

CASE STUDY
Laboratori de l'Agència de
Salut Pública de Barcelona

GC Orbitrap MS/MS technology adds extra certainty to food safety and environmental testing



Thermo Scientific™ Q Exactive™ GC
Orbitrap™ GC-MS/MS System

“Confidence in analytical results is extremely important because they have legal and financial consequences. We need to eliminate chances of false positive or false negatives. Though we routinely use triple quadrupole instruments, if we get any inconclusive results, we go to GC Orbitrap MS/MS technology to add additional specificity and sensitivity to our workflows.”

— Dr. Nuria Cortés-Francisco, Laboratori de l'Agència de
Salut Pública de Barcelona

“Having another technology to confirm results is making our work easier by ensuring there are no errors due to matrix interferences. And, with flexible scope accreditation, we have to accept any analytical request, set up a method, and provide results as soon as possible.”

— Dr. Nuria Cortés-Francisco

Food safety and environmental testing labs face two important obstacles when using traditional triple quadrupole GC-MS methods. First, it is difficult to reach very low limits of quantitation (LOQs) for some emerging compounds of concern, such as polybrominated diphenyl ethers (PBDEs), in complex sample matrices without time-consuming sample concentration prior to analysis. High molecular weight compounds such as BDE-209 pose an additional challenge, because the sensitivity of triple quadrupole systems drops off at higher molecular weights. Achieving certainty in results using a practical confirmatory method or alternative technology is extremely important when triple quadrupole methods produce ambiguous results.

These are among the challenges faced by the Laboratori de l'Agència de Salut Pública de Barcelona, the laboratory chartered with supporting food safety and environmental surveillance programs for Barcelona, Spain. With ISO/IEC 17025: 2005, accreditation, including flexible scope accreditation by the National Entity of Accreditation (ENAC), and frequent new EU alerts about potential contamination, the laboratory must also rapidly respond to emerging food safety and environmental matters.

Using the Thermo Scientific Q Exactive GC Orbitrap GC-MS/MS system, the laboratory realizes the additional certainty provided high specificity and sensitivity, substantially reducing the chance false negative and false positive results.

GC Orbitrap MS/MS technology solves specificity and sensitivity challenges

Food safety and environmental testing methods often rely on triple quadrupole GC and LC-MS methods. These methods usually provide the sensitivity needed to meet regulatory requirements, but when they don't because of matrix interferences, or when a confirming method is needed, orthogonal technology with enhanced specificity and sensitivity is desirable. This is why the Laboratori de l'Agència de Salut Pública de Barcelona adopted a Q Exactive GC Orbitrap GC-MS/MS approach to confirming suspect triple quadrupole GC-MS results.

According to Dr. Nuria Cortés-Francisco, “the concern we had with our triple quadrupole methods was matrix interferences, where in some cases we were not confident in our results because the confirming ion ratio requirements weren't met. Based on triple quadrupole method requirements, you would say it's a negative, but with an obvious peak we were concerned that the negative was false. In these cases, we confirm our triple quadrupole results using the Q Exactive GC Orbitrap GC-MS/MS system. With a triple quadrupole system you only obtain the nominal mass so you can't be sure if it's the target compound or matrix interferences.”

To develop the protocol, the laboratory built a HRAM database that includes the retention times, exact masses, and confirmatory ions for target compounds of interest. Using the database, the laboratory can quickly set up a method for a confirmatory analysis. The confirmatory method uses three ions—one for quantification and two for confirmation to compare two ion ratios—exceeding the SANTE guidelines that require two ions measured by HR MS and one ion ratio.

