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Semiconductor fabrication analysis

Ion chromatography applications summary notebook

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Introduction

Ionic contamination is a big concern in semiconductor manufacturing processes and in finished devices because small amounts of contamination (parts-per-billion (ppb) to parts-per-million (ppm) concentrations) can cause corrosion, erosion, electromigration, and shorting in devices, on wafers or in final individual electronic components. Ion chromatography (IC) is an efficient analytical technique that can quickly determine both trace and major constituents of a wide variety of process contaminants in the semiconductor industry. This technique provides a convenient means of quantifying common inorganic anions and cations, certain organic additives, transition metals, polyvalent ions, and organic chelating agents. With online IC process monitoring, these components can be determined quickly at low parts-per-trillion levels for immediate remediation steps.

In this Application Notebook, IC methods to monitor both the front end (semiconductor wafer fabrication) and back end (semiconductor packaging and PCB board integration) processes are described.

Semiconductor Fabrication Process

Wafer Cleaning

A recurrent step in the fabrication of an integrated circuit is to clean and prepare the silicon wafer for a subsequent process step. Complete cleaning of semiconductor surfaces requires that particulates, organic films, and adsorbed metal ions be removed. Most cleaning procedures are based on immersion in liquid baths of semiconductor grade solvents or other chemical reagents. Ion chromatography is used to monitor the ionic contamination on the bare wafers before and after cleaning. The cleanliness of the cleaning solvents and solutions is monitored as incoming solvents and during the cleaning processes as part of process control.

Photo-lithography

For patterning of the device structures on the wafer, UV optical lithography is used. Resist is deposited on the wafers and then the resist is illuminated through a mask in a scanner and subsequently developed in a developer track. The control of the lithography steps is extremely critical as the smallest critical dimensions (CD) of the active devices are defined in these steps. CD, resist profiles, line edge roughness, etc. need to be measured. To ensure a reliable lithography process, the clean room air also needs to be well controlled. Small amounts of refractory hydrocarbon contamination can be detrimental to the lithography process (lens contamination). Traces of acids, bases and resist solvents such as PGMEA or ethyl lactate in the clean room (CR) air can also have negative impacts by creating defects on the wafers.

Ion chromatography is used to measure ionic contaminants in resist strippers and developers. Trace anions and cations are routinely determined in resists by first extracting the ionic content with a solution of eluent (mobile phase) and then injecting the subsequent solution into the IC system.

Etching

The wafer, covered with the photoresist pattern, is etched with mixed acid etchants to transfer the resist pattern into the underlaying films and layers. The etch rate and depth are dependent upon the ratio of acids in mixed acid etchants. Wet chemistry methods for the determination of acetic, nitric, phosphoric, and hydrofluoric acids in the mixed etchants are both labor intensive and time consuming. Ion chromatography provides a rapid, accurate, and automated analytical technique for this analysis.

Ion Implantation

During this step, ionized impurity atoms (boron, phosphorous, arsenic) are implanted into the exposed wafer to dope and electrically activate the various silicon regions of the transistors. Control of the ion precursor gases is critical to avoid spurious co-implants.

Damascene Metallization

The electrical connections between the transistors of the active devices on the wafer are realized by a damascene metallization process in which an insulating film (such as SiO_2) is deposited and patterned using lithography and etch. Metal is then deposited into the vias and trenches of the dielectric film using an electrolytic metal plating process and, finally, excess metal is removed and the wafer surface is planarized by chemical mechanical planarization (CMP). Typically, copper is the metal of choice for the interconnects. Tungsten is also used for local interconnects in addition to transistor contact levels. The CMP process uses polishing slurry and polishing pads as consumables. After this process, particles and contaminates must be removed. The cleaning process involves chemical cleaning solutions and often polyvinyl alcohol (PVA) brushes to remove submicron particles from the surface of the wafers. The components used (slurries, cleaning chemistries, pads, and brushes) are often analyzed for trace contaminants by IC.

Semiconductor Packaging Process

Die Attach

Die attach adhesives are polymers used to attach the silicon chips (dies) to the "package", which includes contact leads to connect the silicon chip to another device or a printed circuit board. These die attach adhesives must also meet acceptable cleanliness levels to minimize contamination of the package leads and silicon die. As such, military specifications (MIL-STD-883, Method 5011 and MIL-A- 87172) require the use of IC to measure chloride, sulfate, and sodium in these die adhesives. Manufacturers of die attach adhesives use Thermo Scientific™ Dionex™ IC systems to comply with regulations to determine concentrations of sodium, chloride, fluoride, and potassium.

Leadframes

Silicon chips are connected via a wire bond to leadframes. Leadframes are used to connect the integrated circuit to the exterior of the device. Ionic contaminants on the surfaces of leadframes are extracted by deionized water or a mixture of deionized water and isopropyl alcohol and determined by IC analysis.

Wire Bond

Electrical connections between the silicon chips and the leadframe pins are created using wire (typically gold) that is bonded by thermosonic, thermopression, or ultrasonic processes.

Encapsulation

Silicon chips are encapsulated with plastic coating for mechanical and environmental protection. Contaminants entrained in the molding compounds can diffuse out of the molding compound into the integrated circuit and catalyze metallization corrosion.

Printed circuit board mounting

Wire connections from printed circuit boards to the leads of the semiconductor package are made by soldering using soldering fluxes. The remaining residues may contain potentially damaging ionic contamination. The ionic content is typically extracted by immersing or spraying the die-package with deionized water or an alcohol-deionized water mixture. The resulting solution is analyzed by IC for inorganic halide and organic acid residues. The ionic content in solder fluxes has decreased in concert with increased manufacturing demands to produce clean devices. "Fluxless" soldering has been found to leave detrimental concentrations of ionic residues on devices.

Ultrapure Water

Deionized (DI) water is used throughout the fabrication of an integrated circuit, which may be exposed to DI water hundreds of times during its manufacture. Because contaminants in DI water can distort normal dopant profiles, create inversion layers or cause shorts, or other circuit malfunctions, it is extremely important that semiconductor pure water be maintained at its highest attainable quality. Ion chromatography is used to maintain the best attainable quality water in semiconductor fabrication facilities and is the only analysis technique recommended by Semiconductor Equipment and Materials International (SEMI) for inorganic anions. Several methods have been used to achieve the sensitivity needed for trace ion determinations in ASTM Type II water* or SEMI grade DI water:

1) Direct injection of large volume samples usually 1 to 2 mL

2) Injection of a large volume sample previously concentrated on a concentrator column, usually <5 mL

3) AutoPrep, which combines large volume sampling (10 mL) with automated sample preparation, automated standard calibration, and inline water purification to achieve ng/L to low µg/L concentrations

Applications in this section include those discussing:

- Direct Injection of a Large Sample Volume
- Concentration of a Large Volume Sample
- AutoPrep with Inline Water Purification

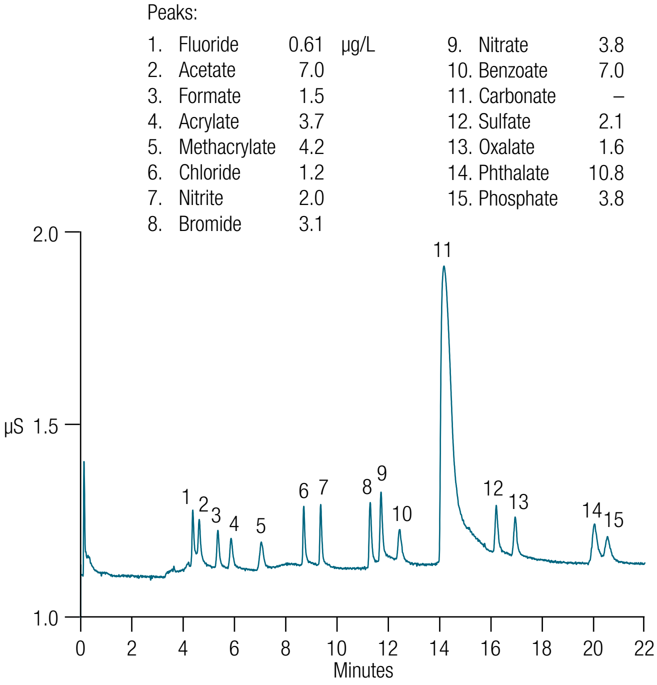
*ASTM Specification D 5127, ASTM International. 100 Barr Harbor Drive, West Conshohocken, PA 19428.

Direct Injection of a Large Sample Volume

Determination of Trace Anions in High-Purity Waters by Ion Chromatography with the Dionex IonPac AS17 Column Using High-Volume Direct Injection with the Dionex EG40 Eluent Generator

This application note describes an IC method using a microbore Thermo Scientific™ Dionex™ IonPac™ AS17 anion-exchange column to determine trace concentrations of common inorganic anions, low-molecular-weight organic acids, as well as acrylate, methacrylate, benzoate, and phthalate. These contaminants can come from some of the following sources: cleaning agents, adhesives, oils, mold release agents, and solder fluxes. Anionic contamination is known to cause corrosion in microelectronic circuitry. In this application note, the analytes are detected by suppressed conductivity with a 2 mm Thermo Scientific™ Dionex™ ASRS™ ULTRA Anion Self Regenerating Suppressor operated in the "Gas-Assisted Recycle" mode. The high-volume, direct-injection technique is used to achieve sensitive detection at low- to sub-µg/L levels without the use of a concentrator column or loading pump and valve.

Conditions	
Eluent:	Potassium hydroxide (Dionex EG40 Eluent Generator as the source)
Temperature:	30 °C
Dionex EG40 Offset Volume:	0 µL
Eluent Flow Rate:	0.5 mL/min
Detection:	Suppressed conductivity, Dionex ASRS ULTRA Gas-Assisted Recycle mode
Dionex ASRS Current Setting:	100 mA
Expected Background Conductivity:	1 µS (40 mM KOH)
Expected System Backpressure:	15.2–16.6 MPa (2200–2400 psi)
Sample Volume:	1 mL

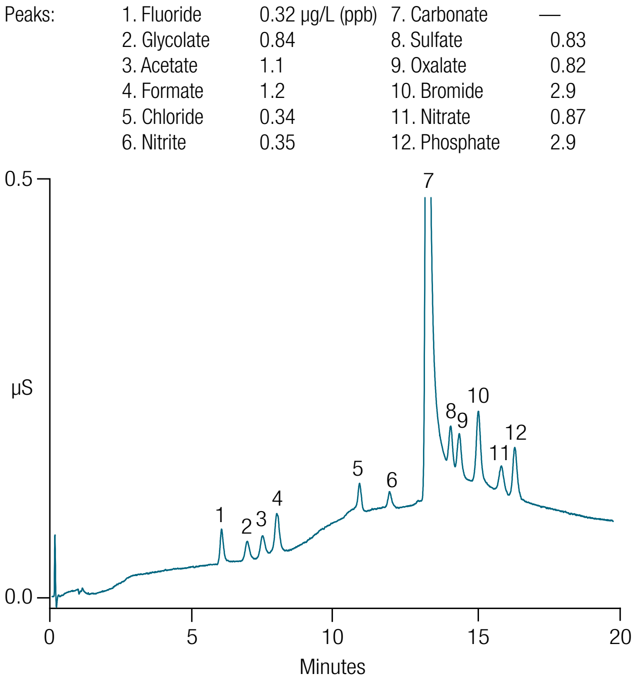


Trace anion determination using the Dionex IonPac AS17 column with Method 1.

