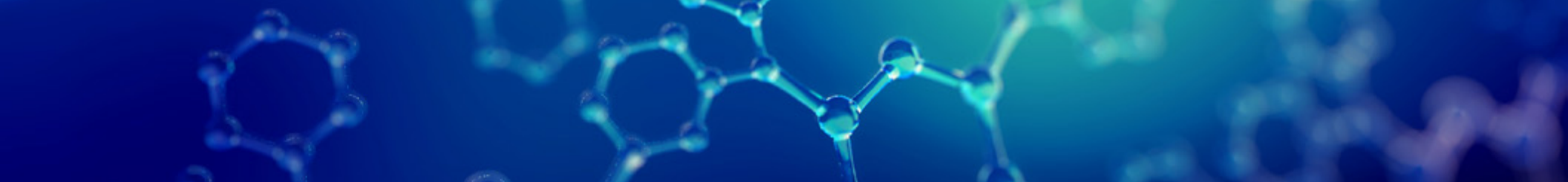


IC-MS Applications Compendium



On the cover: Katie Banaszewski, Director of Quality, NOW Foods, Bloomingdale, Illinois, United States

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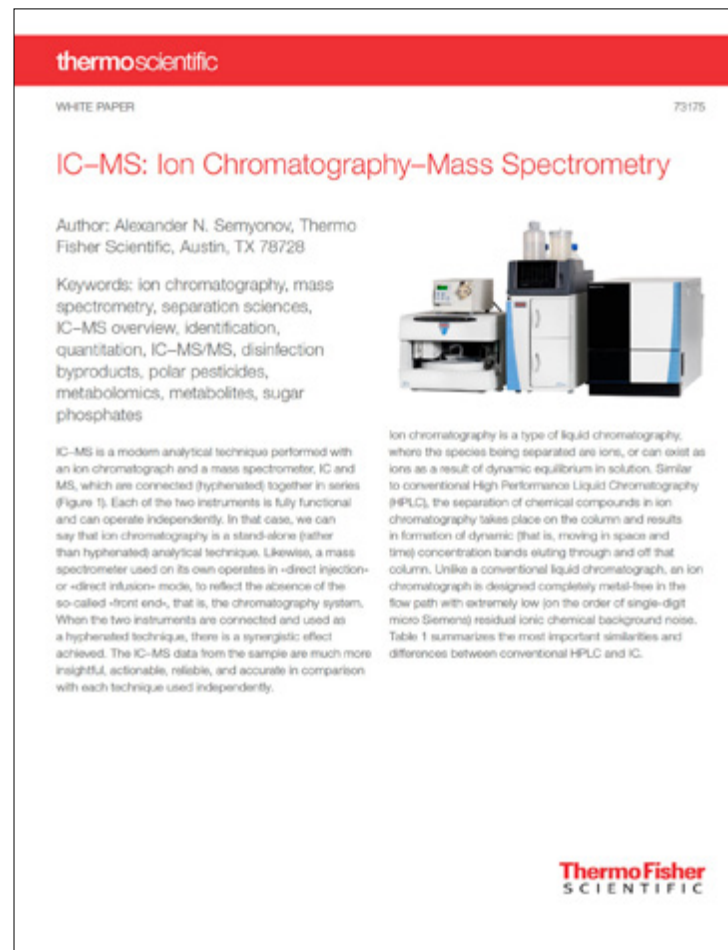
O-glycan profiling using HPAE-PAD hyphenated with a high-resolution accurate mass (HRAM) mass spectrometer

Primary metabolite analysis of plant material using a triple quadrupole MS coupled to a monolith anion-exchange column

IC-MS: Ion Chromatography–Mass Spectrometry

Overview

IC-MS is a modern analytical technique performed with an ion chromatograph and a mass spectrometer, IC and MS, which are connected (hyphenated) in series. Each of the instruments is fully functional and can operate independently. In that case, we can say that ion chromatography is a stand-alone (rather than hyphenated) analytical technique. Likewise, a mass spectrometer used on its own operates in «direct injection» or «direct infusion» mode, to reflect the absence of the so-called «front end», that is, the chromatography system. When the two instruments are connected and used as a hyphenated technique, there is a synergistic effect achieved. The IC-MS data from the sample are much more insightful, actionable, reliable, and accurate in comparison with each technique used independently.

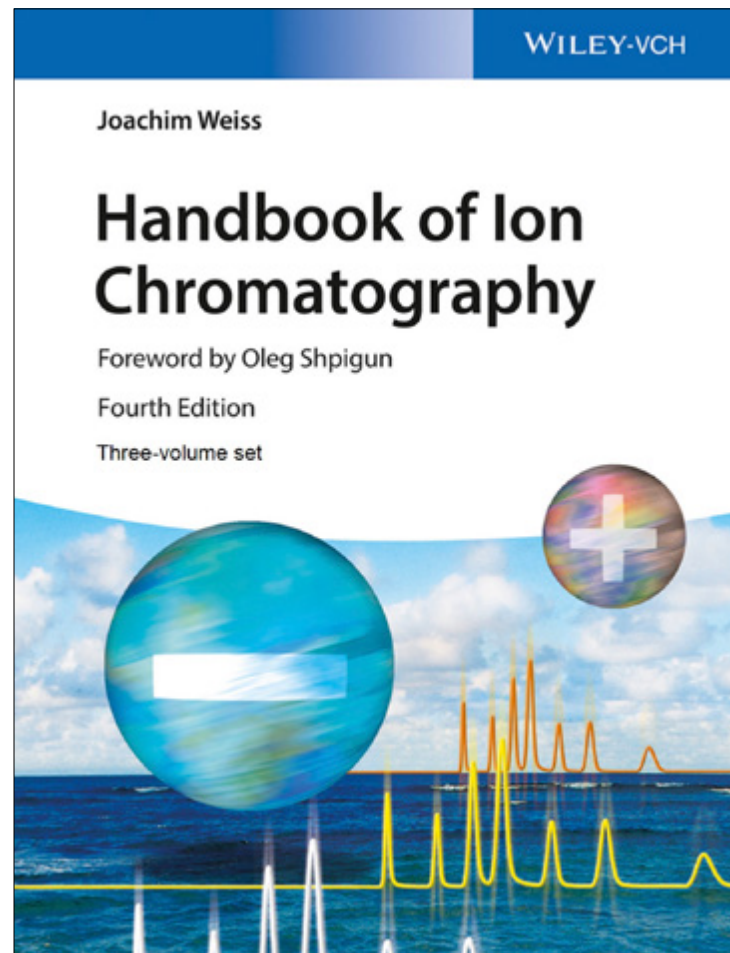


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Excerpt from 2016 Joachim Weiss Handbook of Ion Chromatography

Overview

The second important hyphenation is the coupling of an ion chromatograph to a mass spectrometer; this combination provides the analyst with information on analyte structure and molecular weight. While mass-selective detection in gas chromatography became routine years ago, the coupling of a liquid chromatograph to a mass spectrometer used to be problematic, because the relatively large amounts of liquid mobile phase are not compatible with the high vacuum in the ion source of a mass spectrometer. To solve these incompatibility problems, various types of LC–MS interfaces have been developed. The discussion below focuses on the electrospray interface.

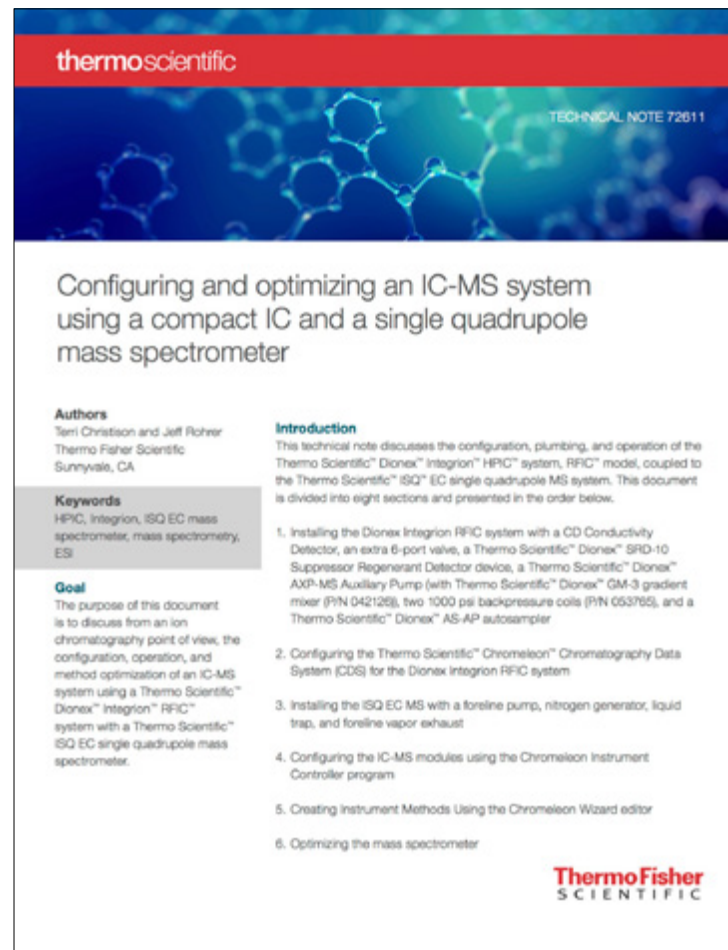


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Configuring and optimizing an IC-MS system

Overview

The purpose of this document is to discuss from an ion chromatography point of view, the configuration, operation, and method optimization of an IC-MS system using a Thermo Scientific™ Dionex™ Integrion™ RFIC™ system with a Thermo Scientific™ ISQ EC single quadrupole mass spectrometer.



[View the full technical note](#)

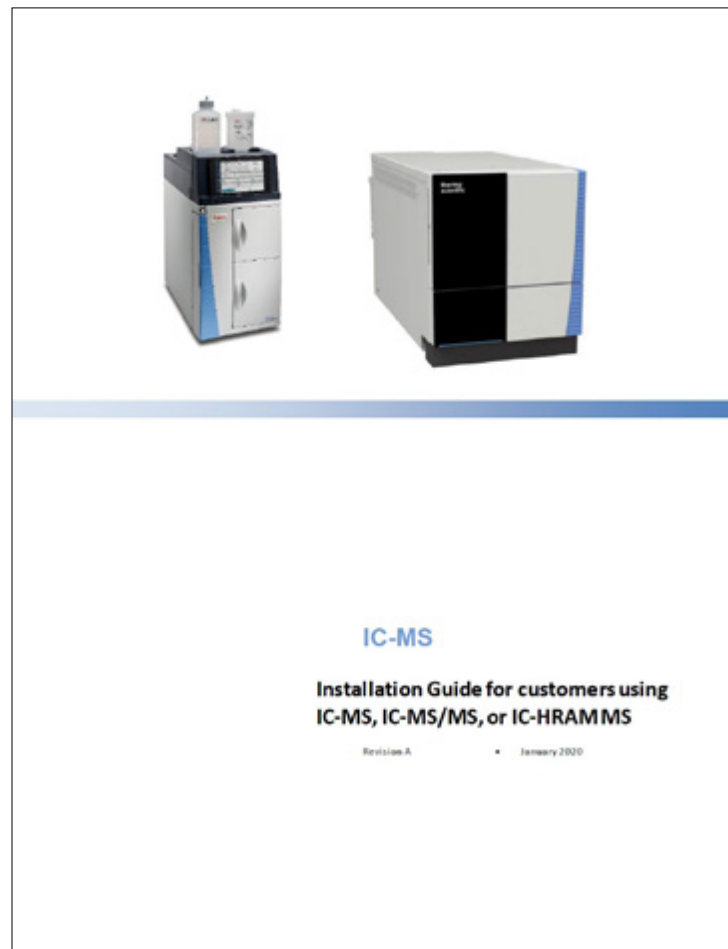
IC-MS Installation Guide

Overview

This manual provides information about IC-MS on Thermo Scientific ion chromatography (IC) systems coupled to Thermo Scientific mass spectrometry (MS) systems.

The layout of this manual is designed to provide quick reference to the sections of interest to the user. To obtain a full understanding of your detector, read this manual thoroughly.

This manual also contains safety messages, precautionary statements, and special notices that when followed properly can prevent personal injury, damage to the detector, or loss of data.

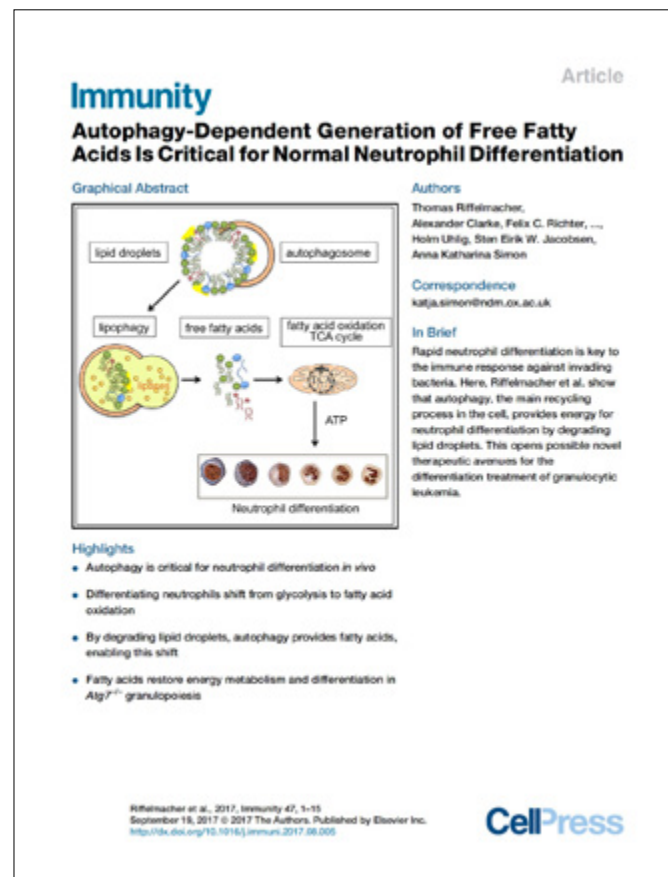


[View the full installation guide](#)

Autophagy-Dependent Generation of Free Fatty Acids Is Critical for Normal Neutrophil Differentiation

Overview

Neutrophils are critical and short-lived mediators of innate immunity that require constant replenishment. Their differentiation in the bone marrow requires extensive cytoplasmic and nuclear remodeling, but the processes governing these energy-consuming changes are unknown. While previous studies show that autophagy is required for differentiation of other blood cell lineages, its function during granulopoiesis has remained elusive. Here, we have shown that metabolism and autophagy are developmentally programmed and essential for neutrophil differentiation *in vivo*. Atg7-deficient neutrophil precursors had increased glycolytic activity but impaired mitochondrial respiration, decreased ATP production, and accumulated lipid droplets. Inhibiting autophagy-mediated lipid degradation or fatty acid oxidation alone was sufficient to cause defective differentiation, while administration of fatty acids or pyruvate for mitochondrial respiration rescued differentiation in autophagy-deficient neutrophil precursors. Together, we show that autophagy-mediated lipolysis provides free fatty acids to support a mitochondrial respiration pathway essential to neutrophil differentiation.

