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Shimadzu Journal

Vol 11
ISSUE1

Environmental Analysis

SJ25_0111 ISSN 2188-0484

Dear Readers,

Masami Tomita

General Manager,
Analytical & Measurement Instruments Division

2025 marks Shimadzu's 150th anniversary. Since its founding, Shimadzu has remained dedicated to addressing societal challenges in collaboration with key opinion leaders (KOLs), guided by our corporate philosophy: "*Contributing to Society through Science and Technology.*" For example, as part of our commitment to global sustainability, Shimadzu actively works to combat environmental issues such as climate change, biodiversity loss, and marine plastics pollution. These issues significantly impact daily life and economic activities, prompting the implementation of international initiatives to address them.

Another major topic of concern are PFAS (per- and polyfluoroalkyl substances), the focus of this issue. Known for their exceptional resistance to heat, water, and oil, PFAS are widely used across various industries. However, these same properties make them highly persistent in natural environments and biological systems, raising serious concerns about their risks to the environment and human health. In this article, we explore the efforts of researchers who are at the forefront of developing urgent countermeasures worldwide. We also showcase innovative applications leveraging cutting-edge technologies to tackle this pressing issue.

We begin with an interview with Dr. Dong Liang of the Chinese National Research Center for Environmental Analysis and Measurement (CNEAC). In this interview, he discusses their mission, current challenges in PFAS analysis, and key environmental conservation measures. Next, Dr. Ruth Marfil-Vega from Shimadzu Scientific Instruments shares insights into the challenges faced by scientists conducting PFAS analysis and how Shimadzu's solutions are helping to overcome them.

In addition to these interviews, we present a range of advanced PFAS analysis applications powered by Shimadzu's state-of-the-art technologies. These include methods for analyzing PFAS in drinking water using on-line SPE coupled with LC-MS/MS, EPA-compliant analysis, Q-TOF screening analysis, and innovative approaches for achieving efficient, high-accuracy analysis. We also highlight Shimadzu's latest environmental initiatives and introduce groundbreaking new products, including the LCMS-TQ RX series, Brevis GC-2050, and GCMS-QP2050.

This issue showcases advanced technologies and research tackling the global challenges posed by PFAS. As part of Shimadzu's ongoing commitment to sustainability and problem solving, we strive to reduce environmental impacts and build a better future. We hope this journal serves as a valuable resource, offering insights and inspiration to researchers, industry professionals, and innovators working to address these critical challenges. By fostering collaboration and knowledge sharing, we can drive meaningful progress toward a more sustainable future.

Yours Sincerely,

150 YEARS ANNIVERSARY

Environmental Analysis Issue1

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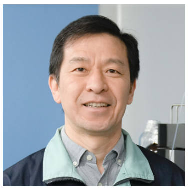

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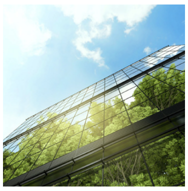

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Interview 1

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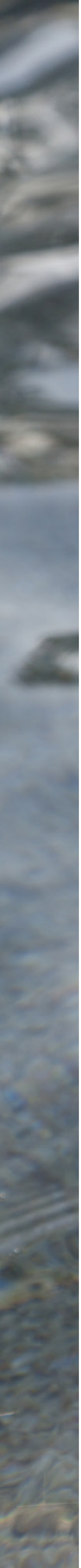


Interview 1 Interview with Dr. Liang DONG

“We are loyal fans of Shimadzu’s mass spectrometers.”

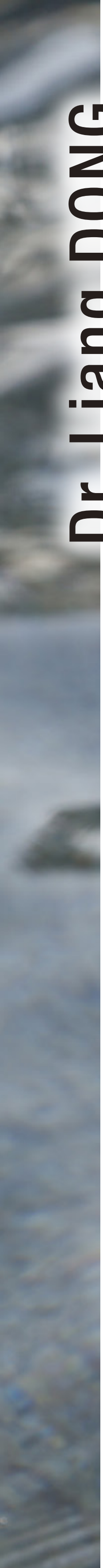
We recently had the opportunity to sit down with Dr. Liang DONG from the Chinese National Research Center for Environmental Analysis and Measurement (CNEAC), with whom we’ve had a long-standing collaboration since 1996. During our conversation, we explored the important work they’re doing to protect the environment, including CNEAC’s mission, the challenges they face with PFAS analysis, and China’s innovative approach to emerging pollutants.

Dr. Liang DONG, thank you very much for spending some time for this interview. First, could you provide an overview of Chinese National Research Center for Environmental Analysis and Measurement’s mission and the role your laboratory plays in ensuring environment safety?

The Chinese National Research Center for Environmental Analysis and Measurement (CNEAC) was founded in 1984 and is affiliated with the Ministry of Ecology and Environment. It’s currently managed by the China-Japan Friendship Center for Environmental Protection. The center mainly focuses on developing environmental testing methods for new pollutants, characteristic pollutants, and complex samples. It also plays a key role in supporting ecological and environmental monitoring and specialized investigations, while providing technical support for quality control in environmental testing organizations. Additionally, CNEAC conducts research on methods for assessing environmental damage and offers related technical services. CNEAC is home to the Ministry’s Key Laboratory for Dioxin Pollution Control. In recent years, the center has really leveraged its expertise in emerging pollutants. It has provided significant technical support for pilot monitoring and pollution prevention efforts. This includes creating standard analysis methods for new pollutants, conducting investigations, tracing sources, evaluating risks, and supporting pollution control in key river basins and other areas.

Could you share some insights on China’s current approach to environmental protection, particularly regarding water quality and soil pollution?

China’s environmental protection efforts are centered around three major battles – “Blue Sky,” “Clear Water,” and “Clean Land.” These focus on air, water, and soil pollution control. Significant progress has been made in tackling air pollution, particularly in reducing PM2.5 levels. In terms of water pollution, notable strides have been made, especially in eliminating black and odorous water bodies. However, soil pollution remains a major challenge, particularly when it comes to investigating and managing contaminated land, especially for construction purposes. The national soil pollution survey completed in 2019 showed that the rapid pace of economic development has left many gaps in soil pollution control. As cities expand, industrial and mining businesses located on the outskirts have been shut down or relocated, with the land now being repurposed for commercial and residential development. Therefore, investigating and managing soil and groundwater contamination has become a key



priority in China's environmental protection agenda. The government is also paying increasing attention to the management of emerging pollutants.

PFAS is a significant global concern. What innovative technologies and strategies are being implemented in your country to enhance water quality protection?

The Ministry of Ecology and Environment places great importance on the management of emerging pollutants like PFOA, PFOS, and PFHxS. In the "List of Key Control New Pollutants" (2023 edition) released at the end of 2022, one of the most critical groups of compounds is PFAS. Currently, the "Sanitary Standards for Drinking Water" (GB5749-2022) set the limits for PFOA and PFOS at 80 and 40 ng/L, respectively. The upcoming revision of the "Surface Water Environmental Quality Standards" (GB3838-2002) will likely include new limits for PFOA and PFOS. In addition to PFOA and PFOS, the concentration of PFHxS in some surface waters is also showing an upward trend, which is something that deserves attention.

How do you anticipate these advancements will impact the environment and public health in the coming years?

Due to the near non-degradability of perfluorinated compounds, and the fact that some PFAS precursors can transform into PFAS, it is crucial to conduct targeted investigations and monitoring in key regions and watersheds. This will help assess the environmental and public health risks associated with PFAS. Strengthening control at the source is the fundamental solution. As the use of compounds like PFOA, PFOS, and PFHxS is phased out, the residual levels of PFAS in China are expected to decrease, resulting in a lower overall risk to both the environment and public health.

Which Shimadzu instruments are currently being used in your laboratory for PFAS analysis, and what are their primary applications?

We use the Shimadzu HPLC/MS/MS8050. This instrument is primarily employed for the development of analytical methods for PFAS, antibiotics, and brominated flame retardants. It is also used in the monitoring and investigation of PFAS in key regional watersheds and environmental areas. Shimadzu's mass spectrometers are highly reliable. CNEAC also owns a series of Shimadzu instruments, including the GCMS-QP5000, GCMS-QP2010, GCMS-QP2010 Plus, GCMS-QP2010 Ultra, GCMS-QP2020, and GCMS-TQ8050 for various other analyses.

What are some of the key challenges you face in your daily operations, and how do you address them?

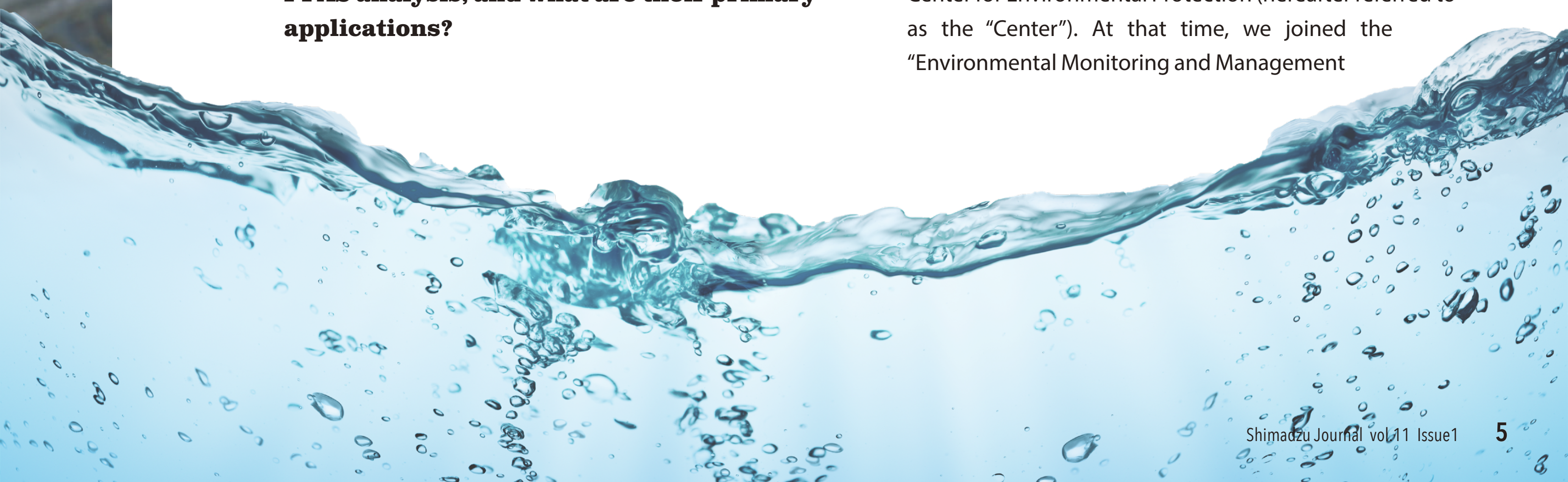
The most significant challenge in PFAS analysis is the complicated and time-consuming sample preparation. This step is not only labor-intensive but also frequently results in low recovery rates and high blanks. Our team consistently works to improve and refine the process to mitigate these issues.

What advancements or improvements would you like to see in your industry?

I would like to see further improvements in the sensitivity of HPLC/MS/MS systems. Additionally, the ability to perform direct injection On-line SPE analysis would greatly simplify the sample preparation process. This would be a significant breakthrough for environmental testing professionals.

We've had the pleasure of collaborating with you and the National Environmental Analysis and Testing Center for over 10 years. How has this long-term partnership influenced your work, and what are some of the key outcomes from it?

Our collaboration with Shimadzu began in 1996, following the establishment of the China–Japan Friendship Center for Environmental Protection (hereafter referred to as the "Center"). At that time, we joined the "Environmental Monitoring and Management



in East Asia” international cooperation project initiated by the United Nations University, which aimed to conduct research and analysis on trace organic pollutants in the environment. This project was sponsored by Shimadzu Corporation, and over the 20 years of cooperation, it played a crucial role in advancing our analytical technologies for persistent organic pollutants and other trace organic substances.

On June 29, 2011, we officially launched the “CNEAC-Shimadzu Environmental Protection Cooperative Research Laboratory,” marking the beginning of a trusted and supportive technical partnership. Through this cooperative laboratory, we've leveraged our respective strengths to deepen our collaboration. This has included active exchanges in technology and personnel training, resulting in significant research outcomes, such as the development of standard analytical methods for persistent organic pollutants.

We've successfully co-developed several key analytical methods, including:

- “Determination of PBDEs in Water Quality by GC/MS” (HJ 909-2017)
- “Determination of PBDEs in Soil and Sediment by GC-MS” (HJ 952-2018)
- “Determination of Perfluorooctane Sulfonate and Perfluorooctanoic Acid and Their Salts in Water Quality by Isotope Dilution/LC-MS/MS (Triple Quadrupole)” (HJ 1333-2023)
- “Determination of Perfluorooctane Sulfonate and Perfluorooctanoic Acid and Their Salts in Soil and Sediment by Isotope Dilution/LC-MS/MS (Triple Quadrupole)” (HJ 1334-2023)

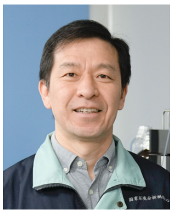
The two sides have successfully organized the “Environmental Research Cooperative Laboratory Technical Workshop” for nine sessions. In 2017, Director Ren Yong of the Center met with Shimadzu President Teruhisa Ueda, and both parties had a productive discussion, agreeing to further strengthen our collaboration. In 2018, the Center and Shimadzu signed an agreement to enhance communication and cooperation in environmental protection, aiming to complement each other's strengths and jointly develop new environmental analysis technologies and methods, as well as applications for environmental analysis equipment.

In January 2024, Director Chen Ming of the Center met with Koki Aoyama, the president of Shimadzu China, and both sides agreed to engage in deeper, practical cooperation to support high-level ecological environmental protection. At the beginning of 2025, CNEAC and Shimadzu renewed the agreement for the Environmental Protection Cooperative Research Laboratory.

Are there any upcoming projects or research areas where you anticipate further collaboration with our company?

Environmental monitoring is fundamental to addressing new pollutants, and it relies on accurate, reliable standard analytical methods. CNEAC looks forward to continuing our collaboration with Shimadzu in the development of standard analytical methods for new pollutants, such as antibiotics and brominated flame retardants, further contributing to the improvement of the overall system for new pollutant standards.

Thank you for sharing your insights and feedback. We are committed to exceeding your expectations moving forward. Thank you very much.



▶Liang DONG, Ph.D.

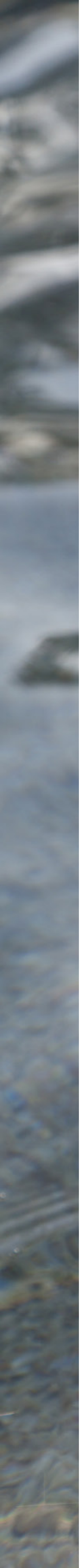
Researcher at the National Research Centre of Environmental Analysis and Measurement, Director of the Key Laboratory for Dioxin Pollution Control of MEE, Member of the first National Expert Committee for Hazardous Waste Identification, Leading Talent in National Environmental Protection Field, First batch of training and teaching experts of MEE (Ministry of Ecology and Environment).

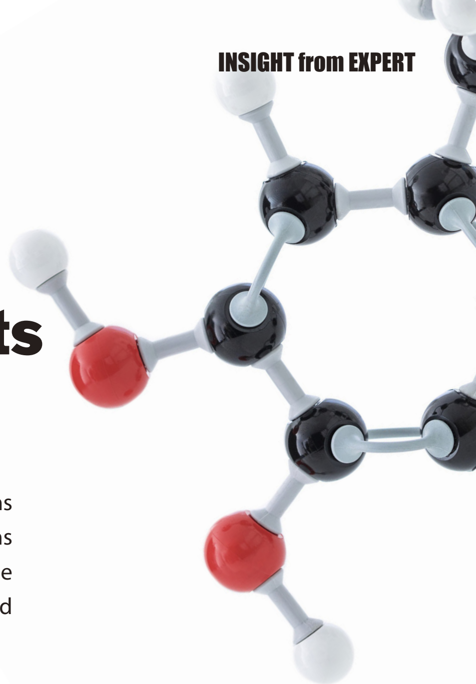
Undertaking various scientific research projects, including national key research and development projects, state-funded special projects for innovative methods of the Ministry of Science and Technology, state-funded special projects for the development of major instruments and equipment, pro bono scientific research projects, international cooperation projects, and standard revision projects etc.

Conducting research mainly on various organic pollutants in environmental media such as soil, water, air, and particulate matter, especially on the analysis and testing of persistent organic pollutants, pollution distribution, residue characteristics, and environmental behaviors.

Having been awarded with many prizes such as the Second Prize of Science and Technology of Environmental Protection twice, the First Prize of Scientific and Technological Progress in Shandong Province, the Third Prize of China Association for Analysis and Testing, the Second Prize of China Instrument and Meter Society.

Having published over 100 papers, more than 20 standards, 6 patents, and 5 works.





Interview 2 Interview with **Dr. Ruth Marfil-Vega**

“Challenges and requirements for environmental labs.”

We spoke with Dr. Ruth Marfil-Vega, Senior Market Manager for Environmental Solutions at Shimadzu Scientific Instruments Inc., USA. She provides insights into key questions regarding PFAS analysis for environmental laboratories. Dr. Marfil-Vega discusses the challenges faced by these laboratories as they rapidly adapt to address PFAS issues and comply with emerging regulatory requirements.

What are the main challenges your customers are facing right now regarding PFAS analysis?

Environmental laboratories work very close with water and wastewater treatment plants, consulting engineering firms, regulators and the public. The current challenges that everyone is facing right now, relate to three major topics: public concern—we all have easier access to information and lack of proper scientific communication is causing the public to voice more concerns about PFAS as well as to be more distress about them. The lack of resources to mitigate PFAS are becoming available slowly. Funding, building treatment facilities, personnel—everything must be in place while the scientific knowledge and the regulations are still being built around PFAS.

Regarding monitoring, labs are now facing the challenge to analyze new compounds with lower detection limits and in response to developing regulations, achieving shorter turnaround times, in addition to the other challenges that everyone is facing, like supply chain disruption and workforce replacement. Modern instruments for the analysis of PFAS like LC-MS/MS can generate the required data. However, the labs need time to secure the funding, install the instruments, and have the personnel ready to start generating PFAS data.

Dr. Ruth Marfil-Vega

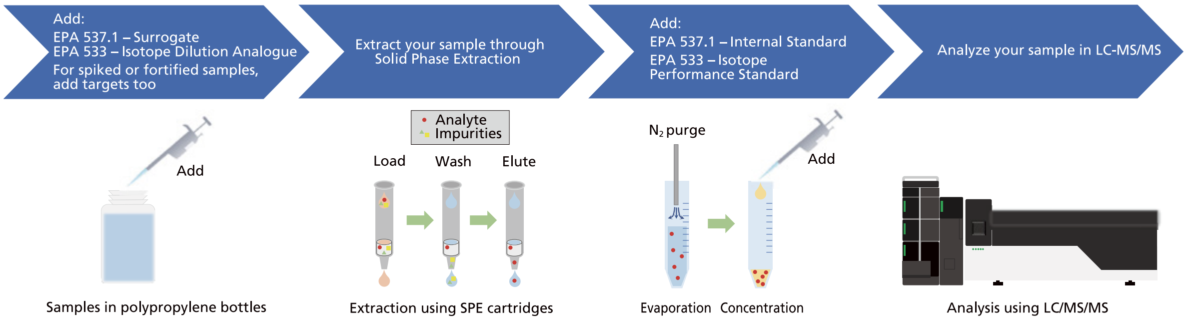
What new requirements and demands are environmental labs facing?

In April 2024, the U.S. Environmental Protection Agency published the PFAS National Primary Drinking Water Regulation for 6 specific PFAS: PFOA, PFOS, PFHxS, PFNA, GenX and PFBS. Maximum Contaminant Level (MCL) from this regulation allowed in tap water for the first five compounds listed ranges between 4 and 10 ng/L. For samples containing two or more of PFHxS, PFNA, GenX and PFBS a Hazard Index calculated based on their concentrations and factors established by EPA must be less than 1. This means that laboratories must be able to quantify lower concentrations than the MCLs; specifically, EPA has published Practical Quantitation Levels laboratories must meet between 3 and 5 ng/L, depending on the compound. Additionally, regulations for wastewater, sludge/biosolids and air emissions are also being developed at federal level in the United States. Some states have already published them. In general, the trend that environmental labs are experiencing is that they need to report lower and lower concentrations at a faster rate, so decisions can be made for regulation and remediation.

How is Shimadzu supporting customers with their PFAS Analysis efforts?

In anticipation to the lower reporting limits, Shimadzu re-evaluated the analysis of PFAS by methods EPA 537.1 and 533 with our LC-MS/MS. A common practice in environmental labs is to optimize methods to meet sensitivity, run times and QA requirements, but sometimes the instrument is not pushed to its maximum performance. With the lower reporting limits to respond to this regulation, the instrument setup has to be perfect, at its best. We have done the hard work for our customers. With our method package, everyone interested in compliance analysis will be ready to start the initial demonstration of capabilities, as the method will help them with that. We have similar resources for other methods, such as EPA 1633 and ASTM D8421 and D8535.

LC/MS/MS Method Package for PFAS in Drinking Water



Learn more
LC/MS/MS Method Package for PFAS in Drinking Water

Dr. Ruth Marfil-Vega

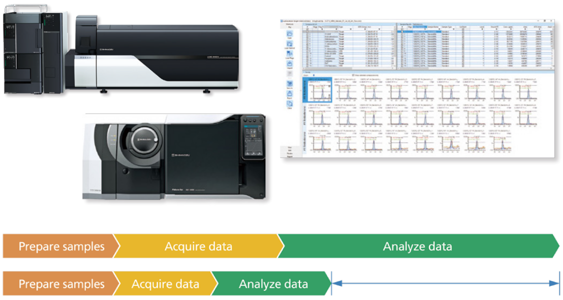
Quantifying the presence of per and polyfluoroalkyl substances of PFAS efficiently in different types of environmental samples such as water, soil, animal tissue and so on might seem an overwhelming task. Shimadzu collaborates with scientists to ease the analytical workflows and with our ready-to-use vetted methods we have minimized the three most important bottlenecks in the analytical process.

