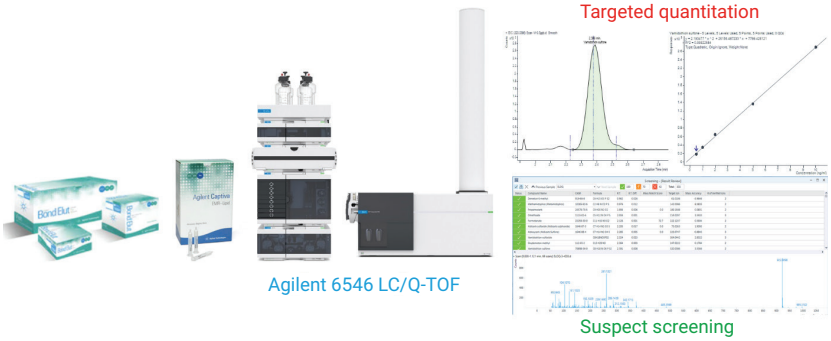
Application Note

Food and Beverage Testing

Agilent

Trusted Answers

Simultaneous Suspect Screening and Quantitation of Pesticides in a Complex Matrix Using an Agilent 6546 LC/Q-TOF



Authors

Trung Quoc Pham, Minh Trung Tran, Dat Ho Tuan, Dao Dinh Loc, Nguyen Thi Linh, Phan Xuan Dai, and Anh Tuan Le
IndoChina Center of Excellence and
Red Star Vietnam Company, Ltd.
Ho Chi Minh City, Vietnam

Boonraksa Srisawang
Agilent Technologies, Inc.
Singapore, Singapore

Abstract

In recent years, the intersection of pesticide concerns, environmental pressures, and a globalized market has made food safety a hot issue. Liquid chromatography/quadrupole time of flight mass spectrometry (LC/Q-TOF MS) is increasingly used to analyze food for authenticity and for contaminants such as pesticides because of its unparalleled precision, sensitivity, and ability to provide comprehensive insights into complex matrices.

This application note describes a methodology for screening and simultaneous quantitation of hundreds of pesticides in black pepper, a complex food matrix. The comprehensive workflow solution includes sample preparation with an Agilent Bond Elut QuEChERS extraction kit and Agilent Captiva Enhanced Matrix Removal–General Pigment Dry (EMR–GPD) cleanup material. Separation and detection were carried out with an Agilent 1290 Infinity II LC system and an Agilent 6546 LC/Q-TOF MS, and data analysis was done with the Agilent MassHunter Personal Compound Database and Library (PCDL). The workflow solution can confidently perform target quantitation and suspect screening, achieving excellent reproducibility, mass accuracy, and linearity. Combining target quantitation and suspect screening workflows reduces the complexity of data analysis and the time needed to review the results.

Introduction

Black pepper is a flowering vine in the family *Piperaceae*, grown mainly for its fruit and seeds. It is known as the "king of spices", as it is used around the world, and is of interest for its potential health benefits, such as enhancing digestion, lowering inflammation, and antioxidant effects[1-3]. Farmers have used pesticides in high concentrations and frequency to obtain high yields and protect many crops from pests, parasites, and insects. As a result, pesticide residues are found in many agricultural products, including black pepper. To control product quality and protect consumer health, regulatory organizations worldwide such as the European Union (EU) and the Codex Alimentarius Commission have set maximum residue limits (MRLs) for pesticides in black pepper.[4]

According to the EU's SANTE/11312/2021 guidelines[5], spices are among the "difficult or unique commodities" that pose a challenge for the analytical chemist when testing multiresidue pesticides. The matrix interferences are complex and diverse, including carbohydrates, fats, piperine, terpenes, and terpenoids.[6] There are two approaches to overcome this challenge: 1) high dilution to reduce the matrix effect or 2) the use of extensive sample cleanup to achieve a cleaner extract. However, if a large-dilution method is used, an analytical instrument with ultra-high sensitivity is required.

The triple quadrupole is the gold standard in quantitation, with the highest sensitivity and an excellent dynamic range, but it has some limitations. The first is its unit mass resolution; it also has a very low mass accuracy, and sample reanalysis for new compounds is not possible. In recent years, ultra-high performance liquid chromatography (UHPLC) combined with high-resolution mass spectrometry (HRMS) has widely been used in high-throughput screening of pesticide residues in food due to its high mass accuracy and resolution, and because it has less dependence on chemical standards.[7] However, for the LC/Q-TOF instrument, the sample extract solution must be cleaned up to ensure that the interference components do not affect the acquisition of mass spectrometry data on trace compounds such as pesticides.

This application note describes a detailed approach for the simultaneous screening and quantification of 302 pesticides in black pepper samples. The All Ions MS/MS scanning mode (data-independent acquisition) on the 6546 LC/Q-TOF MS was used for high-throughput screening and accurate quantitation.[8,9] For sample preparation, an extensive but simple sample cleanup approach was chosen with Agilent Captiva EMR–GPD.[10]

Experimental

Chemicals and reagents

Optima LC/MS-grade methanol, water, formic acid, ammonium formate, acetonitrile (HPLC gradient-grade), and glacial acetic acid were purchased from Sigma-Aldrich (St. Louis, Missouri, United States). Ultrapure water (18.2 MΩ-cm) was produced by a Milli-Q system from Millipore Sigma (Burlington, Massachusetts, United States).

Purine and HP-921 (hexakis [1H, 1H, 3H-tetrafluoropropoxy] phosphazene), used as reference masses for the LC/Q-TOF MS instrument during analysis, were provided by Agilent Technologies (part number G1969-85001, Santa Clara, CA, USA).

Standard solutions

Each of 302 pesticide standard (purity > 98%) or stock (1,000 mg/L) solutions were purchased from Sigma-Aldrich (St Louis, MO, USA) or LGC Standards (Teddington, Middlesex, UK). The stock solution of 10 mg/L of 10 mL standard mixture of all analytes was prepared by adding the appropriate volume from primary stock to a 10 mL volumetric flask and filling to volume with acetonitrile.

Extraction protocol

The following products and equipment were used for sample preparation:

- Agilent Bond Elut QuEChERS EN extraction kit (part number 5982-5650CH)
- Agilent Captiva EMR-GPD cartridge (part number 5610-2091)
- Agilent Vac Elut 20 manifold (part number 12234105)
- Centrifuge
- Vortexer

The protocol for sample preparation is shown in Figure 1 and described in more detail as follows:

1. Weigh 2 g ± 0.1 g of sample into a 50 mL polytetrafluoroethylene centrifuge tube.
2. Add 10 mL of ultrapure water, shake well, vortex for 1 minute, and leave for 15 minutes.
3. Add 10 mL of acidified acetonitrile (1% acetic acid) and vortex for 5 minutes for proper interaction of analytes and solvent.
4. Add QuEChERS EN extraction salts and ceramic homogenizer.
5. Put caps on the tubes and shake vigorously for 5 minutes.
6. Centrifuge for 10 minutes at 4,000 rpm.

7. Transfer 1 mL of supernatant into a 15 mL centrifuge tube and mix with 100 µL of water with 1% formic acid.
8. Pass the sample through the Captiva EMR–GPD cartridge and elute by gravity.
9. After all of the sample is passed through the cartridge, apply high pressure (~5 psi) for 1 minute to dry the EMR–GPD cartridge completely.
10. Mix the eluent well and perform LC/Q-TOF analysis.

Matrix-matched calibration

Matrix-matched calibration curves were prepared by postspiking the intermediate standard solution into a matrix blank. Preparation of matrix-matched calibration levels was identical to solvent standards preparation, except using a matrix blank instead of an acetonitrile solvent blank. In this study, five levels of concentration were used to build the matrix-matched calibration curves, including 0.5, 1, 2, 5, and 10 µg/L in matrix blank solution (2.75, 5.5, 11, 27.5, and 55 µg/kg in black pepper, respectively).

Instrument parameters

A 1290 Infinity II LC system coupled to a 6546 LC/Q-TOF MS (high resolution of 60,000 at *m/z* 2,722 [positive] and *m/z* 2,834 [negative], was used in this research. The operating parameters of the LC and HRMS are described in Table 1.

The Q-TOF MS was operated in positive ionization mode with an Agilent Dual Jet Stream electrospray ionization (ESI) source. Data-independent All Ions acquisition in positive mode was used. With this method, the Q-TOF cycled through three different MS-only scans at a rate of 4 Hz: one with 0 V CE, one with 10 V CE, and one with 20 V CE.

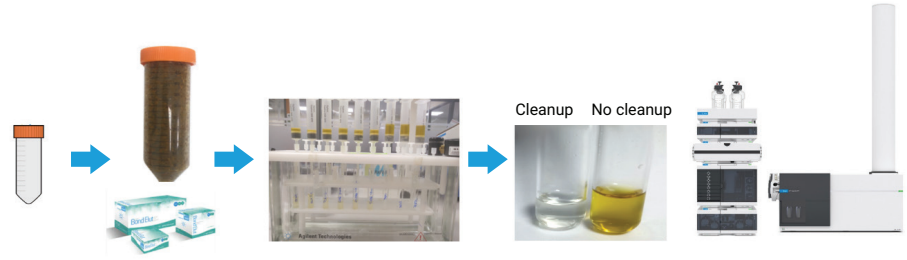


Figure 1. Black pepper sample preparation workflow using Agilent Bond Elut QuEChERS EN extraction kit followed with Agilent Captiva EMR–GPD pass-through cleanup.

Table 1. Chromatography conditions and MS parameters.

