APGC Advances Ultra-Trace Toxins Analysis in Feed and Food

Waters Atmospheric Pressure Gas Chromatography (APGC) source coupled with Mass Spectrometry (MS) improves the detection and quantification of dioxins in feed and food samples at the Institute of Quality Standard and Testing Technology for Agro Products (Beijing), China

Technology: Atmospheric Pressure Gas Chromatography (APGC)

FEED AND FOOD SAFETY AT IQSTAP (BEIJING)

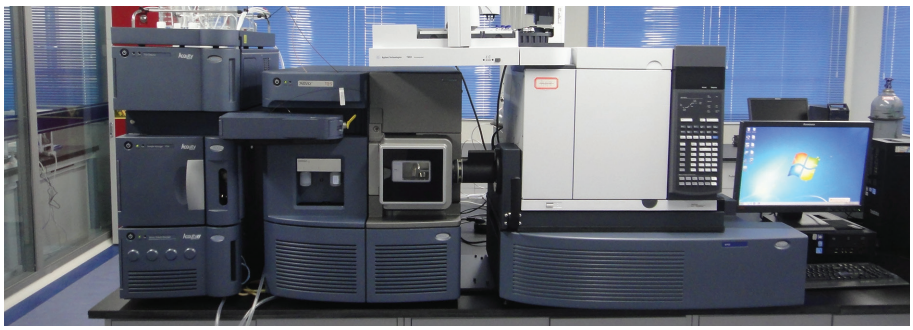
The Institute of Quality Standard and Testing Technology for Agro Products (IQSTAP) in Beijing, China, is dedicated to achieving further improvements of feed safety in China. It's a responsibility with worldwide implications, as China is a leading importer, producer, and consumer of animal feeds and feed ingredients, affecting food quality and safety around the globe.

IQSTAP focuses on feed resource development and utilization, feed and animal product safety, ecological environment safety, quality and safety of animal products. Researchers investigate all facets of the feed industry, but mainly focus on feed biotechnology, biochemical engineering and extraction, animal nutrition and feed science, feed processing technology, feed testing and safety evaluation, and feed economy and information.

In IQSTAP's Dioxin Testing Laboratory, eight scientists focus on the analysis of persistent organic pollutants such as dioxins in feed and agricultural products. The laboratory is part of the Institute of Agricultural Quality Standards and Testing Technology at the Chinese Academy of Agricultural Sciences, and it supports Chinese government agencies and provides third-party services to the private sector.

Researchers at the Dioxin Testing Laboratory analyze feed and agricultural products, as well as environmental samples that can affect these products. The laboratory officially obtained China National Accreditation Service for Conformity Assessment (CNAS) accreditation in 2014 and began its work to perform – and improve – dioxin analysis in feed and food.

IQSTAP's accumulation of scientific research since that time has supported the advancement of technology in feed and food inspection analysis.



Waters instruments at the Institute of Quality Standard and Testing Technology for Agro Products in Beijing, China.

WORKING WITH WATERS

Dr. Xiaomin Li, Assistant Professor, and the IQSTAP Dioxin Testing Laboratory have a long-standing relationship with Waters™ that dates from 2007. IQSTAP laboratories are equipped with Waters software and instrumentation for HRGC/HRMS, GC-MS/MS, UPLC-MS/MS, and now APGC-MS/MS.

Dr. Li explains: *"Waters software and instrumentation are extremely reliable, and Waters engineers are very dedicated. We work closely with Waters personnel, and they are very supportive of our mission. Our team feels that Waters is very trustworthy."*

IQSTAP's Dioxin Testing Laboratory using Waters APGC Technology.

Most of IQSTAP's work uses what's considered the international gold standard in this field – high-resolution gas chromatography (GC) coupled with mass spectrometry (MS).

Working with Waters,™ IQSTAP has found the use of Atmospheric Pressure Gas Chromatography (APGC) enables its scientists to improve the detection and quantification of toxins in feed and food samples – a technological advancement that serves their mission to advance dioxin analysis. IQSTAP is now poised to share these developments with the greater scientific community.

Dr. Xiaomin Li, IQSTAP Assistant Professor, explains: *"APGC is a very sensitive detection system for the accurate determination of dioxins and furans at regulatory levels. We work closely with other scientific researchers and testing organizations. Often personnel from other laboratories visit us to study our methodology, and we introduce them to the methods and experiences that we have found work better."*

GLOBAL IMPACT OF DIOXINS IN FEED AND FOOD

Polychlorinated dibenzo-*p*-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) are toxic organic pollutants that take a long time to break down once they are present in the environment. These toxins are found throughout the world, and they accumulate in the food chain, mainly in the fatty tissue of animals.

