Speciation Applications Summary

Ion Chromatography

Trace Elemental Species Separation and Detection

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Contents

The Ion Chromatography System

Innovative IC Solutions for All Applications and Performance Needs

Thermo Scientific™ Dionex™ IC Systems have led the analytical instrument industry for over 30 years with solutions that represent state-of-the art technological advancements and patented technologies. Our products have evolved over many generations, with each new product providing enhanced performance, greater reliability, and easier operation.

Our High-Pressure Ion Chromatography HPIC™ Systems, including the Thermo Scientific Dionex ICS-5000+ system, are optimized for flexibility, modularity, and ease-of-use, combining the highest chromatographic resolution with convenience. Additionally, Capillary IC takes convenience to a whole new level.

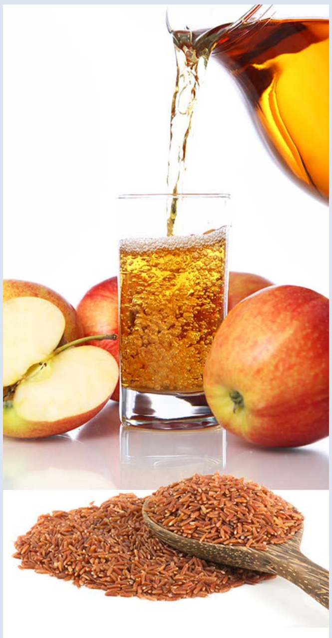
The Thermo Scientific Dionex ICS-4000 is the world's first dedicated capillary high-pressure Reagent-Free™ (RFIC™) IC system, delivering high-pressure IC on demand with a system that is always ready for the next analysis.

Reagent-Free IC systems eliminate daily tasks of eluent and regenerant preparation in turn saving time, preventing errors, and increasing convenience. RFIC-EG systems use electrolytic technologies to generate eluent on demand from deionized water, and to suppress the eluent back to pure water to deliver unmatched sensitivity.

At the heart of our ion chromatography is a unique set of column chemistries that provide high selectivities and efficiencies with excellent peak shape and resolution. Thermo Scientific™ Dionex™ IonPac™ chromatography columns address a variety of chromatographic separation modes including ion exchange, ion exclusion, reversed-phase ion pairing, and ion suppression. Our column chemistries are designed to solve specific applications, and we offer a variety of selectivities and capacities for simple and complex samples. Additionally, our Dionex IonPac column line is available in standard bore, microbore and capillary formats for the ultimate application flexibility. Learn more about our IC systems and consumables at www.thermoscientific.com/dionex.

The complete Thermo Scientific Dionex IC Systems family

Speciation Analysis



Separate and Quantify Different Element Chemical Forms

The need to distinguish between chemical forms of an element has become 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,* can determine an element's various chemical forms, and thus deliver 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 followed by trace elemental detection and quantification using ICP-MS. This combined method is termed Ion Chromatography-Inductively Coupled Plasma Mass Spectrometry (IC-ICP-MS).

ICP-MS is a multi-element spectrometry method to determine 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 thousand [ppt] detection). This technique leverages the combination of an ICP source with a mass spectrometer. The ICP is a high temperature source that decomposes and atomizes molecules, then ionizes the atoms. The mass spectrometer separates and detects these ions.

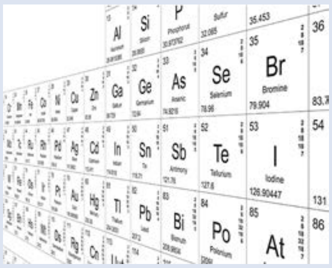
The process of ion chromatography allows ions and polar molecules to be separated on the basis of their charge, size, and polarizability. This specific method determines ionic species mainly with a conductivity detector, but can also be used with other types of detectors. With its metal-free fluidic flow path, ion chromatography is ideally suited to elemental speciation analysis. Furthermore, the system can analyze a range of compounds, from anionic and cationic contaminants to disinfection by-products. These are all important indicators of quality in environmental waters, the pharmaceutical industry, and food applications; many of which are toxic and need to be regulated.

