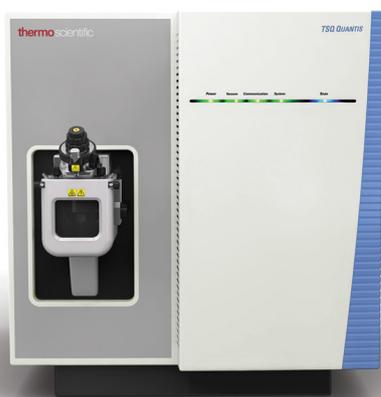




Meeting the challenges in pesticide residues analysis with the TSQ Quantis Triple Quadrupole Mass Spectrometer



The Thermo Scientific™ TSQ Quantis™ triple-stage quadrupole mass spectrometer provides reliability, confidence, and sensitivity to overcome analytical challenges in food analysis. Even in the most difficult matrices and applications, the TSQ Quantis MS delivers best-in-class quantitation day after day, with confidence.

Introduction

The analysis of pesticide residues is one of the most commonly performed and challenging routine analyses in food control laboratories. Maximum residue levels (MRLs) are set by individual countries and vary by pesticide and commodity. Therefore, food supplies, including imported and exported products, must be monitored to check for compliance with these MRLs to ensure that consumers are not being exposed to pesticide levels that are harmful to their health. As new pesticides are introduced by the agrochemical industry and MRLs are lowered, the analysis of pesticide residues in food matrices becomes even more complex and requires precise, fast, and sensitive approaches.

For laboratories that run multi-analyte panels, there is pressure to increase the number of components and lower the detection limits while maintaining the quality standards set by ISO 17025 and without increasing production cost or time. ISVEA srl has been a leader in providing analytical services of excellence for the agri-food sector for years and is highly specialized in wine analysis. ISVEA wanted to improve the performance of their current multi-residue pesticide method by doubling the number of analytes, increasing sensitivity, and achieving higher throughput, while maintaining the QuEChERS sample preparation specified by reference method UNI EN 15662:2018.

The original assay used by ISVEA involved a multi-class method of 300+ components divided between LC-MS/MS (about 100 molecules, 20 min) and GC-MS/MS (about 200 molecules, 70 min) with an LOQ of 10 ppb. An upgraded method was developed on a Thermo Scientific™ Vanquish™ Flex Binary UHPLC system coupled with a Thermo Scientific™ TSQ Quantis™

triple quadrupole mass spectrometer. The high sensitivity, SRM speed, and robustness of the TSQ Quantis mass spectrometer combined with the excellent retention time reproducibility and reliability of the Vanquish Flex UHPLC system provided the optimal system for this type of routine analysis.

Using the TSQ Quantis Triple Quadrupole Mass Spectrometer

The chemists at ISVEA were able to develop a quantitative method for more than 400 pesticides with a 15-minute chromatographic run and an LOQ of 2.5 ppb for 95% of the analytes. One of the challenges was complying with the stringent criteria set by the EU SANTE guidelines for the validation of multi-residue methods, including the definition of the LOD/LOQ and the confirmation of positive residues, in particular, the SRM number. With the TSQ Quantis mass spectrometer, the chemists at ISVEA achieved an LOQ of 10 ppb on almost all the components sought respecting the ion ratio confirmation criteria.

Table 1. Validation parameters and criteria*

Parameter	What/how	Criterion	Cross reference to AQC document
Sensitivity/linearity	Linearity check from five levels	Deviation of back-calculated concentration from true concentration $\leq \pm 20\%$	C14-C19
Matrix effect	Comparison of response from solvent standards and matrix-matched standards	**	C22-C24
LOQ	Lowest spike level meeting the method performance criteria for trueness and precision	\leq MRL	G6
Specificity	Response in reagent blank and blank control samples	$\leq 30\%$ of RL	C42
Trueness (bias)	Average recovery for each spike level tested	70–120%	G3,G6
Precision (RSD _r)	Repeatability RSD _r for each spike level tested	$\leq 20\%$	G3, G6
Precision (RSD _{wR})	Within-laboratory reproducibility, derived from on-going method validation / verification	$\leq 20\%$	G3, G6
Robustness	Average recovery and RSD _{wR} derived from on-going method validation / verification	See above	G6, C40-C44
Ion ratio	Check compliance with identification requirements for MS techniques	Table 4	Section D
Retention time		± 0.1 min	D2

*From: Guidance document on analytical quality control and method validation procedures for pesticides residues and analysis in food and feed, European Commission, SANTE/11813/2017, 21-22 November 2017 rev.0.

**In the case of more than 20% signal suppression or enhancement, matrix effects need to be addressed in the calibration (C22 C30)

Another critical concern for this type of analysis is the reproducibility, or stability, of the entire system (instrumental response and retention times) in a complex matrix, such as wine or olive oil—the core business of ISVEA. For a private laboratory, instrument downtime caused by equipment failure or instrument maintenance is costly. Therefore, the minimum maintenance time required by the TSQ Quantis system is critically important to improve throughput and reduce costs. After six months of continuous 24 hours per day use, both the mass spectrometer and the UHPLC system provided unparalleled robustness.

