

## SGS AXYS and Waters Validate APGC as New Methodology for Dioxin and Environmental Contaminants Analysis

Waters APGC coupled with the Xevo TQ-XS Triple Quadrupole Mass Spectrometer offers unique advantages for dioxin and environmental contaminant analysis as the U.S. EPA updates its guidelines

### Atmospheric Pressure Gas Chromatography (APGC), Xevo TQ-XS Triple Quadrupole Mass Spectrometer

#### POPS AND CEC ANALYTICAL SERVICES AT SGS AXYS

For more than 40 years, SGS AXYS has specialized in the measurement of known or suspected persistent organic pollutants (POPs) and contaminants of emerging concern (CECs) in environmental, biota and product samples. The company offers state-of-the-art mass spectrometry (MS) analysis for more than a thousand compounds of interest to its clients in both the public and private sectors.

Located in Sidney, British Columbia, Canada, the company's expertise extends from a wide variety of fully commercialized tests for organochlorine legacy compounds like dioxins and polychlorinated biphenyls (PCBs), to analysis of emerging and pre-regulatory contaminants. SGS AXYS's isotope dilution techniques and quality programs ensure data of industry-leading quality, detection limits, and defensibility. The analytical services company holds multiple analysis and matrix accreditations with American (NELAC) and Canadian (CALA) national bodies.

Dr. Bharat Chandramouli, Environment, Health and Safety Product Manager and Senior Scientist at SGS AXYS, describes the company's services: *"We work in the more specialized and challenging technical markets. Other labs conduct routine dioxins analysis for regulatory purposes, but we've always kept one step ahead by specializing in more challenging work where low backgrounds are needed, cleanups are more difficult, and sample handling is very challenging."*



Mr. Xinhui Xie carried out the instrumental development on the project.

#### WORKING WITH WATERS

Waters™ and SGS AXYS have a history of working closely together on other projects, and the analytical services laboratory has invested significantly in Waters instrumentation and software over the years. As SGS AXYS began looking for new technology to address U.S. EPA requests for dioxin analysis options, Waters was a natural partner.

SGS AXYS personnel arranged to complete in-house testing of the Waters™ APGC™ technology coupled with the Waters™ Xevo™ TQ-XS Triple Quadrupole Mass Spectrometer for a year to test the instrumentation's production capabilities, before the instrument was purchased in September 2019. The instrumental development was carried out by SGS AXYS Senior Instrument Chemist Mr. Xinhui Xie and the entire initiative was managed by senior Scientist Dr. Coreen Hamilton.



**“We have worked with Waters’ instruments for years, so the chance to try out their APGC tandem mass spectrometry (MS/MS) instrumentation was an excellent collaborative opportunity for us.”**

MS. DEBORAH CLARK

*Operations Manager at SGS AXYS*

SGS AXYS is consistently first to market amongst North American commercial environmental laboratories in analysis of emerging contaminants (e.g., full congener PCBs in 1986; dioxins in 1987 polybrominated diphenyl ethers (PBDEs) in 1995; and per- and polyfluoroalkyl substances (PFAS) in 2002). The company continually develops quantitative approaches for contaminants of emerging concern under full ISO 17025 compliance.

Using Waters Atmospheric Pressure Gas Chromatography (APGC) technology coupled with the Waters Xevo TQ-XS Triple Quadrupole Mass Spectrometer, scientists at SGS AXYS worked closely with Waters personnel to develop and validate an alternate procedure for polychlorinated dibenzo dioxins and furans by EPA Method 1613B using GC-MS/MS. This procedure was developed at the request of the US EPA to bring alternatives to magnetic sector mass spectrometry for dioxin analysis that met all quality control criteria and included all the performance checks, interference checks and qualitative identification criteria from EPA Method 1613B.

