

Growing Demand for Rapid Food Contaminant Detection Spurs New Method Development at NofaLab

Waters Xevo TQ-S and TQ-S micro systems combined with APGC help scientists improve method development for rapid contamination analysis at NofaLab in the Netherlands

TECHNOLOGY: WATERS XEVO TQ-S and TQ-S MICRO SYSTEMS

FOOD, FEED, AND ENVIRONMENTAL SAFETY AT NOFALAB

NofaLab BV is an independent sampling and testing laboratory specializing in the field of food, feed and environmental safety. The company offers the most extensive range of analytical methods for food and feed with the fastest turnaround time in the world.

Founded in April 2004, the company's 160+ employees work in food and feed-related applications, including analysis of quality parameters for oil and fat, 3-MCPD (including esters), mineral oil, heavy metals, pesticides, micro-organisms, organic contaminants (dioxins, PCB, PAH and VOC), GMO, mycotoxins, Phthalates, Bisphenol A, alkaloids, biofuels, and dyes. The company has seen a sharp increase in business in recent years. Tighter regulations require more monitoring, and NofaLab customers are sending more samples for analysis.

Located at Schiedam in the Port of Rotterdam, NofaLab is ideally placed to serve customers at the principal gateway to Europe. Other locations in the Ukraine and Black Sea also serve customers in eastern Europe, Africa, and the Far East. NofaLab became part of the Cotecna Inspection S.A. business family in 2017.

Wim Broer, NofaLab science and development manager, explains how advances in atmospheric pressure gas chromatography (APGC) has helped the company meet the growing needs of its customers.



NofaLab Laboratory, Schiedam, The Netherlands.

WORKING WITH WATERS

Since 2011 NofaLab has employed the Waters® Xevo® TQ-S in combination with APGC. In 2015, the company also began using the Waters Xevo TQ-S micro. The increased sensitivity of the Xevo TQ-S instruments enables NofaLab scientists to quantify and confirm trace components at even lower levels in the most complex samples.

Many analyses cross the boundary between liquid chromatography (LC) and gas chromatography (GC). The Waters Xevo TQ-S instruments enable complete screening of samples using both LC and GC separations, making these instruments valuable for scientists who need to identify, quantify, and confirm the broadest range of compounds in the most complex and challenging samples.



Wim Broer, science and development manager, NofaLab.

GROWING DEMAND FOR RAPID CONTAMINANT DETECTION AT NOFALAB

Recent news reports and legislative activity regarding contaminants in food and feed have spurred greater interest in contaminant detection services. NofaLab has quickly adapted to these market trends, and its locations at key ports have positioned the company to rapidly respond to the growing demand.



“The inspections for contaminants such as dioxins, pesticides, and heavy metals are a large and important part of our work – along with quantitative analyses, of course, such as saponifiability, peroxides and free fatty acids. We also check on the cleaning of container ships and logistics. This is part of our total package.”

MARCEL BRUGGEMAN

Director of NofaLab

NofaLab scientists saw the need to develop methods to identify a broader array of food or environmental contaminants with total confidence. Speed mattered. Promptly responding to new demand from the market is part of NofaLab's growth strategy. Once the samples are in the lab, the company reports results within 24 hours – or in emergency situations, even within a few hours. The company also offers support for its customers 24 hours a day, 7 days a week.

Recent technology advances are a game changer for the company. In 2009 the company analyzed 2–3 samples/day for approximately 60 pesticides. Today, they typically analyze 40–50 samples/day – and for as many as 800 pesticides.

Wim Broer, NofaLab science and development manager explains: *“The pressure for our customers is getting bigger and bigger. Five years ago, this type of analysis could take two or even three weeks. Nowadays, by optimizing the logistics and robust fast methods, we deliver these analytical results within a day.”*

And Broer sees demand continuing to grow, requiring NofaLab to keep developing new and innovative analytical methods that enable the company to meet their customers' changing needs.