Improved Determination of Trace Anions in High Purity Waters by High-Volume Direct Injection with the EG40 Eluent Generator

This application update describes the use of the Thermo Scientific™ Dionex™ EG40 potassium hydroxide (KOH) eluent generator with the Thermo Scientific™ Dionex™ IonPac™ AS15-5µm (3 x 150 mm) column for trace anion analysis. The Dionex EG40 eluent generator generates high-purity and carbonate-free hydroxide eluents on-line to improve the method performance for determination of target analytes at trace levels. The high-volume direct injection technique is used to achieve sensitive detection at low to sub-µg/L levels without the use of a concentrator column, sample loading pump, and additional valve. The Dionex IonPac AS15-5µm column is used to separate common inorganic anions and low molecular weight organic acids in less than 20 min. This column provides improved peak efficiencies, resulting in lower detection limits than the Thermo Scientific™ Dionex™ IonPac™ AS15-9µm column (4 x 250 mm).

Conditions	
Eluent:	Potassium hydroxide (Dionex EG40 as the source)
Temperature:	30 °C
EG40 Offset Volume:	0 µL
Pump Program:	See Table 1
Eluent Flow Rate:	0.7 mL/min
Detection:	Suppressed conductivity, Dionex ASRS ULTRA, Gas-Assisted Recycle mode
ASRS Current Setting:	100 mA
Expected Background Conductivity:	1 µS (60 mM KOH)
Expected System Backpressure:	13.8 MPa (2,000 psi)
Sample Volume:	1 mL



Trace anion determination using the Dionex IonPac AS15-5µm (3 × 150 mm) column.

Download the full application update

Table 1. Pump program.

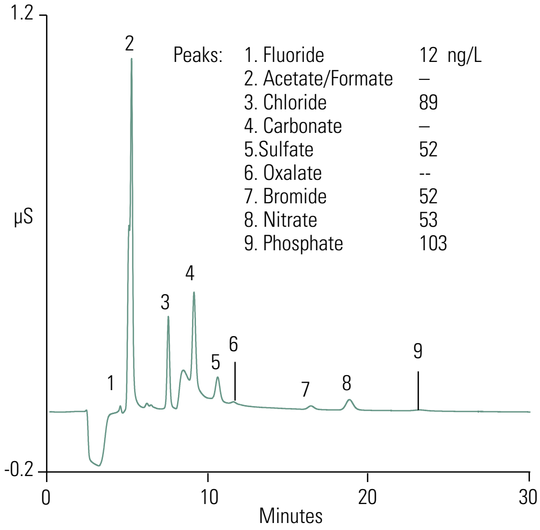
Time (min)	Flow (mL/min)	A (%)	Valve	EG40 Conc (mM)	Comments
Initial	0.70	100	Load	60.0	60 mM KOH
0.00	0.70	100	Load	7.0	Load sample loop, equilibrate 7.0 mM KOH for 5 min
5.00	0.70	100	Inject	7.0	Inject
10.00	0.70	100	Inject	7.0	
17.00	0.70	100	Inject	60.0	60 mM KOH
21.00	0.70	100	Load	60.0	60 mM KOH

Determination of Trace Anions in Ultrapure Water Using Capillary Ion Chromatography

Trace anion determinations in ultrapure water (UPW) are important for the power and electronics industries. Ionic contamination in the electronics industry causes poor yields and early failures of important semiconductor and disk drive components.

This technical note discusses the application of capillary IC, the Thermo Scientific™ Dionex™ AS-AP autosampler, and the Thermo Scientific™ Dionex™ ICW-3000 Online Water Purifier for trace-level analysis, and provides specific guidance for laboratory techniques that are essential for achieving good results when performing ultratrace analyses.

Conditions	
Columns:	Thermo Scientific™ Dionex™ IonPac™ AG15, 0.4 × 50 mm Thermo Scientific™ Dionex™ IonPac™ AS15, 0.4 × 250 mm
Water Purifier:	Dionex ICW-3000 Online Water Purifier
Eluent Source:	Thermo Scientific™ Dionex™ EGC-KOH capillary eluent generator
Eluent:	30 mM KOH
Concentrator:	Thermo Scientific™ Dionex™ IonSwift™ MAC-100
Vol. Conc.:	100 µL
Carbonate Removal Device:	Thermo Scientific™ Dionex™ CRD-200 capillary carbonate removal device
Column Temp:	30 °C
Flow Rate:	10 µL/min
Detection:	Suppressed conductivity, Thermo Scientific™ Dionex™ ACES™ Anion Capillary Electrolytic Suppressor, external water mode



Trace anions standard by concentrating 100 µL on the Dionex IonSwift MAC-100 concentrator column.

Download the full technical note

Determination of Trace Anions in High-Purity Water Using Two-Step Isocratic Ion Chromatography

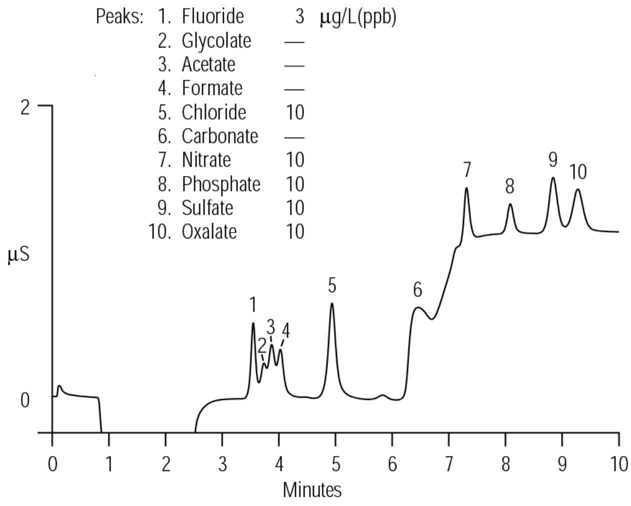
Reliable methods to quantify anionic contaminants in high-purity water are essential to both the semiconductor and power generation industries. Anionic contamination is known to cause metal corrosion in microelectronic circuitry. To respond quickly to changing conditions, those monitoring anionic contamination at trace levels require a fast method. This application note describes a rapid, high-volume, direct-injection technique to isocratically separate the trace anions using a borate eluent on a Thermo Scientific™ Dionex™ IonPac™ AS14 2 mm separation column. A step change in the borate eluent concentration provides higher eluent concentrations later in the run, thereby reducing the total run time.

Conditions	
Columns:	Dionex IonPac AS14 Analytical, 2 × 250 mm (P/N 046129) Dionex IonPac AG14 Guard, 2 × 50 mm (P/N 046138)
Trap Column:	Thermo Scientific™ Dionex™ IonPac™ ATC Trap, 2 mm (Replaced by Dionex IonPac ATC-3, 4 × 35 mm, P/N 079932)
Eluent A:	9 mM Boric acid/ 6.75 mM Sodium hydroxide
Eluent B:	40 mM Boric acid/ 30 mM Sodium hydroxide

Pump Program:

Time	*%A*	*%B*	*Comments*
Initial	100	0	Equilibrate initial eluent
0	100	0	Load sample loop
5.00	100	0	Inject
8.50	100	0	
8.51	0	100	Step to stronger eluent
15.00	0	100	

Eluent Flow Rate:	0.75 mL/min
Detection:	Suppressed Conductivity, Thermo Scientific™ Dionex™ ASRS™ (2 mm), AutoSuppression™ external water mode SRS Current Setting: 300 mA
Expected Background Conductivity:	2–4 µS
Expected System Backpressure:	17.2 MPa (2,500 psi)
Sample Volume:	1 mL



Trace anion standard.

Determination of Trace Anions in High Purity Waters by High Volume/Direct Injection Ion Chromatography

There has been considerable interest in the determination of anions at trace levels by IC. For high-purity water used in semiconductor processing, Semiconductor Equipment and Materials International (SEMI) recommends the use of IC for tracking trace ionic contaminants from 0.025 to 0.5 µg/L.

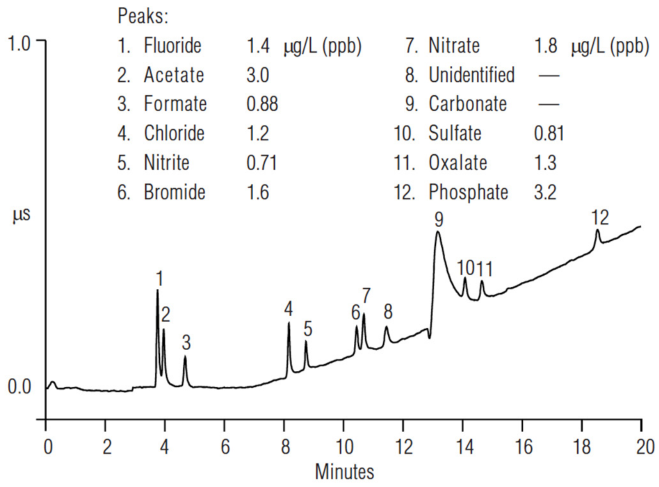
To determine ions from mg/L (ppm) down to mid-µg/L (ppb) levels using IC, a sample size of 10 to 50 µL is sufficient. If lower levels of determination are required, a preconcentration or trace enrichment technique is typically utilized. However, preconcentration has several disadvantages. Compared with a direct injection method, additional hardware is required, such as a concentrator column used to preconcentrate the ions of interest and a sample pump used for loading the sample. An additional valve is often required for switching the concentrator column in and out of line with the analytical column. Extra time is required for the preconcentration step. Also, analyte loading efficiency can be compromised when additives are present.

This application note describes the use of a gradient separation using the Thermo Scientific™ Dionex™ IonPac™ AS11 column for the determination of trace anions by high volume/direct injection.

Experimental Conditions	
Columns:	Dionex IonPac AS11 Analytical, 2 mm i.d. Dionex IonPac AG11 Guard, 2 mm i.d. Dionex IonPac ATC Anion Trap Column, 2 mm i.d.
Eluents:	1) DI H_2O 2) 5 mM NaOH 3) 100 mM NaOH Gradient program—see Table 1.
Flow Rate:	0.5 mL/min
Sample Volume:	750 µL
Detection:	Suppressed conductivity, Dionex ASRS Suppressor 2 mm, external water mode, 300 mA current

Table 1. Gradient program.

Time (min)	%E1	%E2	%E3	Comments
0	0	65	35	Initial 38 mM NaOH
1.99	0	65	35	End regeneration
2.00	90	10	0	Start 0.5 mM NaOH
9.00	90	10	0	End equilibration
11.50	90	10	0	0.5 mM to 5.0 mM NaOH
15.00	0	100	0	5.0 mM to 26 mM NaOH
29.00	0	78	22	End at 26 mM NaOH
29.10	0	65	35	Step to 38 mM NaOH



Trace anion determination.

Concentration of Large-Volume Samples

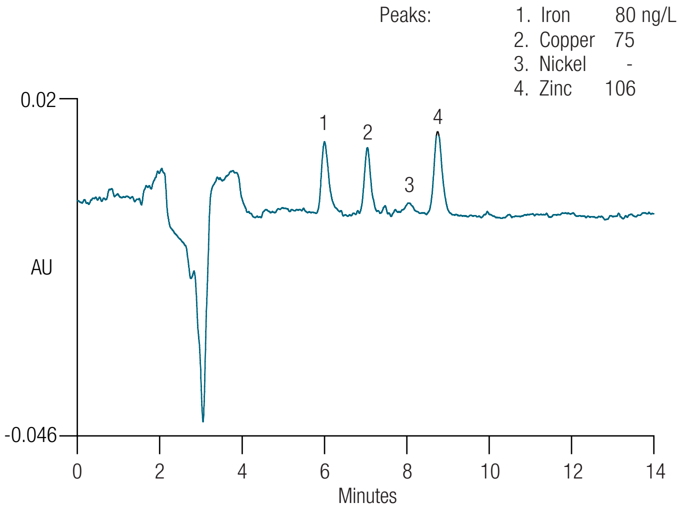
Determination of Transition Metals at PPT Levels in High-Purity Water and SC2 (D-clean) Baths

Metal atoms and conductive particle contaminants are undesirable and potentially damaging in semiconductor manufacturing processes. Oxidative cleaning baths and large volumes of ultrapure rinse water are used to remove metallic contamination from wafer surfaces. For optimal cleaning efficiency, the concentrations of iron and other metals in cleaning solutions should be minimized.

