

Speciation Applications Summary Ion Chromatography

Elemental Species Separation and Detection



Table of contents

Introduction

Speciation Analysis

Complete inorganic analysis

Periodic Table of Total Inorganic Analysis	4
Arsenic	
IC-ICP-MS speciation analysis of As in apple juice using the Thermo Scientific iCAP Q ICP-MS	5
ICP-ICP-MS speciation analysis of As in organic brown rice syrup (ORBS) using the Thermo Scientific iCAP Q ICP-MS	6
Arsenic speciation in human urine by hyphenated ion chromatography (IC) and inductively coupled plasma mass spectrometry (ICP-MS)	7
Determination of inorganic arsenic in rice using IC-ICP-MS	8
Bromine	
Speciation of bromine compounds in ozonated drinking water using ion chromatography and	
inductively coupled plasma mass spectrometry	9
Chromium	
Speciation analysis of Cr(III) and Cr(VI) in drinking waters using anion exchange chromatography	
coupled to the Thermo Scientific iCAP Q ICP-MS	10
The migration of elements from toys and speciation of chromium (VI) in toy material using a low cost IC-ICP-MS Solution	11
lodide	
Determination of iodide and iodate in sov- and milk-based infant formulas	12

З

Mercury

Total and speciation analysis of mercury in contact lens solutions by ICP-MS	14				
Total and speciation analysis of mercury in contact lens solutions by ICP-MS					
Innovative analytical technologies	15				

Introduction



Speciation Analysis

Distinguishing between chemical forms of an element is critical for multiple industries, including the food, environmental, and pharmaceutical sectors. In the past, measuring the total amount of an element was sufficient. Unfortunately, the effects of an element extend far beyond its absolute amount. Different forms of an element can exhibit very different physiochemical properties, including varying toxicities. The process of separation and quantification of different chemical forms of an element, more specifically termed speciation analysis, delivers a better understanding of the environmental or health-related impact associated with a particular sample. Speciation analysis can be split into two components: separation of individual ionic species by ion chromatography (IC), and trace elemental detection and quantification using inductively coupled plasma mass spectrometry

(ICP-MS). This combined method is termed ion chromatographyinductively coupled plasma mass spectrometry (IC-ICP-MS).

Benefits of IC-ICP-MS

- Metal-free IC separates the individual species without contributing trace metal contamination
- IC-ICP-MS acts as a highly sensitive and element-specific detection system

The process of IC allows ions and polar molecules to be separated on the basis of their charge, size, and polarizability. This specific method determines ionic species mainly using a conductivity detector, but can also be used with other types of detectors. With its metal-free fluidic flow path, IC is ideally suited to elemental speciation analysis. Furthermore, the system can determine a range of compounds, from anionic to cationic contaminants, many of which are toxic and regulated.

The concentrations of these compounds are important quality indicators for environmental waters, pharmaceuticals, and food. ICP-MS is a multi-element spectrometry method that determines total elemental concentrations without bias towards metal species. The technique provides rapid and robust total element concentration determinations in various types of samples with high sensitivity (sub-part per trillion [ppt] detection). Combining an ICP ion source with a mass spectrometer (either quadrupolebased or utilizing magnetic and electrostatic sectors), an ICP-MS can detect almost any element in the periodic table in many different sample matrices. The ICP is a high temperature source that decomposes and atomizes molecules, then ionizes the atoms. The mass spectrometer separates and detects these ions.



The Thermo Scientific iCAP RQ with the Thermo Scientific Dionex IC family of instruments

Complete inorganic analysis



More Capabilities Together



Periodic Table of Total Inorganic Analysis

H																	He
li	Be											В	C	N	0	F	Ne
Na	Mg											AI	Si	P	S	CI	Ar
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	ľ	Xe
Cs	Ba	La	Hf	Ta	W	Re	Os	T	Pt	Au	Hg	=	Pb	Bi	Po	At	Rn
Fr	Ra	Ac															
				Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu
				Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	ŀ

IC-ICP-MS speciation analysis of As in apple juice using the Thermo Scientific iCAP Q ICP-MS



Introduction

Recent media reports in the US have claimed that some apple juices may contain high levels of arsenic. However, in these determination studies, only the total arsenic concentration was assessed; no detailed investigation of the chemical form of the element was carried out. This is an important distinction since the inorganic forms of arsenic (As (III) and As (V)) are highly toxic, while the organic forms (e.g. arsenobetaine) are not considered to be toxic. Typical levels of total arsenic found in apple juice are lower than the U.S. EPA drinking maximum contaminant level (MCL) of 10 ng/g so apple juice is generally considered safe and is currently not regulated. However, as a consequence, the FDA is currently reviewing data, and may eventually lower its current guidelines.