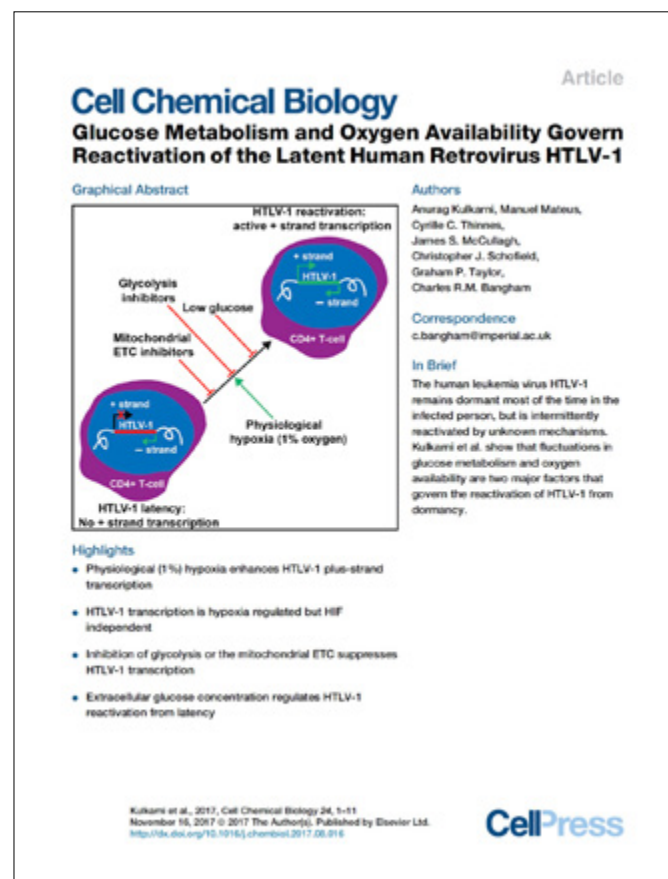


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Glucose Metabolism and Oxygen Availability Govern Reactivation of the Latent Human Retrovirus HTLV-1

Overview

The human retrovirus HTLV-1 causes a hematological malignancy or neuroinflammatory disease in 10% of infected individuals. HTLV-1 primarily infects CD4⁺ T lymphocytes and persists as a provirus integrated in their genome. HTLV-1 appears transcriptionally latent in freshly isolated cells; however, the chronically active anti-HTLV-1 cytotoxic T cell response observed in infected individuals indicates frequent proviral expression *in vivo*. The kinetics and regulation of HTLV-1 proviral expression *in vivo* are poorly understood. By using hypoxia, smallmolecule hypoxia mimics, and inhibitors of specific metabolic pathways, we show that physiologically relevant levels of hypoxia, as routinely encountered by circulating T cells in the lymphoid organs and bone marrow, significantly enhance HTLV-1 reactivation from latency. Furthermore, culturing naturally infected CD4⁺ T cells in glucose-free medium or chemical inhibition of glycolysis or the mitochondrial electron transport chain strongly suppresses HTLV-1 plus-strand transcription. We conclude that glucose metabolism and oxygen tension regulate HTLV-1 proviral latency and reactivation *in vivo*.



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Identification of Circulating Genomic and Metabolic Biomarkers in Intrahepatic Cholangiocarcinoma

Overview

Intrahepatic cholangiocarcinoma (ICC) is an aggressive cancer arising from the bile ducts with a need for earlier diagnosis and a greater range of treatment options. KRAS/NRAS mutations are common in ICC tumours and 6–32% of patients also have isocitrate dehydrogenase 1 and 2 (IDH1 and IDH2) gene mutations associated with metabolic changes. This feasibility study investigated sequencing circulating tumour DNA (ctDNA) combined with metabolite profiling of plasma as a method for biomarker discovery in ICC patients. Plasma was collected from four ICC patients receiving radio-embolisation and healthy controls at multiple time points. ctDNA was sequenced using Ampliseq cancer hotspot panel-v2 on Ion Torrent PGM for single nucleotide variants (SNV) detection and with Illumina whole genome sequencing for copy number variants (CNV) and further targeted examination for SNVs. Untargeted analysis of metabolites from patient and control plasma was performed using liquid chromatography coupled with high-resolution tandem mass spectrometry (LC-MS/MS). Metabolite identification was performed using multi-parameter comparisons with analysis of authentic standards, and univariate statistical analysis was performed to identify differences in metabolite abundance between patient and control samples. Recurrent somatic SNVs and CNVs were identified in ctDNA from three out of four patients that included both NRAS and IDH1 mutations linked to ICC. Plasma metabolite analysis revealed biomarker metabolites associated with ICC and in particular 2-hydroxyglutarate (2-HG) levels were elevated in both samples from the only patient showing a variant allele in IDH1. A reduction in the number of CNVs was observed with treatment.

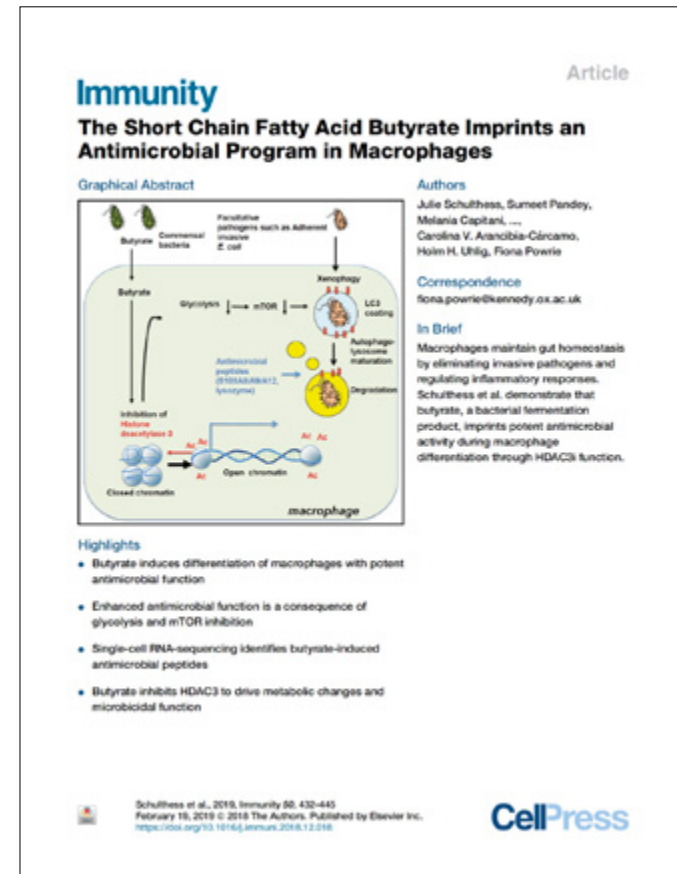


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The Short Chain Fatty Acid Butyrate Imprints an Antimicrobial Program in Macrophages

Overview

Host microbial cross-talk is essential to maintain intestinal homeostasis. However, maladaptation of this response through microbial dysbiosis or defective host defense toward invasive intestinal bacteria can result in chronic inflammation. We have shown that macrophages differentiated in the presence of the bacterial metabolite butyrate display enhanced antimicrobial activity. Butyrate-induced antimicrobial activity was associated with a shift in macrophage metabolism, a reduction in mTOR kinase activity, increased LC3-associated host defense and antimicrobial peptide production in the absence of an increased inflammatory cytokine response. Butyrate drove this monocyte to macrophage differentiation program through histone deacetylase 3 (HDAC3) inhibition. Administration of butyrate induced antimicrobial activity in intestinal macrophages in vivo and increased resistance to enteropathogens. Our data suggest that (1) increased intestinal butyrate might represent a strategy to bolster host defense without tissue damaging inflammation and (2) that pharmacological HDAC3 inhibition might drive selective macrophage functions toward antimicrobial host defense.

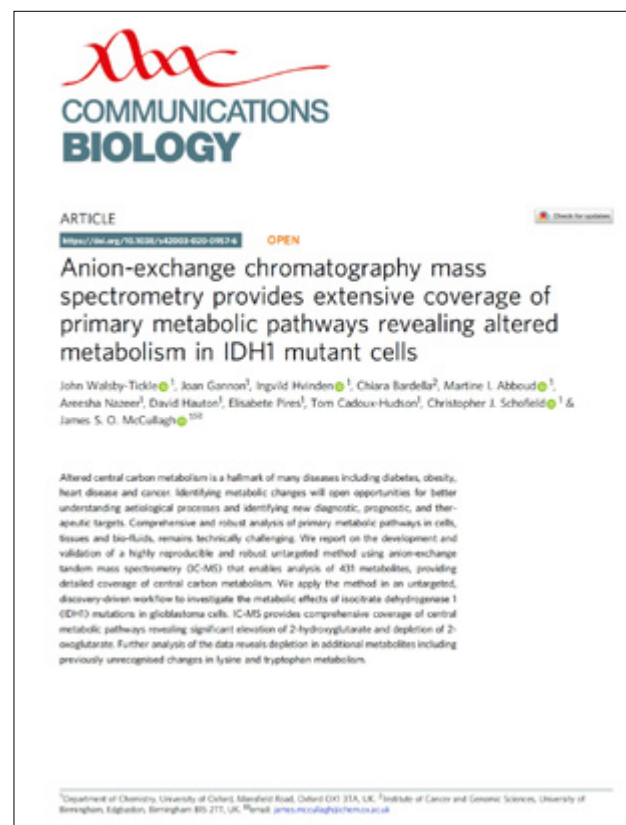


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Anion-exchange chromatography mass spectrometry provides extensive coverage of primary metabolic pathways revealing altered metabolism in IDH1 mutant cells

Overview

Altered central carbon metabolism is a hallmark of many diseases including diabetes, obesity, heart disease and cancer. Identifying metabolic changes will open opportunities for better understanding aetiological processes and identifying new diagnostic, prognostic, and therapeutic targets. Comprehensive and robust analysis of primary metabolic pathways in cells, tissues and bio-fluids, remains technically challenging. We report on the development and validation of a highly reproducible and robust untargeted method using anion-exchange tandem mass spectrometry (IC-MS) that enables analysis of 431 metabolites, providing detailed coverage of central carbon metabolism. We apply the method in an untargeted, discovery-driven workflow to investigate the metabolic effects of isocitrate dehydrogenase 1 (IDH1) mutations in glioblastoma cells. IC-MS provides comprehensive coverage of central metabolic pathways revealing significant elevation of 2-hydroxyglutarate and depletion of 2-oxoglutarate. Further analysis of the data reveals depletion in additional metabolites including previously unrecognised changes in lysine and tryptophan metabolism.



[View the full article](#)

Roles of metal ions in the selective inhibition of oncogenic variants of isocitrate dehydrogenase 1

Overview

Cancer linked isocitrate dehydrogenase (IDH) 1 variants, notably R132H IDH1, manifest a 'gain-of-function' to reduce 2-oxoglutarate to 2-hydroxyglutarate. High-throughput screens have enabled clinically useful R132H IDH1 inhibitors, mostly allosteric binders at the dimer interface. We report investigations on roles of divalent metal ions in IDH substrate and inhibitor binding that rationalise this observation. Mg^{2+}/Mn^{2+} ions enhance substrate binding to wt IDH1 and R132H IDH1, but with the former manifesting lower Mg^{2+}/Mn^{2+} KMs. The isocitrate- Mg^{2+} complex is the preferred wt IDH1 substrate; with R132H IDH1, separate and weaker binding of 2-oxoglutarate and Mg^{2+} is preferred. Binding of R132H IDH1 inhibitors at the dimer interface weakens binding of active site Mg^{2+} complexes; their potency is affected by the Mg^{2+} concentration. Inhibitor selectivity for R132H IDH1 over wt IDH1 substantially arises from different stabilities of wt and R132H IDH1 substrate- Mg^{2+} complexes. The results reveal the importance of substrate-metal ion complexes in wt and R132H IDH1 catalysis and the basis for selective R132H IDH1 inhibition. Further studies on roles of metal ion complexes in TCA cycle and related metabolism, including from an evolutionary perspective, are of interest.



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Ultra-trace determination of oxyhalides in ozonated aquacultural marine waters by direct injection ion chromatography coupled with triple-quadrupole mass spectrometry

Overview

A direct, robust, accurate, and highly sensitive method for oxyhalide species in natural waters, including seawater, using suppressed ion chromatography coupled with mass spectrometry (IC-MS) is described. The method utilized a high capacity, high efficiency anion-exchange column (Thermo Scientific™ Dionex™ IonPac™ AS11-HC, 4 mm, 2 × 250 mm), with the separation achieved using an electrolytically generated potassium hydroxide gradient, delivered at 0.380 mL min⁻¹. Applying the method, detection limits for iodate, bromate, and chlorate in seawater after direct sample injection (20 µL injection volume, samples diluted 10-fold) were 11, 30, and 13 ng L⁻¹ (ppt), respectively. Standard addition calibrations to ozonated seawater samples were linear, in all cases $r^2 > 0.999$ ($n = 10$), with intra-day repeatability of 3.7, 11.2, and 1.8 % ($n = 10$) for a low-level standard mixture (0.30 µg L⁻¹ of iodate, 0.15 µg L⁻¹ of bromate, and 1.50 µg L⁻¹ of chlorate). The method was applied to the analysis of seawater samples taken pre- and post-disinfection points within a recirculating aquacultural system. Iodate, bromate, and chlorate were detected as the main oxyanionic disinfection byproducts, demonstrating the practical utility of the new method as a valuable tool for monitoring changes to seawater composition following disinfection treatments.



