



# Pesticide residues analysis for commercial food testing laboratories

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# Our innovative technologies help food testing laboratories stay ahead of the curve

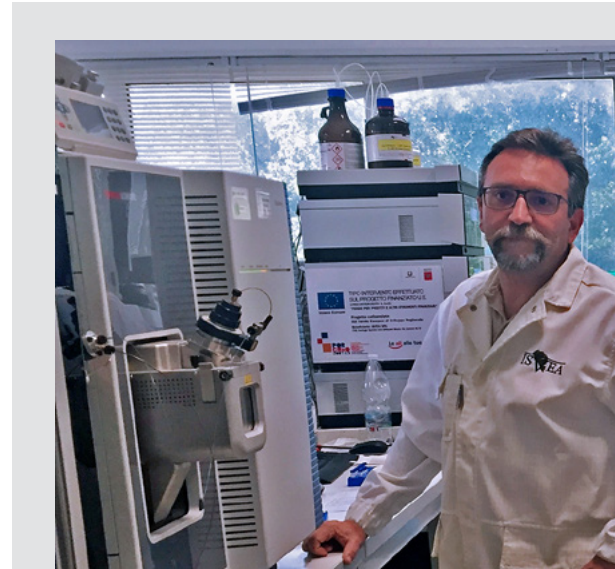
## Introduction

Pesticide residues testing of food is necessary to ensure compliance with maximum residue levels (MRLs), or tolerance levels, or even lower levels specified by customers. Analytical laboratories are also expected to detect, quantitate, and identify hundreds of different pesticides with diverse physicochemical properties in hundreds of different sample types, and this can be challenging.

This pesticide residues analysis compendium features the most up-to-date application notes from Thermo Fisher Scientific and highlights the latest instrument and software innovations designed to increase productivity in a commercial food testing laboratory.

These robust methods were developed with global compliance in mind. Wherever possible, method validation was performed to meet the requirements of MRL levels, MRL residue definitions, and the method performance criteria of the EU SANTE guidelines (Guidance document on analytical quality control and method validation procedures for pesticide residues and analysis in food and feed).

## Compliance | Productivity | Robustness



**Alessandro Cavaglioni, Laboratory Manager, ISVEA, Italy**  
(ISVEA srl is an analytical laboratory for the agri-food sector and is highly specialized in wine analysis).

“...ISVEA needs partners with experts who are able to support the company in challenging goals...”

“...we analyzed thousands of samples and remember only a dozen cases for which it was necessary to cross check results with complementary technology...”

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APPLICATION SUMMARY AN10586

# Ultra low level quantification of pesticides in baby foods using an advanced triple quadrupole GC-MS/MS system

Richard Law, Aaron Lamb, Paul Silcock, and Cristian Cojocariu, Thermo Fisher Scientific, Runcorn, UK

## Keywords

European Regulation 396/2005, Commission Directive 2006/125/ EC, European Commission 2002/657/EC, SANCO/12571/2013, European Commission 788/2012/ EC, pesticide, food, QuEChERS, Vanquish Flex system, TSQ Quantis MS, TraceFinder software

## Goal

To assess the quantitative performance of the Thermo Scientific™ TSQ™ 9000 triple quadrupole GC-MS/MS system fitted with the Advanced Electron Ionization (AEI) source for the analysis of pesticide residues at ultra low levels in baby food.

## Method


The method performance was tested in accordance with the SANTE/11813/2017 guidance document. Samples of carrot/potato and apple/pear/banana baby food were extracted using the citrate-buffered QuEChERS protocol using Thermo Scientific™ HyperSep™ dispersive solid phase extraction (dSPE) products. Liquid injections of the sample extracts were performed using a Thermo Scientific™ TriPlus™ RSH™ autosampler.

Data were acquired, processed, and reported using Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) software, which allows instrument control, method development, quantitative/qualitative analysis, and customizable reporting all within one package.

Part Number	Description
26096-1420	Thermo Scientific™ TraceGOLD™ TG-5SilMS 30 m × 0.25 mm I.D. × 0.25 µm film capillary column
26096-1425	5 m integrated guard column
453T2120	Siltek six baffle PTV liner

## Conclusion

- Accurate, quantitative analysis of over 200 pesticides over up to five orders of magnitude (0.025–250 µg/kg), showing outstanding LODs and linear response.
- Robustness displayed over approximately 400 consecutive injections of sample matrix (1 g/mL), with SANTE compliance at the default MRL throughout.
- High sensitivity providing the real possibility to dilute the sample extract, thus limiting matrix contamination and system maintenance, leading to an increase in laboratory productivity.

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APPLICATION SUMMARY AN10432

## Three-fold increase in productivity for pesticide residue analysis in baby food using fast triple quadrupole GC-MS/MS

Cristian Cojocariu and Paul Silcock, Thermo Fisher Scientific, Runcorn, UK

Michael T. Hetmanski and Richard J. Fussell, Food and Environment Research Agency (FERA), York, UK

### Keywords

Pesticide analysis, baby food, GC-MS/MS, TraceFinder software, food safety

### Goal

To assess the performance and productivity of the Thermo Scientific™ TSQ™ 8000 Evo\* GC-MS/MS for pesticide residues analysis.

### Method

Baby food samples were extracted using the citrate buffered QuEChERS protocol. A TSQ 8000 Evo triple quadrupole GC-MS/MS instrument coupled with a Thermo Scientific™ TRACE™ 1310 GC was used. Sample introduction was performed with a Thermo Scientific™ TriPlus™ RSH autosampler, and chromatographic separation using a Thermo Scientific™ TraceGOLD™ TG-5SiIMS capillary column. Data were acquired and processed using Thermo Scientific™ TraceFinder™ software, a single software package that integrates instrument control, method development functionality, and quantitation focused workflows.

Part Number	Description
26096-1300	TraceGOLD TG-5SiIMS 15 m × 0.25 mm I.D. × 0.25 µm film capillary column
453A2342	SSL single taper liner


### Conclusion

Acceleration of sample analysis is made possible by:

- direct analysis of acetonitrile extracts with no need for an additional solvent exchange step.
- shorter GC run times using fast data acquisition with the EvoCell fast collision cell technology.
- comprehensive detection of target pesticides and nontargeted pesticides using simultaneous full scan and SRM data acquisition. Additional pesticides were identified by searching the full scan data against the NIST library.

Excellent sensitivity was achieved. All pesticides were detected and identified at a concentration of 5–10 ng/g with IDL values of 0.2–3.7 ng/g.