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Arsenic Speciation in Apple Juice

Equipment

- Dionex ICS-5000 IC System*
- Dionex IonPac AS7 column, 2 mm i.d. × 250 mm
- iCAP Q ICP-MS *Dionex ICS-5000* HPIC system can be used for equivalent results

iCAP Q Operating Parameters					
Forward Power:	1550 W				
Nebulizer Gas:	0.80 L/min				
Injector:	2 mm I.D., quartz				
Interface	Ni sampler and skimmer				
QCell He Gas Flow:	4.8 mL/min				
QCell KED:	2 V				
75As Dwell Time:	100 ms				

Dionex ICS-5000 IC Operating Parameters					
Elution:	Gradient				
Mobile Phase:	A: 20 mmol/L Ammonium carbonate B: 200 mmol/L Ammonium carbonate				
Injection Volume:	20 µL				
Duration:	15 min				

Analysis IC-ICP-MS

Results See tables below.

As species concentrations, method detection limits (MDLs) and total arsenic concentrations in two of the apple juice samples analyzed

	AsB	DMA	As ³⁺	AsC	MMA	As ⁵⁺	Sum of Total Species	Total As
MDL	0.002	0.004	0.005	0.004	0.011	0.001	-	0.005
Juice 3	ND	ND	0.5 ± 0.01	ND	ND	0.7 ± 0.01	1.2	1.7 ± 0.05
Juice 4	ND	0.4 ± 0.05	0.3 ± 0.01	ND	0.1 ± 0.05	0.7 ± 0.01	1.5	1.8 ± 0.05

All concentrations have units of ng/g. ND indicates not detected.

Spike recovery for six arsenic species in apple juice

Species	Expected (ng/g)	Found (ng/g)	Recovery (%)
AsB	2.19	2.27	104
DMA	1.40	1.15	82
As (III)	1.35	1.38	102
AsC	1.94	1.87	94
MMA	1.09	1.13	104
As (V)	1.10	1.07	98

IC-ICP-MS speciation analysis of As in organic brown rice syrup (ORBS) using the Thermo Scientific iCAP Q ICP-MS



Introduction

Media reports and scientific publications on the determination of arsenic (As) in foodstuffs have sparked renewed interest from consumer groups and politicians leading to responses from national regulatory bodies. Based on recent reports, the FDA began carrying out a study on As in rice and rice products, including organic brown rice syrup, OBRS, an ingredient in a variety of organic foods. In this study, OBRS samples were analyzed for their total arsenic content by ICP-MS and then subsequently by IC-ICP-MS to determine the concentration of six arsenic species: the two toxic inorganic species As (III) and As (V), and four organic species that are considered harmless.

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Arsenic Speciation in Organic Brown Rice Syrup

Equipment

- Dionex ICS-5000 IC System*
- Dionex IonPac AS7 column, 2 mm i.d. × 250 mm
- iCAP Q ICP-MS**
- *Dionex ICS-5000+ HPIC system can be used for
- equivalent results **iCAP RQ ICP-MS can be used for equivalent results

iCAP Q Operating Parameters				
Forward Power:	1550 W			
Nebulizer Gas:	0.80 L/min			
Injector:	2 mm I.D., quartz			
Interface	Ni sampler and skimmer			
QCell He Gas Flow:	4.8 mL/min			
QCell KED:	2 V			
75As Dwell Time:	100 ms			

Dionex ICS-5000 IC Operating Parameters Elution: Gradient Mobile Phase: A: 20 mmol/L Ammonium carbonate B: 200 mmol/L Ammonium carbonate Injection Volume: 20 μL Duration: 15 min

Analysis

IC-ICP-MS

Results See table below.

