# Fast Screening and Quantification of Pesticide Residues Using a Comprehensive LC-MS Solution: The Pesticide Explorer Collection – Standard Quantitation

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## Key Words
Pesticide Explorer Collection, 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, UltiMate 3000, TSQ Endura, TraceFinder

## Goal
To present a fully tested LC-MS/MS workflow for rapid and robust quantification of more than 250 pesticides below maximum residue limits (MRLs) with sensitivity, accuracy, and precision that meets stringent EU guidelines.

## Introduction
Pesticides are chemicals used on crops to protect them from the negative activity of pests. Inappropriate application of pesticides can have adverse effects on health; therefore, determination and quantification of pesticide residues in foods and food products is an important part of routine food control. The European Union (EU) legislation (European Regulation 396/2005 and Commission Directive 2006/125/EC) requires an extensive and comprehensive study determining pesticides in various products of plant and animal origin. The requirements for low limits of quantification (LOQ) of pesticides pose significant analytical challenges, especially for some complicated food matrices.

This study presents a multi-residue analysis method enabled by Thermo Scientific™ Pesticide Explorer Collection Standard Quantitation Solution, comprising liquid chromatography–triple-stage mass spectrometry (LC-MS/MS), for rapid and robust quantitation of more than 250 pesticides below their required maximum residue limits (MRL). This comprehensive solution includes the Thermo Scientific™ QuEChERS sample preparation kit, Dionex™ UltiMate™ 3000 LC system, TSQ Endura™ triple quadrupole mass spectrometer, TraceFinder™ software, Accucore™ aQ column, and method parameters to provide a start-to-finish workflow for pesticide analysis. The method results address the stringent EU guidelines concerning sensitivity, accuracy and precision.

## Experimental
### Overview
The workflow overview from sample preparation through LC-MS/MS analysis is shown in Figure 1. Samples were homogenized and extracted according to the European EN 15662 QuEChERS protocol prior to injection into the LC-MS/MS system.[1,2] The ready-to-use QuEChERS sample preparation kit containing extraction tubes and associated protocol was used for sample preparation. Identification of pesticide residues was based on retention time, the presence of a minimum of two product ions, and ion-ratio confirmation using selected reaction monitoring (SRM) of characteristic transition ions. Quantification was calculated using matrix-matched calibration. All method performance criteria were established according to the relevant EU guidelines.[3-7]

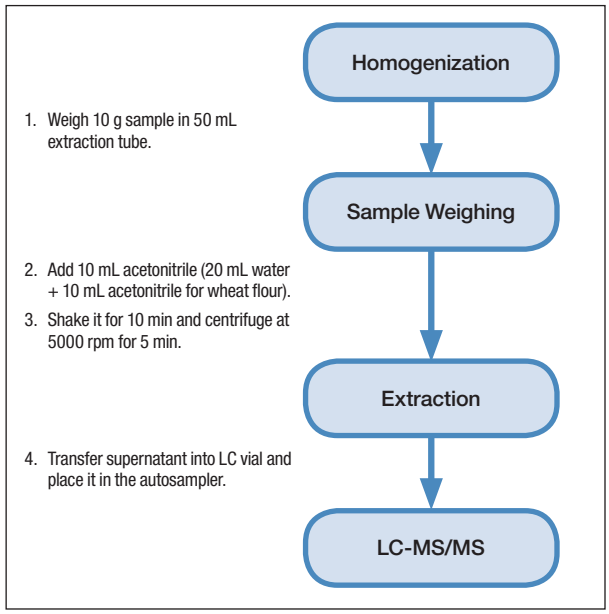


Figure 1. Workflow overview.

**Pesticide Explorer Collection:**
www.thermoscientific.com/Pesticides-LCMS

## Method Supplies

Table 1 lists reagents, instruments, and consumables used.

The pesticides standards were purchased from Sigma-Aldrich ® (Germany) and Lab. Instruments Srl (CASTELLANA GROTTE, Italy). Quality control materials used were FAPAS #T19140 (lettuce puree), FAPAS #19110 (lettuce puree), FAPAS #T19142 (melon puree), and FAPAS #T0983 (wheat flour). FAPAS samples were selected primarily based on their content of target pesticides. However, due to limited availability, some of the matrices are different from the matrices spiked and analyzed (i.e. lettuce and melon puree versus strawberry and leek).

Table 1. Reagents, instruments, and consumables.

| Method Supplies | Fisher Scientific Part Number/ Source |
|---|---|
| Reagents | |
| Acetonitrile (ACN), LC/MS grade | AC61514-0025 |
| Ammonium formate, 99% | AC40115-2500 |
| Formic acid, Optima™ LC/MS grade | A117-50 |
| Methanol, Optima™ LC/MS grade | A456-212 |
| Purified water | Obtained from Thermo Scientific™ Barnstead™ Easypure II™ water system |
| Water, LC-MS grade | AC61515-0025 |
| Instruments | |
| TSQ Endura triple quadrupole mass spectrometer | |
| UltiMate 3000 RSLC | |
| **Method Supplies** | **Thermo Fisher Scientific Part Number** |
| Consumables | |
| QuEChERS extraction tube, 50 mL, 250 pack | 60105-216 |
| Accucore aQ column 100 x 2.1 mm, 2.6 µm | 17326-102130 |

## Sample Preparation

Blank matrix samples [strawberry (SB), wheat flour (WF) and leek (LK)] used for validation experiments were purchased in local retail stores and were homogenized with an Ultra-Turrax homogenizer and extracted prior to fortified sample preparation. Matrix extracts were used as matrix blank samples and for preparation of matrix-matched calibration standards. Ready to use QuEChERS extraction kits were used for sample preparation, and contained 4 g $MgSO_4$, 1 g NaCl, 1 g trisodium citrate dehydrate, and 0.5 g sodium citrate for buffered extraction of target compounds. The same QuEChERS sample preparation protocol was applied to all three of the matrices analyzed, however a modification was made for flour in which water was added to wet the matrix. No cleanup was used.

Homogenization of matrices was performed using the following steps:

1. A relatively large amount of each matrix (~500 g) was placed into an appropriately sized beaker and labeled.
2. A G25 dispergation tool was attached to the Ultra-Turrax homogenizer. (Note: For better recovery for some unstable compounds cryogenic homogenization is advised. [8]).
3. Homogenization was performed at middle rotation speed (speed level 2–3) to create smooth homogenate.

Sample extraction was performed using the following steps:

1. 10 g sample was weighed into a 50 mL QuEChERS extraction tube.
2. 10 mL ACN was added to the SB and LK samples. For WF, 20 mL water was added to completely wet samples, and then 10 mL ACN was added.
3. Samples were shaken for 10 min on a horizontal shaker and centrifuged at 5000 rpm for 5 min.
4. The supernatant was collected and 1 mL was transferred into a LC vial for instrumental analysis.

### LC-MS/MS Analysis

LC-MS/MS analysis was carried out using an UltiMate 3000 RSLC system coupled to a TSQ Endura triple quadrupole mass spectrometer. TraceFinder software (revision 3.2 SP2) was used for instrument control, analysis, data review, and reporting. The LC conditions and gradient are shown in Tables 2 and 3. The LC gradient was optimized to reduce analysis time to 15 minutes, while maintaining good chromatographic separation.