\*Equivalent or better performance with the new Thermo Scientific TSQ 9000 GC-MS/MS system

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# Sensitive and selective analysis of fipronil residues in eggs using Thermo Scientific GC-MS/MS triple quadrupole technology

Cristian Cojocariu, Joachim Gummersbach, and Susanne Sales, Thermo Fisher Scientific

## Keywords

Fipronil, fipronil sulfone, fipronil desulfinyl, eggs, food safety, sensitivity, selected reaction monitoring, GC-MS/MS, triple quadrupole gas chromatography

## Goal

To demonstrate the excellent sensitivity, selectivity, and linearity achieved for the analysis of fipronil and its metabolites in eggs using the Thermo Scientific™ TSQ™ 8000 Evo\* triple quadrupole GC-MS system.

## Method


In the experiments described, a TSQ 8000 Evo triple quadrupole mass spectrometer was coupled to a Thermo Scientific™ TRACE™ 1310 GC for gas-phase separation of target compounds. Injection of acetonitrile extracts of homogenized eggs was performed automatically using a Thermo Scientific™ TriPlus™ RSH™ autosampler. Chromatographic separation was achieved on a Thermo Scientific™ TraceGOLD™ TG-5SiIMS GC capillary column. Data was acquired using full-scan and timed selective reaction monitoring (t-SRM) modes and processed with Thermo Scientific™ TraceFinder™ software.

Part Number	Description
26096-1420	TraceGOLD TG-5SiIMS 30 m × 0.25 mm I.D. × 0.25 µm film capillary column
26096-1425	5 m integrated guard column
453T2120	Siltek six baffle PTV liner

## Conclusion

- The results of these experiments show that by using the Thermo Scientific TSQ 8000 Evo triple quadrupole GC-MS system, excellent sensitivity, selectivity, and linearity were achieved for fipronil, fipronil sulfone, and fipronil desulfinyl.
- All compounds were detected at 0.2 µg/kg (LOD) and identified at 0.5 µg/kg (LOQ), which is five times below the EU statutory MRL for the sum of fipronil and fipronil sulfone.
- The results presented in this work are compliant with the SANTE/11813/2017 analytical quality control guidelines for pesticides and demonstrate that the TSQ 8000 Evo GC-MS/MS system provides sensitive and confident detection and quantification of pesticide residues in difficult matrices

\*Equivalent or better performance with the new Thermo Scientific TSQ 9000 GC-MS/MS system

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APPLICATION SUMMARY AN65609

# A sensitive and robust analytical solution for pesticide residues analysis in apple using GC-(AEI)-MS/MS

Subodh Kumar Budakoti, Sarvendra Pratap Singh, and Dasharath Oulkar, Thermo Fisher Scientific

## Keywords

Advanced electron ionization (AEI), apple, Chromeleon CDS, pesticide residues, QuEChERS, targeted quantitation, TSQ 9000 GC-MS/MS system

## Goal

To develop an analytical solution for trace-level quantitation of 211 pesticides in apple using gas chromatography coupled to triple quadrupole mass spectrometry. The optimized method was validated following EU SANTE/11813/2017 guidelines and evaluated for maximum residue levels (MRLs) compliance with the Food Safety and Standards Authority of India (FSSAI) and the European Commission (EC).

## Method


QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) sample preparation method followed by GC-MS analysis using the Thermo Scientific™ TSQ™ 9000 triple quadrupole GC-MS/MS system equipped with the advance electron ionization source (AEI).

Data acquisition and processing were carried out using Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) software.

Part Number	Description
60105-341	QuEChERS Salts (2007.01)
60105-203	150 mg anhydrous magnesium sulfate, 50 mg primary secondary amine
26096-1420	Thermo Scientific™ TraceGOLD™ TG-5SilMS 30 m × 0.25 mm I.D. × 0.25 µm film capillary column
26096-1425	5 m integrated guard column
453A1925UI	SSL splitless liner, single taper, deactivated

## Conclusion

- The optimized method offered excellent sensitivity (LOQ = 0.001 mg/kg) for more than 95% of compounds, significantly below the current regulatory requirement (0.01 mg/kg).
- The linearity range (0.0001–0.050 mg/kg) offered excellent  $R^2$  (>0.99) with <20% residuals, ion ratio within  $\pm 30\%$ , recoveries within 70–120%, and precision (<20% RSD).
- At least 40 injections (standards, samples, blank) could be completed in a day (24-hour cycle) providing <10% RSD.
- The method validation data were within the acceptance criteria of the SANTE guidelines and comply with the EU and FSSAI MRLs requirements by achieving excellent low limits of quantitation (LOQ).

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APPLICATION SUMMARY AN72952

# Large scale screening and quantitation of pesticide residues in rice using GC-(EI)-MS/MS

Subodh K. Budakoti, Sarvendra P. Singh, and Dasharath Oulkar, Thermo Fisher Scientific

## Keywords

TraceFinder, pesticide residues, rice, QuEChERS, TSQ 9000 GC-MS/MS system, targeted screening, quantification

## Goal

To develop a screening solution followed by quantitation of 159 pesticides in rice by using gas chromatography-triple quadrupole mass spectrometry. The optimized method was validated in accordance with the SANTE guidelines and fulfills the Food Safety and Standards Authority of India (FSSAI) and the European Commission (EC) maximum residue levels (MRLs) for rice.


## Method

Extraction of residues was performed by the widely used QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) acetonitrile extraction method. Any residues in the extract were determined using the Thermo Scientific™ TRACE™ 1310 gas chromatograph coupled to a Thermo Scientific™ TSQ™ 9000 triple quadrupole GC-MS/MS system equipped with the ExtractaBrite™ source and vacuum probe interlock (VPI) technology. The data acquisition and processing were carried out using Thermo Scientific™ TraceFinder™ software. The optimized method was validated according to the SANTE/11813/2017 guidelines.

Part Number	Description
80020-415-500	Anhydrous magnesium sulfate, Thermo Scientific™
80020-424	Sodium acetate, Thermo Scientific™
80020-416-100	PSA (primary secondary amine), Thermo Scientific™
26096-1420	Thermo Scientific™ TraceGOLD™ TG-5SilMS 30 m × 0.25 mm I.D. × 0.25 µm film capillary column
453A1925	SSL splitless liner, single taper, deactivated

## Conclusion

- The VPI NeverVent™ technology, which allows changing of the ion source and column without venting the system, and the reduced downtime from the robustness of the ExtractaBrite ion source further enhance productivity.
- Using this approach, at least 40 injections (standards, samples, blank) could be completed in a day (24-hour cycle).
- This validated method data meets the requirement of the SANTE guidelines and complies with the EU as well as FSSAI MRLs requirement by achieving excellent lower limits of quantitation (LLOQ).

