

# Protecting tomorrow

Environmental contaminant analysis compendium



# Solve more environmental contaminant challenges together

Given the ever-increasing pressure on budgets, higher workloads, demand for faster results, and the need for differentiated revenue streams and customer acquisition, the life of an environmental testing lab is harder than ever before. You need to increase sample throughput and reduce cost while delivering high quality results.

To maintain your laboratory's reputation for quality and ensure regulatory compliance, it is imperative you provide an accurate account of the contaminants present in each sample. Furthermore, producing timely/accurate test results consistently while adhering to regulatory methods and controlling costs with new samples arriving daily is a challenge. Disruptions to daily routines caused by instrument downtime, analysis errors, and unforeseen training needs produce ripple effects that can significantly impact your ability to manage workloads efficiently.

Laboratory managers continue to rank expanding laboratory services, developing staff, and improving their work environment as their top goals; while they focus improving higher throughput/productivity, system efficiency and developing new testing services. The top challenges that continue to hold them back include:

- Complexity of testing requirements
- Time consuming sample prep and ongoing employee training
- Keeping up with changing regulations
- Better management of data
- Instrument maintenance/downtime

We have you covered—from sample receipt to regulatory reporting—with the most comprehensive chromatography, mass spectrometry, and spectroscopy instrumentation in the world. Our complete portfolio contains instrumentation, software, applications, columns, and consumables for organic, inorganic, metals, and even emerging contaminants. Everything is designed to ensure accuracy and limit unnecessary interruptions, because so much is riding on your success—not just optimizing productivity in the lab and saving bottom lines but safeguarding the environment as well.

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# Organic contaminants

Organic regulatory methods for analysis of VOCs in environmental samples are largely the same as they were twenty years ago. Only recently have updates been introduced that allow environmental labs to improve current approaches to VOC analysis. Mirroring the pace of regulatory updates, many environmental testing laboratories rely on outdated instruments because of their ability to meet regulatory method requirements with minimal interruption. Although this strategy has provided adequate productivity in the past, many of the workhorse instruments used today are approaching, or in, obsolescence now. The unavailability of spare parts, absence of support and degradation in performance after years of use creates risk for environmental testing labs whose accreditation and reputation depends on accuracy of sample analysis and availability of instrumentation for timely results reporting. Replacement of aging systems with updated technology provides labs with the ability to

increase efficiency and possibly exceed current sample analysis volumes. Additionally, improved technology allows laboratories to take advantage of the flexibility provided by regulatory method updates, unlocking paths to quicker sample turnaround.

Modernize your volatile and semi-volatile workflows to meet tomorrow's regulatory requirements. Don't let unplanned instrument downtime, sample reanalysis, operator error, or inaccurate results impact your profitability and reputation. Discover strategies to meet sensitivity requirements while optimizing throughput and flexibility. Or—improve profitability with consolidated multi-class GC-MS methods. **Access common EPA and other regional regulatory methods your team needs to meet the challenges of VOC analysis for environmental samples.**

Click on note titles to jump to page

Matrix							US EPA	Title
Drinking Water	Surface Water	Waste Water	Soil	Sediment	Air	Other		
●	●	●	●	●	●		8270E	<a href="#">Analysis of multiple matrices with a single calibration curve for polycyclic aromatic hydrocarbons (PAHs) with the ISQ 7610 GC-MS system following EPA Method 8270E</a>
●	●	●	●	●	●		8270	<a href="#">Optimized analysis of semi-volatile organic compounds (SVOC) in environmental samples in compliance with US EPA Method 8270 utilizing GC-MS</a>
●		●	●				8260	<a href="#">Analysis of VOCs according to US EPA Method 8260</a>
			●				8260	<a href="#">Regulatory analysis of volatile organic compounds in soil using purge and trap coupled to single quadrupole GC-MS</a>
	●	●					8260 & 524.2	<a href="#">Regulatory analysis of volatile organic compounds in water using purge and trap coupled to single quadrupole GC-MS</a>
			●	●			3545A	<a href="#">A more cost-efficient extraction method for polycyclic aromatic hydrocarbons (PAH) in sediments and soils using accelerated solvent extraction (ASE)</a>
			●				1668	<a href="#">A rapid, sensitive, and consolidated method for PCBs and PAHs using GC-MS/MS for US EPA Method 1668</a>

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Click on note titles to jump to page

Matrix							US EPA	Title
Drinking Water	Surface Water	Waste Water	Soil	Sediment	Air	Other		
●	●						524.2	Uninterrupted analysis of volatile organic compounds in drinking water using single quadrupole GC-MS
					●		TO-15	A rapid method for the analysis of air toxics based on US EPA Method TO-15
	●							Automated sample preparation followed by sensitive analysis by GC-MS/MS for environmental contaminants in surface waters
						●		DualData XL DFS Magnetic Sector GC-HRMS high throughput analysis of polychlorinated dioxins/furans (PCDD/Fs)
					●			Using EA-IRMS isotope fingerprints to track sources of PM <sub>2.5</sub> in air pollution
●	●	●	●					Robust and cost-effective analysis of TPH in water and soil using GC-FID to meet various regulated methods
			●	●				Tracing pollutants in soil and sediment using GC-IRMS carbon isotope fingerprint
	●	●		●				Supporting the Nitrate Directive by using isotope fingerprints for detecting the sources of nitrate pollution
			●					Consolidated analysis of soil contaminants—Four-fold increase in the sample throughput with GC high-resolution accurate mass
			●					Robust analysis of PAHs and PCBs in soil with over 500 repeat injections using Orbitrap Exploris GC
●								Determination of polycyclic aromatic hydrocarbons in drinking water at ppt levels by Solid Phase Micro Extraction Arrow coupled with GC-MS
			●					A consolidated method for the analysis of VOCs in soil by HS-GC-MS in analytical testing laboratories
●	●		●					An automated approach for the determination of gasoline range organics (GRO) in water by gas chromatography coupled with static headspace sampling
●	●		●					Robust and cost-effective analysis of TPH in water and soil using GC-FID to meet various regulated methods
					●			Completely cryogen-free monitoring of ozone precursors, air toxics, and oxygenated volatile organic compounds in ambient air in a single run
●								In-Tube Extraction Dynamic Headspace (ITEX-DHS) sampling technique coupled to GC-MS for sensitive determination of odorants in water
	●	●	●	●		●		Reproducible trace analysis of PCBs in environmental matrices using triple quadrupole GC-MS/MS
			●	●	●			Case study: TSQ 9610 GC-MS/MS delivers increased sample throughput in an analytical laboratory



APPLICATION NOTE 000455

## Analysis of multiple matrices with a single calibration curve for polycyclic aromatic hydrocarbons (PAHs) following EPA Method 8270E

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### Goal

The aim of this application note is to demonstrate the wide dynamic range and the robustness of the Thermo Scientific™ ISQ™ 7610 single quadrupole mass spectrometer, using the new Thermo Scientific™ XLXR™ detector, coupled to a Thermo Scientific™ TRACE™ 1610 gas chromatograph, for the analysis of 19 polycyclic aromatic hydrocarbons (PAHs) in soil and water, according to the United States Environmental Protection Agency (EPA) Method 8270E.

### Introduction

Polycyclic aromatic hydrocarbons (PAHs) are organic compounds consisting of carbon and hydrogen atoms. Chemically the PAHs comprise two or more aromatic rings bonded in linear, cluster, or angular arrangements, resulting in a wide diversity of physical, chemical, and toxicological properties. PAHs are ubiquitous and can contaminate soil, air, sediments, and water and are resistant to environmental degradation. These compounds are found in fossil fuel sources and manmade chemicals and are derived from the incomplete

combustion of organic matter used for human activities (such as vehicle emissions, rubber, plastics, and cigarettes). PAHs have toxic effects because of their chemical structure and act as a carcinogen or endocrine disrupter. Due to their toxicity, they are monitored in the environment with strict regulations.

### Conclusion

The results of the experiments described here demonstrate the ISQ 7610 GC-MS system meets and exceeds the EPA Method 8270E requirements for the analysis of PAHs in environmental samples.

 [View the full application note](#)



APPLICATION NOTE 000761

## Optimized analysis of semi-volatile organic compounds (SVOC) in environmental samples in compliance with US EPA Method 8270 utilizing GC-MS

Amit Gujar, Kenneth M Free, Tim Anderson, Thermo Fisher Scientific, USA;  
Adam Ladak, Thermo Fisher Scientific, UK

### Introduction

Semi-volatile organic compounds (SVOCs) are ubiquitous in our environment and raise concerns regarding health for humans and wildlife. They are released in various everyday processes, including manufacturing and agriculture. Thus, the analysis of SVOCs in extracts of many matrices, such as solid waste, soil, air, and water, is commonly performed by environmental testing laboratories. Regulations are in place worldwide to monitor the presence of SVOCs in environmental samples to protect the environment and human exposure. The United States Environmental Protection Agency (EPA) released the first SVOC method by gas chromatography/mass spectrometry (Method 8270) at the end of 1980.<sup>1</sup> EPA Method 8270D and derivative methods are now used in environmental labs globally to monitor SVOCs.

Analytical testing laboratories that follow EPA 8270 methodology for the analysis of SVOCs must comply with several requirements before starting the analysis. These include the ability for the MS instrument to pass decafluorotriphenyl phosphine (DFTPP) tuning criteria, relative retention time (RRT) of target analytes criteria, linearity of calibration curve criteria, and suggested minimum response factors. This application note shows how the Thermo Scientific™ ISQ™ 7610 single quadrupole GC-MS system can meet EPA Method 8270D requirements.

One of the unique features of the ISQ 7610 single quadrupole MS systems is NeverVent™ technology, which allows laboratories to minimize instrument downtime with fast removal of the ion source for maintenance and to change the analytical column without venting the system. The ISQ 7610 MS also introduces a new detector, the Thermo Scientific™ XLXR™ detector system, with a wider linear dynamic range and longer lifetime compared to previous generation detectors.

### Conclusion

The Thermo Scientific ISQ 7610 single quadrupole GC-MS system with the Thermo Scientific™ ExtractaBrite™ ion source and the innovative NeverVent technology is the perfect solution to perform EPA Method 8270D.

 [View the full application note](#)



APPLICATION NOTE 73769

## Analysis of VOCs according to US EPA Method 8260

Adam Ladak, Terry Jeffers, Dwain Cardona, Thermo Fisher Scientific, USA;  
David Lee, Thermo Fisher Scientific, Runcorn, UK; Amy Nutter, Teledyne Tekmar, Mason, OH, USA

### Goal

An analytical method that meets the requirements outlined in US EPA Method 8260D utilizing US EPA Methods 5030 and 5035 preparation methods for the quantitation of purgeable organic compounds (POCs) in wastewater and soil, using the Teledyne Tekmar Atomx XYZ purge and trap (P&T) system along with a Thermo Scientific™ ISQ™ 7000 Mass Spectrometry (MS) system coupled with a Thermo Scientific™ TRACE™ 1310 Gas Chromatograph (GC) and Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS). Method linearity, method detection limit (MDL), and Initial Demonstration of Capability (IDC) were assessed to evaluate method performance.

### Introduction

Volatile organic compounds or VOCs are common in modern life and come from both human-made and natural sources, but the human-made sources of VOCs in populated and industrialized areas are the main contributors to environmental pollution. These VOCs are produced in the processing of, or as, paints, adhesives, petroleum products, pharmaceuticals, and refrigerants. Many of these compounds contaminate our environment today and cause negative health effects to humans when they are exposed to elevated levels. Analytical testing laboratories must monitor the environment to ensure the public is not exposed to elevated levels of VOCs. The latest version of EPA Method 8260—EPA Method 8260D—is used to monitor a variety of solid waste matrices for the presence of VOCs.

To perform EPA Method 8260, all method acceptance criteria must be achieved. These criteria include linearity <20% relative standard deviation (RSD), a minimum response factor (RF), and MDLs for a wide range of target compounds. The analytical method must produce consistent results and be reproducible from day to day, with a continuing calibration verification (CCV) analyzed every 12 hours while samples are being performed. As the method covers varying matrices, it is important that the performance criteria are met in all samples of interest.

### Conclusion

The combined analytical solution of the TRACE 1310 GC coupled with the ISQ 7000 system and the Atomx XYZ P&T system provides clear performance and cost saving advantages for analytical testing laboratories that analyze environmental samples following the EPA Method 8260 requirements.

 [View the full application note](#)



APPLICATION NOTE 000377

## Regulatory analysis of volatile organic compounds in soil using purge and trap coupled to single quadrupole GC-MS

Terry Jeffers, Thermo Fisher Scientific, USA; Adam Ladak, Thermo Fisher Scientific, UK;  
Amy Nutter, Teledyne Tekmar, Mason, OH, USA

### Goal

To demonstrate the compliance of the Teledyne Tekmar Atomx XYZ purge and trap (P&T) system along with a Thermo Scientific™ ISQ™ 7610 single quadrupole mass spectrometer coupled to a Thermo Scientific™ TRACE™ 1610 gas chromatograph (GC) and Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) for the determination of volatile organic compounds (VOCs) in soil according to the Chinese Method HJ 605. Linearity, detection limit, precision, and accuracy were assessed to evaluate the method performance. A long-term study was also performed to demonstrate the robustness of the method.

### Introduction

Analytical testing laboratories must monitor the environment to ensure that the public are not exposed to elevated levels of volatile organic compounds (VOCs). VOCs are generated by humans and produced in the processing of, or as, adhesives, petroleum products, paints, refrigerants, and pharmaceuticals. These compounds are of major concern globally, as they can contaminate the

environment and cause negative health effects in humans who are exposed to elevated levels. There are several worldwide regulations in place to monitor the levels of VOCs in soil. The United States Environmental Protection Agency (US EPA) has defined Method 8260 to monitor the levels of VOCs in soil.

### Conclusion

The results shown in this study demonstrate that the TRACE 1610 GC coupled to the ISQ 7610 single quadrupole MS and the Atomx XYZ P&T provides a suitable tool for analytical testing laboratories analyzing environmental soil samples in compliance with the HJ 605 method.

 [View the full application note](#)



APPLICATION NOTE 000305

## Regulatory analysis of volatile organic compounds in water using purge and trap coupled to single quadrupole GC-MS

Terry Jeffers, Thermo Fisher Scientific, USA; Adam Ladak, Thermo Fisher Scientific, UK;  
Amy Nutter, Teledyne Tekmar, Mason, OH, USA

### Goal

To demonstrate the compliance of the Teledyne Tekmar Atomx XYZ purge and trap (P&T) system along with a Thermo Scientific™ ISQ™ 7610 single quadrupole mass spectrometer coupled to a Thermo Scientific™ TRACE™ 1610 gas chromatograph (GC) and Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) for the determination of volatile organic compounds (VOCs) in ground water, surface water, and wastewater according to the Chinese Method HJ 639. Linearity, detection limit, precision and accuracy were assessed to evaluate the method performance. A long-term study was also performed to demonstrate the robustness of the method.

### Introduction

Volatile organic compounds (VOCs) are a group of chemicals with different properties that are generated from natural processes, industrial activities, petroleum, and household products. These compounds are of major concern as they are important pollutants contributing to the ground-level ozone formation and thus posing a risk to human health. Regulatory agencies all

over the world have published several methods to support analytical testing laboratories dealing with VOCs analysis. Purge and trap coupled to gas chromatography–mass spectrometry (GC-MS) is the method of choice for analysis of VOCs as it offers the advantage of sample pre-concentration combined with water removal, therefore improving sensitivity, increasing column lifetime, and reducing peak interferences that can occur when moisture enters the GC system. Some of the most common methods are defined by the US Environmental Protection Agency (US EPA) and include Method 8260 and Method 524.2.

### Conclusion

The results shown in this study demonstrate that the TRACE 1610 GC coupled to the ISQ 7610 single quadrupole MS and the Atomx XYZ P&T provides a suitable tool for analytical testing laboratories analyzing environmental water samples in compliance with the HJ 639 method.

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CUSTOMER APPLICATION SUMMARY 72995

## A more cost-efficient extraction method for polycyclic aromatic hydrocarbons (PAH) in sediments and soils using accelerated solvent extraction (ASE) for US EPA SW-846 Method 3545A

Zhe Xia, Nicole Klaassen, Ifeoluwa Idowu, and Gregg Tomy, University of Manitoba, Winnipeg, MB, Canada

### Goal

To verify the suitability of a one-step extraction method to improve laboratory efficiency and reduce costs as well as solvent consumption.

### Introduction

Accelerated solvent extraction (ASE) is an extraction method that is accepted by the US Environmental Protection Agency (US EPA) for extracting solid matrices. During the extraction procedure, solvent is pumped into heated extraction cells under high pressure for a prescribed period of time. After extraction, solvent containing analytes are delivered to a collection vial for further cleanup, evaporation, or analysis. While the extraction process is fully automated, additional manual procedures typically using adsorption chromatography are needed to further clean up sample extracts. These methods are often time-consuming, labor-intensive, and use large volumes of solvents.

### Conclusion

The one-step extraction is suitable and applicable for PAH extractions in sediments and soils and meets US EPA SW-846 Method 3545A requirements for Pressurized Fluid Extraction.

- Results of recoveries are similar for from the one-step extraction method for 16 priority PAHs in the CALA and SRM 1944 samples.
- Recoveries are in the range of 70% to 120%.
- Precision values (expressed as % relative standard deviation) are less than 20%.



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APPLICATION NOTE 000540

## A rapid, sensitive, and consolidated method for PCBs and PAHs using GC-MS/MS for US EPA Method 1668 and revisions

Delphine Thomas, Bénédicte Gauriat, Aristide Ganci, Jean-François Garnier, Thermo Fisher Scientific, Les Ulis, France; Adam Ladak, Thermo Fisher Scientific, Hemel Hempstead, UK

### Goal

The purpose of the proposed method is to demonstrate the quantitative performance of the Thermo Scientific™ TSQ™ 9610 triple quadrupole GC-MS/MS system equipped with the Thermo Scientific™ Advanced Electron Ionization (AEI) source for the analysis of PAHs and PCBs in a single run.