Table 2. LC conditions.

| LC conditions | |
|---|---|
| Injection volume | 1 µL |
| Column temperature | 25 °C |
| Flow rate | 300 µL/min |
| Analytical column | Accucore aQ column, 100 x 2.1 mm, 2.6 µm |
| Run time | 15 minutes |
| Tray temperature | 10 °C |
| Needle-cleaning solvent | 20% Methanol in water |
| Sample loop | 100 µL |
| Mobile phases | A: Water with 5 mM ammonium formate and 0.1% formic acid |
| | B: Methanol with 5 mM ammonium formate and 0.1% formic acid |

Table 3. LC gradient.

| Time (min) | Flow (mL/min) | A% | B% |
|---|---|---|---|
| 0 | 0.300 | 100 | 0 |
| 0.5 | 0.300 | 100 | 0 |
| 7 | 0.300 | 30 | 70 |
| 9 | 0.300 | 0 | 100 |
| 12 | 0.300 | 0 | 100 |
| 12.1 | 0.300 | 100 | 0 |
| 15 | 0.300 | 100 | 0 |

The TSQ Endura triple quadrupole mass spectrometer was operated in timed-SRM mode. All SRM traces (parent, qualifier, quantifier ion) were individually tuned for each target analyte by direct infusion of each working standard solution. The mass spectrometer settings are provided in Table 4. For convenience and fast method implementation, the complete method including SRM settings is included with the Pesticide Explorer Collection Standard Quantitation Configuration.

Table 4. MS settings.

| MS settings | |
|---|---|
| Ionization mode | Heated electrospray (HESI) |
| Scan type | Timed-SRM |
| Polarity | Positive/Negative switching |
| Spray voltage for Positive mode | 3700 V |
| Spray voltage for Negative mode | 2500 V |
| Sheath gas pressure | 30 arbitrary units (Arb) |
| Aux gas pressure | 6 Arb |
| Sweep gas pressure | 1 Arb |
| Ion transfer tube temperature | 325 °C |
| Vaporizer temperature | 350 °C |
| CID gas pressure | 2 mTorr |
| Cycle time | 0.5 s |
| Q1 resolution (FWHM) | 0.7 |
| Q3 resolution (FWHM) | 0.7 |
| Chrom filter | 3 s |

## Results and Discussion

To evaluate method performance, three matrices, strawberry, leek (the most complex), and wheat flour, were analyzed. European Union guidelines for single laboratory validation and pesticide residue analysis were used to establish method performance criteria, including linearity, matrix effect, LOD, LOQ, precision, and trueness (bias). All method performance parameters were compared to the relevant legislative requirements and MRLs. For compounds containing more than one isoform, only one performance criterion was established.

Figure 2 shows the LC-MS/MS chromatogram of the strawberry extract spiked with more than 250 pesticides at a concentration of 100 µg/kg (1 µL injection). Despite the short chromatographic run time (15 min), good separation and detection of the pesticide compounds were achieved using the timed-SRM mode. With timed-SRM, data acquisition for a particular target compound is performed in a short retention time window around the known compound retention time. Timed-SRM significantly reduces the number of SRM transitions that are monitored in parallel within a certain retention time window. A longer measurement time (dwell time) is therefore available for each transition, resulting in higher sensitivity and lower quantitation limits, improved RSDs and more data points per chromatographic peak—in this case a minimum of 10 to 12 data points.

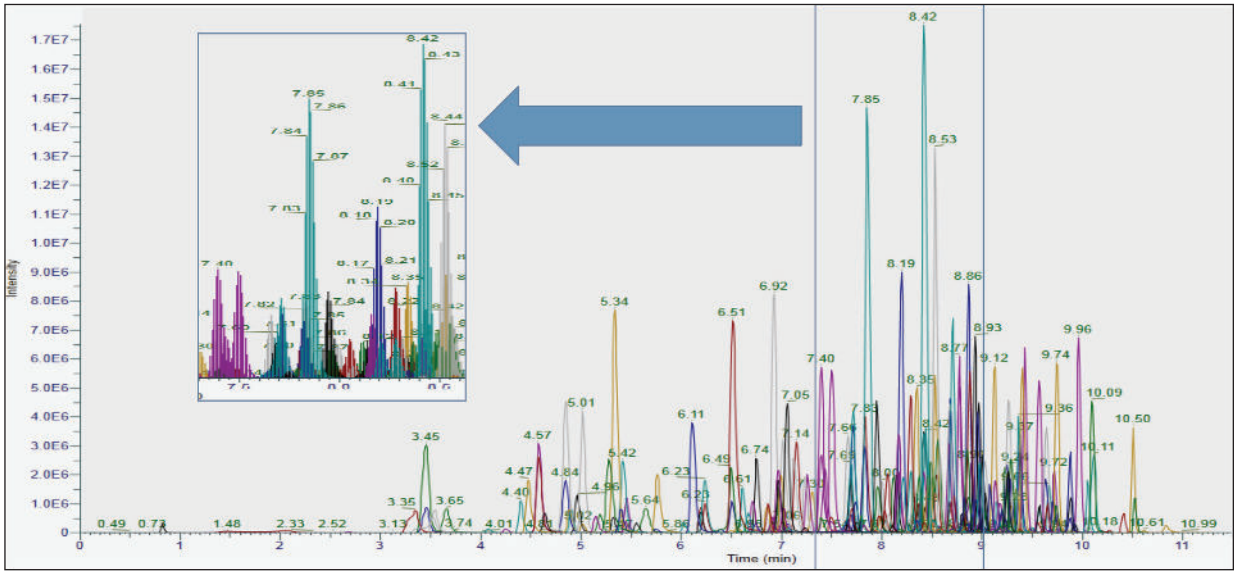


Figure 2. The LC-MS/MS chromatogram of more than 250 pesticides spiked into strawberry extract at 100 µg/kg shows good separation of compounds. Enough scans across the chromatographic peak were obtained throughout the chromatogram.

For the three matrices, including the very complex leek matrix, the LOD and LOQ values obtained demonstrated that the method enabled quantification of target pesticides below regulated MRLs. Table 5 presents the method LODs and LOQs for the target pesticides in the matrices tested. Table 6 compares the LOQ values obtained with the MRLs for selected pesticides. The pesticides selected in Table 6 represent different ionization modes and a range of retention times across the chromatogram. All compounds were detected and quantified below established MRLs.

Table 5. Method performance: LODs and LOQs (µg/kg) for target pesticides by matrix tested. LOQs were estimated taking into account reproducibility (RSDs≤ 15%) and ion ratio criteria.