The Thermo Scientific™ iCAP Q™ ICP-MS system represents a unique platform to determine the total elemental concentration of a sample. The iCAP Q 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 Q series houses a proprietary collision/reaction cell with low mass cut-off so unwanted species do not pass through the quadrupole mass filter. 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.

Learn more about speciation analysis at www.thermoscientific.com/speciation.

The Thermo Scientific iCAP Q with the Thermo Scientific Dionex ICS-5000+ IC System

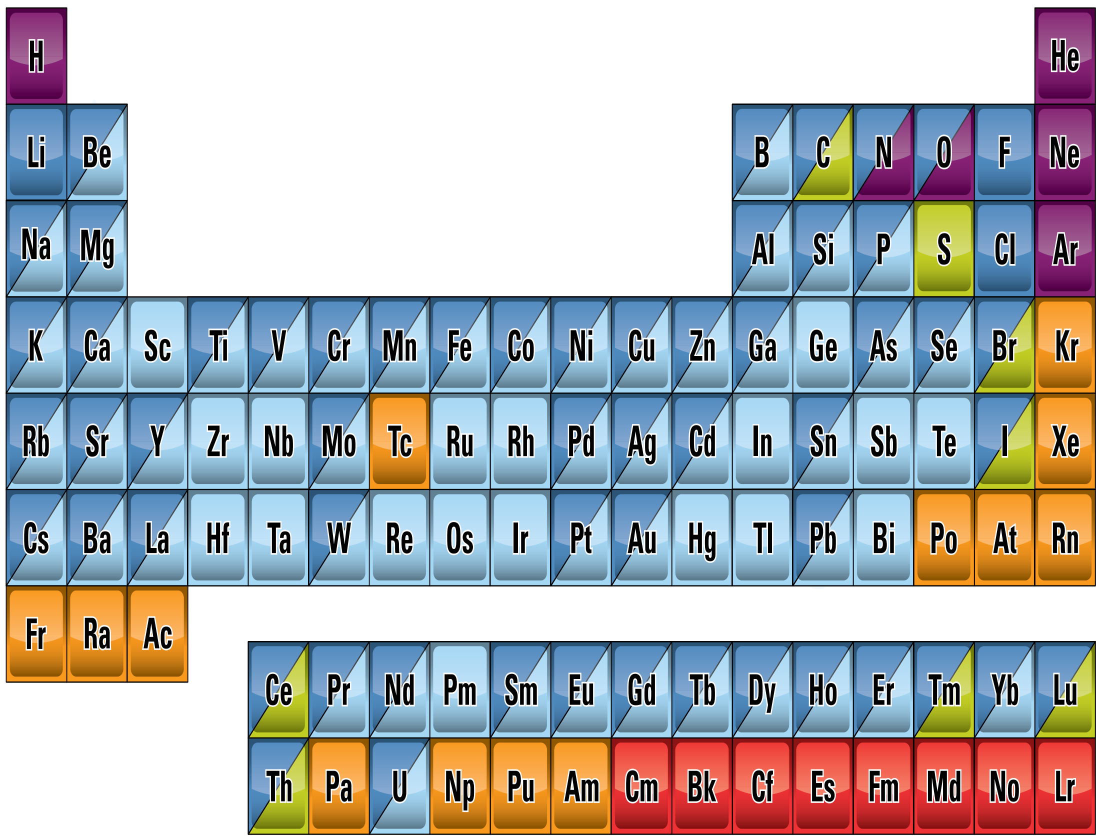
Complete Inorganic Analysis



More Capabilities Together

- AA/ICP/ICP-MS
- IC
- ICP/ICP-MS
- Unstable elements
- ICP-MS only
- Not measurable

Periodic Table of Total Inorganic Analysis



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 US 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*
- 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
Dwell Time:	100 ms