### Introduction

Polychlorinated biphenyls (PCBs) and polyaromatic hydrocarbons (PAHs) are toxic organic pollutants that can contaminate various food and environmental matrices, including soils, water, sun cream, and salmon. These compounds are produced because of natural and human processes. PCBs and PAHs are resistant to environmental degradation and can be transported over long distances. From the environment, they can enter the food chain where they are persistent and can bio-accumulate (vPvB).<sup>1</sup> GC-MS, and increasingly GC-MS/MS, are being utilized by analytical testing laboratories to analyze PAHs and PCBs. GC-MS/MS offers an increase in selectivity, allowing matrix interferences to be significantly reduced. This produces more confidence in sample results and allows an increase in productivity.

There are several challenges associated with the analysis of PAHs and PCBs in various matrices. First and foremost, the regulatory limits and requirements must be met consistently. Often separate methods are utilized, which adds to the number of analyses per sample and reduces sample capacity. Sample preparation is also time consuming and requires a large sample volume to achieve the regulatory limits. One of the major challenges is the separation of the critical isobaric pairs of both PCBs and PAHs. Eliminating the overlap of such isobarics by chromatographic separation can lead to long analysis times, which can reduce the instrument productivity.

### Conclusion

The results obtained in these experiments demonstrate that the TSQ 9610 mass spectrometer equipped with the NeverVent AEI ion source in combination with the Thermo Scientific™ TRACE™ 1610 GC and the Thermo Scientific™ AI/AS 1610 liquid autosampler delivers consistent and reliable analytical performance for analysis of PAHs and PCBs in water samples.

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APPLICATION NOTE 000525

## Uninterrupted analysis of volatile organic compounds in drinking water using single quadrupole GC-MS for US EPA Method 524.2

Terry Jeffers, Thermo Fisher Scientific, USA; Adam Ladak, Thermo Fisher Scientific, UK; Amy Nutter, Teledyne Tekmar, Mason, OH, USA

### Goal

Demonstration of a routine analytical method that meets the requirements outlined in US EPA Method 524.2 for the quantitation of volatile organic compounds in drinking water, using the Teledyne Tekmar Atomx XYZ purge and trap (P&T) system along with a Thermo Scientific™ ISQ™ 7610 MS system coupled with a Thermo Scientific™ TRACE™ 1610 Gas Chromatograph (GC) and Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS). Method linearity, method detection limit (MDL), precision, and accuracy were assessed to evaluate method performance.

### Introduction

It is essential that analytical testing laboratories monitor surface water, ground water, and drinking water for the presence of volatile organic compounds (VOCs). VOCs are human-made contaminants used and produced in the processing of, or as, paints, adhesives, petroleum products, pharmaceuticals, and refrigerants. If they are released into surface or ground water from industrial activities, they can have an adverse effect on natural environment, and ultimately the public. It is extremely important that analytical laboratories ensure both accurate and rapid detection and quantitation of VOCs to ensure public safety. US EPA Method 524.2 is widely used in environmental analysis labs to test water samples for VOCs. The method tests for a wide range of VOCs, including the four trihalomethane disinfection by-products that have sufficiently

high volatility and low water solubility, so that they can be removed from water samples with P&T procedures.

### Conclusion

The combined solution of the TRACE 1610 GC coupled with the ISQ 7610 system and the Atomx XYZ P&T system provides clear advantages for US EPA Method 524.2. The Atomx XYZ concentrator's efficient trap cooling design reduces sample cycle time, enabling an increase in sample throughput. The moisture control system improves water vapor removal, thereby reducing peak interference and increasing GC column life span. The ISQ 7610 VPI and ExtractaBrite ion source allow users to exchange ionization sources and analytical columns without venting the instrument, significantly reducing instrument downtime and minimizing sample analysis interruptions. The Thermo Scientific™ XLXR™ detection system on the ISQ 7610 mass spectrometer provides an extended linear dynamic range, allowing extended calibration curves to be run on the system. The detector also has an extended lifetime, which significantly reduces the need for replacement.

 [View the full application note](#)



# A rapid method for the analysis of air toxics based on US EPA Method TO-15

Terry Jeffers, David Lee and Dwain Cardona, Thermo Fisher Scientific

## Goal

To demonstrate an alternative chromatographic approach to US EPA Method TO-15 for the rapid determination of toxic organic compounds in ambient air using a combined thermal desorption gas chromatography mass spectrometry (TD-GC-MS) solution from Markes International and Thermo Fisher Scientific.

## Introduction

US EPA Method TO-15 is an established method used for the measurement of hazardous air pollutants (HAPs) also known as air toxics. This subset of volatile organic compounds (VOCs) is collected and analyzed using canister sampling methods and thermal desorption gas chromatography mass spectrometry (TD-GC-MS). Despite the popularity of canister sampling, maintaining system integrity and obtaining consistent results can be difficult for routine air analysis labs. Ingress of water to the analytical instrument can negatively impact analyte response and repeatability, as well as reduce the lifetime of the column and detector. In addition to this, traditional canister pre-concentration technologies are challenged by the wide ranges of sample compound concentrations and by the varying temperatures and humidity at sampling locations.

In this application note, we demonstrate the combined use of an innovative trap-based water removal device, alternative column choice, and a robust TD-GC-MS configuration to overcome the challenges experienced in the analysis of volatile air toxics in accordance with US EPA Method TO-15 and provide advantages in laboratory productivity.

## Conclusion

- An improved method is presented for US EPA TO-15 that saves time and in turn improves resource earning potential.
- Consistent performance is most notably attributed to the combination of the KORI-xr moisture management system, the robust performance of the Thermo Scientific™ ExtractaBrite™ ion source, and the rapid separation Thermo Scientific™ TraceGOLD™ TG-VMS column.
- Effective separation and quantitation of 60 HAPs from 0.5 to 50 ppb were realized in under 13 minutes.
- Excellent linearity, RRF RSDs, and precision results comfortably achieved all method acceptance criteria. RSDs were well below the replicate precision requirement of <25%.
- MDL confirmation for n=7 0.5 ppb standards showed absence of interference from excessive moisture with lower method detection limits than required by the published method.

 [View the full application note](#)



APPLICATION SUMMARY 10591

## Automated sample preparation followed by sensitive analysis by GC-MS/MS for environmental contaminants in surface waters

Cees Bijsterbosch, Het Waterlaboratorium, Haarlem, The Netherlands; Cedric Wissel, Interscience BV, Breda, The Netherlands; Cristian Cojocariu and Inge de Dobbeleer, Thermo Fisher Scientific

### Goal

To establish the feasibility of automating the workflow for analyzing semi-volatiles with GC-MS/MS in surface waters.

### Introduction

Environmental contaminants remain a constant cause for concern with the public and there is a need for many laboratories to analyze samples in a fast and cost-effective way. Laboratories look for options to save on solvent costs and to minimize sample preparation time. However, time-saving and cost-driven measurements should not compromise the analytical results in terms of sensitivity, robustness, or quality controls. Consequently, to be able to minimize costs and efforts from the laboratory, the ideal analytical system typically needs to demonstrate: the capability of handling the automation of sample preparation, the flexibility of injection modes (such as large volume injections), and the sensitivity required for detection at the ultra low level. In this study we demonstrate the Thermo Scientific™ TSQ™ 9000 triple quadrupole mass spectrometer configured with the Advanced Electron Ionization (AEI) source and Thermo Scientific™ Triplus™ RSH™ autosampler for the automated sample preparation and subsequent detection of contaminants at ultra low levels in a surface water matrix.

### Conclusion

The experimental results show that automated sample preparation with the TriPlus RSH autosampler combined with the TSQ 9000 triple quadrupole GC-MS/MS system with the AEI source is a powerful, cost-saving configuration:

- Can be completely automated with excellent repeatabilities
- Saves solvents and avoids exposure to solvents for the analyst
- Provides a very high level of sensitivity and excellent linearity
- Allows the laboratory to save time on intensive sample preparation

The easy-to-use method has low detection limits, excellent repeatability, and linearity for a large number of contaminants in surface water samples.

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TECHNICAL NOTE SUMMARY 30174

## DualData XL DFS Magnetic Sector GC-HRMS high throughput analysis of polychlorinated dioxins/furans (PCDD/Fs)

Heinz Mehlmann, Dirk Krumwiede, Thermo Fisher Scientific, Bremen, Germany

### Goal

To demonstrate how to increase sample throughput for dioxin/furan analyses by using the DualData XL Acquisition configuration of the Thermo Scientific™ DFS™ Magnetic Sector GC-HRMS.

### Introduction

Magnetic Sector High Resolution GC-MS is the gold standard for high sensitivity analysis of Dioxins and other POPs. Already for decades it has been proving its proficiency in this field of analysis and thus became the established analysis technique available nowadays in leading Dioxin laboratories throughout the world. Together with technical improvements allowing the routinely detection of low femtogram amounts of highly toxic compounds like 2378-TCDD developments in software tools for instrument control as well as data evaluation have led to strong improvements as for ease-of-use and productivity with this analytical technology.

### Conclusion

It has been demonstrated that the DualData XL Module for the DFS Magnetic Sector GC-HRMS allows a higher sample throughput by no loss in performance such as peak shape or sensitivity. The GC separation integrity, ruggedness, and long-term stability of the column switching system have been proven in unattended sample.

- Increase of productivity up to double sample throughput
- Excellent peak shape using MCD wafer technology
- No loss in sensitivity compared to a standard dual GC system
- Applicable to different POPs such as dioxins, PCBs, and PBDEs

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APPLICATION BRIEF 30482

## Using EA-IRMS isotope fingerprints to track sources of PM<sub>2.5</sub> in air pollution

Christopher Brodie, Thermo Fisher Scientific, Bremen, Germany

### Goal

Trace the origin of PM<sub>2.5</sub> particles in air pollution using carbon, nitrogen and sulfur isotope fingerprints.

### Introduction

Air pollution, also known as haze events, is a growing global issue that results from a high concentration of sub-micron particles (PM<sub>2.5</sub>) in air in the form of sulfate aerosols and nitrous oxides derived from fossil fuel burning, ozone and volatile organic compounds. PM<sub>2.5</sub> particles have a high scattering coefficient on solar radiation and combined with high humidity, reduce visibility significantly. These factors impact the surface temperature of the earth, thereby affecting weather and climate, and carry serious implications for human cardiovascular and respiratory health. In order to improve air quality, it is important to identify the source of air pollutants (referred to as source apportionment) and application of isotope fingerprint analysis using isotope ratio mass spectrometry (IRMS) is an informative and rapid technique for achieving this.

### Conclusion

Using the EA IsoLink IRMS System for source identification of PM<sub>2.5</sub> particles in air pollution, laboratories provides:

- A rapid analytical technique for source identification using isotope fingerprints
- An efficient, automated technique for source identification that does not require sample chemical clean up, with its associated high labor and consumable costs
- A reliable analytical technique that produces high quality data comparable with published literature

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APPLICATION NOTE 74086

## Robust and cost-effective regulatory analysis of TPH in water and soil using GC-FID

Jane Cooper, David Lee, Zoe Julian, Thermo Fisher Scientific, UK  
Josh Marshall, Jonathan Williams, Dŵr Cymru Welsh Water, UK

### Goal

The aim of this work is to demonstrate a simple, cost-effective analytical solution for the routine determination of total petroleum hydrocarbons in water and soil samples, for implementation at the high-throughput Welsh Water laboratory, UK, by using a Thermo Scientific™ TRACE™ 1310 Gas Chromatograph and the Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS).

### Introduction

Total petroleum hydrocarbons (TPH) describe a complex mixture of several hundred organic chemical compounds that originates from crude oil and range from light gas products to heavy fuel oils that can potentially contaminate the environment.

Compounds known to exist in some TPH fractions can affect blood, the immune system, liver, spleen, kidneys, developing fetus, and the lungs. Other TPH, such as some mineral oils, are not very toxic and are used within the food industry.

Current sample preparation and analytical technologies used for the analysis of TPH involve methods such as Soxhlet extraction for soils and liquid-liquid extraction for water samples. These methods are time consuming and require large amounts of organic solvents, with detection using infrared spectroscopy after extraction with Freon™ 113 or tetrachloromethane, which are toxic solvents.

Various regulated methods considering the analysis of TPH are available, including ISO 16703:2011, EN ISO 14039:2004, EN ISO 9377:2000, and ASTM D7678-11.

### Conclusion

The results obtained demonstrate the TRACE 1310 GC-FID in combination with the TriPlus RSH autosampler and Chromeleon CDS offer a rapid and robust solution to laboratories that analyze TPH in environmental samples as evidenced by good chromatographic separation, excellent linearity, repeatability and robustness and the utility for various water types.



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APPLICATION BRIEF 30587

## Tracing pollutants in soil and sediment using GC-IRMS carbon isotope fingerprint

Mario Tuthorn, Dieter Juchelka, Christopher Brodie, Thermo Fisher Scientific, Bremen, Germany

### Goal

Discriminate between pyrogenic and petrogenic PAHs using  $^{13}\text{C}$  isotope fingerprints.

### Introduction

Polycyclic aromatic hydrocarbons (PAHs) are persistent pollutants in the air, soils and sediments. Many PAHs are toxic, carcinogenic or mutagenic to organisms and humans, depending upon their structure, placing them among the extensively monitored organic contaminants. PAHs are present in the environment from natural sources including fossil fuels, such as coal, from volcanic eruptions and forest fires. In addition, PAHs derive from anthropogenic sources, as a by-product of vehicle emissions, burning of fireplace wood and fuel oils and various industrial processes.

The major sources of PAHs to the environment are pyrogenic and petrogenic processes. Pyrogenic PAHs are formed by incomplete combustion of organic material, such as during cracking process, combustion of motor fuels or wood in forest fires, while petrogenic PAHs are formed during crude oil maturation

or released due to fuel transportation spills. Although the sources of PAHs are generally well-known, investigating whether PAHs originate from oil, wood or coal is important in order to reduce the health risk which they pose and for source identification. PAHs are especially inspected in relation to oil and gas explorations as their presence in the aquatic environment can be easily distributed in the food web, for example.

### Conclusion

Isotope fingerprints analyzed by GC-IRMS enable to differentiate between pyrogenic and petrogenic PAHs. With the Thermo Scientific™ GC IsoLink II™ IRMS System, laboratories gain an effective analytical solution based on fast, reliable analysis with full automation. GC-IRMS can be a powerful tool for environmental forensics investigations for potentially carcinogenic pollutants.

 [View the full application brief](#)



APPLICATION BRIEF 30768

## Supporting the Nitrate Directive by using isotope fingerprints for detecting the sources of nitrate pollution

Oliver Kracht, Christopher Brodie, Mario Tuthorn, Thermo Fisher Scientific, Germany  
Andrew Smith, National Environmental Isotope Facility, British Geological Survey, UK

### Goal

Demonstrate how by analyzing nitrogen and oxygen isotopes of nitrates we can identify sources of pollution.

### Introduction

Nitrates are a key nutrient for plants and are largely applied in agriculture to enhance crop productivity. They can be supplied as a solid, in the form of a mineral fertilizer, or as liquid manure. Increasing population size and the growing demand for food has resulted in the excessive use of nitrates in food production. This over application pollutes our environment on local levels, but also spreads globally through oceanic and atmospheric systems.

Consequentially, contamination of the hydrosphere affects human health through drinking water quality and environmental health due to increased algae growth linked to eutrophication. Alongside agriculture, the other major sources of nitrate pollution are human sewage and industrial process waste. Wastewater treatment plants can remove nitrates from wastewater by bacterial denitrification processes with varying degrees of success depending on the region. If the treatments are done poorly, high concentrations of ammonia, ammonium and nitrate can enter the environment, degrading ecosystem health.

In order to protect our health from these pollutants it is critical to find ways of determining the sources of pollution, allowing for appropriate mitigation strategies. In this application brief we report how isotope fingerprints support the Nitrate Directive by offering a tool for nitrate source identification and tracking changes in our environment, as performed in different nitrates studies.

### Summary

Using nitrate isotope fingerprints, it is possible to monitor water quality and trace the sources of pollution related to human activity, including the use of fertilizers and wastewater discharges allowing scientists to inform policy makers on the best strategies for improving water quality and preserving our environment, as guided by the EU Water Framework Directive.

 [View the full application brief](#)



APPLICATION NOTE 10731

## Consolidated analysis of soil contaminants—Four-fold increase in the sample throughput with GC high-resolution accurate mass

Aaron Lamb, Dominic Roberts, and Cristian Cojocariu, Thermo Fisher Scientific, Runcorn, UK

### Goal

Assess the quantitative performance and productivity advantages of consolidating the analysis of PAHs and PCBs using the Thermo Scientific™ Orbitrap Exploris™ GC in addition to screening of unknown soil contaminants.

### Introduction

Polychlorinated biphenyls (PCBs) and polyaromatic hydrocarbons (PAHs) are toxic organic compounds that can contaminate soils, air, sediments, and water as a result of natural and anthropogenic processes. PCBs and PAHs are resistant to environmental degradation and can be transported over long distances. Moreover, due to their lipophilicity these chemicals can undergo biomagnification and accumulation in the food chain and can pose significant health risks to humans. Their toxicity even at very low concentrations means that their presence in the environment needs to be monitored so that the risk of uptake of these compounds into the food chain and subsequently into human populations is minimized.

More recently it has become apparent that oxidized and substituted derivatives of PAHs (such as oxy and methyl PAHs) have similar or increased toxicities compared to non-substituted versions; therefore, governments have already begun monitoring them in soil and particulate matter. Nitrogen, sulfur, and oxygen-containing polyaromatic heterocycles (NSO-PAHs) are another class of compounds that have gained interest due to their ubiquitous presence in the environment and lack of data on their toxicities.