Improved analytical methods are needed to monitor metals in cleaning baths and rinse water. This application note describes a method for determining low ng/L amounts of transition metals in high-purity water and semiconductor bath solutions.

Conditions

Columns:	Dionex IonPac CS5A Analytical, 2 × 250 mm (P/N 052576) Dionex IonPac CG5A Guard, 2 × 50 mm (P/N 052836) Thermo Scientific™ Dionex™ IonPac™ TCC-2 Concentrator, 3 × 35 mm (P/N 043103)
Eluent:	PDCA
Eluent Flow Rate:	0.3 mL/min
LC-30 Temperature:	30.0 °C
Postcolumn Reagent:	0.06 g of PAR in 1 L Dionex MetPac PAR Postcolumn Diluent
Postcolumn Flow Rate:	0.15 mL/min
Concentrator Pump Flow Rate:	2.0 mL/min
Run Time:	15 min
Detection:	Visible, High setting, 530 nm
System Backpressure:	1700–2000 psi



SC2 batch (30 mL concentrated) containing 1 mL HCl / 5 mL H_2O_2 / 494 mL H_2O.

Determination of Silicate in High-Purity Water Using Ion Chromatography and Online Sample Preparation

The water used in the manufacture of semiconductors and other modern electronic components must be extremely pure. This water is often referred to as ultrahigh-purity water (UHPW). The production of UHPW includes deionization to remove corrosive strong acid anions, e.g., chloride and sulfate. Deionization cartridges exhaust their capacity over time and require replacement. When these deionization cartridges begin to fail, one of the first ions to break through the cartridge and contaminate the water is silicate ion. Silicate can break through while the water still yields an acceptable resistivity reading of 18 MΩ-cm. Therefore, silicate is a good early indicator of cartridge depletion.

In this application note, silicate is determined by first preconcentrating a large volume of UHPW on a Thermo Scientific™ Dionex™ IonPac™ AG4A-SC column, then separating the concentrated sample on a Thermo Scientific™ Dionex™ IonPac™ AS17 column set. The Thermo Scientific™ Dionex™ AutoPrep™ system is used to prepare the low-level standards for calibration and automatically generate the calibration curve.

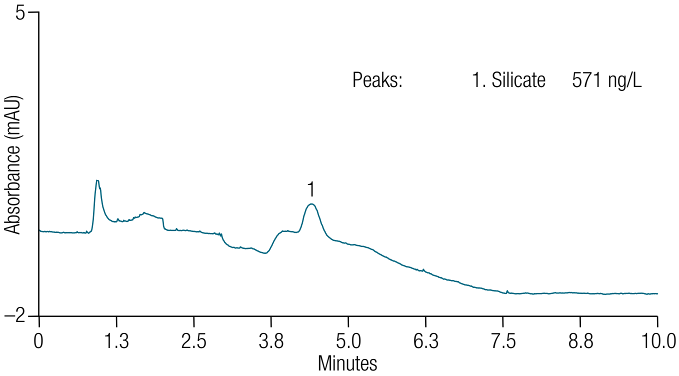
Conditions

Concentrator:	Dionex IonPac AG4A-SC, 4 × 50 mm (P/N 043175)
Columns:	Dionex IonPac AG17 guard, 4 × 50 mm (P/N 055684) Dionex IonPac AS17 analytical, 4 × 250 mm (P/N 055682)
Eluent Source:	Thermo Scientific Dionex EGC II KOH Eluent Generator cartridge with the Thermo Scientific Dionex CR-ATC Continuously Regenerated Anion Trap column

Eluent Concentration:

Time (min)	*Concentration (mM)*	*Curve*
–20.0	50	5
–7.1	50	5
–7.0	1	5
0.0	1	5
2.0	10	6
4.0	20	4
10.0	20	5

Large Loop Volume:	20 mL
Temperature:	30 °C
Detection:	Visible absorbance at 410 nm
Noise:	≤100 mAU
Postcolumn Conditions:	20 mM Sodium molybdate 0.2 N Nitric acid 6 mM Sodium lauryl sulfate Reaction Coil: 1500 µL (P/N 042630) Flow Rate: 0.5 mL/min



Chromatogram of laboratory DI water.

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AutoPrep with Inline Water Purification

Configuring the Dionex Integrion HPIC System for Trace Anion Determinations in Ultrapure Water

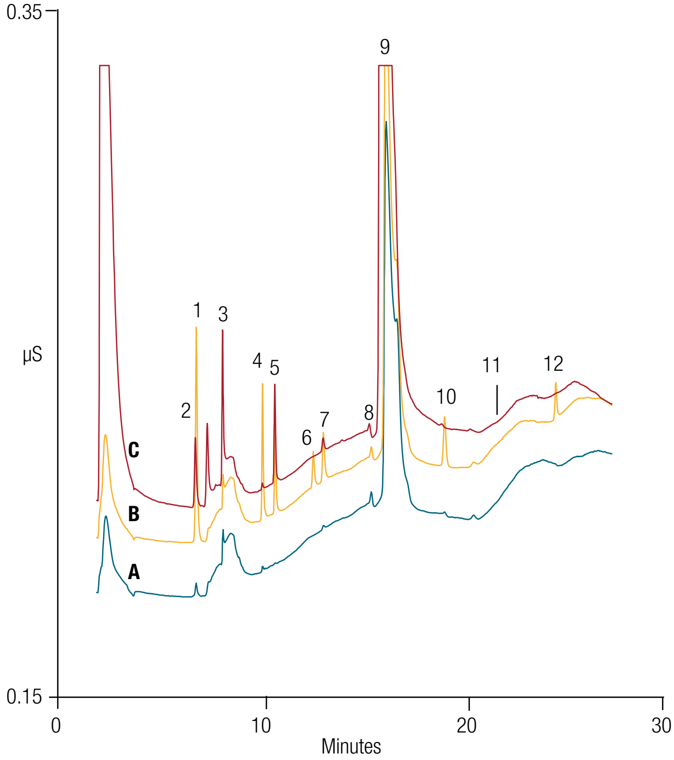
The latest advancement in IC instrumentation, the high pressure (HP) Thermo Scientific™ Dionex™ Integrion™ HPIC™ system, can operate continuously, at up to 5000 psi, for both 4 mm and 2 mm i.d. column formats when using electrolytic eluent generation. When combined with the advantages and ease-of-use of a Reagent-Free™ ion chromatography (RFIC™) system with automated calibration and water purification, this system permits low baseline contamination with excellent reproducibility, thereby yielding greater quantification accuracy, greater sensitivity, and consistently reliable results.

In this technical note, we provide installation recommendations for the Dionex Integrion HPIC system configured for trace anion determinations (ng/L to µg/L) using the Thermo Scientific™ Dionex™ AutoPrep™ module and the Thermo Scientific™ Dionex™ EWP Electrolytic Water Purifier. The results highlight the use of the Dionex EWP Electrolytic Water Purifier and Dionex AutoPrep modules to achieve ng/L sensitivity, as well as some of the instrument, column, and suppressor features.

Chromatographic conditions

Columns:	Dionex IonPac AG17-C guard, 4 × 50 mm Dionex IonPac AS17-C separation 4 × 250 mm
Eluent:	KOH gradient, See Table 2
Eluent Source:	Dionex EGC 500 KOH eluent cartridge, Dionex CR-ATC 600 trap column and high pressure degas module
Flow Rate:	1.0 mL/min
Column Temperature:	35 °C
Detection/ Suppressor Compartment:	15 °C
Detection:	Suppressed conductivity, Dionex AERS 500e suppressor, 4 mm, external water mode (driven by system pump)
Concentrated Volume:	Standards: Incremental additions of 10 µL
Sample Volume:	10 mL
Sample Flow Rate:	~3.5 mL/min (Dionex AS-HV Autosampler pump)
Standard Flow Rate:	~0.5 mL/min (Dionex AutoPrep small loop, gravity)
Concentrator:	Dionex IonPac UTAC-LP2, 3 × 35 mm
Run Time:	Calibration standards: 41.5 min
Samples and Check Standards:	51.5 min
Background Conductance:	< 1 µS
Noise:	< 1 µS
System Backpressure:	~2200 psi

Peaks:		A	B	C (ppt)
	1. Fluoride	11	54	18
	2. Acetate	--	--	--
	3. Formate	--	--	--
	4. Chloride	12	55	13
	5. Nitrite	13	55	18
	6. Bromide	--	54	--
	7. Nitrate	5	53	7
	8. System	--	--	--
	9. Carbonate	--	--	--
	10. Sulfate	--	53	--
	11. Oxalate	--	--	--
	12. Phosphate	--	53	--



Comparison of the system blank (A), a 50 ppt standard (B), and DI water (C).

Configuring the Dionex Modular HPIC System with an Inline Water Purifier and Inline Calibration for Trace Anion Determinations in Ultrapure Water

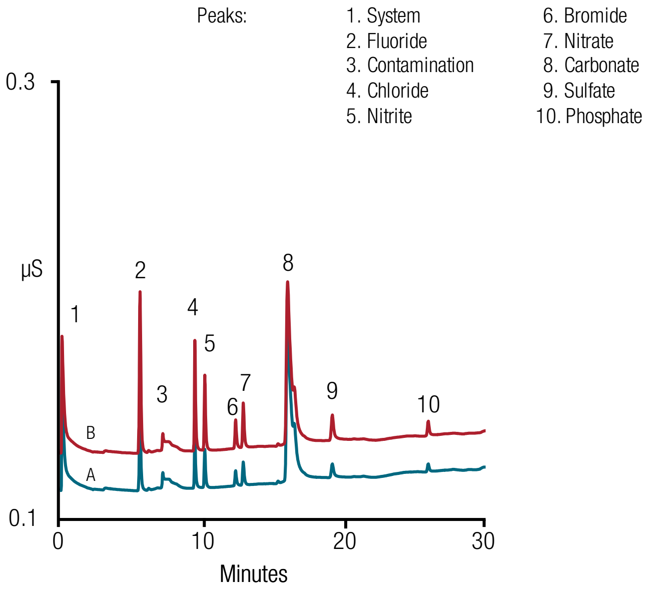
In the electronics industry, ionic contamination, in the parts-per-trillion (ppt, ng/L) to parts-per-billion (ppb, µg/L) concentrations, is a major concern. This contamination can cause corrosion-related failures, poor product quality, low product yields, and shortened product life. Although ionic contamination methods are well established, ionic contamination is increasingly important and more challenging as the devices decrease in size. Therefore, monitoring and minimizing ionic contamination is important; however, trace ion analysis continues to be challenging. The lab environment and IC system cleanliness can significantly impact the baseline contamination, limiting the analytical sensitivity in trace analysis methods.

This technical note demonstrates the configuration of one system of the Thermo Scientific™ Dionex™ ICS-5000^{+} dual IC systems* with the Thermo Scientific™ Dionex™ AS-HV high volume autosampler, the Thermo Scientific™ Dionex™ EWP Electrolytic Water Purifier, and Thermo Scientific™ Dionex™ AutoPrep module to determine trace anions in ultrapure water.