| | Name | Polarity | RT | Strawberry | | Leek | | Flour | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | LOD | LOQ | LOD | LOQ | LOD | LOQ |
| **1** | 2,4-D | - | 7.6 | 5 | 10 | 5 | 10 | 2 | 5 |
| **2** | Abamectin b1a ($NH_4$) | + | 10.2 | 0.1 | 0.2 | 0.3 | 1 | 0.3 | 1 |
| **3** | Acephate | + | 2.9 | 0.5 | 1 | 0.5 | 1 | 1 | 3 |
| **4** | Acetamiprid | + | 5.6 | 0.1 | 0.5 | 0.3 | 1 | 0.1 | 0.3 |
| **5** | Acibenzolar-S-methyl | + | 8.8 | 1 | 2 | 2 | 5 | 0.1 | 0.3 |
| **6** | Alachlor | + | 8.9 | 1 | 5 | 1 | 5 | 1 | 3 |
| **7** | Aldicarb sulfone | + | 4.8 | 0.5 | 1 | 0.5 | 1 | 0.3 | 1 |
| **8** | Allethrin | + | 8.7 | 0.3 | 1 | 0.3 | 1 | 1 | 3 |
| **9** | Ametryn | + | 7.8 | 0.2 | 0.6 | 0.1 | 0.3 | 0.1 | 0.3 |
| **10** | Aminocarb | + | 3.5 | 0.05 | 0.1 | 0.05 | 0.1 | 0.03 | 0.1 |
| **11** | Ancymidol | + | 7.1 | 0.5 | 1 | 0.5 | 1 | 0.1 | 0.3 |
| **12** | Anilofos | + | 9.1 | 0.03 | 0.1 | 0.03 | 0.1 | 0.1 | 0.3 |
| **13** | Aramite ($NH_4$) | + | 9.7 | 0.03 | 0.1 | 0.03 | 0.1 | 0.03 | 0.1 |
| **14** | Atrazine | + | 7.7 | 0.1 | 0.5 | 0.1 | 0.5 | 0.03 | 0.1 |
| **15** | Azaconazole | + | 8.0 | 0.5 | 1 | 0.1 | 0.5 | 0.2 | 0.6 |
| **16** | Azamethiphos | + | 6.7 | 0.05 | 0.1 | 0.3 | 1 | 0.1 | 0.3 |
| **17** | Azinphos-ethy | + | 8.8 | 5 | 10 | 5 | 10 | 0.3 | 1 |
| **18** | Azinphos-methyl | + | 8.2 | 0.5 | 1 | 1 | 5 | 1 | 5 |
| **19** | Azoxystrobin | + | 8.2 | 0.003 | 0.01 | 0.003 | 0.01 | 0.1 | 0.3 |
| **20** | Bendiocarb | + | 6.9 | 0.3 | 1 | 0.3 | 1 | 0.3 | 1 |
| **21** | Benodanil | + | 7.7 | 0.1 | 0.5 | 0.1 | 0.3 | 0.1 | 0.3 |
| **22** | Benoxacor | + | 8.1 | 0.3 | 1 | 0.3 | 1 | 0.3 | 1 |
| **23** | Bensulfuron methyl | + | 8.1 | 0.1 | 0.5 | 0.3 | 1 | 0.3 | 1 |
| **24** | Bentazon | - | 6.7 | 0.3 | 1 | 0.2 | 0.6 | 0.1 | 0.3 |
| **25** | Benzoximate | + | 9.3 | 0.1 | 0.5 | 0.3 | 1 | 0.3 | 1 |
| **26** | Benzoylprop-ethyl | + | 9.2 | 0.1 | 0.5 | 0.3 | 1 | 0.3 | 1 |
| **27** | Bifenazate | + | 8.7 | 0.3 | 1 | 2 | 5 | 2 | 5 |
| **28** | Bitertanol | + | 9.3 | 0.5 | 2 | 0.5 | 2 | 2 | 5 |
| **29** | Boscalid | + | 8.4 | 0.5 | 1 | 0.05 | 0.1 | 0.1 | 0.3 |
| **30** | Brodifacoum | + | 10.4 | 0.1 | 0.5 | 0.2 | 0.5 | 0.2 | 0.6 |
| **31** | Bromacil | + | 6.9 | 0.3 | 1 | 0.3 | 1 | 0.3 | 1 |
| **32** | Bromoxynil | + | 7.6 | 0.1 | 0.5 | 0.2 | 0.6 | 0.1 | 0.3 |
| **33** | Bromuconazole | + | 8.7 | 0.5 | 1 | 0.5 | 1 | 1 | 3 |
| **34** | Bupirimate | + | 8.8 | 0.5 | 1 | 0.5 | 1 | 0.3 | 1 |
| **35** | Buprofezin | + | 9.7 | 0.2 | 0.5 | 0.3 | 1 | 0.3 | 1 |
| **36** | Butachlor | + | 9.8 | 0.2 | 0.6 | 1 | 3 | 2 | 5 |
| **37** | Butafenacil ($NH_4$) | + | 8.7 | 0.1 | 0.3 | 0.1 | 0.3 | 0.03 | 0.1 |
| **38** | Butocarboxim sulfoxide | + | 3.5 | 0.5 | 1 | 1 | 3 | 1 | 3 |
| **39** | Butoxycarboxim | + | 4.8 | 0.1 | 0.3 | 0.1 | 0.3 | 0.3 | 1 |
| **40** | Carbaryl | + | 7.3 | 0.3 | 1 | 0.3 | 1 | 0.3 | 1 |
| **41** | Carbendazim | + | 4.6 | 0.2 | 0.5 | 0.2 | 0.5 | 0.1 | 0.3 |
| **42** | Carbetamide | + | 6.6 | 0.03 | 0.1 | 0.1 | 0.5 | 0.3 | 1 |
| **43** | Carbofuran | + | 6.9 | 0.03 | 0.1 | 0.03 | 0.1 | 0.03 | 0.1 |
| **44** | Carbofuran-3-hydroxy | + | 5.4 | 0.3 | 1 | 0.3 | 1 | 0.3 | 1 |
| **45** | Carfentrazone-ethyl | + | 9.0 | 0.3 | 1 | 0.03 | 0.1 | 0.1 | 0.3 |
| **46** | Carpropamid | + | 9.2 | 0.3 | 1 | 0.3 | 1 | 0.1 | 0.3 |
| **47** | Chlorantraniliprole | + | 8.0 | 0.3 | 1 | 0.3 | 1 | 0.3 | 1 |