Total As concentrations of >100 ng/g were found in three analyzed samples.

	⁷⁵ As (ng/g)
OBRS Sample #1	118 ± 7
OBRS Sample #2	136 ± 7
OBRS Sample #3	107 ± 11



IC-ICP-MS chromatogram of arsenic species found in ORBS sample. As (III) was the most abundant species detected.

Arsenic speciation in human urine by hyphenated ion chromatography (IC) and inductively coupled plasma mass spectrometry (ICP-MS)



Introduction

The different As species can be classified as inorganic arsenic (iAs) and organic arsenic compounds. Exposure to iAs as the sum of arsenite As (III) and arsenate As(V) is a major concern for public health authorities worldwide. The need for As speciation is important because the concentration of As may be interpreted in a riskassessment context differently if As arises from exposure to iAs species (As (III) and As (V)), methylated As (MMA and DMA) or organic As (AsBet and AsChol). The measurement of the total As concentration (which comprises both inorganic and organic As) cannot be used solely to explain the biological and toxic effects of As and judge potential hazards. Thus, the determination of the individual concentration of each As species, by using the speciation approach, becomes critical. The aim of this work is to develop and validate a method for the simultaneous separation and quantification of six different As species - namely AsBet, DMA, As(III), AsChol, MMA and As(V) – in human urine samples.

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Arsenic Speciation in Human Urine

Equipment

- Dionex ICS-5000 IC System*
- Dionex IonPac AG7 column, 4mm i.d. × 50 mm, Dionex IonPac AS7 column, 4 mm i.d. × 250 mm
- iCAP Q ICP-MS**
- *Dionex ICS-5000* HPIC system can be used for equivalent results
- **iCAP RQ ICP-MS can be used for equivalent results

iCAP Q Operating Parameters

Interface:	Ni cones with High Matrix (3.5 mm) insert			
QCell He Gas Flow:	4.8 mL/min			
Nebulizer and Spray Chamber:	Quartz cyclonic spray chamber			
Forward Power:	1550 W			
Nebulizer Flow Rate:	1.17 L/min			

Concentrations in µg/L of the six As species after the chromatographic separation.

Subject	AsBet	DMA	As(III)	AsChol	ММА	As(V)
1	73.1	4.44	0.26	0.51	0.75	<lod< td=""></lod<>
2	27.3	1.75	<lod< td=""><td><lod< td=""><td>0.53</td><td><lod< td=""></lod<></td></lod<></td></lod<>	<lod< td=""><td>0.53</td><td><lod< td=""></lod<></td></lod<>	0.53	<lod< td=""></lod<>
3	66.6	0.40	<lod< td=""><td><lod< td=""><td>0.38</td><td><lod< td=""></lod<></td></lod<></td></lod<>	<lod< td=""><td>0.38</td><td><lod< td=""></lod<></td></lod<>	0.38	<lod< td=""></lod<>
4	22.4	5.50	0.47	<lod< td=""><td>0.50</td><td>0.75</td></lod<>	0.50	0.75
5	11.4	2.66	<lod< td=""><td><lod< td=""><td>0.52</td><td><lod< td=""></lod<></td></lod<></td></lod<>	<lod< td=""><td>0.52</td><td><lod< td=""></lod<></td></lod<>	0.52	<lod< td=""></lod<>
6	27.2	3.33	0.90	0.32	0.79	0.37
7	243	3.65	<lod< td=""><td>0.37</td><td>0.52</td><td><lod< td=""></lod<></td></lod<>	0.37	0.52	<lod< td=""></lod<>
8	769	4.30	0.41	0.31	1.71	<lod< td=""></lod<>
9	17.8	1.11	<lod< td=""><td>0.34</td><td>0.41</td><td><lod< td=""></lod<></td></lod<>	0.34	0.41	<lod< td=""></lod<>
10	188	3.44	<lod< td=""><td>1.20</td><td>0.39</td><td><lod< td=""></lod<></td></lod<>	1.20	0.39	<lod< td=""></lod<>



Dionex ICS-5000 IC Operating Parameters					
Elution:	Gradient				
Mobile Phase:	A: Water B: 100 mmol/L Ammonium carbonate - 3% methanol, pH 10.3				

Injection Volume:	100 μL
Flow Rate:	1.0 mL/min
Duration:	14 min

Analysis

IC-ICP-MS

Results

See table and figure below.