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APPLICATION SUMMARY AN72980

# A quantitative determination of pesticide residues in chili powder using GC-MS/MS

Sarvendra P. Singh, Subodh K. Budakoti, and Dasharath P. Oulkar, Thermo Fisher Scientific

## Keywords

Quantitation, pesticides, chili powder, TraceFinder, QuEChERS, TSQ 9000 GC-MS/MS system, ExtractaBrite source

## Goal

To set up a complete analytical solution to enable commercial food testing laboratories to analyze pesticide residues in chili powder, in compliance with the requirements of the Food Safety and Standards Authority of India (FSSAI).


## Method

A QuEChERS method for the extraction was followed by dispersive solid phase extraction (dSPE) clean-up with graphitized carbon black (GCB) material. Chromatographic separation and data acquisition were carried out using the Thermo Scientific™ TRACE™ 1310 gas chromatograph coupled with a Thermo Scientific™ TSQ™ 9000 triple quadrupole mass spectrometer. Thermo Scientific™ TraceFinder™ 4.1 software was used for instrument control, data acquisition and processing, data review, and reporting.

Part Number	Description
80020-413-100	C18 Octadecyl endcapped, Thermo Scientific™
80020-415-500	Anhydrous magnesium sulfate, Thermo Scientific™
80020-424	Sodium acetate, Thermo Scientific™
80020-416-100	PSA (primary secondary amine), Thermo Scientific™
80020-417-100	GCB (graphitized carbon black), Thermo Scientific™
26096-1420	Thermo Scientific™ TraceGOLD™ TG-5SilMS 30 m × 0.25 mm I.D. × 0.25 µm film capillary column
453T2120	Siltek six baffle PTV liner

## Conclusion

- The method complies with the MRL requirements of the FSSAI and the method performance criteria of the EU SANTE guidelines.
- The LOQ (0.025 mg/kg) is much lower than the MRLs, except for endosulfan, without compromising data quality.
- Identification and confirmation with retention time, ion ratio, recoveries (70–120%) and precision (<20%) offered by the method are within acceptance criteria of SANTE.
- Excellent retention time stability was demonstrated with dicofol in chili powder, assessed across n=25 consecutive matrix injections in a batch.

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APPLICATION SUMMARY AN72981

# A selective and sensitive method for quantification of pesticide residues in wheat using GC-(EI)-MS/MS

Sarvendra P. Singh, Subodh K. Budakoti, and Dasharath P. Oulkar, Thermo Fisher Scientific

## Keywords

TraceFinder, pesticide residues, wheat, QuEChERS, TSQ 9000 GC-MS/MS system, quantitation, triple quadrupole, gas chromatography

## Goal

To demonstrate a comprehensive analytical solution for selective detection and accurate quantification of 148 pesticide residues in wheat matrix by using gas chromatography-triple quadrupole mass spectrometry. The proposed analytical solution is validated in accordance with the SANTE guidelines, and overall results are checked for compliance with maximum residue levels (MRLs) set by the Food Safety and Standards Authority of India (FSSAI) as well as the European Commission (EC) for wheat.


## Method

The extraction of pesticide residues was performed using the QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) method. Pesticide residues in the extracts were detected, identified, and quantified using the Thermo Scientific™ TRACE™ 1310 gas chromatograph coupled to a Thermo Scientific™ TSQ™ 9000 triple quadrupole GC-MS/MS system, in accordance with the SANTE/11813/2017 guidelines. The data acquisition and processing were carried out by using Thermo Scientific™ TraceFinder™ software.

Part Number	Description
80020-415-500	Anhydrous magnesium sulfate, Thermo Scientific™
80020-424	Sodium acetate, Thermo Scientific™
80020-416-100	PSA (primary secondary amine), Thermo Scientific™
26096-1420	Thermo Scientific™ TraceGOLD™ TG-5SilMS 30 m × 0.25 mm I.D. × 0.25 µm film capillary column
453A1925	SSL splitless liner, single taper, deactivated

## Conclusion

- The results are compliant with the SANTE guidelines and can meet the testing requirements for compliance with the EU and FSSAI MRLs.
- Quantitative results for 148 pesticides in wheat matrix in a single injection were achieved.
- The VPI NeverVent™ technology, which allows changing of the ion source and column without venting the system, and the reduced downtime from the robustness of the ExtractaBrite ion source further enhance productivity.
- The method provides the high throughput required by commercial food testing laboratories.
- By following this approach, at least 38 injections (standards, samples, blank) could be completed in a day (24-hour cycle).

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APPLICATION SUMMARY AN73039

# Large-scale screening and quantitation of pesticide residues in milk using GC-(EI)-MS/MS

Sarvendra Pratap Singh, Subodh Kumar Budakoti, Dasharath Oulkar, Thermo Fisher Scientific

## Keywords

TraceFinder, pesticide residues, milk, QuEChERS, TSQ 9000 GC-MS/MS system, quantitation, ExtractaBrite source

## Goal

To set up a complete analytical solution to enable commercial food testing laboratories to analyze 155 pesticide residues in milk using gas chromatography-tandem mass spectrometry. The optimized method was validated per SANTE validation guidelines and assessed for MRL compliance per the Food Safety and Standards Authority of India (FSSAI) as well as the European Commission (EC).


## Method

A citrate buffered QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) method was used, and 25 mg/mL PSA and 50 mg/mL C18 were used for clean-up to remove the interferences from protein and fat. A Thermo Scientific™ TRACE™ 1310 gas chromatograph was coupled with a Thermo Scientific™ TSQ™ 9000 triple quadrupole mass spectrometry equipped with a Thermo Scientific™ ExtractaBrite™ electron ionization (EI) source and vacuum probe interlock (VPI) technology. The data acquisition and processing were carried out by using Thermo Scientific™ TraceFinder™ 4.1 software.

Part Number	Description
60105-216	EN 15662 QuEChERS Extraction kit, Thermo Scientific™
80020-413-100	C18 Octadecyl endcapped, Thermo Scientific™
80020-415-500	Anhydrous magnesium sulfate, Thermo Scientific™
80020-416-100	PSA (primary secondary amine), Thermo Scientific™
26096-1420	Thermo Scientific™ TraceGOLD™ TG-5SilMS 30 m × 0.25 mm I.D. × 0.25 µm film capillary column
453A1925	SSL spitless liner, single taper, deactivated

## Conclusion

- This application note offers a total solution for the trace-level quantification of 155 pesticide residues in milk by using a Thermo Scientific GC-EI-MS/MS system.
- The use of a citrate buffered QuEChERS method for extraction and clean-up followed by analysis using GC-MS/MS decreased downtime and increased productivity for commercial food testing laboratories.
- This validated method data meets the requirements of the SANTE guidelines. Also, this method complies with the EU and FSSAI MRL requirements by achieving an excellent lower limit of quantification.