Dionex ICS-5000 IC System 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

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

Analysis

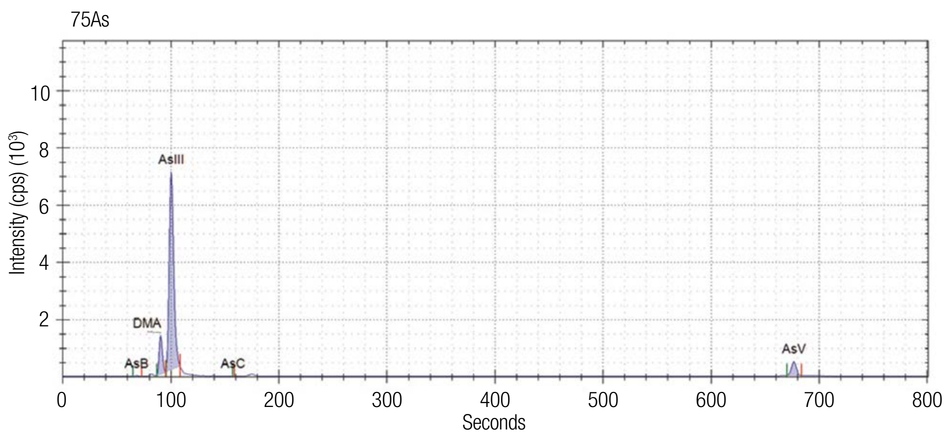
IC-ICP-MS

Results

See table and figure 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.

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 IC system*
- Dionex IonPac AG7 column, 2 mm I.D. × 50 mm
- iCAP Qc 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.
QCell He Gas Flow:	4.8 mL/min
QCell KED:	2 V
Dwell Time:	100 ms

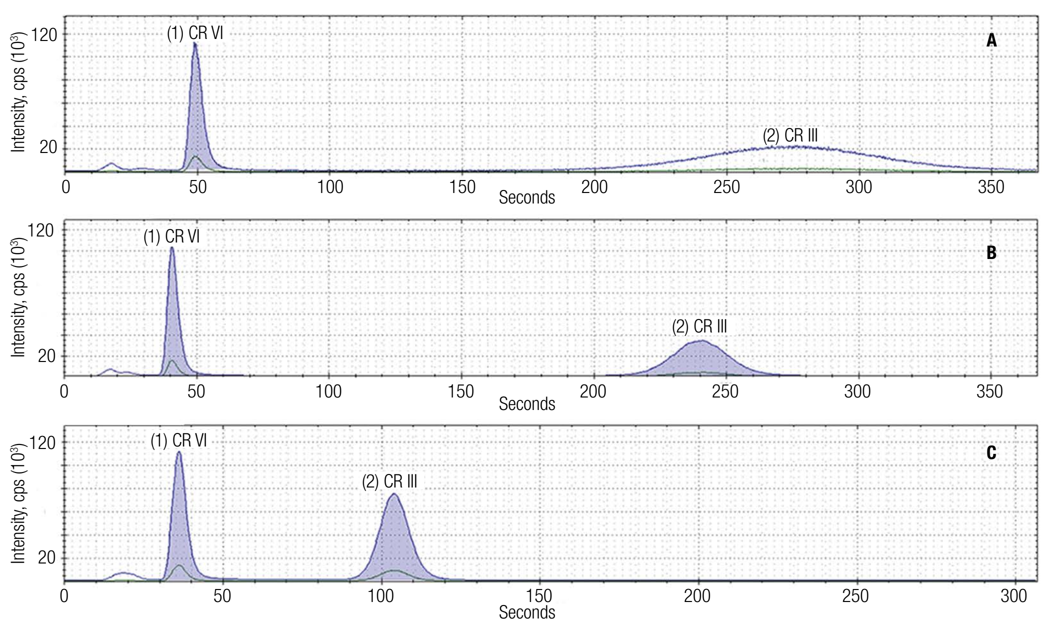
Dionex ICS-5000 IC System Operating Parameters	
Elution:	Isocratic
Mobile Phase:	0.4 mol/L HNO_3
Flow Rate:	400 µL/min
Injection Volume:	20 µL
Duration:	150 s

Analysis

IC-ICP-MS

Results

See figures and table 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

Conc. spiked (ng/g)	Cr (VI)		Cr (III)	
	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

Determination of Iodide and Iodate in Soy- and Milk-Based Infant Formulas

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.