IMPROVED SELECTIVITY OF ATMOSPHERIC PRESSURE GAS CHROMATOGRAPHY (APGC)

In 2011 Broer was one of the first in the field to explore the possibility of using APGC as a method for pesticide analysis with the Waters Xevo TQ-S. He found the increased sensitivity of the Xevo TQ-S instrument enabled NofaLab scientists to quantify and confirm trace components at even lower levels in the most complex samples.

APGC proved to be a robust and sensitive technique that made it easier to increase throughput while providing reliable results. NofaLab began using APGC for analyzing a broad range of pesticides in matrices like edible oil, fatty acids, herb, spices, and others.

Typically, multiresidue pesticide analysis targets several hundred pesticides in a single sample. Due to their varying physicochemical characteristics, these analytes can require different chromatography and ionization modes, including liquid and/or gas chromatography. APGC is a soft ionization process similar to chemical ionization (CI).

Routine testing laboratories – like NofaLab – strive for efficient and reliable sample throughput methodologies, where generic analytical conditions are essential. Due to certain limitations with respect to duty cycles associated with quadrupole technology, restrictions in the number of multiple reaction monitoring (MRM) transitions can exist when using *“gold standard”* tandem quadrupole mass spectrometry techniques.

Recent advancements in technology, however, are overcoming this limitation. This allows for an increasing number of MRMs achievable per injection, while providing the continued sensitivity and selectivity required for confirmation and quantification in routine multi-residue analyses.

Furthermore, the analysis of pesticides by APGC technology allows for improved selectivity when generating MRM transitions in comparison with significant fragmentation experienced with electron impact (EI) gas chromatography. Operating the gas chromatography system at atmospheric pressure provides increased scope for ionization modes – namely charge and proton transfer.

That's valuable for companies like NofaLab. Additionally, Broer explains another practical benefit of the APGC method and the Xevo TQ-S: *“The instrument is very sensitive, so you have the opportunity to inject less sample. That makes the GC stay clean longer and helps reduce the cost of materials.”*

PUTTING THE TECHNOLOGY TO THE TEST

Broer used APGC to develop a method that would increase the number of pesticides detected in the variety of samples typical of NofaLab's customers.

BACKGROUND AND CHALLENGE

Since 2011 NofaLab has employed the Xevo TQ-S in combination with APGC to successfully develop a commodity independent method for the determination of pesticides, originally validating 279 pesticides.

Broer wanted to find one robust analytical method that combines gas chromatography (GC) with mass spectrometry (MS) for the analysis of more than 500 pesticides in all the commodities NofaLab typically analyzes for its customers, which include:

- Fruit and vegetables
- Seeds
- Vegetable oil, fat, and fatty acids
- Grain
- Herbs and spices
- Meat and seafood
- Animal oil, fat, and fatty acids
- Eggs and eggs products
- Milk and milk products
- Animal food

The study's goal was to achieve:

- LOD less than 5 µg/kg
- RSDR less than 20%
- Recovery between 70 and 120%

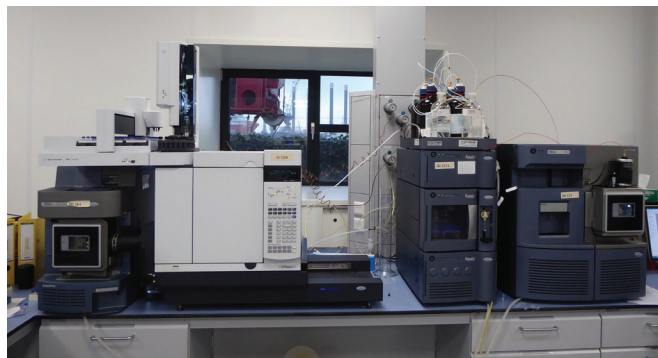
If successful, the new method could potentially result in huge time and cost savings for NofaLab and its customers.

Experimental

This experiment attempted to validate 498 pesticides according to the optimized conditions of the Xevo TQ-S micro. Sample preparation is key.