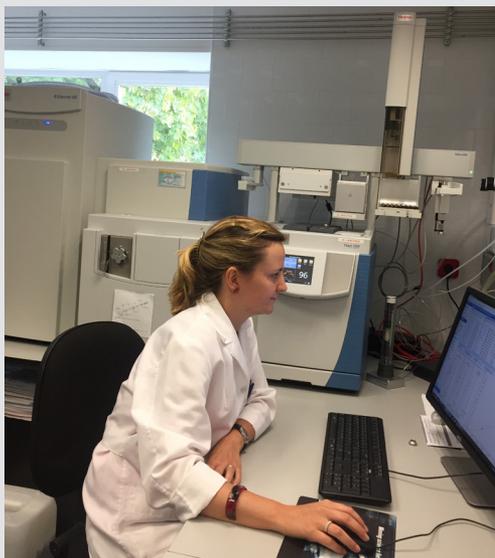


Photo courtesy of Laboratori de l'Agència de Salut Pública de Barcelona

“All suspicious results from low-resolution GC-MS triple quadrupole instruments are now confirmed by the Q Exactive GC Orbitrap GC-MS/MS system. Using both full-scan and SIM methods, and our HRAM compound database, confirmation of results is extremely fast and effective.”

— Dr. Nuria Cortés-Francisco

Confirming suspect results for propargite

In 2016, Dr. Cortés-Francisco applied the Q Exactive GC Orbitrap GC-MS/MS system protocol to address an alert for propargite in oranges. When analyzed using traditional QuEChers sample preparation and triple quadrupole GC-MS, most of the samples tested negative

for propargite. However, some results were inconclusive. All of the samples had some signal for both target ion transitions, but the confirming ion ratio criteria were only met in some cases. Using the database, the laboratory promptly set up a Q Exactive GC Orbitrap GC-MS/MS method, reanalyzed the samples, and confidently determined one sample positive.

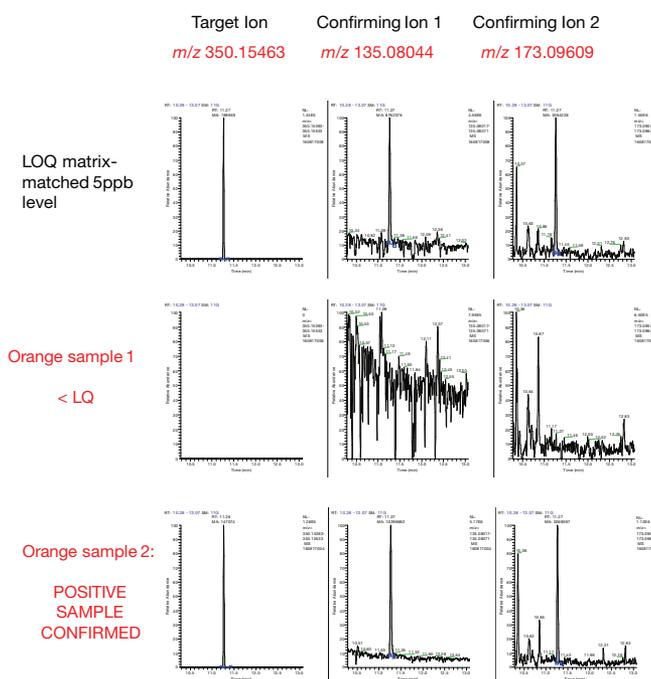


Figure 1. Q Exactive GC Orbitrap GC-MS/MS system analysis of orange samples for propargite (MRL for propargite is 0.01 mg/kg.) When some triple quadrupole results were inconclusive, the samples were reanalyzed using the Q Exactive GC Orbitrap GC-MS/MS system, which was subsequently able to distinguish between the matrix and pesticide signals, confirming the positive findings.

“The Q Exactive GC system provides high selectivity as we can use very high resolution at scan speeds amenable to GC, and its sensitivity is comparable to the best triple quadrupole systems.”

— Dr. Nuria Cortés-Francisco

Achieving low detection limits for difficult PBDEs

Though brominated compounds have been analyzed in environmental applications for years, in 2014 the EU published a recommendation that these compounds should also be analyzed in foods down 0.01 ng/g. Of the brominated compounds targeted, one of the largest with ten bromines—BDE-209—is difficult to analyze using triple quadrupole GC-MS due to its limited sensitivity for higher molecular weight compounds in matrices. For this reason, some labs have chosen to perform the analysis using magnetic sector methods.