PCBs and PAHs (and derivatives) are typically analyzed by gas chromatography (GC) coupled to mass spectrometry (MS). The challenges for the analysis of PAHs and PCBs are the requirement for complicated and costly sample preparation such as Soxhlet extraction. Often long chromatographic separations (>40 min per sample) are required, which overall will result in low sample throughput and high cost of analysis.

### Conclusions

The results of the experiments presented demonstrate that modified QuEChERS methods and the Thermo Scientific™ TriPlus™ RSH autosampler in combination with the Orbitrap Exploris GC provides an ideal solution for analytical testing laboratories looking to improve productivity and deliver confident results.

 [View the full application note](#)



TECHNICAL NOTE 10728

# Robust analysis of PAHs and PCBs in soil with over 500 repeat injections using Orbitrap Exploris GC high-resolution accurate mass

Aaron Lamb, Dominic Roberts, and Cristian Cojocariu, Thermo Fisher Scientific, Runcorn, UK

## Goal

The scope of this study was to test a simplified Thermo Scientific™ GC-Orbitrap™ analytical method over a large number of consecutive injections of soil samples to assess if it can meet the demands of routine trace analysis in soil samples.

## Introduction

Polychlorinated biphenyls (PCBs) and polyaromatic hydrocarbons (PAHs) are toxic organic pollutants that can contaminate soils, air, sediments, and water as a result of natural and human processes. PCBs and PAHs are resistant to environmental degradation and can be transported over long distances. PAHs and PCBs from the environment can enter food chains where they are very persistent and very bioaccumulative (vPvB).

PAHs and PCBs have numerous congeners, many of which have identical masses. For this reason, gas chromatography-mass spectrometry is the analytical technique of choice for their separation and quantification. Higher mass PAHs are prone to poor peak shape in gas chromatography, making peak integration challenging by impacting chromatographic resolution and sensitivity, leading to higher limits of detection and quantification. Also, the routine analysis of PAHs and PCBs in complex soil matrices requires consumables and instrumentation that deliver exceptional degrees of stability in terms of peak

areas, response factors, ion ratios, and mass accuracy so that multiple batches of samples can be analyzed day in and day out with minimal instrument maintenance, such as liner change, column trimming, MS maintenance, or tuning.

In this work the performance of the Thermo Scientific™ Orbitrap Exploris™ GC was demonstrated for the analysis of PAH and PCBs in complex soil matrices by 500 repeat injections. Orbitrap Exploris GC is ideal for routine environmental screening methods because of its ability to meet the required sensitivity and in full scan, which enables consolidation of methods through combining compound classes into a single acquisition.

## Conclusion

Results demonstrate the Orbitrap Exploris GC-MS system and sample preparation enable the consistent, uninterrupted performance needed in fast-paced routine environments looking at increasing productivity while reducing instrument downtime and the cost per sample. Demonstrated excellent system repeatability, stable QC ion ratios, mass accuracy stability, and more.

 [View the full technical note](#)



APPLICATION NOTE 10558

# Determination of polycyclic aromatic hydrocarbons in drinking water at ppt levels by Solid Phase Micro Extraction Arrow coupled with GC-MS

Manuela Bergna, Giulia Riccardino, Thermo Fisher Scientific, Rodano, Italy

## Goal

Fully automatized method for extraction and quantification of low level PAHs from drinking water using Thermo Scientific™ SPME Arrow technology.

## Introduction

Polycyclic aromatic hydrocarbons (PAHs) are considered priority environmental contaminants as they are ubiquitous persistent organic pollutants (POPs). They originate from incomplete combustion of natural deposits (such as oil, coal, tar, wood, and petroleum) and artificial sources (such as fuels, vehicle emissions, rubber, plastics, and cigarettes).

Environmental protection agencies strictly regulate the presence of PAHs in air, water, soil, industrial, and food because this group of chemicals varies in structure and toxicity. The analysis of drinking water for the presence of PAHs is important as coal tar is normally used to protect distribution pipes from corrosion and therefore can be considered the main source of PAHs in drinking water. Different maximum concentration limits are set by local regulatory agencies.

PAHs are present at very low levels (ppb and sub-ppb levels), so extraction of these compounds from matrices that are often complex is time-consuming, expensive, and a potential source of error and cross contamination. Solid phase extraction (SPE) and liquid-liquid extraction (LLE) are the most common techniques used for isolating PAHs from matrices. SPE cartridges are expensive and not reusable, while LLE requires a large volume of organic solvents for extraction procedures. Alternative techniques such as stir bar sorptive extraction (SBSE) and solid phase micro extraction (SPME) minimize sample preparation, eliminate solvent consumption, and reduce analysis cost.

## Conclusion

Overall performance demonstrates PAHs trace detection with minimal sample preparation, outstanding compound linearity over the concentration range 1–500 ng/L, and excellent peak area repeatability (%RSDs < 15). The innovative SPME Arrow fibers design ensures robustness and reliability towards mechanical stress even under fast stirring speed.



[View the full application note](#)





APPLICATION NOTE 73742

## A consolidated regulatory (or US and Chinese regulated) method for the analysis of VOCs in soil by HS-GC-MS in analytical testing laboratories

David Lee and Cristian Cojocariu, Thermo Fisher Scientific, Runcorn, UK

### Goal

To develop and test a consolidated analytical method for the analysis of volatile organic compounds in soil using headspace (HS) gas chromatography (GC) single quadrupole mass spectrometry (MS), following the US EPA 5021 sample preparation guidelines and aligned with the requirements of HJ642-2013 and HJ736-2015 Chinese standard methods.

### Introduction

Volatile organic compounds (VOCs) are defined by the United States Environmental Protection Agency (US EPA) as “any compound of carbon, excluding carbon monoxide, carbon dioxide, carbonic acid, metallic carbides or carbonates, and ammonium carbonate, which participate in atmospheric photochemical reactions.” VOCs are ubiquitous in the environment and they consist of many different classes, including both naturally occurring and man-made. Anthropogenic VOCs are generally used in fragrances, solvents, paints, cleaning materials and as fuels. Many of these compounds are considered toxic for the environment and can cause negative health effects to humans. Compounds, such as trichloroethene and toluene, are of particular concern for the environment, especially when linked to contaminated soils and sediments which to remove to a safe limit would require costly site remediations. Detecting the level of VOC contamination in these types of samples can trigger the decision to potentially start site remediation, a costly process that relies on the accuracy of VOC measurements.

There are several methods available for the analysis of VOCs including US EPA Method 524.4 for analysis in drinking water, TO-15 for analysis in air, and 8260 for analysis in a wide variety of matrices including water and soil. US EPA Method 5021 provides a general purpose method for sample preparation of VOCs in soil and is utilized as the preparation method for Chinese standards HJ642-2013 and HJ736-2015.

### Conclusion

Laboratories analyzing VOCs in environmental samples can consolidate three methods into one, increasing throughput three-fold by running one 30 minute method instead of two methods each >40 minutes, therefore significantly reducing the time from sampling to reporting and the need for multiple GC-MS systems and analysts, decreasing the cost per sample. The HS-GC-MS method presented here allows for simultaneous determination of 61 VOCs in <30 minutes per sample, using the Thermo Scientific™ TriPlus™ 500 HS with the Thermo Scientific™ TRACE™ 1310 GC and Thermo Scientific™ ISQ™ 7000 single quadrupole MS. The analytical results obtained in the experiments performed here comply with the HJ methods requirements with performance parameters.



[View the full application note](#)





APPLICATION NOTE 10702

## An automated approach for the determination of gasoline range organics (GRO) in water by gas chromatography coupled with static headspace sampling

Giulia Riccardino, Cristian Cojocariu, Thermo Fisher Scientific, Runcorn, UK

### Goal

The aim of this application note is to demonstrate the quantitative performance of the Thermo Scientific™ TriPlus™ 500 Gas Chromatography Headspace (HS) Autosampler for the determination of gasoline range organics in water.

### Introduction

Gasoline range organics (GRO) refer to hydrocarbons with a carbon range from C6 to C10 that have boiling points ranging from 60 °C to 170 °C. These chemicals are often present in the environment, especially in ground water and soil, mainly as a consequence of contamination incidents. The source of contamination can be human errors and accidents (such as oil spills) that occur when handling, storing, or transporting oil and oil products. If GRO are detected, the level of contamination needs to be determined by using quantitative analytical methods; therefore, this represents a routine application for environmental analysis laboratories. GRO are highly volatile compounds that can be easily extracted from the matrix without the need for time-consuming sample preparation. Therefore, the analytical technique of choice for this application is headspace sampling coupled to gas chromatography and mass spectrometry and/or flame ionization detection.

The headspace sampling technique coupled with gas chromatography-FID detection was employed to assess method sensitivity, precision, robustness, and linearity for quantitative assessment of GRO in water.

### Conclusions

The results demonstrate the suitability of the TriPlus 500 HS autosampler in combination with the Thermo Scientific™ TRACE™ 1310 GC-FID for GRO analysis in environmental samples and the data shows a reliable analytical tool allowing environmental laboratories to produce consistent results with outstanding analytical performance for GRO quantitative analysis in water samples.



[View the full application note](#)





APPLICATION NOTE 74086

## Robust and cost-effective regulatory analysis of total petroleum hydrocarbons in water and soil using GC-FID

Jane Cooper, David Lee, Zoe Julian, Thermo Fisher Scientific, Runcorn, UK;  
Josh Marshall, Jonathan Williams, Dŵr Cymru Welsh Water, UK

### Goal

The aim of this work was to develop a cost-effective, GC-FID approach for the analysis of TPH, while maintaining robustness for high-throughput analytical testing, that uses hexane as the extraction solvent, solid phase extraction (SPE) for water samples, and a sonication extraction for soil samples, followed by GC-FID analysis using hydrogen as the carrier gas.

### Introduction

Total petroleum hydrocarbons (TPH) describe a complex mixture of several hundred organic chemical compounds that originates from crude oil and range from light gas products to heavy fuel oils that can potentially contaminate the environment.

Compounds known to exist in some TPH fractions can affect blood, the immune system, liver, spleen, kidneys, developing fetus, and the lungs. Other TPH, such as some mineral oils, are not very toxic and are used within the food industry.

Current sample preparation and analytical technologies used for the analysis of TPH involve methods such as Soxhlet extraction for soils and liquid-liquid extraction for water samples. These methods are time consuming and require large amounts of organic solvents, with detection using infrared spectroscopy after extraction with Freon™ 113 or tetrachloromethane, which are toxic solvents.

Various regulated methods considering the analysis of TPH including ISO 16703:2011, EN ISO 14039:2004, EN ISO 9377:2000, and ASTM D7678-11.

### Conclusions

The results obtained demonstrate that the Thermo Scientific™ TRAC™E 1310 GC-FID in combination with the Thermo Scientific™ TriPlus™ RSH autosampler and Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) software offer a rapid and robust solution to laboratories that analyze TPH in environmental samples.

 [View the full application note](#)



APPLICATION NOTE 10706

## Completely cryogen-free regulatory monitoring of ozone precursors, air toxics, and oxygenated volatile organic compounds in a single run

Jane Cooper, Thermo Fisher Scientific, Runcorn, UK; Laura Miles, Markes International Ltd., Llantrisant, UK

### Goal

To demonstrate the applicability of a cryogen-free method composed of a Markes™ CIA Advantage-xr™ canister autosampler, a Markes Kori-xr™ water removal device, and a Markes UNITY-xr™ thermal desorber coupled to a dual-column Thermo Scientific™ ISQ™ 7000 single quadrupole GC-MS, for the analysis of ozone precursors, air toxics, and oxygenated volatile organic compounds in ambient air in accordance to local regulations.

### Introduction

In December 2017, the Chinese Ministry of Environmental Protection issued a document relating to the Environmental Air Volatile Organic Compound Monitoring Program (EA-VOC-MP), which requires the monitoring of 117 compounds comprising three main categories of hazardous airborne volatile pollutants, ozone precursors, air toxics, and oxygenated volatiles compounds.

Obtaining good peak shape and chromatographic separation for this combined compound list typically requires cryogenic cooling of the GC column, with the associated cost and inconvenience (in addition, many thermal desorption (TD) systems also require cryogen).

Evaluate the quantitative analysis of this challenging 117-compound target list without the use of liquid nitrogen or other cryogen, and with cycle times of less than 60 minutes per sample. The analytical system comprises a canister autosampler, water removal device, thermal desorber, and dual-column GC-MS/FID configured for heart-cut 2D-GC separation. Together, these enable the monitoring of samples at 100% relative humidity, offer optimum responses for the three C<sub>2</sub> and two C<sub>3</sub> hydrocarbon isomers using FID, as well as con dent compound identification and high sensitivity for the remaining compounds monitored using MS.

The analytical system used provides fully automated analysis for up to 27 sample channels and offers excellent method detection limits, retention time stability, reproducibility and linearity. When combined with the optimised chromatographic method and the overlap mode available (in which the next sample is loaded to the focusing trap while the current GC analysis is still running), sample-to-sample cycle times of less than 60 minutes can be achieved, maximizing laboratory productivity. The method supports analysis in accordance with US EPA Method TO-17 and Chinese EPA Method HJ 644.

 [View the full application note](#)



APPLICATION NOTE 73471

## In-Tube Extraction Dynamic Headspace (ITEX-DHS) sampling technique coupled to GC-MS for sensitive regulatory determination of odorants in water

Samantha Bowerbank, Northumbria University, UK; Andrea Romano, Thermo Fisher Scientific, Italy

### Goal

This study aims to demonstrate the applicability of the In-Tube Extraction Dynamic Headspace (ITEX-DHS) technique coupled to gas chromatography-mass spectrometry (GC-MS) for the determination of trace-level odorants in water. Through dynamic headspace enrichment achieved by means of the ITEX technique, the target analytes isoborneol (IB), 2-methyl-isoborneol (2-MIB), 2,4,6-trichloroanisole (2,4,6-TCA), and geosmin were extracted and pre-concentrated to reach sub-ppt level limits of detection (LOD).

### Introduction

The presence of malodorous compounds can impart an unpleasant smell to drinking water, negatively affecting consumer experience and generating complaints to water companies. Volatile organic compounds (VOCs) isoborneol, 2-methyl-isoborneol (2-MIB), 2,4,6-trichloroanisole (2,4,6-TCA), and geosmin carry disagreeable earthy and musty olfactory notes. These malodors are perceived by the human nose at concentrations of a few parts per trillion (ppt), whereas international standards ISO 17943 and GB 5749 request method detection limits of 10 ppt for 2-MIB and geosmin.

To reach such low detection levels, several approaches to extract and enrich the analytes prior GC-MS analysis have been published. Solventless extraction techniques such as Solid-Phase Microextraction (SPME) and Purge and Trap are currently successfully applied in routine water testing labs mainly due to the extraction efficiency, high level of automation, and extraction time compatible with routine sample throughput. In particular, the recent development of the SPME Arrow, capable of higher sorption volume and higher fiber robustness compared to classical SPME, offers a relatively inexpensive, more sensitive, and more reliable solution for odor compounds detection in water.

### Conclusion

A sensitive method was developed for the analysis of four odorants commonly found in water: isoborneol, 2-methylisoborneol, 2,4,6-trichloroanisole, and geosmin. Key parameters for the optimization of ITEX-DHS were identified, namely extraction strokes, extraction trap temperature, volume and speed, and desorption trap temperature and speed. The sensitivity obtained exceeds the requirement of 10 ng/L of the international standards ISO 17943 and GB 5749, maintaining a limited cycle time of 12 min.

 [View the full application note](#)



APPLICATION NOTE 000561

## Reproducible trace analysis of PCBs in environmental matrices using triple quadrupole GC-MS/MS

Kjell Hope, Pacific Rim Laboratories Inc., Canada; Giulia Riccardino, Thermo Fisher Scientific, Milan, IT; Adam Ladak, and Paul Silcock, Thermo Fisher Scientific, Hemel Hempstead, UK

### Goal

The aim of this application note is to demonstrate the performance of the Thermo Scientific™ TSQ™ 9610 triple quadrupole mass spectrometer coupled to the Thermo Scientific™ TRACE™ 1610 GC for the determination of polychlorinated biphenyls (PCBs) in environmental samples.

### Introduction

Polychlorinated biphenyls (PCBs) are a group of industrial organic chemicals consisting of carbon, hydrogen, and chlorine atoms.<sup>1</sup> Due to their non-flammability, chemical stability, high boiling point, and electrical insulating properties, PCBs were used in hundreds of industrial and commercial applications including electrical, hydraulic equipment, plasticizers, plastics, rubber products, and dyes. The production of these compounds has been banned in the United States since 1977 because of their persistence in the environment and their tendency to enter the food chain and bioaccumulate in living organisms due to their lipophilicity. There are currently 209 known PCB congeners that can be divided into two groups according to their structural and toxicological characteristics: the non-dioxin-like PCBs (non-DL-PCB) representing the majority of the PCB congeners with a lower degree of toxicity and the dioxin-like PCBs (DL-PCBs) representing the 12 most toxic congeners,

which have structure and toxicity similar to dioxins. DL-PCBs are classified as persistent organic pollutants (POPs), and they have been regulated under the Stockholm Convention for POPs since 2001. Following the Clean Water Act (CWA) in 1972, the United States Environmental Protection Agency (US EPA) developed an analytical method, EPA Method 1668 and following revisions, that can be applied for the determination of PCBs in wastewater, surface waters, soil, sediments, biosolids, and tissue matrices using gas chromatography coupled to high-resolution gas chromatography/high-resolution mass spectrometry (HRGC/HRMS).

### Conclusion

The results obtained in these experiments demonstrate that the TSQ 9610 mass spectrometer equipped with the NeverVent AEI ion source in combination with the TRACE 1610 GC and the Thermo Scientific™ AI/AS 1610 liquid autosampler represents a reliable tool for analysis of PCBs in environmental samples.