### INCREASING ANALYTICAL OPTIONS FOR THE U.S. EPA

Current U.S. EPA guidance recognizes high-resolution gas chromatography (HRGC) and high-resolution mass spectrometry (HRMS) as the ‘gold standard’ for dioxin and furan analysis in aqueous, solid and tissue matrices under the EPA Method 1613B.<sup>1</sup>

However, despite the benefits of high-resolution mass spectrometry for dioxin analysis, the magnetic sector instruments traditionally used for this technique come with some business challenges for analytical services companies – including the need for a high degree of expertise to operate them and significant costs, which limit their widespread use.

As more advanced technology has emerged, the U.S. EPA began to explore alternatives appropriate for dioxin analysis by working with industry leaders like SGS AXYS to identify promising new methods. It’s a role SGS AXYS is comfortable with, having helped to establish other analytical methods that were eventually adopted as part of regulatory agency guidelines, including the U.S. EPA.

Dr. Chandramouli describes SGS AXYS’s perspective on these regulatory guideline changes: *“We saw the opportunity to shape a market and get early adoption in place for these U.S. EPA changes. One of the strategies we’ve taken to mitigate risk on emerging contaminants and techniques is to go in with a regulatory partner to provide the official stamp of approval.”*

Ms. Clark explains the company’s long-term strategy in working with the U.S. EPA on new methodology for dioxin analysis: *“SGS AXYS has consistently worked to advance technology for service of clients. We’ve seen for the last five years or so that an alternative to magnetic sector high-resolution instruments would be desirable.*

SGS AXYS looked at several different analytical techniques before shortlisting APGC-MS/MS. APGC uses an atmospheric pressure chemical ionization (APCI) ionization source for Waters mass spectrometers. The ionization is ‘soft’ – meaning that less fragmentation is observed for many compounds when compared with techniques such as electron ionization (EI). Reduced fragmentation can offer higher sensitivity and specificity, therefore simplifying precursor ion selection in MS/MS analyses.



**“From my perspective on the technical side, we kept our options extremely wide open during our initial search. We didn’t want to throw anything out that had a possibility of working. We needed the same level of sensitivity and defensibility, however. Those were our primary goals.”**

DR. BHARAT CHANDRAMOULI

*Environment, Health and Safety Product Manager and Senior Scientist at SGS AXYS*

The Waters Xevo TQ-XS Triple Quadrupole Mass Spectrometer with APGC was already enabling laboratories in food, feed, and environmental industries to make increasingly sensitive measurements from complex samples, informing contamination incidences and allowing updated recommendations to be made on regulatory limits.

APGC has gradually gained popularity in Europe and is now increasingly considered an equivalent method to the gold standard for dioxin analysis. In 2014, the European Union (EU) passed legislation recognizing the use of triple quadrupole GC-MS/MS, with either EI or APCI, as a confirmatory tool for checking compliance with maximum levels of dioxins in food and feed (589/2014/EU and 709/2014).<sup>2</sup> This was the result of an extensive validation study by an EU working group, and it was the first official regulatory method that began the switch from magnetic sector technology and included APCI. The regulation also began the allowance of the use of MS/MS instead of magnetic sector for confirmatory dioxin analysis.

SGS AXYS scientists believed APGC-MS/MS was an attractive alternative for EPA Method 1613B because of its ability to provide high performance, ease-of-use allowing quick reaction to meet demands and increase throughput, access to ultra-trace quantification, comprehensive MS and MS/MS, and high-resolution separations. The company has many more operators that are familiar with tandem quad MS/MS than magnetic sector, and access to a larger pool of potential candidates when expanding its headcount.



**“We were looking for the next generation of technology that could meet the demands of EPA Method 1613B. We evaluated more than one instrument and more than one platform. Waters APGC technology seemed to be a good option.”**

MS. DEBORAH CLARK  
*Operations Manager at SGS AXYS*

### APGC-MS/MS ANALYSIS FOR DIOXINS

Compared with magnetic sector instruments, APGC-MS/MS shows promise as a more user- and budget-friendly technology that is likely to make dioxin analysis more accessible to testing laboratories across the world, therefore helping to secure global and environmental food safety. Additionally, the Waters Xevo TQ-XS instrument is equipped with unique StepWave technology, enabling detection limits at ultra-trace levels. This allows compliance with regulatory limits and the ability to inject less sample matrix, reducing effects of contamination on the GC-MS system and therefore increasing uptime.