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APPLICATION SUMMARY AN65906

## Automated micro-SPE clean-up for GC-MS/MS analysis of pesticide residues in cereals

Sarvendra Pratap Singh, Subodh Kumar Budakoti, Dasharath Oulkar, Thermo Fisher Scientific

### Keywords


Pesticide residues, cereals, QuEChERS, micro-solid phase extraction ( $\mu$ SPE), advanced electron ionization (AEI), targeted quantitation, TSQ 9000 GC-MS/MS system, Chromeleon Chromatography Data System

### Goal

To assess the suitability of an automated micro-solid phase extraction ( $\mu$ SPE) clean-up of QuEChERS extracts for the determination of pesticide residues in cereal samples by gas chromatography coupled to triple quadrupole mass spectrometry.

### Method

The cleaned-up extracts were analyzed using a Thermo Scientific™ TSQ™ 9000 triple quadrupole GC-MS/MS system equipped with the advanced electron ionization source (AEI). The sample introduction and automated clean-up were performed using a Thermo Scientific™ TriPlus™ RSH robotic autosampler configured with a liquid injection tool as well as with the dedicated  $\mu$ SPE tool and cartridges tray for automated clean-up. Data acquisition and processing were carried out using the Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) software, version 7.2.

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Part Number	Description
60105-341	QuEChERS Salts (2007.01) mylar pouch 6 g magnesium sulfate (anhydrous), 1.5 g sodium acetate, Thermo Scientific™ HyperSep™
60101-45GC	$\mu$ SPE GC cartridges 45 mg: 20 mg MgSO <sub>4</sub> , 12 mg PSA, 12 mg C18 and 1 mg CarbonX
26096-1420	TraceGOLD™ TG-5SilMS 30 m x 0.25 mm I.D. x 0.25 $\mu$ m film capillary column
453A1925UI	SSL splitless liner, single taper, deactivated

### Conclusion

- The experiments demonstrate that automated  $\mu$ SPE compared to dSPE with weighing the sorbents can significantly reduce the sample preparation time by 40 to 50% and increase sample throughput in commercial testing laboratories by more than 1.5 times considering a batch of 10 samples.
- Replacing the manual d-SPE procedures with  $\mu$ SPE delivers optimum recovery and precision while reducing the risk of human errors.
- Excellent linearity was obtained using matrix-matched calibration standards over a concentration range of 0.0025 to 0.1 mg/kg with R<sup>2</sup> values mostly >0.995 and %RSD of residuals <5%.
- The performance has been checked with six replicates of pre-spiked samples at 0.01 mg/kg. The results (%recovery and %RSD) were in the range of 70 to 120% and <20%, respectively, and thus in compliance with the EU SANTE guideline criteria.



APPLICATION SUMMARY AN10541

## Multi-residue pesticide screening in cereals using GC-Orbitrap mass spectrometry

Dominic Roberts and Paul Silcock, Thermo Fisher Scientific, Runcorn, UK; Mette Erecius Poulsen, European Union Reference Laboratory for Pesticide Residues in Cereals and Feedstuffs, Technical University of Denmark, Denmark

### Keywords

Pesticides, QuEChERS, cereals, GC Orbitrap mass spectrometry, screening, quantitation, accurate mass, high resolution, TraceFinder software

### Goal

To demonstrate the performance of the Thermo Scientific™ Exactive™ GC Orbitrap™ mass spectrometer for the analysis of GC-amenable pesticides in cereals (wheat, barley, oat, rye, and rice).


### Method

Cereal samples (barley, oat, rice, rye, and wheat) were ground (or milled) to flour and then extracted using a citrate buffered QuEChERS procedure. In all experiments, an Exactive GC Orbitrap mass spectrometer was used. Automatic sample injection was performed using a Thermo Scientific™ TriPlus™ RSH™ autosampler, and chromatographic separation was obtained with a Thermo Scientific™ TRACE™ 1310 GC and a Thermo Scientific™ TraceGOLD™ TG-5SiIMS capillary column with a 5 m integrated guard. Data were acquired using Thermo Scientific™ TraceFinder™ software.

Part Number	Description
26096-1420	TraceGOLD TG-5SiIMS 30 m × 0.25 mm I.D. × 0.25 µm film capillary column
26096-1425	5 m integrated guard column
453T2120	Siltek six baffle PTV liner

### Conclusion

- All 105 pesticides were detected at 10 µg/kg (5 µg/L in vial). 96% of the 525 pesticide/matrix combinations were confirmed at <20 µg/kg (<10 µg/L in vial) with excellent linearity and in full compliance with the EU SANTE method performance criteria.
- The full scan acquisition permits efficient targeted data processing by use of a compound database and has the capability to easily add further analytes into the method scope.
- Consistent sub-ppm mass accuracy was achieved for all compounds over a wide concentration range, ensuring that compounds are detected with high confidence at low and high concentration levels.
- Repeated injections of a wheat matrix at 10 µg/kg showed that the system is able to maintain a consistent level of performance over an extended period of time as is demanded by a commercial testing laboratory.

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APPLICATION SUMMARY AN10584

# The quantitative power of high-resolution GC-Orbitrap mass spectrometry for the analysis of pesticides and PCBs in food

Dominic Roberts, Richard Law, and Paul Silcock, Thermo Fisher Scientific, Runcorn, United Kingdom  
Jim Garvey, Department of Agriculture, Food and the Marine, County Kildare, Ireland

## Keywords

Pesticides, fruits and vegetables, GC Orbitrap mass spectrometry, quantitation, accurate mass, TraceFinder software

## Goal

To demonstrate the quantitative performance of the Thermo Scientific<sup>™</sup> Exactive<sup>™</sup> GC Orbitrap<sup>™</sup> mass spectrometer for the analysis of GC-amenable pesticides and PCBs in grape and onion samples.

## Method

Grape and onion samples were extracted using the mini-Luke procedure<sup>1</sup>. Automatic sample injection was performed using a Thermo Scientific<sup>™</sup> TriPlus<sup>™</sup> RSH autosampler, and chromatographic separation was performed using a Thermo Scientific<sup>™</sup> TRACE<sup>™</sup> 1310 GC system. Data was acquired and processed using Thermo Scientific<sup>™</sup> TraceFinder<sup>™</sup> software, which allows easy instrument control, method development, and quantitation capabilities.