| | Name | Polarity | RT | Strawberry | | Leek | | Flour | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | LOD | LOQ | LOD | LOQ | LOD | LOQ |
| **48** | Chlorbromuron | + | 8.6 | 0.2 | 0.5 | 0.3 | 1 | 0.3 | 1 |
| **49** | Chlorfenvinphos | + | 9.1 | 0.03 | 0.1 | 0.05 | 0.1 | 0.1 | 0.3 |
| **50** | Chlorfluazuron | + | 10.1 | 0.3 | 1 | 0.3 | 1 | 0.1 | 0.3 |
| **51** | Chloridazon (pyrazone) | + | 5.6 | 0.3 | 1 | 0.3 | 1 | 0.3 | 1 |
| **52** | Chlormequat | + | 0.7 | 0.2 | 0.5 | 0.03 | 0.1 | 0.01 | 0.03 |
| **53** | Chlorotoluron | + | 7.7 | 0.3 | 1 | 0.3 | 1 | 0.3 | 1 |
| **54** | Chloroxuron | + | 8.8 | 0.2 | 0.5 | 0.2 | 0.6 | 0.1 | 0.3 |
| **55** | Chlorpyrifos | + | 9.9 | 0.03 | 0.1 | 0.1 | 0.3 | 0.3 | 1 |
| **56** | Cinosulfuron | + | 6.7 | 0.03 | 0.1 | 0.2 | 0.6 | 0.001 | 0.005 |
| **57** | Clethodim | + | 9.5 | 0.3 | 1 | 2 | 6 | 2 | 5 |
| **58** | Clomazone | + | 8.2 | 0.03 | 0.1 | 0.03 | 0.1 | 0.1 | 0.3 |
| **59** | Clothianidin | + | 5.2 | 0.3 | 1 | 0.5 | 2 | 1 | 3 |
| **60** | Coumaphos | + | 9.2 | 0.3 | 1 | 0.3 | 1 | 0.3 | 1 |
| **61** | Crotoxyphos ($NH_4$) | + | 8.4 | 0.03 | 0.1 | 0.1 | 0.3 | 0.3 | 1 |
| **62** | Cumyluron | + | 8.7 | 0.03 | 0.1 | 0.3 | 1 | 0.1 | 0.3 |
| **63** | Cyanazine | + | 6.7 | 0.03 | 0.1 | 0.3 | 1 | 0.1 | 0.3 |
| **64** | Cyazofamid | + | 8.9 | 0.03 | 0.1 | 0.3 | 1 | 0.3 | 1 |
| **65** | Cycloate | + | 9.5 | 0.3 | 1 | 0.3 | 1 | 0.1 | 0.3 |
| **66** | Cycluron | + | 7.9 | 0.3 | 1 | 0.3 | 1 | 0.1 | 0.3 |
| **67** | Cyflufenamid | + | 9.2 | 0.3 | 1 | 0.1 | 0.3 | 0.1 | 0.3 |
| **68** | Cyromazine | + | 1.7 | 2 | 5 | 2 | 5 | 5 | 10 |
| **69** | Demeton-S-methyl sulfone | + | 4.4 | 0.03 | 0.1 | 0.1 | 0.3 | 0.1 | 0.3 |
| **70** | Desmedipham | + | 8.0 | 10 | 30 | 2 | 5 | 10 | 20 |
| **71** | Desmethyl-pirimicarb | + | 5.0 | 0.1 | 0.5 | 0.2 | 0.6 | 0.3 | 1 |
| **72** | Desmetryn | + | 7.1 | 0.1 | 0.5 | 0.2 | 0.6 | 0.2 | 0.6 |
| **73** | Diclobutrazol | + | 8.8 | 0.3 | 1 | 1 | 3 | 0.1 | 0.3 |
| **74** | Dicrotophos | + | 4.9 | 0.3 | 1 | 1 | 3 | 1 | 3 |
| **75** | Diethofencarb | + | 8.1 | 0.2 | 0.5 | 0.3 | 1 | 0.1 | 0.3 |
| **76** | Difenacoum | + | 10.1 | 0.03 | 0.1 | 0.2 | 0.6 | 0.1 | 0.3 |
| **77** | Difenoconazole | + | 9.4 | 0.2 | 0.5 | 0.03 | 0.1 | 0.03 | 0.1 |
| **78** | Diflubenzuron | + | 9.0 | 0.3 | 1 | 0.1 | 0.3 | 0.03 | 0.1 |
| **79** | Dimefuron | + | 8.0 | 0.1 | 0.3 | 0.3 | 1 | 0.3 | 1 |
| **80** | Dimethametryn | + | 8.9 | 0.1 | 0.3 | 0.1 | 0.3 | 0.2 | 0.6 |
| **81** | Dimethenamid | + | 8.4 | 0.3 | 1 | 0.1 | 0.3 | 0.1 | 0.3 |
| **82** | Dimethoate | + | 5.5 | 0.03 | 0.1 | 0.1 | 0.3 | 0.1 | 0.3 |
| **83** | Dimethomorph | + | 8.3 | 0.3 | 1 | 0.03 | 0.1 | 0.1 | 0.3 |
| **84** | Dimoxystrobin | + | 9.0 | 0.2 | 0.5 | 0.03 | 0.1 | 0.1 | 0.3 |
| **85** | Diniconazole | + | 9.4 | 0.3 | 1 | 0.3 | 1 | 0.3 | 1 |
| **86** | Dinotefuran | + | 3.7 | 0.3 | 1 | 0.3 | 1 | 0.3 | 1 |
| **87** | Dithiopyr | + | 9.5 | 0.3 | 1 | 2 | 5 | 0.3 | 1 |
| **88** | Diuron | + | 7.5 | 0.03 | 0.1 | 0.1 | 0.3 | 0.3 | 1 |
| **89** | DNOC | - | 7.7 | 0.3 | 1 | 1 | 3 | 0.3 | 1 |
| **90** | Dodemorph | + | 8.1 | 0.3 | 1 | 1 | 3 | 0.3 | 1 |
| **91** | Epoxiconazole | + | 8.9 | 0.3 | 1 | 0.03 | 0.1 | 0.1 | 0.3 |
| **92** | Esprocarb | + | 9.7 | 0.03 | 0.1 | 0.1 | 0.3 | 0.1 | 0.3 |
| **93** | Etaconazol | + | 8.8 | 0.3 | 1 | 0.3 | 1 | 0.1 | 0.3 |
| **94** | Ethiofencarb | + | 8.2 | 0.3 | 1 | 2 | 5 | 1 | 3 |
| **95** | Ethiofencarb-sulfone | + | 4.9 | 0.3 | 1 | 0.3 | 1 | 0.1 | 0.3 |