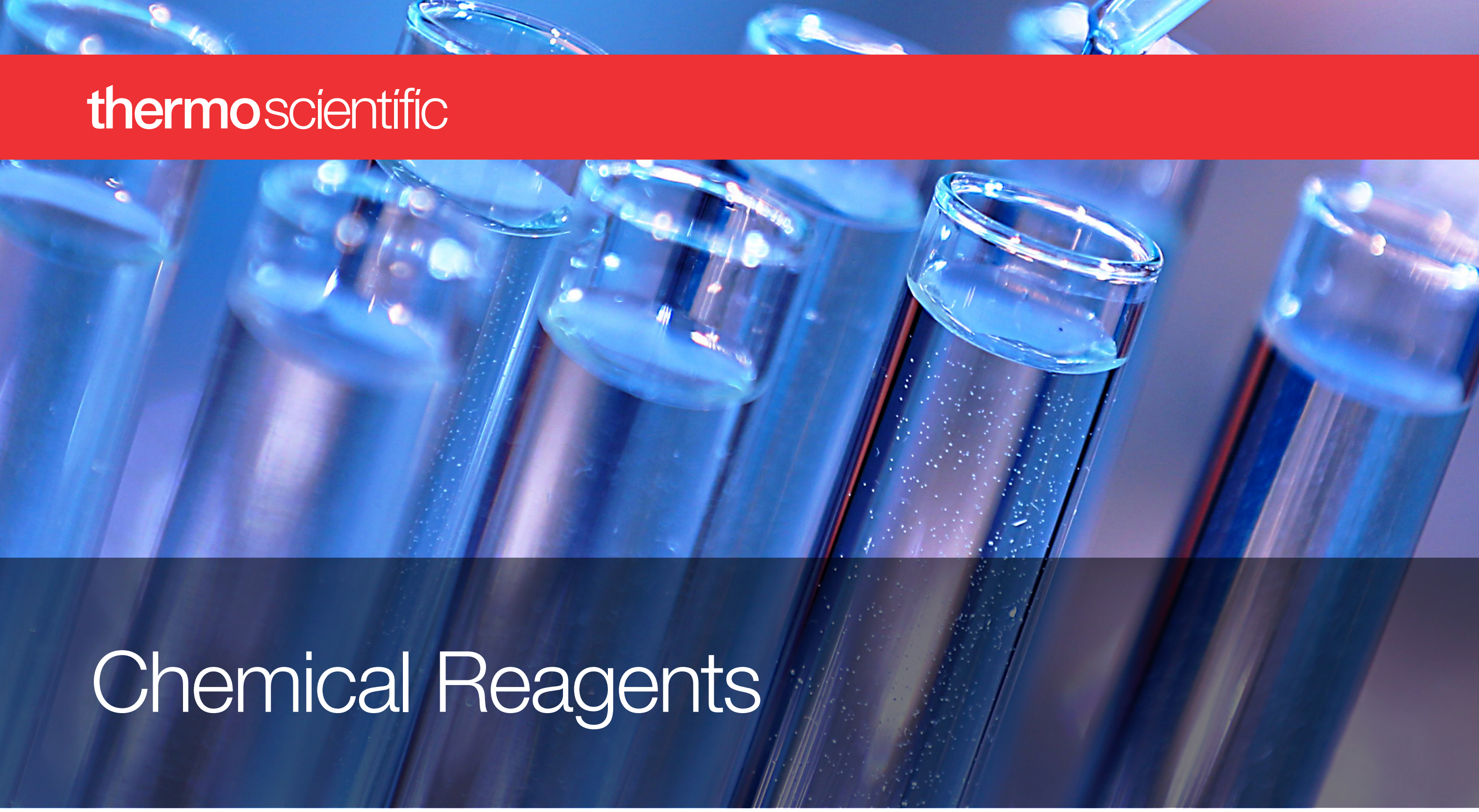
Chromatographic Conditions			
Columns:	Thermo Scientific™ Dionex™ IonPac™ AG17-C guard, 4 × 50 mm Dionex IonPac AS17-C separation, 4 × 250 mm		
Gradient:		Time	KOH (mM)
	Samples**	-21.5	50
	Calibration/EWP*	-15.0	50
		-15.1	1
		0.0	1
		4.0	1
		10.0	12.5
		20.0	20
		25.5	35
		30.0	35
Eluent Source:	Dionex EGC 500 KOH eluent cartridge, Thermo Scientific™ Dionex CR-ATC™ 500 and high pressure degas module		
Flow Rate:	1.0 mL/min		
Column Temperature:	35 °C		
Detection/Suppressor Compartment:	15 °C		
Detection:	Suppressed conductivity, Dionex AERS 500e suppressor, 4 mm, external water mode (driven by system pump)		
Concentrated Volume:	Standards: Incremental additions of 10 µL; Sample Volume: 10 mL		
Sample Flow Rate:	~3.5 mL/min (Dionex AS-HV autosampler peristaltic pump)		
Standard Flow Rate:	~0.5 mL/min (Dionex AutoPrep small loop, gravity)		
Concentrator:	Dionex IonPac UTAC-LP2, 3 × 35 mm		
Run Time:	Calibration Standards: 41.5 min; Samples and Check Standards: 51.5 min		
Background Conductance:	<1 µS		
Noise:	<1 nS		
System Backpressure:	~2200 psi		

* Equivalent or improved results can be achieved using a Thermo Scientific™ Dionex™ ICS-6000 HPIC™ system.

** The sample run times include loading and equilibration 21.5 min before the injection. Standards require less loading time, so the run time is 15 min before injection.



50 ppt (A) and 100 ppt (B) trace anion standards in ultrapure water using the Dionex AutoPrep system small loop.



Chemical Reagents

Chemical reagents, acids, bases and solvents are used to clean wafer surfaces and prepare the wafer for subsequent process steps. Trace metals in these reagents can potentially cause yield and device reliability problems. Ion chromatography allows the determination of parts per billion and even parts per trillion level concentrations of contaminants in chemical reagents. Microbore ion chromatography improves the detection limits for trace ions in concentrated reagents. Thermo Scientific™ Dionex™ solvent-compatible ion exchange columns permit reliable determination of trace ions in semiconductor solvents, such as isopropanol, acetone and methanol.

Applications in this section include those involving:

- Solvents
- Oxidizers
- Acids
- Bases
- Transition Metals

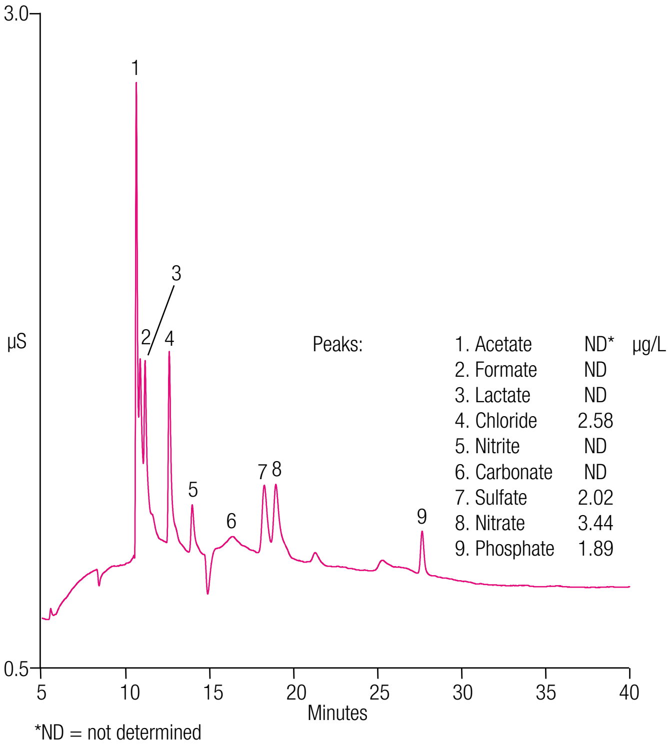
Solvents

Determination of Trace Anions in Organic Solvents Using Matrix Elimination and Preconcentration

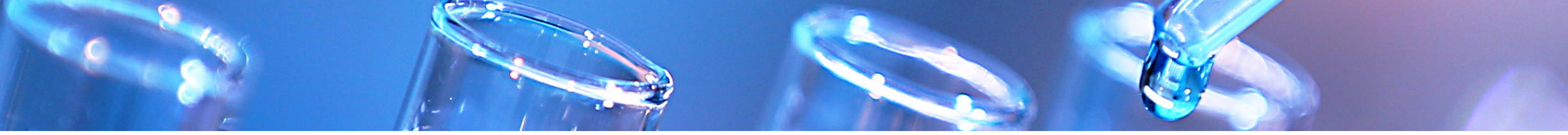
Anion contamination introduced during manufacturing processes can ruin semiconductors and computer components. Trace contaminants in solvents used during manufacturing can cause short circuits, defects in deposition, and corrosion. Component defects such as these reduce yields, which increases manufacturing costs and waste. As device geometries shrink, even lower levels of contamination become problematic. The computer and semiconductor industries need analytical methods to determine trace anions in solvents to help them identify contamination at different stages of manufacturing, so they can take action to prevent future contamination.

This update to AN85 describes an automated method that improves consistency and lowers detection limits, using the Dionex ICS-3000 DP Dual Pump, EG Eluent Generator, and DC Detector/Chromatography modules* with updated suppressor, trap, concentrator, and analytical column technology. This method successfully determines trace anions from high ng/L to low µg/L concentrations in isopropyl alcohol, methanol, acetone, and *N*-methyl-2-pyrrolidone, with improved method detection limits, from a 60% smaller sample injection.

*Equivalent or improved results can be achieved using the Thermo Scientific™ Dionex™ ICS-6000 HPIC™ system.



Chromatogram of a chloride, sulfate, nitrate, and phosphate standard, 2 µg/L each, in methanol.



Conditions	
Sample preparation: System 2	
Trap Column:	Thermo Scientific™ Dionex™ IonPac ATC-HC, 9 × 75 mm(P/N 059604)
Pump 2 Flow Rate:	2.0 mL/min
Carrier:	Degassed deionized water
Matrix Elimination:	10 mL[a]
Peristaltic Pump Flow Rate:	1.2 mL/min (AS-HV)
Inj. Volume:	2 mL
Sample Load Volume:	5 mL (Enter in Sequence)
Rinse Volume:	7 mL (Flush)
AS-HV Sample Load Mode:	Pull
Typical System Backpressure:	~900 psi
Prep. Time with Overlap:	11 min
Prep. Time without Overlap:	17 min

[a] 20 mL when determining anions in NMP mg/L stock standards.

Conditions	
Analytical: System 1	
Columns:	Thermo Scientific™ Dionex™ IonPac AG18 guard, 2 × 50 mm (P/N 060555) Dionex IonPac AS18 analytical, 2 × 250 mm (P/N 060553)
Flow Rate:	0.25 mL/min
Eluent:	22 mM KOH from -2 to 15 min, 22 to 48 mM KOH from 15 to 25 min, 65 mM KOH from 35 to 40 min
Eluent Source:	EGC II KOH
Column Temp.:	30 °C
Concentrator:	Thermo Scientific™ Dionex™ IonPac UTAC-ULP1, 5 × 23 mm (P/N 063475)
Detection:	Suppressed Conductivity, Dionex ASRS ULTRA II Anion Self-Regenerating Suppressor 2 mm, recycle mode (Replaced by Thermo Scientific™ Dionex™ AERS™ 500 (2 mm), P/N 082541), 41 mA
Carbonate Removal:	Dionex CRD 200 device, 2 mm (P/N 062986) installed between the suppressor and the detector
Typical Background:	<2 µS
Typical System Backpressure:	~2200 psi
Typical Noise:	<3 nS
Run Time:	33 min
Total Analysis Time:	44–50 min

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Oxidizers

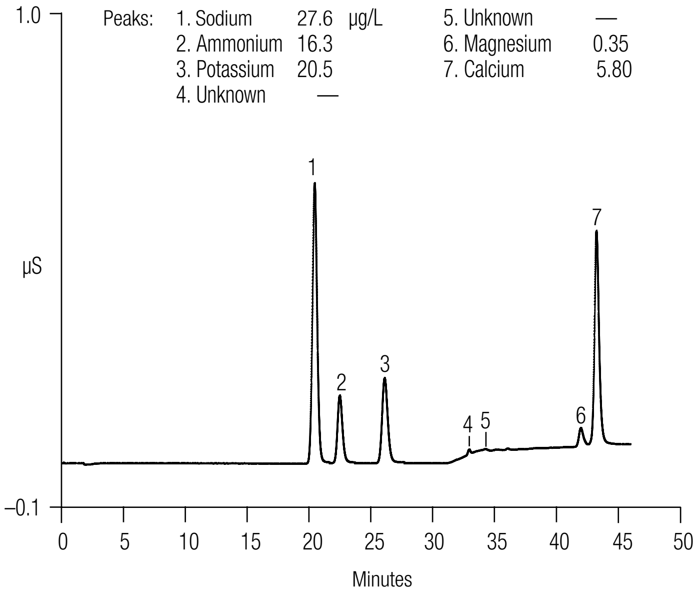
Determination of Cations and Amines in Hydrogen Peroxide by Ion Chromatography Using a RFIC (Reagent-Free) System

Hydrogen peroxide is an essential chemical in the fabrication of integrated circuit and microcircuit devices. Maximum allowable contaminant levels for semiconductor-grade hydrogen peroxide can be as low as <100 ppt (ng/L) per individual inorganic cation. A number of semiconductor manufacturers include specifications for maximum allowable levels of <1 ppb (µg/L) for trimethylamine and related amines.