Part Number	Description
26096-1420	Thermo Scientific <sup>™</sup> TraceGOLD <sup>™</sup> TG-5SilMS 30 m × 0.25 mm I.D. × 0.25 μm film capillary column
26096-1425	5 m integrated guard column
453T2120	Siltek six baffle PTV liner

## Conclusion

- Sensitive and robust full-scan analysis allows for easy and flexible data acquisition and processing.
- All 95 compounds were detected at levels below the MRL, with calculated limits of detection of <2 μg/kg for most compounds (92 of the 95 compounds).
- Excellent linearity was demonstrated with  $R^2 > 0.99$  and average response factors RSD% <20 across the 8-point (1–200 μg/kg) matrix-matched calibration series, which ensures accurate quantitation. No internal standards were used to correct the response.
- Blind analysis of a grape and onion sample showed reliable detection and accurate quantitation of spiked compounds.

## Reference

1. Lozano, A.; Kiedrowski, B.; Scholten, J.; de Kroon, M.; de Kok, A.; Fernández-Alba, A.R. Miniaturisation and optimisation of the Dutch mini-Luke extraction method for implementation in the routine multi-residue analysis of pesticides in fruits and vegetables. *Food Chemistry*. 2016, 192, 668–681.

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APPLICATION SUMMARY AN64971

## Robustness, reproducibility, reliability with best-in-class sensitivity: Increased confidence in targeted quantitation of pesticides in food matrices

Katerina Bousova, Michal Godula, Claudia Martins, Charles Yang, Ed George, Neloni Wijeratne, Thermo Fisher Scientific

### Keywords

Pesticide Explorer Collection, European Regulation 396/2005, Commission Directive 2006/125/ EC, SANCO/12571/2013, European Commission 788/2012/EC, pesticide, food, QuEChERS, Vanquish Flex LC system, TSQ Quantis MS, TraceFinder software

### Goal

To present a fully tested LC-MS/MS methodology for rapid and robust quantitation of more than 250 pesticides below maximum residue levels (MRLs) with sensitivity, accuracy, and precision that meet stringent EU guidelines.

### Method

Samples were homogenized and extracted according to the European EN 15662 QuEChERS protocol prior to injection. LC-MS/MS analysis was carried out using a Thermo Scientific™ Vanquish™ Flex Binary UHPLC system coupled to a Thermo Scientific™ TSQ Quantis™ triple quadrupole mass spectrometer. TraceFinder software was used for instrument control, analysis, data review, and reporting. The LC gradient was optimized to reduce analysis time to 15 minutes, while maintaining good chromatographic separation.

### Conclusion

- The method results comply with the stringent EU guidelines concerning sensitivity, accuracy, and precision.
- Rapid and robust quantitation of more than 250 pesticides in leek at or below their respective MRLs was achieved.
- The LC-MS/MS system selectivity and sensitivity enabled analysis of only 1 µL sample, without the need for dispersive SPE sample clean-up or sample dilution.



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Part Number	Description
60105-216	QuEChERS extraction tube, 50 mL, 250 pack
17326-102130	Thermo Scientific™ Accucore™ aQ column 100 × 2.1 mm, 2.6 µm





APPLICATION SUMMARY AN65606

# Trace level quantitation of pesticide residues in high water content vegetables using LC-(HESI)-MS/MS

Ramiz M.R. Azad and Dasharath Oulkar, Thermo Fisher Scientific

## Keywords

QuEChERS, LC-MS/MS, pesticide residues, cucumber, tomato, TraceFinder software, TSQ Quantis MS

## Goal

To demonstrate a simple and sensitive method for trace level quantification of pesticides in high water content vegetables by using LC-MS/MS. The optimized method was validated per the SANTE guidelines and evaluated for the Food Safety Standards Authority of India (FSSAI) as well as European Commission (EC) maximum residue levels (MRLs) compliance in tomato and cucumber.


## Method

Following the QuEChERS method for extraction, optimization and method validation of a multi-residue method for pesticides in tomato and cucumber was performed using LC-MS/MS with the Thermo Scientific™ TSQ Quantis™ triple quadrupole mass spectrometer. The data acquisition and processing were carried out using Thermo Scientific™ TraceFinder™ software.

Part Number	Description
60105-341	QuEChERS Salts (2007.01)
60105-203	150 mg anhydrous magnesium sulfate, 50 mg primary secondary amine
17326-102130	Thermo Scientific™ Accucore™ aQ column, 100 × 2.1 mm, 2.6 μm

## Conclusion

- Using this approach, at least 70 injections (standards, samples, blank) could be completed in a day (24-hour cycle) and increase the overall high throughput of the commercial food testing laboratory.
- The method performance was evaluated at the 1× (0.01 mg/kg, reporting limit), and 0.5× reporting limit. These two levels, recoveries, and precision data meet the SANTE guideline criteria.
- This method complies with the EU as well as FSSAI MRLs requirements.
- This application offers an analytical solution for quantitation of 166 pesticide residues in tomato and 184 in cucumber.

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APPLICATION SUMMARY AN65684

# Multi-pesticide residues analyses of QuEChERS extracts using an automated online $\mu$ SPE clean-up coupled to LC-MS/MS

Long Sun, Qilei Guo, Cristina C. Jacob, Claudia P.B. Martins, Richard Fussell, Thermo Fisher Scientific

## Keywords

Pesticide residues analysis, LC-MS/MS, QuEChERS, online  $\mu$ SPE, clean-up, automation

## Goal

To demonstrate the feasibility of an automated online sample clean-up solution coupled to LC-MS/MS for rapid and robust quantitation of multi-pesticide residues in food matrices.


## Method

After extraction using either the citrate buffered or acetate buffered versions of the QuEChERS methods, automated  $\mu$ SPE clean-up was performed using the robotic Thermo Scientific™ TriPlus™ RSH autosampler system. The LC-MS/MS system comprised a Thermo Scientific™ Vanquish™ Horizon Binary UHPLC system interfaced with a Thermo Scientific™ TSQ Fortis™ triple quadrupole mass spectrometer equipped with a HESI ionization probe. Chromatographic separation was carried out on a Thermo Scientific™ Accucore™ AQ column. The acquired data were processed using Thermo Scientific™ TraceFinder™ 4.1 software, and Thermo Scientific™ Freestyle™ 1.5 software was used for qualitative purposes.

Part Number	Description
60101-30LC	Miniaturized SPE cartridges, (QuEChERS blend for LC)
C4011-LV1	Collection vials (500 $\mu$ L, fused insert snap-top)
C4011-59	Star-slit snap-it caps
17326-102130	Thermo Scientific™ Accucore™ aQ column, 100 mm $\times$ 2.1 mm; 2.6 $\mu$ m

## Conclusion

- The automated  $\mu$ SPE clean-up afforded spike recoveries within 70–120% with corresponding %RSDs below 20% for 98% of the target pesticides in grape, 98% in tea, and 96% in rice extracts.
- The automated  $\mu$ SPE showed reduced matrix effects when compared with d-SPE.
- Analyte responses in complex matrices (black tea) were stable for at least 200 consecutive injections without maintenance.
- The sample throughput is maximized.
- The risk of human error is minimized.
- The efficiency of commercial testing laboratories is greatly improved by replacing the need for a manual clean-up procedure with fully automated and unified operations. The SPE cartridge is applicable to a wide diversity of sample types, so there is reduced need to use different clean-up regimes for different sample types.