Parameter	Value
LC conditions	
HPLC	Agilent 1290 Infinity II LC with built-in degasser, autosampler with temperature control, and column temperature control compartment
Column	Agilent ZORBAX RRHD Eclipse XDB-C18, 80 Å, 2.1 × 100 mm, 1.8 µm (p/n 981758-902)
Guard Column	Agilent ZORBAX Eclipse XDB-C18, 80 Å, 2.1 mm, 1.8 µm UHPLC guard (p/n 821725-903)
Column Temperature	35 °C
Mobile Phase	A) H_2O containing 5 mM ammonium formate and 0.1% formic acid (v/v) B) MeOH containing 5 mM ammonium formate and 0.1% formic acid (v/v)
Flow Rate	0.3 mL/min
Injection Volume	5 µL
Gradient Elution Profile	Time (min) %A %B 0.0 98 2 0.5 98 2 1.0 50 50 4.0 35 65 10.0 5 95 12.0 0 100 16.0 0 100 18.0 98 2 Post time: 5 min
ESI Q-TOF MS conditions	
MS	Agilent 6546 LC/Q-TOF MS with Agilent Dual Jet Stream ESI source
Polarity	Positive ionization
Drying Gas Temperature	325 °C
Drying Gas Flow Rate	8 L/min
Nebulizer Gas Pressure	20 psi
Sheath Gas Temperature	375 °C
Sheath Gas Flow Rate	12 L/min
Capillary Voltage	4,000 V
MS Scan Range	*m/z* 50 to 1,000
Reference Ions	*m/z* 121.0509/922.0098
Mode	All Ions MS/MS at three levels collision energy (0, 10, 20 V); 4 spectra/min

Data analysis and method validation

Data processing: In this study, the quantitative method, PCDL database, and workflow for suspect screening were built based on the workflow described in application note 5994-0738EN.[11] Furthermore, to extract maximum information from profile data and more quickly process data, the acquisition LC/Q-TOF data were converted to SureMass format using Agilent MassHunter Quantitative Analysis software version 10.0.

Qualitative screening method evaluation: Based on the SANTE/11312/2021 guideline, the screening detection limit (SDL) of the method was determined by prespiked samples with a series of concentrations for each pesticide, with 20 replicates at concentration levels of 2, 5, and 10 µg/kg. After that, sample preparation was performed as described in the extraction protocol section. The criteria for determining SDL, as described in the SANTE guideline, is the lowest level at which an analyte has been detected (the shift of retention time of the analyzed compound ± 0.1 minute and accurate mass for the precursor ion and at least one fragment ion with mass accuracy within ± 5 ppm. For pesticides with *m/z* less than 200, up to 1 milliDaltons mass deviation is allowed) in at least 95% of the samples.

Quantitative method evaluation: Method validation was performed for black pepper according to the SANTE/11312/2021 recommendations for calibration curve linearity, method recovery, and precision.

Results and discussion

Chromatography and data quality

The total ion chromatography (TIC) of the black pepper samples and TIC for each level of collision energy is shown in Figure 2. There is a similar TIC among the three levels of energy. In terms of target compounds, the 6546 LC/Q-TOF MS shows high mass accuracy across the gradient of the mobile phase. Figure 2 shows the sample chromatogram plotted against the mass accuracy of the analytes at their given retention time. The green dots are the mass accuracy of 10 µg/L in a matrix solution. Nearly all the analytes are within ± 5 ppm mass error (red line). These results give confidence in the instrument performance for analyzing pesticides in complex matrices such as black pepper.

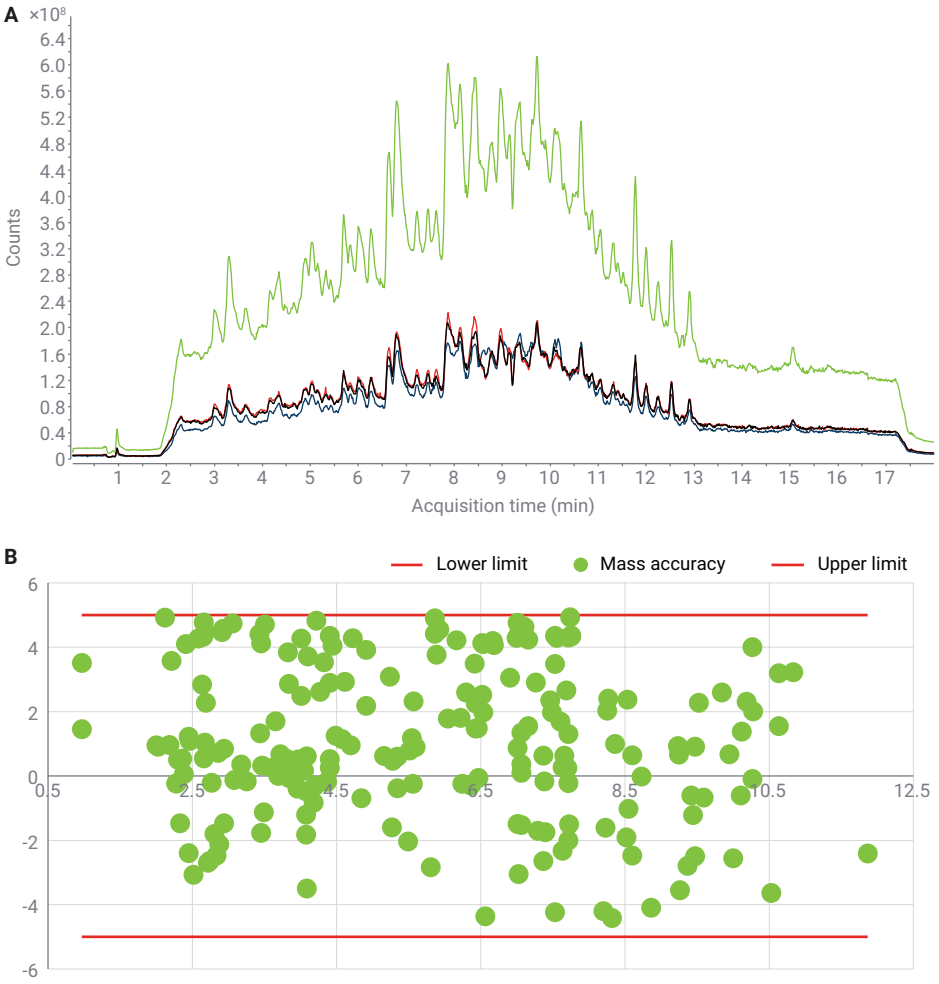


Figure 2. Total ion chromatogram (TIC) of a black pepper sample (A) and the mass accuracy of priority target ions in black pepper matrix at 10 ppb plotted against their retention times (B).

Determination of the screening detection level

According to the SANTE/12682/2019 guidelines, the spiked black pepper samples were prepared and analyzed at a series of concentrations for each pesticide, with 20 replicates at each concentration level (2, 5, and 10 µg/kg).

As shown in Figure 3, most of the 302 pesticide compounds have an SDL value that is ≤ 5 µg/kg. Specifically, 153 pesticides have an SDL value of 2 µg/kg and 117 pesticides have an SDL value of 5 µg/kg. Only 32 compounds have an SDL value of 10 µg/kg.

Quantitative pesticides in black pepper

To validate the developed analytical method for 302 pesticides in black pepper, experiments to determine limit of quantification (LOQ), linearity of calibration, accuracy (%recovery), and precision (relative standard deviation or %RSD) were performed.

Calibration curve

Because the 6546 LC/Q-TOF instrument has dual channels (high and low gains) in the acquisition board, each spectrum is combined with the high- and low-gain channels. This allows for a higher effective saturation rate, and thus a greater linear dynamic range. The HiGain channel has 12x the abundance of the LoGain channel if scaled individually. Thus, the curve fit with the quadratic model was used to construct the calibration curve. With a dynamic range from 0.5 to 10 µg/L in matrix solution, 100% of target compounds (302 pesticides) met the calibration curve requirement of $R^2 \geq 0.99$ (Figures 4 and 5). Additionally, the accuracy of the regression equation was evaluated based on the deviation between theoretical and calculated concentrations at all calibration levels. The results show that the quantitative accuracy of each calibrator was less than or equal to ± 20%. These accuracy levels indicate that the LC/Q-TOF 6546 instrument meets the requirements of SANTE/11312/2021 for the analysis of pesticides.

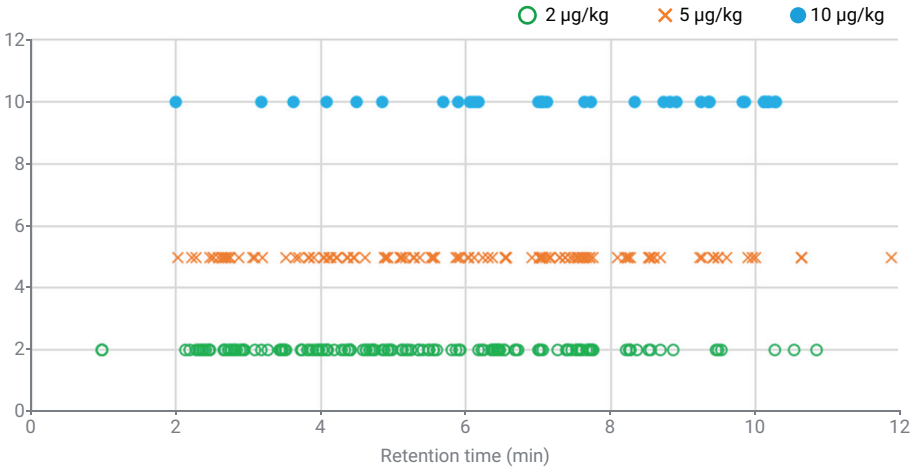


Figure 3. Screening detection limit (SDL) value of each pesticide.