Determination of inorganic arsenic in rice using IC-ICP-MS

Introduction

The ability of rice to accumulate relatively large quantities of arsenic in its grain is well established. Generally, rice contains dimethylarsenic acid (DMA), small amounts of monomethylarsonic acid (MMA) and inorganic arsenic (iAs). The arsenic content and the species present depend on the rice variety, soil and growth conditions. Whereas inorganic arsenic is highly toxic and carcinogenic, DMA is far less toxic and the main arsenic containing compound in fish, arsenobetaine, is considered to be non-toxic. One of the most efficient methods for identification and quantification of inorganic arsenic is the combination of strong-anion-exchange chromatography for separation with ICP-MS for specific arsenic detection. This Application Note describes an IC-ICP-MS method that is well suited for the determination of inorganic arsenic in rice and rice products with excellent detection limits and stability of retention time.

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Inorganic Arsenic in Rice

Equipment

- Dionex ICS-5000 IC System*
- Dionex IonPac AS7 column, 2 mm i.d. × 250 mm
- iCAP Q ICP-MS**
- *Dionex ICS-5000* HPIC system can be used for equivalent results
- **iCAP RQ ICP-MS can be used for equivalent results

ICAP Q Operating ParametersInterface:Ni sampler and skimmerQCell He Gas Flow:4.5 mL/minNebulizer and SprayMicromist and Quartz cyclonic spray chamberChamber:Micromist and Quartz cyclonic spray chamberForward Power:1550 WNebulizer Flow Rate:1.3 L/min

Dionex ICS-5000 IC Operating Parameters		
Elution:	Gradient	
Mobile Phase:	A: Water B: 200 mmol/L Ammonium carbonate	
Injection Volume:	25 μL	
Flow Rate:	0.5 mL/min	
Duration:	10 min	

Analysis
IC-ICP-MS

Results

See table and figure below.

Results for Limit of Detection (LOD), Limit of Quantification (LOQ), Background Equivalent Concentration (BEC), Certified Reference Material (CRM) and spike recovery.

	DMA	ММА	iAs
LOD (µg·kg ⁻¹ solution)	0.030 ± 0.017		0.11 ± 0.039
LOD (µg·kg ⁻¹ rice) incl. DL of 90	2.7 ± 1.5		10 ± 3.6
BEC ($\mu g \cdot k g^{-1}$ solution, from program)	0.019 ± 0.0040	0.0047 ± 0.0035	0.085 ± 0.0025
NIST 1568a (n=6)	181 ± 7.9	11 ± 4.9	118 ± 11.2
Literature	143–185		80–115
BCR 211 (n=8)	118 ± 3.6	14 ± 0.91	122 ± 2.1
BCR-211 (certified)	119 ± 13		124 ± 11
Spike Recovery % (n=6)	96 ± 3.2	103 ± 2.0	104 ± 3.4



Example chromatogram showing automatic identification and peak area determination for As-species.

Speciation of bromine compounds in ozonated drinking water using ion chromatography and inductively coupled plasma mass spectrometry



Introduction

Bromine speciation in drinking water is required worldwide by major regulatory bodies. A maximum contaminant level (MCL) of 10 μ g/L in the US for bottled drinking water and in the EU of 3 µg/L for natural mineral water and spring water treated with ozonation are stipulated for the bromate anion, which is toxic and carcinogenic. Differentiating bromate from bromide (relatively non-toxic) is therefore important due to the toxicity differences between the two species.

U.S. EPA Method 321.8 provides an analytical procedure for bromate determination in drinking water using IC-ICP-MS. In this Application Note, a Thermo Scientific[™] Dionex[™] ICS-5000 IC is coupled to a Thermo Scientific[™] iCAP[™] RQ ICP-MS to determine bromine species in drinking water.