| | Name | Polarity | RT | Strawberry | | Leek | | Flour | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | LOD | LOQ | LOD | LOQ | LOD | LOQ |
| **96** | Ethiofencarb-sulfoxide | + | 5.0 | 0.03 | 0.1 | 0.03 | 0.1 | 0.1 | 0.3 |
| **97** | Ethiprole | + | 8.3 | 0.3 | 1 | 1 | 3 | 1 | 3 |
| **98** | Ethirimol | + | 6.2 | 0.3 | 1 | 0.1 | 0.5 | 0.1 | 0.3 |
| **99** | Ethofumesate | + | 8.3 | 0.3 | 1 | 5 | 20 | 5 | 20 |
| **100** | Ethoxyquin | + | 7.7 | 0.3 | 1 | 1 | 3 | 0.3 | 1 |
| **101** | Etofenprox ($NH_4$) | + | 10.5 | 0.03 | 0.1 | 0.3 | 1 | 1 | 3 |
| **102** | Etoxazole | + | 10.0 | 0.02 | 0.05 | 0.03 | 0.1 | 0.1 | 0.3 |
| **103** | Etrimfos | + | 9.1 | 0.3 | 1 | 0.1 | 0.3 | 1 | 3 |
| **104** | Fenamidone | + | 8.3 | 0.03 | 0.1 | 0.3 | 1 | 0.3 | 1 |
| **105** | Fenamiphos | + | 8.9 | 0.03 | 0.1 | 0.03 | 0.1 | 0.03 | 0.1 |
| **106** | Fenarimol | + | 8.8 | 0.3 | 1 | 0.1 | 0.3 | 0.1 | 0.3 |
| **107** | Fenazaquin | + | 10.5 | 0.1 | 0.3 | 1 | 3 | 0.5 | 1.5 |
| **108** | Fenbuconazole | + | 8.9 | 0.3 | 1 | 0.3 | 1 | 2 | 5 |
| **109** | Fenhexamid | + | 8.7 | 0.003 | 0.01 | 1 | 3 | 0.3 | 1 |
| **110** | Fenobucarb | + | 8.2 | 0.03 | 0.1 | 0.1 | 0.3 | 0.1 | 0.3 |
| **111** | Fenoxanil | + | 9.4 | 0.1 | 0.3 | 0.03 | 0.1 | 0.1 | 0.3 |
| **112** | Fenoxycarb | + | 9.0 | 0.1 | 0.5 | 0.03 | 0.1 | 0.03 | 0.1 |
| **113** | Fenpyroximat | + | 10.1 | 0.003 | 0.01 | 0.01 | 0.03 | 0.03 | 0.1 |
| **114** | Fensulfothion | + | 7.8 | 0.03 | 0.1 | 0.03 | 1 | 0.3 | 1 |
| **115** | Fenthion | + | 9.2 | 0.3 | 1 | 1 | 3 | 0.3 | 1 |
| **116** | Fenthion-sulfoxide | + | 7.3 | 0.06 | 0.2 | 0.1 | 0.3 | 0.1 | 0.3 |
| **117** | Fenuron | + | 5.3 | 0.1 | 0.3 | 0.1 | 0.3 | 0.03 | 0.1 |
| **118** | Flazasulfuron | + | 8.1 | 0.1 | 0.3 | 0.3 | 1 | 0.03 | 0.1 |
| **119** | Florasulam | + | 6.2 | 0.1 | 0.3 | 0.03 | 0.1 | 0.1 | 0.3 |
| **120** | Fluazifop | + | 8.3 | 0.3 | 0.6 | 0.3 | 1 | 1 | 3 |
| **121** | Fluazinam | - | 9.7 | 0.03 | 0.1 | 0.3 | 1 | 0.1 | 0.3 |
| **122** | Flubendiamide | + | 9.0 | 1.5 | 5 | 1.5 | 5 | 5 | 10 |
| **123** | Flufenacet | + | 8.8 | 0.1 | 0.3 | 0.3 | 1 | 0.1 | 0.3 |
| **124** | Flufenoxuron | + | 9.9 | 0.3 | 1 | 0.1 | 0.3 | 0.3 | 1 |
| **125** | Flumetsulam | + | 5.3 | 0.1 | 0.3 | 0.3 | 1 | 0.3 | 1 |
| **126** | Fluometuron | + | 7.5 | 0.3 | 1 | 0.1 | 0.3 | 0.03 | 0.1 |
| **127** | Fluopicolide | + | 8.5 | 0.3 | 1 | 0.1 | 0.3 | 0.1 | 0.3 |
| **128** | Fluopyram | + | 8.7 | 0.03 | 0.1 | 0.03 | 0.1 | 0.1 | 0.3 |
| **129** | Fluorochloridone | + | 8.7 | 0.3 | 1 | 0.3 | 1 | 1 | 3 |
| **130** | Fluoxastrobin | + | 8.7 | 0.2 | 0.5 | 0.3 | 1 | 0.1 | 0.3 |
| **131** | Fluquinconazole | + | 8.7 | 0.3 | 1 | 0.5 | 1 | 0.3 | 1 |
| **132** | Flusilazole | + | 9.0 | 0.2 | 0.5 | 0.2 | 0.5 | 0.2 | 0.6 |
| **133** | Flutriafol | + | 7.7 | 0.1 | 0.3 | 0.3 | 1 | 0.3 | 1 |
| **134** | Forchlorfenuron | + | 8.0 | 0.1 | 0.5 | 0.1 | 0.3 | 0.1 | 0.3 |
| **135** | Formetanate hydrochloride | + | 3.4 | 0.3 | 1 | 0.3 | 1 | 0.1 | 0.3 |
| **136** | Formothion | + | 6.6 | 2 | 5 | 2 | 5 | 3 | 10 |
| **137** | Fosthiazate | + | 7.4 | 0.1 | 0.3 | 0.1 | 0.3 | 0.01 | 0.03 |
| **138** | Fuberidazole | + | 5.4 | 0.2 | 0.6 | 1 | 3 | 0.3 | 1 |
| **139** | Furathiocarb | + | 9.6 | 0.3 | 1 | 5 | 10 | 1 | 3 |
| **140** | Griseofulvin | + | 7.7 | 0.3 | 0.6 | 0.3 | 1 | 0.3 | 1 |
| **141** | Halofenozide | - | 8.4 | 0.3 | 0.6 | 0.3 | 1 | 0.01 | 0.03 |
| **142** | Haloxyfop | + | 8.9 | 0.3 | 1 | 0.1 | 0.3 | 0.03 | 0.1 |
| **143** | Haloxyfop-methyl | + | 9.4 | 0.02 | 0.05 | 0.3 | 1 | 0.1 | 0.3 |

| | Name | Polarity | RT | Strawberry | | Leek | | Flour | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | LOD | LOQ | LOD | LOQ | LOD | LOQ |
| **144** | Heptenophos | + | 7.9 | 0.3 | 1 | 1 | 3 | 0.3 | 1 |
| **145** | Hexaconazole | + | 9.2 | 0.3 | 1 | 0.5 | 1.5 | 0.3 | 1 |
| **146** | Hexaflumuron | - | 9.5 | 0.3 | 1 | 0.3 | 1 | 0.1 | 0.3 |
| **147** | Hexazinone | + | 7.0 | 0.03 | 0.1 | 0.1 | 0.3 | 0.1 | 0.3 |
| **148** | Hexythiazox | + | 9.9 | 0.03 | 0.1 | 0.1 | 0.3 | 0.1 | 0.3 |
| **149** | Imazalil | + | 7.7 | 0.3 | 1 | 0.1 | 0.3 | 0.3 | 1 |
| **150** | Imazaquin | + | 7.0 | 0.2 | 0.6 | 0.3 | 1 | 0.1 | 0.3 |
| **151** | Imazethapyr | + | 6.5 | 0.03 | 0.1 | 0.1 | 0.3 | 0.1 | 0.3 |
| **152** | Imibenconazole | + | 9.8 | 0.1 | 0.3 | 0.3 | 1 | 1 | 3 |
| **153** | Imidacloprid | + | 5.1 | 0.1 | 0.3 | 0.1 | 0.3 | 0.5 | 1.5 |
| **154** | Indoxacarb | + | 9.4 | 0.3 | 1 | 1 | 3 | 0.1 | 0.3 |
| **155** | Ioxynil | - | 8.1 | 1 | 3 | 0.3 | 1 | 1 | 3 |
| **156** | Iprovalicarb | + | 8.7 | 0.3 | 1 | 1 | 3 | 1 | 3 |
| **157** | Isocarbophos | + | 7.8 | 0.3 | 1 | 0.1 | 0.3 | 2 | 5 |
| **158** | Isoprocarb | + | 7.7 | 0.2 | 0.6 | 0.3 | 1 | 0.3 | 1 |
| **159** | Isoprothiolane | + | 8.5 | 0.3 | 1 | 0.1 | 0.3 | 1 | 3 |
| **160** | Isoproturon | + | 7.8 | 0.1 | 0.3 | 0.1 | 0.3 | 0.03 | 0.1 |
| **161** | Isoxaben | + | 8.4 | 0.02 | 0.05 | 0.03 | 0.1 | 0.1 | 0.3 |
| **162** | Isoxadifen-ethyl | + | 9.0 | 0.3 | 1 | 0.3 | 1 | 0.1 | 0.3 |
| **163** | Kresoxim-methyl | + | 9.0 | 0.3 | 1 | 1 | 3 | 2 | 5 |
| **164** | Lenacil | + | 7.7 | 0.03 | 0.1 | 0.3 | 1 | 0.3 | 1 |
| **165** | Malaoxon | + | 7.0 | 0.1 | 0.3 | 0.5 | 1.5 | 0.1 | 0.3 |
| **166** | Mandipropamid | + | 8.4 | 0.3 | 1 | 0.3 | 1 | 0.3 | 1 |
| **167** | MCPA | - | 7.8 | 0.5 | 2 | 1 | 3 | 2 | 5 |
| **168** | Mefenacet | + | 8.7 | 0.03 | 0.1 | 0.03 | 0.1 | 0.03 | 0.1 |
| **169** | Mepiquat chloride | + | 0.8 | 0.1 | 0.3 | 0.03 | 0.1 | 0.03 | 0.1 |
| **170** | Mepronil | + | 8.6 | 0.1 | 0.3 | 0.1 | 0.3 | 0.03 | 0.1 |
| **171** | Metamitron | + | 5.4 | 0.3 | 1 | 1 | 3 | 1 | 3 |
| **172** | Metazachlor | + | 7.7 | 0.3 | 1 | 0.1 | 0.3 | 0.1 | 0.3 |
| **173** | Metconazole | + | 7.2 | 0.1 | 0.3 | 0.1 | 0.3 | 0.1 | 0.3 |
| **174** | Methabenzthiazuron | + | 8.0 | 0.1 | 0.3 | 0.03 | 0.1 | 0.03 | 0.1 |
| **175** | Methamidophos | + | 2.1 | 0.3 | 1 | 1 | 3 | 1 | 3 |
| **176** | Methiocarb | + | 8.4 | 0.2 | 0.6 | 0.1 | 0.3 | 0.1 | 0.3 |
| **177** | Methiocarb-sulfone | + | 5.8 | 0.1 | 0.3 | 0.1 | 0.3 | 0.1 | 0.3 |
| **178** | Methiocarb-sulfoxide | + | 5.3 | 0.03 | 0.1 | 0.1 | 0.3 | 0.1 | 0.3 |
| **179** | Methomyl | + | 4.2 | 0.03 | 0.1 | 0.1 | 0.3 | 0.3 | 1 |
| **180** | Methoprotryne | + | 7.9 | 0.1 | 0.3 | 0.1 | 0.3 | 0.3 | 1 |
| **181** | Methoxyfenozide | + | 8.6 | 0.1 | 0.3 | 1 | 3 | 1 | 3 |
| **182** | Metobromuron | + | 7.8 | 0.1 | 0.3 | 1 | 3 | 0.3 | 1 |
| **183** | Metolachlor | + | 8.9 | 0.3 | 1 | 0.1 | 0.3 | 0.3 | 1 |
| **184** | Metolcarb | + | 6.6 | 0.2 | 0.6 | 0.3 | 1 | 0.3 | 1 |
| **185** | Metosulam | + | 7.1 | 0.1 | 0.3 | 0.3 | 1 | 0.1 | 0.3 |
| **186** | Metoxuron | + | 6.4 | 0.3 | 1 | 2 | 5 | 1 | 3 |
| **187** | Metrafenone | + | 9.3 | 0.1 | 0.3 | 0.3 | 1 | 0.3 | 1 |
| **188** | Metsulfuron-methyl | + | 7.0 | 0.3 | 1 | 0.1 | 0.3 | 0.1 | 0.3 |
| **189** | Mevinphos | + | 6.0 | 0.03 | 0.1 | 1 | 3 | 0.1 | 0.3 |
| **190** | Mexacarbate | + | 4.8 | 0.03 | 0.1 | 0.1 | 0.3 | 0.3 | 1 |
| **191** | Monocrotophos | + | 4.6 | 0.2 | 0.6 | 0.1 | 0.3 | 0.1 | 0.3 |