First, you probably know that PFAS are everywhere. When sample contamination happens our LC-MS instrument can be quickly ruled out as the source. We have conducted extensive testing backed by our customers experience to demonstrate minimal to no contribution to PFAS concentration from our hardware. And when PFAS are introduced in the system, the use of a delay column makes sure that the PFAS background does not interfere with the analysis If highly concentrated sample is injected in the LC-MS/MS, the design of the autosampler and hardware enables minimal carry over and fast cleanup of the instrument by flashing it gas with solvents commonly used in the laboratory.

PFAS: Essentials for Analysis by LC-MS/MS

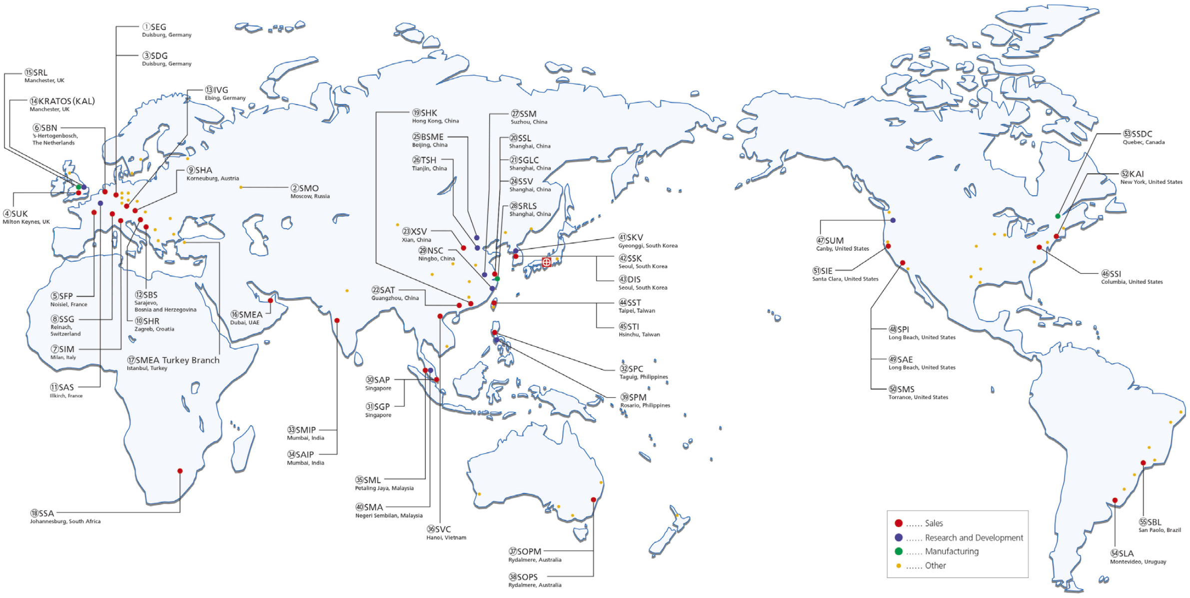
Second, the ever-growing list of PFAS analyzed as targets, surrogates and internal standards makes the data processing and review tedious and time consuming. However, with LabSolutions Insight you can streamline the process and focus on samples that need critical attention. You'll get your results faster with LabSolutions Insight data review screens that can be customized and with the automated QA/QC flagging for EPA methods 537.1 and 533, as well as other methods for PFAS analysis.

Faster Data Review for Dramatically Higher Productivity



Learn more
LabSolutions Insight

And third, for those moments when you need technical support, you need to add a new PFAS target to your methods, or you just need to update reports with the latest QA parameters required by EPA. We'll be there with you. Our team with combined decades of sustained experience in the analysis of PFAS. at Shimadzu and commercial laboratories, utilities research labs and more always provides unmatched support throughout the life of our LCMS.



Dr. Ruth Marfil-Vega

If you could give one key piece of advice about PFAS analysis, what would it be?

There is a wealth of knowledge out there based on 15-20 years of work in research and routine testing laboratories to help you demystify PFAS analysis and get your lab up and running quickly. If you are new to PFAS or LC-MS/MS analysis, just reach out to your trusted resources, and you will be fine.

Thank you for the valuable information on PFAS analysis within the environmental analysis field. Could you also shed some light on the PFAS analysis in drinking water, as our next edition will be touching this topic?

Analysis of PFAS in drinking water, as well as other regulated and unregulated contaminants, is a topic that I am enthusiastic about. Before I joined Shimadzu I worked on that field for many years. The occurrence of PFAS in drinking water and how to treat them is a complex topic. From the testing perspective, as I mentioned in the previous question, we have a lot of knowledge collected over decades. And we can use that knowledge to help labs getting their labs up and running and in supporting the treatment and compliance with regulation. I'm looking forward to reading what you have prepared for the next edition!

Related content

PFAS Q&A For Environmental Labs

PFAS Analysis Demystified

▶ Dr. Ruth Marfil-Vega has 20+ years of expertise in environmental chemistry and engineering in the US and Europe. She has worked with the EPA and the utilities industry to design and execute comprehensive solutions for environmental and water quality issues, including PFAS, micro- and nanoplastics and other emerging and regulated contaminants. Ruth has continued working with environmental stakeholders since she joined Shimadzu six years ago to ensure that analytical instrumentation and workflows enable their success. She graduated from the University of Valladolid (Spain; BSc in Chemistry), and from the University of Cincinnati (PhD in Environmental Science and Engineering).

Environmental Analysis

Determination of various PFAS in drinking water using on-line SPE coupled to LC-MS/MS

Anja Grüning
Shimadzu Europa GmbH

User Benefits

- Single vendor solution for UHPLC and MS system
- Quantification of 44 PFAS in ng/L range using an on-line SPE approach
- All PFAS requested by the EU directive 2020/2184[1] on the quality of water intended for human consumption are covered

Introduction

Perfluoroalkyl and polyfluoroalkyl substances (PFAS) refer to a class of more than 4000 individual chemicals that have been widely used since the 1950s, e.g. as fire retardants, food packaging materials or non-stick coatings. These compounds offer heat-resistant, and oil- and waterrepellant properties as well as chemical and thermal stability, resistance to UV light and weathering. Due to their anthropogenic origin, PFAS cannot be degraded, and hence they accumulate and can now be detected ubiquitously in the environment. Since drinking water is considered to be an important source of human PFAS intake, testing drinking water for PFAS levels has been essential for several years now.

This Application News demonstrates the determination of all PFAS requested in the EU directive 2020/2184[1] on the quality of water intended for human consumption in an appropriate concentration range. Furthermore, the analysis includes 24 additional PFAS and 22 internal standard using the same method based on an on-line SPE approach which omits additional sample preparation steps.

Materials and Methods

Fast, sensitive and robust LC-MS/MS systems provide the basis for routine analysis in drinking water laboratories. For the described application, a Shimadzu LCMS-8060NX triplequadrupole mass spectrometer coupled with a Nexera™ X3 UHPLC system was used (Figure 1, Figure 3).

44 PFAS standards and one IS-mixture (ISO 21675-LSS) were purchased (Wellington Laboratories / neochema). Stock solutions of these PFAS were diluted with methanol and combined to a single standard mixture with a final a concentration of 1ng/µL for each compound. Further dilutions of this mixture were produced and spiked into Evian water to prepare calibration samples in drinking water in the concentration range from 0.5 ng/L to 100 ng/L. Bottled Evian water was chosen as dinking water matrix as no noticeable PFAS could be detected in the blank. All samples (except blanks) were spiked with IS to a final concentration of 20 ng/L.

No further sample preparation is required. 1 mL of sample is injected directly on a SPE-trap column.

Analysis was performed within 15 minutes using MRM acquisition with at least two transitions for each compound (except PFBA, PFMPA, PFMBA, HPFHpA where only one transition is available). Analytical conditions are listed in Table 1. The optimized MRM transitions are summarized in Table 2.

Since PFAS can be present in reagents, glassware, pipettes, tubing, degassers and other parts from the LC-MS/MS instrument, the use of a solvent delay column is necessary. Small C18 columns are placed between mixer and autosampler respectively between mixer and valve to delay possible PFAS contaminations and separate them from sample-derived PFAS.

To minimize adsorption of (especially long chain) PFAS to the surface LabTotal Vials (P/N 227-34001-01) with PP-caps, with aluminium septa (P/N 961-10030-31) were used.

Table 1. Analytical conditions

Mass Spectrometer	: LCMS-8060NX
Ionization	: Electrospray Ionization (ESI), negative
Interface Voltage	: -1 kV
Focus Voltage	: -2 kV
Heating Gas	: 15 L/min
DL Temp.	: 150 °C
Interface Temp.	: 300 °C
Nebulizing Gas	: 3 L/min
Drying Gas	: 3 L/min
Heat Block	: 400 °C
Dwell-/Pause-time	: 4 (3 for IS) / 1 msec
CID	: 270 kPa
UHPLC	: Nexera X3
Pump A (Analytical)	: 2 mM ammonium acetate in H_2O
Pump B (Analytical)	: 10 mM ammonium acetate in Methanol
Pump C (Trap)	: H_2O + modifier (sample loading)
Pump D (Trap)	: Methanol (washing of SPE and delay column)
Analytical column	: Shim-pack™ Scepter 1.9 μm, C18-120, 2.1 x 50 mm
Delay column	: Shim-pack™ GIST HP 3 μm, C18-AQ, 3 x 30 mm
Trap column	: EVOLUTE® Express ABN on-line SPE cartridge
Injection Volume	: 1000 μL
Cooler temperature	: 15 °C
Column Oven	: 50 °C

Figure 1. LCMS-8060NX coupled to a Nexera™ X3 system

Results

Calibration curves were calculated using weighted (1/conc) linear regression. The linearity ranges from 0.5 ng/L (resp. 1 or 2.5 ng/L) – 100 ng/L (50 ng/L for PFNS) with an R^2 of at least 0.99 for all PFAS. The lowest calibration point (0.5 ng/mL) can be determined in 77.3% of all PFAS (Table 2). Exemplary calibration curves and respective MRMchromatograms at 1 ng/L are shown in Figure 2.

Two control samples at 5 ng/l and 25 ng/L were analyzed in three-fold to measure analytical reproducibility. The percentage relative standard deviation was typically lower than 20% (for >95% of the determined compounds resp. QCs) from these measurements (Table 3).

Table 2. MRM transitions and calibration information

Acronym	Compound name	RT	Type	Quantifier	Qualifier	ISTD	Calibration range	Unit	R^2
10:2 FTS	1H,1H,2H,2H-perfluorododecane sulfonic acid	9.64	Target	627.00>607.00	627.00>80.90	PFDoDA-IS	1 - 100	ng_L	0.9953
11Cl-PF3OUdS	11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	9.471	Target	630.90>451.05	630.90>82.95	PFDoDA-IS	0.5 - 100	ng_L	0.9958
3.7-DMPFOA	3,7-dimethylperfluorooctanoic acid	8.796	Target	469.00>269.00	469.00>219.05	PFNA-IS	0.5 - 100	ng_L	0.9975
4:2 FTS	1H, 1H, 2H, 2H-perfluorohexane sulfonic acid	6.806	Target	327.10>307.00	327.10>80.95	PFHxA-IS	0.5 - 100	ng_L	0.9995
6:2 FTS	1H, 1H, 2H, 2H-perfluorooctane sulfonic acid	8.112	Target	427.10>407.00	427.10>80.90	6:2 FTS-IS	0.5 - 100	ng_L	0.9994
6:2 FTS-IS		8.113	ISTD	428.90>408.90	428.90>80.95	----	----	ng_L	----
8:2 diPAP	8:2 Fluorotelomer phosphate diester	10.597	Target	989.10>543.15	989.10>96.95	8:2 diPAP-IS	2.5 - 100	ng_L	0.9914
8:2 diPAP-IS		10.596	ISTD	992.80>96.85	992.80>544.90	----	----	ng_L	----
8:2 FTS	1H, 1H, 2H, 2H-perfluorodecane sulfonic acid	8.996	Target	527.10>507.00	527.10>80.90	8:2 FTS-IS	1 - 100	ng_L	0.9989
8:2 FTS-IS		8.993	ISTD	529.00>508.95	529.00>80.95	----	----	ng_L	----
9Cl-PF3ONS	9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid	8.826	Target	530.90>351.10	530.90>82.90	PFOS-IS	0.5 - 100	ng_L	0.9984
DONA	4,8-dioxa-3H-perfluorononanoic Acid	7.68	Target	377.10>251.00	377.10>84.95	PFHpA-IS	0.5 - 100	ng_L	0.9967
FOUEA/8:2 FTUCA	2H-perfluoro-2-decenoic acid	8.736	Target	457.10>393.20	457.10>343.05	FOUEA-IS	0.5 - 100	ng_L	0.9980
FOUEA-IS	2H,2H,3H,3H-perfluorundecanoic acid	8.735	ISTD	459.00>394.05	459.00>344.00	----	----	ng_L	----
H4PFUNA		9.133	Target	491.10>367.20	491.10>387.05	PFOS-IS	1 - 100	ng_L	0.9980
HPFHpA	7H-perfluorheptanoic acid	6.545	Target	345.10>281.00	----	PFHpA-IS	0.5 - 100	ng_L	0.9982
N-Et-FOSA	N-ethylperfluorooctanesulfonamide	10.167	Target	526.00>169.05	526.00>219.05	N-Et-FOSA-IS	0.5 - 100	ng_L	0.9995
N-Et-FOSAA	N-ethylperfluorooctanesulfonamidoacetic acid	9.345	Target	584.00>419.00	584.00>526.00	N-Et-FOSAA-IS	0.5 - 100	ng_L	0.9974
N-Et-FOSAA-IS		9.339	ISTD	589.00>419.10	589.00>530.90	----	----	ng_L	----
N-Et-FOSA-IS		10.163	ISTD	531.10>169.00	531.10>219.20	----	----	ng_L	----
NFDHA	Nonafluoro-3,6-dioxaheptanoic acid	6.75	Target	201.00>84.95	295.10>201.00	PFHxA-IS	0.5 - 100	ng_L	0.9980
N-Me-FOSA	N-methylperfluorooctanesulfonamide	9.976	Target	511.90>169.00	511.90>219.05	N-Me-FOSA-IS	0.5 - 100	ng_L	0.9956
N-Me-FOSAA	N-methylperfluorooctanesulfonamidoacetic acid	9.176	Target	569.90>418.95	569.90>512.15	N-Me-FOSAA-IS	0.5 - 100	ng_L	0.9973
N-Me-FOSAA-IS		9.168	ISTD	572.90>419.10	572.90>515.10	----	----	ng_L	----
N-Me-FOSA-IS		9.968	ISTD	515.00>169.00	515.00>219.00	----	----	ng_L	----
PEESA	Perfluoro(2-ethoxyethane)sulfonic acid	6.538	Target	315.00>135.00	315.00>82.90	PFHxA-IS	0.5 - 100	ng_L	0.9941
PFBA*	Perfluorobutanoic acid	4.759	Target	213.00>169.00	----	PFBA-IS	1 - 100	ng_L	0.9993
PFBA-IS		4.754	ISTD	216.90>172.00	----	----	----	ng_L	----
PFBS*	Perfluorobutane sulfonic acid	6.142	Target	299.00>79.90	299.00>98.90	PFBS-IS	0.5 - 100	ng_L	0.9996
PFBS-IS		6.139	ISTD	301.90>79.80	301.90>98.80	----	----	ng_L	----
PFDA*	Perfluorodecanoic acid	8.994	Target	513.00>469.00	513.00>219.05	PFDA-IS	0.5 - 100	ng_L	0.9975
PFDA-IS		8.997	ISTD	519.00>473.90	519.00>219.00	----	----	ng_L	----
PFDoA / PFDoDA*	Perfluorododecanoic acid	9.624	Target	613.00>568.95	613.00>169.10	PFDoDA-IS	0.5 - 100	ng_L	0.9981
PFDoDA-IS		9.619	ISTD	614.90>570.10	614.90>269.10	----	----	ng_L	----
PFDoS / PFDoDS*	Perfluorododecane sulfonic acid	9.853	Target	699.00>98.90	699.00>79.90	PFDoDA-IS	1 - 100	ng_L	0.9908
PFDS*	Perfluorodecane sulfonic acid	9.312	Target	598.80>79.95	598.80>98.85	PFDoDA-IS	1 - 100	ng_L	0.9995
PFHpA*	Perfluoroheptanoic acid	7.59	Target	363.10>319.00	363.10>169.00	PFHpA-IS	0.5 - 100	ng_L	0.9979
PFHpA-IS		7.591	ISTD	367.00>322.10	367.00>169.00	----	----	ng_L	----
PFHpS*	Perfluoroheptane sulfonic acid	8.167	Target	448.90>79.90	448.90>98.90	PFHxS-IS	0.5 - 100	ng_L	0.9981
PFHxA*	Perfluorohexanoic acid	6.886	Target	313.10>269.00	313.10>119.00	PFHxA-IS	0.5 - 100	ng_L	0.9988
PFHxA-IS		6.883	ISTD	317.90>273.00	317.90>120.10	----	----	ng_L	----
PFHxDA	Perfluorohexadecanoic acid	10.455	Target	813.00>769.00	813.00>169.00	PFHxDA-IS	2.5 - 100	ng_L	0.9943
PFHxDA-IS		10.454	ISTD	814.90>769.90	814.90>369.00	----	----	ng_L	----
PFHxS*	Perfluorohexane sulfonic acid	7.637	Target	398.90>79.95	398.90>98.90	PFHxS-IS	0.5 - 100	ng_L	0.9985
PFHxS-IS		7.636	ISTD	402.00>79.90	402.00>98.80	----	----	ng_L	----
PFMBA	Perfluoro-4-methoxybutanoic acid	6.265	Target	279.10>84.95	----	PFPeA-IS	0.5 - 100	ng_L	0.9987
PFMPA	Perfluoro-3-methoxypropanoic acid	5.209	Target	228.90>84.95	----	PFBA-IS	0.5 - 100	ng_L	0.9995
PFNA*	Perfluorononanoic acid	8.606	Target	463.00>418.95	463.00>219.00	PFNA-IS	0.5 - 100	ng_L	0.9996
PFNA-IS		8.605	ISTD	471.90>427.00	471.90>223.00	----	----	ng_L	----
PFNS*	Perfluorononane sulfonic acid	8.984	Target	549.10>79.90	549.10>98.90	PFUnDA-IS	0.5 - 50	ng_L	0.9947
PFOA*	Perfluorooctanoic acid	8.144	Target	413.20>369.00	413.20>169.05	PFOA-IS	0.5 - 100	ng_L	0.9967
PFOA-IS		8.142	ISTD	421.00>376.10	421.00>172.00	----	----	ng_L	----
PFOcDA / PFODA	Perfluorooctadecanoic acid	10.75	Target	913.00>868.95	913.00>169.00	PFHxDA-IS	1 - 100	ng_L	0.9965
PFOS*	Perfluorooctane sulfonic acid	8.606	Target	498.90>79.90	498.90>98.90	PFOS-IS	0.5 - 100	ng_L	0.9981
PFOSA / FOSA	perfluorooctane sulfonamide	9.313	Target	497.90>77.90	497.90>478.15	FOSA-IS	0.5 - 100	ng_L	0.9974
PFOSA-IS		9.312	ISTD	505.90>78.00	505.90>172.00	----	----	ng_L	----
PFOS-IS		8.603	ISTD	506.90>79.90	506.90>98.80	----	----	ng_L	----
PFPeA / PFPA*	Perfluoropentanoic acid	5.94	Target	263.10>219.00	263.10>69.10	PFPeA-IS	0.5 - 100	ng_L	0.9991
PFPeA-IS		5.946	ISTD	267.90>223.00	267.90>69.10	----	----	ng_L	----
PFPeS / PFPS*	Perfluoropentane sulfonic acid	6.992	Target	349.20>79.95	349.20>98.95	PFHxS-IS	0.5 - 100	ng_L	0.9978
PFTeDA	Perfluorotetradecanoic acid	10.096	Target	713.00>669.05	713.00>169.05	PFTeDA-IS	0.5 - 100	ng_L	0.9967
PFTeDA-IS		10.102	ISTD	714.90>670.00	714.90>368.90	----	----	ng_L	----
PFTrDA*	Perfluorotridecanoic acid	9.878	Target	663.00>619.00	663.00>169.00	PFDoDA-IS	0.5 - 100	ng_L	0.9988
PFTrDS*	Perfluorotridecane sulfonic acid	10.067	Target	749.00>99.10	749.00>79.90	PFDoDA-IS	1 - 100	ng_L	0.9915
PFUnDA*	Perfluoroundecanoic acid	9.332	Target	563.00>518.95	563.00>269.05	PFUnDA-IS	0.5 - 100	ng_L	0.9947
PFUnDA-IS		9.333	ISTD	570.00>524.90	570.00>268.90	----	----	ng_L	----
PFUnS / PFUnDS*	Perfluoroundecane sulfonic acid	9.601	Target	649.00>79.95	649.00>98.95	PFDoDA-IS	0.5 - 100	ng_L	0.9962

* PFAS requested in EU directive 2020/2184[1] on the quality of water intended for human consumption

