

Queen's University Belfast

Bringing speed and reliability to food safety testing

In the effort to ensure the safety of food, testing laboratories face a non-negotiable outcome: certainty. But when the line between what's safe and what's dangerous is defined in terms of parts-per-billion of a targeted (natural) contaminant, achieving certainty can be a challenge.

Every sample that enters a food safety testing lab requires the full expertise of the analysts in order to reach testing limits like these. But what happens when more than a few, or a few dozen, or even a few hundred samples show up? What if it's 30,000 tons of perishable grain that has just arrived on a cargo ship, all of which has to be sampled, tested, verified as safe, and released as soon as possible for downstream food production?

The Institute for Global Food Security (IGFS) team at Queen's University, Belfast is all too familiar with this stress-inducing threesome of certainty, urgency, and massive scale. It's their job to help develop the sampling and testing methods needed to quickly and consistently assess mycotoxin levels in large grain shipments. Mycotoxins, including aflatoxins and ochratoxin, are naturally occurring byproducts of certain fungi that can grow on grain stored under less-than-ideal conditions, posing a potentially serious health threat to humans and livestock.

Making large-scale analysis manageable

"You might say we do testing in a big way," Dr. Nick Birse, an IGFS researcher said. "We're very much real-world oriented here, because our work has quantifiable impact on the agricultural sector in Northern Ireland. We're different from certain commercially oriented testing labs, where throughput equates to profit; this is not a massive money-making enterprise for us. We're part of a collaborative partnership called Food Fortress, set up in part by Queen's University. The methodologies we develop are driven by a social responsibility to support the local agri-food sector and promote public health."

To make it all work, Dr. Brett Greer, who led the development and implementation of the testing method, relies on analytical instrumentation that delivers speed and reliability—qualities he's come to associate with Agilent LC/MS platforms such as the 6495D triple quadrupole LC/MS and the intelligent reflex data-dependent sample reinjection feature.



Dr. Nick Birse and Dr. Brett Greer

The Institute for Global Food Security (IGFS) team
Queen's University
Belfast

Dr. Birse added, "If, say, 10,000 tons of grain are being unloaded on a conveyor belt, we'll take half-kilogram samples every few minutes. Obviously, that's a lot of material to test, so we simplify the process by combining three or four samples together. If that composite batch passes, we don't worry about those samples; if it doesn't, we look at them individually. The sensitivity of the 6495D adds a lot of confidence to that approach."

The perishable nature of the tested material places significant pressure on the speed of the analysis; the longer these materials remain in storage, the more vulnerable they are to the kind of fungal contamination their testing seeks to uncover. Dr. Greer developed the method in order to test materials quickly enough so that any issues can be flagged up by the time the material has arrived at its processing destination.

"With intelligent reflex, Dr. Qiqi He, who is responsible for routine analysis, can do a really quick screen using a rapid method, and if the data show a problem with one of the samples, it can be automatically routed through the conventional longer method," Dr. Birse explained. "We use a 3 cm column for screening, and it gives us such good baseline separation and sharp peaks that we almost don't need the 10 cm column, at least for certain analytes."

Using a shorter column means faster run times and less solvent use, leading to lower analysis costs and faster turnaround times. Dr. Birse put these advantages in perspective.

"Running samples on the screening column takes maybe four minutes, compared to 14 minutes for the longer column, so being able to send fewer samples through the full method is significant," he said. "It isn't unusual for maybe only 7–10% of a batch of samples to be over the limit and require further analysis. The time and solvent the team save by using the prescreening approach definitely add up. The instrument probably uses somewhere between 30% and 80% less solvent, even if an unusually large number of samples need further testing."

Achieving greater efficiency through automated analysis

While saving time and resources on a per-sample basis adds significant value, intelligent reflex also allows users to automate the review of screening results and deciding which samples need to be reinjected for further analysis," Dr. Birse said. "If they're making those decisions based on a calibration curve, they're going to come to the same conclusions as intelligent reflex. The time savings associated with automating that evaluation and doing everything in an unattended way without compromising the results has had a big impact. We set things up at the end of the day for overnight sample runs, with intelligent reflex evaluating the results and managing any reinjections that are needed. The system also lets us automate routine calibration and equilibration tasks overnight, so when the analyst arrives in the morning, the instrument is already in the sweet spot and ready for a full day of method development tasks. When we were manually evaluating our results, several hours of every day went toward looking over the previous night's results, reinjecting samples, and re-optimizing the system. It's almost like getting two instruments for the price of one."

*"When you put the 6495D and intelligent reflex together, it's an easy package to recommend," he added.
"The calibrations are reliable, the instrument is stable, and the results are consistently excellent. It's good technology that works."*

There's also a bigger-picture aspect to testing agricultural products on such a large scale. The data Dr. Greer and his team generate with this methodology are providing unique insights into mycotoxin risk and mitigation.

"The team's approach is giving us a broader understanding of mycotoxin prevalence," Dr. Birse said. "It's valuable lens for looking at correlations between mycotoxin levels and things like weather patterns, farming practices across regions, and import routes. We're getting a much better understanding of where things come from, what the risks are, and how we can address them. We hope that will help enable a shift toward a more proactive approach to food safety."

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