The management of the data, both from the point of view of traceability and the processing of analytical results, is a key point for the efficiency of the laboratory. For this reason, particular attention was paid to the evaluation of data processing software. Thermo Scientific™ TraceFinder™ software easily manages data acquisition and data processing of large numbers of analytes and samples, integrating efficiently with the already existing LIMS interface.

Conclusion

With the use of the Vanquish Flex UHPLC/TSQ Quantis mass spectrometer system, ISVEA realized its objective of a fast, sensitive, and efficient multi-class analysis with quantitative monitoring of more than 400 substances simultaneously, validated as required by ISO 17025. In

the future, ISVEA expects to further expand the number of pesticides and improve the sensitivity of the analysis.

During the development of the method and its validation, ISVEA received great support from both the Thermo Fisher Scientific Sales and Application teams to make this a successful collaboration.

Who is ISVEA?

ISVEA srl is an analytical laboratory that has been a leader in providing analytical services of excellence for the agri-food sector for years and is highly specialized in wine analysis. ISVEA offers a vast catalog of services and can customize solutions for customers, in particular in enological production chains and other agriculture fields.

ISVEA's close collaboration with local authorities, universities, and research institutes helps it effectively respond to the analytical needs of the environmental and food safety markets. The laboratory is accredited according to the standard UNI CEI EN ISO IEC 17025. Currently, ISVEA works with about 3000 companies in the Italian and European wine sector.

ISVEA is a multi-purpose analysis laboratory authorized by the Ministry of Agricultural, Food, and Forestry Policies for the certification of wine and oil and is accredited by ACCREDIA.



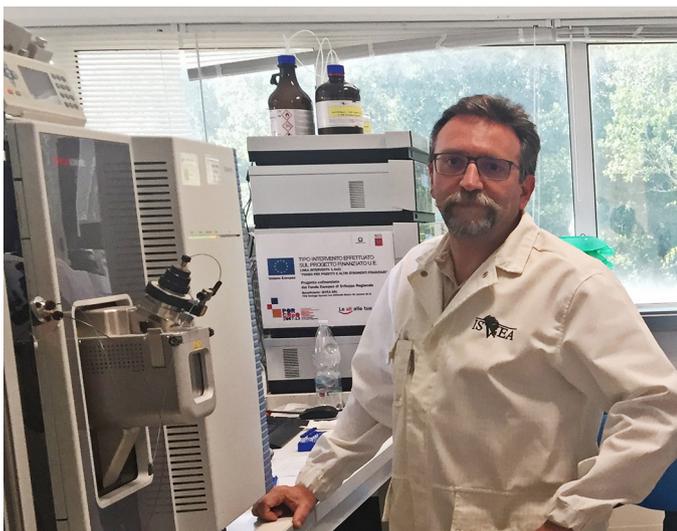
ISVEA srl analytical laboratory, which is highly specialized in wine analysis

Questions: Alessandro Cavaglioni, Laboratory Manager

What current work are you involved in and what does your experience bring to ISVEA?

I have been the manager of the ISVEA laboratory for 21 years. My duties are multiple, but one of the most important for business strategy is the choice of analytical instruments. Over the years I have always tried to identify the most suitable analytical technology for each analysis. This has led to a strong instrumental differentiation in the laboratory, which provides the advantages of being able to respond quickly to analytical requests from customers and of increasing company knowledge in various techniques.

“Over the years I have always tried to identify the most suitable analytical technology for each analysis.”



ISVEA Laboratory Manager Alessandro Cavaglioni

What is your experience with TSQ triple quadrupole technology for quantitation and screening? What increases your confidence in pesticide identification?

We purchased the first LC-MS/MS in 2008 for the analysis of pesticides in wine, a matrix rich in interferences. Since that time we have analyzed thousands of samples. Normally the control of one confirmation transition (in addition to the quantification one) is sufficient. With two SRM transitions per component, the more complex cases are solved. From the quantitative point of view, the matrix effect can cause ion suppression of the signal, although this effect becomes less significant over the years with the increase in instrument performance. The use of matrix-matched calibration and internal standards helps to minimize some matrix effect issues.

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

Do you feel that TSQ triple quadrupole technology can help food testing companies identify contamination, and what kind of barriers do you see that will limit the use of this technology?

Yes, for sure. Today this technique is one of the best to quantify micro-contaminants we already know. This is also its limitation: with this technique it is impossible to search for compounds that were not initially envisioned.



TSQ Quantis triple quadrupole mass spectrometer

Looking to the future what do you think other scientists in food safety labs need to know about LC-MS/MS?

One of the technical improvement points is the reduction of the signal suppression effect due to the matrix. It would be useful to know if sample introduction systems are being studied for this.

Would you recommend this technology to other scientists analyzing pesticides for food safety, not just for import but also export?

Yes. It is a very useful preventive control because many pesticides that are allowed in one country are banned in others, and goods can be stopped at customs. Moreover, with a robust and fast method, the response time is very quick.

“...with a robust and fast method, the response time is very quick.”

Why did ISVEA choose to work with Thermo Fisher Scientific? How do you feel about the current collaboration?

ISVEA needs partners with experts who are able to support the company in challenging goals, such as doubling the number of components analyzed in a single run with high reliability and robustness. Thermo Fisher Scientific, with its instruments and applications specialists, is a company that can guarantee all of these requirements.



Laboratory technicians Franca Congiu (foreground) and Francesca Barbetti (background)

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