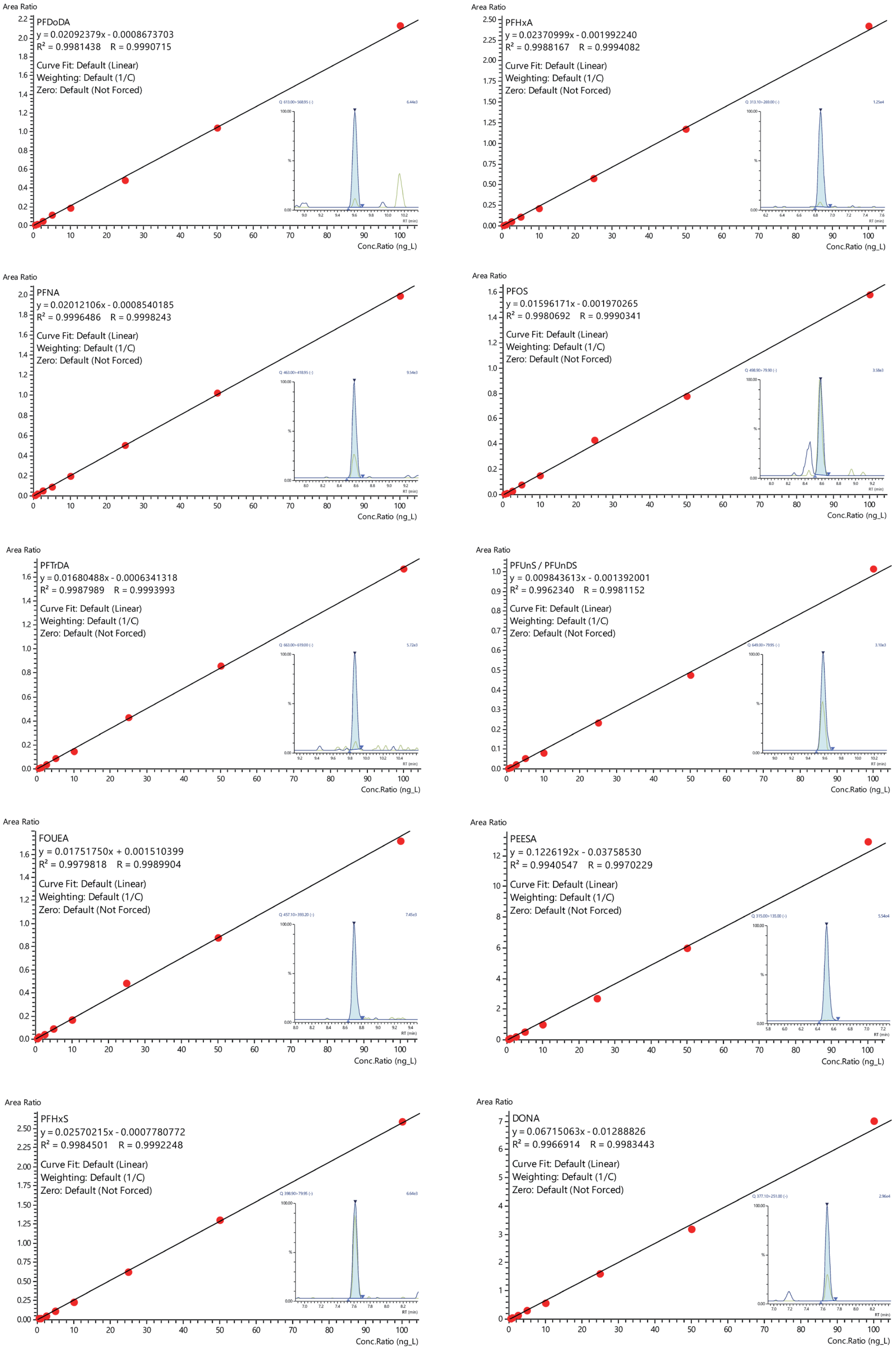


Figure 2. Exemplary calibration curves ranging from 0.5– 100 ng/L and a typical chromatogram at 1 ng/L level

Table 3. Reproducibility of QC samples

	10:2 FTS		11Cl-PF3OUdS		3.7-DMPFOA		4:2 FTS		6:2 FTS		8:2 diPAP		8:2 FTS		9Cl-PF3ONS		DONA		FOSA		FOUEA	
	QC low		QC low		QC low		QC low		QC low		QC low		QC low		QC low		QC low		QC low		QC low	
	5 ng/L		5 ng/L		5 ng/L		5 ng/L		5 ng/L		5 ng/L		5 ng/L		5 ng/L		5 ng/L		5 ng/L		5 ng/L	
	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy
QC L1	5.31	106.23	5.19	103.81	4.77	95.3	4.72	94.32	4.98	99.55	3.61	72.1	6.9	137.93	4.98	99.55	4.67	93.36	4.6	91.96	4.02	80.45
QC L2	5.46	109.24	4.62	92.33	4.08	81.59	4.73	94.56	4.75	95.07	4.45	89.06	5.43	108.58	4.29	85.86	4.78	95.53	4.78	95.55	4.65	92.94
QC L3	5.04	100.72	4.11	82.14	4.37	87.5	4.57	91.48	5.55	110.98	4.71	94.15	5.53	110.58	4.29	85.79	4.48	89.52	4.68	93.59	4.87	97.48
Mean		105.39		92.76		88.13		93.46		101.87		85.1		119.03		90.4		92.81		93.7		90.29
SD		4.32		10.84		6.88		1.71		8.21		11.54		16.4		7.92		3.04		1.79		8.82
%RSD		4.1		11.68		7.8		1.83		8.06		13.56		13.78		8.77		3.28		1.92		9.76
	QC high		QC high		QC high		QC high		QC high		QC high		QC high		QC high		QC high		QC high		QC high	
	25 ng/L		25 ng/L		25 ng/L		25 ng/L		25 ng/L		25 ng/L		25 ng/L		25 ng/L		25 ng/L		25 ng/L		25 ng/L	
	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy
QC H1	26.11	104.45	21.67	86.68	19.91	79.63	25.11	100.44	26.79	107.17	26.08	104.3	25.13	100.54	24.43	97.71	23.23	92.92	25.59	102.35	24.58	98.33
QC H2	28.41	113.64	24.83	99.33	21.29	85.16	24.93	99.71	26.17	104.69	20.85	83.42	26.86	107.44	24.36	97.43	24.12	96.49	25.52	102.09	25.1	100.42
QC H3	21.79	87.16	20.43	81.71	21.28	85.11	25.78	103.1	25.34	101.36	19.04	76.15	28.41	113.63	21.26	85.05	25.08	100.31	23.74	94.97	25.88	103.53
Mean		101.75		89.24		83.3		101.09		104.4		87.96		107.2		93.4		96.57		99.8		100.76
SD		13.45		9.09		3.18		1.78		2.92		14.61		6.55		7.23		3.7		4.19		2.62
%RSD		13.22		10.18		3.82		1.76		2.79		16.61		6.11		7.74		3.83		4.2		2.6

	H4PFUNA		HPFHpA		PFDoS		PFTrDS		N-Et-FOSA		N-Et-FOSAA		NFDHA		N-Me-FOSA		N-Me-FOSAA		PEESA		PFBA	
	QC low		QC low		QC low		QC low		QC low		QC low		QC low		QC low		QC low		QC low		QC low	
	5 ng/L		5 ng/L		5 ng/L		5 ng/L		5 ng/L		5 ng/L		5 ng/L		5 ng/L		5 ng/L		5 ng/L		5 ng/L	
	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy
QC L1	5.21	104.23	5.23	104.52	4.56	91.14	4.17	83.31	4.86	97.25	4.22	84.31	4.63	92.68	4.56	91.22	4.83	96.64	4.29	85.71	4.75	94.96
QC L2	4.82	96.45	5.33	106.7	4.07	81.3	5.92	118.41	4.54	90.86	4.8	95.96	4.67	93.32	5.25	104.93	5.27	105.45	4.42	88.45	4.86	97.29
QC L3	5.57	111.42	4.92	98.37	4.22	84.33	3.88	77.54	4.24	84.87	3.62	72.41	4.29	85.78	5.1	102	6.04	120.77	4.36	87.23	4.53	90.58
Mean		104.03		103.19		85.59		93.08		90.99		84.23		90.59		99.38		107.62		87.13		94.28
SD		7.49		4.32		5.04		22.12		6.19		11.77		4.18		7.22		12.21		1.37		3.4
%RSD		7.2		4.19		5.89		23.76		6.81		13.98		4.61		7.27		11.34		1.58		3.61
	QC high		QC high		QC high		QC high		QC high		QC high		QC high		QC high		QC high		QC high		QC high	
	25 ng/L		25 ng/L		25 ng/L		25 ng/L		25 ng/L		25 ng/L		25 ng/L		25 ng/L		25 ng/L		25 ng/L		25 ng/L	
	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy
QC H1	27.56	110.24	26.32	105.26	21.13	84.52	20.06	80.26	24.91	99.63	24.67	98.68	24.04	96.16	24.63	98.53	27.48	109.94	21.6	86.41	25.02	100.09
QC H2	25.61	102.43	26.94	107.75	24.8	99.19	19.47	77.89	26.51	106.05	21.96	87.86	23.64	94.57	25.58	102.32	24.1	96.39	22.57	90.27	24.36	97.45
QC H3	24.41	97.65	27.49	109.97	17.95	71.82	21.95	87.78	23.24	92.94	27.96	111.84	24.43	97.73	21.9	87.58	24.8	99.21	22.86	91.45	24.09	96.37
Mean		103.44		107.66		85.18		81.98		99.54		99.46		96.15		96.15		101.85		89.38		97.97
SD		6.36		2.35		13.7		5.17		6.56		12.01		1.58		7.66		7.15		2.64		1.91
%RSD		6.14		2.19		16.08		6.3		6.59		12.08		1.64		7.96		7.02		2.95		1.95

	PFBS		PFDA		PFDoDA		PFDS		PFHpA		PFHpS		PFHxA		PFHxDA		PFHxS		PFMBA		PFMPA	
	QC low		QC low		QC low		QC low		QC low		QC low		QC low		QC low		QC low		QC low		QC low	
	5 ng/L		5 ng/L		5 ng/L		5 ng/L		5 ng/L		5 ng/L		5 ng/L		5 ng/L		5 ng/L		5 ng/L		5 ng/L	
	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy
QC L1	4.71	94.2	5.26	105.22	5.01	100.29	5.01	100.1	4.91	98.21	5.13	102.62	4.81	96.29	3.13	62.53	4.65	92.93	4.7	94.09	4.69	93.73
QC L2	4.97	99.45	5.72	114.45	4.93	98.7	6.32	126.32	4.66	93.28	4.99	99.73	4.7	94.03	4.78	95.55	4.86	97.2	4.66	93.29	4.92	98.41
QC L3	4.6	92.07	5.29	105.88	4.1	81.9	6.85	137.01	4.5	89.93	5.02	100.39	4.45	89.06	3.06	61.24	4.81	96.17	4.97	99.3	4.85	96.97
Mean		95.24		108.52		93.63		121.14		93.81		100.91		93.13		73.11		95.43		95.56		96.37
SD		3.8		5.15		10.19		18.99		4.16		1.51		3.7		19.44		2.23		3.26		2.4
%RSD		3.99		4.74		10.88		15.67		4.44		1.5		3.97		26.6		2.34		3.42		2.49
	QC high		QC high		QC high		QC high		QC high		QC high		QC high		QC high		QC high		QC high		QC high	
	25 ng/L		25 ng/L		25 ng/L		25 ng/L		25 ng/L		25 ng/L		25 ng/L		25 ng/L		25 ng/L		25 ng/L		25 ng/L	
	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy
QC H1	24.31	97.23	23.81	95.22	24.84	99.37	24.35	97.39	24.46	97.84	25.69	102.75	23.93	95.7	23.96	95.82	24.97	99.9	25.4	101.59	25.6	102.38
QC H2	24.92	99.69	27.11	108.43	24.84	99.35	28.56	114.23	25.97	103.86	24.81	99.22	24.39	97.54	24.88	99.51	24.94	99.75	25.02	100.08	25.23	100.92
QC H3	23.97	95.89	26.51	106.05	21.72	86.86	24.29	97.17	26.63	106.54	25.64	102.55	24.14	96.57	24.16	96.64	25.54	102.15	24.38	97.52	25	100
Mean		97.6		103.24		95.19		102.93		102.75		101.51		96.61		97.33		100.6		99.73		101.1
SD		1.93		7.04		7.21		9.79		4.45		1.98		0.92		1.94		1.35		2.06		1.2
%RSD		1.97		6.82		7.58		9.51		4.33		1.95		0.95		1.99		1.34		2.06		1.19

	PFNA		PFNS		PFOA		PFODA		PFOS		PFPeA		PFPeS / PFPS		PFTeDA		PFTrDA		PFUnDA		PFUnS / PFUnDS	
	QC low		QC low		QC low		QC low		QC low		QC low		QC low		QC low		QC low		QC low		QC low	
	5 ng/L		5 ng/L		5 ng/L		5 ng/L		5 ng/L		5 ng/L		5 ng/L		5 ng/L		5 ng/L		5 ng/L		5 ng/L	
	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy
QC L1	4.68	93.6	4.72	94.32	4.65	92.96	3.93	78.6	4.15	82.93	4.69	93.86	4.62	92.35	4.65	92.93	4.64	92.84	4.25	85.01	4.12	82.47
QC L2	4.78	95.64	5.56	111.23	4.92	98.38	5.92	118.39	4.87	97.4	4.76	95.21	4.68	93.68	3.67	73.33	4.18	83.7	4.24	84.76	3.51	70.23
QC L3	4.45	89.1	5.65	112.91	4.78	95.54	5.34	106.85	5.3	106.06	4.83	96.53	4.97	99.34	3.85	77.05	3.93	78.69	4.71	94.22	2.84	56.84
Mean		92.78		106.15		95.62		101.28		95.46		95.2		95.13		81.1		85.08		88		69.85
SD		3.35		10.28		2.71		20.47		11.69		1.34		3.71		10.41		7.17		5.39		12.82
%RSD		3.61		9.69		2.84		20.21		12.24		1.4		3.9		12.84		8.43		6.13		18.35
	QC high		QC high		QC high		QC high		QC high		QC high		QC high		QC high		QC high		QC high		QC high	
	25 ng/L		25 ng/L		25 ng/L		25 ng/L		25 ng/L		25 ng/L		25 ng/L		25 ng/L		25 ng/L		25 ng/L		25 ng/L	
	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy	Conc.	Accuracy
QC H1	21.94	87.75	27.02	108.1	24.01	96.06	24.63	98.51	25.73	102.94	25.35	101.42	23.74	94.96	25.32	101.29	24.77	99.08	25.75	103.02	22.83	91.33
QC H2	24.48	97.93	26.94	107.75	23.77	95.06	28.41	113.63	25.38	101.52	24.92	99.68	24.11	96.43	23.47	93.88	23.64	94.56	24.69	98.77	25.7	102.81
QC H3	24.36	97.45	27.99	111.94	22.88	91.52	26.33	105.3	24.68	98.73	24.75	98.99	25.88	103.52	19.6	78.39	22.32	89.28	23.37	93.48	17.16	68.63
Mean		94.38		109.26		94.21		105.82		101.06		100.03		98.31		91.19		94.31		98.42		87.59
SD		5.74		2.33		2.39		7.57		2.14		1.25		4.58		11.68		4.91		4.78		17.39
%RSD		6.09		2.13		2.53		7.16		2.12		1.25		4.66		12.81		5.2		4.85		19.86

Figure 3. Scheme of the NexeraTM on-line SPE LCMS-8060NX system

The Package

Main Unit		
LCMS-8060NX	TQ Mass spectrometer	
Nexera X3	Liquid chromatograph CBM-40 DGU-405 2x LC-40D X3 LC-40B X3 SIL-40C X3 CTO-40S 2x Reservoir Tray	
Accessory		
Valve	FCV-0206H3	
Mixer	2x Mir20 µL	
Loop	1 mL	
Main Consumables		
Shim-pack Scepter C18		(50 mm x 2.1 mm I.D., 1.9 µm; P/N 227-31012-03)
Shim-pack GIST HP C18-AQ (2x)		(30 mm x 3.0 mm I.D., 3 µm; P/N 227-30766-01)
EVOLUTE® Express ABN on-line SPEcartridge (Biotage)		(30 mm x 2.1 mm I.D; P/N OSPE-620-32150)
Shimadzu LabTotal Vial for LC/LCMS		(P/N 227-34001-01)
PP-caps, with aluminium septa		(P/N 961-10030-31)

This part number is available in the EU area only. If you are in another territory, please contact for your Shimadzu local office

Software and Libraries
LabSolutions LCMS
LabSolutions Insight

Conclusions

This application note describes an on-line SPE LC-MS/MS method to monitor 44 PFAS and 22 internal standards in drinking water. Using the LCMS-8060NX coupled with a Nexera UHPLC system equipped for on-line SPE a highly robust and sensitive method for routine PFAS analysis in drinking water which omits additional sample preparation steps is demonstrated.

References

1. DIRECTIVE (EU) 2020/2184 OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 16 December 2020 on the quality of water intended for human consumption

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Environmental Analysis

Untargeted Screening of Per- and Polyfluoroalkyl Substances by HRAM-DIA Method on LCMS-9030

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[2] Internship Student from Faculty of Science, National University of Singapore

User Benefits

- A sensitive untargeted screening method was established based on HRAM-DIA data acquisition on LCMS-9030. The method was successfully verified with 14 PFAS standards at 1 ng/mL in water.
- Data analysis was performed using LabSolutions Insight Explore™ – Analyze. PFAS-like species could be extracted by specific elemental settings which functions as mass defect filtering for PFAS. This procedure was used for an unknown sample, and 16 PFAS-like species were discovered and characterized.

Introduction

Contamination of per- and polyfluoroalkyl substances (PFAS) are found everywhere in water, soils, sediments, fish, foods, textiles and human blood etc. Targeted screening and quantitation of PFAS in drinking water by LC-MS/MS are established in reference to the US EPA Method 537 and ISO 21675: 2019, ASTM D7979 etc. Such MRM-based methods are widely adopted in the analysis of up to 29 PFAS or more [1]. However, PFAS represents a large collective of compounds [2] and many of them are not determined by the existing methods. This study aims to establish a method of untargeted screening for both known and undiscovered PFAS in water samples. The method was established based on LC-Q-TOF data, i.e., HRAM spectrum (MS) and DIA deconvolution spectrum (MS/MS), relying on the specific mass defect feature of PFAS [3, 4] using LabSolutions Insight Explore – Analyze program. Fourteen PFAS including PFOA and PFOS were used as standards for verifying the performance of this HRAM-DIA method in terms of detection sensitivity and identification. The established approach was applied to real water sample analysis and the found PFAS-like species were further characterized via database and library searches, and structural elucidation using LabSolutions Insight Explore – Assign program.

Experimental

Reagents, PFAS standards and samples

Acetonitrile (LCMS grade) and methanol (LCMS grade) were obtained from commercial suppliers. Ammonium acetate (>99%) of LCMS grade was used as additives in the mobile phase prepared from Milli-Q water. Sixteen PFAS standards (Table 2) were purchased from Wellington Laboratories and Apollo scientific. M-PFOS (with $^{13}C_4$) and M-PFOA (with $^{13}C_4$) were used as internal standards in method development. Water samples were collected and subjected to analysis for the discovery of unknown PFAS in the study.

LC-Q-TOF analytical conditions

Details of the analytical conditions on LCMS-9030, Q-TOF system (Shimadzu Corporation, Japan) are shown in Table 1. LabSolutions v5.114 and LabSolutions Insight Explore v3.8 SP4 were used for data acquisition in MS and DIA mode and data processing of HRAM spectra and DIA MS/MS spectra for efficient detection and identification of targeted and untargeted PFAS.

Tabl 1. Analytical conditions of PFAS on LCMS-9030

LC Conditions	
Column	Shim-pack™ Velox, C18 (2.1x100 mm, 2.7 µm)
Flow Rate	0.4 mL/min
Mobile Phase	A: 5 mM Ammonium acetate in water B: Acetonitrile
LC gradient	B: 10% (0-0.5 min) → 85% (8.5 min-9 min) 10% → (9.1 min-12 min) → stop
Delay column	Shim-pack Velox, C18 (2.1x50 mm, 2.7 µm)
Oven Temp.	40°C
Injection Vol.	50 µL

Interface Conditions and MS mode	
Interface	ESI Heated
Interface Temp.	300°C
DL Temp.	250°C
Heat Block Temp.	400°C
Nebulizing Gas	3 L/min (N2)
Heating Gas Flow	10 L/min (Air)
Drying Gas Flow	10L/min (N2)
MS mode	MS (-), m/z 100~1000 DIA (-), m/z 50~1000; with CE 25V and Spread (+/-) 20V Loop time: 1.01 sec

Untargeted screening method

As shown in Table 1, MS and DIA events were set up for data acquisition. Data analysis and processing were performed with the LabSolutions Insight Explore suite, which include Analyze and Assign etc. The Analyze is for the deconvolution of DIA data to generate a precursor list and provide various functions of in-depth data analysis such as deconvoluted MS/MS spectrum, formula prediction and library search etc. The Assign is for identification and structural elucidation, which links to database searches such as ChemSpider and PubChem. Both Analyze and Assign were highly efficient and flexible in data processing and result display. Two PFAS libraries were installed and used in this study: (1) an in-house PFAS HRMS MS/MS library including spectra of 34 PFAS standards [4] and (2) MS-DIAL PFAS_Neg library [5].