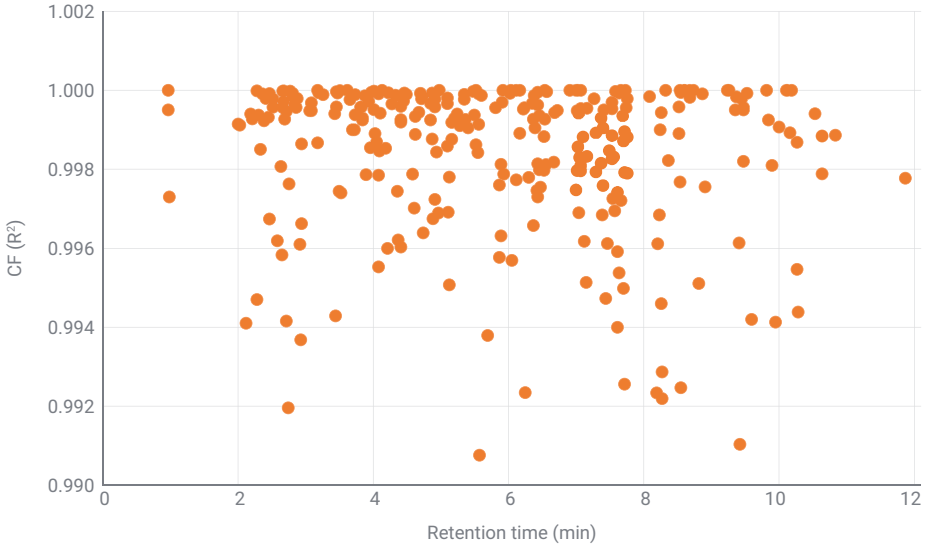
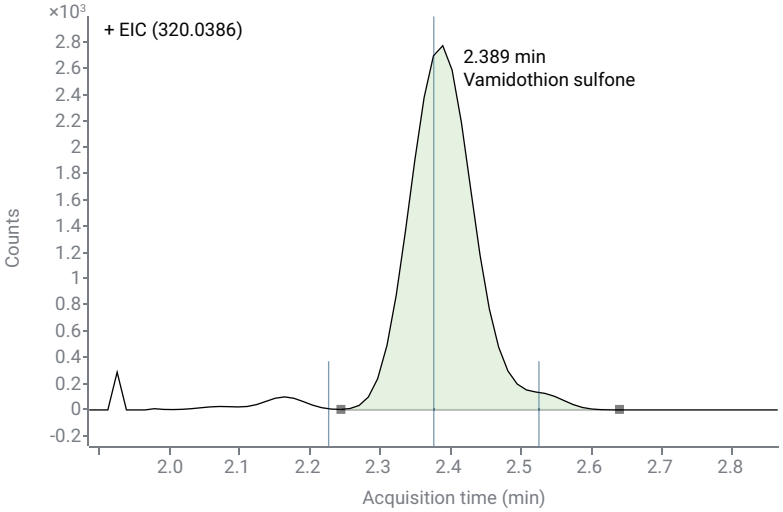
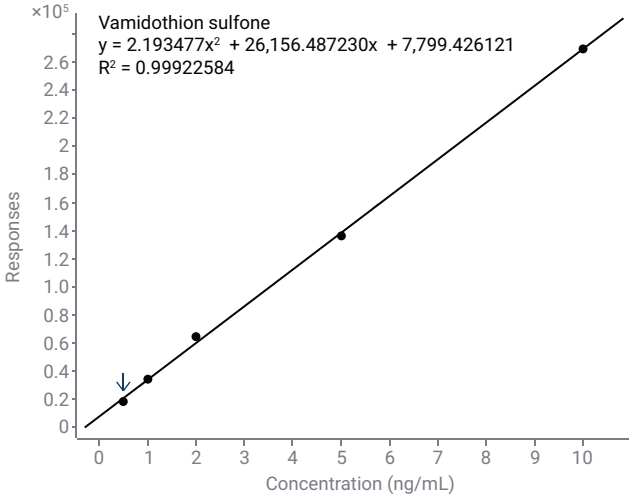
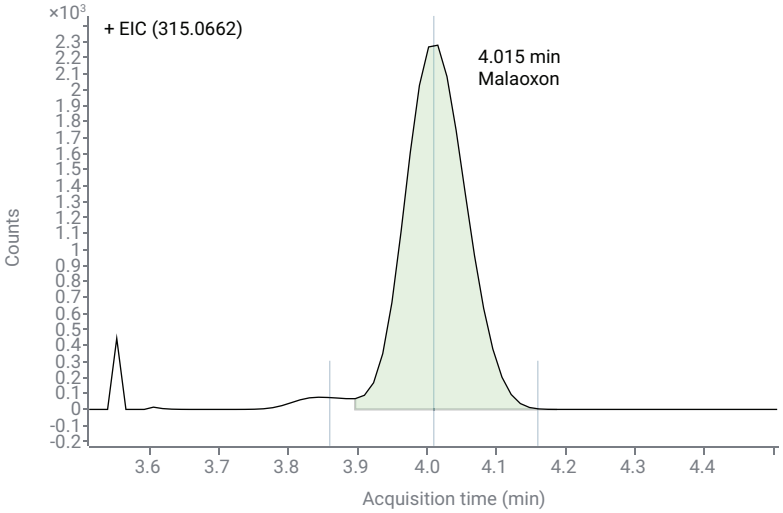
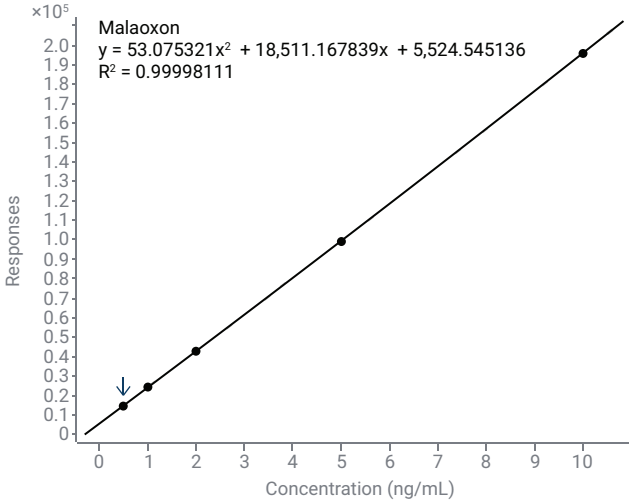


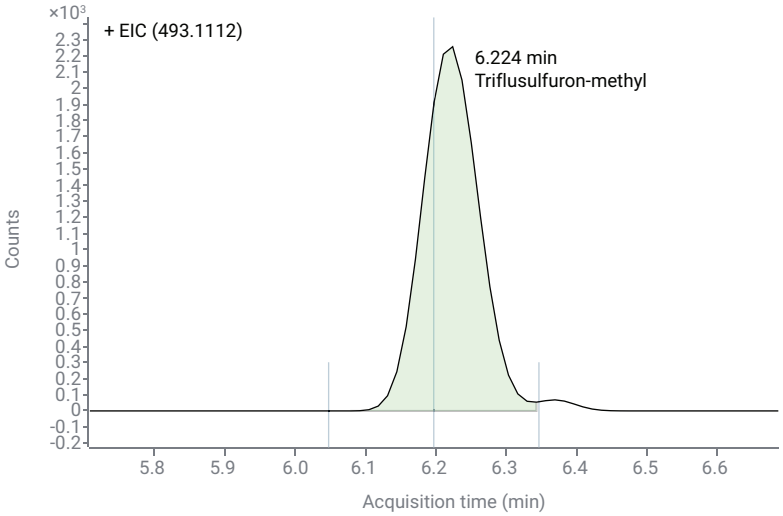
Figure 4. Method performance evaluation in terms of the calibration curve.

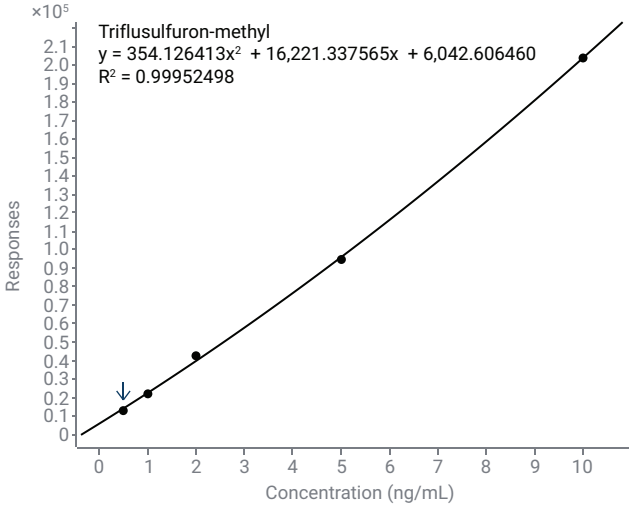

×10³
+ EIC (320.0386)
2.389 min
Vamidothion sulfone
Counts
Acquisition time (min)


×10⁵
Vamidothion sulfone
y = 2.193477x² + 26,156.487230x + 7,799.426121
R² = 0.99922584
Responses
Concentration (ng/mL)


×10³
+ EIC (315.0662)
4.015 min
Malaoxon
Counts
Acquisition time (min)


×10⁵
Malaoxon
y = 53.075321x² + 18,511.167839x + 5,524.545136
R² = 0.99998111
Responses
Concentration (ng/mL)


×10³
+ EIC (493.1112)
6.224 min
Triflusulfuron-methyl
Counts
Acquisition time (min)


×10⁵
Triflusulfuron-methyl
y = 354.126413x² + 16,221.337565x + 6,042.606460
R² = 0.99952498
Responses
Concentration (ng/mL)