| | Name | Polarity | RT | Strawberry | | Leek | | Flour | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | LOD | LOQ | LOD | LOQ | LOD | LOQ |
| **192** | Monolinuron | + | 7.5 | 0.03 | 0.1 | 0.1 | 0.3 | 0.1 | 0.3 |
| **193** | Napropamide | + | 8.9 | 0.1 | 0.3 | 0.3 | 1 | 0.03 | 0.1 |
| **194** | Neburon | + | 9.1 | 0.3 | 1 | 0.1 | 0.3 | 2 | 5 |
| **195** | Nicosulfuron | + | 6.9 | 0.3 | 1 | 0.1 | 0.3 | 0.1 | 0.3 |
| **196** | Nuarimol | + | 8.3 | 0.3 | 1 | 0.3 | 1 | 1 | 3 |
| **197** | Ofurace | + | 7.0 | 0.3 | 1 | 0.3 | 1 | 0.3 | 1 |
| **198** | Omethoate | + | 3.3 | 0.1 | 0.3 | 0.1 | 0.3 | 0.1 | 0.3 |
| **199** | Oxadixyl | + | 6.5 | 0.3 | 1 | 0.3 | 1 | 0.3 | 1 |
| **200** | Oxamyl (NH4) | + | 4.0 | 0.1 | 0.3 | 0.1 | 0.3 | 0.01 | 0.05 |
| **201** | Paclobutrazol | + | 8.5 | 0.3 | 1 | 0.3 | 1 | 1 | 3 |
| **202** | Penconazole | + | 9.1 | 0.1 | 0.3 | 0.1 | 0.3 | 0.2 | 0.6 |
| **203** | Pencycuron | + | 9.4 | 0.1 | 0.3 | 0.1 | 0.3 | 0.1 | 0.3 |
| **204** | Phenmedipham | + | 8.0 | 2 | 5 | 2 | 5 | 5 | 10 |
| **205** | Phenthoate | + | 9.0 | 0.1 | 0.3 | 0.1 | 0.3 | 0.3 | 1 |
| **206** | Phoxim | + | 9.3 | 0.3 | 1 | 2 | 5 | 2 | 5 |
| **207** | Picoxystrobin | + | 9.0 | 0.03 | 0.1 | 0.3 | 1 | 0.3 | 1 |
| **208** | Piperonyl butoxide | + | 9.8 | 0.003 | 0.01 | 0.1 | 0.3 | 0.03 | 0.1 |
| **209** | Piperophos | + | 9.4 | 0.03 | 0.1 | 0.03 | 0.1 | 0.03 | 0.1 |
| **210** | Pirimicarb | + | 6.2 | 0.1 | 0.3 | 0.3 | 1 | 0.3 | 1 |
| **211** | Pirimiphos-methyl | + | 9.3 | 0.03 | 0.1 | 0.03 | 0.1 | 0.03 | 0.1 |
| **212** | Primisulfuron-methyl | + | 8.6 | 0.2 | 0.5 | 0.3 | 1 | 0.1 | 0.3 |
| **213** | Prochloraz | + | 9.3 | 0.1 | 0.3 | 0.1 | 0.3 | 0.3 | 1 |
| **214** | Profenophos | + | 9.6 | 0.03 | 0.1 | 0.01 | 0.03 | 0.03 | 0.1 |
| **215** | Promecarb | + | 8.5 | 0.1 | 0.3 | 0.3 | 1 | 0.1 | 0.3 |
| **216** | Prometon | + | 7.4 | 0.03 | 0.1 | 0.1 | 0.3 | 0.3 | 1 |
| **217** | Prometryn | + | 8.4 | 0.2 | 0.6 | 0.2 | 0.6 | 0.2 | 0.6 |
| **218** | Propamocarb | + | 3.5 | 0.03 | 0.1 | 0.03 | 0.1 | 0.03 | 0.1 |
| **219** | Propazine | + | 8.3 | 0.1 | 0.3 | 0.3 | 1 | 0.3 | 1 |
| **220** | Propetamphos | + | 8.6 | 0.3 | 1 | 1 | 3 | 0.03 | 0.1 |
| **221** | Propiconazole | + | 9.2 | 0.1 | 0.3 | 0.1 | 0.3 | 0.1 | 0.3 |
| **222** | Propoxur | + | 6.9 | 0.3 | 1 | 0.3 | 1 | 0.03 | 0.1 |
| **223** | Propyzamide | + | 8.6 | 0.3 | 1 | 0.3 | 1 | 1 | 3 |
| **224** | Prosulfocarb | + | 9.6 | 0.1 | 0.3 | 0.3 | 1 | 0.1 | 0.3 |
| **225** | Pymetrozine | + | 3.5 | 0.1 | 0.3 | 0.1 | 0.3 | 0.1 | 0.3 |
| **226** | Pyraclostrobin | + | 9.3 | 0.03 | 0.1 | 0.1 | 0.3 | 0.01 | 0.03 |
| **227** | Pyrimethanil | + | 8.3 | 0.3 | 1 | 0.3 | 1 | 0.3 | 1 |
| **228** | Pyroxsulam | + | 7.0 | 0.1 | 0.3 | 0.03 | 0.1 | 0.1 | 0.3 |
| **229** | Quinoxyfen | + | 10.1 | 0.03 | 0.1 | 0.1 | 0.3 | 0.1 | 0.3 |
| **230** | Quizalofop-ethyl | + | 9.6 | 0.03 | 0.1 | 0.1 | 0.3 | 0.1 | 0.3 |
| **231** | Quizalofop-p | + | 8.9 | 0.3 | 1 | 2 | 5 | 2 | 5 |
| **232** | Resmethrin | + | 10.3 | 1 | 3 | n | n | 10 | 20 |
| **233** | Rimsulfuron | + | 7.4 | 0.03 | 0.1 | 0.3 | 1 | 0.3 | 1 |
| **234** | Rotenone | + | 8.9 | 0.3 | 1 | 0.3 | 1 | 0.3 | 1 |
| **235** | Schradan | + | 5.8 | 0.03 | 0.1 | 0.3 | 1 | 0.03 | 0.1 |
| **236** | Sethoxydim | + | 9.7 | 2 | 5 | 50 | 100 | 2 | 5 |
| **237** | Simeconazole | + | 8.8 | 0.3 | 1 | 5 | 10 | 5 | 10 |
| **238** | Simetryn | + | 7.1 | 0.3 | 1 | 0.3 | 1 | 0.3 | 1 |
| **239** | Spinosad A | + | 9.3 | 0.3 | 1 | 0.3 | 1 | 0.3 | 1 |