This application note describes the use of a Thermo Scientific™ Dionex™ IonPac™ CS17 column to determine trace cations and amines in hydrogen peroxide with a large-loop injection. The Dionex IonPac CS17 column separates amines without the organic solvent eluent modifier needed for separating amines when using older cation-exchange IC columns. Using an RFIC system, this application easily determines sub-µg/L concentrations of cations and amines.

Conditions

Column:	Dionex IonPac CS17 4 × 250 mm (P/N060557)
Guard:	Dionex IonPac CG17 4 × 50 mm (P/N 060560)
Eluent:	Dionex EGC-MSA, 0.8 mM isocratic to 28.5 min, gradient to 4 mM at 37 min, gradient to 8 mM at 50 min
Flow Rate:	1 mL/min
Temperature:	30 °C
Detection:	Suppressed conductivity, Thermo Scientific™ Dionex™ Cation Atlas™ Electrolytic Suppressor (CAES™), recycle mode
Inj. Volume:	1 mL
Background:	<1 µS



RFIC Analysis of hydrogen peroxide Sample #1.

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Acids

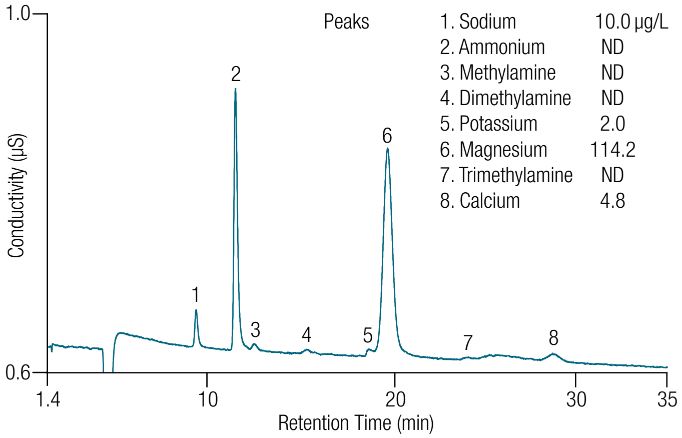
Determination of Trace Cations in Concentrated Acids Using AutoNeutralization Pretreatment and Ion Chromatography

Determination of cations in concentrated acids is important because some cations can combine with anionic contaminants to form insoluble compounds that can cause plating and etching defects in electronics manufacturing processes.

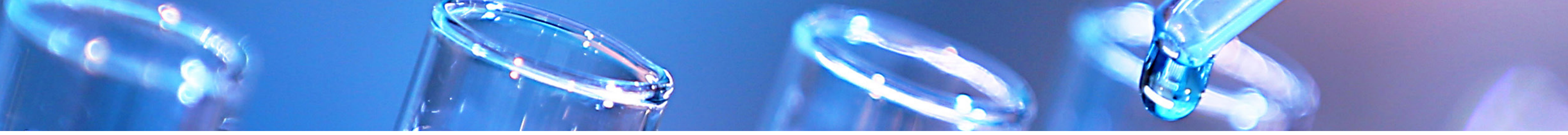
Ion chromatography with suppressed conductivity detection is the preferred method for determining ionic species, especially at µg/L and ng/L concentrations. Direct injection of concentrated acid samples overloads an IC column with the acid cation, resulting in poor chromatography, making it difficult or impossible to quantify cations. AutoNeutralization eliminates the need to dilute the concentrated acid prior to analysis and allows µg/L and ng/L anion determinations. This technique neutralizes the strong acid or base in a sample.

In this application note, AutoNeutralization is performed using dual pumps, detectors, and valves of the Thermo Scientific™ Dionex™ ICS-3000* IC system for easier configuration without the need for external pumps or valves. The system configurations, plumbing, valve operations, and program are described.

*Equivalent or improved results can be achieved using the Thermo Scientific™ Dionex™ ICS-6000 HPIC™ system.



Trace levels of cations in 10% hydrofluoric acid using AutoNeutralization.



Conditions

Sample preparation:	*System 2*
Trap Column:	Dionex IonPac CTC-1, 9 × 24 mm (P/N 040192)
Flow Rate:	0.5 mL/min
Carrier:	Degassed deionized water
Inj. Volume:	100 µL
Neutralization:	Dionex CSRN II Cation Self-Regenerating Neutralizer, external water mode, 500 mA. (Replaced by Thermo Scientific™ Dionex™ CERS™ 500e Cation Electrolytically Regenerated Suppressor for External Water Mode (4 mm) P/N 302663)
Run Time:	1.4 min for AutoNeutralize and Concentrate (needs to be determined experimentally)
Typical System Backpressure:	~800 psi

Analytical:	*System 1*
Columns:	Dionex IonPac CG16 guard (5 × 50 mm, P/N 057574) and Dionex IonPac CS16 analytical (5 × 250 mm, P/N 079805)
Flow Rate:	1.0 mL/min
Eluent:	28 mM methanesulfonic acid
Eluent Source:	Dionex EluGen EGC II MSA
Temperature:	30 °C
Concentrator:	Thermo Scientific™ Dionex™ IonPac™ TCC-ULP1, 5 × 23 mm (P/N 063783)
Detection:	Suppressed Conductivity, Dionex CSRS ULTRA II Cation Self-Regenerating Suppressor, recycle mode, 82 mA
Typical Background:	<0.5 µS
Typical System Backpressure:	~2200 psi
Typical Noise:	<0.5 nS
Total Run Time:	35 min
Rinse Program:	
System 2:	Same conditions without Concentrate
System 1:	Bypassed
Inj. Valve 1:	Inject position
Total Run Time:	10 min

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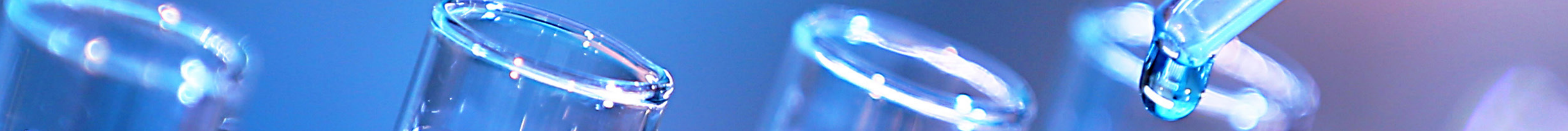


Determination of Trace Anions in Concentrated Hydrofluoric Acid

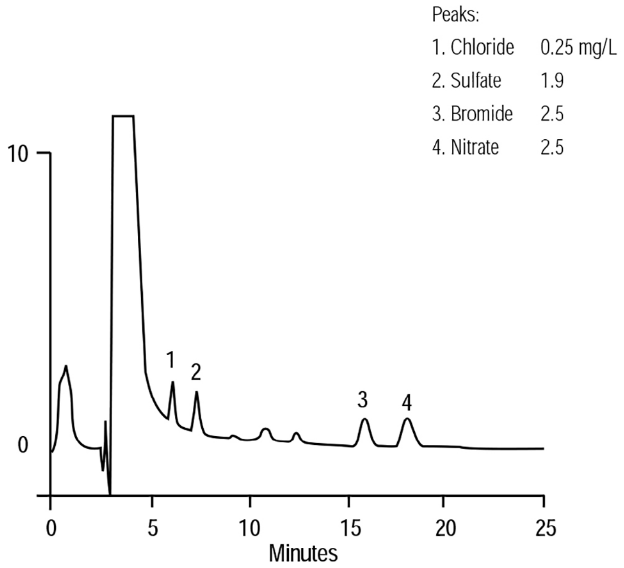
The determination of trace anions in concentrated hydrofluoric acid has been a difficult analytical challenge. In the past, labor-intensive manual preconcentration methods were required prior to analytical measurements. Typical preconcentration procedures involved evaporation of a specific large volume of sample for 6 to 10 hours on a hot plate before transfer to a volumetric flask and analysis. Also, a class-100 clean room environment was normally required during sample pretreatment.

In this application note, trace anions were separated from the high concentration of fluoride by matrix elimination using a Thermo Scientific™ Dionex™ IonPac™ AC10 concentrator column prior to analytical separation. The retained anions of interest were eluted from the Dionex IonPac AC10 concentrator and separated on a Thermo Scientific™ Dionex™ IonPac™ AS10 column. The detection limits (MDL) using this method are 25 to 50 ppb for most trace anions in 5% hydrofluoric acid.

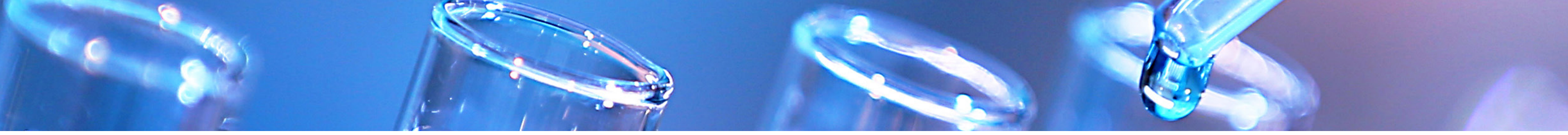
Conditions	
Columns:	Dionex IonPac AS10 (2 mm) analytical Dionex IonPac AG10 (2 mm) guard Dionex IonPac AC10 (2 mm) concentrator Thermo Scientific™ Dionex™ IonPac™ ATC-1 trap (2 mm), 2 required
Eluent 1:	Deionized water
Eluent 2:	400 mM Sodium hydroxide
Rinsing Reagent:	70% v/v Methanol
Eluent Flow Rate:	0.25 mL/min
Rinsing Flow Rate:	1.0 mL/min
Injection Volume:	10 mL
Detection:	Suppressed conductivity
Regenerant:	50 mN Sulfuric acid (needed for AMMS only)
Suppressor:	Thermo Scientific™ Dionex™ AMMS (2 mm) or Thermo Scientific™ Dionex™ Anion Self-Regenerating Suppressor (ASRS) (2 mm)
Regenerant Flow Rate:	10 mL/min

Gradient Program:

Time	*E1*	*E2*	*V5*	*V6*
0.0	75	25	0	0
2.5	75	25	1	0
12.0*	75	25	0	1



Determination of trace anions in 5% hydrofluoric acid by IC.