Broer explains: *"We work with samples from 1 kilo to sometimes 20 kilograms. For the pesticide analysis, you only need one gram of it, but that one gram must say everything about the kilos we have received – kilos which, in turn, say everything about the whole load."*

A QuEChERS extraction was performed on ten different commodities on five different days. All pesticides were spiked with maximum residue level (MRL) (in general 10 µg/kg) and 150 µg/kg. For the study, Broer and team used 1 gram of sample based on solid material (0.5 gram for oil and fat) and performed a standard QuEChERS extraction. The level of water in the extraction solution was set to 10 mL.



Waters Xevo TQ-S system in the NofaLab laboratory.

The samples were put in a freezer until the water was frozen. After the clean-up, the samples were further cleaned by chemical filtration over a PSA elution column. The amount of sample on-column was 0.5 mg. The pesticide MRL-level is 5 µg.

Outcome

According to study results, 460 pesticides were validated – achieving a LOD less than 5 µg/kg, a RSDR less than 20%, and recovery between 70 and 120%.

An additional 38 pesticides failed validation due to low recoveries. Those pesticides – including nitro- and chlorophenols, fenhexamid, acephate, captan, and folpet – showed too much interaction with the PSA elution column.

Overall, 92% of the studied pesticides are commodity independent, and 94% when herbs and spices are excluded – a beneficial result for NofaLab because of the variety of commodities the company typically analyzes for its customers.



"We put a lot of effort into matrix-independent extraction. If that is possible, you can also analyze matrix-independent. The fewer variations you have in the extractions, the easier it becomes in the analysis. Meanwhile, we have such a robust method that it does not matter whether it is a fatty acid or ginger powder or sunflower oil."

WIM BROER

NofaLab science and development manager

IMPROVED SENSITIVITY FOR PESTICIDE DETECTION

The study found APGC in combination with the Xevo TQ-S or TQ-S micro instruments is a robust and sensitive technique for analyzing a broad range of pesticides in several matrices, including edible oil, fatty acids, herb, spices, and other commodities.

As a result, the study concluded:

- This sensitivity provides the opportunity to develop a commodity independent extraction (increasing the L/S during the extraction).
- Less injection of absolute matrix results in a longer stable and reliable chromatographic separation.
- Less absolute matrix shows less influence of the ionization process in the source.
- The source is in fact maintenance-free.

Improved pesticide detection: The increase in the number of pesticides detected could potentially help NofaLab expand its services to customers. The Xevo TQ-S micro showed equivalent results for the standard 300 pesticides when compared with the Xevo TQ-S. Additionally, the Xevo TQ-S micro enabled the extension of the number of analytes to 500 in one run.

As Broer summarizes concisely: *"It's quite a lot."*

NofaLab researchers believe there is room for even more analytes in one run (up to 1,000). Broer believes this space could also be used to shorten the chromatographic run.



"The system was very sensitive – much more sensitive than we were used to. That means you gain a lot of sensitivity for a lot of compounds."

WIM BROER

NofaLab science and development manager

Ionization: One advantage of APGC is the ionization is "soft," meaning that less fragmentation is observed for many compounds when compared with techniques such as electron impact (EI).

Reduced fragmentation can give higher sensitivity and specificity, therefore simplifying pre-cursor ion selection in MS/MS analyses.

That's a big advantage for NofaLab scientists, who needs to analyze a broad range of pesticides in variety of matrices.

Easy LC to GC changeover: A big advantage of the Xevo instruments is the quick and simple changeover from LC to GC with one APGC method. Since APGC is not a vacuum technique equilibration time between techniques is kept to a minimum.

This means that the analysis can be tailored to business demands, maximizing up-time and instrument utilization. It also gives researchers the widest possible coverage from analyses.

With the increased throughput, the time savings when switching from LC to GC adds up quickly. Broer explains: *"Within a very short time period, we were up and running."*

The study results suggest continued APGC method development holds significant benefits for NofaLab's business expansion.

Broer summarizes the impact of this innovative APGC method with the Waters Xevo TQ-S instruments: *"We've expanded the number of pesticides we can analyze and the number of clients we can serve. We're getting good qualitative and quantitative detection of pesticides."*

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