| | Name | Polarity | RT | Strawberry | | Leek | | Flour | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | LOD | LOQ | LOD | LOQ | LOD | LOQ |
| **240** | Spiromesifen | + | 9.9 | 0.3 | 1 | 1 | 3 | 1 | 3 |
| **241** | Spirotetramat | + | 8.7 | 0.03 | 0.1 | 0.1 | 0.3 | 0.3 | 1 |
| **242** | Spiroxamine | + | 8.6 | 0.1 | 0.3 | 0.3 | 1 | 0.3 | 1 |
| **243** | Sulfotep | + | 9.1 | 0.1 | 0.3 | 0.3 | 1 | 0.3 | 1 |
| **244** | Sulprofos | + | 9.9 | 1 | 3 | 1 | 3 | 2 | 5 |
| **245** | Tebuconazole | + | 9.1 | 0.3 | 1 | 0.03 | 0.1 | 0.1 | 0.3 |
| **246** | Tebufenozide | + | 9.0 | 0.3 | 1 | 1 | 3 | 2 | 5 |
| **247** | Tebufenpyrad | + | 9.7 | 0.03 | 0.1 | 0.3 | 1 | 0.1 | 0.3 |
| **248** | Tebuthiuron | + | 7.1 | 0.03 | 0.1 | 0.1 | 0.3 | 0.3 | 1 |
| **249** | Teflubenzuron | + | 9.4 | 1 | 3 | 2 | 5 | 5 | 10 |
| **250** | Tepraloxydim | + | 8.7 | 1 | 3 | 5 | 10 | 5 | 10 |
| **251** | Terbumeton | + | 7.5 | 0.1 | 0.3 | 0.1 | 0.3 | 0.1 | 0.3 |
| **252** | Terbuthylazine | + | 8.4 | 0.1 | 0.3 | 0.1 | 0.3 | 0.1 | 0.3 |
| **253** | Terbutryn | + | 8.5 | 0.1 | 0.3 | 0.3 | 1 | 0.3 | 1 |
| **254** | Tetraconazole | + | 8.8 | 0.3 | 1 | 0.3 | 1 | 0.1 | 0.3 |
| **255** | Tetramethrin | + | 9.7 | 0.3 | 1 | 2 | 5 | 1 | 3 |
| **256** | Thiabendazole | + | 5.3 | 0.3 | 1 | 0.3 | 1 | 0.3 | 1 |
| **257** | Thiacloprid | + | 6.1 | 0.03 | 0.1 | 0.03 | 0.1 | 0.03 | 0.1 |
| **258** | Thiamethoxam | + | 4.5 | 0.03 | 0.1 | 0.1 | 0.3 | 0.1 | 0.3 |
| **259** | Thidiazuron | + | 7.1 | 0.3 | 1 | 0.3 | 1 | 0.3 | 1 |
| **260** | Thiobencarb | + | 9.4 | 0.1 | 0.3 | 0.1 | 0.3 | 0.1 | 0.3 |
| **261** | Thiophanate-methyl | + | 6.9 | 0.3 | 1 | 0.3 | 1 | 0.3 | 1 |
| **262** | Tolfenpyrad | + | 9.7 | 0.03 | 0.1 | 0.1 | 0.3 | 0.1 | 0.3 |
| **263** | Tralkoxydim | + | 9.9 | 0.1 | 0.3 | 0.03 | 0.1 | 0.03 | 0.1 |
| **264** | Triadimefon | + | 8.6 | 1 | 3 | 5 | 10 | 1 | 3 |
| **265** | Triadimenol | + | 8.5 | 0.3 | 1 | 0.3 | 1 | 1 | 3 |
| **266** | Triazophos | + | 8.7 | 0.01 | 0.05 | 0.1 | 0.3 | 0.1 | 0.3 |
| **267** | Trichlorfon | + | 5.2 | 1 | 3 | 1 | 3 | 1 | 3 |
| **268** | Tricyclazole | + | 6.5 | 0.03 | 0.1 | 0.1 | 0.3 | 0.1 | 0.3 |
| **269** | Tridemorph | + | 9.2 | 0.3 | 1 | 2 | 5 | 0.1 | 0.3 |
| **270** | Trietazine | + | 8.8 | 0.3 | 1 | 0.1 | 0.3 | 0.1 | 0.3 |
| **271** | Trifloxystrobin | + | 9.4 | 0.03 | 0.1 | 0.03 | 0.1 | 0.03 | 0.1 |
| **272** | Triflumizole | + | 9.6 | 0.03 | 0.1 | 0.3 | 1 | 0.3 | 1 |
| **273** | Vamidothion | + | 5.4 | 0.01 | 0.03 | 0.03 | 0.1 | 0.1 | 0.3 |
| **274** | Zoxamide | + | 9.2 | 0.1 | 0.3 | 0.3 | 1 | 0.3 | 1 |

Table 6. Comparison of the method LOQ to the MRL for selected pesticides.

| Analyte | MRL (µg/kg) | | | LOQ (µg/kg) | | |
|---|---|---|---|---|---|---|
| | Strawberry | Leek | Flour | Strawberry | Leek | Flour |
| **Acephate** | 10 | 10 | 10 | 1 | 1 | 1 |
| **Azoxystrobin** | 50000 | 10000 | 300 | 0.01 | 0.01 | 0.3 |
| **Carbaryl** | 50 | 10 | 500 | 1 | 1 | 1 |
| **Dimethomorph (sum of isomers)** | 50 | 1500 | 10 | 1 | 0.1 | 0.3 |
| **Diniconazole** | 50 | 10 | 10 | 1 | 1 | 1 |
| **Oxamyl** | 50 | 10 | 10 | 0.3 | 0.3 | 0.05 |
| **Pencycurone** | 50 | 50 | 50 | 0.3 | 0.3 | 0.3 |
| **Pyraclostrobin** | 100 | 700 | 200 | 0.1 | 0.3 | 0.03 |
| **Spinosad A** | 50 | 500 | 1000 | 1 | 1 | 1 |
| **Zoxamide** | 50 | 20 | 20 | 0.3 | 1 | 1 |

The relative standard deviation (RSD) is an important qualitative parameter that can be used instead of signal-to-noise ratio to provide a better estimate of LODs and LOQs. For pesticide residue analysis, SANCO/12571/2013 specifies repeatability criteria of 20% RSD for all compounds within the method scope. For example, as shown in Figure 3, 2.9% RSD was obtained for seven replicate injections of benzaton in the leek matrix at 10 µg/kg. The method RSDs at the MRLs were below 15%, establishing method reproducibility.