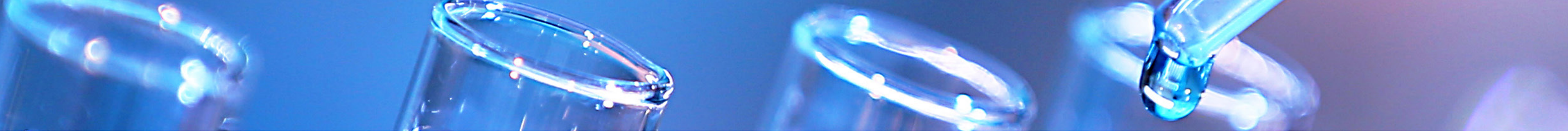


Determination of Trace Anions in Hydrofluoric Acid, Ammonium Fluoride, and a Buffered Oxide Etchant

Concentrated hydrofluoric acid (HF) is used in the semiconductor and electronics industries, both alone and as one component of a buffered oxide etchant (BOE), to remove oxide layers during device production. This HF must be of high purity, especially with respect to anionic impurities that can damage the semiconductor (e.g., chloride and sulfate). In 1997, Semiconductor Equipment and Materials International (SEMI) specified that HF must have chloride and sulfate concentrations <200 µg/L and nitrate and phosphate concentrations <100 µg/L. Simply diluting the HF to a concentration that will not overload the anion-exchange column does not allow enough sensitivity to determine the contaminating anions.

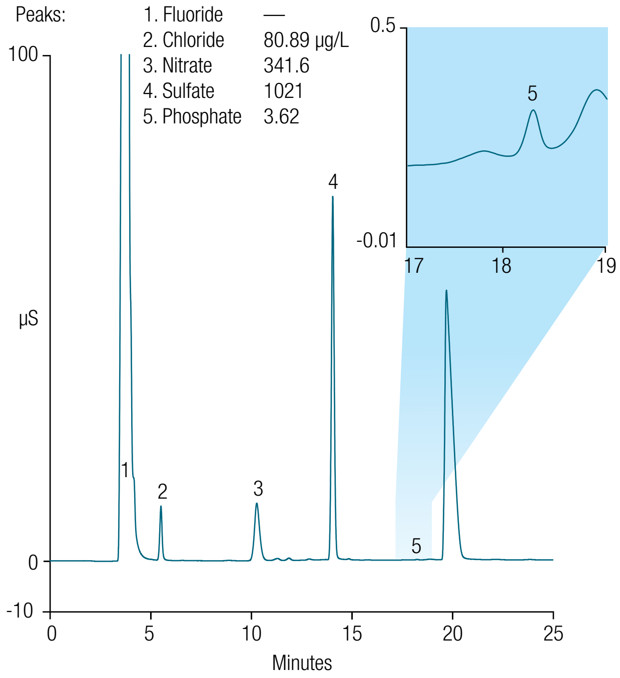
This technical note describes determination of low concentrations of strong acid anions in HF. The method design also allows determination of low concentrations of strong acid anions in an HF/ammonium fluoride mixture (BOE), and ammonium fluoride.

Conditions	
Ion Exclusion	
Column:	Dionex IonPac ICE-AS1, 9 × 250 mm (P/N 043197)
Eluent:	Deionized water
Trap Column:	Dionex ATC-HC, 9 × 75 mm (P/N 059604)
Flow Rate:	See Table 4
Ion Chromatography	
Analytical Column:	Dionex IonPac AS11-HC, 2 × 250 mm (P/N 052961)
Guard Column:	Dionex IonPac AG11-HC, 2 × 50 mm (P/N 052963)
Concentrator Column:	Dionex IonPac AG11-HC, 4 × 50 mm (P/N 053962)
Eluent Source:	Dionex EGC II KOH cartridge (This has been replaced by the Dionex EGC III KOH cartridge, P/N 074532) with Thermo Scientific™ Dionex™ CR-ATC trap column (P/N 060477)
Gradient:	See Table 4
Flow Rate:	0.38 mL/min
Sample Volume:	500 µL
Column Temperature:	30°C
Pressure:	~2100 psi
SRS Current:	30 mA
Detection:	Suppressed conductivity, Thermo Scientific™ Dionex™ Anion Self-Regenerating Suppressor™ ASRS™ 300 (P/N 64555), 2 mm, external water mode

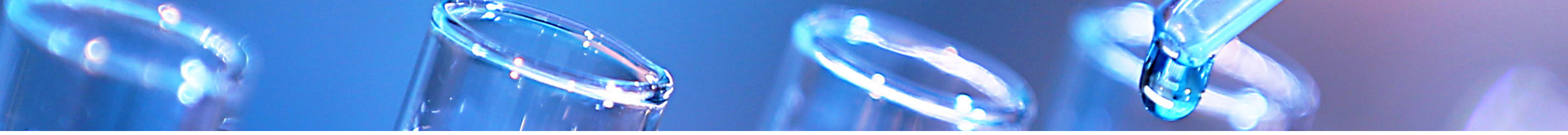


Sample preparation, analysis, and ICE column cleaning program description.

Program	Time (min)	Flow Rate Pump 2	Injection Valve Position	10-Port Valve Position	Eluent Concentration (mM)	Remark
Sample Preparation	Init	0.5	Load	A	8	Dionex AS Autosampler starts loading sample to sample loop.
	0	0.5	Inject	A	8	Start fraction collection by flushing sample to concentrator.
	11	Off	Inject	A	8	End fraction collection by switching off pump 2.
Analysis and ICE Column Cleaning	Init	Off	Load	B	8	Dionex AS Autosampler starts loading 25% formic acid to the sample loop for ICE column cleaning.
	0	0.8	Inject	B	8	Start ICE column cleaning and sample analysis. The ICE column is cleaned by flushing 25% formic acid from the sample loop to the column.
	7	0.8	Inject	B	8	
	15	0.8	Inject	B	8	
	30	0.8	Inject	B	30	
	35	0.5	Inject	B	30	
	40	0.5	Inject	B	8	



Determination of anions in ammonium fluoride.

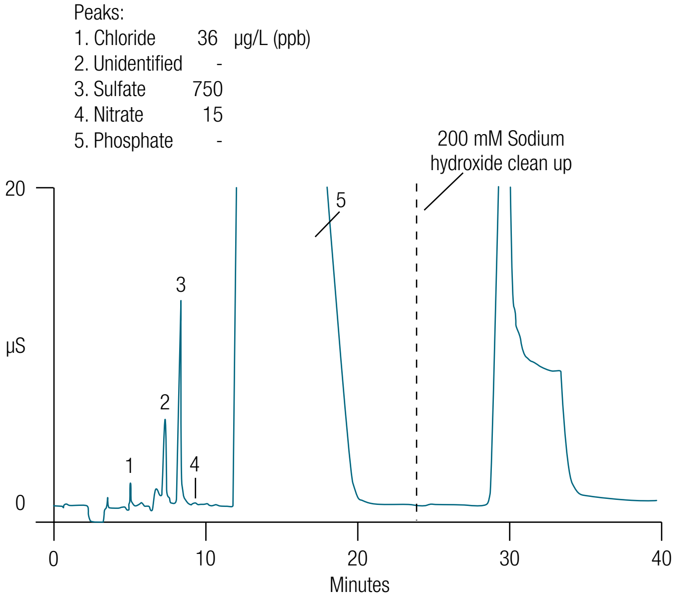


The Determination of Trace Anions in Concentrated Phosphoric Acid

The determination of trace anions in phosphoric acid is hampered by a large excess of phosphate ion. Diluting the concentrated sample overcomes the problem of a large concentration of the interfering matrix ion, but lacks the required sensitivity for the contaminant ions of interest. An improved method for determining trace anions in concentrated phosphoric acid has been developed to overcome this problem. Trace inorganic anions are separated from the high concentration of phosphate by an ion-exclusion separation prior to an ion-exchange separation.

This technical note describes the theory, set up, and analytical procedure for the determination of chloride, nitrate, and sulfate at sub-mg/L (ppm) levels in 85% (w/w) phosphoric acid.

Conditions	
Ion Exclusion	
Column:	Dionex IonPac ICE-AS6
Trap Column:	Dionex IonPac AG10, 4 mm
Eluent:	Deionized water
Flow Rate:	0.50 mL/min
Ion Chromatography	
Analytical Column:	Dionex IonPac AS11-HC, 2 mm
Guard Column:	Dionex IonPac AG11-HC, 2 mm
Concentrator Column:	Dionex IonPac AG11-HC, 4 mm
Eluent:	20 mM sodium hydroxide, step to 200 mM sodium hydroxide
Flow Rate:	0.38 mL/min
Sample Volume:	200 µL
Detection:	Suppressed conductivity, Dionex ASRS, AutoSuppression™ external water mode
Suppressor Current Setting:	300 mA
Expected System Backpressure:	16.5 MPa (2400 psi) (with concentrator column in line)
Expected Background Conductivity:	2–3 µS



Determination of trace anions in high purity 85% phosphoric acid.

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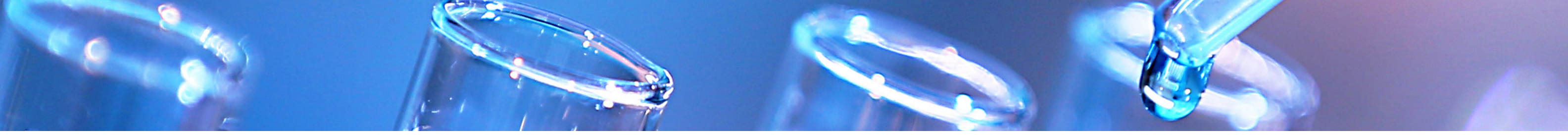
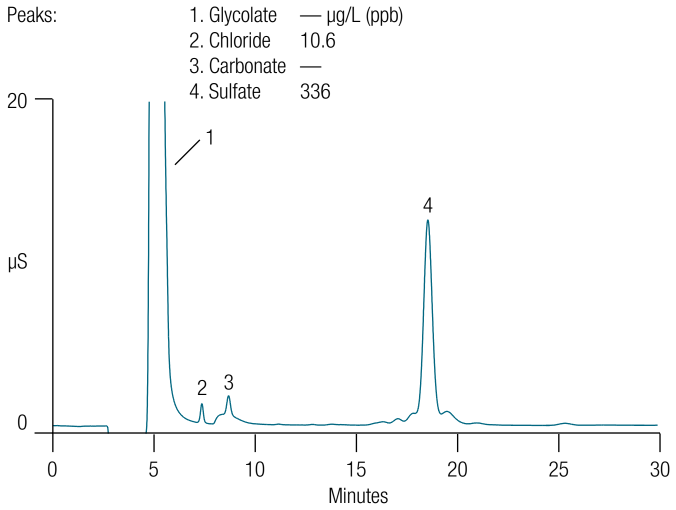


Determination of Trace Anions in Concentrated Glycolic Acid

This technical note describes a reliable method to determine trace chloride and sulfate in glycolic acid. The presence of these ions in glycolic acid that is used for soldering fluxes can corrode electronic parts. Determination of these ions is hampered by the large excess of glycolate ion. Diluting the concentrated sample reduces the concentration of the interfering matrix ion, but results in a lack of sensitivity for the contaminant ions of interest. An improved method for determining trace anions in concentrated weak acids has been developed to overcome this problem. Trace inorganic anions of strong acids are separated from the high concentration of glycolate by an ion exclusion separation prior to an ion exchange separation.

This technical note covers the theory, set up, and analytical procedure for the determination of trace chloride and sulfate at sub-mg/L (ppm) levels in 0.71–7.5% (v/v) glycolic acid.

Conditions	
Ion Exclusion	
Analytical Column:	Dionex IonPac ICE-AS6
Trap Column:	Dionex IonPac AG10, 4 mm
Eluent:	Deionized water
Flow Rate:	0.55 mL/min
Ion Chromatography	
Analytical Column:	Dionex IonPac AS9-HC, 2 mm
Guard Column:	Dionex IonPac AG9-HC, 2 mm
Concentrator Column:	Dionex IonPac AG9-HC, 4 mm
Eluent:	8 mM Sodium carbonate 1.5 mM Sodium hydroxide
Flow Rate:	0.25 mL/min
Sample Volume:	750 µL
Detection:	Suppressed conductivity, Dionex ASRS, AutoSuppression™ external water mode
Suppressor Current Setting:	100 mA
Expected System Backpressure:	13.8 MPa (2000 psi) (with concentrator column in line)
Expected Background Conductivity:	20 µS



Trace anions in high purity glycolic acid.