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Bromate in Drinking Water

Equipment

- Dionex ICS-5000 IC System*
- Dionex IonPac AS19 column,
 2 mm i.d. × 250 mm, Dionex IonPac AG19 column, 2 mm i.d. × 50 mm
- iCAP RQ ICP-MS
- *Dionex ICS-5000* HPIC system can be used forequivalent results

iCAP RQ Operating Parameters		
QCell He Gas Flow:	4.5 mL/min	
Cool Gas:	14 L/min	
Auxiliary Flow:	0.8 L/min	
Nebulizer:	1.13 L/min	
Spray Chamber:	2.6 °C	
Forward Power:	1550 W	
Nebulizer Flow Rate:	1.3 L/min	
Injector:	2 mm i.d.	
KED Voltage:	2.5 V	

Elution: 10 mM KOH from 0–25 min,

Dionex ICS-5000 IC Operating Parameters

EIUTIOU	10 IIIWI KUTI II0III 0–23 IIIIII,
	45 mM from 25–30 min,
	10 mM from 35 min*
Injection Volume:	100 µL
Flow Rate:	0.3 mL/min
Duration:	35 min

Analysis

IC-ICP-MS

Results

See tables below.

Results for bromate in KED mode of the six water samples (spiked and unspiked).

Water	Average Unspiked Sample Result (µg·L ⁻¹)	Samples Spiked with 1 µg·L ⁻¹ Bromate (µg·L ⁻¹)	Recovery %
1	N/A	0.999	99.9
2	1.218	2.221	100.3
3	1.732	2.729	99.7
4	N/A	1.000	100.0
5	N/A	0.999	99.9
6	1.127	2.124	99.7



Bromate at EPA recommended method detection limit (MDL) of 0.3 μ g/L⁻¹.

Speciation analysis of Cr (III) and Cr (VI) in drinking waters using anion exchange chromatography coupled to the Thermo Scientific iCAP Q ICP-MS



Introduction

Both the United States EPA and the European Union have specified maximum admissible chromium concentrations in their respective drinking water directives. As with many other trace elements, chromium (Cr) is typically found in more than one chemical form, each of which with different chemical properties and behavior, such as bioavailability and toxicity. For chromium, Cr (III) is essential to human beings and involved in different processes in the body while Cr (VI) is highly toxic. Total Cr content therefore in, for example, a drinking water sample does not provide sufficient information to evaluate potential hazards to populations exposed to it. In order to provide this critical information a supporting speciation analysis is required to determine the amounts of the different Cr species in the sample.

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Chromium Speciation in Drinking Water

Equipment

- Dionex ICS-5000*
- Dionex IonPac AS7 column, 2 mm i.d. × 250 mm
- iCAP Q ICP-MS**
- *Dionex ICS-5000+ HPIC system can be used for equivalent results
- **iCAP RQ ICP-MS can be used for equivalent results

iCAP Q Operating Parameters**		
Forward Power:	1550 W	
Nebulizer Gas:	0.80 L/min	
Injector:	2 mm I.D., quartz	
Interface:	Ni sampler and skimmer	
QCell He Gas Flow:	4.8 mL/min	
QCell KED:	2 V	
Dwell Time:	100 ms	

Dionex ICS-5000 IC System Operating Parameters** Elution: Isocratic Mobile Phase: 0.4 mol/L HNO₃ Flow Rate: 400 μL/min Injection Volume: 20 μL Duration: 150 s **Operating parameters used in analysis based on configurations in

**Operating parameters used in analysis based on configurations in AN 43126

Analysis IC-ICP-MS

Results

See table and figures below.



Cr(III) and Cr(VI) chromatograms obtained using 0.2 (A), 0.3 (B) and 0.4 (C) mol/L nitric acid as mobile phase. Note that the x-axis in (C) has been shortened to 300 s.

Recovery of Cr (VI) and (III) species from drinking water samples

Cono onikod (ng/g)	Cr (VI)		Cr (III)	
conc. spikeu (iig/g)	Found (ng/g)	Recovery (%)	Found (ng/g)	Recovery (%)
2.34 of each	2.31 ± 0.01	99 ± 1	2.35 ± 0.02	100 ± 1
6.03 Cr (VI); 1.90 Cr (III)	6.01 ± 0.02	100 ± 1	2.00 ± 0.01	105 ± 1
1.87 Cr (VI); 6.20 Cr (III)	1.85 ± 0.01	99 ± 1	6.15 ± 0.03	99 ± 1

The migration of elements from toys and speciation of chromium (VI) in toy material using a low cost **IC-ICP-MS** solution



Introduction

Hexavalent chromium (Cr (VI)) is highly toxic and classed as a carcinogen. Trivalent chromium (Cr (III)), however, is essential for enzymatic function in human metabolism. Discrimination between oxidation states is therefore essential for an accurate risk assessment of chromium in materials we are exposed to.