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# Rapid analysis of fipronil and fipronil sulfone in eggs by liquid chromatography and triple quadrupole mass spectrometry

Susanne Sales, Siegrun Mohring, and Michal Godula, Thermo Fisher Scientific

## Keywords

Fipronil, fipronil sulfone, eggs, LC-MS, Accucore aQ column, TSQ Quantis MS, UltiMate 3000 RSLC

## Goal

To develop a quick and simple method for the determination of fipronil and fipronil sulfone in eggs using an in-house modified QuEChERS acetonitrile extraction protocol and LC-MS/MS determination.


## Method

A modified QuEChERS acetonitrile extraction protocol was used. The Thermo Scientific™ UltiMate3000™ RSLC with a binary gradient pump was connected to a Thermo Scientific™ TSQ Quantis™ triple quadrupole mass spectrometer. Data evaluation was performed using Thermo Scientific™ TraceFinder™ 4.1 software.

Part Number	Description
60105-340	Thermo Scientific™ HyperSep™ dispersive SPE Mylar pouch 4000 mg magnesium sulfate and 1000 mg NaCl, 50 pk
60105-367-SP	C18 dSPE pouches
12198721	Magnesium sulfate, 99%, for analysis, anhydrous
17326-102130	Thermo Scientific™ Accucore™ aQ column, 100 mm × 2.1 mm, 2.6 μm

## Conclusion

- The limit of quantification and identification of 0.0005 mg/kg for each of fipronil and fipronil sulfone is 5x below the EU statutory MRL for the sum of fipronil and fipronil sulfone.
- The results are in full compliance with the SANTE11945/2015 analytical quality control guidelines for pesticides.
- Sample preparation, using a modified QuEChERS protocol, takes around 15 min and allows high throughput analysis.
- Using Accucore aQ column technology, the run time could be shortened to 8 min while maintaining excellent separation efficiency and robustness.
- The study proves very good robustness of the system, allowing uninterrupted analysis of up to 100 egg samples without the need for any system maintenance.

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APPLICATION SUMMARY AN72953

# Development and validation of single residue method for dithianon in apple and apple juice using LC-MS/MS

Ramiz M.R. Azad and Dasharath Oulkar, Thermo Fisher Scientific

## Keywords

UHPLC, TraceFinder software, pesticide residues, apple, apple juice, QuEChERS, LC-MS/MS

## Goal

To develop and deliver a complete solution that can be implemented for commercial testing laboratory analysis of dithianon in apple and apple juice by using LC-MS/MS.


## Method

Following the QuEChERS method for extraction, analysis was performed using the Thermo Scientific™ Vanquish™ Flex Binary UHPLC system coupled with the Thermo Scientific™ TSQ Quantis™ triple quadrupole mass spectrometer. Thermo Scientific™ TraceFinder™ 4.1 software was used for instrument control, data acquisition, data processing, data review, and reporting.

Part Number	Description
60105-341	QuEChERS Salts (2007.01)
25002-102130	Thermo Scientific™ Hypersil GOLD™ column, 100 × 2.1 mm, 1.9 μm

## Conclusion

- This optimized method fulfills the EU and FSSAI MRLs compliance requirements by achieving an excellent lower limit of quantitation (0.001 mg/kg) without compromising the data quality.
- The method provides highly sensitive, robust, accurate, and precise results for dithianon residue in apple and apple juice.
- This method also demonstrates a simple way for identification and quantitation of dithianon per the regulatory SANTE guideline requirements.
- The optimized protocol could result in high throughput in commercial food testing laboratories.

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APPLICATION SUMMARY AN72961

# Screening and quantitation of pesticide residues in rice using LC-(HESI)-MS/MS

Ramiz M. R. Azad and Dasharath Oulkar, Thermo Fisher Scientific

## Keywords

TraceFinder, pesticide residues, rice, QuEChERS, LC-MS/MS, TSQ Quantis MS

## Goal


To develop a screening solution followed by quantitation of 160 pesticide residues in rice matrix using liquid chromatography-triple quadrupole mass spectrometry. The optimized method was validated in accordance with the SANTE guidelines and in compliance with the requirements of FSSAI and the European Commission (EC) MRLs.

## Method

Extraction was carried out using the acetic acid buffered version of the QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) method. The Thermo Scientific™ Vanquish™ Flex Binary UHPLC system was coupled with a Thermo Scientific™ TSQ Quantis™ triple quadrupole mass spectrometer, which included the Heated Electrospray Ionization (HESI) source. The data acquisition and processing were carried out by using Thermo Scientific™ TraceFinder™ software. The optimized method was validated according to the SANTE/11813/2017 guidelines.

## Conclusion

- This method provides a solution for the simultaneous screening and quantitation of pesticide residues (160 pesticides) in rice.
- Use of the QuEChERS method for extraction followed by LC-MS/MS analysis could increase the overall high throughput of the commercial food testing laboratory.
- By following this approach, at least 50 injections (standards, samples, blank) could be completed in a 24-hour cycle.
- This validated method data meets the SANTE guidelines. Also, this method complies with the EU and FSSAI MRL requirements by achieving an excellent lower limit of quantitation (LOQ).

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Part Number	Description
60105-341	QuEChERS Salts (2007.01)
25002-102130	Thermo Scientific™ Hypersil GOLD™ column, 100 × 2.1 mm, 1.9 μm





APPLICATION SUMMARY AN72982

## A simple and robust method for trace level quantitation of pesticide residues in wheat grain using LC-MS/MS

Ramiz M. R. Azad, Dasharath Oulkar, and Ashutosh Pathak, Thermo Fisher Scientific

### Keywords

TraceFinder, pesticide residues, wheat grain, QuEChERS, LC-MS/MS, TSQ Quantis MS

### Goal


To deliver a total solution for trace level quantitation of 145 pesticide residues in wheat grain by using liquid chromatography-tandem mass spectrometry. The optimized method was validated per the SANTE guidelines and evaluated for the fulfillment of the Food Safety and Standards Authority of India (FSSAI) as well as the European Commission (EC) MRLs compliance in wheat grain.

### Method

The QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) method was used for extraction. The Thermo Scientific™ Vanquish™ UHPLC system was coupled to a Thermo Scientific™ TSQ Quantis™ triple quadrupole mass spectrometer, which included the Heated Electrospray Ionization (HESI) source. The data acquisition and processing were carried out by using Thermo Scientific™ TraceFinder™ software. The optimized method was validated according to the SANTE/11813/ 2017 guidelines.