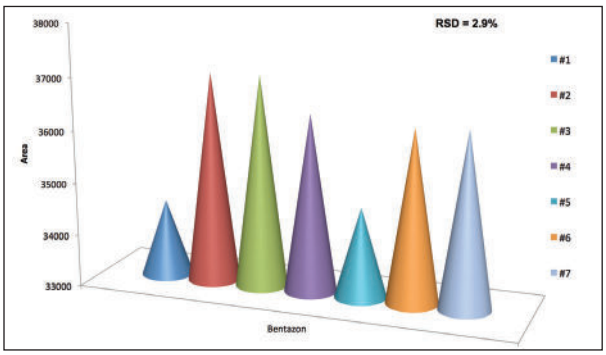


Figure 3. An RSD of 2.9% was obtained for seven replicate injections of bentazon in leek matrix at 10 µg/kg.

When using ESI, matrix effects can challenge accurate quantitation of pesticides. Though there are different strategies to compensate for these effects, the results presented in this application note are based on matrix-matched calibration. Figure 4 shows the effect of matrix on peak area. Although ion suppression is observed in the leek and wheat flour matrices, the method proved effective regardless of the matrix analyzed.

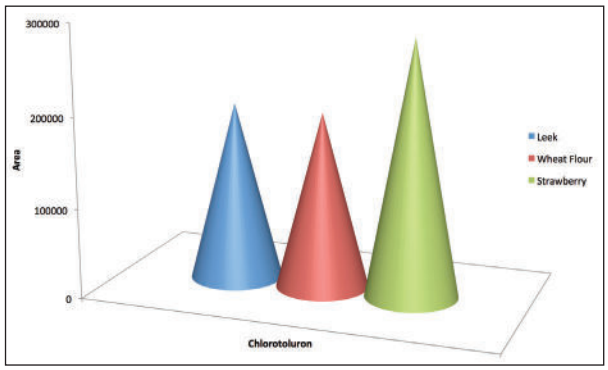


Figure 4. Matrix effects on peak area of chlorotoluron.

QuEChERS sample preparation offers a convenient and effective approach for extraction of pesticide residues in food matrices. The robust procedure has a number of compelling advantages: high recoveries, accurate results, high sample throughput, low solvent and glassware usage, reduced labor and bench space, and lower reagent costs. As shown in Figure 5, the percent recoveries achieved for selected pesticides at the 10 µg/kg level were acceptable and generally between 80 and 110% in the matrices analyzed. The pesticides selected represent results typical of all pesticides studied.

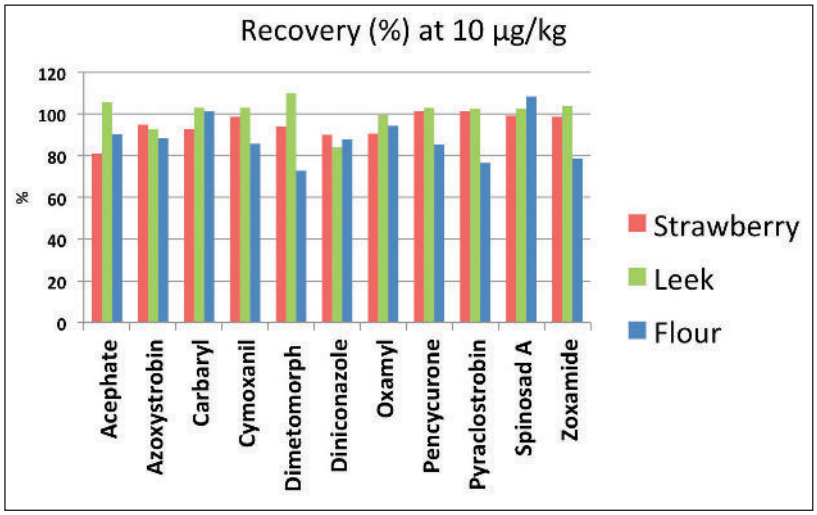


Figure 5. Recovery (%) of selected pesticides at the 10 µg/kg-level by matrix.

The quality control samples FAPAS #T19140 (lettuce puree), FAPAS #19110 (lettuce puree), FAPAS #T19142 (melon puree) and FAPAS #T0983 (wheat flour) were analyzed for their content of target pesticides to provide external quality control for method validation. As shown in Table 7, the measured target analyte values consistently fell within the acceptance range with acceptable %RSD values.

Table 7. External quality control (FAPAS) results for the relevant compounds.

| Analyte | Fapas No. | Fapas Matrix | Assigned value (µg/kg) | Acceptance range (µg/kg) | Measured value (µg/kg) | RSD (%) |
|---|---|---|---|---|---|---|
| **Carbaryl** | T19142 | Melon Puree | 89.0 | 49.9–128.2 | 91.1 | 1.1 |
| **Diniconazol** | | | 52.3 | 29.3–75.3 | 59.7 | 9.0 |
| **Zoxamide** | | | 91.7 | 51.4–132.1 | 108.4 | 3.0 |
| **Pencycuron** | T19140 | Lettuce Puree | 73.2 | 41.0–105.4 | 45.9 | 6.0 |
| **Thiamethoxam** | | | 48.8 | 27.3–70.3 | 36.3 | 9.1 |
| **Azoxystrobin** | 19110 | Lettuce Puree | 188.0 | 110–265 | 132.5 | 15.4 |
| **Dimetomorph (sum of isomers)** | | | 181.0 | 106–256 | 160.1 | 11.9 |
| **Propyzamide** | | | 197.0 | 116–277 | 195.1 | 16.5 |
| **Azoxystrobin** | T0983 | Wheat Flour | 383.0 | 241–524 | 361.2 | 1.7 |
| **Fenhexamid** | | | 110.0 | 61–158 | 125.4 | 10.4 |
| **Imazalil** | | | 161.0 | 93–229 | 157.2 | 8.2 |
| **Thiabendazole** | | | | 49.3–126.7 | 67.6 | 7.3 |

## Conclusion

Regulations of the European Union pose some significant challenges to the analytical methods quantifying pesticide residues in complex matrices. This application note described a multi-residue LC-MS/MS method that uses the TSQ Endura triple quadrupole mass spectrometer-based Pesticide Explorer Collection Standard Quantitation solution for rapid and robust quantitation of more than 250 pesticides in fruit and vegetable matrices at their respective MRLs. For convenience and fast method implementation, the complete instrument and data processing method including SRM settings is included with the Pesticide Explorer Collection start-to-finish workflow solution.

The method results were shown to comply with the stringent guidelines set forth in SANCO/12571/2003 concerning sensitivity, accuracy, and precision. In 15 minutes, all target pesticides were detected and quantified in food matrices below established MRLs. Method RSDs at the MRLs were below 15%, establishing the method's reproducibility. Percent recoveries achieved at the 100 µg/kg-level using a standard QuEChERS sample preparation protocol were in general between 80 and 110%. The QuEChERS sample extraction procedure enabled analysis of only 1 µL sample, without need for dispersive SPE sample cleanup or sample dilution, with increased robustness and throughput.

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