Ms. Clark comments on the implementation of the APGC Xevo TQ-XS at SGS AXYS: *“The whole process was collaborative and involved teamwork – everything from generating data and learning about what the instrument could do, to having Waters people here on site and available via email and conference calls.”*

### WORKING WITH WATERS

Working closely with Waters, SGS AXYS scientists tested the capabilities of the APGC-MS/MS technology with the Waters Xevo TQ-XS Triple Quadrupole Mass Spectrometer.

The project’s goal was to develop and validate an alternative procedure for dioxin analysis using MS/MS rather than HRMS. SGS AXYS and Waters personnel worked together to adapt U.S. EPA Method 1613B protocols and criteria to MS/MS – showing equivalency of results in terms of sensitivity, linearity, selectivity, accuracy, and precision.

Dr. Chandramouli explains: *“It’s the first new approach for regulatory dioxin analysis in North America in 25 years. So, this is a big deal. Europe, of course, has been doing MS/MS work on dioxins for quite a while. But not North America, except for a few academic labs here and there.”*

## BEYOND DIOXIN ANALYSIS

With the APGC-MS/MS method validated to meet all the requirements of the U.S. EPA Method 1613B, SGS AXYS is in the process of validating an EI-GC-MS/MS option for the EPA ahead of the ATP submission, and finalized its purchase of the APGC Xevo TQ-XS. SGS AXYS operations scientists have now shifted their focus from testing the instrumentation to evaluating the breadth of its possibilities in a commercial production environment.



**"The opportunity to work on a newer platform, to really see what we can do with the new technology, has been very exciting. Our operations staff is already requesting a second instrument so we can have one for routine analysis, and one for more development work. They are always asking 'What can we do next?' So, it has been great for the team to be able to experiment with this technology."**

**MS. DEBORAH CLARK**

*Operations Manager at SGS AXYS*

The operations team at SGS AXYS has regular performance evaluation cycles and with the company's new accreditation for dioxins, PCBs, and pesticides on the APGC instrument against multiple quality standards, the company is looking to expand APGC-MS/MS product development, sales and marketing efforts.

*"We are currently in a busy production period, but we have further development plans in 2020 on the instrument to develop and validate methods that are still being run on magnetic sector systems,"* comments Ms. Clark.

## BUSINESS BENEFITS

In addition to the technical capabilities of the Waters APGC system, SGS AXYS's long-standing relationship with Waters was a factor in the analytical services company's decision to buy the new APGC-MS/MS instrumentation.

Ms. Clark explains: *"We have a lot of experience with other Waters instrumentation and software, which informed part of the decision to invest in this APGC technology."*

The timescale from instrument purchase to full-time production is expected to take a couple of years. The initial aim was to validate the APGC-MS/MS method for dioxin analysis internally and with Waters and obtain accreditation. Dr. Chandramouli explains: *"Having that U.S. EPA method in place and published confirming this technique as an equivalent method to HRMS is what will give our clients the confidence to switch, and to use the method beyond pilot tests."*

The team in British Columbia submits proposals for new instrumentation in the capital budget each year, for approval by SGS Global, headquartered in Geneva. Together with the management team at SGS AXYS, Ms. Clark compiles a justification for large instrument purchases for submission to the Geneva team.

*"SGS Global views the AXYS lab as a Centre of Excellence, so they trust that they will see the return on an instrument purchase when we say they will,"* comments Ms. Clark.