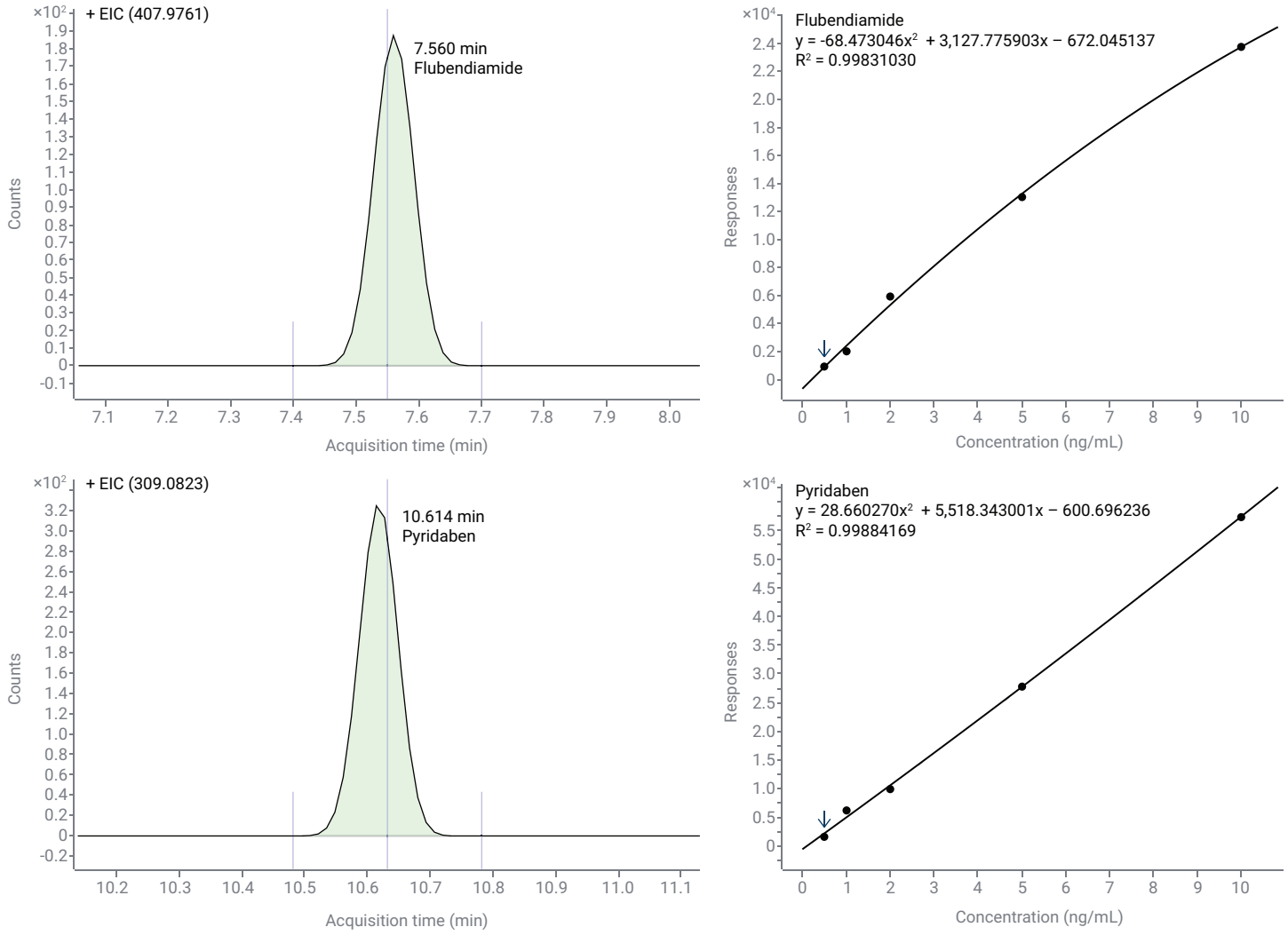


Figure 5. The extracted ion chromatograms and matrix-matched calibration curves for early, middle, and last elution pesticide compounds.

Determination of limit of quantification

According to SANTE/11312/2021, LOQ is defined as the lowest concentration spike for the sample in which the recovery is from 40 to 120%, and repeatability (RSD) and reproducibility (RSD) are both less than 20%. The method is meaningful when the LOQ is equal to or less than the MRL of the compounds. In compliance with EU regulations, the MRL for the majority of pesticides in pepper matrices is set at 50 µg/kg. In this study, the LOQ value of each pesticide was determined by prespiking at 10, 20, and 50 µg/kg (15 samples over two days).

Figure 6 and Table 2 (Appendix) give information about the number of pesticides whose recovery efficiency and RSD value met the requirements of SANTE at three levels of prespiked concentrations. Overall, there were 229 pesticides with LOQ values of 10 µg/kg, representing 76% of the target list (302 compounds), 25 compounds with LOQ of 20 µg/kg, and 22 compounds with LOQ of 50 µg/kg because of low sensitivity, matrix interferences, and positive occurrence in black pepper matrix.

Method accuracy and precision

In the process of analyzing pesticide residues in food matrices, target recovery is the key indicator for the performance and accuracy of the sample preparation method. According to EU regulations, the lowest MRL established for pesticides in pepper matrix is 50 µg/kg. Therefore, prespiked samples at three levels of concentration: 10 µg/kg (LOQ), 20 µg/kg (2 LOQ), and 50 µg/kg (5 LOQ) were used

to evaluate target recovery. According to the method performance acceptability criteria in SANTE guidelines, the average recovery must not be lower than 30% or above 140% if they are consistent (RSD ≤ 20%). The acceptable recovery range was even more stringently defined from 40 to 120% with RSD ≤ 20%. The mean recovery based on nine technical replicates (n = 9) of prespiked samples for each concentration is shown in Figures 6 and 7.

Figure 6 illustrates the number of pesticide compounds with mean recoveries that meet the requirement SANTE/11312/2021 at three concentration levels. At prespiked 10 µg/L (LOQ), there are 204 compounds that have an average recovery ranging from 70 to 120%, accounting for 67.5% of the total target list. Furthermore, if the acceptable recovery range was defined from 40 to 120% with RSD ≤ 20%, 229 pesticides met this criterion, which accounted for 75.8% of the total compounds.

A dramatic increase in the number of pesticides with an average recovery ranging from 40 to 120% was witnessed at prespiked 2 LOQ and 5 LOQ samples. At the 2 LOQ spike, 254 compounds had a mean recovery of 40 to 120%. Meanwhile, at the 5 LOQ spike, there were 91.3% of pesticides with 40 to 120% recovery with RSD value ≤ 20%.

The recovery results confirmed that the developed sample preparation method provided acceptable analyte recoveries for most targeted pesticides in black pepper. The unacceptable recoveries of the failed pesticides were mainly related to the interaction between cleanup material, target compounds, and matrix interferences in the black pepper.

Repeatability (RSD)

Method precision was evaluated using the intrabatch recovery repeatability with nine technical replicates of prespiked LOQ, 2 LOQ, and 5 LOQ. As shown in Figure 7, the %RSD of 229 pesticides mostly fluctuates between 10 and 17% at prespiked LOQ. Furthermore, 84 and 91% of target compounds in black pepper provided RSD_r ≤ 20% at prespiked 2 LOQ and 5 LOQ, respectively, which demonstrates the consistent performance of the sample preparation method.

Reproducibility (RSD)

The reproducibility of the analytical method for pesticides that were satisfactory in terms of recovery

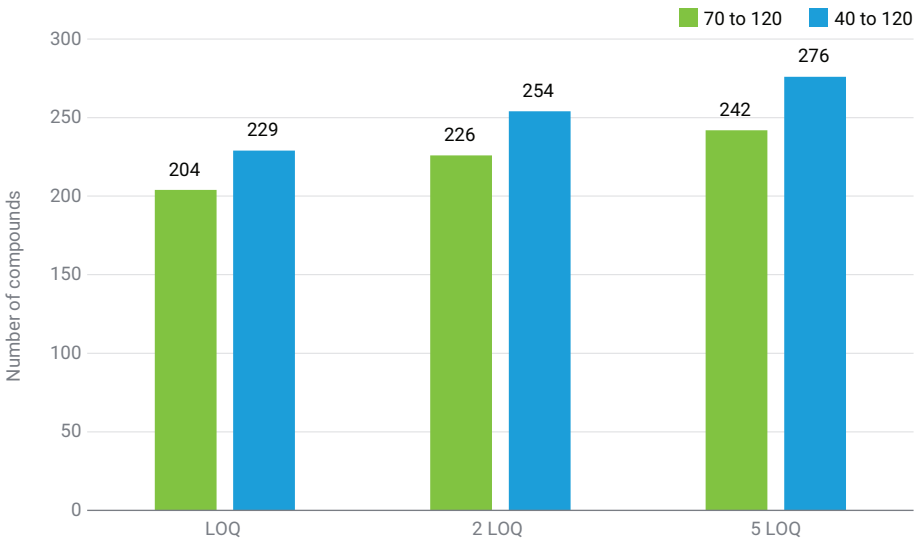


Figure 6. Prespiked quality control (QC) recovery distribution of all 302 targets in black pepper.

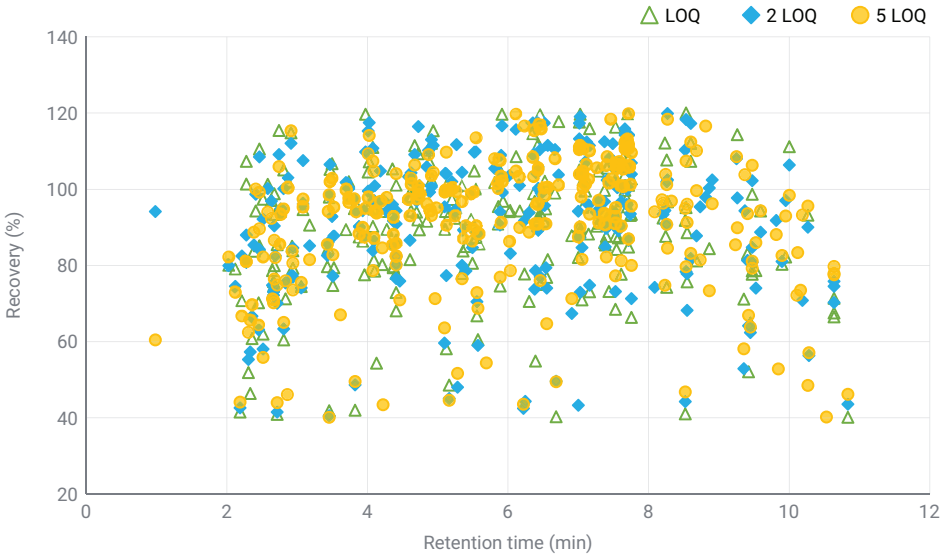


Figure 7. Recovery distribution of pesticide compounds at three levels, prespiked.

performance and repeatability according to the SANTE guidelines was also evaluated at three concentration levels. The $\%RSD_{wr}$ was calculated based on the recoveries of six replicates of prespiked QCs across two batches, prepared by two lab scientists using different lots of sample matrices on different days. Overall, the pesticide compounds met the criteria for SANTE recovery, which had good reproducibility at all three concentrations used for evaluation. The average RSD_{wr} values fluctuated between 12 and 15% (Figure 8). The results indicate that this method is suitable for analyzing a large group of pesticides in a complex matrix and provides consistent quantitative results for day-to-day routine analyses.