 [View the full application note](#)



CASE STUDY 000411

## Deliver increased sample throughput in an analytical laboratory with GC-MS/MS

Pacific Rim Laboratories

### Introduction

Analytical testing laboratories that analyze environmental and food samples are under pressure to produce consistent data at low levels of detection for compounds in a variety of matrices. Pacific Rim Laboratories in Canada faces these challenges every day. Pacific Rim is an analytical testing laboratory that analyzes a wide range of environmental and food samples for trace level contaminants. Kjell Hope, a technical research chemist at Pacific Rim explained, “We look at samples in matrixes that include soil, air (emission/ ambient), water, sediment, tissue, and food. We analyze a wide range of contaminants including dioxins, organochlorine pesticides (OCP), polyaromatic hydrocarbons (PAH), polychlorinated biphenyls (PCB), polybrominated diphenyl ethers (PBDEs) and tributyltin (TBT). Here at Pacific Rim, we typically process between 8,000 to 10,000 samples per year.”

First and foremost, Pacific Rim Laboratories must deliver accurate results that adhere to the regulations followed by their clients. Hope gave more detail on this, “Ninety percent of our methods are based on US EPA methods for compliance. Since we investigate new compounds, some may not have official US EPA or EU regulations attached, and we base those methods on ISO 17025 best practices.”

### Conclusion

Ensuring the regulatory limits for environmental and food analysis can be met day in and day out is extremely important for analytical testing laboratories. The Thermo Scientific™ TSQ™ 9610 Triple Quadrupole GC-MS/MS System offers significant advantages for targeted quantitative analysis in complex matrices, including:

- Sensitivity to exceed regulatory requirements
- Increase in sample throughput
- Reduction in training
- Consolidation of analytical methods
- Confidence in results and reduction of re-analysis
- Extended uptime with robust operation

 [View the full case study](#)

# Inorganic, trace elemental and by-product contaminants introduction

Regulations are increasingly stringent and the list of harmful substances that must be tested for is growing, requiring the most accurate and sensitive analysis solutions. We can help speed up method development to address the variety of analytical methods needed to qualitatively or quantitatively determine the inorganic, trace elemental and by-product composition of solids or liquids; including inorganic components such as cations, metals, anions or inorganic salts.

By utilizing highly sensitive and selective modern instrumentation in conjunction with pre-developed methods, we can help you modernize your test menu and achieve faster set-up to results while meeting the trace level analytical

measurement of inorganic elements, trace metals or by-product contaminants you or your clients need; ranging from parts per million (ppm) down to parts per trillion (ppt) levels.

Our analytical testing solutions and methods can help identify and measure trace compounds within solid and liquid samples to the precision and accuracy your local regulatory requirements specify while enabling operational improvements. Our breadth of solutions means we can help you address a large range of environmental sample types and easily expand your capabilities.

Click on note titles to jump to page

Matrix							US EPA	Title
Drinking Water	Surface Water	Waste Water	Soil	Sediment	Air	Other		
			●	●			6010D	US EPA SW-846 Method 6010D using the Thermo Scientific iCAP 7400 ICP-OES Duo
●							557	Extending the quantitative performance of US EPA Method 557 for haloacetic acids, bromate, and dalapon in water using an IC-MS/MS workflow
●							557	Case study: Fast, direct analysis of disinfection byproducts, including nine haloacetic acids (HAA9), bromate, and dalapon using IC-MS/MS per US EPA Method 557
●							557	Fast, verified and validated determination of nine haloacetic acids, bromate, and dalapon at trace levels by tandem IC-MS/MS
●							551.1	Analysis of halogenated disinfection byproducts and chlorinated solvents in drinking water by GC-dual ECD
●							332.0	Tomorrow's quantitation: robust, reproducible quantitation workflows of perchlorate in water with IC-MS/MS
●							300.0 & 300.1	Determination of inorganic anions in drinking water using a compact ion chromatography system for US EPA Method 300.1
●							300.0 & 300.1	Municipal drinking water analysis by fast IC for US EPA Method 300.0 and 300.1

*Continued on next page*

Click on note titles to jump to page

Matrix							US EPA	Title
Drinking Water	Surface Water	Waste Water	Soil	Sediment	Air	Other		
●	●	●					300.0 & 300.1	<a href="#">Low-cost determination of inorganic cations and ammonium in environmental waters for US EPA Method 300.1</a>
●							200.8	<a href="#">Fully automated, intelligent, high-throughput elemental analysis of drinking waters using SQ-ICP-MS for US EPA 200.8</a>
●							200.7	<a href="#">US EPA Method 200.7 using the Thermo Scientific iCAP PRO XPS Duo ICP-OES</a>
●	●						200.7	<a href="#">Rapid, interference-free analysis of water according to US EPA Method 200.7</a>
●								<a href="#">Regulatory analysis of environmental waters for cations and ammonium using a compact ion chromatography system</a>
●	●	●						<a href="#">Determination of water pollutants using photometric analysis for compliance with ISO 15923-1</a>
	●	●						<a href="#">Multi-element analysis of surface and waste waters using the Thermo Scientific iCAP TQ ICP-MS with prepFAST autodilution</a>
●		●				●		<a href="#">Direct analysis of environmental samples using ICP-MS with argon gas dilution (AGD)</a>
●	●	●						<a href="#">Fast and robust assessment of water quality using ICP-OES for DIN EN/ISO 11885:2009</a>
		●						<a href="#">Simultaneous determination of tetrabutylammonium and tributylamine in wastewater by IC/LC-MS</a>
●								<a href="#">Discovery of emerging disinfection by-products in water using gas chromatography coupled with Orbitrap-based mass spectrometry</a>

# US EPA SW-846 Method 6010D using the Thermo Scientific iCAP 7400 ICP-OES Duo

James Hannan, Thermo Fisher Scientific, Hemel Hempstead, UK

## Goal

This application note describes the performance of the Thermo Scientific™ iCAP™ 7400 ICP-OES Duo for the analysis of environmental sample types by the US EPA Method 6010D. The iCAP 7400 ICP-OES Duo achieves short analysis times per sample whilst maintaining compliance with the protocol.

## Introduction

On 21 October 1976, the United States (US) congress enacted the Resource Conservation and Recovery Act (RCRA), which governs the disposal of solid and hazardous waste. Guideline methods for the analysis of these types of samples are collated under 'Test methods for evaluating solid waste, physical/chemical methods' Solid Wastes (SW)-846. One of the specified methods is US Environmental Protection Agency (EPA) Method 6010D 'Inductively Coupled Plasma-Optical Emission Spectrometry'. This method prescribes the use of Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES) instrumentation for the determination of 31 elements in groundwaters, industrial and organic wastes, soils, sludges and sediments. All samples, except filtered groundwater, must be digested prior to analysis using either US EPA Method

3050B 'Acid Digestion of Sediments, Sludges, and Soils' or US EPA Method 3051A 'Microwave Assisted Digestion of Sediments, Sludges, Soils, and Oils'. US EPA Method 6010D is used within the USA and its variants are widely used in other regions, therefore the ability to undertake this type of analysis using US EPA Method 6010D represents an important benchmark for any ICP-OES instrument.

## Conclusion

The Thermo Scientific iCAP 7400 ICP-OES Duo exceeds the requirements needed to meet the US EPA Method 6010D protocol. The instrument minimizes spectral interferences and reduces stray light and enables higher sensitivity and lower noise; producing a high signal to background ratio. Using the method as described, the sample analysis was 2 minutes 35 seconds; reducing system waste and minimizing both the cost of analysis and its environmental impact. The simplicity of the Qtegra ISDS Software enables complex procedures to be easily adhered to for maximum quality control.

 [View the full application note](#)

## Extending the quantitative performance of US EPA Method 557 for haloacetic acids, bromate, and dalapon in water using an IC-MS/MS workflow

Charles Maxey, Neloni Wijeratne, Scott Peterman, Claudia Martins, Ed George, Thermo Fisher Scientific, San Jose, CA; Alex Semyonov, Thermo Fisher Scientific, Austin, TX

### Goal

Development and easy implementation of a robust, reliable, and reproducible work ow solution for analysis and quantitation of nine haloacetic acids, bromate, and dalapon in water using ion chromatography coupled to a triple quadrupole MS.

### Introduction

Clean drinking water is becoming scarcer, and any contamination can result in long lasting damage to human health. Strategies used to purify water include mechanical measures and disinfection, which utilizes chlorination to remove microbial content. The disinfection process, however, can introduce by-products that can result in health risks. The primary class of compounds associated with drinking water contamination is disinfection by-products (DBPs). Haloacetic acids (HAAs) are a subgroup of DBPs, which are specifically linked to cancer and other issues.

Continued research focused on identifying more potentially harmful compounds requires analytical workflows to be responsive to new requirements determining DBP concentration levels prior to release. Standard analytical methods coupling liquid chromatography to triple quadrupole mass spectrometers to perform selected-reaction monitoring (SRM) are the preferred methods

due to the selectivity, sensitivity, and analysis speed. Traditional separation methods using liquid chromatography (LC) often require costly derivatization to effectively separate target compounds prior to ionization and analysis. Ion chromatography (IC), however, effectively and efficiently retains and separates the polar compounds, improving the overall IC-MS/MS workflow to meet current regulations as well as maximize work ow robustness.

### Conclusion

A straightforward fast IC-MS/MS method resulting in reproducible, sensitive quantitation of the HAAs to exceed US EPA method requirements. Compared to the conventional US EPA methods using GC with electron capture, IC-MS/MS offers significant time savings for sample preparation. All analytes in this assay were detected to the lowest calibration level, and accuracy is within the US EPA specified criteria. The resolution between the matrix peaks and HAAs is excellent, allowing for minimum interference in detection and ensuring a cleaner ion source of the MS.

 [View the full application note](#)

## Fast, direct analysis of disinfection byproducts, including nine haloacetic acids (HAA9), bromate, and dalapon using IC-MS/MS per US EPA Method 557

Erie County Public Health Laboratory

### Goal

Determine if moving from a GC-ECD to an IC-MS/MS analytical method for haloacetic acid analysis can save substantial analyst time performing extractions.

### Introduction

Clean drinking water is critically important to human health. Typically, both mechanical and chemical processes are needed to ensure drinking water quality; however, the by-products of chemical processes can include chlorinated to halogenated acids (HAAs) and bromate among others. Because excessive consumption of these compounds can result in severe health issues, drinking water regulations require determination of the concentrations of disinfection by-products (DBPs) prior to release to the public.

Depending on the analytical method chosen, analysis and quantitation of DBPs can pose several challenges including time-consuming and tedious sample preparation and poor recoveries. The Erie County Public Health Laboratory has addressed these challenges with the adoption of a direct-injection ion chromatography-tandem mass spectrometry (IC-MS/MS) method that uses the Thermo Scientific™ Dionex™ ICS-5000 HPIC™ System coupled to the Thermo

Scientific™ Quantiva™ triple quadrupole mass spectrometer (equivalent to the Thermo Scientific™ Dionex™ ICS-6000 HPIC™ system and Thermo Scientific™ TSQ Fortis™ triple quadrupole mass spectrometer). Quantitation of target DBPs in drinking water per US EPA Method 557 is robust, reliable, reproducible, and requires no sample preparation, allowing significant time, hazardous waste, and cost savings. Most significantly, Erie County Public Health Laboratory has reduced HAA determination time from approximately four hours to about 45 min per sample.

### Conclusion

By eliminating lengthy extractions, shortening chromatographic separations, and using eluent generation, the Thermo Fisher Scientific total solution enables the laboratory to analyze 20 samples per day, meeting US EPA batch size recommendations.

 [View the full case study](#)

## Fast, verified and validated determination of nine haloacetic acids, bromate, and dalapon at trace levels by tandem IC-MS/MS

Xin Zhang, Charanjit Saini, Chris Pohl, and Yan Liu, Thermo Fisher Scientific, Sunnyvale, CA

### Goal

To identify and quantify low concentrations of haloacetic acids, bromate, and dalapon in drinking water according to US EPA Method 557 using a Thermo Scientific™ Dionex™ ICS-6000 ion chromatography system and a Thermo Scientific™ Dionex™ IonPac™ AS31 column coupled with triple quadrupole electrospray mass spectrometry

### Introduction

Haloacetic acids (HAAs) are a class of undesirable disinfection by-products (DBPs) formed during the disinfection of drinking water in which routine water disinfectants such as chlorine or chloramine are used to kill pathogenic microorganisms. There are nine major HAAs: monochloroacetic acid (MCAA), dichloroacetic acid (DCAA), trichloroacetic acid (TCAA), monobromoacetic acid (MBAA), dibromoacetic acid (DBAA), tribromoacetic acid (TBAA), bromochloroacetic acid (BCAA), bromodichloroacetic acid (BDCAA), and chlorodibromoacetic acid (CDBAA). Because of their suspected carcinogenicity, mutagenicity, as well as developmental, reproductive, and hepatic toxicity, the World Health Organization (WHO) has established guidelines for these DBPs in drinking water. In the US, these guidelines are regulated by the Environmental

Protection Agency (EPA) as a part of the Safe Drinking Water Act (SDWA). In 1998, the Stage 1 Disinfectants and DBPs Rule (Stage 1 DBPR) was published, which set the limit for total trihalomethanes (TTHM) at 80 µg/L and, for the first time, set the maximum contamination levels for the sum of the five HAAs (HAA5: MCAA, DCAA, TCAA, MBAA, and DBAA) at 60 µg/L. It also sets a maximum contaminant level goal (MCLG) for dichloroacetic acid (DCAA) to zero and trichloroacetic acid (TCAA) to 30 µg/L. In the Stage 2 DBPR, the MCLG for TCAA was reduced to 20 µg/L and MCAA was set at 70 µg/L. Consequently, efforts have been made to develop fast and accurate analytical methods to monitor concentration, behavior, and distribution of HAAs in water.

### Conclusion

A fast, sensitive, and simple method was developed for direct analysis of nine HAAs, bromate, and dalapon in drinking water samples using IC-MS/MS without sample pretreatment.

 [View the full application note](#)



APPLICATION SUMMARY 73454

# Analysis of halogenated disinfection byproducts and chlorinated solvents in drinking water by GC-dual ECD for US EPA Method 551.1

David Lee and Cristian Cojocariu, Thermo Fisher Scientific, Runcorn, UK

## Goal

To assess the performance of the Thermo Scientific™ TRACE™ 1310 Gas Chromatograph with dual column and dual ECD setup for the analysis of halogenated disinfection byproducts and chlorinated solvents in drinking water.

## Introduction

Most countries across the world have set regulatory limits for halogenated disinfection byproducts and chlorinated solvents in drinking water supplies as these chemicals can have serious health effects if present above certain levels. The chlorinated solvents can enter the water supply through accidental spillage, leakage from disposal sites, or deliberate discharge from factories, whereas disinfection byproducts are formed via chemical reactions with organic material present during the water treatment process.

Traditionally, the analytical method of choice for the preparation, detection, and quantification of such compounds is liquid-liquid extraction of water using an organic solvent, followed by analysis using gas chromatography (GC) coupled to an electron capture detector (ECD). Detection limits using ECDs are typically in the femtogram region and can be more sensitive than mass spectrometry for halogenated compounds. The identification of the analytes is then confirmed by running the extract again on a second column phase or by mass spectrometry.

## Conclusion

This simple, cost-effective yet robust and sensitive analytical configuration can be easily implemented in commercial testing laboratories for the assessment of halogenated disinfection byproducts and chlorinated solvents in drinking water samples to support US EPA Method 551.1 and the United Kingdom Drinking Water Inspectorate requirements.

- Excellent peak shape and chromatographic resolution were obtained with simultaneous peak identity and confirmation thanks to the dual-column dual-detector configuration.
- Outstanding sensitivity with femtogram on column IDL levels were achieved.
- Excellent linearity was obtained with  $R^2$  values  $\geq 0.995$  and AvCF %RSD  $< 11$  for all analytes.
- Quantification of real water samples, spiked and unspiked, resulted in compound recoveries between 80 and 120% and %RSD of calculated concentration  $< 10$  for  $n=7$  replicates of spiked sample for all investigated analytes.



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## Tomorrow's quantitation: robust, reproducible quantitation workflows of perchlorate in water with IC-MS/MS

Neloni Wijeratne, Claudia Martins, Jonathan Beck, Thermo Fisher Scientific, San Jose, CA;  
Debadeep Bhattacharyya, Thermo Fisher Scientific, Boston, MA;  
Beau Sullivan, Tae Pak, Andrey Khorst, Santa Clara Valley Water District, San Jose, CA

### Goal

Development and easy implementation of a robust, reliable, and reproducible workflow solution for the analysis and quantitation of perchlorate in water using a triple-stage quadrupole mass spectrometer (MS).

### Introduction

Clean drinking water in today's world is becoming scarce, and any contamination can result in long-lasting damage to human health. Besides purifying water by means of mechanical measures, disinfection also plays an essential role in ensuring the supply of clean drinking water. Drinking water goes through an extensive disinfection process to ensure high quality; however, contaminants continue to cause some severe health risks. In 1997, low-level perchlorate contamination (<50 ng/mL or parts per billion) was discovered in the Western US. Since then, perchlorate has been found in sites scattered around the nation. Although the US Environmental Protection Agency has not established a regulation for perchlorate in drinking water, it has placed perchlorate on the contaminant candidate list (CCL) and the unregulated contaminants monitoring rule (UCMR).

### Conclusion

The presence of perchlorate can cause significant health issues, especially in drinking water. Analysis and confident quantitation of perchlorate in water can pose several challenges, especially in view of their increasing complexity. A reagent-free IC system coupled with the Thermo Scientific™ TSQ Fortis™ MS is a powerful platform solution that can enable development of robust, reproducible, fast, and sensitive quantitation assays for perchlorate in water.