 [View the full article](#)



Determination of cations and amines in alkanolamine scrubbing solutions

Overview

Determinations of alkanolamines and inorganic cations by IC-ESI MS were demonstrated on the Dionex IonPac CS19-4 μ m cation-exchange column using the Dionex Integrion IC and ISQ EC single quadrupole mass spectrometer. The method had good sensitivity for sodium and ammonium hydrate adducts to single-digit μ g/L, despite the small injection volume of 2.5 μ L, while also assaying up to 2000 mg/L of the matrix amine. The method showed good accuracy for EA, DEA, and MDEA, as demonstrated by the recovery experiments. IC-MS provided the necessary detection selectivity to ensure an interference-free quantitation of low levels of inorganic cations in the presence of high concentrations of alkanolamines. Through the use of the HESI-II interface, no make-up solvent was needed to support the desolvation process, thus reducing the complexity and costs of the analytical approach.

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APPLICATION NOTE 72609

Using ion chromatography with electrospray ionization mass spectrometry for the determination of cations and amines in alkanolamine scrubbing solutions

Authors
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Sunnyvale, CA

Keywords
HPLC, Integrion, Dionex IonPac CS19-4 μ m column, ISQ EC mass spectrometer, single quadrupole mass spectrometer, Dionex CERS 500e suppressor, inorganic cations, IC-MS, ESI

Goal
To confirm identities of μ g/L to mg/L concentrations of ammonium, calcium, and alkylamines in high mg/L concentrations of alkanolamines by IC-ESI MS

Introduction
Natural gas (methane) is an important energy resource, accounting for 21.6% of the total energy consumed in 2015, and is primarily used for heating, cooking, vehicle fuel, raw materials for various chemicals and plastics production, and to generate electricity.¹ As a result of hydraulic fracturing of previously inaccessible shale deposits, the United States has moved from a moderate producer to the highest producer of natural gas, followed by Russia, Iran, Canada, and Qatar.² However, many of the wells that use hydraulic fracturing contain sour gas, which contains undesirably high concentrations of hydrogen sulfide (H_2S) (>5.7 mg/m³) and high concentrations of carbon dioxide (CO_2) gas.² Sour crude natural gas is very acidic, toxic, and highly corrosive, requiring treatment with amine-rich scrubber solutions to neutralize the CO_2 and remove the H_2S gas impurities.^{3,4}

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Determination of perchlorate by U.S. EPA Method 332.0 using IC-MS

Overview

This application note evaluated the performance of a Thermo Scientific™ Dionex™ Integrion™ HPIC™ System coupled with an ISQ EC MS to fulfill the requirements of EPA Method 332. The results showed this new IC-MS platform can successfully implement EPA Method 332 for the determination of perchlorate in drinking water. This study demonstrated great sensitivity, good precision and accuracy, and ruggedness while all conditions were met, along with passing all other QC requirements needed in EPA Method 332.

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APPLICATION NOTE 72587

Determination of perchlorate by U.S. EPA Method 332.0 using a compact ion chromatography system coupled with mass spectrometry

Authors
Beibei Huang and Jeffrey Rohrer
Thermo Fisher Scientific,
Sunnyvale, CA, USA

Keywords
Dionex Integrion HPIC, Dionex IonPac AS20 column, ISQ EC Single Quadrupole Mass Spectrometer, IC-MS, drinking water analysis

Goal
To identify and quantitate trace levels of perchlorate in raw and finished drinking waters according to U.S. EPA Method 332.0 using a compact high-pressure ion chromatography (HPIC) system coupled with a single quadrupole mass spectrometer

Introduction
Perchlorate has been used as an oxidizer in rockets, munitions, and fireworks since the 1950s. It has been found to cause thyroid dysfunction and has been linked to tumors in humans. Perchlorate is regulated under the Safe Drinking Water Act (2011) although the Maximum Contaminant Level Goal (MCLG) and Maximum Contaminant Level (MCL) have not yet been established. Additionally, numerous U.S. states have promulgated enforceable standards for the levels of perchlorate in drinking water. Massachusetts and California have established standards for perchlorate levels in drinking water of 2 µg/L and 6 µg/L, respectively. More than 10 other states have also set advisory levels or health-based goals for perchlorate in drinking water ranging from 1 to 18 µg/L.

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Determination of oxyhalides and bromide in drinking water using IC-MS

Overview

The IC-MS method described here augments US EPA Method 300.1 Part B (IC-CD) for bromate analysis. Adding the single quadrupole mass spectrometer to the Method 300.1 Part B setup provides advantages over the traditional setup regarding sensitivity and selectivity. IC-MS also offers positive identification of analytes, which is particularly advantageous for complex matrices. This method can simultaneously determine other oxyhalides (chlorite, chlorate) and bromide and thus can deliver Method 300.1 Part B data, confirm peak identification, and provide more sensitive and accurate detection of bromate. Coupling MS with CD takes advantage of the strengths of each detector.

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APPLICATION NOTE 72888

Determination of oxyhalides and bromide in drinking water using a compact ion chromatography system coupled with a single quadrupole mass spectrometer

Authors
Jingli Hu and Jeffrey Rohrer
Thermo Fisher Scientific,
Sunnyvale, CA, USA

Keywords
IC-MS, Dionex Integron HPLC, Dionex IonPac AS19-4µm column, disinfection byproducts, US EPA Method 300.1 Part B, bromate, chlorite, chlorate, ISQ EC mass spectrometer, AERS 500e suppressor, drinking water

Goal
To develop a method to determine oxyhalides and bromide in drinking water by coupling IC with single quadrupole mass spectrometry (IC-MS)

Introduction
Most municipal water authorities disinfect their water to provide their communities with safe drinking water. The most common chemical disinfectants are chlorine, chlorine dioxide, chloramine, and ozone.¹ However, these disinfectants can react with naturally occurring material in the water to form unintended disinfection byproducts (DBPs), which may pose health risks. For example, chlorination of drinking water can produce trihalomethanes, haloacetic acids, and chlorate. Similarly, chlorine dioxide treatment generates inorganic oxyhalides, chlorite, chlorate, and other DBPs. Chlorate may also be generated in the presence of chloramine.² Ozone reacts with natural sources of bromide, which may be found at various levels in water supplies, to produce bromate. Of these DBPs, bromate has been identified by the International Agency for Research on Cancer as an animal carcinogen and potential human carcinogen.³ The World Health Organization (WHO) has estimated an excess lifetime cancer risk of 1 in 10,000 (i.e., in a population of one million, an additional one hundred people are expected to contract cancer over their lifetime) for drinking water containing bromate at 20 µg/L.⁴

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Fast determination of oxyhalides and bromide in drinking water using single quadrupole IC-MS

Overview

This study demonstrates that oxyhalides and bromide can be determined accurately in municipal drinking water and bottled water using a fast isocratic 15-min IC-MS method. The method provides an advantage over the traditional IC-CD method concerning sensitivity and selectivity, offering positive analyte-identification, particularly for complex samples.



Fast determination of oxyhalides and bromide in drinking water using a compact ion chromatography system coupled with a single quadrupole mass spectrometer

Authors
Jingli Ha and Jeffrey Roher
Thermo Fisher Scientific,
Sunnyvale, CA, USA

Keywords
IC-MS, isocratic, Dionex Integron HPLC, Dionex IonPac AS19-4um column, disinfection byproducts, US EPA Method 300.1 Part B, bromate, chlorite, chlorate, ISO IEC mass spectrometer, AERS 500e suppressor, drinking water, RPIC system

Introduction
Most municipal water authorities disinfect their water in order to provide their communities with safe drinking water. The most common chemical disinfectants are chlorine, chlorine dioxide, chloramine, and ozone.¹ However, these disinfectants can react with naturally occurring material in the water to form unintended disinfection byproducts (DBPs), which may pose health risks. For example, chlorination of drinking water can produce trihalomethanes, haloacetic acids, and chlorate. Similarly, chlorine dioxide treatment generates inorganic oxyhalides, chlorite, chlorate, and other DBPs. Chlorate may also be generated in the presence of chloramine.² Ozone reacts with natural sources of bromide, which may be found at various levels in water supplies, to produce bromate. Of these DBPs, bromate has been identified by the International Agency for Research on Cancer as an animal carcinogen and potential human carcinogen.³ The World Health Organization (WHO) has estimated an excess lifetime cancer risk of 10^{-4} , 10^{-5} , 10^{-6} for drinking water containing bromate at levels of 20, 2, and 0.2 $\mu\text{g/L}$, respectively.⁴ Major regulatory bodies worldwide, including the U.S. Environmental Protection Agency (EPA), European Commission (EC), U.S. Food and Drug Administration (FDA), and World Health Organization (WHO), have set the maximum allowable concentration for bromate in drinking water at 10 $\mu\text{g/L}$.⁵ In Europe, the limit was lowered to 3 $\mu\text{g/L}$ for bottled natural mineral and

Goal
To develop a fast, isocratic method to determine oxyhalides and bromide in drinking water by coupling IC with single quadrupole mass spectrometry (IC-MS)



 [View the full application note](#)



Determination of low-level haloacetic acids, bromate, and dalapon in drinking water using IC-MS

Overview

This study evaluated an IC-MS method for the determination of all nine haloacetic acids, bromate, and dalapon in drinking water. The method used a Dionex ICS-6000 HPIC system with DP pump and Dionex IonPac AS31 anion-exchange column coupled with suppressed conductivity detection and a single quadrupole mass spectrometer ISQ EC detector.

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APPLICATION NOTE 73342

Determination of low-level haloacetic acids, bromate, and dalapon in drinking water using IC-MS

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

Keywords: Single quadrupole MS, Dionex IonPac AS31 column, HAAAs, HAA5, disinfection byproducts, ISQ EC, monochloroacetic acid (MCAA), monobromoacetic acid (MBAA), dichloroacetic acid (DCAA), bromochloroacetic acid (BCAA), dibromoacetic acid (DBAA), trichloroacetic acid (TCAA), bromodichloroacetic acid (BDCAA), chlorodibromoacetic acid (CDBAA), tribromoacetic acid (TBAA)

Goal
To develop a method to determine haloacetic acids, bromate, and dalapon in drinking and surface waters using an ion chromatography (IC) system coupled with single quadrupole mass spectrometer (IC-MS)

Introduction
Disinfection treatment is essential to eliminate waterborne disease-causing microorganisms from drinking water. Municipal water authorities most commonly disinfect water using chemical disinfectants such as chlorine, chlorine dioxide, chloramine, and ozone.¹ However, these disinfectants can react with naturally occurring material in the water to form unintended disinfection byproducts (DBPs). For example, chlorination of drinking water can produce trihalomethanes, haloacetic acids (HAAAs), and chloric bromate is formed when disinfecting ozone reacts

with natural sources of bromide. These DBPs may pose health risks. For example, long term ingestion of bromate or haloacetic acids may cause cancer.

Dalapon, a herbicide used to control grasses in a wide variety of crops, can be introduced to waterways from runoff. People who for many years drink water containing dalapon above the maximum contaminant level (MCL), the highest level of a contaminant that is allowed in drinking water, could experience minor kidney changes.²

To ensure drinking water safety, major regulatory bodies worldwide, including the U.S. Environmental Protection Agency (EPA), European Commission (EC), U.S. Food and Drug Administration (FDA), and World Health Organization (WHO), have set the maximum allowable concentrations for toxic compounds and microorganisms in drinking water.³⁻⁵

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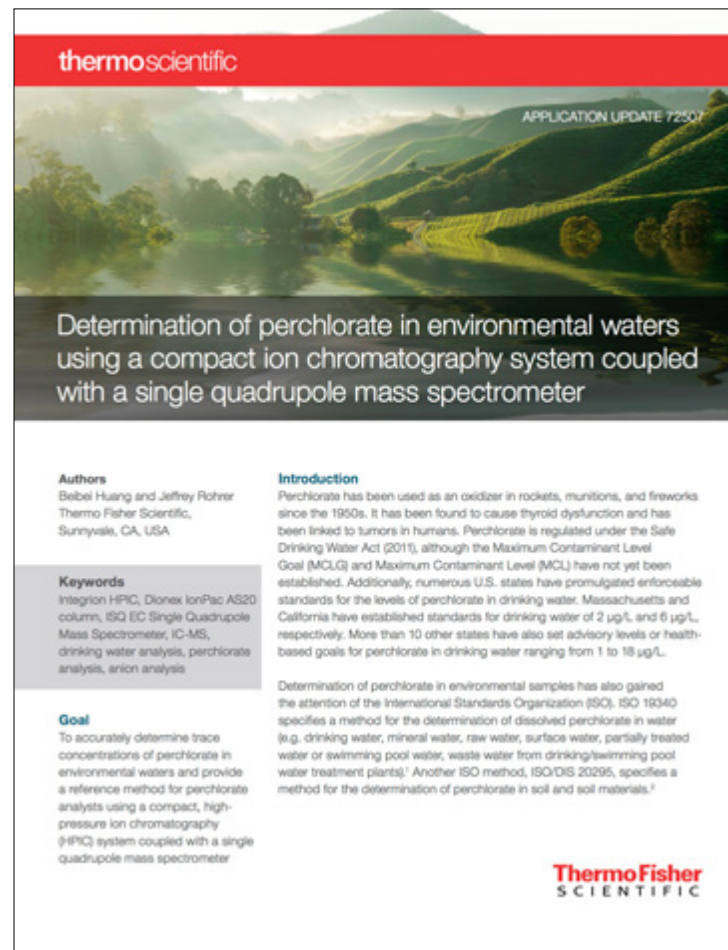
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Determination of perchlorate in environmental waters

Overview

This application update demonstrates an improved approach for the determination of perchlorate in environmental waters using IC-MS. The ISQ EC MS provides lower perchlorate detection limits in high-ionic-strength matrices than conductivity detection alone. Separation of perchlorate from the matrix ions combined with the use of a diverter valve has resulted in a method that minimizes interferences for environmental water samples. The recovery study demonstrated good method accuracy. The ionization interface of the ISQ EC MS instrumentation eliminates the need to add organic solvent to enhance ionization and thus detection.