LC screener analysis

MassHunter Quantitative Analysis software 10.0 enables one method to analyze a batch of samples for both quantitation of priority targets and suspect screening. The LC Screener tool displays compounds identified in the analytical sample that satisfy the specified conditions (green), need to be reviewed (yellow), and are not identified (red). For example, the analysis results for a pepper sample from Phu Quoc are shown in Figure 9, and the outlier parameters were set up according to SANTE/11312/2021. Pirimicarb was selected here, so its results were shown in the analysis panels. The middle left panel shows an average full spectrum at the time when pirimicarb elutes. The middle right panel shows the theoretical isotopes for pirimicarb (red) from the reference pattern library overlaid with the experimental data (blue).

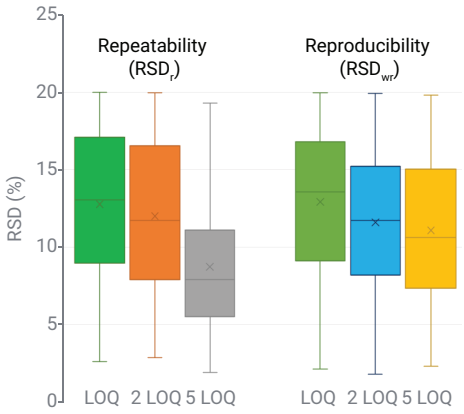


Figure 8. Method performance evaluation in terms of repeatability (n = 9), and reproducibility (n = 6).

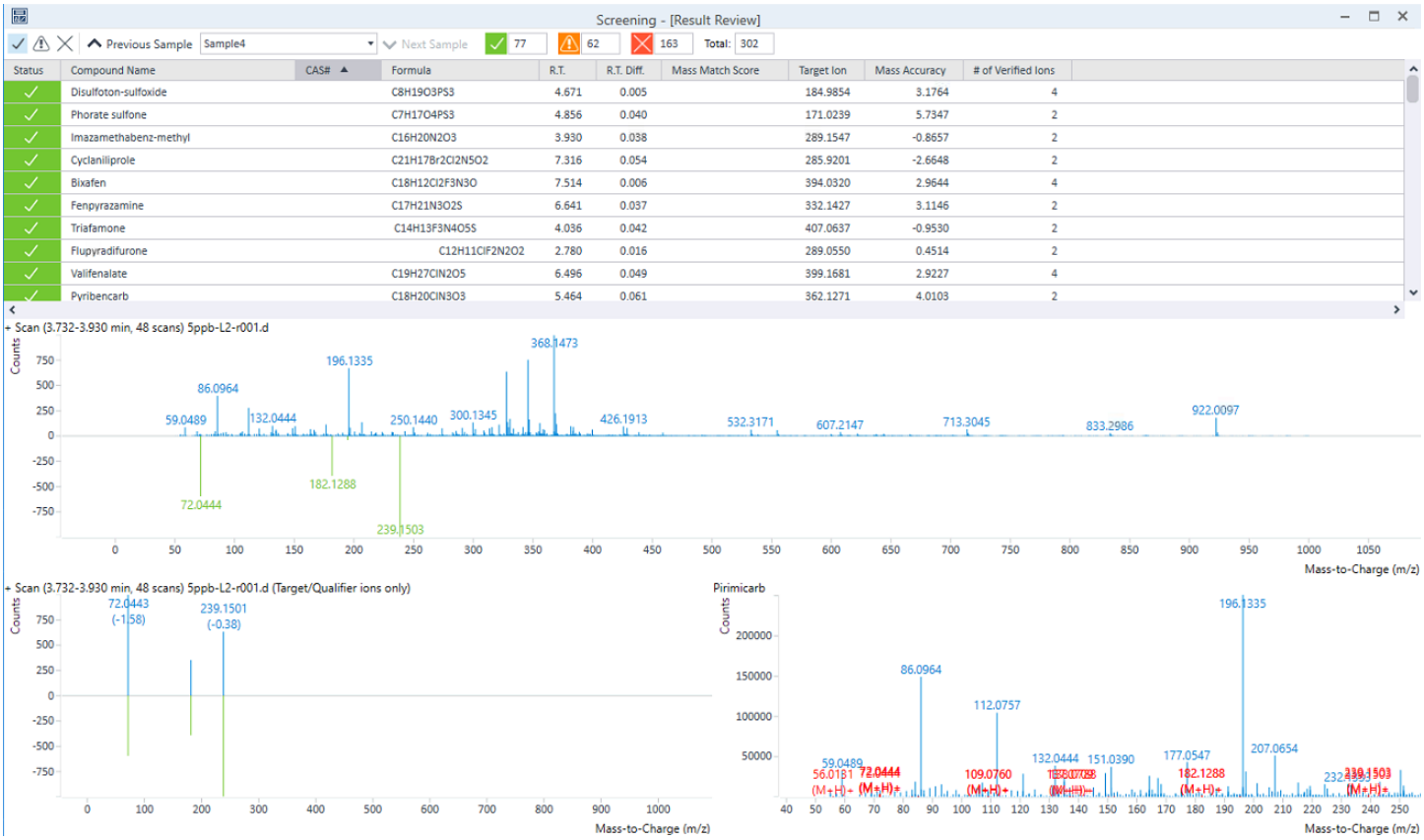


Figure 9. The LC Screener tool window in Agilent MassHunter Quantitative Analysis software shows a list of analytes that are positively identified or need review in the selected sample.

When quickly screening for suspect compounds that do not have confirmed retention times or fragments, the isotopic pattern match in the lower right panel of Figure 9 can be helpful in determining the confidence of identification.

Conclusion

A comprehensive solution in terms of food safety control has been developed that includes authenticity testing and screening for toxic compounds in complex food matrices using an Agilent 6546 LC/Q-TOF high-resolution mass spectrometer. This application note describes a method for the simultaneous screening and quantification of 302 pesticide residues in black pepper using a 6546 LC-Q/TOF. The method developed for the black pepper matrix has a sensitive and reliable screening capacity, with most screening detection limits at or below 5 µg/kg. The method also has an accurate and robust quantitation capacity, with most limits of quantification at or below 10 µg/kg and relative standard deviation below 20%. This method can also be expanded to include many other food matrices of plant origin for qualitative and quantitative pesticide residue screening.

References

1. Platel, K.; Srinivasan, K. Influence of Dietary Spices and Their Active Principles on Pancreatic Digestive Enzymes in Albino Rats. *Nahrung* **2000**, *44*, 42–46.
2. Srinivasan K. Antioxidant Potential of Spices and Their Active Constituents. *Crit. Rev. Food Sci. Nutr.* **2014**, *54(3)*, 352–72.
3. Kunnumakkara, A. B.; Sailo, B. L.; Banik, K.; Harsha, C.; Prasad, S.; Gupta, S. C.; Bharti A. C.; Aggarwal, B. B. Chronic Diseases, Inflammation, and Spices: How Are They Linked? *J. Transl. Med*. Jan 25 **2018**, *16(1)*, 14.
4. European Commission EU Pesticide Residues database, **2022**. https://ec.europa.eu/food/plant/pesticides/eu-pesticides-database/start/screen/mrls
5. Guidance SANTE 11312/2021 – Analytical Quality Control and Method Validation Procedures for Pesticide Residues Analysis in Food and Feed. **2021**.
6. Lee, J.-G.; *et al.* Chemical Composition and Antioxidant Capacity of Black Pepper Pericarp. *Applied Biological Chemistry* **2020**, *63(1)*.
7. Agilent Technologies, Improving Food Safety Analysis with LC/Q-TOF. *LC & GC* **2020**.
8. All Ions MS/MS Workflow Guide. *Agilent Technologies*, publication number 5991-1994EN, **2013**.
9. Xie, Y.; Chen, H.; Fan, C.; Ge, L.; Huo, S.; Lu, M. Simultaneous Screening and Quantification of Pesticide Residues in Red Cabbage. *Agilent Technologies application note*, publication number 5994-3123EN, **2021**.
10. Aimei Zou, L. Z., Wong, A.; Garnica, R.; Sian Gan, C. Analysis of 510 Pesticides in Black Pepper, Using Captiva EMR Sequential Pass-Through Cleanup and LC/MS/MS. *Agilent Technologies application note*, publication number 5994-4768EN, **2022**.
11. Yannell, K. E.; Chen, K. Enhanced Food Safety Testing: A Pesticide Screening Methodology Using the Agilent 6546 LC/Q-TOF and MassHunter Quantitative Analysis Software 10.0 LC/Q-TOF Screener Tool. *Agilent Technologies application note*, publication number 5994-0738EN, **2019**.

Appendix

Table 2. The list of target pesticide compounds and their recoveries at prespiked 10, 20, and 50 µg/kg (n = 9) in black pepper matrix.