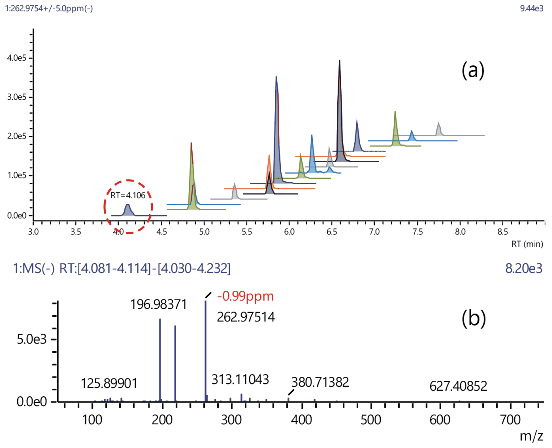


Figure 1. (a) XICs of 14 PFAS and 2 ISTD (1 ng/mL each); (b) MS spectrum of the 1st peak (PFPA).

Results and Discussion

1. Detection of PFAS by HRAM

Table 2 shows the results of detection of 14 PFAS and 2 ISTD in pure water from HRAM data (event 1) acquired on LCMS-9030 using the method as described above. The XIC chromatograms of 1 ng/mL sample is displayed in Figure 1a and the spectrum of the first XIC peak (PFPA) is shown in Figure 1b. The lowest detectable concentrations by HRAM are 0.01 ng/mL for 11 PFAS, 0.02 ng/mL for PFDA, 0.05 ng/mL for PFPA and 0.1 ng/mL for PFTrA and PFTeA. The mass accuracy is better than (+/-) 3.3 ppm, with most compounds less than (+/-) 2 ppm. Linear calibration curves were established from the lowest concentrations to 5 ng/mL for all the PFAS with R^2 between 0.94 and 0.99. PFOA, PFOS and their isotope labelled standards (M-PFOA, M-PFOS) could also be detected at 0.01 ng/mL level. These results indicate that a highly-sensitive screening and quantitation method could be established based on HRAM on LCMS-9030.

Table 2. Sixteen PFAS information, accurate mass, measured error, RT and calibration curve range on LCMS-9030

PFAS (Abbr.)	Formula	CAS No.	[M-H]-(Meas.)	[M-H]-(Calc.)	Error (ppm)	RT (min)	Range (ng/mL)	R^2
PFPA	$C_5HF_9O_2$	2706-90-3	262.9751	262.9760	-3.27	4.12	0.05 ~ 5	0.989
PFBS	$C_4F_9SO_3H$*	29420-49-3	298.9421	298.9430	-3.01	4.86	0.01 ~ 5	0.941
PFHxA	$C_6HO_2F_{11}$*	307-24-4	312.9718	312.9728	-2.91	4.88	0.01 ~ 5	0.981
PFHpA	$C_7HF_{13}O_2$*	375-85-9	362.9687	362.9696	-2.20	5.36	0.01 ~ 5	0.982
PFOA	$C_8HF_{15}O_2$*	335-67-1	412.9655	412.9664	-1.99	5.77	0.01 ~ 5	0.982
M-PFOA	$^{13}C_4C_4HF_{15}O_2$	N.A.	416.9791	398.9366	-1.73	5.77	0.01 ~ 5	0.982
PFHxS	$C_6F_{13}HO_3S$*	82382-12-5	398.9358	462.9632	-1.75	5.86	0.01 ~ 5	0.950
PFNA	$C_9HF_{17}O_2$*	375-95-1	462.9624	512.9600	-1.60	6.13	0.01 ~ 5	0.983
PF-3,7-DMOA	$C_{10}HF_{19}O_2$	172155-07-6	468.9692	512.9600	-1.83	6.27	0.01 ~ 5	0.985
PFDA	$C_{10}HF_{19}O_2$*	335-76-2	512.9592	498.9302	-1.58	6.47	0.02 ~ 5	0.988
PFOS	$C_8F_{17}O_3HS$*	4021-47-0	498.9296	502.9436	-1.22	6.59	0.01 ~ 5	0.955
M-PFOS	$^{13}C_4C_4F_{17}O_3HS$	N.A.	502.9429	562.9568	-1.31	6.59	0.01 ~ 5	0.969
PFUnA	$C_{11}HF_{21}O_2$*	2058-94-8	562.9562	598.9238	-1.12	6.80	0.02 ~ 5	0.998
PFDS	$C_{10}HF_{21}SO_3$	2806-15-7	598.9232	662.9505	-1.09	7.24	0.01 ~ 5	0.987
PFTrA	$C_{13}HO_2F_{25}$*	72629-94-8	662.9497	712.9473	-1.27	7.43	0.1 ~ 5	0.991
PFTeA	$C_{14}HO_2F_{27}$*	376-06-7	712.9463	262.9760	-1.42	7.75	0.1 ~ 5	0.983

* Targeted PFAS by EPA 537 method

2. Detection of PFAS by DIA

The DIA data of the same data set was processed with the LabSolutions Insight Explore - Analyze to generate a long list of precursors via deconvolution, followed by applying predicting formula. To look for PFAS-like compounds and species, the key settings of elements include F: 6~50, H: 1~5, O: 1~5, C: 1~50, S: 0~1 and N: 0~1. These settings restrict the elemental composition of candidate with a negative mass defect and could be used to find PFAS-like species [4]. Figure 2 shows an example of this approach. A precursor peak (*m/z*262.9751) generated from DIA data (Figure 2a) appeared at the same RT as PFPA in MS XIC (Figure 1a). The mass defect measured is -24.9 mDa, which is very close to the calculated mass defect of PFPA (-24.0 mDa). The corresponding deconvoluted MS/MS spectrum as shown in Figure 2b matches perfectly to the PFPA MS/MS spectrum in the PFAS library (Figure 2c). These results confirm that PFPA of 1 ng/mL spiked in water can be detected firmly via the DIA analysis approach.

The detection and identification results of the 14 spiked PFAS in water are compiled into Table 3. As can be seen from the Table, all the 14 PFAS were detected and identified via the above approach using the Analyze.

The results indicate that PFAS can be detected from DIA data, which is the basis to use the HRMS-DIA method for untargeted screening of PFAS in water samples.

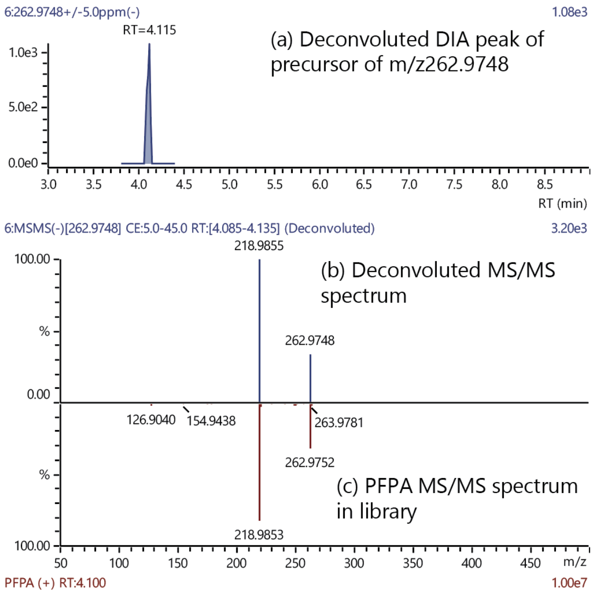


Figure 2. Detection of PFPA (1 ng/mL in water) by DIA peak (a) and deconvoluted spectrum (b), which matches to the library spectrum of PFPA (c)

Table 3. Detection and identification of 14 PFAS (1 ng/mL) from DIA data using Analyze program

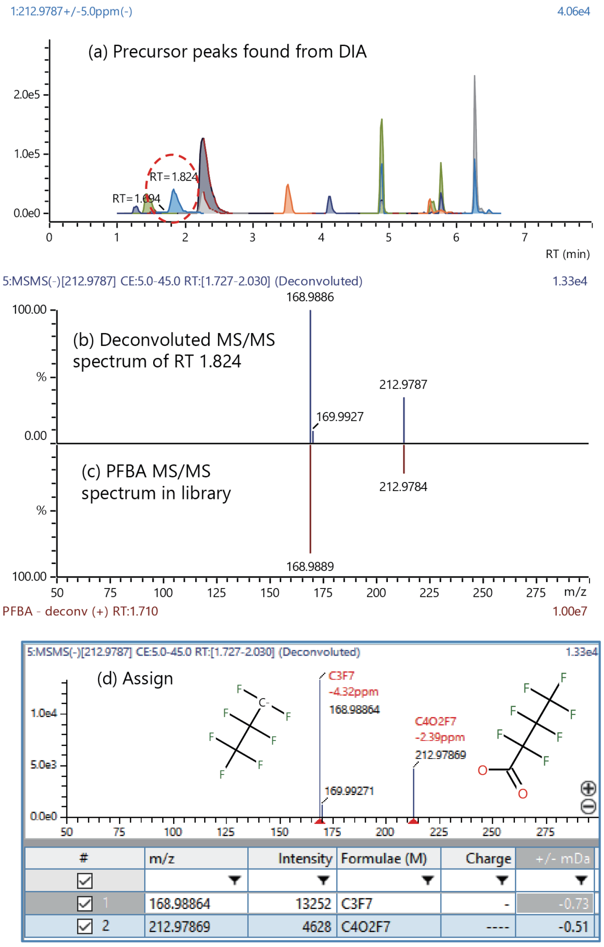
PFAS (Abbr.)	PFAS Formula	Precursor generated from DIA data	Precursor RT (min)	Precursor ion formula obtained	Deconvoluted spectrum from DIA data	PFAS Lib Search
PFPA	$C_5HF_9O_2$	262.9748	4.12	$[C_5HO_2F_9\text{-}H]^-$	262.9748, 218.9855	Confirmed
PFBS	$C_4F_9SO_3H$	298.9421	4.86	$[C_4HO_3F_9S\text{-}H]^-$	298.9421, 98.9552, 79.9565	Confirmed
PFHxA	$C_6HO_2F_{11}$	312.9721	4.88	$[C_6HO_2F_{11}\text{-}H]^-$	312.9721, 268.9819, 118.9918	Confirmed
PFHpA	$C_7HF_{13}O_2$	362.9695	5.35	$[C_7HO_2F_{13}\text{-}H]^-$	362.9695, 318.9791, 168.9888	Confirmed
PFOA	$C_8HF_{15}O_2$	412.9656	5.77	$[C_8HO_2F_{15}\text{-}H]^-$	412.9656, 368.9757, 218.9857, 168.9884	Confirmed
PFHxS	$C_6F_{13}HO_3S$	398.9357	5.85	$[C_6HO_3F_{13}S\text{-}H]^-$	398.9357, 118.9918, 98.9548, 79.9567	Confirmed
PFNA	$C_9HF_{17}O_2$	462.9625	6.14	$[C_9HO_2F_{17}\text{-}H]^-$	462.9625, 418.9725, 218.9856, 168.9885	Confirmed
PF-3,7-DMOA	$C_{10}HF_{19}O_2$	468.9693	6.26	$[C_9HF_{19}\text{-}H]^{-*}$	468.9693, 446.9687, 268.9822	Confirmed
PFDA	$C_{10}HF_{19}O_2$	512.9592	6.46	$[C_{10}HO_2F_{19}\text{-}H]^-$	512.9592, 468.9694, 268.9823, 218.9855, 168.9884	Confirmed
PFOS	$C_8F_{17}O_3HS$	498.9294	6.58	$[C_8HO_3F_{17}S\text{-}H]^-$	498.9294, 168.9883, 118.9921, 98.9551, 79.9565	Confirmed
PFUnA	$C_{11}HF_{21}O_2$	562.9565	6.80	$[C_{11}HO_2F_{21}\text{-}H]^-$	562.9565, 518.9660, 318.9789, 268.9818, 218.9848, 168.9882	Confirmed
PFDS	$C_{10}HF_{21}SO_3$	598.9232	7.24	$[C_{10}HO_3F_{21}S\text{-}H]^-$	598.9232	Confirmed
PFTrA	$C_{13}HO_2F_{25}$	662.9486	7.43	$[C_{13}HO_2F_{25}\text{-}H]^-$	662.9486, 618.9597	Confirmed
PFTeA	$C_{14}HO_2F_{27}$	712.9465	7.75	$[C_{14}HO_2F_{27}\text{-}H]^-$	712.9465, 668.9566, 168.9880	Confirmed

* PF-3,7-DMOA is ionized to form $[M\text{-}HCOO]^- = [C_9HF_{19}\text{-}H]^-$

3. Untargeted screening by HRAM-DIA method

The above data processing using Analyze was adopted for untargeted screening of PFAS in unknown samples. The obtained DIA data of a water sample was processed with Analyze and a long list of precursors (>800) was produced. By applying formula prediction with specific elemental settings as described above to all precursors, 16 PFAS-like precursors were generated (Table 4), which feature with the characteristic negative mass defects. Upon applying library search, five candidates were found in the PFAS libraries: PFBA (SI=94%), PFCA-unsaturated (SI=65%), 6:2 fluorotelomer sulfonic acid (SI=55%), PFOA (SI=61%) and PF-3,7-DMOA (SI=76%). In addition, PFCA-diether H_substituted was found to match to *m/z*626.9530 spectrum with a very low SI (23%). For such poor library matched and the remaining totally un-matched species, their identities could rely only on the characterization through structural elucidation analysis using the Assign program.

The 16 PFAS-like precursor peaks extracted from DIA data via Analyze are displayed in Figure 3a. Taking the peak at 1.824 min (*m/z*212.9787) as an example, the deconvoluted MS/MS spectrum and library search are shown in Figure 3b and 3c. The results confirm the presence of PFBA in the sample. If the deconvoluted MS/MS spectrum was sent to Assign program which links to database search (ChemSpider), heptafluorobutyric acid (CAS No.: 375-22-4) was found as a matched structure and the precursor as well as a fragment were annotated (Figure 3d). It is actually same as perfluorobutanoic acid (PFBA). Another representative example is candidate #15, which was detected at 6.26 min with a precursor ion of *m/z*446.9676. PFAS Library search did not generate any result. The deconvoluted MS/MS spectrum was sent to Assign with inputting a formula of C_9HOF_{17} for searching in ChemSpider database. The Assign program generated a list of



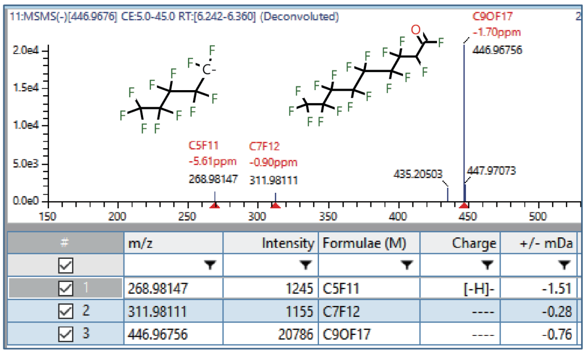
#	m/z	Intensity	Formulae (M)	Charge	+/- mDa
☑	▼	▼	▼	▼	▼
☑ 1	168.98864	13252	C3F7	-	-0.73
☑ 2	212.97869	4628	C4O2F7	----	-0.51

Figure 3. (a) Detection of unknown PFAS-like compounds from DIA, (b) deconvoluted spectrum of peak at RT 1.824, (c) matches to library spectrum of PFBA; (d) Assign to Heptafluorobutyric acid structure and fragment (same as PFBA).

Table 4. Untargeted screening for detection and identification of PFAS from a water sample by HRAM-DIA on LCMS-9030

Candidate #	Precursor from DIA	RT (min)	Precursor ion Formula	Error (ppm)	Deconvoluted DIA MS/MS spectrum	PFAS Library search	ID by Assign (ChemSpider)
1	402.9979	1.26	$[C_{13}H_4OF_{12}\text{-}H]^-$	-4.6	402.9979	Not found	(2E)-2,3,4,4,5,5,6,6,7,7,7-Undecafluoro -1-(4-fluorophenyl)-2-hepten-1-one
2	404.0146	1.43	$[C_{15}H_5NOF_{10}\text{-}H]^-$	1.9	404. 0146, 376.9950	Not found	N-[2,3,5,6-Tetrafluoro-4-(trifluoromethyl) phenyl]-2-(trifluoromethyl) benzamide
3	212.9787	1.82	$[C_4HO_2F_7\text{-}H]^-$	-2.4	212.9787, 168.9886	Perfluorobutanoic acid (PFBA)	Heptafluorobutyric acid
4	220.9864	2.23	$[C_8H_2O_3F_4\text{-}H]^-$	-1.6	220.9864, 138.9956, 79.9567	Not found	2,3,5,6-Tetrafluoro-4-formylbenzoic acid
5	219.9835	2.27	$[C_5HNO_2F_6\text{-}H]^-$	-1.5	219.9835, 81.9525	Not found	4-Cyano-2,2,3,3,4,4-hexafluorobutanoic acid
6	247.9786	3.51	$[C_6HNO_3F_6\text{-}H]^-$	-0.8	247.9786, 219.9838, 79.9566	Not found	bis(trifluoromethyl)-1,2-oxazole-4-carboxylic acid
7	196.9836	4.12	$[C_4HOF_7\text{-}H]^-$	-3.6	196.9836, 130.9923, 80.9951, 68.9951	Not found	butanal, heptafluoro-
8	246.9805	4.88	$[C_5HOF_9\text{-}H]^-$	-2.4	246.9805, 180.9887, 130.9918, 118.9919	Not found	2,2,3,3,4,4,5,5,5-Nonafluoropentanal
9	626.9530	4.88	$[C_{12}H_2O_4F_{22}\text{-}H]^-$	0.1	626. 9530, 354.9605, 312.9722, 268.9822	PFCA-diether H_substituted	No result
10	224.9786	4.89	$[C_5HO_2F_7\text{-}H]^-$	-2.8	224.9786, 174.9816	PFCA-unsaturated	1,1,1,3,5,5,5-Heptafluoro-2,4-pentanedione
11	374.9738	4.89	$[C_{10}H_5O_3F_9S\text{-}H]^-$	-1.4	374.9738, 312.9720, 268.9821	Not found	Phenyl 1,1,2,2,3,3,4,4,4-nonafluoro-1-butanesulfonate
12	426.9681	5.60	$[C_8H_5O_3F_{13}S\text{-}H]^-$	0.4	426.9677	6:2 Fluorotelomer sulfonic acid	1H,1H,2H,2H-PERFLUOROOCTANESULFONIC ACID
13	346.9742	5.77	$[C_7HOF_{13}\text{-}H]^-$	-1.6	346.9742, 280.9816, 96.9595	Not found	2,2,3,3,4,4,5,5,6,6,7,7-Dodecafluoroheptanoylfluoride
14	412.9654	5.77	$[C_8HO_2F_{15}\text{-}H]^-$	-2.5	412.9654, 368.9753, 218.9856, 168.9887	Perfluorooctanoic acid (PFOA)	Perfluorooctanoic Acid (PFOA)
15	446.9676	6.26	$[C_9HOF_{17}\text{-}H]^-$	-1.7	446. 9676, 311.9811, 268.9815	Not found	2,3,3,4,4,5,5,6,6,7,7,8,8,9,9,9-Hexadecafluorononanoyl fluoride
16	468.9697	6.26	$[C_9HF_{19}\text{-}H]^-$	-3.8	468.9697, 446.9678, 268.9822, 218.9854, 168.9886	PF-3,7-DMOA	2,2,3,4,4,5,5,6,6,7,8,8,8-tridecafluoro-3,7-bis(trifluoromethyl)octanoic acid, C10HF19O2

candidates for the formula and spectrum. One of most-likely candidate is 2,3,3,4,4,5,5,6,6,7,7,8,8,9,9,9-Hexadecafluorononanoyl fluoride. As can be seen in Figure 4, the precursor ion and two fragments match the structure essentially. However, it is worth to note that, although the identification results using Assign program and ChemSpider database may provide reference structures and information, the results are not considered as conclusions and further structural analysis is required.



#	m/z	Intensity	Formulae (M)	Charge	+/- mDa
☑					
☑ 1	268.98147	1245	C5F11	[-H]-	-1.51
☑ 2	311.98111	1155	C7F12	----	-0.28
☑ 3	446.96756	20786	C9OF17	----	-0.76

Figure 4. Deconvoluted MS/MS spectrum of precursor peak at 6.26 min (*m/z*446.9676); Assign: 2,3,3,4,4,5,5,6,6,7,7,8,8,9,9,9-Hexadecafluorononanoyl fluoride, C_9HOF_{17}, from ChemSpider.

Conclusion

In this study, an untargeted screening method based on HRAM-DIA data acquisition was established on LCMS-9030. The DIA data obtained was deconvoluted using the LabSolutions Insight Explore – Analyze to generate a list of precursors. Then, PFAS-like species were extracted by applying formula prediction with specific elemental settings. This approach was successfully verified with 14 PFAS standards at 1 ng/mL in water and applied to analyze an unknown water sample. Sixteen PFAS-like species were found. PFAS library search and structural elucidation using the Assign program were conducted for these PFAS-like species.

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Environmental Analysis

Analysis of Per-and Polyfluoroalkyl Substances (PFAS) using the LCMS-8050 Triple Quadrupole Mass Spectrometer According to EPA Draft Method 1633

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User Benefits

- Limits of Quantitation listed in EPA Draft 1633 were readily achieved with the Shimadzu LCMS-8050, a system that delivers high-quality data, throughput, and affordability.
- The robust LCMS-8050 performs well with complex environmental matrices, such as wastewater samples used in this study.
- This efficient instrument is field upgradeable to higher sensitivity instruments, which makes it a versatile and future-proof solution for PFAS analysis.