The European Union directive for toy safety (2009/48/EC) mandates limits for Cr (VI) content that are analytically challenging with commonly used methods such as High Pressure Liquid Chromatography (HPLC) coupled to Inductively Coupled Plasma Mass Spectrometry (ICP-MS). In this Application Note, the use of Ion Chromatography (IC) is shown to be a powerful alternative to HPLC, allowing the analysis of both Cr species in the low ng L-1 range. A method is presented for total elemental quantification (TEQ) and stabilization and quantification of Cr (VI) after migration from toy material at European Union 2009/48/EC directive required levels.

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Chromium (VI) in Toys

Equipment

- Dionex ICS-900 IC System*
- Dionex NG1 column, 2 mm i.d. × 50 mm, IonPac AG7 column, 2 mm × 50 mm
- iCAP RQ ICP-MS
- *Dionex Aquion IC system can be used for equivalent or improved results

iCAP Q Operating Parameters

Forward Power:	1550 W
Nebulilzer:	Microflow PFA-ST for elemental analysis and PFA-LC for speciation
Nebulizer Gas:	0.99 L/min
Nebulizer:	1.13 L/min
QCell He Gas Flow:	4.4 mL/min
QCell KED:	3 V

exavalent Chromium concentration found in the various toy samples.					
Sample	Amount spiked [ng mL ⁻¹]	Found Injection 1 [ng mL-1]	Found Injection 2 [ng mL-1]	Found Injection 3 [ng mL-1]	Average Recovery [%]
Finger paint	0.27	0.26	0.30	0.34	110 ± 15
TiO ₂	0.27	0.24	0.25	0.26	92 ± 4
lnk 1	0.27	0.23	0.27	0.25	88 ± 3
Ink 2	0.10	0.09	0.10	0.09	87 ± 4



Chromatogram of an ink sample.

Dionex ICS-5000 IC Operating Parameters		
Elution:	0.35 mol/L HNO ₃	
Injection Volume:	10 µL	
Flow Rate:	0.4 mL/min	
Duration:	5 min	

Analysis

IC-ICP-MS

Results

See table and figure below.

Determination of iodide and iodate in soy- and milk-based infant formulas

App



Introduction

Accurate measurement of iodine in food matrices requires a robust iodine extraction method and a sensitive analytical method for iodine quantification. This application note includes the acetic acid digestion method for iodide extraction, coupled with an IC-Pulsed Amperometric Detection (PAD) method for iodide detection first developed in an archived version of this application note. The IC method coupled with electrochemical detection allows for selective and sensitive determination of iodide in complex matrices. The acid digestion procedure to extract iodide was optimized for milk- and soy-based infant formulas. In addition, sample preparation conditions to convert iodate to iodide for determining total iodine (i.e., iodide and iodate) are presented.

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Iodide and Iodate Speciation in Infant Formula

Equipment

• Dionex ICS-5000 IC system* including: Gradient pump

DC Detector Chromatography Compartment

ED Electrochemical Detector without cell (P/N 079830)

ED Electrode, Ag, with gasket and polishing kit (P/N 079856)

Ag/AgCl reference electrode (P/N 061879)

Dionex AS or AS-AP Autosampler

- Dionex IonPac AG11 guard, 4 × 50 mm (P/N 44078)
- Dionex IonPac AS11 analytical, 4 × 250 mm (P/N 44076)
- Thermo Scientific[™] Dionex[™] OnGuard[™] II RP Cartridges, 2.5 cc (P/N 057084)
- EO Eluent Organizer with two 2 L plastic bottles and pressure regulator
- Vial Kit, 0.3 mL polyprop with caps and septa (P/N 055428)
- Micro Tubes 1.5 mL, type D, without skirted base, screw cap assembled, sterile (Sarstedt[™] P/N 72.692.005 or equivalent)
- Thermo Scientific[™] Nalgene[™] narrow-mouth bottle, HDPE/PP, 1000 mL (P/N 2002-0032)
- Nalgene polystyrene lab filter unit, 500 mL upper capacity with 1000 mL receiver capacity 0.2 micron, 75 mm membrane diameter (P/N 154-0020)
- *Dionex ICS-5000* HPIC system can be used for equivalent results