Compound	Recovery (%)		
	LOQ (10 µg/kg)	2 LOQ (20 µg/kg)	5 LOQ (50 µg/kg)
2,6-Dichlorobenzamide	101.25 ± 9.92	100.19 ± 12.17	93.98 ± 9.14
Acephate	41.55 ± 6.27	42.64 ± 5.77	44.12 ± 3.84
Acetamiprid	71.23 ± 6.65	75.41 ± 6.16	75.98 ± 8.29
Aldicarb sulfoxide (Aldicarb Sulphoxide)	70.72 ± 7.51	82.5 ± 12.27	66.76 ± 8.3
Aldoxycarb (Aldicarb Sulfone)	101.39 ± 8.31	80.79 ± 15.73	80.96 ± 15.76
Ametoctradin	41.05 ± 5.47	44.24 ± 5.74	46.78 ± 2.84
Amitraz	26.58 ± 6.59	34.61 ± 3.52	40.21 ± 1.61
Atrazine-2-hydroxy (Hydroxy Atrazine)	18.6 ± 7.41	22.29 ± 2.78	21.97 ± 2.28
Atrazine-desethyl (Desethylatrazine)	90.58 ± 13.13	85.18 ± 10.48	81.5 ± 5.66
Atrazine-desisopropyl	93.42 ± 17.81	82.52 ± 9.15	82.08 ± 7.76
Azaconazole	77.86 ± 8.82	80.04 ± 7.96	76.52 ± 4.57
Azinphos-ethyl (Guthion Ethyl)	D	D	97.99 ± 15.86
Azoxystrobin	101.79 ± 7.37	105.5 ± 4.99	108 ± 3.06
Beflubutamid	108.23 ± 16.77	100.27 ± 11.74	100.71 ± 7.59
Benodanil	112.08 ± 8.86	100.63 ± 7.21	93.15 ± 7.64
Bensulide	94.08 ± 11.83	111.93 ± 19.77	102.78 ± 10.78
Benzobicyclon	75.02 ± 6.39	77.02 ± 3.89	76.04 ± 2.47
Bifenazate-diazene	D	D	110.13 ± 6.97
Bitertanol	96.71 ± 16.13	102.47 ± 10.22	84.56 ± 16.34
Bixafen	87.61 ± 10.05	105.91 ± 18.93	91.1 ± 12.89
Boscalid (Nicobifen)	70.54 ± 13.38	93.84 ± 12.35	88.72 ± 7.15
Bromacil	78.48 ± 14.78	100.86 ± 13.68	94.21 ± 8.07
Bromuconazole(II)	D	D	105.26 ± 17.16
Bupirimate	105.08 ± 8.94	113.26 ± 15.61	110.33 ± 7.38
Buprofezin	114.35 ± 19.53	97.71 ± 16.15	89.88 ± 12.84
Butamifos	87.65 ± 7.3	90.93 ± 17.68	103.88 ± 11.98
Butoxycarboxim	107.33 ± 13.97	87.98 ± 15.94	80.98 ± 15.14
Butralin (Sutralin)	D	70.82 ± 11.45	93.26 ± 18.64
Buturon	97.28 ± 9.1	101.99 ± 6.45	100.2 ± 5.89
Cadusafos	95.16 ± 13.68	78.37 ± 15.48	79.68 ± 11.33
Carbaryl	105.36 ± 11.72	96.01 ± 9.87	85.42 ± 8.24
Carbendazim (Azole)	61.95 ± 8.8	58.06 ± 5.72	55.87 ± 5.76
Carbetamide	101.44 ± 13.23	100.14 ± 8.63	97.68 ± 3.29
Carbophenothion	D	D	83.38 ± 8.03
Carboxin	101.01 ± 15.29	90.92 ± 6.29	85.87 ± 2.54
Carfentrazone-ethyl	99.71 ± 18.22	115.82 ± 21.34	112.35 ± 7.16
Chlorantraniliprole	66.74 ± 11.86	70.45 ± 11.96	72.89 ± 3.61
Chlordimeform	97.34 ± 19.44	92.17 ± 15.5	82.24 ± 15.85
Chlorfluazuron (Chlorfluazuron)	31.8 ± 31.39	56.38 ± 8.08	57.08 ± 10.58
Chloridazon (PAC)	79.28 ± 4.51	76.93 ± 7.5	73.58 ± 7.83
Chlorimuron-ethyl	11.48 ± 9.07	17.78 ± 7.07	20.17 ± 5.13
Chlorthiamid	D	D	43.66 ± 17.83

Compound	Recovery (%)		
	LOQ (10 µg/kg)	2 LOQ (20 µg/kg)	5 LOQ (50 µg/kg)
Chlorthiophos	D	D	72.22 ± 13.07
Chlortoluron (Chlorotoluron)	91.62 ± 13.12	105.89 ± 5.8	96.14 ± 4.41
Clodinafop-propargyl	77.7 ± 12.12	105.92 ± 15.9	100.85 ± 6.21
Cloquintocet-mexyl	64.94 ± 3.5	62.37 ± 2.69	63.79 ± 3.03
Clothianidin	85.46 ± 17.09	70.1 ± 13.08	74.87 ± 10.31
Cyanazine (Fortrol)	89.39 ± 17.08	101.76 ± 15.64	99.77 ± 9.67
Cyantraniliprole	43.06 ± 18.09	75.89 ± 14.44	70.98 ± 7.84
Cyazofamid	115.88 ± 10.68	111.47 ± 13.99	109.99 ± 4.65
Cyclaniliprole	99.68 ± 18.28	122.49 ± 18.1	93.68 ± 7.21
Cyenopyrafen	D	D	73.52 ± 13.59
Cyflufenamid	D	108.56 ± 46.57	97.26 ± 13.96
Cyflumetofen	D	102.43 ± 18.93	96.19 ± 7.62
Cymoxanil (Curzate)	D	107.52 ± 20.13	95.34 ± 16.25
Cyprazine	89.46 ± 14.62	95.37 ± 16.51	99.2 ± 6.75
Cyprodinil	68.46 ± 11.81	73.16 ± 8.48	77.25 ± 4.28
Cyromazine	35.76 ± 15.77	17.83 ± 8.54	13.03 ± 2.35
DEET/Diethyltoluamide	94.13 ± 15.39	104.14 ± 10.15	99.81 ± 2.61
Demeton-S-methyl	8.44 ± 4.36	15.03 ± 2	18.94 ± 2.14
Demeton-S-methylsulfone	94.27 ± 5.51	98.66 ± 7.17	100.06 ± 8.31
Desmedipham	84.99 ± 8.73	78.56 ± 9.48	90.46 ± 10.16
Desmethyl Norflurazon	89.32 ± 13.93	101.02 ± 7.33	98.56 ± 5.83
Desmetryne	79.39 ± 4.05	85.32 ± 3.41	88.15 ± 4.49
Desthio-prothioconazole(I)	91.09 ± 12.86	94.72 ± 12.53	91.37 ± 10.69
Diclobutrazol (Diclobutrazol)	95.36 ± 15.75	100.01 ± 10.76	106.15 ± 7.86
Diclosulam	22.36 ± 9.83	22.73 ± 9.46	23.15 ± 5.87
Diethofencarb	104.4 ± 11.66	108.92 ± 6.93	91.37 ± 7.89
Difenoconazole	75.67 ± 11.52	68.23 ± 10.73	96.09 ± 5.3
Diflubenzuron	73.15 ± 13.33	91.01 ± 15.28	101.66 ± 16.32
Diflufenican	D	95.37 ± 15.25	81.44 ± 6.62
Dimefox	98.85 ± 14.82	73.61 ± 14.27	76.45 ± 13.61
Dimefuron	100.03 ± 14.16	109.81 ± 10.16	113.53 ± 8.59
Dimepiperate	D	97.93 ± 18.51	116.6 ± 9.31
Dimethachlor	92.13 ± 12.63	99.02 ± 8.03	98.72 ± 4.43
Dimethenamide - P	94.28 ± 15.99	100.95 ± 10.37	103.2 ± 5.75
Dimethoate	114.82 ± 11.91	122.08 ± 13.48	125.37 ± 16.63
Dimethomorph(E)	94.48 ± 18.14	83.17 ± 14.8	78.61 ± 13.11
Dimethomorph(Z)	91.95 ± 14.05	89.25 ± 7.36	91.02 ± 6.42
Dimetilan	101.91 ± 6.64	102.93 ± 6.44	100.3 ± 6.49
Dimoxystrobin	106.5 ± 7.78	106.55 ± 9.17	107.24 ± 5.17
Dioxacarb	107.18 ± 8.13	117.5 ± 20.84	114.1 ± 7.45
Disulfoton-sulfone	110.26 ± 20.69	110.22 ± 7.87	109.09 ± 3.56
Disulfoton-sulfoxide	91.98 ± 10.39	97.57 ± 9.94	100.61 ± 5.61
Ditalimfos (Plondrel)	D	D	40.66 ± 10.38
Diuron	83.76 ± 16.2	89.33 ± 9.01	87.15 ± 6.17
Dodemorph	D	D	D
EDPP/Edifenphos	93.4 ± 16	106.88 ± 8.45	95.67 ± 5.92