Monitoring the presence of these contaminants is vitally important for food safety, particularly because PCDDs and PCDFs are highly toxic and can cause cancer, reproductive and developmental problems, damage to the immune system, and hormone interference.

These toxins are not commercially produced, but instead are formed when organic compounds are incinerated in the presence of chlorine. They occur in a wide range of industrial activities such as chemical manufacturing, production of iron and steel, paper and pulp bleaching, waste incineration, leaded gasoline combustion, and many others.

Their presence in the global food chain has led to increased interest in detecting and quantifying these contaminants in feed and food supplies. Most human exposure to dioxins is through food, mainly meat and dairy products, fish and shellfish. Due to the bioaccumulative nature of these compounds, it is essential to monitor them at ultra-trace levels in food and environmental samples.

China implemented a revised regulatory and registration system for imported feed and feed additives in 2015. The World Health Organization (WHO) has conducted human-based risk assessments, setting toxic equivalent factors for these compounds.

Dioxins are restricted internationally under the Stockholm Convention. Additionally, the European Union has limited or prohibited the use of many of these compounds and has set standards on acceptable levels in feed and food supplies.

These regulations are continually reviewed and updated as new research becomes available. The established presence of PCDDs and PCDFs in the food chain makes ongoing monitoring of these contaminants in feed and food essential to ensure that the levels do not exceed the allowable concentration, which has sparked scientific interest in improving dioxin analysis methods.

IQSTAP's Dioxin Testing Laboratory is dedicated to improving its methodology for analyzing the most toxic PCDDs and PCDFs. It's a mission that faces several complications – including the need for sensitivity and accuracy, as well as matrix interference.

> "The uniqueness of dioxin analysis means that each dioxin laboratory is faced with the complexity of the sample matrix, the effective purification of the sample, and the quasi-deterministic quantitative problem of the target compound."
>
> DR. XIAOMIN LI
> *IQSTAP Assistant Professor*

Scientists at the Dioxin Testing Laboratory are continually working to advance the analysis of dioxins by developing new methods for detection and quantification of contaminants in feed and food samples. IQSTAP uses the international gold standard method for detecting PCDDs and PCDFs – Isotope dilution high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS) method. The European Commission has established that GC-MS/MS may be used as a confirmatory method for PCDD and PCDF detection.

Working closely with Waters, IQSTAP's Dioxin Testing Laboratory evaluated the potential use of APGC as a new analytical technology to advance the detection and quantification of PCDDs and PCDFs in feed and food samples.

IMPROVED SENSITIVITY AND SELECTIVITY OF APGC

Tightening regulations on contaminants in feed and food have sparked the need for greater sensitivity from analytical techniques like GC-MS. Additionally, reducing the injected volume of samples has been shown to minimize matrix effects and the contamination on instrumentation – two factors that directly affect dioxin analysis.

Working with Waters to identify new technology to improve the detection and quantification of contaminants in feed and food samples, the IQSTAP Dioxin Testing Laboratory scientists developed a GC-MS/MS with APGC technique to analyze 17 of the most toxic PCDDs and PCDFs. They found APGC-MS/MS was a very sensitive detection system for the accurate determination of dioxins and furans at regulatory levels.

Dr. Li explains: *"The unique source design of Waters APGC instrumentation is particularly suitable for the analysis of ultra-trace organic substances such as dioxins where laboratories need increased sensitivity and rapid results. It provides an alternative approach to classical HRGC/HRMS methods for the analysis of PCDDs and PCDFs."*

The IQSTAP team found several significant advantages to APGC when compared with traditional dioxin analysis methodology.

Improved ability to measure complex matrices: With its ability to detect contaminant limits at ultra-trace levels, APGC enabled IQSTAP scientists to achieve compliance with regulatory limits on the presence and quantity of toxic PCDDs and PCDFs. The increased sensitivity of APGC coupled with MS, enabled the Dioxin Testing Laboratory researchers to quantify and confirm trace contaminants at even lower levels in the most complex samples. Additionally, because they could inject less sample matrix, the APGC technique reduced the effects of contamination on the instrumentation – and therefore increased uptime.

Dr. Xiaomin Li working in IQSTAP's Dioxin Testing Laboratory, Beijing.