Keywords

Per- and Polyfluorinated Alkyl Substances, PFAS, Perfluorinated Compounds, PFCs, PFOA, PFOS, Triple Quad, EPA Draft 1633, EPAM1633, aqueous samples, water, wastewater

Abstract

This application note demonstrates that the Shimadzu LCMS-8050 meets and exceeds the Quality Assurance and Quality Control criteria and performance specified in Environmental Protection Agency (EPA) draft method 1633 for the analysis of Per- and Polyfluoroalkyl Substances (PFAS) in environmental samples. All analytes were reliably quantitated at or less than the Limit of Quantitation (LOQ) reported in the EPA draft method. This work proves the suitability of the Shimadzu LCMS-8050 for accurate and robust analysis of PFAS in routine laboratories in accordance with present demands in sensitivity and throughput. The method presented here can be optimized for other matrices or additional compounds.

Introduction

The EPA is continuing to standardize methods for the analysis of PFAS in environmental samples using Liquid Chromatography with Triple Quadrupole Spectrometry (LC-MS/MS). Shimadzu offers a full line of Ultra-Fast LC-MS/MS (UFMS™) systems for quantitating PFAS at environmentally relevant concentrations. EPA Draft Method 1633 (EPAM1633)[1] is the latest method proposed by the EPA in 2022 for the quantitation of targeted PFAS in aqueous, solid, biosolid, and tissue samples.

This application note, part of the comprehensive suite of Shimadzu's vetted solutions for the analysis of PFAS[2], demonstrates the performance of the Shimadzu LCMS-8050 for the quantitation of PFAS in accordance with EPAM1633. The LCMS-8050 was selected for this work because of its known dependability to deliver the sensitivity and robustness expected by modern environmental laboratories for the routine analysis of PFAS using methods requiring sample preparation by Solid Phase Extraction (SPE), like EPAM1633, or with a large-volume injection (*e.g.*, EPA 8327, ASTM 8421-22).

Experimental Approach and Instrumentation

This application note describes the analysis of the 40 target PFAS compounds, 23 Extracted Internal Standards (EIS), and 7 Non-Extracted Internal Standards (NIS) included in EPAM1633 in reagent water and wastewater using the Shimadzu LCMS-8050. PFAS types and acronyms used in EPAM1633 and this work are listed in Table 1. All standards used in the work presented here were purchased from Wellington Laboratories (PFAC-MXF, PFAC-MXG, PFAC-MXH, PFAC-MXI, PFAC-MXJ, MPFAC-HIF-ES, MPFAC-HIF-IS) as native mixes.

Table 1. Target analytes, EIS, and NIS included in this method

Type	Name	Type	Name
Target	PFBA	EIS	13C4-PFBA
Target	PFPeA	EIS	13C5-PFPeA
Target	PFHxA	EIS	13C5-PFHxA
Target	PFHpA	EIS	13C4-PFHpA
Target	PFOA	EIS	13C8-PFOA
Target	PFNA	EIS	13C9-PFNA
Target	PFDA	EIS	13C6-PFDA
Target	PFUnA	EIS	13C7-PFUnA
Target	PFDoA	EIS	13C2-PFDoA
Target	PFTrDA	EIS	13C2-PFTeDA
Target	PFTeDA	EIS	13C3-PFBS
Target	PFBS	EIS	13C3-PFHxS
Target	PFPeS	EIS	13C8-PFOS
Target	PFHxS	EIS	13C2-4:2FTS
Target	PFHpS	EIS	13C2-6:2FTS
Target	PFOS	EIS	13C2-8:2FTS
Target	PFNS	EIS	13C8-PFOSA
Target	PFDS	EIS	D3-NMeFOSA
Target	PFDoS	EIS	D5-NEtFOSA
Target	4:2FTS	EIS	D3-NMeFOSAA
Target	6:2FTS	EIS	D5-NEtFOSAA
Target	8:2FTS	EIS	D7-NMeFOSE
Target	PFOSA	EIS	D9-NEtFOSE
Target	NMeFOSA	EIS	13C3-HFPO-DA
Target	NEtFOSA	NIS	13C3-PFBA
Target	NMeFOSAA	NIS	13C2-PFHxA
Target	NEtFOSAA	NIS	13C4-PFOA
Target	NMeFOSE	NIS	13C5-PFNA
Target	NEtFOSE	NIS	13C2-PFDA
Target	HFPO-DA	NIS	18O2-PFHxS
Target	ADONA	NIS	13C4-PFOS
Target	9Cl-PF3ONS	--	TDCA
Target	11Cl-PF3OUdS		
Target	3:3 FTCA		
Target	5:3 FTCA		
Target	7:3 FTCA		
Target	PFEESA		
Target	PFMPA		
Target	PFMBA		
Target	NFDHA		

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Standards and Calibration Curve Preparation

The native PFAS mixtures PFAC-MXF, PFAC-MXG, PFAC-MXH, PFAC-MXI, PFAC-MXJ, and methanol were mixed in a ratio of 20:10:10:10:12.5:37.5 for preparing Target Standard Solution I. Target standard solution II was prepared by diluting Target Standard Solution I 10 times with methanol. Native PFAS Mixtures MPFAC-HIF-ES, MPFAC-HIF, and methanol were mixed in a ratio of 15:15:270 for preparing the Internal standard solution. Calibration standards were prepared by mixing Target Standard Solution I, Target Standard Solution II, Internal Standard Solution, and methanol to achieve the desired concentrations listed in EPA draft method 1633 and Table 2 (targets: 0.2 – 250 ng/mL; EIS: 1.25 – 25 ng/mL; NIS: 1.25 – 5 ng/mL).

Sample Preparation

500 mL of wastewater effluent or ultra-pure water were collected into pre-cleaned polyethylene bottles and weighed. 25 µL of EIS stock solution was added to each sample. Pre-conditioning of the SPE cartridges with 0.1% methanolic ammonium, methanol, and reagent water was performed according to the product manual (Inert Sep mini MA-2: 5010-27235, 280 mg). Samples were loaded onto the SPE cartridges at 5 mL/min. Sample bottles were rinsed with 10 mL of reagent water and those rinses were loaded onto the SPE cartridges. The cartridge was dried with nitrogen gas and the analytes were eluted with 5 mL of 0.1% methanolic ammonium. 25 µL of NIS was added to each extract.

Instrument and operational conditions

The LC-MS/MS analysis was performed using a Shimadzu Nexera ultrahigh pressure liquid chromatography (UHPLC) system coupled with a triple quad LCMS-8050. A delay column was used in this work as the essential modification of hardware for minimizing possible PFAS background contamination from LC and solvents[3].

A description of the LC-MS/MS parameters is included in Table 3. Sample-to-sample run time of 20 minutes includes the re-equilibration for both the delay and analytical columns after final wash out with concentrated acetonitrile to flush the column, remove background residual contaminants, and restore column performance before starting the next run.

All compound parameters, including precursor ion, product ions, and collision energies, were optimized using flow injection analysis (i.e., bypassing the analytical column) using LabSolutions software. Optimized retention times and precursor and product ions for multiple reaction monitoring (MRMs) are listed in Table 3.

Table 3. Chromatography and mass spectrometer conditions

Parameter	Value
LCMS	Shimadzu LCMS-8050
Analytical column	Shim-pack GIST-HP C18 2.1 × 50 mm, 3 µm
Delay column	Shim-pack GIST C18 3.0 × 50 mm, 5 µm
Column oven temp	40˚C
Injection volume	2 µL
Mobile phase	A: 2 mM Ammonium Acetate in 5 % (v/v) Acetonitrile in reagent water B: Acetonitrile
Gradient flow rate	0.4 mL/ Min
Run time	20 minutes
Nebulizing gas flow	3 L/ Min
Heating gas flow	15 L/Min
Interface temperature	190℃
Desolvation line temperature	200℃
Heat block temperature	300℃
Drying gas flow	5 L/ Min
Acquisition cycle time	16 min
Total MRMs	72

Table 2. Retention time, precursor ion, product ions, and calibration range used in this method.

Type	Name	RT	Precursor Ion	Product Ion-1	Product Ion-2	Concentration CS1 (ng/mL)	Concentration CS7 (ng/mL)
Target	PFBA	2.024	213.0	169.0	N/A	0.8	250
Target	PFPeA	3.57	263.0	219.0	69.0	0.4	125
Target	PFHxA	4.906	313.0	269.0	119.0	0.2	62.5
Target	PFHpA	7.708	363.0	319.0	169.0	0.2	62.5
Target	PFOA	8.64	413.0	369.0	169.0	0.2	62.5
Target	PFNA	9.119	463.0	419.0	219.0	0.2	62.5
Target	PFDA	9.511	513.0	469.0	219.0	0.2	62.5
Target	PFUnA	9.901	563.0	519.0	269.0	0.2	62.5
Target	PFDoA	10.461	613.0	569.0	319.0	0.2	62.5
Target	PFTrDA	11.345	663.0	619.0	168.9	0.2	62.5
Target	PFTeDA	12.237	713.0	669.0	168.9	0.2	62.5
Target	PFBS	4.841	299.0	80.0	99.0	0.2	62.5
Target	PFPeS	7.766	349.0	80.0	99.0	0.2	62.5

Type	Name	RT	Precursor Ion	Product Ion-1	Product Ion-2	Concentration CS1 (ng/mL)	Concentration CS7 (ng/mL)
Target	PFHxS	8.776	399.0	80.0	99.0	0.2	62.5
Target	PFHpS	9.294	449.0	80.0	99.0	0.2	62.5
Target	PFOS	9.709	499.0	80.0	99.0	0.2	62.5
Target	PFNS	10.2	549.0	80.0	99.0	0.2	62.5
Target	PFDS	10.956	598.9	80.0	99.0	0.2	62.5
Target	PFDoS	12.524	698.9	80.0	99.0	0.2	62.5
Target	4:2FTS	4.404	327.0	307.0	80.9	0.8	250
Target	6:2FTS	8.398	427.0	407.0	80.9	0.8	250
Target	8:2FTS	9.323	527.0	507.0	80.9	0.8	250
Target	PFOSA	11.438	498.0	78.0	478.0	0.2	62.5
Target	NMeFOSA	13.178	512.0	219.0	169.0	0.2	62.5
Target	NEtFOSA	13.475	526.0	219.0	169.0	0.2	62.5
Target	NMeFOSAA	9.491	570.0	419.0	483.0	0.2	62.5
Target	NEtFOSAA	9.647	584.0	419.0	526.0	0.2	62.5
Target	NMeFOSE	13.016	616.0	59.0	N/A	2	625
Target	NEtFOSE	13.305	630.0	59.0	N/A	2	625
Target	HFPO-DA	5.693	285.0	169.0	185.0	0.8	250
Target	ADONA	8.209	377.0	251.0	85.0	0.8	250
Target	9Cl-PF3ONS	10.032	530.9	351.0	353.0	0.8	250
Target	11Cl-PF3OUdS	11.728	630.9	451.0	453.0	0.8	250
Target	3:3 FTCA	2.937	241.0	177.0	117.0	1	312.5
Target	5:3 FTCA	6.076	341.0	237.0	217.0	5	1562.5
Target	7:3 FTCA	8.952	441.0	317.0	337.0	5	1562.5
Target	PFEESA	5.943	315.0	135.0	83.0	0.4	125
Target	PFMPA	2.681	229.0	85.0	N/A	0.4	125
Target	PFMBA	3.916	279.0	85.0	N/A	0.4	125
Target	NFDHA	4.715	295.0	201.0	85.0	0.4	125
EIS	13C4-PFBA	2.025	217.0	172.0	172.0	10	10
EIS	13C5-PFPeA	3.568	268.0	223.0	223.0	5	5
EIS	13C5-PFHxA	4.904	318.0	273.0	120.0	2.5	2.5
EIS	13C4-PFHpA	7.707	367.0	322.0	322.0	2.5	2.5
EIS	13C8-PFOA	8.64	421.0	376.0	376.0	2.5	2.5
EIS	13C9-PFNA	9.119	472.0	427.0	427.0	1.25	1.25
EIS	13C6-PFDA	9.51	519.0	474.0	474.0	1.25	1.25
EIS	13C7-PFUnA	9.899	570.0	525.0	525.0	1.25	1.25
EIS	13C2-PFDoA	10.458	615.0	570.0	570.0	1.25	1.25
EIS	13C2-PFTeDA	12.236	715.0	670.0	670.0	1.25	1.25
EIS	13C3-PFBS	4.835	302.0	80.0	99.0	2.5	2.5
EIS	13C3-PFHxS	8.775	402.0	80.0	99.0	2.5	2.5
EIS	13C8-PFOS	9.71	507.0	80.0	98.9	2.5	2.5
EIS	13C2-4:2FTS	4.405	329.0	309.0	80.9	5	5
EIS	13C2-6:2FTS	8.397	429.0	409.0	80.9	5	5
EIS	13C2-8:2FTS	9.323	529.0	509.0	80.9	5	5
EIS	13C8-PFOSA	11.438	506.0	78.0	78.0	2.5	2.5
EIS	D3-NMeFOSA	13.174	515.0	219.0	168.9	2.5	2.5
EIS	D5-NEtFOSA	13.467	531.0	219.0	168.9	2.5	2.5
EIS	D3-NMeFOSAA	9.489	573.0	419.0	419.0	5	5
EIS	D5-NEtFOSAA	9.645	589.0	419.0	419.0	5	5
EIS	D7-NMeFOSE	12.997	623.1	59.0	59.0	25	25
EIS	D9-NEtFOSE	13.282	639.1	59.0	59.0	25	25
EIS	13C3-HFPO-DA	5.688	287.0	169.0	185.0	10	10
NIS	13C3-PFBA	2.023	216.0	172.0	N/A	5	5
NIS	13C2-PFHxA	4.905	315.0	270.0	119.0	2.5	2.5
NIS	13C4-PFOA	8.637	417.0	172.0	N/A	2.5	2.5
NIS	13C5-PFNA	9.118	468.0	423.0	N/A	1.25	1.25
NIS	13C2-PFDA	9.51	515.0	470.0	N/A	1.25	1.25
NIS	18O2-PFHxS	8.775	403.0	84.0	N/A	2.5	2.5
NIS	13C4-PFOS	9.708	503.0	80.0	99.0	2.5	2.5
--	TDCA	8.401	498.3	124.0	80.0		

Results and Discussion

Calibration was performed for all targeted PFAS using a seven-point calibration curve and following the recommendations included in EPA draft method 1633; concentrations ranged from 0.2 – 250 ng/mL for targets, 1.25 – 25 ng/mL for EISs, and 1.25 – 5 ng/mL as shown in Table 3. The LOQs for each target compound are listed in Table 3 and equivalent to CS1; all LOQs meet the concentration listed in EPAM1633.

Figure 1 shows the MRM transitions from CS1; this figure demonstrates the separation and peak shape of targets at the lowest concentration standard included in the calibration curve.

Table 4 summarizes signal-to-noise (S/N), accuracy, and instrument linearity for the initial calibration, and the instrument detection limit (IDL) for all target compounds. The S/N of CS1 ranged from 8 to >30,000; this demonstrates that lower concentrations of the targeted PFAS could be easily measured with the LCMS-8050. Accuracy for all target compounds in CS1 ranged between 80% and 116%, exceeding results reported and accepted in EPAM1633. The instrument linearity for the calibration curve was evaluated by calculating the relative standard error (RSE); RSE for all target compounds was ≤20% in accordance with EPAM1633. The IDLs and %RSDs at the IDLs for each target are shown in Table 4. The IDLs ranged between 1.9 ng/mL (7:3 FTCA) and 0.01 ng/mL (multiple analytes), approximately one order of magnitude lower than the LOQs.

Repeatability was evaluated at CS1 (n=7), CS4 (n=6) and CS7 (n=6). In CS1, the lowest calibration standard, 75% of the targeted compounds showed a %RSD of less than 20%; the %RSD of 12.5% of the targets ranged between 20% and 30%, and %RSD was >30% for the remaining 12.5% of the targets (5 compounds).

The %RSD for all targets in CS4 and CS7 (i.e., the mid-point and high point of the calibration curve, respectively) was <20%, with most compounds showing a %RSD of less than 10%.

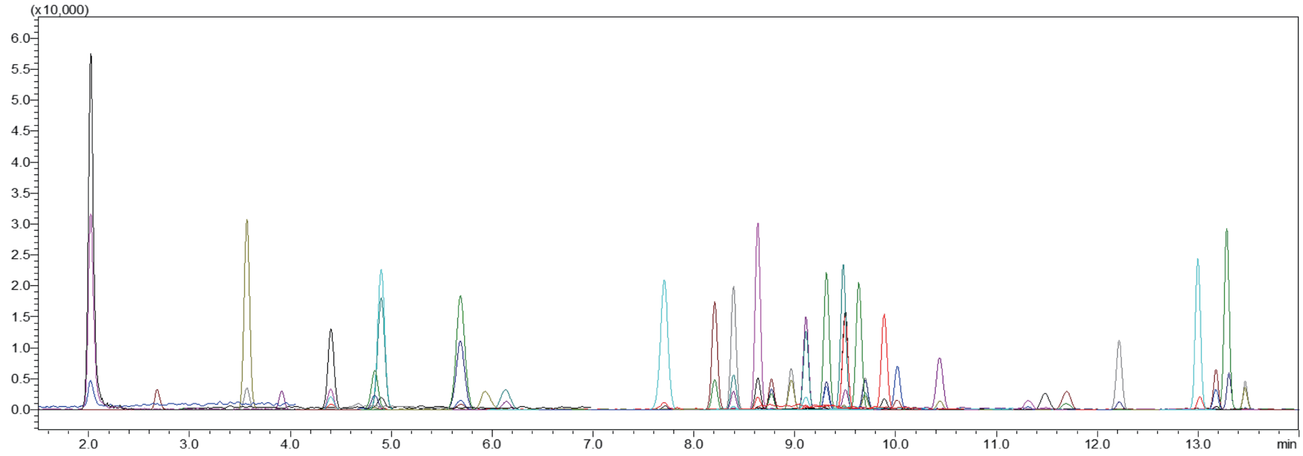


Figure 1. MRM transitions of all PFAS (target, EIS, NIS) at the lowest calibration standard (CS1) concentration.

Table 4. Method performance summary of neat standards using the Shimadzu LCMS-8050.

Name	CS1 Average S/N (n=7)	%Accuracy CS1 (n=7)	RSE	IDL ng/mL (n=7)	%RSD IDL (n=7)
PFBA	12.36	102.68	3.64	0.16	5.77
PFPeA	18.73	105.25	2.98	0.18	12.52
PFHxA	25.93	106.18	4.75	0.13	21.55
PFHpA	7.85	102.14	4.77	0.11	15.03
PFOA	16.56	103.79	8.42	0.08	11.19
PFNA	26.59	105.89	7.06	0.10	15.65
PFDA	32.95	105.47	9.36	0.07	11.79
PFUnA	44.23	104.42	7.51	0.10	15.78
PFDoA	56.80	99.14	6.67	0.15	20.17
PFTrDA	47.26	109.92	6.73	0.11	16.01
PFTeDA	145.08	86.49	7.07	0.08	10.89

Name	CS1 Average S/N (n=7)	%Accuracy CS1 (n=7)	RSE	IDL ng/mL (n=7)	%RSD IDL (n=7)
PFBS	106.61	104.01	8.32	0.12	23.82
PFPeS	57.58	116.00	8.97	0.12	16.54
PFHxS	287.07	103.13	9.56	0.18	31.19
PFHpS	96.74	100.94	10.68	0.07	10.80
PFOS	12.97	104.09	11.42	0.21	50.62
PFNS	32.95	113.14	10.88	0.12	16.77
PFDS	101.25	94.83	11.37	0.18	34.16
PFDoS	62.67	109.76	6.70	0.17	19.79
4:2FTS	28989.63	83.92	15.20	0.24	9.70
6:2FTS	24990.49	80.44	15.82	0.50	26.45
8:2FTS	34754.78	108.43	10.40	0.32	12.78
PFOSA	304.11	113.56	11.92	0.22	35.66
NMeFOSA	107.66	110.25	10.66	0.14	19.22
NEtFOSA	537.58	107.01	9.04	0.12	15.94
NMeFOSAA	111.13	102.36	5.69	0.09	15.71
NEtFOSAA	18.72	112.22	10.59	0.18	34.30
NMeFOSE	150.32	92.74	6.52	0.85	11.82
NEtFOSE	158.18	101.23	6.01	0.38	5.63
HFPO-DA	626.89	100.05	4.20	0.22	9.04
ADONA	2149.52	101.71	3.84	0.13	5.14
9Cl-PF3ONS	486.71	94.21	4.70	0.16	6.11
11Cl-PF3OUdS	671.33	111.72	9.08	0.35	14.03
3:3 FTCA	3924.23	104.45	8.49	0.31	12.02
5:3 FTCA	196.34	112.09	6.11	0.84	5.14
7:3 FTCA	1179.93	111.16	5.90	1.89	11.20
PFEESA	87.20	105.06	3.19	0.11	7.91
PFMPA	476.40	104.14	2.67	0.09	6.87
PFMBA	38489.86	113.02	7.73	0.14	10.19
NFDHA	2035.17	85.56	8.37	0.24	17.57

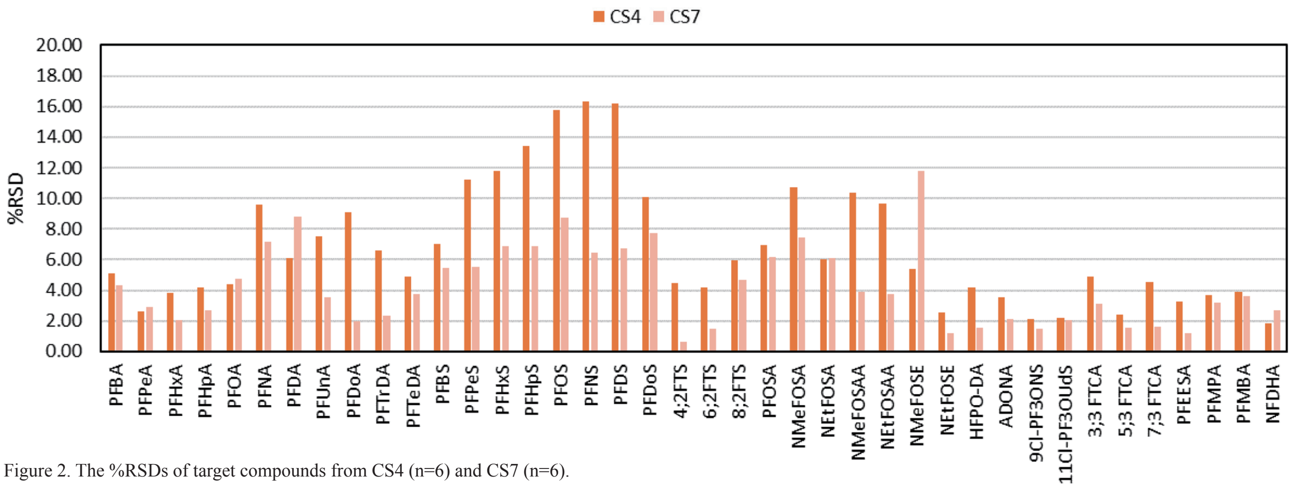


Figure 2. The %RSDs of target compounds from CS4 (n=6) and CS7 (n=6).