Reagents and Standards

- Deionized (DI) water, type I reagent grade, 18 MΩ-cm resistivity or better filtered through a 0.2 µm filter immediately before use
- Nitric acid
- Sodium iodide
- Sodium iodate
- Ascorbic acid
- Acetic acid

Dionex ICS-5000 IC System Operating Conditions

Flow Rate:	1.5 mL/min
Injection Volume:	100 µL
Column Temp:	30 °C
Backpressure:	1000 psi
Flush Volume:	1000 µL
Detection:	PAD
Cell Temp:	30°C
Background:	2–10 nC
Working Electrode:	Silver working electrode
Reference Electrode:	Mode: Ag/AgCL Noise: 3–5 pC

Analysis

IC-PAD

Results

See chromatograms below.



Left: Determination of iodide in DI water (A), milk-based infant formulas (B–E), and soy-based infant formula (F). Right: Chromatogram of iodide in infant formula (A) and infant formula spiked with iodate (B).

Combining the synergies of ion chromatography and inductively coupled plasma to identify mercury contamination in herbal medicine



Introduction

Both mercury and lead are neurologic toxins and bioaccumulators, targeting brain, and other organs which can cause birth defects and sometimes death. Therefore, not only must the patients be rapidly diagnosed and treated to minimize the damage to their health but the source of their illness must be also rapidly identified to prevent other future cases. This application note demonstrates the advantages of using ion chromatography with inductively coupled plasma mass spectrometry for mercury speciation.

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Mercury Speciation in Herbal Medicines

Equipment

- Dionex ICS-1600* IC System including: Dionex AS-AP Autosampler (P/N 074921)
 VWD Variable Wavelength Detector 3400 (P/N 070221)
- Dionex IonPac CS5A mixed cation/anion exchange column set
- Inductively Coupled Plasma Mass Specrometer (ICP-MS)
- * The Thermo Scientific[™] Dionex[™] Integrion[™] HPIC[™] system can be used for equivalent results.

Standards and Reagents

- Acetic acid
- Sodium perchlorate
- Cystine

Results of mercury determinations in contaminated herbal medicines

Analysis

IC-ICP-MS

Results

In this mercury poisoning cluster example, the IC analysis provided mercury speciation which defines to potential toxicity based on the toxicity of each species, whereas the ICP-MS analysis provided a fast multi-element screening. This revealed that mercury caused the clinical symptoms, and determined the total mercury contamination. Inorganic mercury determinations are shown in the table below.

Inorganic Mercury				
Sample	Measured (mg/L)	Calculated (wt %)	Calculated (µg/tablet)	Methylmercury / Ethylmercury
Control	-	-	-	-
1	3.69	0.0586	52	ND
2	15.6	0.2265	387	ND
3	4.82	0.0437	117	ND
4	5.31	0.0473	123	ND
5	11.3	0.0708	243	ND

Total and speciation analysis of mercury in contact lens solutions by ICP-MS

Introduction

While there is continual awareness regarding exposure to mercury (Hg) sources in general and MeHg⁺ in particular due to its presence in food samples such as fish, less interest is paid to the potential risk from ethylmercury (EtHg⁺ or EtHgX). One of the main reasons for this is the faster degradation and consequently excretion of EtHg+ in the human body that results in considerably lower chronic toxicity. There remains however potential sources where acute intake of EtHg+ can occur, for example as a consequence of exposure to thiomersal. Thiomersal is used as a bactericide in multi-dose and in other health related products such as eye drops or contact lens solutions. The compound hydrolyzes in aqueous solution to form EtHg⁺ and thiosalycilate which is an effective bactericide.