Furthermore, the APGC technique reduced the cost of tests for contaminants because of its improved sensitivity and selectivity. IQSTAP found APGC used only half the materials needed with the previously used techniques and would analyze them at a lower concentration. The ability to eliminate the matrix effect also reduced the need for time-consuming purification steps – helping the Dioxin Testing Laboratory to provide faster results for time-sensitive analyses.

Soft ionization: Traditionally, the determination of persistent organic pollutants used electron ionization (EI) for fragmenting the molecules and generating charged ions for detection. But some of the compounds of interest for IQSTAP scientists were not suitable for "hard" ionization analysis such as EI.

That's because the EI technique sometimes gave rise to identical ions for different compounds, so its selectivity is limited. At the same time, the EI technique can make it difficult to see the whole molecule, and it can result in the same fragment for compounds with different numbers of bromine atoms. That presented the team with the challenge to overcome the limitations of EI for feed and food safety analysis.

However, atmospheric pressure chemical ionization (APCI) technology results in a "soft" ionization process where the molecule is ionized by either proton transfer or charge transfer, rather than by direct electron bombardment. This soft ionization with APGC was extremely beneficial for the IQSTAP team's work because of the reduced fragmentation for many compounds when compared with techniques such as EI.

Reduced fragmentation can give higher sensitivity and specificity, therefore simplifying pre-cursor ion selection in MS/MS analyses. With the APCI interface, a soft and reproducible ionization is favored in GC, typically the protonated molecule and/or the molecular ion is the base peak of the spectrum in most cases.

The IQSTAP scientists also found the analysis of feed and food samples by APGC allows for improved selectivity when generating multiple reaction monitoring (MRM) transitions in comparison to the significant fragmentation experienced with EI gas chromatography. Operating the GC system at atmospheric pressure provides increased scope for ionization mode optimization – namely charge and proton transfer.

That makes APGC an attractive alternative because it opens new possibilities in both quantitative analysis at trace levels and universal screening – both of which require the ability to detect a broad range of contaminants in a variety of matrices.

"The APGC soft ionization method is suitable for the analysis of many easily degradable compounds. That's particularly helpful with dioxin analysis, where EI has significant limitations."

DR. XIAOMIN LI
IQSTAP Assistant Professor

Easy LC to GC changeover: Many analyses cross the boundary between LC and GC. Waters APGC technology offered IQSTAP scientists a 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 the analysis can be tailored to demands, maximizing up-time and instrument utilization. It also gives researchers the widest possible coverage from analyses.

This capability offers significant benefits to laboratories like IQSTAP's Dioxin Testing Laboratory, which use multiple techniques.

Dr. Li explains: *"Its mutual conversion capability with UPLC-MS/MS was a big part of our decision to incorporate APGC in our laboratory. It offers us significant advantages, and it's easy to do."*

Ease of use: The easy-to-use interface of the Waters APGC instrumentation reduces the need for training, as well as speeds up the implementation process in the laboratory – two significant benefits in the dioxin analysis field.

That makes APGC fit easily into the workflows of IQSTAP's Dioxin Testing Laboratory and speeds up productivity.

Dr. Li explains: *"APGC offers us the advantage of being very easy to operate. APGC compared to High Resolution Gas Chromatography (HRGC)/High Resolution Mass Spectrometry (HRMS), is like a photographic card camera vs a Canon high end SLR camera 5D IV."*

ADVANCING DIOXIN ANALYSIS

IQSTAP's Dioxin Testing Laboratory researchers have found APGC is a robust and sensitive technique for analyzing dioxins in feed and food samples, and they see the potential for expanded use in the field. IQSTAP believes incorporating this innovative technique holds great advances for food security both to detect pollutants and monitor their levels to ensure they meet the specifications regulated by the European Union – as well as future standards that develop over time in other countries around the world.

The scientists at IQSTAP plan to continue their work with Waters to improve the analysis of other persistent organic pollutants using APGC by researching the capabilities of APGC and its possibilities for their work.

"APGC has provided us with new ideas for scientific research. It offers an alternative to traditional GC-MS for dioxin analysis, providing the speed and sensitivity we need."

DR. XIAOMIN LI
IQSTAP Assistant Professor

Waters
THE SCIENCE OF WHAT'S POSSIBLE.™

Waters Corporation
34 Maple Street
Milford, MA 01757 U.S.A.
T: 1 508 478 2000
F: 1 508 872 1990
www.waters.com