Two types of aqueous samples were analyzed following the extraction procedure outlined in EPAM1633: ultrapure reagent water (n=5) and wastewater (n=7). Both sample types were spiked at concentrations equal to those in CS1. Precision and accuracy are normally evaluated by spiking the samples with a mid-level concentration. CS1 was used instead to better assess the method performance at a more challenging concentration.

Table 5 shows the %recoveries of the EISs in the ultrapure reagent water and wastewater samples. In ultrapure reagent water, the recoveries from all EISs were within 50% and 150%, except D5-NEtFOSA (48%). The recoveries of the EIS in wastewater were slightly lower for all compounds than in ultrapure reagent water. The %recoveries of the EIS in both types of samples exceed the reported ranges in EPAM1633. Figure 6 shows the recovery of the target compounds spiked at CS1 concentrations in ultrapure reagent water (n=5) and wastewater (n=3), with error bars displaying the %RSD. The %recovery and %RSD for all the targeted PFAS in ultra-pure water and wastewater, except for 3:3 FTCA, are within acceptable ranges for the analysis of PFAS in environmental samples when spiked at the lowest standard of the calibration curve.

Table 5. The %recoveries of EIS in ultra-pure water and wastewater samples.

EIS	% Recovery CS1 Spike in UPW(n=5)	% Recovery CS1 Spike in WW(n=7)
13C4-PFBA	94.09	32.78
13C5-PFPeA	96.48	85.57
13C5-PFHxA	99.69	93.64
13C4-PFHpA	100.51	91.56
13C8-PFOA	104.16	93.28
13C9-PFNA	107.61	98.75
13C6-PFDA	104.58	89.06
13C7-PFUnA	97.69	72.58
13C2-PFDoA	82.12	58.55
13C2-PFTeDA	65.92	42.09
13C3-PFBS	96.04	92.31
13C3-PFHxS	105.44	100.97
13C8-PFOS	109.04	99.40
13C2-4:2FTS	86.72	82.76
13C2-6:2FTS	93.63	83.95
13C2-8:2FTS	93.09	84.67
13C8-PFOSA	72.53	85.68
D3-NMeFOSA	52.33	54.40
D5-NEtFOSA	47.86	44.46
D3-NMeFOSAA	102.61	79.32
D5-NEtFOSAA	91.70	61.64
D7-NMeFOSE	56.78	49.43
D9-NEtFOSE	53.77	45.35

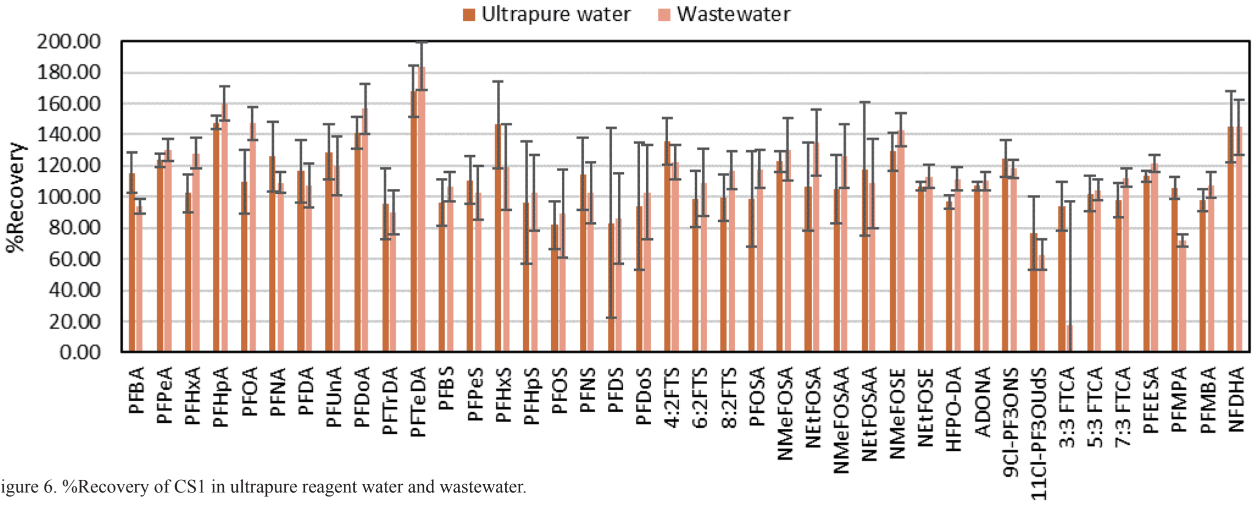


Figure 6. %Recovery of CS1 in ultrapure reagent water and wastewater.

Summary and Conclusions

The Shimadzu LCMS-8050 was evaluated for its ability to analyze PFAS in wastewater samples in accordance with EPAM1633. The results demonstrated the excellent performance of the LCMS-8050 for key quality control and performance parameters defined in the draft method and indicate that lower detection limits are easily achievable.

References

1. United States Environmental Protection Agency, Office of Water. 3rd Draft Method 1633 Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC/MS/MS, 2022. https://nepis.epa.gov/Exe/ZyPURL.cgi?Dockey=P101687F.txt (accessed June 1, 2023)

2. https://www.ssi.shimadzu.com/products/liquid-chromatograph-mass-spectrometry/lc-ms-system/lcms-ms-ms-method-package-pfas/index.html

3. https://www.ssi.shimadzu.com/sites/ssi.shimadzu.com/files/pim/pim_document_file/ssi/technical/technical_reports/16761/2022_PFAS_Essentials_0.pdf

Environmental Analysis

Accurate Analysis of PFOA, PFHxS, and PFOS in Drinking Water Using a Triple Quadrupole LC/MS/MS

Yui Higashi, Nami Iwasa
Solutions COE, Analytical & Measuring Instruments Division,
Shimadzu Corporation

User Benefits

- PFOA, PFOS, and PFHxS concentrations at 0.2 ng/L (concentration in water sample) can be quantitated using an optimized analytical conditions.
- The LCMS-8050RX is equipped with CoreSpray technology, which improves consistency and reliability, so concentrations in drinking water can be quantitated with excellent recovery rates and repeatability for recovery tests (1 ng/L in samples).

Introduction

Perfluorooctanoic acid (PFOA), perfluorohexanesulfonic acid PFHxS), and perfluorooctanesulfonic acid (PFOS) are types of organofluorine compounds that are used for a wide range of applications, such as water repellents, waterproof materials, and surfactants. Due to their chemical stability, there is concern that their environmental persistence could result in harmful accumulations in humans. Therefore, PFOA and PFOS are included in the water quality management goal items, and PFHxS is listed as an item for consideration in Japan.

This article describes the results from analyzing PFOA, PFOS, and PFHxS in drinking water that have been concentrated by a factor of 1000 based on the testing method of the water quality management in Japan.

Sample Preparation

The drinking water samples were pretreated by adding 50 µL of a ^{13}C-labelled internal standard solution (a final concentration of 10 ng/L) and then using an anion exchange solid phase column for solid phase extraction. The eluate from the solid phase column was concentrated by drying with nitrogen gas stream, and then bring the volume to 0.5 mL with methanol for analysis. The sample preparation process is shown in Fig. 1. In this case, AquaTrace automatic solid phase extraction system from GL Sciences was used to automate all the steps, from conditioning to concentrating.

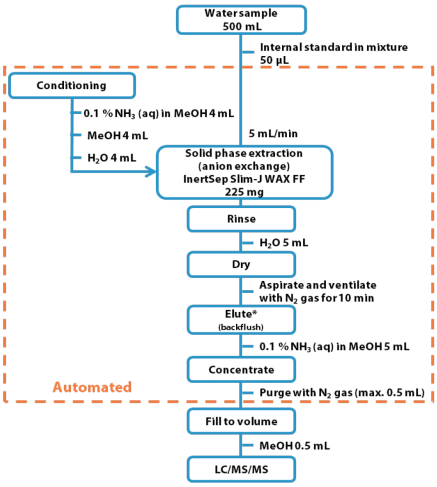


Fig. 1 Sample Preparation Process
(*The solid phase column was manually inverted for elution by backflushing.)

LCMS-8050RX

Samples were analyzed using a triple-quadrupole mass spectrometer, LCMS-8050RX (Fig. 2). The LCMS-TQ8050RX features the new CoreSpray technology, which generates a more uniform nebulizer flow than previous models, and the highly accurate ionization technology enables more consistent measurements.

Fig. 2 LCMS-8050RX

Analytical Conditions

The PFOA, PFHxS, and PFOS were measured based on the HPLC and MS analytical conditions listed in Table 1. Measuring PFAS requires particular care due to the risk of contaminations from the system, the mobile phase, and other sources. For this article, a delay column was installed between the mixer and autosampler to inhibit the effects of PFAS contamination from the system. A reagent intended for PFOS/PFOA analysis was used in the mobile phase.

Table 1 Analytical Conditions

UHPLC (Nexera™-X3 System)

Analytical Column	: Shim-pack™GIST-HPC18-AQ (150 mm ×2.1 mm I.D., 3 µm) (P/N: 227-30765-04)
Solvent Delay Column	: Shim-pack XR-ODS II (2 mm ×75 mm, 2.2 µm) (P/N: 228-41623-91)
Mobile Phase A	: 10 mM Ammonium Acetate in H_2O
Mobile Phase B	: Acetonitrile
Gradient Program	: B 40 % –70 % (13 min) –100 % (13.01 –17 min) – 40 % (17.01 –21 min)
Flowrate	: 0.2 mL/min
Column Temp.	: 40 °C
Injection Volume	: 1 µL
Run Time	: 21 min

MS (LCMS-8050RX)

Ionization	: ESI (Negative mode)	
Mode	: MRM	
Probe Voltage	: -1 kV	
Nebulizing Gas	: 3 L/min	
Drying Gas Flow	: 3 L/min	
Heating Gas Flow	: 15 L/min	
DL Temp.	: 150 °C	
Block Heater Temp.	: 250 °C	
Interface Temp.	: 300 °C	
Probe Position	: +2 mm	
MRM Transition	: PFOA	*m/z* 412.90 > 169.10
	PFHxS	*m/z* 399.00 > 79.95
	PFOS	*m/z* 498.90 > 79.95
	$^{13}C_8$-PFOA	*m/z* 420.90 > 375.85
	$^{13}C_6$-PFHxS	*m/z* 405.00 > 79.95
	$^{13}C_8$-PFOS	*m/z* 506.90 > 80.00

MRM Chromatograms

A standard solution containing 0.2 µg/L each of PFOA, PFHxS, and PFOS (equivalent to 0.2 ng/L in water samples) was measured based on the analytical conditions shown in Table 1. The resulting MRM chromatograms are shown in Fig. 3. With an optimized conditions, good separation from the PFHxS and PFOS branched chains was achieved.

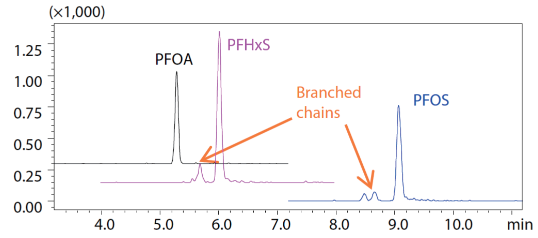


Fig. 3 MRM Chromatograms of Each Component (0.2 µg/L Each)

Calibration Curves

Fig. 4 shows the calibration curves for PFOA, PFHxS, and PFOS that are based on the internal standard method for the concentration range from 0.2 to 20 µg/L (7 points that are equivalent to 0.2 to 20 ng/L in the water samples). The calibration curve correlation coefficient (R) was greater than 0.9996 for all three compounds. Average concentration values at each calibration point (n = 5) were 80 to 120% of the true value, and repeatability was less than 5%RSD of the concentration. The area repeatability values (area %RSD value) at 0.2 µg/L, the lowest point of the calibration curves, was also less than 5%. Excellent calibration performances were confirmed.

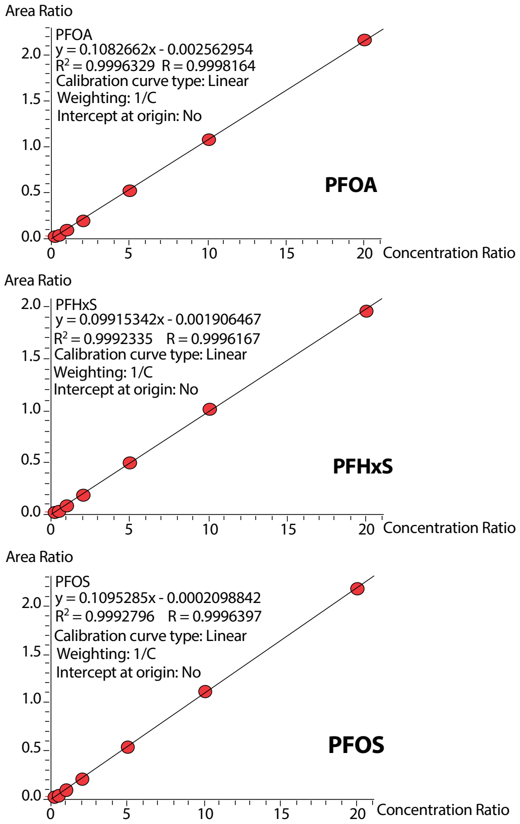


Fig. 4 Calibration Curves for Each Component (0.2 to 20 µg/L)

Recovery Test

Drinking water samples spiked with 1 ng/L or 5 ng/L concentrations of PFOA, PFHxS, or PFOS were prepared and pretreated according to the process indicated in Fig. 2. The results are indicated in Table 2. Recovery rates ranged from 82 to 103% and repeatability was less than 4% of concentration, which were both good results that confirmed drinking water samples can be analyzed accurately.

Table 2 Recovery Test Results (n = 5)

Component	1 ng/L Concentration in Water Sample		5 ng/L Concentration in Water Sample	
	Recovery (%)	Repeatability (%RSD)	Recovery (%)	Repeatability (%RSD)
PFOA	82.4	2.5	82.8	2.2
PFHxS	98.6	3.5	92.8	2.0
PFOS	102.9	2.3	94.2	0.9

Conclusion

By using LCMS-8050RX, adequate sensitivity was achieved for analyzing the concentration of 0.2 ng/L (equivalent to 0.2 ng/L in the water samples).

Good recovery rates and reproducibility results were obtained from recovery tests for drinking water with 1 ng/L concentrations using a 1000-fold concentration process. In this case, sample preparation was automated by using the AquaTrace system, which simplified operations and reduced the labor time. LCMS-8050RX system, which is equipped with CoreSpray technology, provided more consistent ionization and ion loading performance, and highly reliable analytical results were achieved.

The above results confirm that LCMS-8050RX system can be used to analyze PFOA, PFHxS, and PFOS in drinking water efficiently and accurately.

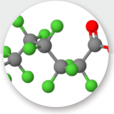
Shimadzu Selection

This is a selection of articles by Shimadzu relating to the environment and PFAS. From posters presented at the international conferences to e-books, they include everything from the latest applications to the latest publications. In these articles, you will find various applications of our instruments as well as cutting-edge technologies. Please obtain the articles of your interest through the links on the titles.

PFAS Analysis: Application Notebook

Per- and polyfluoroalkyl substances (PFAS) are currently of great public health and environmental concern. Because PFAS are ubiquitous and commonly used in materials routinely employed for chemical analysis, laboratories are in need of streamlined protocols to minimize background contamination from these chemicals and quickly generate accurate data. This notebook provides a number of examples showcasing the use of ultra fast LC-MS/MS for the analysis of PFAS according to multiple methods.

watch the video »

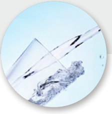


Systematic Study of Techniques to Minimize PFAS Background Interferences

Public concern over PFAS has spurred efforts by EPA, FDA, etc. to standardize PFAS quantification methods in various environmental samples. However, analyzing these ubiquitous contaminants with LC-MS is challenging due to pervasive lab-based PFAS contamination, compromising accuracy. Common culprits include LC components, solvents, and consumables. Achieving sub-ppt level accuracy requires a combination of measures, including rigorously evaluated consumables and a delay column used with a sensitive LCMS system. While other measures are important, the delay column is crucial for minimizing background PFAS interference. It should: 1) have minimal backpressure, 2) reliably delay PFAS, 3) be stable at high pressures, and 4) offer excellent reproducibility. This research will assess the impact of delay columns and consumables on EPA methods 533, 537.1, and 1633.

Analysis of PFAS in water using HS-SPME GCMS

PFAS are environmental pollutants with recognized health and environmental concerns due to their probable toxicity, persistence, and ubiquitousness throughout the environment, and consumer and industrial products. PFAS encompass a large family of chemicals with varying physicochemical properties. These properties not only affect their toxicity and environmental fate, but also complicates chemical analysis.
In this work, a Head-Space Solid Phase Microextraction-Gas Chromatography/Mass Spectrometry (HS-SPME GCMS) analytical method was developed to analyze six classes of volatile PFAS in water. This HS-SPME method presents the advantage of simplifying sample extraction and handling for the analysis of volatile PFAS in water, hence, minimizing operational costs, potential errors, and risk of sample-cross contamination.



Handbooks of Analysis Procedures for EPA methods

Introducing the handbooks for EPA Method 533 and EPA Method 537.1, offering detailed guidance on analysis procedures. They include step-by-step instructions for determining the Minimum Reporting Level (MRL), preparing standard solutions, and setting up QC samples. The handbooks also cover reagent preparation, sample extraction, and calibration curve samples.

Analysis Procedures for EPA method 533

Analysis Procedures for EPA method 537.1

Shimadzu PFAS Website

PFAS are a group of persistent and harmful chemicals that can be commonly found in the environment globally. These anthropogenic chemicals can come from various sources such as manufacturing, industrial applications, food contact materials and consumer products, and eventually end up and accumulate in the water, soil and living organisms. With concerns rising quickly about the health risks associated with PFAS exposure, it is important to ensure proper monitoring of these chemicals.

LC/MS/MS Method Package for PFAS in Drinking Water

The United States Environmental Protection Agency (US EPA) has standardized two methods for the analysis of PFAS in drinking water: EPA Method 537.1 and 533, encompassing a total of 29 PFAS. These methods were used as reference for creating a vetted LC/MS/MS Method Package for Analyzing PFAS in Drinking Water. This method package includes ready-to-use analytical conditions for EPA Methods 533 and 537.1, examples of analytical procedures for the methods, and various other information, such as precautions for sample preparation and analysis. Using this product, 52 PFAS compounds* in drinking water can be analyzed.

* Including internal standard substances, surrogates, etc.

Revealing the Invisible Web

—Solutions for an Efficient Analysis of PFAS—

PFAS are a group of over 6000 synthetic chemicals with fluorinated carbon chains and various functional groups. First produced in the 1940s, they are released into the environment during manufacturing, use, and disposal of PFAS-containing products. Their stable carbon-fluorine bonds make them resistant to degradation, leading to accumulation in the environment and living organisms, earning them the nickname "forever chemicals." Today, PFAS are found in the products we use, the air we breathe, the food we eat, the water we drink, and even inside our bodies. New technologies are being developed to prevent their indefinite environmental presence. These technologies, along with proper PFAS monitoring, are essential to mitigate their harmful effects on humans and ecosystems.

Analysis of Perfluorooctane Sulfonamidoethanols (FOSEs) and Fluorotelomer Alcohols (FTOHs) in Textiles by LC/MS/MS

LC/MS/MS and GC/MS have been used in analyses of various PFCs or PFAS in various environmental samples and consumer products. A LC/MS/MS method was reported previously for simultaneous analysis of 24 PFCs in textiles. Perfluorooctane sulfonamidoethanols (FOSEs) and fluorotelomer alcohols (FTOHs) belong to less-polar PFCs and may degrade to form PFOS and PFOA. Normally, they are analysed using GC/MS with chemical ionisation (CI) due to their non-polar properties. In this study, an alternative method using LC/MS/MS was developed for the analysis of four FTOHs and two FOSEs in textiles to respond to the recent demands in testing analysis for textile and consumer products.

Analytical Solutions for Microplastics

—Aiming to Improve the Marine Environment—

Tiny plastic fragments on the order of several µm to 5 mm are referred to as microplastics. In recent years, there have been growing concerns that microplastics are having a negative impact on marine environments and ecosystems. Not only the microplastic materials themselves but also the additives they contain, as well as harmful substances absorbed in the environment, may have a latent impact on humans via the food chain. Shimadzu provides analytical and measuring instruments for the study of a variety of plastic materials: for R&D, characteristic evaluation of raw materials, quality control for plastic products, and deterioration analysis. With these diverse techniques, Shimadzu provides optimal solutions for microplastics research.