The sensitivity observed for the calibration standard provided by SCVWD was very high, hence, the standards were further diluted with Milli-Q water. All the analytes in this assay were detected to the lowest calibration level and the accuracy is within the criteria. The limit of detection was less than 0.002 µg/L based on the criterion of signal-to-noise ratio of greater than 3. The prepared quality control (QC) samples from ERA were found to be within the vendor acceptance limits of 8.88 to 12.3 µg/L and with % RSDs of 0.7 and 1.5 for parent ions of 99 and 101, respectively. Last, but not least, the optimal performance of the Thermo Scientific™ Dionex™ ICS-5000+ HPIC™ system and TSQ Fortis MS platform solution exhibited excellent reproducibility and quantitation of perchlorate in water samples.

 [View the full application note](#)

# Determination of inorganic anions in drinking water using a compact ion chromatography system for US EPA Method 300.1

Manali Aggrawal and Jeff Rohrer, Thermo Fisher Scientific, Sunnyvale, CA

## Goal

To demonstrate inorganic anion determinations in drinking water samples according to US EPA Method 300.1 (A) using a Thermo Scientific™ Dionex™ Aquion™ IC system.

## Introduction

The determination of inorganic anions in municipal drinking, waste, and bottled waters according to the US Environmental Protection Agency (EPA) Methods 300.0 and 300.1 is one of the most popular and widely used ion chromatography (IC) methods. We used a Thermo Scientific™ Dionex™ Aquion™ ion chromatography system with a Thermo Scientific™ Dionex™ AS-DV Autosampler to determine seven anions (fluoride, chloride, bromide, nitrite, nitrate, phosphate, and sulfate) in drinking water samples. The Dionex Aquion IC system is a simple, inexpensive, and compact platform with straightforward operation for basic ion analysis. In this study, mg/L concentrations of inorganic anions were separated on a 4 × 250 mm Thermo Scientific™ Dionex™ IonPac™ AS22 anion-exchange column using a carbonate/bicarbonate eluent. Eluent was prepared by diluting the Thermo Scientific™ Dionex™ AS22 Eluent Concentrate for ease-of-use and to minimize eluent preparation errors. Following the separation, anions were detected using suppressed conductivity detection with a Thermo Scientific™ Dionex™ AERS™ 500 Carbonate

Electrolytically Regenerated Suppressor. This technical note provides detailed instructions for system set up and operation for determining inorganic anions in samples such as drinking water.

## Conclusion

- In this technical note we demonstrated that US EPA Method 300.1 (Determination of inorganic anions in drinking water by ion chromatography) could be successfully executed on a Dionex Aquion IC system.
- Drinking water samples were successfully analyzed for the seven common inorganic anions.
- The method showed good precision and accuracy for all seven anions with recoveries from 95 to 104%.

 [View the full technical note](#)

# Municipal drinking water analysis by fast IC for US EPA Method 300.0 and 300.1

Terri Christison and Khalil Divan, Thermo Fisher Scientific, Sunnyvale, CA

## Goal

To increase sample throughput for US EPA Methods 300.0 (Part A) and 300.1 (Part A) using the Thermo Scientific™ Dionex™ IonPac™ AS22-Fast anion-exchange column.

## Introduction

The determination of inorganic anions (fluoride, chloride, nitrite, sulfate, bromide, nitrate, and phosphate) in municipal drinking water is one of the most important ion chromatography (IC) applications worldwide. In the United States, water integrity is legislated through the Safe Drinking Water Act (SDWA), which ensures water quality and safety. Other industrialized countries have similar regulations and, therefore, have similar analytical needs.

Since the 1980s, with the approval of EPA Method 300.0 (Part A), Thermo Scientific™ Dionex™ IC methods have been used for compliance testing of inorganic anions. In 1993 and 1997, methods using Thermo Scientific™ Dionex™ IonPac™ AS4A and Thermo Scientific™ Dionex™ IonPac™ AS9-HC anion-exchange columns were specified in Methods 300.0 (Part A) and 300.1 (Part A). As advancements in column technology continued, new columns were proposed, such as the Thermo Scientific™ Dionex™ IonPac™ AS14 column in Dionex AN 133 in 2004. However, both methods have run times of more than 14 min.

## Conclusion

This method using the Dionex IonPac AS22-Fast column provides an equivalent method—using the latest column technology—to the EPA 300.0 (Part A) and 300.1 (Part A) approved methods with the benefits of:

- A 5 min run time
- Reducing cycle time
- Lowering the overall cost
- Increasing the sample throughput

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APPLICATION NOTE 73728

## Low-cost determination of inorganic cations and ammonium in environmental waters for US EPA Method 300.1

Hua Yang and Jeffrey Rohrer, Thermo Fisher Scientific, Sunnyvale, CA

### Introduction

In the United States (US), water quality is regulated through the Safe Drinking Water Act (SDWA) and the Clean Water Act (CWA) and enforced through the United States Environmental Protection Agency (US EPA). As a well-accepted technique, ion chromatography (IC) has been used for compliance monitoring inorganic anions in water, including surface, ground, drinking, and waste waters following US EPA Methods 300 and 300.1. Although the common alkali and alkaline earth cations, and ammonium are not considered primary drinking water contaminants in the US, they are routinely monitored and reported here and are regulated in Europe and Japan. Ammonium is measured in the US for waste water discharge compliance according to the criteria for the protection of aquatic life from the toxic effects of ammonia in freshwater published by US EPA in 2013.

This work demonstrates the determination of cations and ammonium in environmental waters by IC using a Thermo Scientific™ Dionex™ IonPac™ CS16 column set on a Thermo Scientific™ Dionex™ Easion™ Chromatography system in Displacement Chemical Regeneration (DCR) mode. The Dionex Easion IC system is an integrated, single-channel low-cost system designed for isocratic applications with suppressed conductivity detection. Coupled to the Thermo Scientific™ Dionex™ AS-DV Autosampler, it provides a low-cost choice for routine water analysis.

### Conclusion

This work shows the determination of inorganic cations and ammonium in a drinking water and a waste water sample using the Dionex Easion IC system in DCR mode. The integrated Dionex Easion IC system coupled with the Dionex AS-DV autosampler provides a simple and low-cost instrument setup for routine determination of inorganic cations and ammonium in water samples.

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## Fully automated, intelligent, high-throughput elemental analysis of drinking waters using SQ-ICP-MS for US EPA Method 200.8

Marcus Manecki, Daniel Kutscher, Christoph Wehe, Robert Henry, Julian Wills, and Shona McSheehy Ducos, Thermo Fisher Scientific

### Goal

To demonstrate robust high-throughput analysis of environmental samples using SQ-ICP-MS in He-KED mode, in accordance with the requirements of US EPA Method 200.8 revision 5.5 and to demonstrate the performance of the Thermo Scientific™ iCAP™ RQ ICP-MS coupled to the ESI prepFAST™ autodilution system.

### Introduction

EPA Method 200.8 analyses for the quantification of trace metals in drinking and waste waters are performed routinely in many laboratories. Thousands of analyses are performed per week to support the monitoring and control of drinking water contaminants and water quality. Due to the complexity of the standard operating procedure (SOP), skilled technicians are required to setup and prepare the daily analysis, as well as actively monitor the results and perform further sample manipulation as required throughout the analytical run. The need for technical staff is a factor that keeps the overall expense of routinely running the 200.8 method relatively high.

Recent advances in autodilution offer the potential to automate much of the sample preparation and data review with automated re-runs of any samples that do not meet predefined limits. By automatically creating a calibration set of standards from one stock standard and then diluting each sample to a predefined dilution level, an autodilution system can save valuable analysts' time and reduce costs overall through the lowered consumption of utilities and lab supplies.

### Conclusion

It is possible to run the entire analysis (encompassing sample dilution, calibration, and measurement) with minimal manual intervention.

- Successfully validated for use with US EPA Method 200.8
- Readily achieved the goal of 52 EPA Method 200.8 analyses per hour
- Internal standard recovery was well within the EPA Method 200.8 requirements of 60 to 125%

The iCAP RQ ICP-MS in combination with the ESI prepFAST autodilution system is ideal to measure environmental samples in a high throughput commercial testing laboratory.

 [View the full application note](#)

# US EPA Method 200.7 using the Thermo Scientific iCAP PRO XPS Duo ICP-OES

Sanja Asendorf, Thermo Fisher Scientific, Bremen, Germany

## Goal

To describe the use of the Thermo Scientific™ iCAP™ PRO XPS Duo ICP-OES for the analysis of water samples using US EPA Method 200.7.

## Introduction

The analysis and monitoring of natural, produced and drinking waters is essential to ensure both human and environmental health. Levels of permissible contamination are controlled by local, national and international regulations. In the United States of America the Environmental Protection Agency (EPA) is the body responsible to set and regulate national standards for the quality of supplied drinking water and drinking water resources, such as ground waters. The EPA Office of Ground Water and Drinking Water (OGWDW) administers control under the Federal Regulation 40 CFR part 141 & 143. This regulation states that all supplied waters must comply with the Maximum Contaminant Levels (MCL) for the contaminants specified in the National Primary Drinking Water Regulations (NPDWR). The MCL and Maximum Contaminant Level Goals (MCLG) that the EPA defines as the maximum level of an element in drinking water at which no known or anticipated adverse effect on the health of persons would occur. Further contaminants are given suggested maximum values in the National Secondary Drinking Water Regulations (NSDWR) as these elements will affect water properties such as taste and color. The Unregulated Contaminant Monitoring Rule 3 (UCMR-3) requires that measurements are taken and recorded for two areas at every water treatment plant; the metals to be tested and their Maximum Reporting Limits (MRL).

## Conclusion

The Thermo Scientific iCAP PRO XPS Duo ICP-OES demonstrated compliance with the requirements of EPA Method 200.7 for a wide range of water sample types.

- Quantitative recovery can be achieved in a variety of real environmental matrices. All spike recoveries were well within the allowable range of 85 to 115%.
- Relative standard deviations (RSDs) of these measurements were within 5% across the duration of the run.
- Rapid analysis times were achieved. Samples in this study were processed at a speed of 1 sample every 1 minutes and 58 seconds, or 30 samples per hour.
- Fast start up ensures the system is purged and stable within minutes to allow for maximum instrument utilization during a working day.
- Combined features make the iCAP PRO XPS ICP-OES the ultimate instrument for cost-effective elemental analysis.

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APPLICATION NOTE 000056

## Rapid, interference-free analysis of water according to US EPA Method 200.7

Bhagyesh Surekar, Thermo Fisher Scientific, Bremen, Germany; Cristian Cojocariu, Matthew Cassap, Thermo Fisher Scientific, Hemel Hempstead, UK; Sabrina Antonio, Thermo Fisher Scientific, US

### Goal

To develop a complete solution for the fast analysis of various water samples using the Thermo Scientific™ iCAP™ PRO Series ICP-OES Duo system according to the requirements of United States Environmental Protection Agency Method 200.7, including a demonstration of accuracy, robustness, and sensitivity, with application of interference corrections and quality control protocols.

### Introduction

Monitoring natural and drinking waters is essential to ensure the well-being of an entire eco-system and consumer health. Levels of permissible organic and inorganic contaminants are regulated by local, national, and international legislation. In the United States of America, the Environmental Protection Agency (EPA) is responsible for setting and regulating national standards for the quality of drinking water and drinking water sources. In order to assess the quality of drinking, ground, and surface waters for the presence of inorganic contaminants (e.g., trace metals), laboratories follow EPA Method 200.7: Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma–Atomic Emission Spectrometry (also known as Inductively Coupled Plasma–Optical Emission Spectrometry). This method details the preparation and analysis of various types of water samples using ICP-OES.

ICP-OES is widely used for the analysis of major and trace elements in various types of samples (e.g., food, environmental) due to its ability to detect low elemental concentrations, robustness, and wide linear dynamic range.

### Conclusion

Optimizing the EPA 200.7 method for speed and robustness without compromising key performance criteria is possible. The results demonstrate the Thermo Scientific™ iCAP™ PRO XP ICP-OES Duo instrument can meet the requirements of EPA Method 200.7 for method detection limits, linear dynamic range, accuracy, instrument performance with IPC and QCS solution, and robustness. For busy testing laboratories, the workload demand can be met without compromising quality.

 [View the full application note](#)

## Regulatory analysis of environmental waters for cations and ammonium using a compact ion chromatography system

Carl Fisher, Thermo Fisher Scientific, Sunnyvale, CA

### Goal

To determine inorganic cations and ammonium in environmental waters using a compact high-pressure ion chromatography system with a 4 µm particle cation-exchange column to shorten analysis time.

### Introduction

Common alkali and alkaline earth cations are not considered primary drinking water contaminants in the US, but they are monitored and reported by many public water suppliers and are regulated in the EU and Japan.

While calcium and magnesium are essential minerals that have beneficial human health effects, they are routinely measured to determine water hardness, an important parameter for control of corrosion and scaling.

Ammonia is a colorless, pungent gas that is highly soluble in water, where it exists in equilibrium between a molecular form associated with water and the ionized form (the ammonium cation,  $\text{NH}_4^+$ ). Its toxicity to aquatic life depends upon the extent of dissociation, which in turn depends upon temperature and pH. Ammonia can enter environmental waters as a product of anaerobic decomposition of nitrogen-containing compounds or from waste streams containing ammonia. Ammonium, which is formed upon dissolution of ammonia in water, is routinely measured in the US for wastewater discharge compliance monitoring and in the EU and Japan in both wastewater and drinking water.

### Conclusion

- The high pressure capability of the Thermo Scientific™ Dionex™ Integriion™ HPIC™ system allowed a flow rate of 0.9 mL/min to be used, which reduced the run time from 25 min to just over 18 min.
- The high capacity and resolution of the Thermo Scientific™ Dionex™ IonPac™ CS16-4µm column enabled the analysis of a wide range of environmental waters, and resolved trace ammonium in the presence of a 10,000-fold higher concentration of sodium.
- The retention time precision of 0.03% or less and the peak area precision of 0.2% or less for standards in the low mg/L range demonstrated the exceptional reproducibility of the analytical solution presented.

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# Determination of water pollutants using photometric analysis for compliance with ISO 15923-1

Marco Rastetter, Jo Gawler, Mari Kiviluoma, and Annu Suoniemi-Kähärä, Thermo Fisher Scientific

## Goal

To demonstrate compliance with the ISO 15923-1 standard for the determination of water pollutants using an automated photometric procedure.

## Introduction

Clean water in sufficient amounts is an ongoing global challenge. Water resources are experiencing increasing pressure in many parts of the world, requiring countries to improve the management and protection of water ecosystems. In 2000, the European Union's (EU) Water Framework Directive (WFD) announced an integrated approach to protecting water quality, quantity, and the role of habitat. As a result, governmental bodies of different EU member states are required to work with one another to ensure that the requirements of the directive are met and water quality is maintained. Quality criteria include nutritional composition as well as chemical composition, i.e. the level of pollutants.

The International Organization of Standardization (ISO) develops standards to benefit state authorities, regulatory bodies, and industry for the purpose of equitably and durably managing shared water resources. As a network composed of 163 countries, ISO published more than 19,500 international standards covering almost every industry from technology and food safety to agriculture and healthcare. More than 550 standards currently exist to address

water issues and these quality standards provide a common language for water sampling, reporting, and monitoring in order to ensure purity and other desired characteristics that apply to industrial processes as well as natural water.

## Conclusion

- The Gallery and Aquakem analyzer methods meet or exceed the detection limits of the ISO 15923-1 standard.
- The Aquakem and Gallery discrete analyzers rapidly perform each of the seven required ISO 15923-1 tests from one sample; results for one sample are available in 18 minutes.
- One hundred sulfate tests are complete in only 25 minutes.
- These analyzers are designed to use small volumes of water and reagents and are therefore environmentally friendly solutions for the determination of ions and several other water quality parameters, such as alkalinity, total hardness, pH, and conductivity.

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## Multi-element analysis of surface and waste waters using the Thermo Scientific iCAP TQ ICP-MS with prepFAST autodilution

Simon Lofthouse, Claudia Charko, Thermo Fisher Scientific; Kristof Tirez, Vito NV, MOL, Belgium; Johan Annys, Flanders Environment Agency (VMM), Aalst, Belgium

### Goal

To demonstrate routine automated multi-element analysis of environmental samples using triple quadrupole ICP-MS.

### Introduction

The adverse effects of increased concentrations of heavy metals in the environment and their potential impact on human, animal and plant health are of key concern. Consequently, monitoring of the content of these elements in a variety of environmental matrices (ground and surface waters, drinking water, waste waters, soils and sediments) is one of the most frequently performed analyses in the context of environmental legislation. Some of the most common guidelines for different regions are summarized

Elemental analysis of environmental waters usually requires a number of different analyses to cover the different elements depending on element chemistry, interferences and linear range. Ideally, a single technique that could measure all environmentally relevant elements from a single sample, based on an easy sample preparation procedure (e.g. dilution or digestion), and with potential for automated and unattended analysis is desirable.

### Conclusion

Triple quadrupole ICP-MS is a viable tool for commercial testing laboratories performing the analysis of environmental water samples, such as ground water, surface water and waste water.

- In combination with the fully integrated prepFAST in-line dilution system, automatic dilution of all samples effectively minimizes operator interaction and hence frees up valuable resources.
- The Thermo Scientific™ iCAP™ TQ ICP-MS meets the requirements of WAC / III / B / 011 with analyte recoveries of 89–114%.
- The total measurement time for 68 elements, including stabilization time for the use of the different gases, can be limited to less than 3 minutes per sample, assuring the highest throughput.



[View the full application note](#)

## Direct analysis of environmental samples using ICP-MS with argon gas dilution (AGD)

Tomoko Vincent, Thermo Fisher Scientific, Bremen, Germany

### Goal

To demonstrate the applicability of the Thermo Scientific™ iCAP™ RQ ICP-MS with Argon Gas Dilution (AGD) for performing direct, high-throughput analysis of relevant trace elements in complex environmental samples (sea water, waste water, or drinking water).

### Introduction

Analyzing high matrix samples for their trace elements content is a known challenge in ICP-MS. The complexity of the sample matrix can significantly affect system sensitivity, cause intense fluctuation of the internal standard signals, and lead to increased system maintenance with unwanted downtime due to clogging the orifice of the interface cones or tip of the nebulizers.