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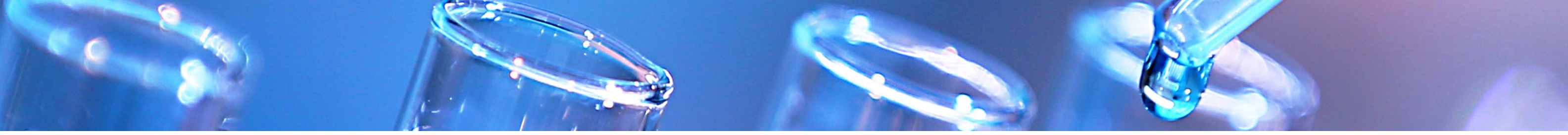
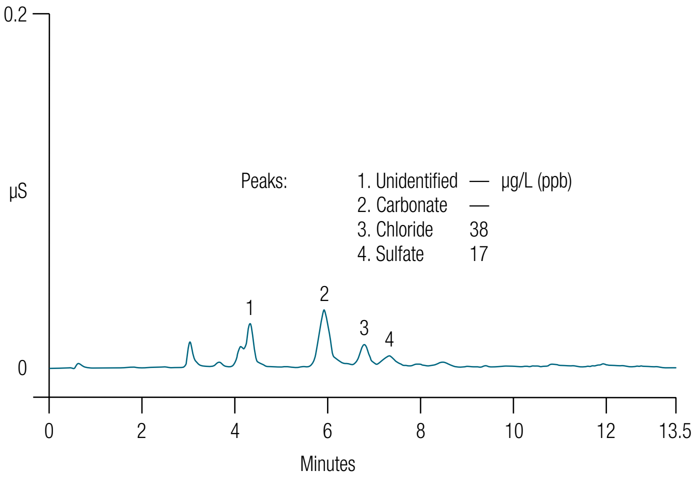


Determination of Trace Anions in High-Nitrate Matrices by Ion Chromatography

Chloride contamination, present in the nitric acid used to etch wafers, can significantly compromise the yield and reliability of semiconductors. Traditional wet chemical methods are neither sensitive nor reliable enough for these demanding trace-level anion determinations. A direct injection method was developed to address this need using a microbore Thermo Scientific™ Dionex™ IonPac™ AS15 column. This application note covers the theoretical background, setup, and analytical procedure for a method to determine chloride, sulfate, and phosphate to low-µg/L (ppb) levels in 0.7% (v/v) nitric acid and other matrices that contain high concentrations of nitrate ion.

Conditions	
Columns:	Dionex IonPac AS15 Analytical, 2 × 250 mm Dionex IonPac AG15 Guard, 2 × 50 mm
Trap Column:	Dionex IonPac ATC-1 Anion Trap Column, 4 mm Dionex IonPac ATC column, 2 mm
Eluent:	Potassium hydroxide (Dionex EG40 Eluent Generator as the source)
Temperature:	Ambient
Dionex EG40 Offset Volume:	0 µL
Pump Program:	See Table 1
Eluent Flow Rate:	0.2 mL/min
Detection:	Suppressed conductivity, Thermo Scientific™ Dionex™ ASRS™ ULTRA Suppressor, AutoSupression External Water Mode or Gas Assisted Recycle Mode
Dionex SRS Suppressor Current Setting:	50 mA
Expected Background Conductivity:	1 µS (48 mM KOH)
Expected System Backpressure:	17.2 MPa (2,500 psi)
Sample Volume:	1 µL



Trace anions in 7000 ppm sodium nitrate.

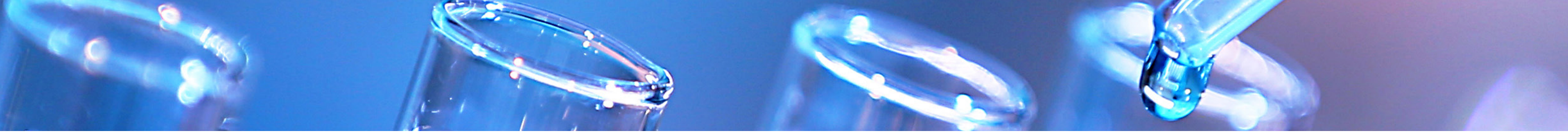


Table 1. Pump program.

Time (min)	Flow (mL/min)	A (%)	Inj. Valve	Column Valve	KOH Conc. (mM)	Comments
0.00	0.20	100	Load	A*	48	48 mM KOH; RDM “On”; Load Sample Loop
4.30	0.20	100	Load	B**	48	RDM “Off”
5.00	0.20	100	Inject	B	48	Inject
20.00	0.20	100	Inject	B	48	End analytical separation
20.01	0.20	100	Inject	B	100	Begin Cleanup step
40.00	0.20	100	Inject	B	100	End Cleanup step
40.01	0.20	100	Inject	B	48	Begin equilibration 48 mM
60.00	0.20	100	Inject	B	48	End

*A = Inject
**B = Load

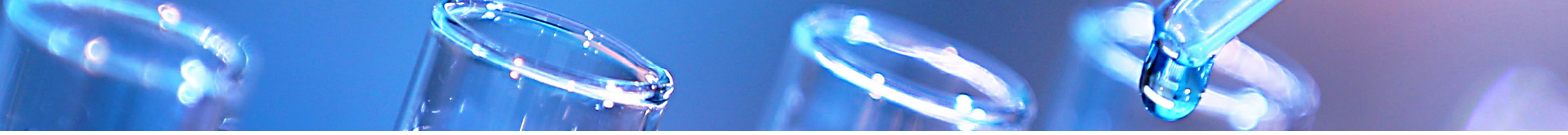
Bases

Determination of Trace Anions in Basic Solutions by Single Pass AutoNeutralization and Ion Chromatography

The electronics industries need analytical methods to determine trace anions in concentrated bases. Anions at very low concentrations can cause corrosion, deposition defects, and electronic shorts in electronic devices. Ion chromatography (IC) with suppressed conductivity detection is the preferred and well-established method for determining ionic species, especially at µg/L and ng/L concentrations. Direct analysis of concentrated base samples is challenging because the high concentrations of the base anion overloads the column, resulting in poor chromatography and quantification. Diluting the concentrated bases prior to anion analysis reduces the column overload issues. However, this dilution sacrifices anion determinations at µg/L and ng/L concentrations.

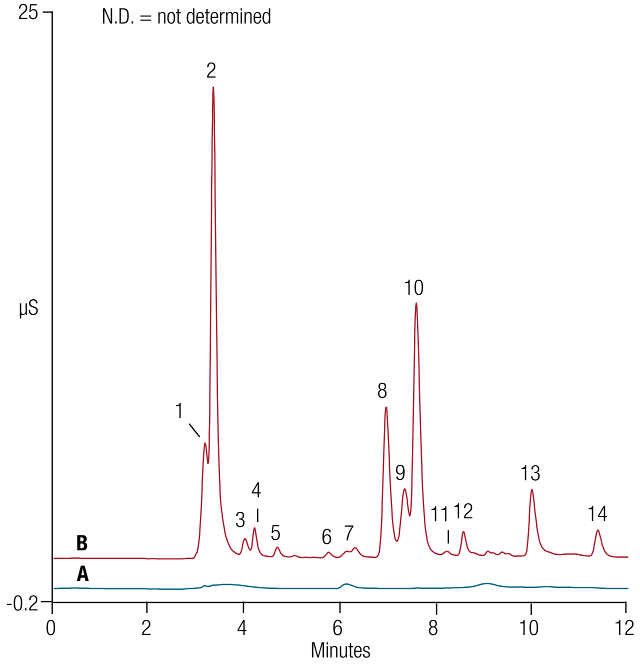
This application note discusses a Single Pass AutoNeutralization method that uses the high static capacity of a 4 mm Thermo Scientific™ Dionex™ AERS™ 500 Anion Electrolytically Regenerated Suppressor to neutralize the base samples in a single pass.

Conditions	
Columns:	Dionex IonPac AG18-4µm, 2 × 30 mm Dionex IonPac AS18-4µm, 2 × 150 mm
Gradient:	KOH gradient, 23 mM KOH (-3–3.8 min), 23–45 mM (3.8–5.5 min), 45 mM (5.5–8 min), 45–23 mM (8.0–8.1 min), 23 mM (8.1–12 min)
Eluent Source:	Dionex EGC 500 KOH Eluent Generator cartridge, Dionex CR-ATC 500 Anion Trap Column (CR-ATC 600 for the Integrion IC system) and high pressure degas module
Flow Rate:	0.25 mL/min
Injection Vol.:	100 µL
Column Temp.:	30 °C
Sample Prep.	
Transfer Water:	Dionex AXP pump at 1 mL/min (Figures 3–7, 9, 10) with Dionex IonPac ATC-HC 500 Anion Trap Column, 9 × 75 mm Dionex AXP pump at 0.5 mL/min (Figures 7 and 8) with Dionex IonPac ATC-HC 500 Anion Trap Column, 9 × 75 mm
Auto-Neutralization:	Single pass, Dionex AERS 500 Anion Electrolytically Regenerated Suppressor, 4 mm, recycle mode
Neutralizing Suppressor Current:	A: 300 mA for amine solutions (Figures 3–7, 9, 10) B: 490 mA for strong base solutions (Figures 7 and 8)
Concentrator:	Dionex UTAC-LP2, 4 × 35 mm
Compartment Temp.:	15 °C
Detection:	Suppressed conductivity, Dionex AERS 500 Anion Electrolytically Regenerated Suppressor, 2 mm, 28 mA, recycle mode
Run Time:	12.5 min
Background conductance:	< 1 µS
Noise:	~ 1 nS
System Backpressure:	~ 3,000 psi



Peaks:	A	B	
1. Acetate	N.D	8. Bromide	564 µg/L
2. Formate	N.D.	9. Unknown	–
3. Chloride	23.1 µg/L	10. Sulfate	515
4. Unknown	–	11. Nitrate	N.D.
5. Unknown	–	12. Unknown	–
6. Unknown	–	13. Phosphate	354
7. Carbonate	N.D.	14. Unknown	–

N.D. = not determined



Trace anions in Deionized water (A) and 0.1 M tetrabutylammonium hydroxide (TBAOH) ACS grade solution (B).

Download the full application note

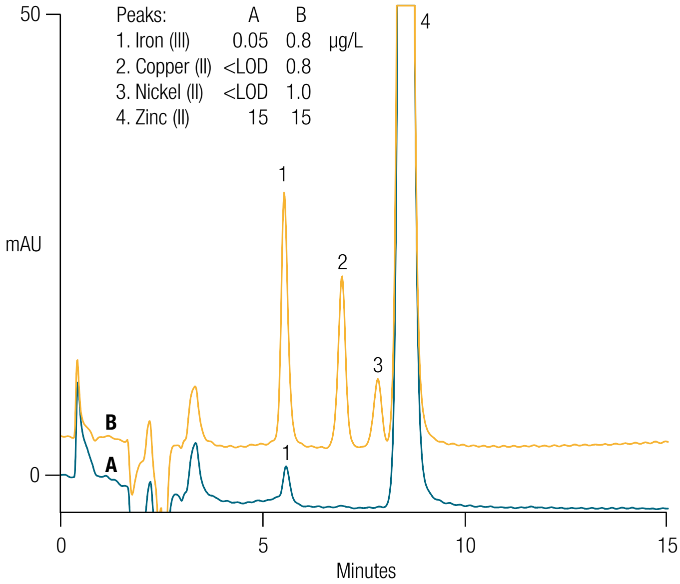
Transition Metals

Fast and Sensitive Determination of Transition Metals in Power Industry Waters Using Ion Chromatography

This application note focuses on the analysis of power plant waters, but the method described here is relevant to other industries, such as semiconductor fabrication, where determination of transition metals is a critical part of the manufacturing process.