[View the full application update](#)



Detection of oxyhalides using IC-MS

Overview

Disinfection treatment is used to protect municipal water from potentially dangerous microbes. The most common chemical disinfectants are chlorine, chlorine dioxide, chloramine, and ozone. These chemical disinfectants can react with natural organic and inorganic matter in the source water to produce disinfection byproducts (DBPs), including oxyhalides (e.g., bromate, chlorate, etc.) that are potentially harmful to humans.



Detection of oxyhalides using a compact ion chromatography system coupled with mass spectrometry

Authors
Beibei Huang and Jeffrey Rohrer
Thermo Scientific, Sunnyvale, CA

Keywords
Integrion HPLC, IC-MS, IonPac AS19-4µm column, ISQ EC mass spectrometer, disinfection byproducts, drinking water analysis

Introduction
Disinfection treatment is used to protect municipal water from potentially dangerous microbes. The most common chemical disinfectants are chlorine, chlorine dioxide, chloramine, and ozone.^{1,2} These chemical disinfectants can react with natural organic and inorganic matter in the source water to produce disinfection byproducts (DBPs), including oxyhalides (e.g., bromate, chlorate, etc.), that are potentially harmful to humans.

This application brief demonstrates a method for detecting trace concentrations of oxyhalides using a Thermo Scientific[®] Dionex[™] Integrion[™] HPLC[™] system with a Thermo Scientific[™] Dionex[™] IonPac[™] AS19-4µm column set for separation, coupled to a Thermo Scientific[™] ISQ[™] EC single quadrupole mass spectrometer. The combination of ion chromatography and mass spectrometry (IC-MS) for oxyhalide detection can maximize the benefits of both chromatography and mass confirmation.

Figure 1 shows chromatograms of a 0.5 mg/L fluoride, 0.4 mg/L iodate, 0.1 mg/L bromate, chlorite, and chlorate standard using suppressed conductivity detection and a MS detector in selected ion monitoring (SIM) mode. The conductivity detector responds to all charged species regardless of their molecular mass. The Dionex IonPac AS19-4µm column is recommended to separate low concentrations of oxyhalides from the significantly higher concentrations of inorganic anions in drinking water.





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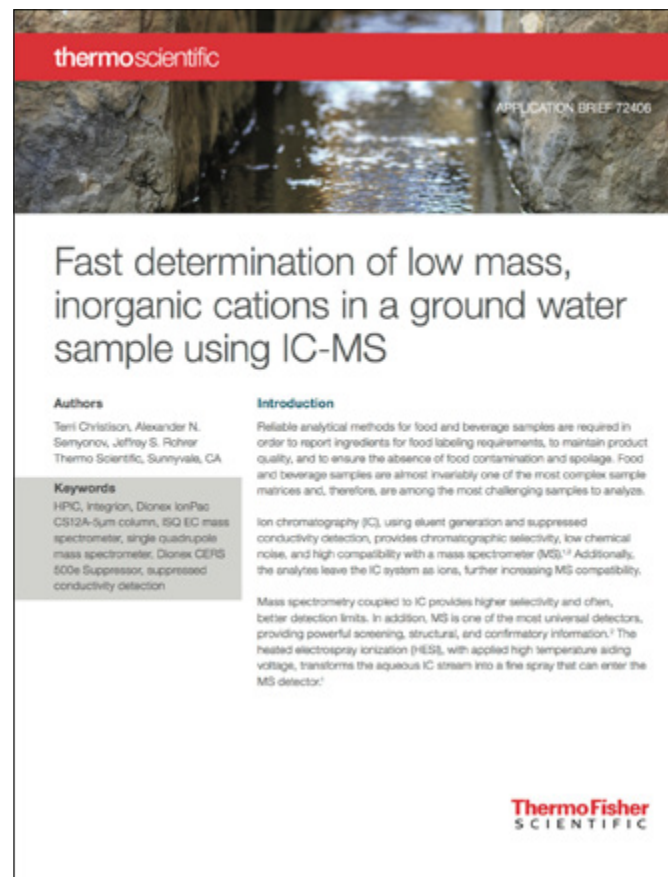




Fast determination of inorganic cations in a ground water sample using IC-MS

Overview

Reliable analytical methods for food and beverage samples are required to report ingredients for food labeling requirements, maintain product quality, and ensure the absence of food contamination and spoilage. Food and beverage samples are almost invariably one of the most complex sample matrices and, therefore, are among the most challenging samples to analyze.



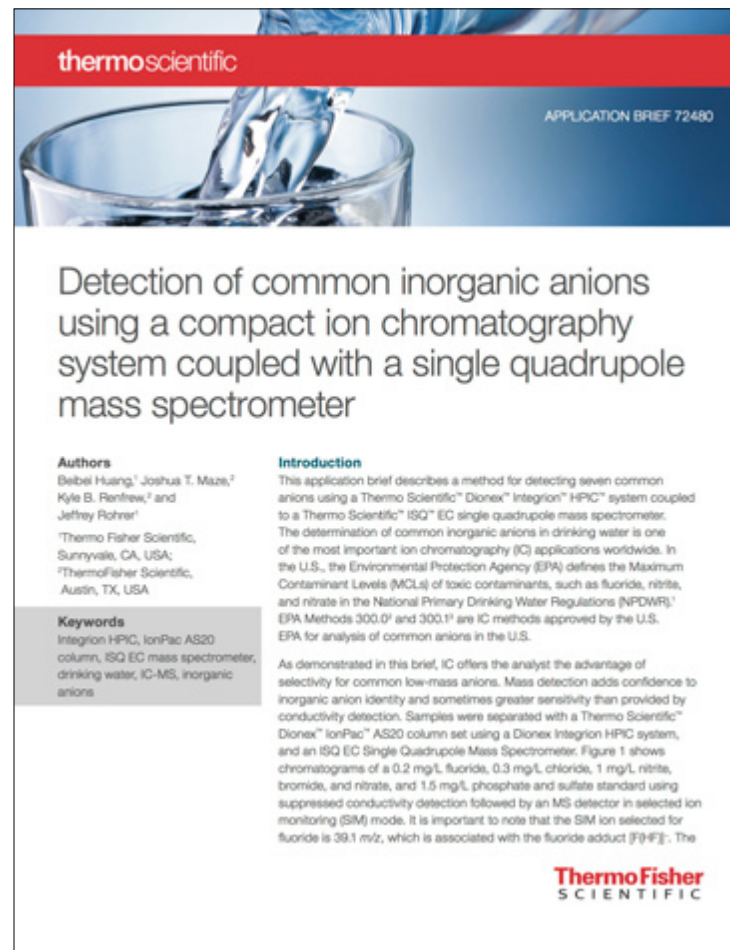
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Detection of common inorganic anions using IC-MS

Overview

This application brief describes a method for detecting seven common anions using a Thermo Scientific™ Dionex™ Integrion™ HPIC™ system coupled to a Thermo Scientific™ ISQ™ EC single quadrupole mass spectrometer. The determination of common inorganic anions in drinking water is one of the most important ion chromatography (IC) applications worldwide. In the U.S., the Environmental Protection Agency (EPA) defines the Maximum Contaminant Levels (MCLs) of toxic contaminants, such as fluoride, nitrite, and nitrate in the National Primary Drinking Water Regulations (NPDWR). EPA Methods 300.0 and 300.1 are IC methods approved by the U.S. EPA for determination of common anions in the U.S.



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Detection of common organic acids using IC-MS

Overview

This application brief demonstrates a method for detecting common organic acids using a Thermo Scientific™ Dionex™ Integron™ HPIC™ system with a Thermo Scientific™ Dionex™ IonPac™ AS11-HC-4µm column set and coupled to a Thermo Scientific™ ISQ™ EC single quadrupole mass spectrometer. Mass spectrometry (MS), as a detection technique for ion chromatography (IC), has recently gained popularity due to the increasing demand for sensitivity, selectivity, confirmation of identity, and structural interpretation. The ISQ EC single quadrupole mass spectrometer increases analytical confidence by providing sensitive detection and mass confirmation in a product that requires less operator training than a typical mass spectrometer.

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APPLICATION BRIEF

Detection of common organic acids using a compact ion chromatography system coupled with mass spectrometry

Authors
Beibei Huang, Jingli Hu, and Jeffrey Rohrer

Keywords
Integron, HPIC, Dionex IonPac AS11-HC-4µm column, ISQ EC single quadrupole mass spectrometer, IC-MS

Introduction
This application brief demonstrates a method for detecting common organic acids using a Thermo Scientific™ Dionex™ Integron™ HPIC™ system with a Thermo Scientific™ Dionex™ IonPac™ AS11-HC-4µm column set and coupled to a Thermo Scientific™ ISQ™ EC single quadrupole mass spectrometer. Mass spectrometry (MS), as a detection technique for ion chromatography (IC), has recently gained popularity due to the increasing demand for sensitivity, selectivity, confirmation of identity, and structural interpretation. The ISQ EC single quadrupole mass spectrometer increases analytical confidence by providing sensitive detection and mass confirmation in a product that requires less operator training than a typical mass spectrometer.

Figure 1 shows chromatograms of 0.5 mg/L each of pyruvate, succinate, malate, and citrate standard using a suppressed conductivity detector (CD) and an MS detector in selected ion monitoring (SIM) mode. The CD responds to all charged species regardless of their molecular mass. If there is a coelution of multiple ionic components, the CD cannot discern them and presents them as one peak.

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Fast determination of nine haloacetic acids, bromate, and dalapon at trace levels in drinking water by IC-MS/MS

Overview

A fast, sensitive, and simple method was developed for direct determination of nine HAAs, bromate, and dalapon in drinking water samples using IC-MS/MS without sample pretreatment. The unique selectivity of the Dionex IonPac AS31 column provides excellent separation of 9 HAAs, bromate, and dalapon from common interference anions such as chloride, sulfate, and carbonate in 35 min. The high sensitivity provided by the MS detection method made it possible to perform the direct injection of drinking water samples, thus eliminating the complexity and variabilities of sample preparation. The use of a hydroxide eluent generator and suppression in the Reagent-Free IC system provides a reliable, economic, and environmentally friendly platform for the separation and detection of HAAs, bromate, and dalapon.

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APPLICATION NOTE 73343

Fast determination of nine haloacetic acids, bromate, and dalapon at trace levels in drinking water samples by tandem IC-MS/MS

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

Keywords: IC-MS/MS, HAAs, HAA5, disinfection byproducts (DBPs), EPA 557, Dionex IonPac AS31 column, Dionex ICS-6000 ion chromatography system, TSQ Fortis triple quadrupole mass spectrometer

Goal
To identify and quantify low concentrations of haloacetic acids, bromate, and dalapon in drinking water according to U.S. 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 byproducts (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 toxicity¹⁻⁴ the World Health Organization (WHO)⁵ has established guidelines for these DBPs in drinking water. In the U.S., 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.⁶ Consequently, efforts have been made to develop fast and accurate

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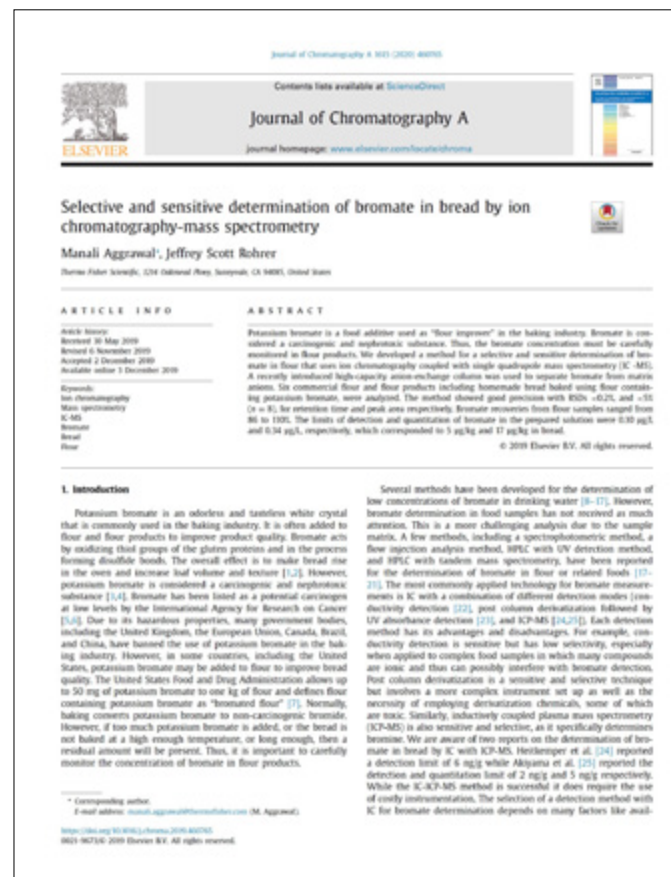
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Selective and sensitive determination of bromate in bread by ion chromatography-mass spectrometry

Overview

Potassium bromate is a food additive used as “flour improver” in the baking industry. Bromate is considered a carcinogenic and nephrotoxic substance. Thus, the bromate concentration must be carefully monitored in flour products. We developed a method for a selective and sensitive determination of bromate in flour that uses ion chromatography coupled with single quadrupole mass spectrometry (IC-MS). A recently introduced high-capacity anion-exchange column was used to separate bromate from matrix anions. Six commercial flour and flour products, including homemade bread baked using flour containing potassium bromate, were analyzed. The method showed good precision with RSDs < 0.2%, and < 5% (n = 8), for retention time and peak area respectively. Bromate recoveries from flour samples ranged from 86 to 110%. The limits of detection and quantitation of bromate in the prepared solution were 0.10 µg/L and 0.34 µg/L, respectively, which corresponded to 5 µg/kg and 17 µg/kg in bread.



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Determination of lactose in lactose-free dairy products using HPAE coupled with PAD and MS dual detection

Overview

Five commercial lactose-free dairy products (milk, half and half milk, cultured milk smoothie, yogurt, and sour cream) were analyzed for their lactose content. With dual detection, lactose was detected down to 0.004% with good precision and accuracy with a recovery range of 85–110%. The combination of PAD and MS detections after HPAE separation allows both the quantification of lactose in low-lactose/lactose-free dairy products and peak confirmation.