A typical environmental sample that is often analyzed for trace elements content is sea water (with average salinity of 3.5%), which contains high amounts of Na, K, Ca, Mg, S, and Cl. In order to analyze such complex samples, dilution has to be performed, either using liquid dilution or, alternatively, using an inert gas such as argon. However, reducing the impact of the matrix by sample dilution is not the only challenge for this analysis. In addition, the different composing elements in the matrix can cause significant interferences (mostly polyatomic in nature), leading to potentially false positive results. As the concentration of

most common contaminants in sea water is at trace or even ultratrace level (below 10 µg·L<sup>-1</sup>), a high sensitivity ICP-MS system with powerful and effective interference removal capabilities is required in order to obtain accurate results.

### Conclusion

The experiment performed demonstrates the iCAP RQ ICP-MS system equipped with AGD and operated in STD and KED modes enables routine analysis of ultratrace multi-elements in complex samples such as sea water. With run cycle time/sample of 2.2 min, matrix interference eliminated without sacrificing sensitivity or robustness the system configuration is ideal to enable high throughput environmental sample analysis.

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## Fast and robust assessment of water quality using ICP-OES for DIN EN/ISO 11885:2009

Sukanya Sengupta, Thermo Fisher Scientific, Germany; Sven Gebhardt, Niedersächsisches Landesgesundheitsamt, Aurich, Germany; Cristian Cojocariu, Thermo Fisher Scientific, UK

### Goal

To demonstrate the suitability of the Thermo Scientific™ iCAP™ PRO Series ICP-OES for fast, sensitive and robust analysis of major and trace elements in various water samples, including drinking water, following the DIN EN ISO 11885:2009 requirements.

### Introduction

Even in small quantities, many elements can have toxic effects when present in drinking supplies or food chains. As such, monitoring the trace elemental composition of drinking, waste, and surface waters (such as rivers, lakes, and ponds) is vital in the maintenance of human, animal, and environmental health.

The approach adopted by many nations to ensure water quality is to work to a set of standard guidelines published by a national body. One such body is the Deutsches Institut für Normung - DIN (German Institute for Standardisation), which is the German member body of the International Organization for Standardization (ISO) and is responsible for setting and defining technical standards. ISO sets standards internationally and one such standard, vital for human and environmental health, is the method specified in DIN EN ISO 11885:2009 Water quality—Determination of selected elements by inductively coupled plasma optical emission spectrometry.

The DIN EN ISO 11885:2009 method contains performance guidelines and recommends the use of inductively coupled plasma optical emission spectroscopy (ICP-OES) as the analytical instrumentation of choice for assessing both major and trace elements in water samples. ICP-OES instruments are particularly well suited for this analysis as they can effectively perform rapid, low level multielement measurements in a single analytical method (compared to single element techniques such as AAS). However, a common challenge for environmental laboratories analyzing large sample numbers each day is optimizing sample throughput without compromising the sensitivity and productivity, while providing high accuracy, precision, and low limits of detection for key elements.

### Conclusion

The suitability of the Thermo Scientific™ iCAP™ PRO XP ICP-OES Duo instrument for water analysis according to the requirements and guidelines described in the DIN EN ISO 11885:2009 method. Fast, highly sensitive, reliable, and robust analysis of different kinds of water sample types was achieved with the use of three different iCAP PRO XP ICP-OES Duo instruments in two different laboratories, demonstrating the excellent data reproducibility, reliability, and robustness of the instruments.

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## Simultaneous determination of tetrabutylammonium and tributylamine in wastewater by IC/LC-MS

Christian Bukh, Thermo Fisher Scientific, Hvidovre, Denmark; Johannes Ritter, Thermo Fisher Scientific GmbH, Vienna, Austria; Detlef Jensen, Thermo Fisher Scientific GmbH, Dreieich, Germany

### Goal

To develop a robust, fast, selective, and sensitive chromatographic method to determine tributylamine and tetrabutylammonium in industrial wastewater.

### Introduction

Trialkyl amines (TAMs) and quaternary ammonium compounds (QACs) are known as environmental contaminants, with negative impacts on aquatic biota. QACs, in particular, are resistant toward biological degradation, which mandates their trace determination. The analytical speciation of TAMs and QACs relies heavily on gas chromatography (GC) and high-performance liquid chromatography (HPLC). Existing approaches describe separate workflows for TAMs and QACs.

TAMs are used as specific organic markers to trace their transport and impact on marine habitats. Valls et al. characterize TAMs as ubiquitous contaminants in the aquatic environment. They have been found as impurities in QACs designed as fabric softeners in laundry detergents and other household products. QACs resist the biological treatment of wastewaters and are found at higher concentrations in urban sewage sludges. TAMs have been identified

in water and air, and to determine low concentrations of individual species in environmental samples GC and HPLC methods are used.

### Conclusion

A robust and easy to use IC/LC-MS application and workflow were developed to allow the separation and detection of tributylamine and tetrabutylammonium in complex industrial wastewaters. The volatile eluent consisting of acetonitrile, water, and a small amount of formic acid supported the ionization of the amine. Due to the polymer-based non-functionalized column, symmetric peaks were obtained without any further eluent additives. LODs and LOQs for both analytes were in the sub mg/L-range, attesting to the high sensitivity of the single quadrupole MS detection. High throughput was achieved with injection cycle times of 9 min. Sample preparation was limited to a simple dilution of the wastewater samples with DI water. This approach and workflow have the potential to be applied to other TAMs and QACs in environmental samples.

 [View the full application note](#)

## Discovery of emerging disinfection by-products in water using gas chromatography coupled with Orbitrap-based mass spectrometry

Cristian Cojocariu, Paul Silcock, Thermo Fisher Scientific, Runcorn, UK; Cristina Postigo, Institute of Environmental Assessment and Water Research, (IDAEA-CSIC) Water and Soil Quality Research Group, Barcelona, Spain; Susan D. Richardson, University of South Carolina, Columbia, SC USA.; Damia Barcelo, Catalan Institute for Water Research (ICRA), Girona, Spain

### Introduction

The disinfection of drinking water is required in order to protect consumers from potential waterborne infectious and parasitic pathogens. Water is commonly treated by adding chemical disinfectants, such as free chlorine, chloramines, chlorine dioxide, and ozone. However, although very effective in removing disease-causing microorganisms, these disinfectants can react with naturally occurring materials in the water and can form disinfection by-products (DBPs) which can be harmful to human health. In particular, compounds containing an iodo-group, i.e., iodinated DBPs (iodo-DBPs), may pose a greater health risk for the population exposed to them than their brominated and chlorinated analogues. In recent years, several chemical classes of low molecular weight iodo-DBPs have been reported; however, many more may be still present in the unknown fraction (~50%) of halogenated material formed during disinfection treatments. Therefore, complete characterization of iodo-DBPs present in DBP mixtures is crucial to further investigate their occurrence in disinfected waters and potential toxicity effects.

### Conclusion

This work has shown the successful application of the Thermo Scientific™ Q Exactive™ GC system for the characterization of iodo-DBPs in disinfected water extracts.

A large number of peaks were detected in the samples analyzed and an exact mass filter in TraceFinder was used to isolate only the compounds containing iodine. Higher concentrations of iodo-DBP were found in the samples exposed to chloramination compared to chlorination treatments.

The EI data obtained can be used for candidate compound identification against existing commercial libraries. Importantly, as often the chemicals detected are not included in such libraries, the consistent sub-ppm mass accuracy measurements will unambiguously determine the elemental composition and subsequent structural elucidation of unknown chemicals.

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# Emerging contaminants introduction

Population growth, recycling, waste materials, climate change, and industrial contamination and other public health crisis such as SARS-CoV-2 create unintended consequences that impact water quality and cause water stress. Analytical testing to ensure water quality and safety is essential for health and the ecosystem including analysis of regulated pollutants like pesticides, semi-volatile organic compounds, metals, disinfection byproducts, and

emerging contaminants like PFAS, hormones, and many unknown chemicals or unexpected epidemiological or microcystins outbreak concerns.

To address changing regulatory requirements faster, including US EPA, EU, Drinking Water Directive (DWD) standards and others, we can help recommend methods and instructions for detection, identification, and analysis of emerging contaminants in different matrices.

Click on note titles to jump to page

Matrix							US EPA	Title
Drinking Water	Surface Water	Waste Water	Soil	Sediment	Air	Other		
	●	●				●	8327	Direct analysis of selected per- and polyfluorinated alkyl substances (PFAS) in ground, surface, and waste water for US EPA Method 8327
●							604, 525, and 8270	Trace analysis of chlorophenolics using triple quadrupole GC-MS
			●				557	Extraction and analysis of poly- and perfluoroalkyl substances (PFAS) from soil
●							545	Reduced injection volume applied to the quantitation of cylindrospermopsin and anatoxin-a in drinking water according to EPA Method 545
●							544	Quantitation of cyanotoxins in drinking water according to EPA 544 guidelines
●							537.1	Determination of per- and polyfluorinated alkyl substances (PFAS) in drinking water using automated solid-phase extraction and LC-MS/MS
●							537.1	Secondary validation study for US EPA Method 537.1 using automated SPE followed by LC-Q Exactive Orbitrap high-resolution accurate MS
●							533	Determination of per- and polyfluorinated alkyl substances (PFAS) in drinking water using automated solid phase extraction and LC-MS/MS
●						●	200.7	Straightforward identification of microplastic particles in bottled water
●								Low level quantification of NDMA and non-targeted contaminants screening in drinking water using GC Orbitrap mass spectrometry
		●						Methodology and validation results for analyzing pesticides in water by large volume PTV GC-MS/MS
						●		A novel high resolution accurate mass Orbitrap-based GC-MS platform for routine analysis of short chained chlorinated paraffins
						●		Pyrolysis-GC-Orbitrap MS - a powerful analytical tool for identification and quantification of microplastics in a biological matrix
						●		Microplastics in the environment
						●		Low-level consistent analysis of PBDEs in environmental and food matrices using triple quadrupole GC-MS/MS
		●						Wastewater surveillance of COVID-19
	●	●				●		AOF by combustion IC – non-targeted complementary determination of PFAS in aqueous samples



APPLICATION NOTE 65397

## Direct analysis of selected per- and polyfluorinated alkyl substances (PFAS) in ground, surface, and waste water for US EPA Method 8327

Cristina C. Jacob, Claudia P.B. Martins, Alan R. Atkins, Richard F. Jack, Thermo Fisher Scientific, San Jose, CA, USA

### Goal

To demonstrate method performance for the PFAS analysis at low levels (ng/L) in a wide variety of non-drinking water matrices by direct analysis and submit data package for EPA 8327 interlaboratory method validation.

### Introduction

Per- and polyfluoroalkyl substances (PFAS) are a group of man-made chemicals that includes perfluorooctanoic (PFOA), perfluorooctyl sulfonic acid (PFOS), and hexafluoropropylene oxide dimer acid (HFPO-DA, which is part of GenX process). PFAS compounds have been manufactured since the 1940s. The most well-known PFAS compounds, PFOA and PFOS, have been the most extensively produced and studied for chemical properties and toxicological effects. Both chemicals are very persistent in the environment and accumulate in the human body over time. It is well documented that exposure to PFAS can lead to adverse human health effects and are found in food packaging material as well as food processing equipment. Plants can accumulate PFAS when grown in PFAS-containing soil and/or water. These compounds are

also found in a wide variety of consumer products such as cookware, food containers (e.g., pizza boxes), and stain repellants. Additional products that lead to routes of exposure include clothing with stain- and water-repellent fabrics, nonstick products (e.g., non-stick coatings), polishes, waxes, paints, and cleaning products. Another major source of PFAS are fire-fighting foams, which are a primary component of groundwater contamination at airports and military bases. More exposure comes from workplace environments, including production facilities or industries (e.g., chrome plating, electronics and manufacturing, or oil recovery).

### Conclusions

The method shows excellent quantitative performance of the Thermo Scientific™ TSQ Altis™ mass spectrometer for PFAS direct analysis in the low ng/L range in non-drinking water matrices.

 [View the full application note](#)



APPLICATION NOTE 000491

## Trace analysis of chlorophenolics using triple quadrupole GC-MS

Kjell Hope, Pacific Rim Laboratories Inc., Canada; Adam Ladak, Thermo Fisher Scientific, Hemel Hempstead, UK

### Goal

To develop and assess a reliable, robust, and future-proof method for the analysis of chlorophenols at trace and ultra-trace levels in water.

### Introduction

Chlorophenolic compounds are widely used in the industrial manufacturing of pharmaceuticals, agricultural chemicals, and dyes. They are ubiquitous in the environment, having been released via pesticides, industrial waste, or the breakdown of chlorinated compounds. Chlorophenolics are also produced as disinfection by-products in drinking water due to the chlorination of phenols. Chlorophenol derivatives such as catechol, chlorocatechols, guaiacol, chloroguaiacols, and syringol exhibit toxic properties including cytotoxic, mutagenic, and cancerogenic activity. Subsequently, substitution of these compounds with chlorine atoms may increase their toxicity and prolong the period of bioaccumulation in living organisms.

Due to the toxicity of these chemicals, there are a number of regulations implemented throughout the world to limit the public's exposure to them. These regulations include the United States Environmental Protection Agency (US EPA) Methods 604, 525, and 8270. However, the EPA Methods only cover a subset of chlorophenolic compounds and do not assess the risk of the entire class of compounds. Limited information is present regarding the toxicity of the wide range of chlorophenolic compounds. Currently, guidelines are given only for 2,4,6-trichlorophenol (2,4,6-TCP) at 2 µg/L in water.

### Conclusion

The results obtained in this study demonstrate that the Thermo Scientific™ TSQ™ 9610 triple quadrupole GC-MS/MS system in combination with the Thermo Scientific™ TRACE™ 1610 GC and the Thermo Scientific™ TriPlus™ RSH autosampler represent a reliable, robust, and future-proof tool for the analysis of chlorophenols at trace and ultra-trace levels in water:

- Extended linearity was achieved thanks to the XLXR detector, which allowed for average coefficients of determination (R<sup>2</sup>) of >0.99 in the range 2.5–10,000 ng/mL and AvCF %RSDs below 10%.
- The optimized design and the high sensitivity of the NeverVent™ AEI ion source allowed for MDLs below 1 ng/L for all the target compounds, which is significantly below the current guideline values for 2,4,6-TCP of 2 µg/L.
- The inertness of the flow path and the precise control of the TriPlus RSH autosampler ensure excellent precision for all compounds with peak area %RSD <10% in a sample spiked at 1 ng/L.



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CUSTOMER APPLICATION NOTE 73937

## Extraction and analysis of poly- and perfluoroalkyl substances (PFAS) from soil

Matthew S. MacLennan, Daniel Ng, David Hope, Pacific Rim Laboratories, B. C. Canada

### Introduction

Recent studies suggest that toxic and highly persistent poly- and perfluorinated alkyl substances (PFAS) are much more prevalent in tissue and soil than in water. The increasing length of perfluoroalkyl chain in PFAS is correlated strongly to lower water solubility/higher adsorption behavior of a particular PFAS molecule in the environment (i.e., migration of PFAS at soil/water/air interfaces) and in remediation/filtration (i.e., choice of filtration media or sorbents). There are over 6,000 PFAS commercially available, many of which have high environmental persistence and have been found in water and soils globally. This poses a significant challenge to developing analytical methods, especially for the extraction of a variety of PFAS from solid matrices such as soil. Previously, we reported unsatisfactory (0–50%) recovery of long-chain PFAS from soil using vortex/sonication.

In the present study, soil was spiked with 24 PFAS (C4-C14 acids, C4-C10 sulfonates, 4:2, 6:2 and 8:2 fluorotelomers, C8 sulfonamide) at 1 ng/g, which were allowed to absorb overnight into the soil samples.

The soil samples were extracted using the Thermo Scientific™ Dionex™ ASE™ 350 Accelerated Solvent Extractor, which produced 70–130% recovery of all PFAS target compounds. Accelerated solvent extraction has outperformed commonly used, manual “shaking” extraction methods under the same conditions.

After ASE extraction, the solution from the ASE sample collection vials underwent clean-up using solid-phase extraction (SPE) and were analyzed on an LC/MS/MS in a 15-min run. Blanks contained no significant amounts of PFAS. Accelerated solvent extraction is demonstrated to be acceptable for the extraction of short- and long-chain PFAS, with a variety of polarities and head-groups, from soil in the range of 1 ng/g to 400 ng/g.

### Conclusion

Accelerated solvent extraction can extract a variety of PFAS from soil including acids, sulfonates, fluorotelomer sulfonates, and sulfonamide compounds. Accelerated solvent extraction shows high potential for effective extraction of the growing list of PFAS from solid samples.

 [View the full application note](#)



APPLICATION NOTE 64972

## Reduced injection volume applied to the quantitation of cylindrospermopsin and anatoxin-a in drinking water according to US EPA Method 545

Ali Haghani, Andy Eaton, Eurofins Eaton Analytical, Monrovia, CA;  
Neloni Wijeratne, Claudia Martins, Thermo Fisher Scientific, San Jose, CA

### Goal

To demonstrate a sensitive, accurate, and reliable LC-MS/MS workflow to quantify anatoxin-a and cylindrospermopsin in drinking water according to EPA Method 545, performed at a reduced injection volume to increase method robustness while maintaining the desired levels of sensitivity.

### Introduction

Cyanobacteria naturally occur in surface waters. Under certain conditions, such as in warm water containing an abundance of nutrients, they can rapidly form harmful algal blooms (HABs). HABs can produce toxins known as cyanotoxins, which can be harmful to humans and animals. Anatoxin-a (also known as Very Fast Death Factor) is a neurotoxin with acute toxic effects and therefore subjected to monitoring and regulation efforts in several countries, including the United States. Cylindrospermopsin is toxic to liver and kidney tissues. As a result, the United States Environmental Protection Agency (EPA) has developed EPA Method 545 for the Unregulated Contaminant Monitoring Rule 4 (UCMR 4) program, which collects data for contaminants suspected to be present in

drinking water but that do not have health-based standards set under the Safe Drinking Water Act (SDWA). This study demonstrates the performance of the new Thermo Scientific™ TSQ Quantis™ triple quadrupole MS platform performance using EPA Method 545: Determination of cylindrospermopsin and anatoxin-a in drinking water by liquid chromatography electrospray ionization tandem mass spectrometry (LC/ESI-MS/MS).