“Because we needed to achieve very low LOQs for PBDEs in many kinds of difficult matrices—seafood, vegetables, meat, eggs, olive oil, etc.—we applied the Q Exactive GC Orbitrap GC-MS/MS system,” noted Dr. Cortés-Francisco. “With a simple method that uses selected ion monitoring (SIM) windows, we monitor not only the main peak but also the isotopic pattern, allowing us to be sure of a positive hit.”

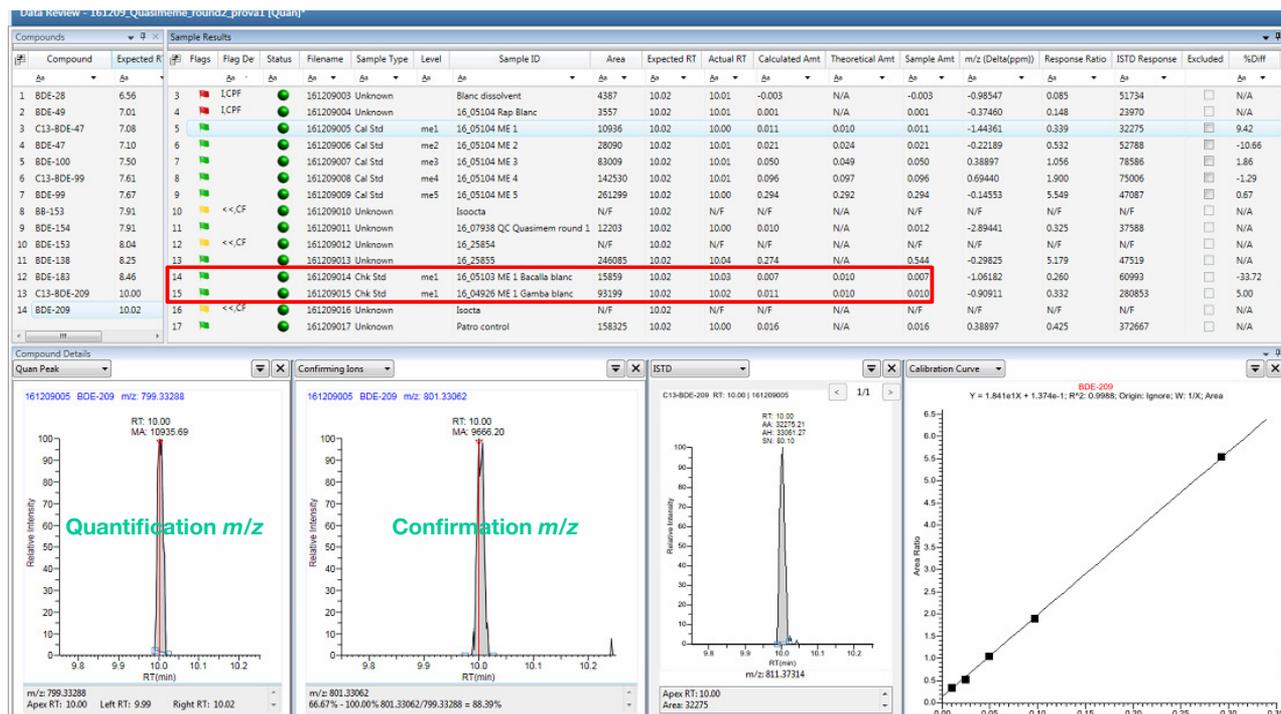
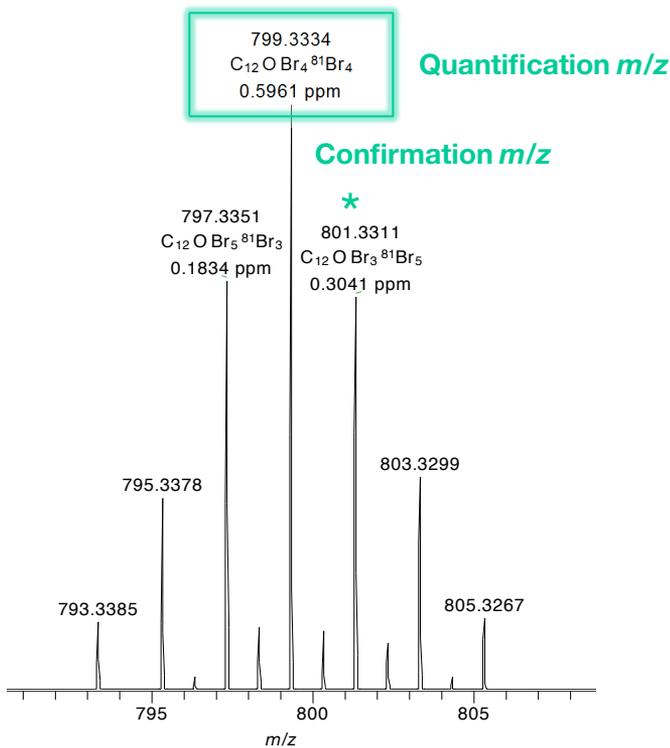