Compound	Recovery (%)		
	LOQ (10 µg/kg)	2 LOQ (20 µg/kg)	5 LOQ (50 µg/kg)
Epoxiconazole (BAS 480F)	88.22 ± 14.58	95.91 ± 18.17	102.56 ± 10.17
Etaconazole	73.08 ± 5.41	74.79 ± 12.48	93.47 ± 7.57
Ethaboxam	94.39 ± 6.95	102.28 ± 4.49	101.24 ± 2.56
Ethametsulfuron-methyl	D	D	D
Ethidimuron (Sulfadiazole)	79.97 ± 12.35	90.39 ± 10.63	86.61 ± 5.76
Ethiofencarb	81.53 ± 15.86	86.58 ± 13.58	93.05 ± 10.05
Ethiofencarb Sulfoxide	75.74 ± 7.09	73.06 ± 7.94	70.96 ± 5.96
Ethion	94.27 ± 8.71	88.7 ± 15.37	94.39 ± 7.13
Ethiprole	108.59 ± 20.8	115.64 ± 8.86	104.81 ± 7.8
Ethirimol	41.85 ± 1.43	40.58 ± 3.63	40.15 ± 2.58
Ethoxysulfuron	22.06 ± 9.56	26.47 ± 8.61	27.63 ± 6.82
Fenamidone	D	D	119.65 ± 11.04
Fenamiphos	85.37 ± 12.02	84.9 ± 12.56	90.81 ± 8.67
Fenamiphos-sulfone	94.74 ± 5.71	97.13 ± 5.14	104.74 ± 2.03
Fenamiphos-sulfoxide	86.37 ± 3.59	94.35 ± 6.01	90.03 ± 2.42
Fenarimol	70.95 ± 7.25	84.66 ± 14.62	81.57 ± 6.21
Fenazaquin	40.13 ± 3.38	43.61 ± 2.47	46.19 ± 1.9
Fenbendazole	40.26 ± 5.84	49.63 ± 3.2	49.51 ± 4.09
Fenbuconazole	104.7 ± 19.61	97.17 ± 17.43	97.09 ± 14.32
Fenchlorphos-oxon	D	D	100.31 ± 10.36
Fenoxycarb	102.22 ± 8.36	91.08 ± 9.56	92.85 ± 7.66
Fenpropidin	D	D	D
Fenpyrazamine	98.96 ± 16.55	110.43 ± 11.81	108 ± 6.37
Fenpyroximate	93.19 ± 9.5	90.02 ± 7.01	95.61 ± 6.64
Fenthion-sulfone	86.77 ± 16.34	94.38 ± 12.95	104.08 ± 9.02
Fenthion-sulfoxide (Mesulfenfos)	94.15 ± 11.23	104.81 ± 7.6	96.85 ± 2.93
Flonicamid	110.54 ± 21.07	108.48 ± 19.93	99.26 ± 7.9
Florasulam	D	D	D
Fluacrypyrim	120.99 ± 14.37	131.28 ± 11.84	107.38 ± 6.37
Flubendazole	48.58 ± 8.07	45.09 ± 5.02	44.64 ± 3.64
Flubendiamide	106.52 ± 10.91	100.4 ± 9.96	105.44 ± 12.28
Flucycloxuron	80.3 ± 8.85	81.03 ± 11.62	82.62 ± 7.36
Flufenacet (Flutamide)	90.25 ± 16.47	87.22 ± 15.21	103.94 ± 15.77
Flufenoxuron	82.22 ± 13.61	97.01 ± 14.07	92.98 ± 9.82
Fluometuron	111.47 ± 10.35	109.1 ± 13.09	106.36 ± 4.16
Fluopicolide	111.38 ± 16	104.24 ± 9.85	96.03 ± 6.23
Flupyradifurone	105.1 ± 9.62	100.12 ± 6.48	94.82 ± 7.63
Flupyrsulfuron-methyl	D	D	D
Fluridone	85.61 ± 3.57	88.69 ± 2.81	88.38 ± 3.67
Flurprimidol	119.56 ± 21.6	110.26 ± 7.67	116.9 ± 6.04
Flurtamone	96.8 ± 7.45	99.15 ± 13.59	102.07 ± 9
Flusilazole	87.6 ± 10.53	89.77 ± 7.58	106.2 ± 12.68
Fluthiacet-methyl	84.85 ± 8.06	86.51 ± 6.91	86.98 ± 2.9
Flutolanil	97.43 ± 6.57	95.99 ± 12.04	110.39 ± 7.77
Flutolanil	96.86 ± 19.18	73.76 ± 12.72	115.28 ± 7.32
Flutriafol	115.34 ± 15.84	121.5 ± 9.25	104.43 ± 4.23

Compound	Recovery (%)		
	LOQ (10 µg/kg)	2 LOQ (20 µg/kg)	5 LOQ (50 µg/kg)
Fluxapyroxad	111.82 ± 8.51	105.91 ± 8.12	105.03 ± 3.69
Forchlorfenuron	D	48.02 ± 5.36	51.65 ± 5.43
Formetanate	79.07 ± 12.07	74.54 ± 6.86	72.95 ± 5.47
Formothion	D	D	67.06 ± 13.08
Fosthiazate	97.95 ± 7.82	104.32 ± 7	102.87 ± 3.09
Fuberidazole	60.48 ± 5.53	63.32 ± 6.39	65.07 ± 7.17
Furalaxyl	90.82 ± 14.45	101.74 ± 10.26	101.58 ± 5.99
Furametpyr	107.03 ± 17.77	99.26 ± 15.95	94.45 ± 9.24
Halosulfuron-methyl	D	43.3 ± 8.12	43.32 ± 7.33
Haloxyfop-methyl	112.09 ± 11.7	81.79 ± 11.43	83.2 ± 5.16
Heptenophos	95.03 ± 17.77	121.72 ± 17.82	99.32 ± 7.36
Hexazinone	81.38 ± 10.73	86.46 ± 5.97	87.39 ± 4.52
Hexythiazox	D	91.79 ± 17.58	88.08 ± 10.97
Imazamethabenz-methyl	119.7 ± 3.57	109.81 ± 5.14	95.04 ± 3.61
Imibenconazole	77.69 ± 6.31	84.77 ± 14.54	79.29 ± 9.4
Imidacloprid	78.61 ± 13.5	74.83 ± 10.51	71.25 ± 6.6
Indaziflam	95.38 ± 9.28	104.18 ± 16.85	104.56 ± 11.07
Indoxacarb	107.34 ± 16.1	117.28 ± 11.74	112.37 ± 12.21
IPC/Propham	101.98 ± 9.42	97.91 ± 11.09	96.55 ± 4.28
Ipconazole(I)	84.38 ± 10.77	100.32 ± 9.46	73.34 ± 5.81
Iprobenfos	84.18 ± 14.73	101.5 ± 19.08	106.37 ± 8.65
Isofenphos	D	D	121.39 ± 13.13
Isofenphos Methyl	119.61 ± 5.17	133.63 ± 19.64	112.88 ± 7.41
Isofenphos Oxon	117.74 ± 8.2	103.75 ± 12.08	101.05 ± 6.96
Isoprothiolane	117.05 ± 11.23	117.33 ± 7.21	105.64 ± 4.26
Isoproturon	93.43 ± 6.77	99.77 ± 5.26	99.65 ± 2.24
Isouron	89.48 ± 5.12	94.22 ± 5.32	97.89 ± 3.19
Isoxaben	114.53 ± 13.95	117.38 ± 10.97	108.33 ± 4.98
Isoxadifen-ethyl (AE F122006)	103.81 ± 15.63	114.17 ± 8.4	110.6 ± 5.94
Kresoxim-methyl	D	111.31 ± 10.92	111.8 ± 8.41
Lactofen	D	D	85.43 ± 16.19
Lenacil	58.16 ± 10.9	77.32 ± 11.99	90.58 ± 9.52
Malaoxon	104.54 ± 6.44	115.29 ± 6.75	109.35 ± 3.3
Malathion	93.25 ± 18.26	98.98 ± 11.65	115.87 ± 6.52
Mandipropamid	77.78 ± 12.75	91.74 ± 15.23	116.57 ± 7.18
Mecarbam	102.44 ± 20.11	111.01 ± 14.41	103.54 ± 6.3
Mepanipyrim	82.1 ± 15.13	94.21 ± 14.4	87.51 ± 6.49
Mephosfolan	94.91 ± 3.56	96.92 ± 5.14	97.49 ± 4.13
Mesosulfuron-methyl	D	D	D
Metaflumizone	D	107.99 ± 8.14	108.58 ± 12.51
Metalaxyl	100.91 ± 17.03	104 ± 10.28	109.7 ± 7.51
Metamitron	83.83 ± 16.47	84.62 ± 5.67	80.61 ± 8.51
Metazachlor	D	D	95.09 ± 11.21
Metconazole	112.14 ± 18.72	94.44 ± 17.84	74.8 ± 8.67
Methabenzthiazuron	D	59.64 ± 8.64	63.61 ± 5.54
Methamidophos (Methamidophos)	D	94.17 ± 11	60.44 ± 10.97

Compound	Recovery (%)		
	LOQ (10 µg/kg)	2 LOQ (20 µg/kg)	5 LOQ (50 µg/kg)
Methidathion	104.81 ± 9.6	109.7 ± 11.15	99.33 ± 6.05
Methiocarb Sulfone	D	D	46.14 ± 6.57
Methiocarb Sulfoxide	74.51 ± 3.83	72.19 ± 6.33	70.18 ± 4.8
Methomyl	72.86 ± 9.35	76.6 ± 6.49	82.59 ± 5.69
Methoprotryne	100.92 ± 3.91	104.42 ± 3.01	96.73 ± 1.89
Metobromuron	35.84 ± 14.53	49.59 ± 17.96	71.3 ± 13.19
Metolachlor	90.32 ± 15.9	94.11 ± 13.46	105.58 ± 13.72
Metosulam	D	D	D
Metoxuron	84.44 ± 6.44	86.06 ± 9.74	94.23 ± 7.27
Metribuzin	54.35 ± 10.56	96.29 ± 10.1	93.74 ± 6.43
Metronidazole	51.87 ± 10.28	55.33 ± 10.99	62.44 ± 8.69
Metsulfuron-methyl	D	D	D
Monocrotophos (Azodrin)	85.16 ± 9.56	84.19 ± 7.29	89.63 ± 8.1
Monuron	77.53 ± 8.02	87.35 ± 6.45	87.65 ± 4.86
Napropamide	99.52 ± 19.44	107.24 ± 15.22	101.37 ± 11.73
Neburon (Phosphoramidothioic Acid)	D	90.37 ± 18.02	81.28 ± 5.01
Norflurazon	92.33 ± 9.93	94.41 ± 4.62	92.96 ± 3.69
Nuarimol	94.05 ± 18.74	103.65 ± 19.96	86.35 ± 10.29
Ofurace	95.03 ± 5.15	99.51 ± 5.26	97.77 ± 5.08
Omethoate	80.02 ± 11.45	79.93 ± 7.09	82.22 ± 7.01
Oryzalin	D	112.39 ± 18.59	106.35 ± 8.43
Oxadixyl	106.7 ± 10.47	92.85 ± 8.18	102.86 ± 7.85
Oxasulfuron	1.62 ± 2.01	2.82 ± 2.61	4.45 ± 4.91
Oxathiapiprolin	98.84 ± 12.68	109.21 ± 6.69	103.34 ± 3.91
Oxycarboxin	94.61 ± 4.78	96.13 ± 6.32	97.44 ± 2.91
Oxydemeton-methyl	60.79 ± 3.01	66.68 ± 5.45	69.73 ± 8.94
Paclobutrazol	54.89 ± 10.12	78.67 ± 8.95	91 ± 5.52
Paraoxon-methyl	79.4 ± 12.55	87.87 ± 15.57	95.33 ± 11.32
Penconazole	66.35 ± 11.77	71.27 ± 9.17	80.01 ± 8.96
Penflufen	96.97 ± 18.36	96.7 ± 11.81	90.36 ± 6.78
Penoxsulam (Penoxalim)	15.84 ± 3.44	24.61 ± 8.02	24.26 ± 6.57
Pentanochlor	87.21 ± 10.2	89.25 ± 9.19	106.4 ± 6.64
Penthiopyrad	100.99 ± 10.18	114.32 ± 16.32	120.8 ± 6.46
Pethoxamid	88.9 ± 11.97	72.92 ± 13.77	90.11 ± 13.73
Phenmedipham	87.35 ± 8.89	88.47 ± 8.3	90.47 ± 3.24
Phenthoate (Fenthoate)	D	105.91 ± 20.47	105.83 ± 11.96
Phorate-sulfone	102.37 ± 20.14	113.05 ± 14.29	103.58 ± 6.88
Phorate-sulfoxide	109.35 ± 20	116.44 ± 14.56	99.53 ± 4.58
Phosmet-oxon	79.7 ± 6.95	82.62 ± 4.84	85.56 ± 5.12
Phosphamidon	105.23 ± 4.44	106.48 ± 4.79	101.98 ± 5.95
Picolinafen	D	94.34 ± 18.71	103.81 ± 12.86
Picoxystrobin	92.17 ± 14.52	102.35 ± 18.94	108.56 ± 9.72
Piperonyl Butoxide	78.64 ± 15.52	74.07 ± 12.67	85.96 ± 7.75
Piperophos	64.91 ± 37.71	79.93 ± 45.13	110.53 ± 18.13
Pirimicarb	95.9 ± 9.49	94.02 ± 6.7	94.08 ± 3.65
Pirimicarb-desmethyl-formamido	87.5 ± 4.58	90.22 ± 3.85	88.38 ± 4.52