### Conclusion

- The method provides a solution for a trace-level accurate quantitation of pesticide residues in wheat grain.
- Use of the QuEChERS method for extraction followed by LC-MS/MS analysis could increase the overall high throughput of the commercial food testing laboratory.
- By following this approach at least 50 injections (standards, samples, blank) could be completed in a day (24-hour cycle).
- This validated method data meets the requirements of the SANTE guidelines. Also, this method complies with the EU and FSSAI MRL requirements by achieving an excellent LOQ.

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Part Number	Description
60105-341	QuEChERS Salts (2007.01)
25002-102130	Thermo Scientific™ Hypersil GOLD™ column, 100 × 2.1 mm, 1.9 μm



APPLICATION SUMMARY AN73016

# Trace-level quantitation of pesticide residues in red chili powder using LC-(HESI)-MS/MS

Ramiz M. R. Azad, Dasharath Oulkar, and Ashutosh Pathak, Thermo Fisher Scientific

## Keywords

Pesticide residues, chili powder, QuEChERS, LC-MS/MS, TSQ Quantis MS, TraceFinder software

## Goal

To develop a method for the trace-level quantitation of pesticides and their metabolite residues in chili powder using liquid chromatography-triple quadrupole mass spectrometry. The optimized method performance was verified in accordance with the EU SANTE guidelines and assessed for compliance with the Food Safety and Standards Authority of India (FSSAI) and EU MRLs in chili powder.


## Method

Following the QuEChERS method for extraction, analysis was performed using the Thermo Scientific™ Vanquish™ Flex UHPLC system coupled to a Thermo Scientific™ TSQ Quantis™ triple quadrupole mass spectrometer (fitted with a heated electrospray ionization (HESI) source). The data acquisition and processing methods were carried out using Thermo Scientific™ TraceFinder™ software version 4.1.

Part Number	Description
80020-415-500	Anhydrous magnesium sulfate, Thermo Scientific™
80020-424	Sodium acetate, Thermo Scientific™
80020-416-100	PSA (primary secondary amine), Thermo Scientific™
80020-417-100	GCB (graphitized carbon black), Thermo Scientific
25002-102130	Thermo Scientific™ Hypersil GOLD™ column, 100 × 2.1 mm, 1.9 μm

## Conclusion

- A robust analytical solution for the trace-level (sub ppb) quantitation of more than 120 pesticides in chili powder is demonstrated.
- The method is suitable for high-throughput commercial food testing laboratories.
- By following this approach, at least 70 injections (standards, samples, blank) could be completed in a day (24-hour cycle).
- The method validation data at the reporting limit (RL) meets the recovery and precision requirement per the SANTE guidelines.
- This method complies with the EU and FSSAI MRL requirements by achieving excellent lower limits of quantitation.

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# Trace level quantitation of pesticide residues in fresh fruits using LC-MS/MS

Ramiz M. R. Azad and Dasharath Oulkar, Thermo Fisher Scientific

## Keywords

TraceFinder software, pesticide residues, grape, apple, QuEChERS, LC-MS/MS, TSQ Quantis MS

## Goal


To provide an analytical solution for the trace level quantitation of 160 pesticides (parent, isomers, and metabolites) in table grapes and in apple using liquid chromatography-tandem mass spectrometry. The optimized method was validated in accordance with the EU SANTE guidelines and further evaluated for compliance with the Food Safety and Standards Authority of India (FSSAI) as well as European Union (EU) MRLs.

## Method

For extraction of residues, the AOAC version of the QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) method was selected as it is widely used for the analysis of pesticide residues in fruit. The Thermo Scientific™ Vanquish™ Flex UHPLC system was coupled with the Thermo Scientific™ TSQ Quantis™ quadrupole tandem mass spectrometer with a heated electrospray ionization (HESI) source. The data acquisition and processing methods were carried out using Thermo Scientific™ TraceFinder™ software version 4.1.

## Conclusion

- A sensitive, robust, and low-cost method for the quantification and identification of 160 pesticides in grape and apple is presented.
- Using this approach, at least 70 injections could be completed in a day (24-hour cycle) to increase the sample throughput of commercial food testing laboratories.
- The validation data fully meets the requirement of the EU SANTE guidelines.
- The color-coded flagging of results outside of the acceptance criteria enables faster processing of the data and automatically identifies results in need of further investigation.
- The optimized method meets the EU and FSSAI MRLs for the LC-amenable pesticides in grape and apple.

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Part Number	Description
60105-341	QuEChERS Salts (2007.01)
25002-102130	Thermo Scientific™ Hypersil GOLD™ column, 100 × 2.1 mm, 1.9 μm





APPLICATION SUMMARY AN73245

## Fast analysis of multi-class pesticides panel in wine using single run LC-triple quadrupole mass spectrometry

Ilaria Palini, Silvia Bani, ISVEA, Poggibonsi, Italy;

Debora D'Addona, Charles T. Yang, Ed George, and Dipankar Ghosh, Thermo Fisher Scientific

### Keywords

SANTE/11813/2017, TSQ Quantis MS, Vanquish Flex UHPLC, pesticides, QuEChERS, sample prep, Pesticide Explorer II

### Goal

To demonstrate a fully tested LC-MS/MS workflow for fast and robust quantification of more than 400 pesticides below maximum residue limits (MRLs) with accuracy and precision that meet the EU SANTE guidelines.

### Method

After extraction of ten milliliters of wine, analysis was carried out using a Thermo Scientific™ QuEChERS sample preparation kit, Thermo Scientific™ Vanquish™ Flex Binary UHPLC system, Thermo Scientific™ TSQ Quantis™ triple quadrupole mass spectrometer, Thermo Scientific™ TraceFinder™ software, and a Thermo Scientific™ Accucore™ aQ C18 Polar Endcapped LC column.

Part Number	Description
60105-344-SP	Thermo Scientific™ HyperSep™ Dispersive SPE 50/pack
17326-102130	Accucore aQ column, 100 × 2.1 mm, 2.6 μm

### Conclusion

- The method allowed ISVEA to quickly quantitate samples with confidence at or below the EU MRL for a wide variety of pesticides, giving their clients added confidence in the safety of their products.
- Rapid and robust quantitation of more than 400 pesticides in wine was achieved at or below their respective MRLs, conforming to the EU SANTE guidelines.
- The LC-MS/MS system enabled analysis of 1 μL sample injections, without the need for dispersive SPE sample clean-up or sample dilution, with increased robustness and throughput.
- The screening and quantitation method showed excellent reproducibility in terms of consistent peak shapes and long column lifetime over 1000 injections, with consistent peak response over time.