### Conclusion

The TSQ Quantis triple quadrupole MS proved to be sensitive, accurate, reproducible, and reliable in the quantitation of cylindrospermopsin, and anatoxin-a in drinking water according to EPA Method 545.

Adequate sensitivity was obtained with 5 µL, 10 µL, and 25 µL injection volumes for drinking water matrices. This represents an up to 10-fold reduction in the injection volume than what the EPA is recommending and results in less matrix injected, thereby reducing maintenance of the LC-MS system.

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APPLICATION NOTE 64968

## Quantitation of cyanotoxins in drinking water according to US EPA Method 544 guidelines

Ali Haghani, Andy Eaton, Eurofins Eaton Analytical, Monrovia, CA;  
Neloni Wijeratne, Claudia Martins, Thermo Fisher Scientific, San Jose, CA

### Goal

To demonstrate a sensitive, accurate, and reliable LC-MS/MS methodology in the quantitation of cyanotoxins in drinking water according to EPA 544 guidelines.

### Introduction

Harmful algal blooms are a major environmental problem in the United States and in other nations. Known as red tides, blue-green algae, or cyanobacteria, harmful algal blooms have severe impacts on human health, aquatic ecosystems, and the economy. As a result, the United States Environmental Protection Agency (EPA) has developed EPA Method 544 for the Unregulated Contaminant Monitoring Rule 4 (UCMR 4) program, which collects data for contaminants suspected to be present in drinking water that lack health-based standard regulation under the Safe Drinking Water Act (SDWA). The quantitative performance of the latest generation of triple quadrupole instruments enhances quantitation for these groups of compounds.

This study demonstrates the performance of the new Thermo Scientific™ TSQ Quantis™ triple quadrupole MS platform via EPA Method 544: Determination of Microcystins in Drinking Water by Solid Phase Extraction (SPE) and Liquid Chromatography Electrospray Ionization and Tandem Mass Spectrometry (LC-ESI-MS/MS).

### Conclusion

The TSQ Quantis triple quadrupole MS proved to be sensitive, accurate, reproducible, and reliable in the quantitation of microcystins and nodularin in drinking water according to the EPA method requirements. Adequate sensitivity was obtained using a 5 µL injection volume.

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APPLICATION NOTE 73346

## Determination of per- and polyfluorinated alkyl substances (PFAS) in drinking water using automated solid-phase extraction and LC-MS/MS for US EPA Method 537.1

Changling Qiu, Xin Zhang, Rahmat Ullah, Wei Chen, and Yan Liu, Thermo Fisher Scientific, Sunnyvale, CA, USA

### Goal

To demonstrate an efficient and reliable solid-phase extraction method with the Thermo Scientific™ Dionex™ AutoTrace™ 280 PFAS Solid-Phase Extraction instrument for the determination of per- and poly-fluorinated compounds in drinking water per US EPA Method 537.1

### Introduction

Per- and polyfluorinated alkyl substances (PFAS) are a group of man-made chemicals including perfluorooctanoic acid (PFOA), perfluorooctanesulfonic acid (PFOS), and GenX chemicals that have been manufactured and used in a variety of industries globally. These compounds have a wide range of commercial product applications including industrial polymers, stain repellents, surfactants, waterproofing products, packaging, and aqueous film forming foams used for firefighting. PFAS are highly soluble in water, chemically stable, persistent in the environment, and can accumulate in the human body over time, leading to adverse human health effects. PFOA and PFOS are no longer manufactured in the US due to their persistence and potential human health risks.

In November 2018, the United States Environmental Protection Agency (US EPA) published Method 537.1 “Determination of selected per- and polyfluorinated alkyl substances in drinking water by solid phase extraction and LC/MS/MS”. The method uses an offline solid-phase extraction (SPE) with liquid chromatography tandem mass spectrometry (LC-MS/MS) to extract, enrich, and determine 18 PFAS in drinking water. Currently most testing laboratories perform the sample extraction manually using a vacuum manifold, which is labor-intensive, time-consuming, and the flow rate through the cartridge is difficult to control. There is a high demand for automation of the SPE procedure.

### Conclusion

This method can be used for the extraction and determination of 18 PFAS in drinking water with a PFAS-safe AutoTrace 280 extraction system and LC-MS/MS while preventing PFAS from leaching into samples during extraction, while delivering consistent and reliable performance to fulfill the requirements of US EPA Method 537.1.

 [View the full application note](#)



APPLICATION SUMMARY 65499

## Secondary validation study for EPA Method 537.1 using automated SPE followed by LC-Q Exactive Orbitrap high-resolution accurate MS

Ali Haghani, Andy Eaton, Eurofins Eaton, Monrovia, CA;

Richard Jack, Maciej Bromirski, Thermo Fisher Scientific, San Jose, CA

### Goal

To demonstrate method performance for per- and polyfluorinated alkyl substances (PFAS) analysis using Thermo Scientific™ Orbitrap™ high-resolution accurate mass spectrometry as an alternative to conventional triple quadrupole instruments for determination of PFAS in drinking water matrices using US EPA Method 537.1.

### Introduction

Within the last decade, liquid chromatography-tandem mass spectrometry (LC-MS/MS) sensitivity has increased by at least a factor of ten and is therefore sensitive enough for quantitation of targeted compounds for validated methods. The ease of use for detecting polar compounds makes LC-MS/MS the technique of choice for analysis of compounds of emerging concern (CECs) in environmental samples. However, with the development of high-resolution accurate mass (HRAM) spectrometers, sensitivity rivals that of triple quadrupole MS instruments and, in addition, mass resolution provides the added benefits of accurate quantitation along with unknown screening capabilities. HRAM using Orbitrap technology combines the sensitivity of a triple quadrupole analyzer for quantitation with the confidence of full scan data for quantitative identification and confirmation similar to MS/MS instruments that participated in a method validation study.

### Conclusion

The method referenced in this application note is rugged and reproducible and shows excellent quantitative performance of the Thermo Scientific™ Q Exactive™ mass spectrometer in Parallel Reaction Monitoring (PRM) mode for US EPA Method 537.1 with enhanced selectivity and specificity.

- Excellent linearity and quantitative accuracy were achieved over the range of 0.1 to 40 ng/L, with correlation coefficients greater than 0.995 for all transitions.
- Precision and accuracy were met by analyzing seven laboratory fortified blanks (LFBs) extracted over three days spiked at 25 ng/L, with <20% RSD and  $\pm 30$  difference achieved.
- Monrovia, CA, tap water was spiked at 25 ng/L, extracted over two batches in duplicates, and analyzed. The %RSD of less than 30% and recoveries of  $\pm 30\%$  of spike amount were met.

 [View the full application note](#)



APPLICATION NOTE 73883

# Determination of per- and polyfluorinated alkyl substances (PFAS) in drinking water using automated solid phase extraction and LC-MS/MS for US EPA Method 533

Xin Zhang, Changling Qiu, Rahmat Ullah, and Yan Liu, Thermo Fisher Scientific, Sunnyvale, CA, USA

## Goal

Demonstrate an efficient and reliable solid phase extraction method with a Thermo Scientific™ Dionex™ AutoTrace™ PFAS, an automated solid-phase extraction (SPE) system, for the determination of per- and poly-fluorinated compounds in drinking water per US EPA Method 533.

## Introduction

Drinking water perfluorinated and polyfluorinated alkyl substances (collectively referred to as PFAS) occurrence studies have typically targeted perfluorooctanoic acid (PFOA) and perfluorooctanesulfonate (PFOS), and as a result, these two are the most commonly detected compounds. This discussion focuses primarily on PFOS and PFOA. However, other compounds including PFBA, PFHxA, PFHpA, PFNA, PFUnDA, and PFHxS have also been detected in drinking water. In December 2019, the United States Environmental Protection Agency (US EPA) took a key step in implementing the PFAS Action Plan by announcing a new validated method for testing PFAS in drinking water. This new validated test method complements other actions the agency is taking under the Action Plan to help communities address PFAS nationwide. It focuses on “short chain” PFAS, those PFAS with carbon chain lengths of four to twelve, and covers PFOS, PFOA, and other common PFAS that have been detected

and reported. Currently most testing laboratories are performing the sample extraction manually using a vacuum manifold, which is labor-intensive and time-consuming.

In addition, the flow rate through the cartridge is difficult to control, which may yield low recoveries or cause false negatives. There is a high demand for automation of the SPE procedure. Here we demonstrate how to automate an SPE system reliably for PFAS in large-volume aqueous samples and saves time, solvent, and labor, while ensuring high reproducibility and productivity for analytical testing laboratories.

## Conclusions

We demonstrated a PFAS-inert automated SPE extraction LC-MS/MS system that can be used for the simultaneous extraction and determination of twenty-five PFAS in drinking water while preventing PFAS from leaching into samples during extraction; while delivering cost savings and uncompromising performance to fulfill US EPA Method 533 requirements.

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APPLICATION NOTE 53167

## Straightforward identification of microplastic particles in bottled water

Thermo Fisher Scientific

### Introduction

Plastics can be formed into an endless variety of shapes with different physical characteristics and that makes them attractive materials for a wide assortment of products. Large and diverse usage results in a significant amount of plastic materials eventually ending up as refuse. The size of the plastic waste can range from large, easily visible, easily handled, and easily detectable macroplastics down to microplastic particles that may be too small to detect with normal vision. The European Chemical Agency (ECHA) defines microplastic particles as particles with dimensions from 5 mm to 1 nm. The source of these microplastics is from primary sources where the plastics have been produced as small particles (such as in some cosmetic products and cleaners) or as secondary products that arise from the mechanical, photochemical, or thermal break down of larger plastic products. Microplastics studies have centered on contamination in marine environments and related areas (sea salt, seafood, and aquatic animals) they also appear in tap water and even in commercial products such as bottled water and other beverages. The detection and identification of microplastic particles are important for determining the possible sources of these contaminants and for evaluating the environmental impact and health risks associated with the different types of materials.

One of the biggest challenges with the analysis of microplastics involves distinguishing microplastic particles from other types of materials and making sure that misidentification does not lead to over-estimating the amount of microplastics in a sample. FTIR and Raman spectroscopy (two different forms of vibrational spectroscopy) are widely used for the unambiguous identification

of different types of polymeric materials. The molecular information reflected in the vibrational spectra of polymer materials provides a molecular fingerprint that can be used with spectral databases to positively identify different types of polymer materials. The spectral information supplied by infrared and Raman spectroscopy is similar but not exactly the same. Infrared spectroscopy depends on changes in dipole moments and is particularly useful for detecting polar functional groups found in many different types of polymer materials (hydroxyl, amines, amides, carbonyls, etc.).

### Conclusion

For spectroscopic analysis it is important to select an isolation technique that allows for subsequent spectroscopic analysis.

- Vibrational spectroscopy provides molecular information and can identify a wide variety of materials including polymeric materials.
- Micro-spectroscopy allows for analysis of microscopic particles; ideal for analysis of microplastic particles
- Infrared and Raman spectroscopy both provide molecular structure information and excel at identifying different aspects of polymer materials. Where one is weak the other is strong but together provide exceptional coverage for identifying a wide variety of different materials.

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APPLICATION NOTE 10530

## Low level quantification of NDMA and non-targeted contaminants screening in drinking water using GC Orbitrap mass spectrometry

Cristian Cojocariu, and Paul Silcock, Thermo Fisher Scientific, Runcorn, UK;  
Maria José Farré, Sara Insa, Damià Barceló, Catalan Institute for Water Research (ICRA),  
Parc Científic i Tecnològic de la Universitat de Girona, 17003 Girona, Spain;

### Introduction

N-nitrosodimethylamine (NDMA) is a semivolatile organic compound that belongs to nitrosamines, an emerging class of drinking water contaminants. NDMA is the main nitrosamine of concern and is classified as a potent carcinogen by the US Environmental Protection Agency as it is known to induce tumors following administration by either ingestion or inhalation. NDMA is formed as a by-product during industrial processes such as chloramination of wastewater and drinking water. NDMA is considered a priority pollutant and various countries around the world have already introduced maximum acceptable concentrations of 9 ng/L and action levels 10 ng/L. It is particularly important that NDMA is detected and accurately quantified in drinking water as even low level of this chemical (e.g., 10 ng/L) can pose human cancer risks, being especially toxic to the liver.

Traditionally, the analytical methodology used for NDMA detection and quantification employs single or triple quadrupole gas chromatography mass spectrometry (GC-MS), or magnetic sectors and high resolution time-of-flight mass spectrometers. However, with these analytical instruments it is difficult to

obtain high selectivity and high sensitivity at the same time. Reduced selectivity can lead to interferences with the matrix and background chemical ions and can result in false positive detection and erroneous quantification of NDMA. This is due to poor selectivity through insufficient resolving power of such instrumentation.

### Conclusion

This study demonstrates excellent quantitative and qualitative performance of the Thermo Scientific™ Exactive™ GC system for the analysis of trace levels of NDMA. NDMA was easily detected and accurately quantified at 1.0 ng/L in the drinking water samples with excellent recovery values. Consistently low (sub ppm) mass deviation from the theoretical NDMA mass was observed at all concentration levels and in all analyzed samples.

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APPLICATION NOTE 10580

## Methodology and validation results for analyzing pesticides in water by large volume PTV GC-MS/MS

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Inge De Dobbeleer, Thermo Fisher Scientific, Breda, The Netherlands

### Goal

Develop and validate a method for analyzing pesticides in water by large volume programmable temperature vaporization (PTV GC-MS/MS).

### Introduction

The European Union (EU) Water Framework Directive was introduced in August 2013, amending EU directive 2000/60/EC and 2008/105/EC to be applied to all EU member states. For the identification of priority substances and the monitoring of different classes of contaminants, such as several pesticide compounds.

EU member states have the flexibility to apply an Environmental Quality Standard (EQS) for an alternative matrix or, where relevant, an alternative biota taxon, for example sub-phylum Crustacean, paraphylum “fish”, class Cephalopoda or class Bivalvia (mussels and clams). The directive encourages the development of novel monitoring methods such as passive sampling and other tools.

In the United Kingdom (UK) specifically, Chemical Investigation Programme 2 (CIP2) UK regulations investigate the occurrence, sources, and removal of trace substances in wastewater treatment facility effluent. This regulation helps to establish priorities for premeditative action to ensure surface waters meet new EQS.

The directive poses challenges to the water laboratories, mainly in terms of monitoring the various compounds at a very low required level. In many laboratories, the approach to reach these levels would require a capital equipment investment as well as a different approach to sampling.

### Overall conclusions

- The method reaches all validation requirements and limits of quantitation for all pesticides, except for heptachlor and its epoxides.
- Excellent linearity was observed over the concentration range tested (40–5000 µg/L) with the coefficient of determination > 0.995.
- Recovery levels were between 80–114% for all compounds, meeting the EU Water Framework Directive regulations.
- Residual standard deviations were met for all compounds, except for heptachlor and its epoxides.
- Quantitation limits for the Water Framework Directive are met, except for heptachlor and its epoxides.

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TECHNICAL NOTE SUMMARY 10539

## A novel high resolution accurate mass Orbitrap-based GC-MS platform for routine analysis of short chained chlorinated paraffins

Cristian Cojocariu Thermo Fisher Scientific, Runcorn, UK

### Goal

To demonstrate the performance of a novel benchtop, high resolution accurate mass Orbitrap™-based GC-MS for the analysis of short chained chlorinated paraffins (SCCPs).

### Introduction

Short Chained Chlorinated Paraffins (SCCPs) are emerging contaminants that, once released, will remain in the environment for long periods of time with the potential to bioaccumulate in living organisms. SCCPs are intentionally manufactured and used as lubricants and coolants in the metal processing industry or as plasticizers and flame retardants in plastic products. Chronic exposure to SCCPs is believed to have harmful and irreversible effects for humans and the environment. As a consequence, SCCPs are listed in the Stockholm convention as chemicals with potential adverse effects and their production and use in Europe is restricted and regulated.

Detection and quantification of SCCPs poses analytical challenges due to the fact that these compounds are present in the environment at low levels, as very complex isomeric mixtures and are difficult to separate chromatographically. Although there is no consensus for the use of a validated analytical procedure for the routine monitoring of SCCPs in environmental samples, there are several analytical methods that are used to detect and quantify SCCPs.

### Conclusion

These preliminary results demonstrate that the Thermo Scientific™ Exactive™ GC is a potential solution to address the difficult challenges related to the detection and quantification of SCCPs due to the excellent sensitivity, linearity, and selectivity in combination with an uncomplicated instrumental setup.

- Low instrumental LODs of ~10 pg/μL, calculated as total homologues response for each of the two SCCPs technical mixtures, can be easily obtained.
- LOQs as low as 3 pg/μL can be reliably obtained for individual homologue groups.
- Excellent linearity was obtained across a total SCCP mixture concentration range of 1 to 10,000 pg/μL, making the Exactive GC an ideal quantification tool.
- The high resolving power of the Exactive GC facilitates sub-ppm mass accuracy at low and high concentrations essential for achieving enough selectivity to confidently separate SCCP specific low mass ions from the interfering background ions (in EI), or higher masses (in NCI), for various SCCP homologue groups.

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APPLICATION SUMMARY 10643

## Pyrolysis-GC-Orbitrap MS - a powerful analytical tool for identification and quantification of microplastics in a biological matrix

Jörn Logemann, Cristian Cojocariu, Thermo Fisher Scientific; Eystein Oveland, Ørjan Bjørøy and Tanja Köge, Institute of Marine Research, Bergen, Norway; Wibke Peters, S+H Analytik, Mönchengladbach, Germany

### Goal

To assess the applicability of pyrolysis-gas chromatography-Orbitrap™ mass spectrometry for the qualitative and quantitative analysis of plastic polymers in complex biological matrices.