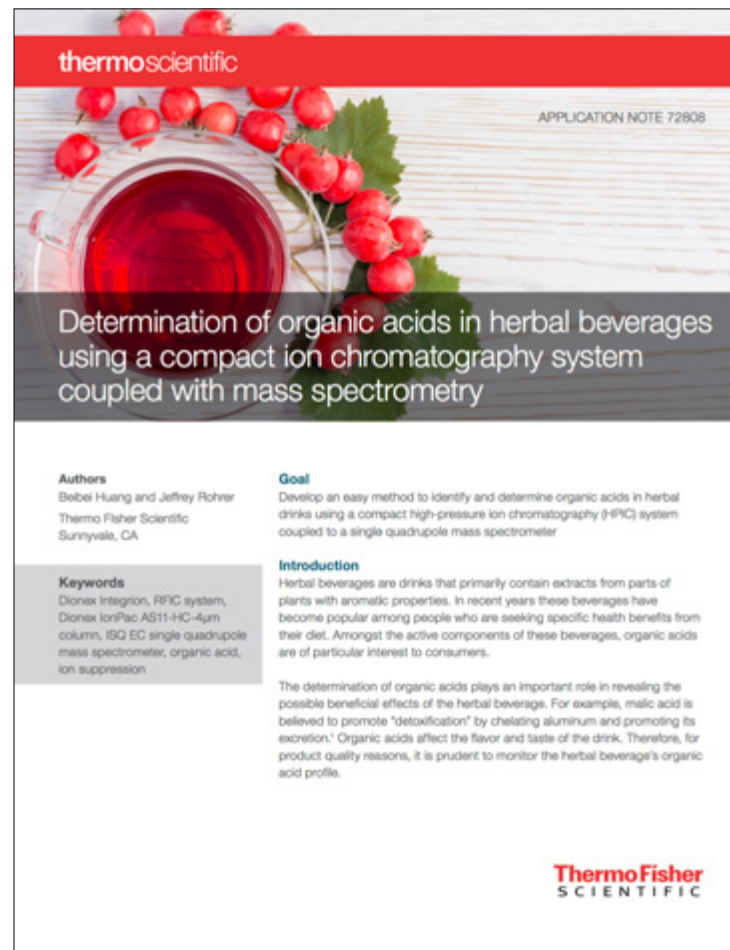
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Determination of organic acids in herbal beverages using IC-MS

Overview

This work uses a Dionex IonPac AS11-HC-4 μ m column on an Integrion IC system that electrolytically generated the eluent for separation coupled to an ISQ EC single quadrupole mass spectrometer. Co-eluting organic acids (OAs) including acetate/glycolate, malate/succinate, and malonate/tartrate were accurately quantified with MS detection. The results showed that this IC-MS method allows the accurate direct determination of OAs in aloe, hawthorn/plum, and goji drinks, especially showing selectivity in detection for co-eluting OAs and confirmation of identity. The ISQ EC MS provides lower detection limits for most OAs except for formate and citrate than conductivity detection alone. A ^{13}C -enriched succinate ($^{13}\text{C}_4$, M+4) internal standard (m/z 121) is recommended to be used for succinate quantitation to improve accuracy and ruggedness when ion suppression occurs.



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Determination of bromate in flour and bread by IC-MS

Overview

Using the high capacity anion exchange Dionex IonPac AS31 column and a large volume injection, low concentrations of bromate were determined in our and bread samples. The addition of an MS detector to the IC system provided high detection specificity and quantification of those samples with an unresolved bromate peak. Six commercial flour and flour products, including homemade bread baked using flour containing potassium bromate, were analyzed. The method showed good precision with RSDs <0.2%, and <5% (n=8), for retention time and peak area, respectively. Bromate recoveries from flour samples ranged from 86% to 110%. The limits of detection and quantitation of bromate in the prepared solution were 0.10 µg/L and 0.34 µg/L, respectively, which corresponded to 5 µg/kg and 17 µg/kg in bread.

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APPLICATION NOTE 73051

Determination of bromate in flour and bread by IC-MS

Authors
Manali Aggarwal and Jeffrey Roehrer
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Sunnyvale, CA

Keywords
IonPac AS31 column, mass spectrometry, ISO EC single quadrupole mass spectrometer, IC-MS, bromate, bread, flour

Introduction
Potassium bromate is a commonly used food additive in the baking industry. It is often added to flour and flour products to make the flour stronger and more extensible. It acts by oxidizing thiol groups of the gluten protein in flour and forming disulfide bonds in the process. The overall effect is to make bread rise in the oven and increase loaf volume and texture.^{1,2} However, potassium bromate is considered a carcinogenic and nephrotoxic substance.^{3,4} Bromate has been listed as a potential carcinogen at low levels by the International Agency for Research on Cancer.^{5,6} Due to its carcinogenic potential, many countries, including the United Kingdom, the European Union, Canada, Brazil, and China, have banned this use of potassium bromate. The US FDA restricts its use and allows up to 50 mg of potassium bromate to 1 kg of flour,⁷ with the belief that the baking process converts potassium bromate to non-carcinogenic bromide. However, if too much potassium bromate is added, or the bread is not baked long enough or at a high enough temperature, then a residual amount will remain. Thus, it is important to carefully monitor the low levels of bromate in flour products. This analysis is challenging due to the complex sample matrices. The most commonly applied technology for bromate determination is IC with a combination of different detection modes (conductivity detection,⁸ post-column derivatization followed by UV absorbance detection,^{9,10} and ICP-MS¹¹⁻¹³).

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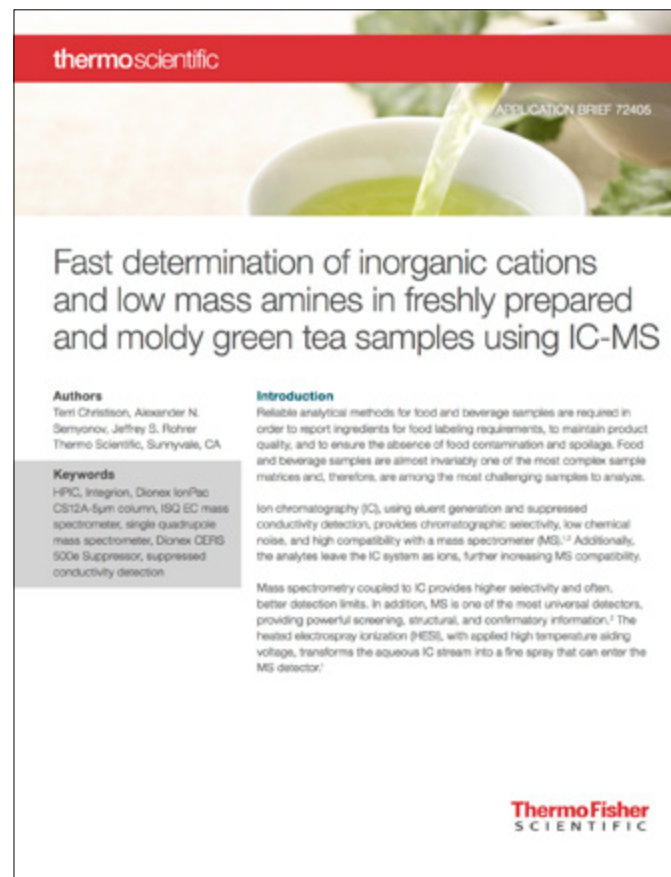




Fast determination of inorganic cations and low mass amines in freshly prepared and moldy green tea using IC-MS

Overview

Reliable analytical methods for food and beverage samples are required to report ingredients for food labeling requirements, maintain product quality, and ensure the absence of food contamination and spoilage. Food and beverage samples are almost invariably one of the most complex sample matrices and, therefore, are among the most challenging samples to analyze.



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Fast determination of inorganic cations and low mass amines in a spoiled cranberry juice sample using IC-MS

Overview

Reliable analytical methods for food and beverage samples are required to report ingredients for food labeling requirements, maintain product quality, and ensure the absence of food contamination and spoilage. Food and beverage samples are almost invariably one of the most complex sample matrices and, therefore, are among the most challenging samples to analyze.

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APPLICATION BRIEF 72404

Fast determination of inorganic cations and low mass amines in a spoiled cranberry juice sample using IC-MS

Authors
Teri Christison, Alexander N. Semynov, Jeffrey S. Roher
Thermo Scientific, Sunnyvale, CA

Keywords
HPLC, Integron, Dionex IonPac CS13A-4um column, ISO EIC mass spectrometer, single quadrupole mass spectrometer, Dionex CEFS 500e Suppressor, suppressed conductivity detection

Introduction
Reliable analytical methods for food and beverage samples are required in order to report ingredients for food labeling requirements, to maintain product quality, and to ensure the absence of food contamination and spoilage. Food and beverage samples are almost invariably one of the most complex sample matrices and, therefore, are among the most challenging samples to analyze.

Ion chromatography (IC), using eluent generation and suppressed conductivity detection, provides chromatographic selectivity, low chemical noise, and high compatibility with a mass spectrometer (MS).^{1,2} Additionally, the analytes leave the IC system as ions, further increasing MS compatibility.

Mass spectrometry coupled to IC provides higher selectivity and often, better detection limits. In addition, MS is one of the most universal detectors, providing powerful screening, structural, and confirmatory information.³ The heated electrospray ionization (HESI), with applied high temperature sliding voltage, transforms the aqueous IC stream into a fine spray that can enter the MS detector.⁴

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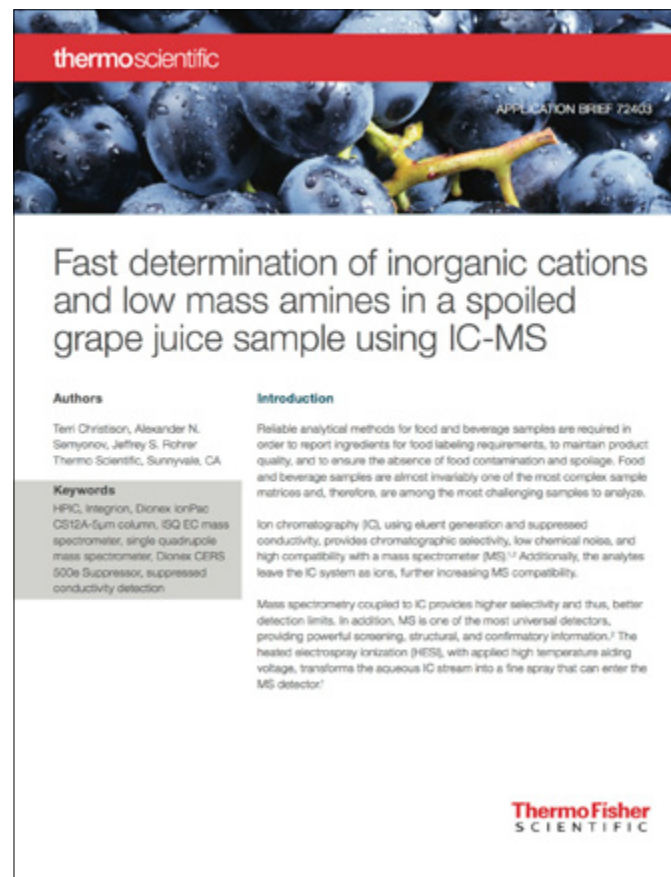
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Fast determination of inorganic cations and low mass amines in a spoiled grape juice sample using IC-MS

Overview

Reliable analytical methods for food and beverage samples are required to report ingredients for food labeling requirements, maintain product quality, and ensure the absence of food contamination and spoilage. Food and beverage samples are almost invariably one of the most complex sample matrices and, therefore, are among the most challenging samples to analyze.



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Fast and sensitive analysis of N-containing bisphosphonates without derivatization using IC-MS

Overview

Bisphosphonates are a class of chemical compounds used in various medical approaches. These active pharmaceutical ingredients (API) are mainly used against bone disorders, including osteoporosis. The older and most used bisphosphonates are etidronate, clodronate, and tiludronate. Now bisphosphonates are used in oncology for treatment of cancer hypercalcemia and metastatic bone diseases. Many new APIs have been developed with the addition of a basic nitrogen in the bisphosphonate side chain. This modification has critically modified the product's potency. For example, pamidronate (N-containing bisphosphonate) is one hundred times more potent than etidronate (non-N-containing bisphosphonate). Regarding the better potency, this new API represents a great potential for doping.

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APPLICATION BRIEF 72910

Fast and sensitive analysis of N-containing bisphosphonates without derivatization using IC-MS

Author
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Keywords
HPLC, ICS-6000, Dionex IonPac
AS18 4µm Fast Column, ISQ
EC mass spectrometer, single
quadrupole mass spectrometer,
Dionex AERS 500 Suppressor,
pharmaceutical compounds

Introduction
Bisphosphonates are a class of chemical compounds used in various medical approaches. These active pharmaceutical ingredients (API) are mainly used against bone disorders including osteoporosis. The older and most used bisphosphonates are etidronate, clodronate, and tiludronate. Now bisphosphonates are used in oncology for treatment of cancer hypercalcemia and metastatic bone diseases. Many new APIs have been developed with the addition of a basic nitrogen in the bisphosphonate side chain. This modification has critically modified the product's potency. For example, pamidronate (N-containing bisphosphonate) is one hundred times more potent than etidronate (non-N-containing bisphosphonate). Regarding the better potency, this new API represents a great potential for doping.

Due to the very polar and strong chelating characteristics of bisphosphonates, their analysis represents a real analytical challenge. The scientific literature reports different approaches to performing bisphosphonates analysis: ion-pairing HPLC with light scattering detection, ion chromatography (IC) with UV indirect detection, HPLC with charged aerosol detection¹ or LC-MS with derivatization². In complex matrices like plasma, only LC-MS with derivatization can provide the required selectivity and sensitivity. However, sample preparation is still required and is a time-consuming step.

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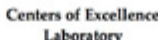
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Determination of perchlorate in honey

Overview

This report demonstrates the use of ISQ-EC makes possible the determination of a trace amount of perchlorate in matrix with high amount of sugars by scanning for perchlorate at specific mass. To show method accuracy, recovery was checked by adding a known amount of perchlorate.



Summary:

Perchlorate in commercially available honey was analyzed on Thermo ICS-6000 ion chromatography system with ISQ-EC single quadrupole mass spectrometer. Below is the result of the analysis.

Brand	Perchlorate in Honey (µg/Kg)
A	1.7
B	3.2
C	3.1

Amount was calculated from SIM scan mass chromatogram at 99 m/z with an internal standard scanned at 107 m/z on ISQ-EC mass spectrometer.