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APPLICATION SUMMARY AN65722

# A multiresidue method for pesticide profiling using an Orbitrap Tribrid mass spectrometer

Ed George, Seema Sharma, Scott Peterman, and Richard Fussell, Thermo Fisher Scientific

## Keywords

Orbitrap ID-X mass spectrometer, Tribrid, pesticide residues screening, nontargeted, high-resolution accurate-mass, Vanquish UHPLC, identification points, part per billion (ppb), Compound Discoverer software, TraceFinder software, quantitation of pesticide residue, intelligent MS, data dependent acquisition, dynamic exclusion, AcquireX workflow

## Goal

To develop a method based on full scan acquisition that enables the detection and quantitation of multiple targeted pesticide residues and simultaneous post-acquisition screening analysis of unknown/unexpected pesticide residues. The resulting method must be able to acquire compound-specific precursor and product ions with sufficient sensitivity, mass accuracy, and resolution to selectively extract ion intensity attributed to compounds of interest. Meeting these requirements will enable accurate quantitation and help to maintain a high degree of reproducibility and robustness for the duration of the study. In addition, the overall LC-MS<sup>n</sup> workflow must be easy to use, applicable to most sample types and matrices, and extremely efficient.

## Method

Chromatographic separation was performed on a Thermo Scientific™ Vanquish™ Flex Binary UHPLC system using a Thermo Scientific™ Accucore™ aQ C18 column. Mass analysis was performed on a Thermo Scientific™ Orbitrap ID-X™

Tribrid™ mass spectrometer using the Thermo Scientific™ AcquireX™ workflow for automated generation of the background exclusion list and management of data-dependent acquisition (DDA) and dynamic exclusion (DE). Full scan data processing was performed with Thermo Scientific™ TraceFinder™ software.

Part Number	Description
17326-102130	Accucore aQ column, 100 × 2.1 mm, 2.6 μm

## Conclusion

- The incorporation of intelligent MS routines managed by the AcquireX workflow removes the burden of manually creating inclusion/exclusion lists previously needed to ensure pesticide detection and confirmation at LOD/LOQ levels.
- The results presented demonstrate the enhanced profiling capability for large numbers of pesticides across a wide range of residue levels substantially lower than existing MRLs.
- By automating the exclusion list generation and implementation, the AcquireX workflow is easy-to-use, amenable to any matrix, and ideal for post-acquisition analysis of known and unknown pesticides or sample-specific compounds.

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APPLICATION SUMMARY AN73204

# Multi-residue analysis of polar anionic pesticides in food samples using a compact ion chromatography system coupled with tandem mass spectrometry (IC-MS/MS)

Yingchen Li, Qilei Guo, Fausto Pigozzo, Richard J. Fussell, and Beibei Huang, Thermo Fisher Scientific

## Keywords

Polar pesticides, QuPPE, glyphosate, fosetyl-AI, bialaphos, phosphonic acid, MPPA, glufosinate, chlorate, HEPA, AMPA, N-acetyl AMPA, N-acetylglufosinate, ethephon, cyanuric acid, N-acetyl-glyphosate, perchlorate, wheat flour, leek

## Goal

To develop and validate an integrated sample-to-result analytical workflow based on ion chromatography (IC) coupled with triple quadrupole mass spectrometry (MS/MS) for the multi-residue determination of polar anionic pesticides and perchlorate in representative food matrices. The performance of this Thermo Scientific™ Anionic Pesticides Explorer workflow must be robust in analysis and the results compliant with EU SANTE/11813/2017 method validation and ongoing quality control guideline criteria. Also, the analysis should meet the residue definitions and maximum residue levels (MRLs) or tolerance values applicable in the European Union, United States, Japan, and China.

## Method


Extraction of the samples was based on a modification of the QuPPE Method. A Thermo Scientific™ Dionex™ Integrion™ HPIC™ system, fitted with a Thermo Scientific™ Dionex™ electrolytic eluent generator cartridge (EGC) and conductivity cell, was coupled to a Thermo Scientific™ Dionex™ AS-AP Autosampler and

Thermo Scientific™ TSQ Altis™ Triple Quadrupole Mass Spectrometer. The system control, data acquisition, and data processing were done using Thermo Scientific™ Chromeleon™ Chromatography Data System software or Thermo Scientific™ TraceFinder™ software.

Part Number	Description
083225	Thermo Scientific™ Dionex™ IonPac™ AG19-4µm guard column, 2 × 50 mm
083223	Thermo Scientific™ Dionex™ IonPac™ AS19-4µm analytical column, 2 × 250 mm

## Conclusion

- This workflow, which is compliant with EU SANTE guidelines, supplies a sensitive and reliable method for simultaneous multiresidue analysis of polar anionic pesticides in complex samples.
- Extensive testing over several months and more than 1500 sample injections has demonstrated the Anionic Pesticides Explorer to be reliable, reproducible, and robust and hence suitable for commercial testing laboratories.

 [View the full application note](#)



# Determination of cationic polar pesticides in homogenized fruit and vegetable samples using IC-HRAM MS

Terri Christison, John E. Madden, and Jeff Rohrer, Thermo Fisher Scientific, Sunnyvale, CA, USA

## Keywords

IonPac CS17 column, RFIC, Reagent-Free IC, Integrion HPIC system, accurate mass spectrometry, multiresidue, IC-MS

## Goal

To demonstrate the determination of six cationic polar pesticides in food samples using ion chromatography with the Thermo Scientific™ Q Exactive™ Hybrid Quadrupole-Orbitrap mass spectrometer.


## Method

Homogenized food samples were prepared according to a simplified version of the EURL FV Quick Polar Pesticides Extraction (QuPPE) method using methanol in place of acidified methanol. Analysis was performed with a Thermo Scientific™ Dionex™ Integrion™ HPIC™ High Pressure system with reagent-free ion chromatography (RFIC) capabilities coupled to a Thermo Scientific™ Q Exactive™ Focus hybrid quadrupole-Orbitrap HRAM-MS with HESI II probe.

Part Number	Description
060563	Thermo Scientific™ Dionex™ IonPac™ CG17 guard column
060561	Thermo Scientific™ Dionex™ IonPac™ CS17 separation column

## Conclusion

- In these experiments, the ddMS Parallel Reaction Monitoring mode used to extract ions of interest from the matrix was effective for qualitative and quantitative determinations in homogenized food samples.
- The IC method demonstrated high accuracy (80% recoveries for most of the samples, with the exception of squash) and sensitivity (LOD <1.1 µg/L). The squash sample exhibited recoveries <80%.
- The HRAM capability easily resolved co-eluting diquat and paraquat using the confirmatory ions, without any indication of suppression.

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