### Introduction

Plastics are synthetic organic polymers, commercially introduced on a large scale starting in the 1950s. Single-use plastics (grocery bags, food packaging, bottles, utensils) are persistent pollutants making up approximately 40% of beach litter. This litter eventually ends up in the marine environment, with an estimated 8 million metric tons of plastic waste entering the oceans worldwide every year. Most plastics have a very long degradation time, and for a timespan up to centuries they end up as macro-, micro- and nanoplastics through weathering. Due to their characteristics and additional content (monomeric residue, plasticizers, flame retardants etc.), micro- and nanoplastics can have complex toxicological effects on marine life through direct ingestion and/or leachates. This might represent a hazard for ecosystems and for human exposure through consumption and inhalation.

### Conclusion

The Thermo Scientific™ Exactive™ GC Orbitrap GC-MS system in combination with pyrolysis has proven to be a very promising analytical technique that opens new possibilities with respect to the analysis of microplastic polymers in biological matrices.

- The Exactive GC Orbitrap GC-MS system demonstrates excellent linear response over a concentration range of 0.05 µg to 50 µg absolute weight for each plastic material with accurate quantitative estimation of microplastic polymers in real samples.
- The high resolving power of the Exactive GC Orbitrap GC-MS system facilitates sub-ppm mass accuracy at low and high concentrations, essential for achieving enough selectivity to confidently separate and identify pyrolysis products and reduces detection limits (ex: polystyrene and polypropylene).
- Full-scan acquisition enables the detection and identification of additional compounds produced during the pyrolysis process of microplastics. Putative identifications require confirmation using analytical standards.

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APPLICATION NOTE 52921

## Microplastics in the environment

Thermo Fisher Scientific

### Introduction

The outer layers of skin contain a significant number of dead cells. Removal of these cells, called exfoliation, produces a cosmetically cleaner appearance, making exfoliation a common practice in facials and other treatments. This can be done aggressively, using sandpaper-like boards, or in a more subtle manner using exfoliating creams or soaps. With a surge of interest in this practice, many cosmetic producers began using microbeads and other materials to enhance exfoliation. Some, such as pumice and salts, are natural products, but they also include synthetic materials such as polymeric microbeads.

The microbeads are typically around 25-100 microns in size – making them visible to the unaided eye. Concerns have been raised about the large number of microbeads released and the slow or negligible breakdown of these materials, and their subsequent entry into the food chain through microorganisms and filter feeders.

FTIR microscopy is an excellent tool for detecting and identifying these materials. The Thermo Scientific™ Nicolet™ iN™5 FTIR microscope was designed for exactly such an application, with extreme simplicity in design and function

and powerful analysis software. This is enhanced by the microATR capabilities which enable the analysis with no sample preparation. Here, we use the Nicolet iN5 microscope and Germanium (Ge) ATR to analyze reference materials and samples extracted from a consumer product.

### Conclusions

The Nicolet iN5 FTIR microscope is ideally suited for the analysis of microbeads on filters. The simplicity of operation targets this at laboratories with minimal microscopy experience. The most common microbeads are around 25-100 microns in size, for which the microscope and its Ge-tip ATR are perfectly suited. Coupled with the Thermo Scientific™ OMNIC™ and OMNIC Specta software, a typical analysis can require no more than a minute, with visual positioning using manual operations, simple contact-and-analyze ATR and the most trusted software in spectroscopy.

 [View the full application note](#)



APPLICATION NOTE 000493

## Low-level consistent analysis of PBDEs in environmental and food matrices using triple quadrupole GC-MS/MS

Kjell Hope, Pacific Rim Laboratories Inc., Canada; Giulia Riccardino, Thermo Fisher Scientific, Milan, IT; Adam Ladak, Paul Silcock, Thermo Fisher Scientific, Hemel Hempstead, UK

### Goal

The aim of this application note is to demonstrate the performance of the Thermo Scientific™ TSQ™ 9610 triple quadrupole mass spectrometer coupled to the Thermo Scientific™ TRACE™ 1610 GC for the determination of polybrominated diphenyl ethers (PBDEs) in environmental and food samples.

### Introduction

PBDEs are classes of polybrominated hydrocarbons existing as mixtures of congeners with similar molecular structures but different chemical and physical properties (e.g., congeners with lower numbers of bromine atoms tend to be more volatile and to bioaccumulate more than higher brominated congeners). Historically these compounds were widely used as flame retardants in a variety of products, such as plastics, furniture, upholstery, electrical equipment, electronic devices, textiles, and other household products, because of their capability to release bromine radicals that reduce both the rate of combustion and dispersion of fire when exposed at high temperatures. These compounds enter the environment through emissions from manufacturing processes, volatilization from various products that contain PBDEs, recycling wastes, and leachate from waste disposal sites. They are considered ubiquitous

persistent pollutants as they have been detected in the airborne particulate matter, bonded to sediments, surface water, fish, and other marine animals, and therefore represent a risk to human health. As a consequence, the use of certain toxic PBDEs with links to cancer and endocrine disruption (including penta-, tetra-, and deca-PBDE) have been prohibited, and are currently listed in the Stockholm Convention inventory of persistent organic pollutants.

### Conclusion

The results obtained in these experiments demonstrate that the TSQ 9610 mass spectrometer equipped with the NeverVent AEI ion source in combination with the TRACE 1610 GC and the Thermo Scientific™ AI/AS 1610 liquid autosampler delivers consistent and reliable analytical performance for analysis of PBDEs in environmental and food samples.

 [View the full application note](#)



SMART NOTE 000035

## Wastewater surveillance of COVID-19

Thermo Fisher Scientific

### Why is wastewater surveillance of COVID-19 necessary?

Wastewater (i.e., sewage) testing for SARS-CoV-2 (the virus that causes COVID-19) can be helpful to public health agencies and government officials as a novel complement to existing COVID-19 surveillance systems. Monitoring wastewater for SARS-CoV-2 is used for early detection of COVID-19 spread within a community. By testing the amount of the virus in sewage water and continuous trend monitoring can serve as an early warning of COVID-19 in communities. The detection of SARS-CoV-2 viral RNA in wastewater has rapidly become a significant tool to address the ongoing pandemic. Wastewater detection of SARS-CoV-2 is particularly useful where significant numbers of asymptomatic infections occur. The fluctuation of virus levels in sewage water can help communities see how well preventive measures are working. Wastewater-based epidemiology (WBE) programs can be an invaluable additional predictive tool for addressing the COVID-19 pandemic.

The data generated by National Wastewater Surveillance System (NWSS) will help public health officials to better understand the extent of COVID-19 infections in communities. Multiple testing methods and laboratory workflows are used to quantify SARS-CoV-2 in wastewater. A balanced nutrient ratio is essential if the microorganisms are to function at maximum efficiency. The most

important of these nutrients are nitrogen and phosphorus. It is helpful to know how many people are contributing to the wastewater so that detected amounts of RNA can be compared or 'normalized' to faecal load. Several options exist that may allow estimation of relative human faecal load including biomarkers such as ammoniacal nitrogen, urea and creatinine. Analysis of chemical and physiochemical reference parameters such as pH and conductivity, ammonia, nitrate, nitrite, total organic nitrogen, ortho-phosphate, and other ions such as chloride and sulfate are important. Total nitrogen, phosphorus, biological oxygen demand, ammonium, and urea have also been proposed as population biomarkers. The survival period of coronavirus in water environments strongly depends on temperature, property of water, concentration of suspended solids and organic matter, pH, salinity, ammonia, organic matter, microbial activity, biofilms, and dose of disinfectant used.

 [View the full smart note](#)



APPLICATION NOTE 73481

## AOF by combustion IC – non-targeted complementary determination of PFAS in aqueous samples

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### Goal

To develop an automated method to determine a range of perfluorinated alkyl substances (PFAS) by combustion IC within environmentally relevant matrices and compare the results with existing highly specific methods based on liquid chromatography mass spectrometry (LC-MS/MS).

### Introduction

PFAS are human-made chemicals, which have been manufactured since the 1940s. The most well-known PFAS compounds, perfluorooctanoic acid (PFOA) and perfluorooctanesulfonic acid (PFOS), have been extensively studied for their chemical properties and toxicological effects. PFAS are used in a wide variety of consumer products, such as nonstick cookware, food containers, stain- and water-repellent fabrics, polishes, waxes, paints, and cleaning products. Other sources of PFAS are firefighting foams, which are primary groundwater contaminants at airports and military bases. Further environmental exposure to PFAS comes from industrial production facilities (e.g., chrome plating, electronics, manufacturing, or oil recovery). Living organisms, including plants, animals, and humans, can accumulate PFAS compounds in their tissue, which can build up over time and impact their health.

### Conclusion

The described AOF approach extends the analytical toolset to determine PFAS in environmental samples, such as surface water, groundwater, and wastewater. While LC-MS/MS and gas chromatography tandem mass spectrometry (GC-MS/MS)-methods use a specified set of PFAS for the evaluation, the adsorption process for the AOF method ensures the extraction and determination of more components—PFAS and other fluorinated compounds—than the previously mentioned approaches.

 [View the full application note](#)

# Nutrient analysis introduction

Determining nutrient levels in drinking water, wastewater, and soil samples is critical for protecting aquatic habitats and maintaining clean and safe drinking water supplies. Utility companies and environmental laboratories must regularly measure elemental phosphorus and nitrogen in sewage water, along with a range of other pollution indicators, to ensure discharge streams are compliant with regulatory standards. Wastewater nutrient analysis can also be used for the assessment of population-level infection, including SARS-CoV-2 surveillance, by providing important biomarker indications for population size.

While traditional wet chemistry techniques, including titrations, flow injection analysis, and other colorimetric techniques, have been used for many years to undertake nutrient analysis of drinking water, wastewater, and soil samples. Traditional approaches are slow, labor-intensive, error prone and often unreliable, involving hazardous reagents that add substantial costs for waste disposal. Learn how your lab can implement automated, high-throughput nutrient analysis for cost-effective results; eliminating the limitations of traditional wet chemistry methods.

Click on note titles to jump to page

Matrix							US EPA	Title
Drinking Water	Surface Water	Waste Water	Soil	Sediment	Air	Other		
●	●					●		<a href="#">Analysis of nitrogen, other nutrient and toxic elements in fertilizers using the Thermo Scientific iCAP 7400 ICP-OES</a>
			●			●		<a href="#">High Accuracy of Nitrogen, Carbon and Sulfur Analysis for Agronomy Applications using the Thermo Scientific FlashSmart Elemental Analyzer</a>
●		●						<a href="#">Automated nutrient analysis and water quality monitoring</a>
●		●	●				Various	<a href="#">Fast, easy and simultaneous nutrient analysis of drinking water, wastewater, and soil samples</a>



APPLICATION NOTE 40833

## Analysis of nitrogen, other nutrient and toxic elements in fertilizers using the Thermo Scientific iCAP 7400 ICP-OES

Matthew Cassap, Thermo Fisher Scientific, Bremen, Germany

### Goal

To demonstrate accurate analysis of nitrogen and other elements in fertilizers, using the Thermo Scientific™ iCAP™ 7400 ICP-OES Duo.

### Introduction

Fertilizers are used to provide major plant nutrients (N, P, K), secondary plant nutrients (Ca, S, Mg) and micronutrients such as B, Mn, Fe, Cu, Zn, Mo and Se. Accurate determination of the composition of fertilizers is essential so that the correct dose can be applied to the soil. An insufficient application of a fertilizer may result in poor crop yield. In contrast, an excessive application may result in environmental damage such as eutrophication – caused by dissolved phosphates and nitrates entering water courses or land contamination from non-nutrient elements within the fertilizers. ICP-OES facilitates the cost effective analysis of fertilizers due to the powerful multi-element capability of this technique. The Thermo Scientific™ iCAP™ 7000 Plus Series ICP-OES enables the simultaneous analysis of all the plant nutrition elements (including nitrogen) as well as potential harmful elements (As, Cd, Cr) in fertilizers.

### Conclusion

In conclusion, all of the results obtained using the iCAP 7400 Duo ICP-OES are in alignment with the mean results (within 2 standard deviations of the mean) of certified values. Any exceptions to these results have occurred if the certified value was calculated from results submitted from less than 5 laboratories (leading to inaccurate statistics) or if a different method was used to determine the results. In these cases, the results obtained on an iCAP 7000 Plus Series ICP-OES are approaching 2 standard deviations of the mean.

 [View the full application note](#)



APPLICATION NOTE 42264

## High Accuracy of Nitrogen, Carbon and Sulfur Analysis for Agronomy Applications using the Thermo Scientific FlashSmart Elemental Analyzer

Dr. Liliana Krotz, Dr. Francesco Leone and Dr. Guido Giazzi, Thermo Fisher Scientific, Milan, Italy

### Goal

To demonstrate the performance of the Thermo Scientific™ FlashSmart™ Elemental Analyzer in agronomy applications.

### Introduction

Nitrogen and carbon determination in soils is important for the evaluation of organic matter and for the determination of fertilizers needed. They also provide significant information on the nutritional elements of plants which are key factors for their future growth. The determination of nitrogen content provides insight on the quality of different types of crop, used for feeding and processing, as well as for N-cycle and N-fixation monitoring in agricultural and environmental research. The differentiation of Total Carbon (TC) and Total Organic Carbon (TOC) is also useful to evaluate the quality of soils while sulfur is an essential component of living matter. The deficiency of sulfur has a negative influence in the growth of vegetables and in the quality of proteins. Also, the carbon / nitrogen ratio of the organic material added to the soil influences the rate of decomposition of organic matter and this results in the release or immobilization of soil nitrogen. If the added organic material contains more nitrogen in proportion to the carbon, then nitrogen is released into the soil from the decomposing organic material, while if the organic material has less nitrogen

in relation to carbon, then the microorganisms will utilize the soil nitrogen for further decomposition and the soil nitrogen will be immobilized and will not be bioavailable. Therefore, cellular carbon and nitrogen metabolism must be tightly coordinated to sustain optimal growth and development for plants.

### Conclusion

The all-in-one Thermo Scientific FlashSmart Analyzer is the optimal solution in agronomy for the quantitative determination of nitrogen, carbon and sulfur, and the C/N ratio, in sample matrices with a wide range of concentrations spanning low to high amounts. Specifically, the Analyzer demonstrates excellent repeatability, reproducibility, accuracy and precision.

The nitrogen, carbon and sulfur data obtained by the FlashSmart Analyzer are inside the tolerance declared for the Reference Materials. No memory effect was observed when changing the sample type, indicating the complete conversion and detection of the nitrogen, carbon and sulfur present in the sample.

 [View the full application note](#)



EBOOK D17523

## Automated nutrient analysis and water quality monitoring

Thermo Fisher Scientific

Wet chemistry, also called bench chemistry or wet chemical analysis, is a form of analytical chemistry that may use multiple methods, such as colorimetry, gravimetry and titration to analyze elements and compounds in liquid samples. Analytical chemistry has developed exponentially in last three decades, still the wet chemical analysis is an integral part of the modern analytical chemistry lab because the wet chemical methods are proven and less expensive. Often, modern instrumentation cannot determine results which many specific wet chemical tests provide.

Discrete photometric systems are precise and easily adaptable for the analysis of water and environmental samples; potentially eliminating the need for wet chemistry instruments includes ing pH/conductivity/ion meter, automated titrator, colorimeter, turbidity meter, spectrophotometer and Flow Injection Analyzer (FIA) or Segmented Flow Analyzers (SFA) or Continuous Flow Analyzer (CFA).

Our analyzers with system reagents provide an automated solution for water analysis saving technician time and reducing errors. Automated discrete analysis enables:

- Faster results—ready for immediate analysis: no reagent priming, no method changeover time
- Testing flexibility—various tests can be performed on each sample; there is no limitation to the number of tests
- Precision and quality—specific measurements with high reproducibility

 [View automated water analysis guide](#)



BROCHURE 000257

## Fast, easy and simultaneous nutrient analysis of drinking water, wastewater, and soil samples

Thermo Fisher Scientific

Laboratories require accurate, efficient, and easy-to-use techniques for nutrient analysis. The Thermo Scientific™ Gallery™ and Gallery™ Plus discrete analyzers are an integrated, automated platform for nutrient analysis that provide a faster, safer, and more reliable replacement to traditional wet chemistry methods for the measurement of:

- Orthophosphate
- Total phosphorous (TP)
- Ammonia
- Nitrates
- Nitrites
- Total oxidized nitrogen (TON)
- Total Kjeldahl nitrogen (TKN)

The Gallery and Gallery Plus discrete analyzers allow nutrient determinations to be run in parallel with both photometric and electrochemical measurement techniques, including pH, conductivity, hexavalent chromium, chloride, and sulfate analyses. By enabling high-throughput measurement of over 20 parameters simultaneously, the analyzers streamline workflows to reduce total run times and increase analytical efficiency. The unique low-volume cuvette design and precision liquid-handling capabilities also accommodate microliter sample and reagent volumes, helping laboratories minimize reagent use and waste to reduce cost-per-analysis.

As fully-automated platforms, the Gallery and Gallery Plus discrete analyzers ensure every test is performed according to precise, predefined workflows. These workflows require no user intervention once the pre-mixed, ready-to-use reagents are loaded into the instrument. Automation not only improves the reliability and sensitivity of results compared with manual wet chemistry techniques, it also boosts laboratory productivity by freeing staff to walk away and work on other tasks. By eliminating the need to handle hazardous reagents, the Gallery systems' automated workflows also better protect the health and safety of laboratory teams. Each test is validated to fulfill the United States Environmental Protection Agency (US EPA) and international regulatory requirements for environmental and drinking water analysis, supporting consistently compliant results.

 [View the full brochure](#)

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