Analytical Solutions for Artificial Photosynthesis

—Photocatalyst Characterization and Product Quantification—

Artificial photosynthesis uses photocatalysts and sunlight to mimic natural photosynthesis, converting light energy into useful compounds. It's seen as a promising renewable energy technology, especially for producing "green hydrogen" without CO_2 emissions. It can also generate compounds like carbon monoxide, alcohols, formic acid, and hydrocarbons by reducing CO_2. However, challenges in cost and efficiency hinder its societal implementation. Achieving a 10% energy conversion efficiency requires developing effective photocatalysts and efficient reaction systems. Shimadzu Corporation supports this technology with advanced analytical measurement techniques.

Towards Carbon Neutrality

With our Products and Technologies, Shimadzu Contributes to Solving Problems in a Diverse Range of Fields. We contribute to technological development and quality control in fields such as hydrogen fuels, biofuels, wind power generation, and other renewable energies, as well as automobiles and storage batteries, which are all indispensable for achieving carbon neutrality. In addition, we are actively adopting renewable energy in our business activities, aiming to reduce CO_2 emissions.

Latest topics 1

Tackling the Global Microplastic Challenge

The term microplastics refers to tiny plastic particles in the environment, with a diameter between 1 μm and 5 mm. Microplastics are divided into two categories: primary microplastics like microbeads and pellets, which are produced industrially at a small size; and secondary microplastics, which become smaller as plastic products are broken down through deterioration from exposure to ultraviolet rays and the application of physical forces by water waves for example. Both types are consumed by living organisms and accumulate in the internal organs. Plastic additives and harmful chemical substances adhering to the surfaces are then released, raising concerns not just about their impact on the human body but on the ecosystem and the environment as a whole. The problem of environmental contamination caused by microplastics requires scientific knowledge, including assessments of the amount and distribution of plastics released into the environment, identifying their sources, and evaluating their effects on organisms and ecosystems. Various research and surveys are being conducted around the world.

However, samples collected from the environment contain not only plastic but also various impurities and adhesions. To obtain correct analytical results, microplastics must be accurately extracted and analyzed by performing a sequence of preparation processes prescribed depending on the sample. Preparation methods for microplastics include acid treatment, in which vegetation, microorganisms, and other organic substances are dissolved; and density separation, which separates the microplastics from sand, clay, and other inorganic substances.

However, preparation processes, which are primarily performed manually, are complicated, and the results differ depending on the analyst, which is a problem.

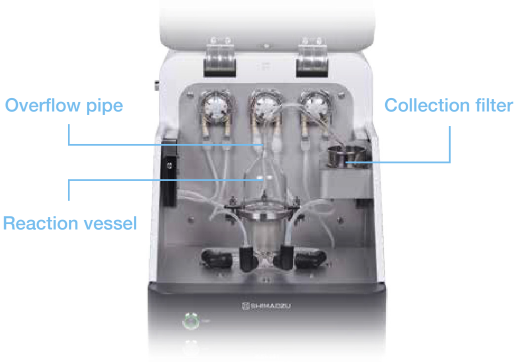
Microplastics collected on the beach

MAP-100 the World's First Microplastic Automatic Preparation Device

In 2023, Shimadzu released the MAP-100. This microplastic automatic preparation device automates typical preparation methods for extracting microplastics from samples of environmental epipelagic water, thereby providing labor savings, and improving reproducibility and safety. For samples containing microplastics obtained from environmental water, the MAP-100 automates the acid treatment and density separation process, as well as the collection of particles in the sample. This instrument complies with the preparation methods noted in the "Guidelines for River and Lake Microplastic Monitoring Methods" released in March 2023 by the Japanese Ministry of the Environment.

Samples that have been prepared are then analyzed using various analytical instruments. Shimadzu provides a variety of instruments for performing analyses and evaluations related to microplastics, from Fourier transform infrared spectrophotometers (FT-IR) for identifying the types of plastics to particle imaging systems for measuring the number and shapes of particles. In addition to these instrument lineups, this product, which automates the preparation process, supports the implementation of high-accuracy analysis work. Shimadzu provides wide-ranging solutions for everything from preparation to analysis and measurement, thereby working to solve the problem of microplastics, which is expanding globally.

For more details, visit MAP-100



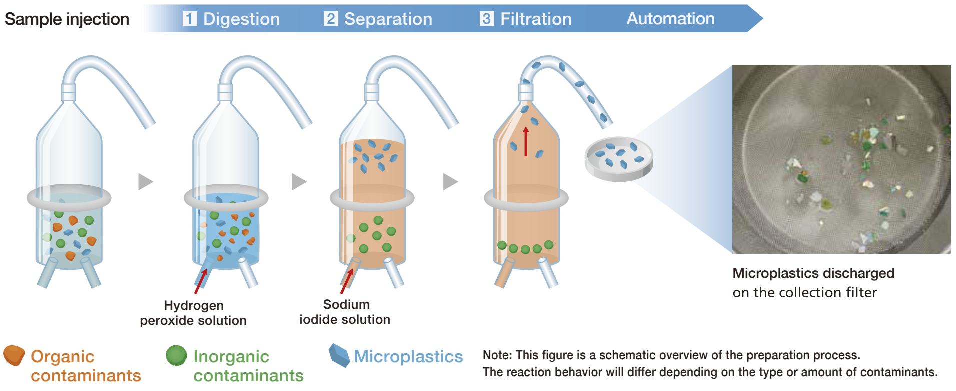
Features

1. Replaces Troublesome Manual Work with a Specialized Unit

The preparation process for microplastic analysis is time-consuming and collecting particles after treatment using tweezers is difficult. This product controls the flow line automatically and collects all the tiny microplastic particles. This not only eliminates dependency on individual skills and heightens the reliability of the analysis, but also eliminates manual labor involving acidic reagents, thereby ensuring safety.

2. Compatibility with Guidelines

Preparation by this product is compliant with the “River and Lake Microplastics Investigative Guidelines” published by the Japanese Ministry of the Environment. Automating the preparation process for a standard microplastics analysis contributes to the assessment of the actual distribution of these microplastics.

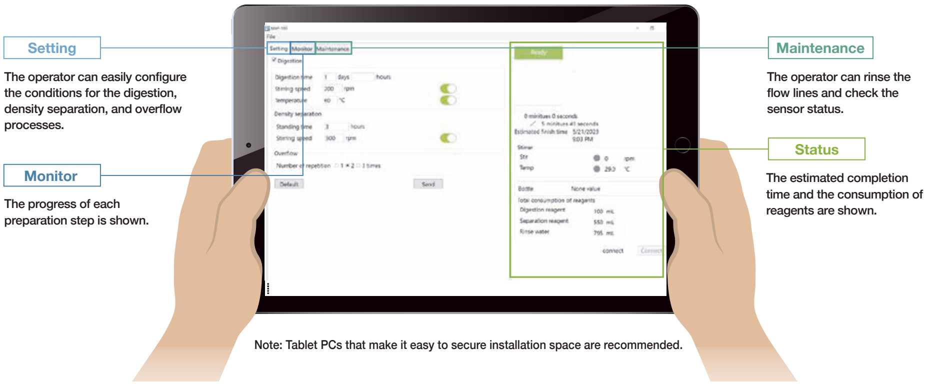


Microplastics discharged on the collection filter

Note: This figure is a schematic overview of the preparation process.
The reaction behavior will differ depending on the type or amount of contaminants.

3. Simple, Intuitive Software

The processing time and conditions must be adjusted according to the amount and condition of substances adhering to the sample. The control software for this product consists of simple, easy-to-operate windows, enabling easy configuration of conditions. In addition, the processing status and finishing time can be checked on screen, improving the efficiency of the user’s workflow.



☑ Digestion
Digestion time 1 days hours
Stirring speed 200 rpm
Temperature 60 ℃
Density separation
Standing time 3 hours
Stirring speed 300 rpm
Overflow
Number of repetition ○ 1 ● 2 ○ 3 times

0 minitues 0 seconds
5 minitues 41 seconds
Estimated finish time 5/21/2023 9:03 PM
Stirrer
Stir 0 rpm
Temp 29.3 ℃
Bottle None value
Total consumption of reagents
Digestion reagent 100 mL
Separation reagent 550 mL
Rinse water 795 mL

Setting
The operator can easily configure the conditions for the digestion, density separation, and overflow processes.

Monitor
The progress of each preparation step is shown.

Maintenance
The operator can rinse the flow lines and check the sensor status.

Status
The estimated completion time and the consumption of reagents are shown.

Note: Tablet PCs that make it easy to secure installation space are recommended.

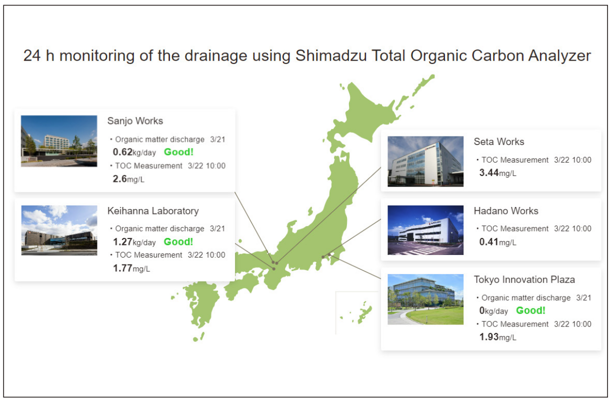
Latest topics 2

Effluent monitoring system that reduces environmental load

Monitoring water discharged from plants and living environment is extremely important for preventing water pollution. Especially, water discharged for business purposes that releases toxic substances into our living environment needs to be controlled following strict standards.

Each business location of Shimadzu Japan uses effluent monitoring systems to monitor the effluent 24 hours a day. The state of business locations in Japan can be checked in real time on the management screen. In Shimadzu Tokyo Innovation Plaza which opened in October 2022, effluent conditions are strictly controlled based on the company's own standards in addition to standards of Kawasaki City. The site uses a system that stops water discharge and stores water in a tank when any abnormality is detected in order to prevent water from being released externally. Using the system, in addition to monitoring, effluent that may burden the environment can be properly treated before draining it.

In order to continuously monitor effluent, each business location of Shimadzu Japan uses the Shimadzu TOC-4200 On-line TOC Analyzer as well as TNP-4200 On-line Total Nitrogen and Phosphorus Analyzer according to the purposes.



24 h monitoring of the drainage using Shimadzu Total Organic Carbon Analyzer

Sanjo Works
- Organic matter discharge 3/21
0.62kg/day Good!
- TOC Measurement 3/22 10:00
2.6mg/L

Keihanna Laboratory
- Organic matter discharge 3/21
1.27kg/day Good!
- TOC Measurement 3/22 10:00
1.77mg/L

Seta Works
- TOC Measurement 3/22 10:00
3.44mg/L

Hadano Works
- TOC Measurement 3/22 10:00
0.41mg/L

Tokyo Innovation Plaza
- Organic matter discharge 3/21
0kg/day Good!
- TOC Measurement 3/22 10:00
1.93mg/L

TOC-4200 in Shimadzu Tokyo Innovation Plaza

Shimadzu Corporation provides monitoring equipment such as flue gas analyzer in addition to equipment for water quality management in order to contribute to the reduction in environmental inventory from various angles.

On-line TOC Analyzer TOC-4200

Shimadzu Tokyo Innovation Plaza

pH monitoring tank (Right) and emergency water tank (Left)

TOC Product Selection Guide

We offer a complete TOC instrument lineup, including our TOC-L series, which feature the highest level of detection sensitivity available by using the combustion catalytic oxidation method; the TOC-4200 online water quality analyzer, which supports a wide range of samples and boasts advanced operability; and our compact eTOC series on-line TOC analyzer, perfect for ultra-pure water measurement.

Product Selection Guide helps you to find the most optimal TOC Analyzer from various models. It takes only 1 minute to see the results.

Click the button below to get started now.

TOC-L Configurator

The TOC-L series consists of a high-sensitivity model with a detection limit of 4 μg/L, making them suitable for various applications, including pure water measurements, and a standard model designed with cost performance in mind. Each analyzer is available as an LCD and keyboard-equipped standalone model as a PC-controlled model.

Click the button below to verify the optimal TOC-L system configuration and request a free quotation.

Latest topics 3

Driving Green Innovation: Shimadzu Group Integrates Cellulose Fibers in Analytical Instruments and Acquires El-design Co., Ltd. for Groundbreaking Packaging Project

Shimadzu Corporation is actively promoting eco-friendly functional materials and systems to reduce our environmental impact. We are committed to prioritizing sustainable practices and advocating for the adoption of green solutions. Together, we can create a greener future.

In a World's First, Components Compounded with Cellulose Fibers are being Adopted for Analytical and Measuring Instruments

In a world's first, Shimadzu Corporation is adopting a cellulose fiber reinforced flame resistant composite resin, a functional, environmentally friendly material, for our analytical and measuring instruments. This resin was created based on GREEN CHIP CMF™*[1] developed collaboratively by Tomoegawa Co., Ltd. (Chuo City, Tokyo) and FP Chemical Industry Co.,Ltd. (Fuji City, Shizuoka Prefecture) in 2020. The development of this plastic*[2] was the result of a challenge by Shimadzu to improve the flame resistance, which was then solved by the three companies. The materials will be used in the configuration units of 15 models in the Nexera series of liquid chromatographs shipped starting in late November.

GREEN CHIP CMF is a sustainable material compounded with cellulose fibers, thereby limiting the use of petroleum derived resins, leading to a reduction in CO_2. Because of its high strength, components can be molded more thinly, reducing the weight of products. However, the exterior and components of analytical instruments and other electrical equipment should be flame resistant from the perspective of safety. Both petroleum derived plastics and plant derived cellulose are flammable, and increasing their respective flame resistance was problematic. Tomoegawa Co., Ltd., FP Chemical Industry Co.,Ltd., and Shimadzu Corporation spent three years developing a compounding technique for combining resin and cellulose, both of which are flammable materials, but increasing their flame resistance while maintaining a certain level of strength.

Shimadzu Corporation is initially adopting the product for their liquid chromatographs, but has plans to increase the usage ratio of sustainable materials in various products.

*1 GREEN CHIP CMF was developed collaboratively by Tomoegawa Co., Ltd. and FP Chemical Industry Co.,Ltd.
*2 UL94_V-0 equivalent.

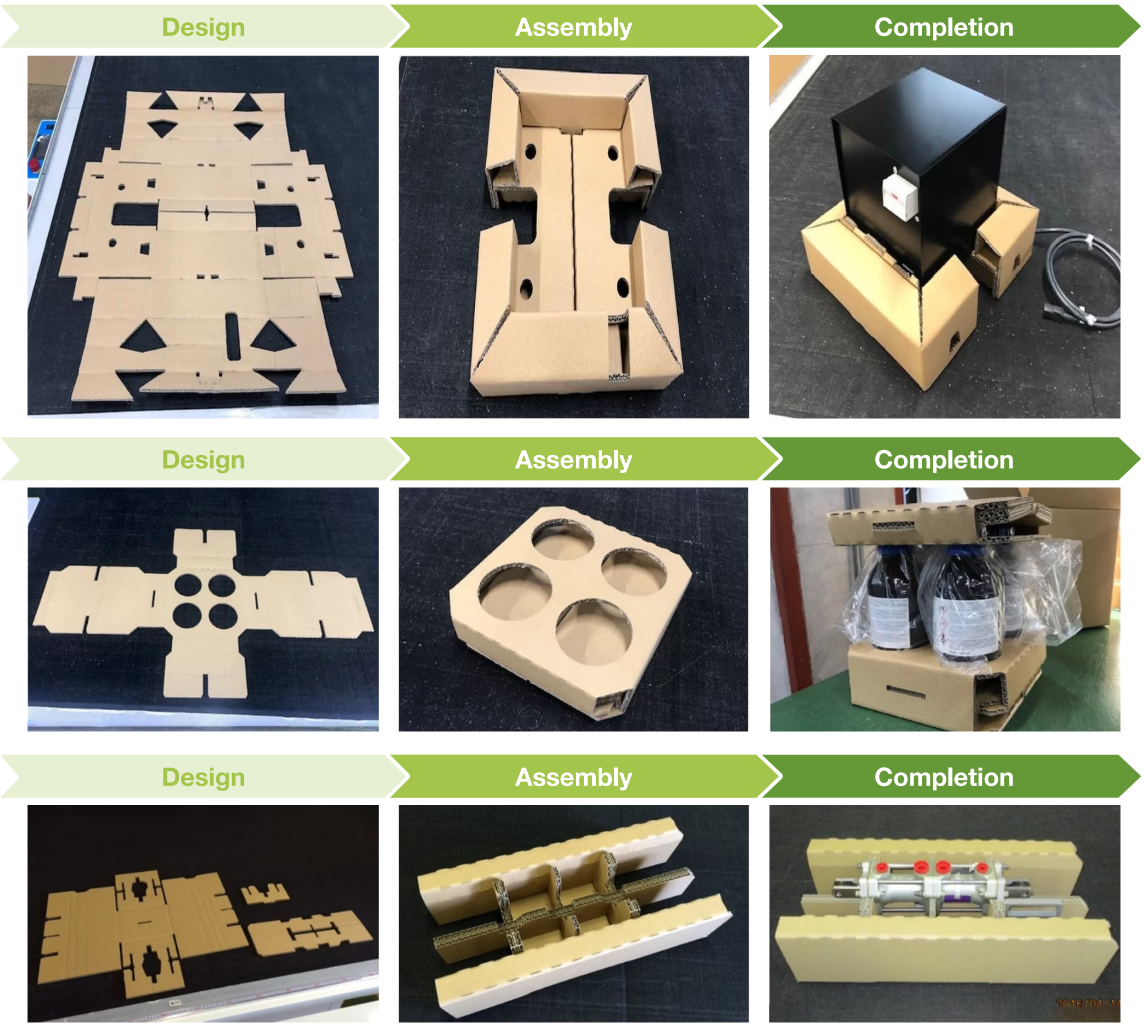
News & Notices
Read the original article

Start of a Revolutionary Packaging Project in the Shimadzu Group to Contribute to the Environment

In June 2023, Shimadzu Corporation began a “Revolutionary Packaging Project” to improve the design of packaging for products. This project will revise the packaging materials and specifications used for products handled by the Shimadzu Group, leading to reduced environmental load and logistics costs. At the end of March 2023, Shimadzu Logistics Service Corporation (SLS), which handles logistics functions for the Shimadzu Group, acquired El-design Co., Ltd. (ELD), which has high level packaging design technology. Shimadzu will promote the Revolutionary Packaging Project company-wide including the development departments and environment departments, mainly led by SLS and ELD. The amount of waste will be reduced by changing from conventional wood and plastic packaging materials to recyclable cardboard boxes.

Additionally, reducing the volume after packaging will improve the loading efficiency for the Shimadzu Group as a whole, leading to reductions in transport fees and packaging fees both in Japan and overseas. Through these series of measures, cost reductions of approximately 100 million yen per year are expected.

News & Notices
Read the original article



Reductions in waste and costs are achieved by changing plastic product guards to cardboard boxes.

It is Shimadzu Corporation’s goal to contribute to a carbon-neutral society and a circular economy. As part of our medium-term management plan, we are reducing the use of petroleum-derived plastics and aiming to minimize CO_2 emissions. By maintaining our focus on sustainability, Shimadzu Corporation is well-positioned to lead the way to a greener future.

Learn more about
Shimadzu’s environmental commitment

Brevis GC-2050 and GCMS-QP2050

Latest topics 4

Release of Brand-new Gas Chromatograph and Gas Chromatograph Mass Spectrometer

For over 65 years, Shimadzu Corporation has delivered gas chromatograph (GC) instruments and solutions. These support detection of low amount components and reliable data generation in the chemical, pharmaceutical and various market fields. Amid customer demands change, we keep innovating and applying new technologies into our products. Additionally, we have collaborated with many knowledgeable scientists and produced dedicated GC systems.

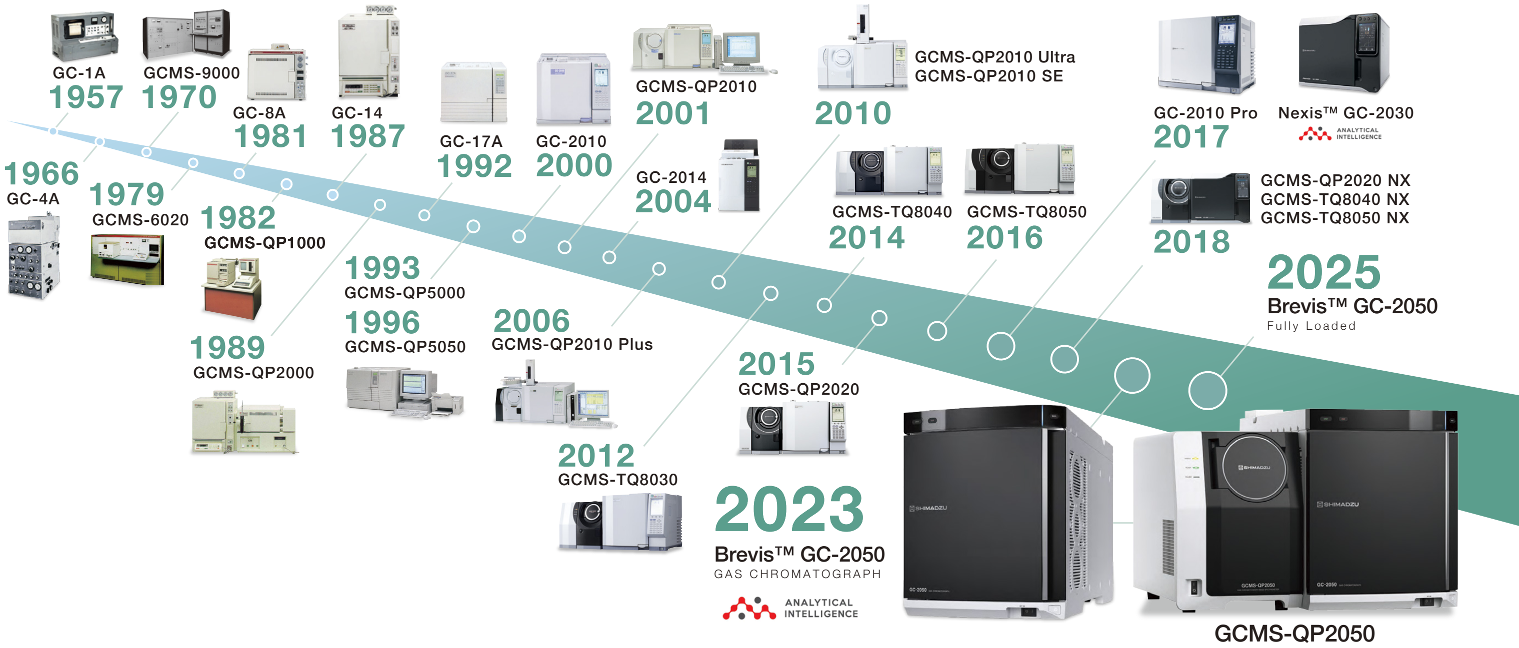
In 1970, we released our first gas chromatograph mass spectrometer, GCMS-9000 which was a magnetic sector type mass spectrometer, in cooperation with Swedish company, LKB Instruments, Inc. Then, we achieved a computational operation system and introduced our own quadrupole type gas chromatograph mass spectrometer (GC-MS).