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Mercury Speciation in Contact Lens Solutions

Equipment

- Dionex ICS-5000 IC System*
- Dionex IonPac CS5A column, 2 mm I.D. × 250 mm
- iCAP Qc ICP-MS *Dioney ICS 5000+ HPIC System of
- *Dionex ICS-5000* HPIC System can be used for equivalent results

icap	Q	Operating	Parameters
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Forward Power:	1550 W	
Nebulizer Gas:	1.05 L/min	
Injector:	2 mm I.D., quartz	
Interface:	Ni sampler and skimmer	
Dwell Time: 10 ms, 100 ms for speciation analysis		
Analysis Mode:	Standard (no cell gas)	

Spike recovery of thiomersal in contact lens solution

Dionex ICS-5000 IC System Operating Parameters		
Elution:	Isocratic	
Mobile Phase:	10 mmol/L NaClO ₄ 10 mmol/L acetic acid 10 mmol/L cystine	
Flow Rate:	0.5 mL/min	
Injection Volume:	20 µL	
Duration:	5 min	

Analysis IC-ICP-MS

Results

See table and chromatogram below.

Sample #	Amount Spiked (mg/kg)	Amount Recovered (mg/kg)	Spike Recovery (%)
1	10.2	10.9 ± 0.04	108
2	18.1	18.8 ± 0.07	104



Chromatographic separation of EtHg+ derived from thimerosal hydrolysis

Innovative analytical technologies

Eluent Generation

RFIC-EG systems have redefined IC by making it possible to just add water to operate an IC system. These systems allow for a simpler and more reliable way to deliver superior results while simultaneously saving time and labor. Eluent generation allows the automatic production of highpurity IC eluents. This is made possible through precise control of the electric current applied to the electrolysis of water to generate hydroxide and hydronium ions. Eluent generation eliminates the need to manually prepare eluents from concentrated acids and bases. The only routine reagent needed is deionized water. Furthermore, because the instrument pump seals and pistons only come in contact with deionized water, overall pump maintenance is significantly reduced.

Learn more about our eluent generation solutions

High Pressure Ion Chromatography

HPIC systems redefine the way IC is performed due to their continuous operation up to 5000 psi when configured as an RFIC system. High backpressure tolerance lets you increase flow rates to maximize your throughput while still benefiting from the advantages of electrolytic eluent generation and suppression. This feature allows the use of new high-efficiency 4 µm particle-size columns, which produce fast run times using 150 mm long columns and high resolution using 250 mm long columns.

Learn more about HPIC

4 µm Particle Columns

Chromatographic separations using packed columns benefit from a high number of theoretical plates per column. The number of theoretical plates can be increased by packing smaller particles into the columns. Typically IC columns use resin particles ranging from 7–9 µm in diameter. Recent developments in resin technology have allowed the use of 4 μm resin particles in ion exchange columns. The benefits of columns packed with smaller particles include more efficient peaks, better resolution, faster run times, easier integration, and more reliable results.

Learn more about 4 µm Particle Columns

Ion Chromatography and RFIC Systems

Which IC system is right for your application and budget? From basic starter-line to highly customizable high-pressure IC systems, feel confident that you are selecting quality Thermo Scientific Dionex products, support, and service from the IC technology innovator and leader.



Dionex Integrion HPIC System



Thermo Scientific[™] Dionex[™] Aquion[™] IC System



Thermo Scientific[™] Dionex[™] ICS-5000⁺ IC System

Learn more about IC systems

Innovative analytical technologies

ICP-MS Systems

The Thermo Scientific[™] iCAP RQ[™] ICP-MS system represents a unique platform to determine the total elemental concentration of a sample. The iCAP RO allows for high sensitivity that can provide single figure ppt detection limits for many elements. As a result, a full mass range analysis can be carried out for routine samples. Additionally, the iCAP RQ series houses a proprietary collision/ reaction cell with low mass cut-off so unwanted species do not pass through the quadrupole mass filter. The iCAP TQ ICP-MS is a triple quadrupole ICP-MS that enables advanced interference removal using reactive gases such as oxygen, and hence allows lower detection limits to be obtained for difficult elements even in challenging samples. When coupled with Dionex IC systems, these techniques successfully provide a complete picture for analyzing both total elemental concentration as well as chemical form of the element of interest.



icap RQ

Learn more about the iCAP RQ



iCAP TQ

Learn more about the iCAP TQ

Find out more at thermofisher.com/speciationanalyzer

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