This report demonstrates the use of ISQ-EC makes trace amount of perchlorate analysis in matrix with high amount of sugars possible by scanning for perchlorate at specific mass. Recovery % was also checked by adding known amount of perchlorate to show accuracy of analysis.

Use of diverter valve between conductivity detector and ISQ-EC is recommended to direct the early eluting sugars to waste.

This application report is published as a proof of principle. No ISO data available for included figures.

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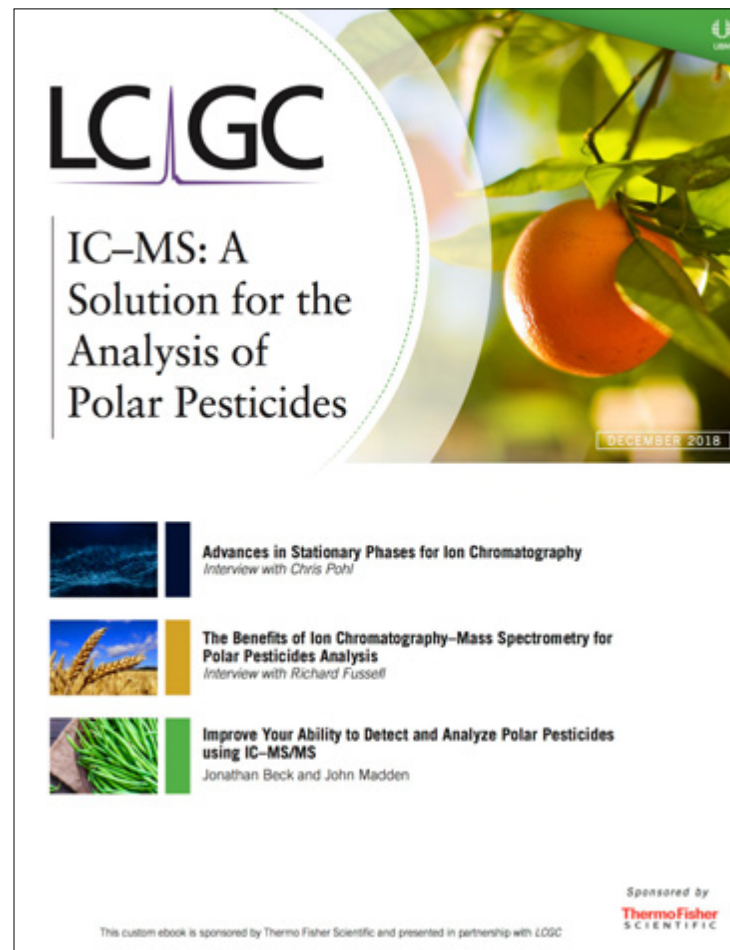




IC-MS: A Solution for the Analysis of Polar Pesticides

Overview

Polar pesticides such as glyphosate are widely used in agriculture, but the analysis of their residues is challenging. LCGC's new ebook on IC-MS: A Solution for the Analysis of Polar Pesticides (sponsored by Thermo Fisher Scientific) explains why ion chromatography-mass spectrometry (IC-MS) systems provide advantages for laboratories tasked with detecting and characterizing polar pesticides in diverse samples.



[View the full eBook](#)





Determination of polar pesticides in grapes using IC-MS/MS

Overview

We introduced a new workflow based on the modified QuPPE method and IC-MS/MS that supports simultaneous multiresidue analysis for polar pesticides in grape samples. The IC-MS/MS method was developed using a Dionex IonPac AS19-4 μ m column set and a compact IC system coupled to a TSQ Quantis triple quadrupole mass spectrometer. The modified QuPPE method used pure methanol instead of acidified methanol to extract the analytes and a Dionex OnGuard II RP cartridge as the clean-up step. To increase sensitivity, electrospray ionization was improved by post-column introduction of acetonitrile into the eluents. Matrix-matched calibration was used to compensate for matrix effects. The results showed that the sensitivity, linearity, retention time precision, and recovery align with the SANTE/11813/2017 method performance criteria. The method provides lower LOQs than EU MRLs. Overall, this workflow supported simultaneous multiresidue analysis of polar pesticides in the grape samples using the modified QuPPE method.

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APPLICATION NOTE 72919

Determination of polar pesticides in grapes using a compact ion chromatography system coupled with tandem mass spectrometry

Authors
Beibei Huang¹, Michael Volny²,
Claudia Martins¹, Alexander
Semyonov¹, and Jeffrey Rohrer¹
¹Thermo Fisher Scientific,
Sunnyvale, CA, USA
²Thermo Fisher Scientific,
San Jose, CA, USA

Goal
To develop and test a method based on ion chromatography (IC) coupled with a triple quadrupole mass spectrometer (IC-MS/MS) for the determination of polar pesticides and their metabolites in grapes. Method performance should be in compliance with statutory maximum residue levels (MRL)/tolerance levels, residue definitions, and relevant guidelines for method validation and analytical quality control.

Introduction
The group of polar ionic pesticides include some of the most frequently used pesticides worldwide.¹ Although these compounds result in residues in food and have been the subject of recent controversy, they have been monitored infrequently in food-testing programs. In the United States, for example, a report by the Government Accounting Office² criticized the responsible government agencies [Environmental Protection Agency (EPA), Food and Drug Administration (FDA), and Department of Agriculture (USDA)] with respect to the lack of testing for glyphosate residues in food. The lack of testing is in part due to the analytical difficulties and higher costs associated with the single-residue methods that were until recently the only options available.

Keywords
AMPA, Glyphosate, Integron, HPLC system, Dionex IonPac AS19-4 μ m column, TSQ Quantis triple quadrupole mass spectrometer, polar pesticides

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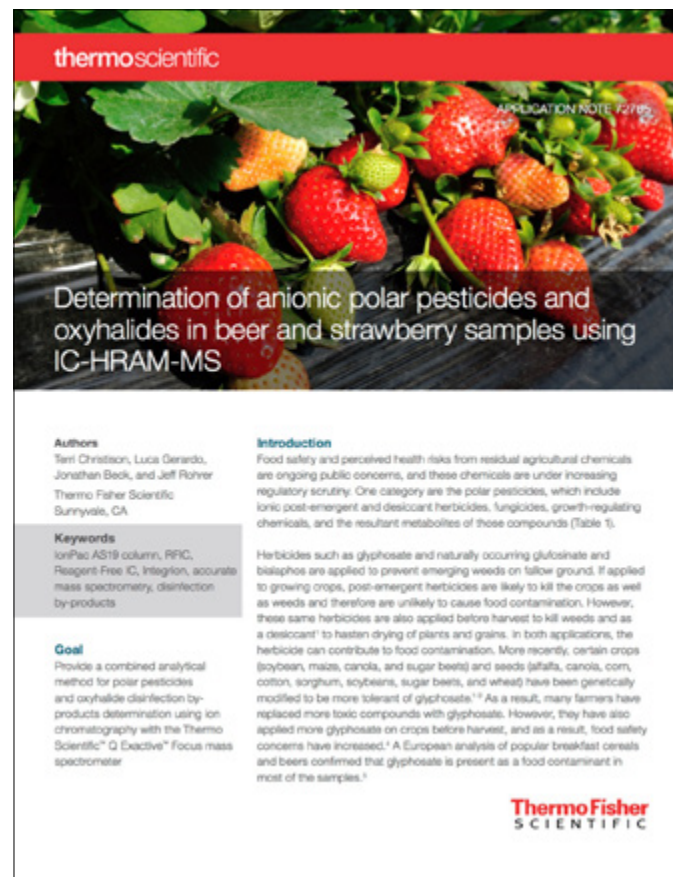


Determination of anionic polar pesticides and oxyhalides in beer and strawberry samples using IC-HRAM-MS

Overview

This application note demonstrates fast direct determinations of 18 analytes that include 14 residual polar pesticides, three DBPs, and perchlorate using IC- HRAM-MS. The method uses a Dionex Integrion HPIC system and Thermo Scientific™ Q Exactive™ Focus Hybrid Quadrupole-Orbitrap Mass Spectrometer. These determinations are essential to the food industry and food regulatory industry to ensure food safety or investigate possible food safety issues.

The method was applied to homogenized strawberry samples and seven diluted beer samples. Bromide at sub-ppb concentrations was found in the strawberries, which could be due to methylbromide, a common strawberry fungicide. Glyphosate at sub-ppb concentrations was determined in three of the beer samples: a rice-barley beer, a non-alcoholic barley beer, and a gluten-free sorghum beer. It is interesting to note that glyphosate was not found in any of the wheat beers despite the common application of glyphosate as a desiccant to wheat crops.



[View the full application note](#)



Determination of cationic polar pesticides in homogenized fruit and vegetable samples using IC-HRAM-MS

Overview

Food safety and perceived health risks from residual agricultural chemicals are an ongoing public concern and a subject of increasing regulatory scrutiny. One such group is known as polar pesticides. These include ionic emergent and desiccant herbicides, fungicides, growth-regulating chemicals, and the resultant metabolites of those compounds. Although pesticide and pesticide residues are commonly determined by GC-MS or HPLC-MS methods, polar pesticides are best determined by ion chromatography-mass spectrometry (IC-MS) methods because of their ionic and non-volatile nature. Food Environmental Research Association (FERA LTD) and others have successfully demonstrated determinations of anionic pesticide residues (glyphosate, glufosinate, fosetyl, and metabolites) in beer, fruit, and vegetables using IC-MS/MS and IC-high-resolution accurate mass (HRAM) MS. Determinations of cationic polar pesticides were also previously demonstrated using IC-MS/MS, however paraquat and diquat were problematic without HRAM-MS. These applications demonstrate the advantages of using IC coupled with MS over other separation methods to separate ionic compounds.



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Determination of histamine, agmatine, and putrescine in wine by ion chromatography-single quadrupole mass spectroscopy

Overview

Biogenic amines have important biological functions, such as regulating growth, controlling blood pressure, and facilitating neural transmittance. Although biogenic amines can occur naturally in many foods (fish, meat, dairy, fruits, vegetables, chocolate, and wine), they are usually present due to microbial activity. Vintners use malolactic fermentation to create the desired flavors. However, certain strains of the lactic acid bacteria necessary for this fermentation can also produce biogenic amines by decarboxylating amino acids. Putrescine, cadaverine, histamine, and agmatine have been found in red wines, and to a lesser extent, in white wines. In healthy persons, dietary histamine is rapidly detoxified by diamine oxidase and other amine oxidases. However, people with histamine intolerance or with low amine oxidase activity can experience minor to serious health effects, ranging from headaches, flushing, rhinitis, and urticaria, to changes in blood pressure (hyper- or hypotension), heart rate (increased heart rate or arrhythmia), and breathing (asthma symptoms). Multiple factors have been surmised to impact concentration and type of biogenic amines occurring in wine, such as storage time and conditions, grape varieties, and contamination by wild type and certain strains of lactic acid bacteria; therefore, it is important to have suitable analytical methods for determining biogenic amines.

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APPLICATION NOTE000164

Determination of histamine, agmatine, and putrescine in wine by ion chromatography-single quadrupole mass spectroscopy

Authors: Terri Christison and Jeff Rohrer
Thermo Fisher Scientific, Sunnyvale, CA, USA

Keywords: Dionex IonPac CS19-4 μ m column, RPLC, Reagent-Free IC, IC-MS, biogenic amines

Goal
Demonstrate determinations of three biogenic amines in wine by ion chromatography coupled with electrospray ionization single quadrupole mass spectrometry

Introduction
Biogenic amines have important biological functions, such as regulating growth, controlling blood pressure, and facilitating neural transmittance. Although biogenic amines can occur naturally in many foods (fish, meat, dairy, fruits, vegetables, chocolate, and wine), they are usually present due to microbial activity.^{1,2} Vintners use malolactic fermentation to create the desired flavors. However, certain strains of the lactic acid bacteria necessary for this fermentation can also produce biogenic amines by decarboxylating amino acids. Putrescine, cadaverine, histamine, and agmatine have been found in red wines, and to a lesser extent, in white wines. In healthy persons, dietary histamine is rapidly detoxified by diamine oxidase and other amine oxidases.^{3,4} However, people with histamine intolerance or with low amine oxidase activity can experience minor to serious health effects, ranging from headaches, flushing, rhinitis, and urticaria, to changes in blood pressure (hyper- or hypotension), heart rate (increased heart rate or arrhythmia), and breathing (asthma symptoms).^{5,6} Multiple factors have been surmised to impact concentration and type of biogenic amines occurring in wine, such as storage time and conditions, grape varieties, and contamination by wild type and certain strains of lactic acid bacteria; therefore, it is important to have suitable analytical methods for determining biogenic amines.

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Determination of glyphosate and AMPA in oat flour by IC-MS

Overview

Glyphosate is the active ingredient in the popular herbicide Roundup® and is widely used for weed control in cultivated and uncultivated areas. There are concerns about its potential adverse effects on human health, such as its potential carcinogenicity. The bacteria in soil break down glyphosate into aminomethylphosphonic acid (AMPA), which may also be toxic to humans. Due to its widespread use, trace amounts of glyphosate residues may be found in various fruits, vegetables, and cereals. The United States Environmental Protection Agency (EPA) set the maximum amount of glyphosate at 30 mg/kg for oats. Glyphosate was reported to be present in oat-based cereals up to 2,837 µg/kg by the Environmental Working Group (EWG), a nonprofit organization⁴ Although the amounts of glyphosate found in oat products were well below the EPA tolerance, the EWG is petitioning the EPA to lower the acceptable amount of glyphosate in oats.

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APPLICATION NOTE000262

Determination of glyphosate and AMPA in oat flour by IC-MS

Authors: Jingli Hu and Jeffrey Rohrer

Keywords: Single quadrupole mass spectrometer, Dionex Integriion HPLC, Dionex IonPac AS10-4µm column, ISQ EC, Dionex ADRS 600 suppressor

Goal
To develop a method to determine glyphosate and AMPA in oat flour by coupling ion chromatography with single quadrupole mass spectrometry (IC-MS)

Introduction
Glyphosate is the active ingredient in the popular herbicide Roundup® and is widely used for weed control in cultivated and uncultivated areas. There are concerns about its potential adverse effects on human health, such as its potential carcinogenicity.¹ The bacteria in soil break down glyphosate into aminomethylphosphonic acid (AMPA), which may also be toxic to humans.² Due to its widespread use, trace amounts of glyphosate residues may be found in various fruits, vegetables, and cereals. The United States Environmental Protection Agency (EPA) set the maximum amount of glyphosate at 30 mg/kg for oats.³ Glyphosate was reported to be present in oat based cereals up to 2,837 µg/kg by the Environmental Working Group (EWG), a nonprofit organization.⁴ Although the amounts of glyphosate found in oat products were well below the EPA tolerance, the EWG is petitioning the EPA to lower the acceptable amount of glyphosate in oats.



