for Aldrin (left) and a zoomed view of the lower range of the calibration curve (right)



Spectral fidelity across the calibration range

Spectral fidelity is critical for both compound identification and accurate quantitation. Poor spectral quality can lead to misidentification, incorrect ion ratios, and inaccurate peak area measurements, ultimately compromising accuracy, reproducibility, and quantitation reliability. Maintaining high-quality spectra across the entire calibration range is therefore essential to ensure confident and defensible results. Figure 4 shows an example of the XIC for a spinach QuEChERS extract post-spiked at 1 and 200 µg/kg. The close match of the molecular ion cluster with sub-1ppm mass accuracy at low and high concentrations reflects the high quality of the spectra.

Figure 4. Spectral fidelity for Pentachloroaniline at 1 and 200 µg/kg in spinach matrix. XICs at each concentration (A) and zoomed-in view of EI mass spectra of the molecular ion cluster at each concentration level, annotated with the measured mass, elemental composition, theoretical mass, and mass accuracy in ppm (B).



Mass accuracy irrespectively of analyte concentration and time

Mass accuracy plays a paramount importance in ensuring correct analyte identification, providing selectivity to discriminate isobaric and co-eluting compounds in complex matrices, reducing false positives when searching libraries. It is therefore important that mass accuracy stays consistent irrespectively of the analyte concentration and the analysis time.
The Orbitrap GC S delivered high reproducible mass accuracy within 3 ppm mass error across the calibration levels as demonstrated in Figure 5 as well as over a long acquisition period of four weeks as shown in Figure 6.

Figure 5. Average mass accuracy for quantifier ions across the calibration levels (0.5-200 µg/kg post-spiked in spinach matrix)

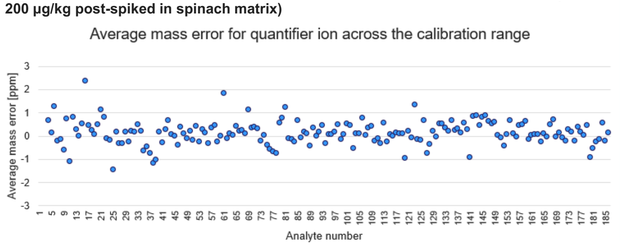
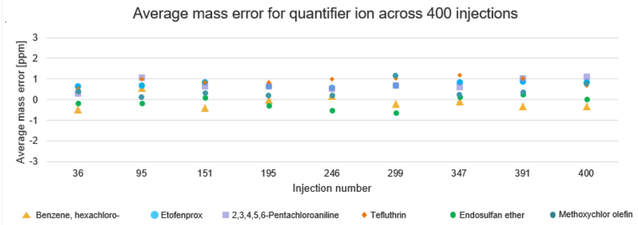


Figure 6. Mass accuracy obtained across a period of four weeks for solvent, matrix-matched calibration standards and injections of diverse matrices post-spiked at 10 µg/kg.



Conclusions

The results obtained in these experiments demonstrate that the Orbitrap Exploris GC S mass spectrometer equipped with NeverVent AEI ion source represents a reliable tool for analysis of pesticides in complex matrices.

- The Orbitrap Exploris GC S offers a simple approach for the multiresidue analysis of pesticides allowing for full-scan acquisition combined with high mass resolving power
- The NeverVent AEI ion source is designed for ultra-trace concentration detection, making it ideal for challenging applications ensuring detection limits at levels at or below 0.5 µg/kg for most of the investigated pesticides
- Confident identification of compounds can be easily achieved through i). high spectral quality, ii). high spectral fidelity, ensuring consistent spectrum independently from concentration, and iii). consistent mass accuracy irrespectively of analyte concentration and time
- System suitability for quantitative analysis was demonstrated with R^2 >0.993, RF %RSD across the entire calibration curve < 20, and IRs within ± 30% of the average of calibration standards, calculated amounts within 20% the expected values, and percentage recovery (%) ranging from 96 to 104.

References

SANTE/11312/2021 v2026, Analytical quality control and method validation procedures for pesticide residues analysis in food and feed. Implemented 01/01/2026

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