GC-MS is mainly used for environmental, food, forensic and chemical markets because their samples are very complex, and require higher selectivity.

In 2020, we reached 50 years anniversary and our triple quadrupole GC-MS was selected for an anti- doping test for the international sports games.

Read the Application

In 2023, we released brand new GC and GC-MS systems. Those have superior compactness and easy-to-use features, which will bring more benefits and possibilities to your labs.



Brevis GC-2050

Providing both Productivity Improvements and Space Savings

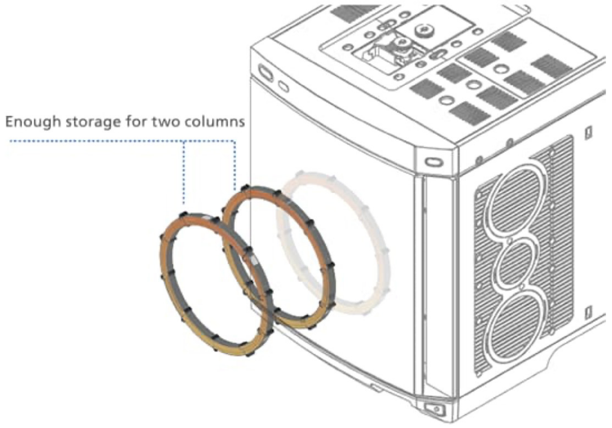
Shimadzu Corporation announces the release of the Brevis GC-2050 gas chromatograph. This instrument provides both productivity improvements and space savings while maintaining a high level of basic functionality and expandability of optional products.

Features

1. Utilization of Laboratory Environments where Space is Limited

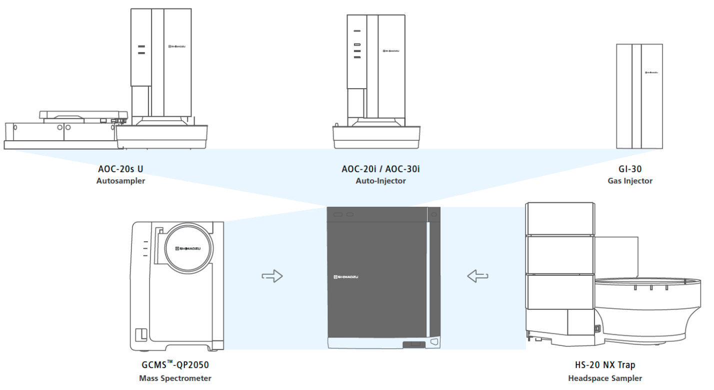
This product achieves a space saving width of just 350 mm. It is 35 % narrower in comparison to the width of the existing Nexis GC-2030.

Additionally, despite its compact size, it can accommodate a standard capillary column and double productivity by simultaneously analyzing samples on two analytical lines. This system is the world's narrowest system that can analyze two concurrent analyses.



2. Highly Expandable to Satisfy Laboratory Needs

A flexible system configuration is supported. Existing analytical methods and consumables can be seamlessly transitioned to the Brevis GC-2050 without being restricted by device configurations.



Injection Units
SPL / WBI / SINJ / PTV / OCI / SPI

Columns
7 inch standard capillary column/packed colum

Pretreatment Devices
AOC-20 / AOC-30 / GI-30 / HS-20 NX Loop/Trap / HS-10 / AOC-6000 plus / TD-30 / PY-3030D

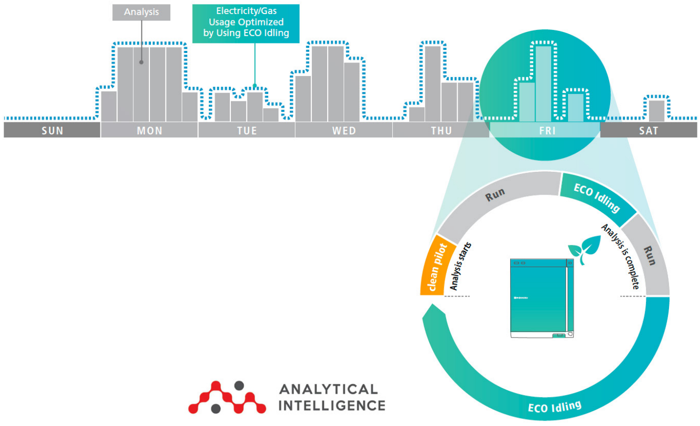
Detectors
FID / TCD / BID / FTD / FPD / ECD / MS

Options
Low temperature control solenoid valve / Gas selector / Hydrogen sensor / External control relay / Multi position valve

Advanced Flow Technology
Detector Splitting / BackFlush

3. ECO Idling: Making Your Usual Analysis Eco-Friendly

The ECO Idling function maintains GC' s accurate analytical performance as usual during analysis, and automatically switches to an energy-saving status when not analyzing. It learns the analysis patterns, visualizes the amount of weekly analysis work, and can also propose an eco operation schedule to laboratory chemists.



GCMS-QP2050

Compact size, High Sensitivity, Durability and Easy Maintenance Heighten the Efficiency of Environmental, Chemical, and Food Analyses

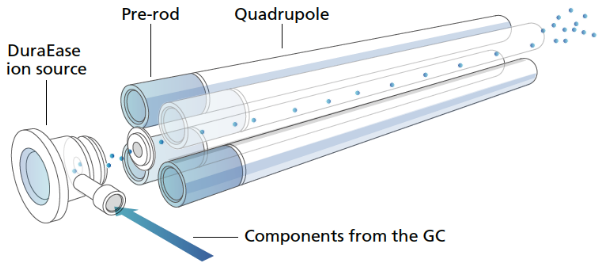
The business environments and needs involved in analysis work change on a continual basis. The next-generation GCMS-QP2050 gas chromatograph mass spectrometer, with its accumulation of impressive Shimadzu technology, will lead the way forward. New value is provided by hardware boasting astounding reliability and stability, and easy-to-operate software equipped with superior automated technology.

Features

1. Robust Engineering Minimizes Maintenance

Contamination-Resistant Ion Optical System

A contamination-resistant ion optical system in the GCMS-QP2050 keeps the frequency of maintenance to a minimum while also enabling highly reliable measurements to be performed for an extended period.



DuraEase Ion Source

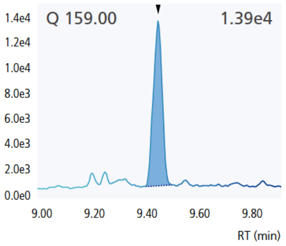
The structure of the next-generation DuraEase ion source is inert and achieves a uniform temperature distribution, resulting in high sensitivity and exceptional durability.

Long-Life Filament

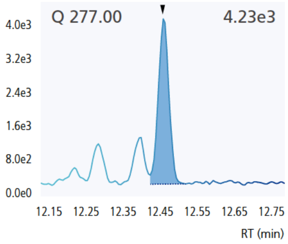
The newly developed long-life filament has an operating life at least five times longer than conventional filaments. There are no concerns about re-analysis or downtime due to sudden filament burnout.

Quadrupole Rods with Pre-Rod

The built-in pre-rod allows only the ions to efficiently pass through, limiting contamination of the quadrupole. In addition, because heating to prevent contamination is not required, it is maintenance-free.



Cadusafos (3 pg/µL)

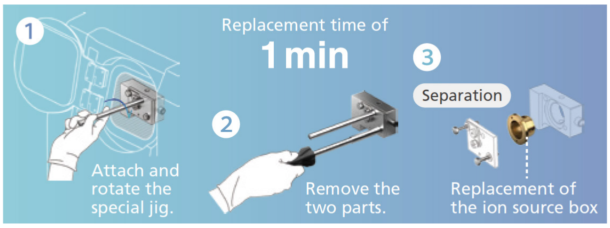


Fenitrothion (3 pg/µL)

2. Easy Maintenance

Ion Source Maintenance Takes Just One Minute

The DuraEase ion source completely re-imagines the conventional ion source to enable more convenient maintenance. The ion source is disposable and no cleaning is required, so maintenance is finished in just one minute.



Easy Startup and Shutdown from the Touch Panel

The vacuum system can be turned ON/OFF and Easy sTop can be performed from the GC touch panel. Operations from a personal computer are not required, so maintenance of the GC injection port, column, and ion source can proceed with ease.

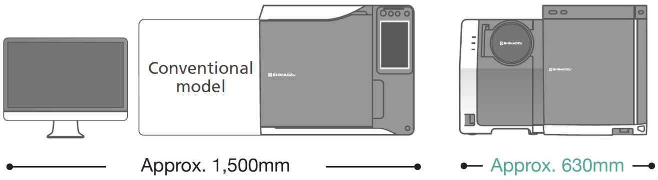
3. A Flexible Fit for Laboratories

Remote System Access

The system can be operated from a personal computer or tablet on the network via a LAN connection. Additionally, analysis and instrument status can be checked while away from the laboratory.

Compact Design Fits Anywhere

The GCMS-QP2050 saves on space. With remote access, there is no need to install a personal computer beside the instrument. This enables a flexible layout in the laboratory.



Latest topics 5

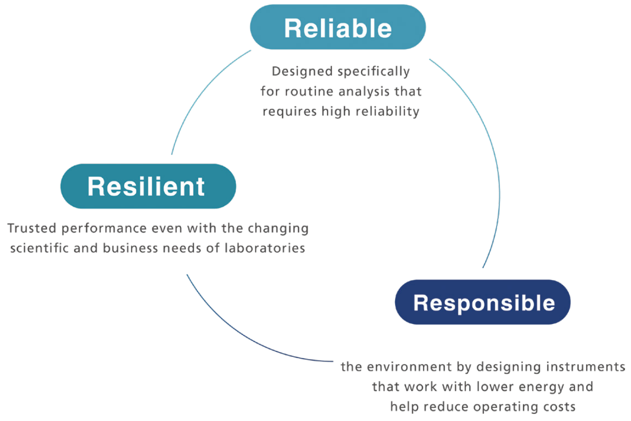
Release of the LCMS-TQ RX Series High-Performance Liquid Chromatograph Mass Spectrometers

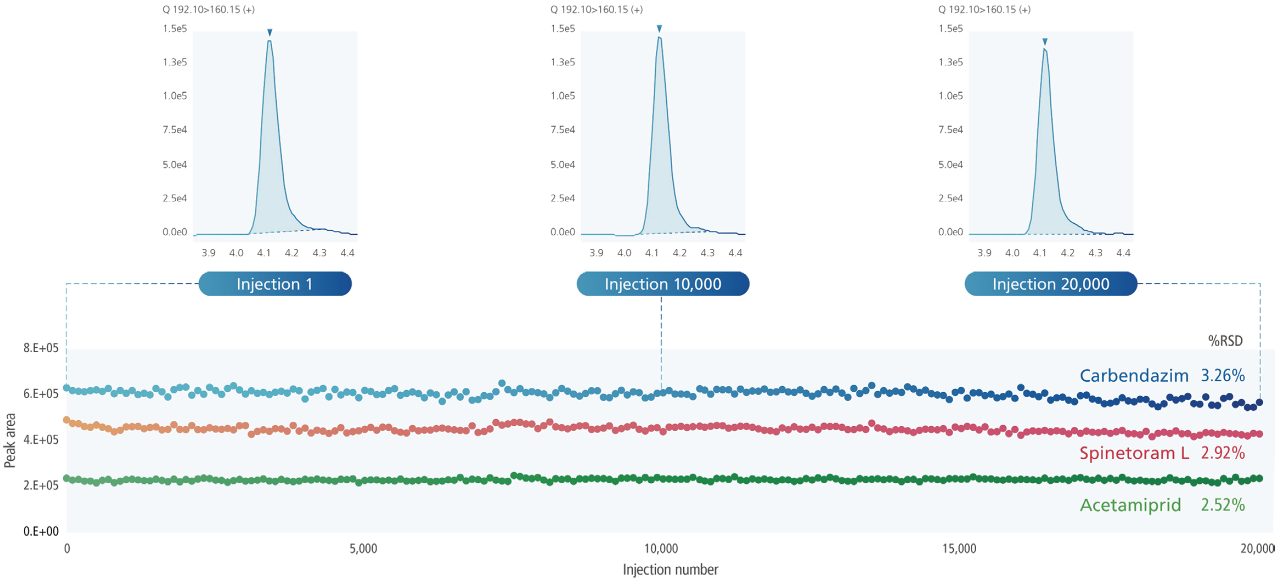
Providing More Reliable Data with High Sensitivity and Stability

Shimadzu announces the release of the LCMS-8045RX, LCMS-8050RX, and LCMS-8060RX triple quadrupole high-performance liquid chromatograph mass spectrometers globally. The three models in the LCMS-TQ RX Series carry onward the basic functionality of Shimadzu triple quadrupole (TQ) liquid chromatograph mass spectrometers (LC-MS) while providing even higher stability and convenient operability. Shimadzu is releasing this series for manufacturers and contract analysis organizations in the pharmaceutical, environmental, and food product sectors.

In the pharmaceutical, environmental, and food product sectors, regulations have become stricter, and there is an increasing demand to obtain reliable data with highly stable instruments. In particular, the global market for LC-MS/MS instruments with their high sensitivity and selectivity is expected to grow by at least 8 % annually, and the objectives and sectors where they are applied have diversified. In routine analysis applications, which account for 40 % of the LC-MS/MS market, several samples are measured consecutively, so data reliability and shortened downtime areimportant. In addition to basic performance, such instruments must provide both stability and operability.

With the LCMS-TQ RX Series, the data reliability has been heightened by stabilizing the sample ionization with the ion source improved. This series is equipped with functions that check the instrument status prior to measurements, and automatically implement calibration (tuning), as well as ecology mode for minimizing standby power consumption, thereby achieving high-efficiency laboratory operations with a low environmental impact. Through this series, Shimadzu will heighten the efficiency of laboratory work related to pharmaceuticals, food product development, and environmental inspection so that we contribute to the growth of research and development in healthcare, green transformation, and other sectors.



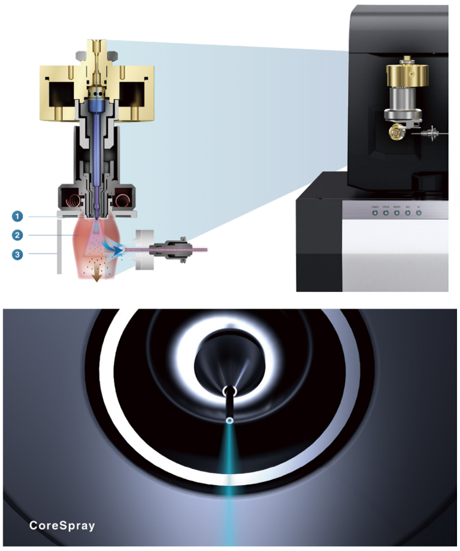


Peak Area Stability in Matrix

Features

1. Improving Data Reliability with High Stability and Measurement Sensitivity

In the LCMS-TQ RX Series, the ion source is equipped with the newly developed CoreSpray nozzle. The stability of the analysis data has been increased by uniformizing the sample spray with improving the shape of the nozzle used to nebulize the sample. Moreover the ionization efficiency of some compounds has been improved by raising the upper limit of nebulizing gas flow rate.



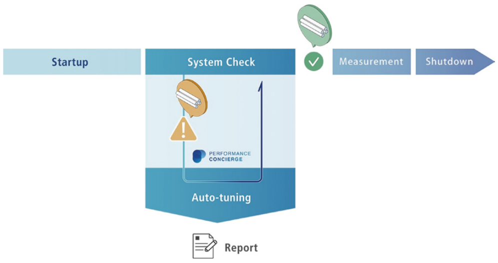
CoreSpray

CoreSpray technology applied to the repeated analysis of pesticides in black tea matrix resulting in enhanced robustness (%RSD less than 3.5%, 20,000 injections). Data were acquired without internal standards and without diverting to waste for the initial polar matrix effects.

Learn more about LCMS-TQ RX Series

2. Maintaining Favorable Instrument Conditions, Heightening the Efficiency of Laboratory Operations

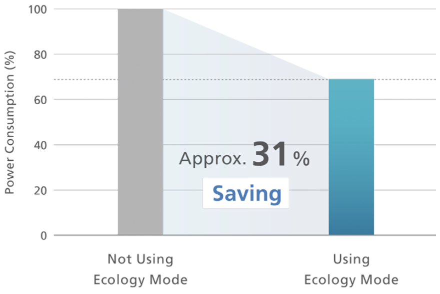
The PERFORMANCE CONCIERGE function checks the mass accuracy, spectral intensity, and other instrument conditions before starting measurements, and automatically implements tuning depending on the results. Consistently maintaining the instrument in optimal condition controls the tuning times and reduces downtime.



Instrument Check

3. Minimization of Power Consumption by the Instrument itself, and Reducing Load on the Lab and the Environment

In ecology mode, the instrument monitors its usage, and if it has not been used for a set period, it automatically shuts down. Conventionally, the inside of the instrument continues to be heated even after measurements are finished. By minimizing this, power consumption is reduced by approximately 31 %.



Comparison of Power Consumption

RELEASED

New Products

Brevis GC-2050

ANALYTICAL INTELLIGENCE

Gas Chromatograph

“Smaller, simpler, and easier to use – without compromising performance.” That’s the demand from analysts. And that’s why Shimadzu developed the Brevis GC-2050. This new space-saving GC delivers uncompromising analytical performance in a modern yet rugged design, easily meeting the analysis needs of laboratories in a range of industries.

Learn more

GCMS-QP2050

ANALYTICAL INTELLIGENCE

Gas Chromatograph Mass Spectrometer

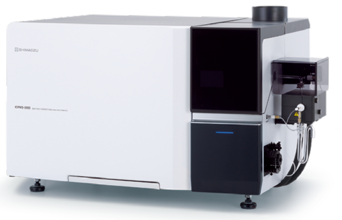
The business environments and needs involved in analysis work change on a continual basis. The next-generation GCMS-QP2050 gas chromatograph mass spectrometer, with its accumulation of impressive Shimadzu technology, will lead the way forward. New value is provided by hardware boasting astounding reliability and stability, and easy-to-operate software equipped with superior automated technology.

Learn more

ICPMS-2040/2050 Series

ANALYTICAL INTELLIGENCE

Inductively Coupled Plasma Mass Spectrometry



Shimadzu’s ICPMS-2040 Series / ICPMS-2050 Series of ICP Mass Spectrometers has achieved a harmonious blend of environmental-friendliness and analytical performance through its advanced proprietary Mini-Torch System. Without the need for any special options, it reduces measurement time, contributing to the optimization of your workflow efficiency. Moreover, the software comes with various functions, options, and maintenance information that minimize operator intervention, revolutionizing the way you work.

Learn more

MAP-100

Microplastic automatic preparation device

The presence of microplastics in environmental surface water such as oceans, rivers, and lakes has attracted international attention as an environmental problem, and many monitoring results have been reported.
The MAP-100 automates the typical steps needed to isolate microplastics. This improves the reproducibility of the analytical workflow, enables lab technicians to focus on other tasks, and makes handling of reagents safer.

Learn more

IRSpirit-X Series

ANALYTICAL INTELLIGENCE

Fourier Transform Infrared Spectrophotometer

IRSpirit FTIR spectrophotometers are extremely compact, but can still be used with standard accessories from Shimadzu and other manufacturers.
IRSpirit instruments come with IR Pilot software, which offers 23 application programs without the need for parameter setup. Measure multiple samples with just a few clicks. You can choose from the highly sensitive IRSpirit-TX, the affordable IRSpirit-LX, and the highly reliable IRSpirit-ZX with its moisture-resistant design.

Learn more

LCMS-TQ RX Series

ANALYTICAL INTELLIGENCE

Triple Quadrupole LC-MS/MS

Innovative technology, exceptional design and new ways of thinking are part of our engineering DNA delivering solutions for the ever-changing needs of any laboratory. As our scientific and business needs change our engineering design evolves and adapts. The result is the RX Series of triple quadrupole LC-MS instruments designed with unmatched capability, redefined reliability and creating a new standard in actionable data.

Learn more

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Printed in Japan, March, 2025