Determining glyphosate and AMPA is challenging due to their high polarity, low volatility, and lack of a chromophore. Glyphosate can be determined in food products by HPLC or GC. However, those methods require tedious and time-consuming derivatization. Ion chromatography with mass spectrometry (IC-MS) is more suitable for glyphosate determinations because these pesticides and their metabolites are ionic. As a result, a direct analysis with no derivatization is possible. An ion chromatography-tandem mass spectrometry (IC-MS/MS) method was developed for the determination of glyphosate, AMPA, and other polar pesticides in food products.⁵

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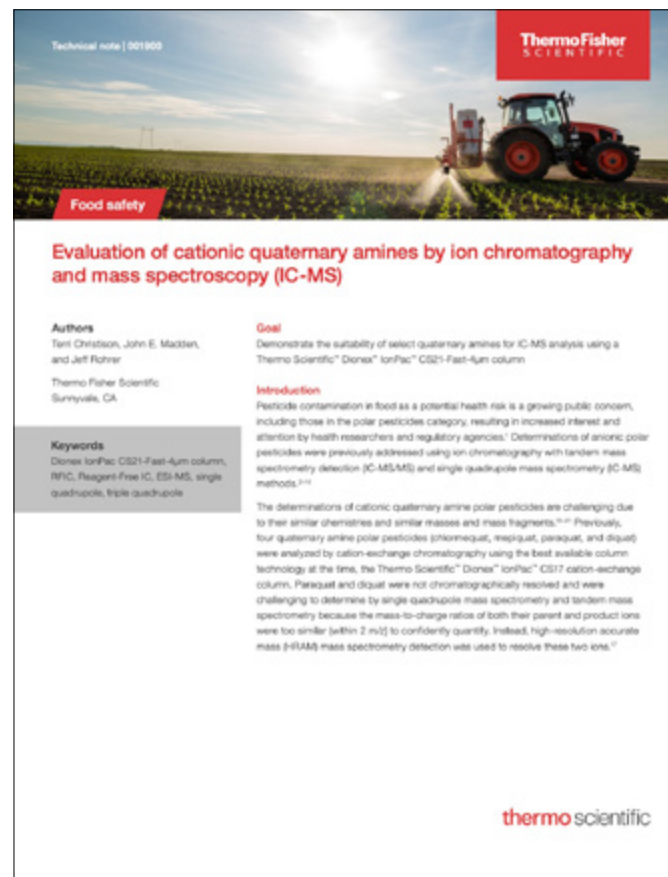
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Evaluation of Cationic Quaternary Amines by Ion Chromatography and Mass Spectroscopy (IC-MS)

Overview

Cationic quaternary amine polar pesticides are particularly challenging due to their similar chemistries, masses, and mass fragments. A series of quaternary amine polar pesticides were analyzed with the Thermo Scientific™ Dionex™ IonPac™ CS21-Fast-4 μ m column set with a Thermo Scientific™ ISQ EC™ single quadrupole mass spectrometer with HESI-II probe. Previously only four quaternary amine polar pesticides were demonstrated, while this application note shows twelve of them are possible with this IC-MS setup.



[View the full technical note](#)



Determination of Cationic Polar Pesticides in Oat Cereals by Ion Chromatography and Electrospray Ionization Mass Spectrometry

Overview

Oat cereal is a very difficult matrix but with the Thermo Scientific™ Dionex™ IonPac™ CS21-Fast-4 μ m column set with a Thermo Scientific™ ISQ EC™ single quadrupole mass spectrometer with HESI-II probe, we can screen Chlormequat, mepiquat, paraquat, and diquat. This provides an accurate method with enough screening sensitivity. For great sensitivity, the use of IC-MS/MS is needed.



Application note | 001956

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ion chromatography

Determination of cationic polar pesticides in oat cereals by ion chromatography and electrospray ionization mass spectroscopy

Authors
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Keywords
Dionex IonPac CS21-Fast-4 μ m column, RPIC, Reagent-free IC, ESI-MS, single quadrupole, diquat, paraquat, mepiquat, chlormequat

Goal
Demonstrate determinations of cationic polar pesticides by ion chromatography coupled with electrospray ionization mass spectrometry

Introduction
Pesticide contamination in food as a potential health risk is a growing public concern, resulting in increased interest and attention by health researchers and regulatory agencies. Collectively grouped into the category of "ionic and Highly Polar Pesticides" are herbicides, fungicides, defoliants, and desiccants. For traditional solvent-based pesticides, the regulatory test methods are typically gas and liquid chromatography combined with mass spectrometry. However, polar pesticides, which are ionic and non-volatile, are much more suitable for methods using ion chromatography (IC) separations combined with mass spectrometry (MS) detection. Consequently, determinations of anionic polar pesticide residues, such as glyphosate, glufosinate, and their degradation products, such as AMPA (aminomethylphosphonic acid), have been successfully demonstrated in beer, fruit, cereals, vegetables, and drinking water by the Food Environmental Research Association (Fera Science Ltd.)^{1,2} and others³⁻⁵ using IC with MS (MS/MS, high-resolution accurate mass (HRAM) MS, or single quadrupole MS).

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Determination of Chlorate and Perchlorate in Powdered Infant Formula using Ion Chromatography-Mass Spectrometry

Overview

Recent reports by the European Food Safety Authority (EFSA) found that current levels of chlorate in drinking water and foods were too high and could negatively impact iodine uptake, especially among infants and children. Chlorate originates from chlorine disinfectants widely and legally used in water treatment and food processing, with drinking water being overwhelmingly the main contributor.

The sample preparation was optimized for effective extraction and cleanup and can be applied to various powdered dairy samples. The method uses a Thermo Scientific™ Dionex™ IonPac™ AS20 column on a Thermo Scientific™ Dionex™ Integriion IC system, which electrolytically generated the eluent for separation, coupled to a Thermo Scientific™ ISQ EC™ single quadrupole mass spectrometer. The method showed good sensitivity for the detection, identification, and quantification of chlorate and perchlorate.

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APPLICATION NOTE73854

Determination of chlorate and perchlorate in powdered infant formula using ion chromatography-mass spectrometry

Authors: Beibei Huang and Jeffrey Rohrer
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Keywords: Integriion HPLC, Dionex IonPac AS20 column, ISQ EC single quadrupole mass spectrometer, IC-MS

Goal
To develop an IC-MS method for the determination of chlorate and perchlorate in powdered infant formula.

Introduction
Food can be contaminated by chlorate and perchlorate during different stages of production. Perchlorate can be present in food via the use of fertilizers, while chlorate can be present due to the use of chlorinated disinfectants during food production. The dairy industry often uses hypochlorite and chlorine dioxide as sanitizing agents. Studies have confirmed that the use of these types of sanitizing agents could result in elevated chlorate levels in final products.

Recent reports by the European Food Safety Authority (EFSA) found that current levels of chlorate in drinking water and foods were too high and could negatively impact iodine uptake, especially among infants and children. Chlorate originates from chlorine disinfectants widely and legally used in water treatment and food processing, with drinking water being overwhelmingly the main contributor.

The EFSA is currently reviewing the MRLs for chlorate and perchlorate in food.¹

Ion chromatography (IC) with suppressed conductivity detection combined with electrospray ionization/ mass spectrometry is one of the most sensitive and robust characterization methods available for chlorate and perchlorate. This Application Note uses a Thermo Scientific™ Dionex™ IonPac™ AS20 column set on a compact IC system (Thermo Scientific™ Dionex™ Integriion™ HPLC system) coupled with a Thermo Scientific™ ISQ EC™ single quadrupole mass spectrometer to determine chlorate and perchlorate in powdered infant formula.

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Determination of Chlorate and Perchlorate in Milk using Ion Chromatography-Mass Spectrometry

Overview

Chlorate and other oxychlorine species have been associated with inhibition of iodine uptake in humans and the formation of methemoglobin. Infants and young children are at high risk due to their greater dependency on milk products and higher consumption/body weight ratio. Chlorate and perchlorate have traditionally been analyzed by ion chromatography (IC). More recently, IC coupled to a mass spectrometer (IC-MS) has become a highly preferred method for determining chlorate and perchlorate in complex sample matrices. MS detection provides greater data confidence while minimizing labor-intensive sample preparation.

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APPLICATION NOTE 73981

Determination of chlorate and perchlorate in milk using ion chromatography-mass spectrometry

Authors: Beibei Huang and Jeffrey Rohrer
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Keywords: Integriion HPIC, Dionex IonPac AS20 column, ISQ EC Single Quadrupole Mass Spectrometer, IC-MS

Goal
To develop an IC-MS method for the determination of chlorate and perchlorate in milk.

Introduction
Chlorate in milk and dairy products can arise from chlorinated water and chlorine-containing disinfectants for cleaning and sanitation of process equipment at both the farm and food processor stages. Perchlorate is an environmental contaminant that occurs naturally, primarily near potash deposits and in arid regions. It can also originate from perchlorate salts in military and industrial products such as solid rocket fuels, explosives, fireworks, and some fertilizers.

Chlorate and other oxychlorine species have been associated with inhibition of iodine uptake in humans and the formation of methemoglobin. Infants and young children are at high-risk due to their greater dependency on milk products and higher consumption/body weight ratio.¹

Chlorate and perchlorate have traditionally been analyzed by ion chromatography (IC). More recently, IC coupled

to a mass spectrometer (IC-MS) has become a highly preferred method for determining chlorate and perchlorate in complex sample matrices. MS detection provides greater data confidence while minimizing labor-intensive sample preparation.

This Application Note uses a Thermo Scientific™ Dionex™ IonPac™ AS20 column set on a compact IC system (Thermo Scientific™ Dionex™ Integriion™ HPIC system) coupled with a Thermo Scientific™ ISQ EC™ single quadrupole MS. With a simple sample preparation, this method offers an easy approach to quantifying chlorate and perchlorate in milk. The Dionex IonPac AS20 column is a high capacity, hydroxide selective anion-exchange column designed to separate perchlorate from cations.

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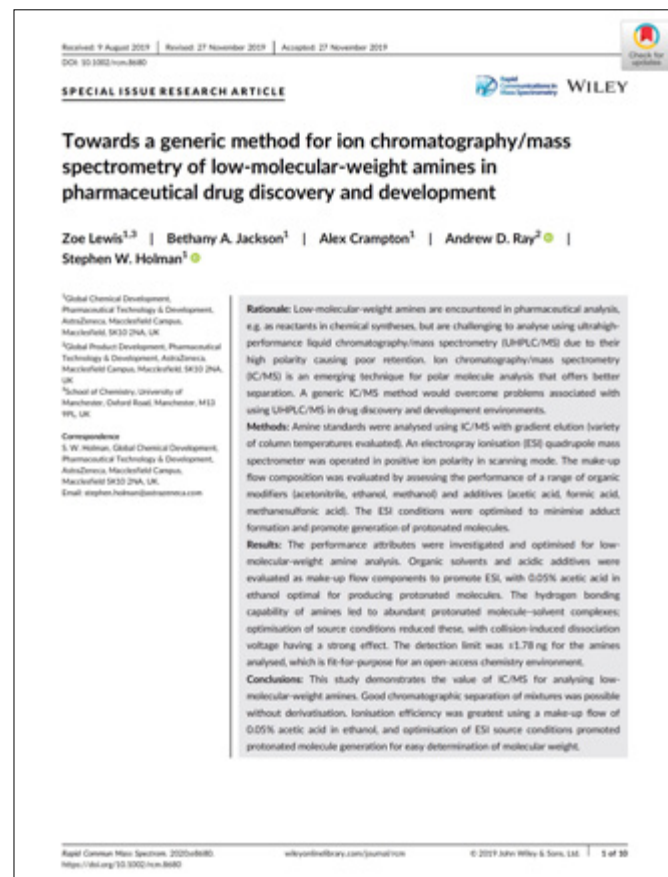
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Towards a generic method for IC–MS of low-molecular-weight amines in pharmaceutical drug discovery and development

Overview

This study demonstrates the value of IC/MS for analysing low molecular-weight amines. Good chromatographic separation of mixtures was possible without derivatisation. Ionisation efficiency was greatest using a make-up flow of 0.05% acetic acid in ethanol, and optimisation of ESI source conditions promoted protonated molecule generation for easy determination of molecular weight.



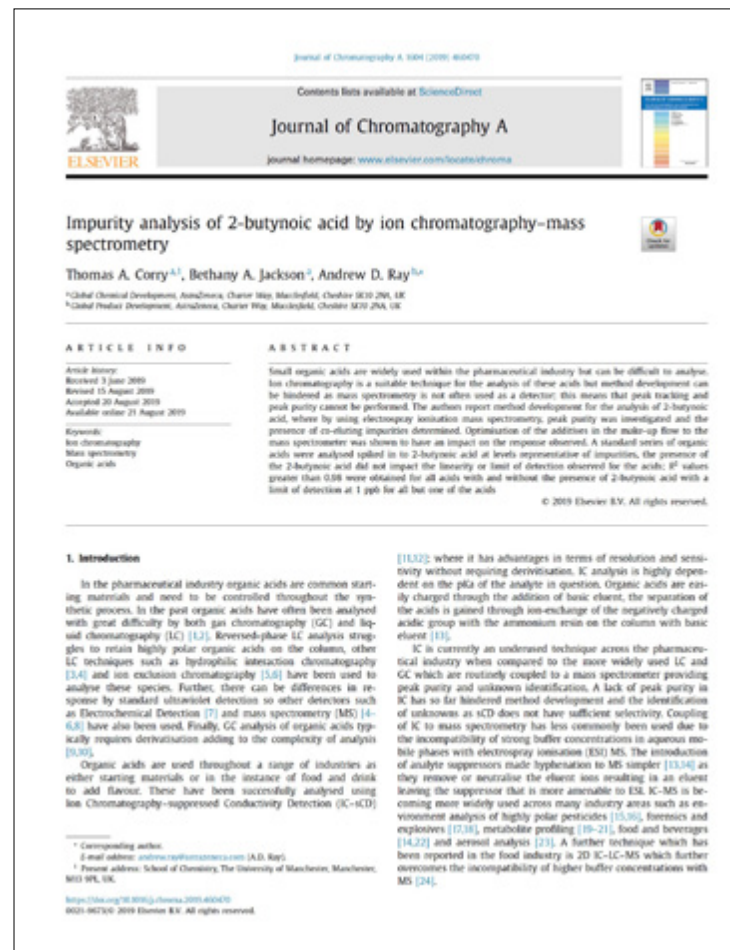
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Impurity analysis of 2-butynoic acid by IC-MS

Overview

Small organic acids are widely used within the pharmaceutical industry but can be difficult to analyse. Ion chromatography is a suitable technique for the analysis of these acids but method development can be hindered as mass spectrometry is not often used as a detector; this means that peak tracking and peak purity cannot be performed. The authors report method development for the analysis of 2-butynoic acid, where by using electrospray ionisation mass spectrometry, peak purity was investigated and the presence of co-eluting impurities determined. Optimisation of the additives in the make-up flow to the mass spectrometer was shown to have an impact on the response observed. A standard series of organic acids were analysed spiked into 2-butynoic acid at levels representative of impurities. The presence of the 2-butynoic acid did not impact the linearity or limit of detection observed for the acids; r^2 values greater than 0.98 were obtained for all acids with and without the presence of 2-butynoic acid with a limit of detection at 1 ppb for all but one of the acids.