Figure 2. Q Exactive GC Orbitrap GC-MS/MS method results. Matrix-matched calibration curve for monkfish sample spiked with BDE-209 from 0.01 to 0.3 ng/g. Courtesy of Laboratori de l'Agència de Salut Pública de Barcelona.

“We’re easily reaching the LOQs for PBDEs, because the Q Exactive GC Orbitrap GC-MS/MS system has the necessary sensitivity even at higher masses, and its high resolution allows us resolve matrix interferences. There are not many labs analyzing PBDE compounds at 0.01 ng/g levels because its nearly impossible, but we are able to do it thanks to the Q Exactive GC Orbitrap GC-MS/MS system.”

— Dr. Nuria Cortés-Francisco



Conclusion

Confidence in the results of food safety and environmental testing is of utmost importance. The Q Exactive GC Orbitrap GC-MS/MS system brings together the power of high-resolution GC and HRAM Orbitrap MS to provide high-specificity, high-sensitivity target compound analyses when GC-MS triple quadrupole methods produce inconclusive results or cannot achieve required detection limits. When a confirming method is needed, the Q Exactive GC Orbitrap GC-MS/MS system is the orthogonal technology to apply.

Figure 3. The Q Exactive GC Orbitrap GC-MS/MS system can acquire a full isotopic pattern for difficult compounds such as BDE-209 (shown here) without sensitivity loss, providing more confidence in results. Courtesy of Laboratori de l'Agència de Salut Pública de Barcelona.



Photo courtesy of Laboratori de l'Agència de Salut Pública de Barcelona

About Nuria Cortés-Francisco

Dr. Nuria Cortés-Francisco received her PhD in Analytical Chemistry from the University of Barcelona. She developed her career at the Spanish Council for Scientific Research, where she mainly worked on the use and study of high-resolution mass spectrometry for the analysis of organic pollutants in environmental and food samples. Since 2014, she has been the Emerging Contaminants and Mass Spectrometry Specialist at the Laboratori de l'Agència de Salut Pública de Barcelona.

About the Laboratori de l'Agència de Salut Pública de Barcelona

The Laboratori de l'Agència de Salut Pública de Barcelona (Laboratory of the Public Health Agency of Barcelona) was created in 1887. Since then, it has evolved to meet the needs of public health in the city, and in 2003 it underwent a transformation with the integration of the human and material resources of the Laboratory of Public Health of the Generalitat de Catalunya in Barcelona.

The laboratory contributes to the identification and control of public health programs in the community. This is done by supporting analytical monitoring programs for food safety and environmental surveillance and involves screening of over 35000 samples per year. The laboratory has more than 1,000 analytical parameters of reference covered by the accreditation ISO 17025: 2005, granted by the National Entity of Accreditation (ENAC). Modern instrumentation and having the scope of flexible accreditation enables the laboratory to rapidly respond to emerging problems in the areas of food safety. The work of the laboratory is based on



Photo courtesy of Laboratori de l'Agència de Salut Pública de Barcelona

collaboration with clients, adaptation to new needs and demands, and the continuous expansion of services and competences. Everyone can access the services offered by the laboratory; both public administrations and companies and individuals that want to ensure the quality and safety of products in accordance with current food legislation.

Find out more at thermofisher.com/OrbitrapGCMS

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