Compound	Recovery (%)		
	LOQ (10 µg/kg)	2 LOQ (20 µg/kg)	5 LOQ (50 µg/kg)
Pirimiphos-ethyl (Pirimifos-ethyl)	83.11 ± 14.62	81.29 ± 15.94	93.58 ± 15.01
Pirimiphos-methyl (Pirimifos-methyl)	107.7 ± 10.82	101.98 ± 14.6	90.8 ± 7.02
Pyridaben	66.48 ± 8.25	74.53 ± 12.4	77.9 ± 10.34
Prochloraz	D	74.25 ± 12.65	94.05 ± 14.83
Procymidone	68.09 ± 8.41	82.35 ± 5.83	80.16 ± 4.07
Profluralin	71.36 ± 9.25	70.27 ± 10.84	79.71 ± 11.74
Propamocarb	14.5 ± 4.06	15.37 ± 2.42	17.47 ± 3.85
Propanil	D	98.25 ± 38.39	89.97 ± 5.69
Propaphos	93.13 ± 8.19	93.51 ± 13.02	101.15 ± 5.56
Propaquizafop	52.06 ± 10.27	64.15 ± 12.09	66.94 ± 5.9
Propargite	111.14 ± 18.87	106.35 ± 15.48	98.4 ± 4.66
Propyzamide (Pronamide)	94.81 ± 15.75	79.41 ± 8.37	90.95 ± 11.49
Pydiflumetofen	91.85 ± 10.03	110.33 ± 16.01	94.94 ± 4.12
Pyraclofos	74.26 ± 12.18	90.7 ± 15.9	101.22 ± 9.67
Pyraclostrobin	92.46 ± 15.28	93.41 ± 18.66	96.38 ± 8.38
Pyrazophos	D	D	96.91 ± 15.29
Pyribencarb	80.6 ± 5.84	84.67 ± 3.92	86.41 ± 3.6
Pyridaben	67.5 ± 7	75.8 ± 14.7	77.54 ± 10.06
Pyridalyl	16.48 ± 9.41	25.77 ± 6.32	30.96 ± 4.37
Pyrifluquinazon	102.12 ± 13.41	95.75 ± 7.54	98.25 ± 4.04
Pyrimethanil	D	90.61 ± 16.3	76.9 ± 7.17
Pyrimidifen	71.14 ± 7.58	52.92 ± 8.48	58.1 ± 6.42
Pyriofenone	88.57 ± 16.32	77.81 ± 13.08	91.29 ± 5.67
Pyroxsulam	D	D	3.24 ± 3.78
Quinalphos (Diethquinalphione) II	107.84 ± 13.43	114.05 ± 11.16	110.73 ± 5.6
Quinoclamine (ACN)	42 ± 4.15	48.65 ± 5.86	49.53 ± 2.14
Quinoxyfen	21.96 ± 15.85	43.6 ± 15.68	52.82 ± 8.92
Rotenone	D	D	82.19 ± 3.51
Saflufenacil	D	D	54.41 ± 6.26
Sebuthylazine	120.68 ± 6.11	116.68 ± 17.56	103.28 ± 9.16
Secbumeton	98.1 ± 6.09	103.18 ± 3.56	97.29 ± 4.05
Silthiofam	116.24 ± 22.35	93.52 ± 12.84	95.2 ± 3.61
Spinetoram	26.09 ± 5.87	31.5 ± 6.23	32.34 ± 4.7
Spirodiclofen	D	D	48.5 ± 9.2
Spirotetramat	87.81 ± 17.33	67.42 ± 13.04	71.33 ± 13.59
Sulfentrazone	D	37.17 ± 14.09	43.46 ± 8.56
Sulfotep	102.64 ± 15.77	103.97 ± 13.49	109.59 ± 8.98
TBZ/Thiabendazole (Thiabendazole)	40.88 ± 3.04	41.54 ± 2.89	44.02 ± 4.3
Tebupirimfos	98.65 ± 18.89	102.28 ± 20.39	106.27 ± 16.03
Tebuthiuron	80.21 ± 12.57	97.02 ± 6.64	84.59 ± 4.53
TEPP/Tetraethyl Diphosphate	74.75 ± 3.32	77.2 ± 4.03	82.82 ± 3.44
Tebufenozide	93.96 ± 17.18	108.14 ± 12.98	118.37 ± 12.83
Terbufos	D	D	85.56 ± 12.27
Terbufos Sulfone	74.62 ± 14.79	94.07 ± 17.56	107.56 ± 7.06
Terbufos Sulfoxide	95.48 ± 11.31	104.65 ± 13.16	100.18 ± 6.26
Terbumeton	93.99 ± 4.49	96.87 ± 3.89	97.52 ± 2.03

Compound	Recovery (%)		
	LOQ (10 µg/kg)	2 LOQ (20 µg/kg)	5 LOQ (50 µg/kg)
Terbuthylazine-desethyl	79.71 ± 10.48	91.73 ± 15.26	92.81 ± 7.3
Terbutryn	92.95 ± 2.45	100.26 ± 4.2	96.24 ± 3.65
Tetraconazole	119.67 ± 8.04	130.08 ± 17.46	131.21 ± 16.11
Thenylchlor	117.63 ± 16.16	117.33 ± 20.19	111.1 ± 12.21
Thiacloprid	74.43 ± 9.69	74.1 ± 4.2	75.58 ± 3.91
Thiamethoxam	70.21 ± 6.74	63.16 ± 8.56	64.45 ± 7.7
Thidiazuron	D	36 ± 8.86	29.23 ± 4.63
Thiodicarb	77.9 ± 5.84	80.27 ± 5.18	82.37 ± 4.18
Thiofanox-sulfone	115.44 ± 22.68	109.1 ± 10.36	105.95 ± 6.92
Thiofanox-sulfoxide	97.05 ± 17.07	96.87 ± 7.51	92.34 ± 5.57
Tolfenpyrad	78.55 ± 13.01	80.85 ± 11.04	81.13 ± 7.32
Triadimefon	81.51 ± 15.63	111.54 ± 19.76	100.53 ± 17.26
Triafamone	110.97 ± 4.56	110.74 ± 5.8	107.38 ± 4.05
Triasulfuron (Logran)	D	D	D
Tribenuron-methyl	D	D	D
Trichlorfon (Dylox) (DEP)	85.02 ± 15.33	77.45 ± 8.39	83.78 ± 9.26
Tricyclazole	35.6 ± 6.69	37.1 ± 4.41	35.73 ± 1.31
Trifloxystrobin	81.18 ± 11.7	87.77 ± 15.16	99.61 ± 11.03
Triflumezopyrim	90.26 ± 7.09	83.95 ± 8.48	78.64 ± 4.04
Triflumizole	D	D	89.46 ± 8.42
Triflumizole Metabolite FM-6-1	97.94 ± 6.19	98.06 ± 5.87	98.52 ± 6.1
Triflusulfuron-methyl	31.47 ± 6.01	42.48 ± 5.64	43.58 ± 3.88
Triasulfuron	60.54 ± 11.51	59.01 ± 10.51	68.78 ± 7.6
Uniconazole-P(I)	76.84 ± 14.48	74.04 ± 14.68	64.69 ± 9.62
Uniconazole-P(II)	85.96 ± 14.71	96.78 ± 10.22	93.34 ± 7.9
Valifenalate	91.57 ± 14.17	117.29 ± 9.3	100.38 ± 3.57
Vamidothion	94.22 ± 4.54	94.92 ± 3.35	93.4 ± 5.64
Vamidothion Sulfone	83.75 ± 4.88	85.35 ± 7.19	88.72 ± 8.37
Vamidothion Sulfoxide	46.38 ± 4.69	57.28 ± 6.74	65.65 ± 9.31
Warfarin	41.68 ± 12.43	44.32 ± 8.53	40.36 ± 2.05

D = Detected, but average recovery or RSD did not meet SANTE guideline.

www.agilent.com

DE23696489

Printed in the USA, March 4, 2024
5994-7214EN

Agilent
Trusted Answers