The justification to SGS Global for purchasing the APGC Xevo TQ-XS Triple Quadrupole Mass Spectrometer was centered on long-term development and implementation, rather than short-term production goals, as Dr. Chandramouli explains: *"This is going to take time, and everyone knows that. If you put together a good justification, they will spend it because they trust that we will deliver what we claim we will."*

Dioxin analysis was the initial priority for SGS AXYS when implementing APGC-MS/MS, but the company has since evaluated the use of this technology for PCBs and chlorinated pesticides, with considerable success and have obtained accreditation for both these methods in Canada and the US. Ms. Clark comments: *"We have had a lot of success with these other contaminants. This was an expensive instrument, so we need to be able to keep it busy all the time."*

*"If the instrument had only worked for dioxins, we would have had to take a much harder look at what system we chose."*

Dr. Chandramouli adds.

## THE FUTURE OF CONTAMINANT ANALYSIS

Now SGS AXYS is looking ahead to how their investment in Waters APGC and the Xevo TQ-XS Triple Quadrupole Mass Spectrometer can provide future business benefits for the company and its customers, including developing and validating additional methods for environmental contaminants analysis.

Staying abreast of technology advances is essential for SGS AXYS to meet their customers' changing needs. The company considers their analytical services – and the instrumentation and software needed to perform them – as an investment that should continually return value to the company, just like any other type of investment.

Dr. Chandramouli describes the company's strategy: *"We have already moved beyond dioxins and validated and accredited HRMS-equivalent methods for PCBs and chlorinated pesticides. We've seen with our chlorinated pesticides, for example, that we've been able to cut analysis time due to the instrument's superior electronic speed and ability to combine two runs in one and provide much higher quality analyses. We will be examining development and validation of methods for brominated flame retardants and multi-residue pesticides in the near future."*

Ms. Clark describes the factors involved: *"A big consideration for us is the practicality of it as a commercial offering. It needed to be robust and to be able to switch easily between methods, not just dioxins. We were looking for a replacement for our magnetic sector MS instruments for all the other analyses we do as well. We also consider things like keeping the same instrument provider, to maintain familiarity with quantification software. If you can avoid having multiple packages in-house, it makes it easier logistically to push work through. We have significant experience with MassLynx, plus TargetLynx, the quantitation software within it, which makes a big difference and influenced the decision-making process. It has everything we needed for processing and reporting dioxins."*



SGS AXYS scientists tested the capabilities of the APGC-MS/MS technology with the Waters Xevo TQ-XS Triple Quadrupole Mass Spectrometer.

*"This is one of my favorite instruments, it's highly sensitive and selective due to the soft ionization with APGC. It is easy to operate, and low-maintenance compared to the magnet sector, which is a big benefit to routine analysis. Also, I am able to switch between methods without any difficulty."*

MR. XINHUI XIE

Senior Instrument Chemist at SGS AXYS

The U.S. EPA approval of a new method that uses APGC-MS/MS for determining dioxins will take advantage of the technological advances made in tandem quadrupole MS over the decades.

For SGS AXYS customers, the new method for U.S. EPA Method 1613B has the potential to improve data quality for contaminants because it has excellent performance, readily meets all standard QA/QC specifications and handles complex sample matrices with ease. The advances in mass spectrometer source design and electronics provide the promise of fewer flags on low-level detects due to decreased noise.

Ms. Clark explains: *"SGS AXYS is committed to developing solutions to better service our customers' long-term needs and we believe the Waters APGC-MS/MS instrumentation can have a significant role."*

## References

1. Method 1613: Tetra-Through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS, *United States Environmental Protection Agency*, September 1994
2. Amending Regulation (EC) No 152/2009 as regards the determination of the levels of dioxins and polychlorinated biphenyls (June 2014) <https://eur-lex.europa.eu/legal-content/EN/TXT/PDF/?uri=CELEX:32014R0709&from=PT>

# Waters

THE SCIENCE OF WHAT'S POSSIBLE.™

Waters, The Science of What's Possible, APGC, and Xevo are trademarks of Waters Corporation. All other trademarks are the property of their respective owners.