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IC-MS for the determination of organic acids in pharmaceutical solutions

Overview

This study illustrates the use of a simplified IC-MS method to facilitate the trace amount determination of highly polar, low molecular weight aliphatic and unsaturated organic acids in pharmaceutical solutions. By improving the identification and quantification of these organic acids, pharmaceutical companies can overcome significant challenges in impurity monitoring.

In this workflow, the Dionex IonPac AS11-HC-4 μ m column supports a highly selective separation of the analytes in question. The high capacity of the column ensures the matrix tolerance needed to determine low concentrations of organic acids, i.e. below 0.1% relative to the chosen 2-butynoic acid matrix. The elevated column temperature (40 °C) improves chromatographic peak efficiencies and prevents using an organic solvent in the mobile phase. The use of continuously regenerated membrane-based suppressors allows the hyphenation of IC with MS, further adding to the analytical selectivity and sensitivity.

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APPLICATION NOTE 73344

IC-MS for the determination of organic acids in pharmaceutical solutions

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Keywords: Ion chromatography, IC,
suppression, aliphatic and unsaturated, RPLC,
Dionex IonPac AS11-HC-4 μ m, ISQ EC single
quadrupole mass spectrometer

Goal
To develop a simplified ion chromatography (IC) application
in conjunction with mass-selective detection to facilitate the
identification and quantification of highly polar aliphatic and
unsaturated organic acids in pharmaceutical solutions

Introduction
High-performance liquid chromatography (HPLC) and
IC are used for a wide range of applications across the
pharmaceutical industry. In drug discovery, for example,
these methods are used to screen drug candidates,
either as standalone tools or in conjunction with mass
spectrometers. HPLC and IC workflows are also used in
pre-clinical development to analyze in vitro and in vivo
samples, and in clinical trials to gather data on drug safety
and efficacy. These techniques also play an essential role
in pharmaceutical manufacturing, including the collection
of quality assurance/quality control (QA/QC) data and the
validation of cleaning procedures.¹

One of the many challenging tasks faced by
pharmaceutical companies is the determination of



short-chained aliphatic and unsaturated organic acids
during development and manufacture. This is necessary
when assessing impurities in a starting material (educt),
determining breakdown products in formulation stability
monitoring, or controlling the cleaning of production lines.
Because these short-chained, low molecular weight
organic acids are hydrophilic and very polar in nature, their
identification and quantification are complicated at trace
levels.

There is a range of analytical techniques available to
quantify organic acids. Ion pair chromatography, ion
suppression chromatography, and anion exchange
chromatography with weak anion-exchange columns
are often used in combination with UV-detection.²⁻⁴
However, these approaches do not offer sufficient
sensitivity and selectivity for the trace-level detection

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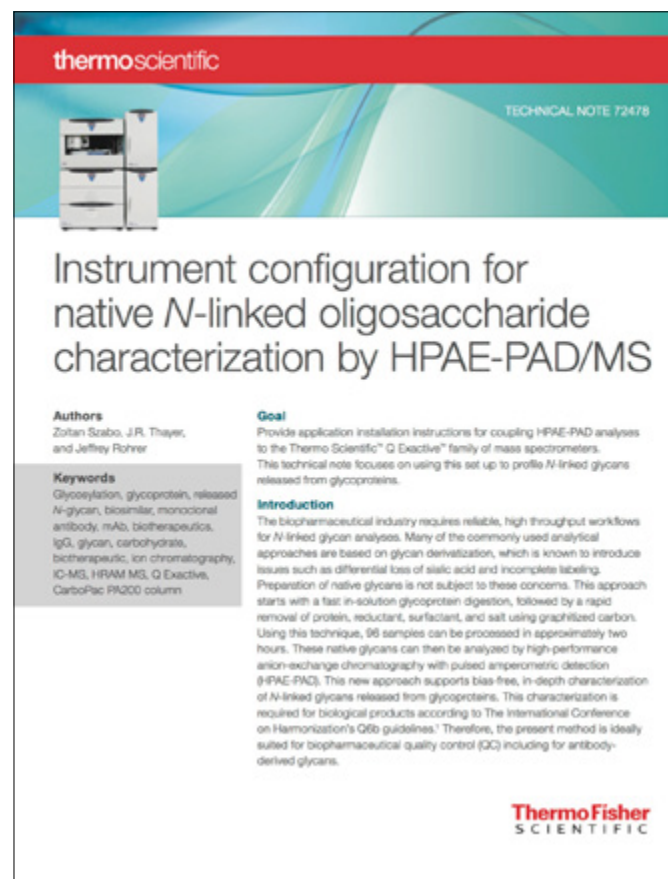




Instrument configuration for native *N*-linked oligosaccharide characterization by HPAE-PAD/MS

Overview

Separation by HPAE is based on charge, linkage and positional isomerism, and fucosylation, resulting in excellent separation of many different glycan isomers. Using this new workflow, 53 unique *N*-glycans were identified from hu-AGP, and *N*-glycans from three different relevant mAbs were annotated, showing many common and several individual features. Adduct formation with acetate enhances MS fragmentation, producing diagnostic ions that support structural elucidation of neutral *N*-glycans. The conditions employed for desalter regeneration deliver reproducible removal of sodium ions. The desalter does not materially degrade glycan resolution between the column and the downstream MS probe. HPAE-PAD/MS is an excellent tool to profile and annotate complex mixtures of native *N*-glycans released from different glycoproteins and provides a different selectivity than other glycan separation techniques that have been coupled to MS.



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Improved profiling of sialylated *N*-linked glycans by ion chromatography-Orbitrap mass spectrometry

Overview

Excellent separation of many different glycans based on their charge, isomerism, fucosylation, and sialylation was achieved using HPAE. This work presents an improved approach for further increasing coverage in glycan analysis using HPAE-PAD coupled to HRAM mass spectrometry. The effect of increased hydroxide concentration and temperature on glycan resolution was studied. The change in hydroxide concentration and column temperature can be exploited for increasing glycan coverage. We find that higher hydroxide concentration leads to improved overall resolution resulting from improved separation of glycan isomers. Lower hydroxide concentration helps resolve certain structural features such as fucosylation.



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APPLICATION NOTE 73063

Improved profiling of sialylated *N*-linked glycans by ion chromatography-Orbitrap mass spectrometry

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Keywords
HPAE-PAD, glycoprotein,
sialylated glycans, Q Exactive,
HPAE-MS, HPLC, CarboPac
PA200 column

Introduction
Protein glycosylation is a key intracellular process that involves interactions of various enzymes and substrates.^{1,2} Glycoproteins are central to several important biological processes. Carbohydrates, especially those linked through an asparagine side chain (*N*-linked), play a critical role in these biological activities. Due to increased understanding of the biological significance of carbohydrates, an increasing number of glycoprotein therapeutics are being developed as treatments for various pathological conditions.

Goal
To describe an HPAE-MS method for improved glycan profiling

Manufacturing glycoproteins in high volume is complex, not only due to the nature of the molecules, but also because it involves a cell-based process. Protein glycosylation is heavily dependent on the manufacturing process. Various factors that influence protein glycosylation include cell type, cell age, and cell culture conditions. Maintaining batch-to-batch consistency of glycoprotein therapeutics requires close attention to quality control. Hence, there is an increased demand for methods to characterize glycoprotein carbohydrates.

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O-glycan profiling using HPAE-PAD hyphenated with a high-resolution accurate mass (HRAM) mass spectrometer

Overview

Glycosylation is an important post-translational modification (PTM) that occurs on nitrogen and oxygen atoms of amino acid residues of proteins. Glycosylation plays an essential role in physiological and pathological cellular functions. Atypical and abnormal glycosylation is linked with different diseases, e.g., cancer, rheumatoid arthritis, diabetes, and cardiac diseases. Thus, it is important to characterize protein glycosylation in biological and clinical specimens. It has been estimated recently that more than 50% of all proteins are glycosylated.

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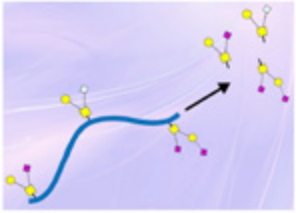
O-glycan profiling using HPAE-PAD hyphenated with a high-resolution accurate mass (HRAM) mass spectrometer

Authors: Manali Aggrawal, Sachin Patil, and Jeffrey Rohrer
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Keywords: Dionex ICS-5000[®], Dionex ICS-6000, Dionex CarboPac PA300 column, Orbitrap, pulsed amperometric detection, Q Exactive, bovine fetuin, bovine thyroglobulin, bovine fibrinogen, mucin

Goal
To evaluate and demonstrate an HPAE-PAD/MS workflow for profiling O-glycans from four different glycoproteins

Introduction
Glycosylation is an important post-translational modification (PTM) that occurs on nitrogen and oxygen atoms of amino acid residues of proteins. Glycosylation plays an essential role in physiological and pathological cellular functions. Atypical and abnormal glycosylation is linked with different diseases, e.g., cancer, rheumatoid arthritis, diabetes, and cardiac diseases.^{1,2} Thus, it is important to characterize protein glycosylation in biological and clinical specimens. It has been estimated recently that more than 50% of all proteins are glycosylated.¹⁻⁴ Two main types of protein



glycosylation have been distinguished: N-glycosylation and O-glycosylation. N-glycosylation is the most abundant PTM in which oligosaccharides (glycans) are attached via an N-glycosidic bond to asparagine (Asn) in the consensus sequence Asn-Xaa-Ser/Thr (Ser, serine; Thr, threonine; Xaa, any amino acid except proline). O-glycosylation is another type of protein glycosylation. For O-glycosylation, the oligosaccharide is linked via an oxygen of serine (Ser) or threonine (Thr). In humans, the most common sugars linked to Ser or Thr are N-acetylglucosamine (GlcNAc) and N-acetylgalactosamine (GalNAc).⁵

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Primary metabolite analysis of plant material using a triple quadrupole MS coupled to a monolith anion-exchange column

Overview

The combination of the 1 mm Thermo Scientific™ Dionex™ IonSwift™ MAX-100 Anion-Exchange column and the API 4000 ESI-MS/MS system provides a simple, rapid, specific, and sensitive method for the simultaneous detection and quantification of several primary metabolites involved in the TCA cycle and glycolysis pathway. Compared to the previously used Dionex IonPac AS11-HC column, the MAX-100 has shorter run times and helps achieve better sensitivity.

Customer Application Note

Primary Metabolite Analysis of Plant Material Using a Triple Quadrupole MS Coupled to a Monolith Anion-Exchange Column

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Introduction
A platform using Dionex ion chromatography (IC) coupled to triple quadrupole mass spectrometry (MS) was developed to measure various metabolites in *Brassica napus* seeds. The IC tandem mass spectrometry (IC-MS/MS) technique provides a good tool to identify and quantify metabolites in a complex matrix. The biological pathways include many homologs and structural isomers with the same mass and similar fragmentations, so it is also essential for the analysis to have a good separation of the different compounds. The new IonSwift™ MAX-100 column (2 × 250 mm) provides good peak shape and separation for a broad spectrum of substances. Comparison with the previous product, the IonPac® AS11-HC column (2 × 250 mm), showed a similar separation in a shorter run time and an enhanced sensitivity for several target analytes.

Equipment
Dionex IC-3000 System including:
EG Eluent Generator
DP Dual Pump with Degasser
AS Autosampler
DC Detector Compartment with Anion Self-Regenerating Suppressor (ASRS®) 300 2 mm Suppressor
API 4000™ Liquid Chromatography (LC)-MS/MS System (AB SCIEX)

Reagents and Standards
Methanol, chloroform, and other biochemicals were obtained from Sigma-Aldrich Chemie GmbH (Taufkirchen, Germany) and Roche Diagnostics GmbH (Mannheim, Germany). Water was purified with the Millipore Milli-Q® Plus System.

Preparation of Samples
Homogenize approximately 30 mg frozen material at liquid nitrogen temperature in 2 mL microcentrifuge tubes (Eppendorf®, Hamburg, Germany) containing two steel beads (AS6, Kontes-Mueninghen, Germany) by grinding in a TissueLyser (Qiagen GmbH, Hilden, Germany) for 45 s at 3000 strokes. After homogenization, extract the samples with 0.5 mL 1:1 (v/v) methanol/chloroform under the same grinding conditions. Extract water-soluble compounds from the CHCl₃ phase by adding 300 µL water, followed by 45 s shaking and centrifugation at 2000 g for 2 min. Filter the upper aqueous/methanol phase with a Vivaclear centrifugal filter (5 µm pore size, SartoriusStedim Biotech, Göttingen, Germany) at 2000 g for 2 min. Re-extract the lower CHCl₃ phase with 200 µL water and add the aqueous/methanol phase to the first phase. Evaporate the extract to dryness using a centrifugal vacuum dryer at 20 °C and redissolve in 1 mL water.

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