Download the Full Version of Application Note 37

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

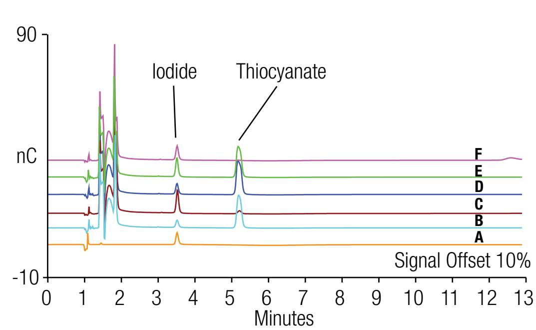
Flow Rate:	1.5 mL/min
Injection Volume:	100 µL
Column Temp:	30°C
Backpressure:	1000 psi
Flush Volume:	1000 µL
Detection:	PAD
Cell Temp:	30°C
Background:	2–10 nC
Working Electrode:	Silver working electrode
Reference Electrode:	Mode: Ag/AgCL Noise: 3–5 pC

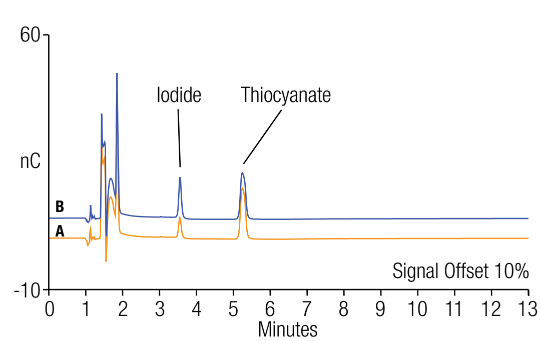
Analysis

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).

Brief of Application Note 43130

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 bio-accumulators, 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)

Standards and Reagents

- Acetic acid
- Sodium perchlorate
- Cystine

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.

Results of mercury determinations in contaminated herbal medicines

	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.

Download the Full Version of Application Note 43141

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

*Dionex ICS-5000+ HPIC System can be used for equivalent results

iCAP Q Operating Parameters

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)

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

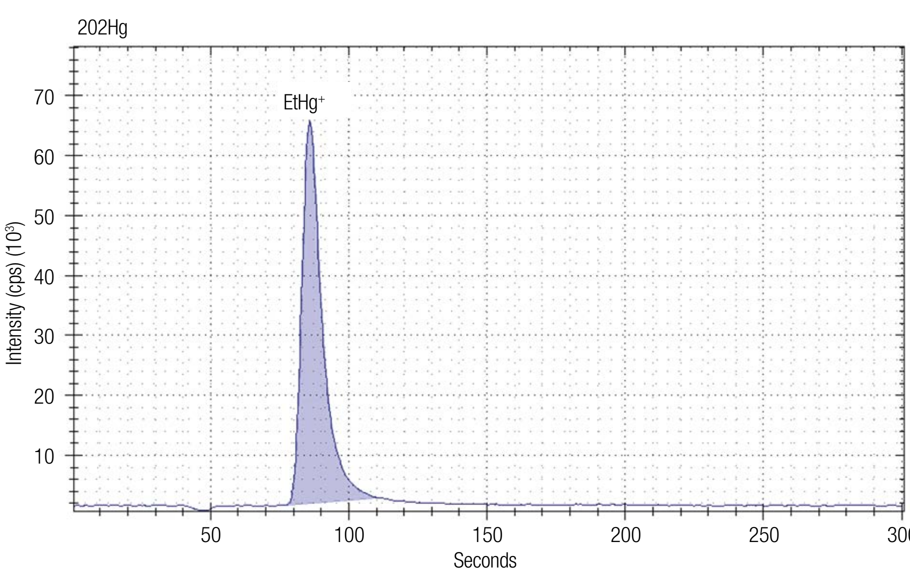
IC-ICP-MS

Results

See table and chromatogram below.

Spike recovery of thiomersal in contact lens solution

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

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