In this application note an IC method with 530 nm absorbance detection was evaluated for the determination of iron (III), copper (II), nickel (II), and zinc (II) at sub- and low-µg/L concentrations in boiling water and the primary cooling circuit of pressurized water nuclear power plant reactors. The Thermo Scientific™ Dionex™ IonPac™ CS5A column with a pyridine-2,6-dicarboxylic acid (PDCA) eluent allowed the separation of the four target transition metals in less than 10 min, even in the presence of high concentrations of boric acid and lithium that constitute the pressurized water reactor (PWR) primary coolant surrogate matrix. Excellent recoveries were achieved for the target analytes in DI water and boric acid/lithium hydroxide matrices, with and without additional zinc added.

Conditions	
Columns:	Dionex IonPac CG5A, 2 × 50 mm Dionex IonPac CS5A, 2 × 250 mm
Eluent:	7.0 mM Pyridine-2,6-dicarboxylic acid (PDCA) 66 mM Potassium hydroxide 5.6 mM Potassium sulfate 74 mM Formic acid
Flow Rate:	0.3 mL/min
Concentrator:	Dionex IonPac TCC-2
Vol. Conc.:	4.7 mL
Column Temp.:	30 °C
Postcolumn:	0.24 mM 4-(2-Pyridylazo) resorcinol (PAR) 1.0 M 2-Dimethylaminoethanol 0.50 M Ammonium hydroxide 0.30 M Sodium bicarbonate
PCR Flow Rate:	0.15 mL/min (40–60 psi)
PC Mixer:	125 µL Knitted reaction coil, 2 mm
Detection:	Absorbance at 530 nm
Noise:	~0.12 mAU/min
Backpressure:	2150 psi
Run Time:	20 min



Chromatogram of transition metals in surrogate PWR matrix (2,000 mg/L B + 3.8 mg/L Li + 15 µg/L Zn^{2+}). Trace A: PWR surrogate matrix. Trace B: PWR surrogate matrix spiked with 0.8 µg/L Fe^{3+}, Cu^{2+}, and 1.0 µg/L Ni^{2+}.

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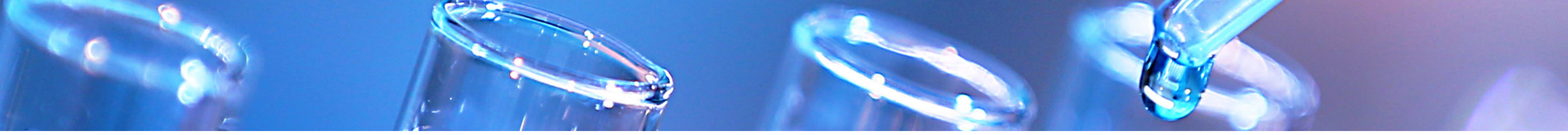
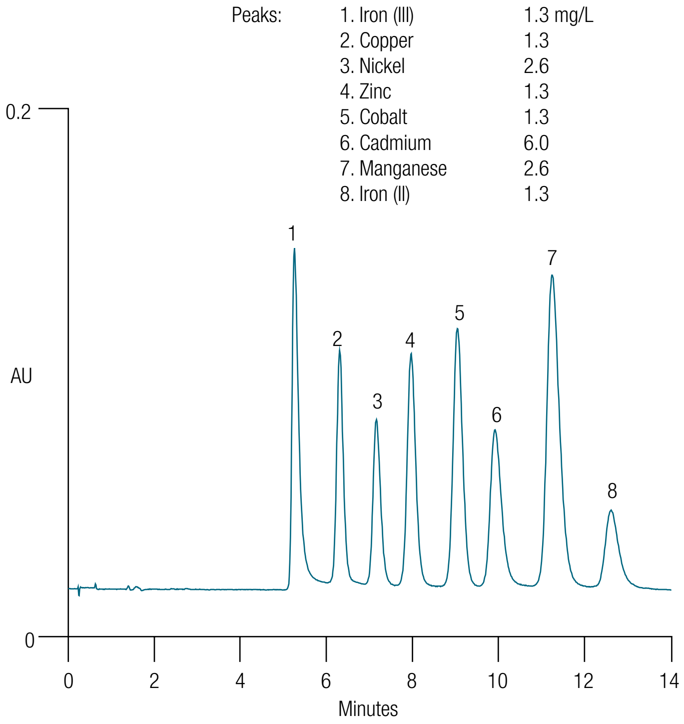


Determination of Transition Metals by Ion Chromatography

Metal ions can exist in several different forms. The factors that determine the form of the metal ion are the extent of complexation and the oxidation state. In many samples, metal ions are present in their hydrated forms. Hydrated metal ions can also be complexed by weak ligands such as organic acids or amino acids. These ligands are generally displaced by the complexing agents used in IC eluents. In this application note, cation-exchange chromatography and with postcolumn reaction and UV detection are used to determine the total of both hydrated and weakly complexed metal ions.

Conditions, Method A	
Columns:	Dionex IonPac™ CS5A Analytical and CG5A Guard
Eluent:	Dionex MetPac PDCA eluent
Flow Rate:	1.2 mL/min
Inj. Volume:	50 µL
Mixing Device:	375 µL knitted reaction coil
Postcolumn Reagent:	0.5 mM PAR, in Dionex MetPac PAR Postcolumn Diluent
Reagent Flow Rate:	0.7 mL/min
Detector Wavelength:	530 nm



Separation of transition metals using Dionex IonPac CS5A column with PDCA, method A conditions.

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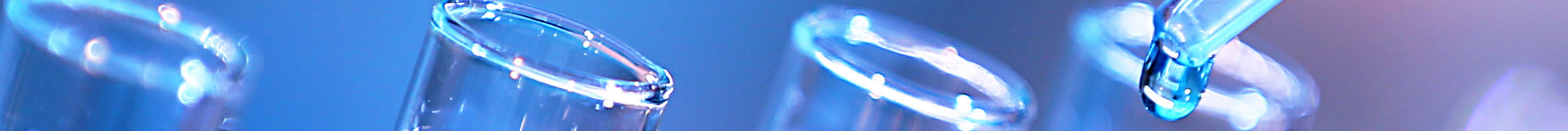


Determination of Trace Transition Metals in Reagent Grade Acids, Bases, Salts, and Organic Solvents Using Chelation Ion Chromatography

The SEMI (Semiconductor Equipment and Materials International) specifications for maximum permitted levels of transition metals in concentrated acids used in fabrication of semiconductor devices is in the range of 0.1 to 1.0 part per billion (ppb). Labor-intensive manual preconcentration methods are usually required prior to analytical measurements. The chelation ion chromatographic technique (chelation IC) provides the sample preconcentration step and direct determination of trace transition metals by ion chromatographic separation and postcolumn derivatization prior to detection.

This application note describes a method for the determination of trace transition metals in trace metal grade reagents by chelation IC. The detection limits for most elements present in concentrated trace-metal grade reagents are in the sub-part-per-billion range.

Chelation concentration–transition metals

Columns:	Dionex MetPac CC-1, Dionex TMC-1
Eluents:	E1: $H_2$0 E2: 2.0 M ammonium acetate, pH 5.4 ± 0.1 E3: 2.0 M nitric acid E4: 0.10 M ammonium nitrate, pH 3.5 ± 0.3

Gradient program, system 1

t (min)	*%E1*	*%E2*	*%E3*	*E4*	*V5*	*V6*	*Flow (mL/min)*
0.0	0	100	0	0	1	0	3.0
0.1	0	100	0	0	1	1	3.0
2.5	0	100	0	0	1	1	3.0
2.6	72	0	28	0	0	1	3.0
5.0	72	0	28	0	0	1	1.0
5.1	0	0	0	100	0	0	3.0
6.6	0	0	0	100	1	0	1.0
6.7	0	0	100	0	1	1	3.0
7.7	0	0	100	0	1	1	3.0
7.8	0	100	0	0	1	1	3.0
9.3	0	100	0	0	1	1	3.0
9.4	100	0	0	0	1	1	0.0

Gradient program, system 2

t (min)	*%E1*	*%E2*	*%E3*	*E4*	*V5*	*V6*	*Flow (mL/min)*
0.0	0	100	0	0	1	0	3.0
2.0	0	100	0	0	0	1	2.0
5.0	0	100	0	0	1	0	3.0
7.0	0	100	0	0	1	0	1.2
7.1	50	0	50	0	1	1	1.2
12.0	50	0	50	0	1	1	1.2
12.1	0	0	0	100	1	1	2.0
13.0	0	0	0	100	0	0	3.0
15.0	0	0	0	100	0	0	3.0
15.1*	0	0	100	0	1	0	4.0
16.0	0	0	100	0	1	0	4.0
17.0	0	0	100	0	1	0	4.0
18.0	0	100	0	0	1	0	0.0

*Begin sample analysis

Analytical chromatography–transition metals

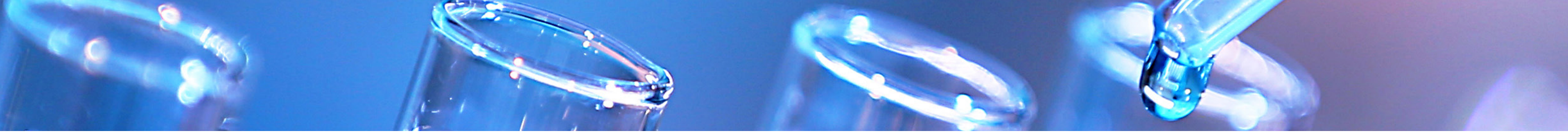
Column:	Dionex IonPac CS5
Eluent:	0.0060 M pyridine-2,6-dicarboxylic acid, 0.090 M acetic acid, 0.040 M sodium hydroxide or 0.050 M oxalic acid, 0.095 M lithium hydroxide
Eluent Flow Rate: 1.0 mL/min	

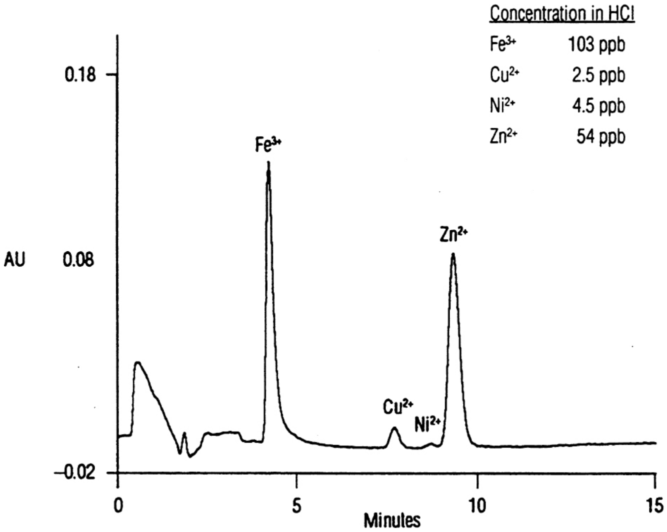
Postcolumn derivatization

Reagent:	4×10^{-4} M 4-(2-pyridylazo) resorcinol 1.0 M 2-dimethylaminoethanol 0.50 M ammonium hydroxide 0.30 M sodium bicarbonate
Reagent Addition:	Membrane reactor or mixing tee
Reagent Flow Rate:	0.5 mL/min
Reactor:	Packed or knitted reaction coil

Detection

Detector:	Visible absorbance, VDM or UDM
Wavelength:	520 or 530 nm
Time Constant:	1 s





Trace metals in hydrochloric acid.

Download the full application note

Plating Baths

Plating baths for copper electrochemical deposition (ECD) need to be of well controlled composition and free of contaminants to ensure a metal deposition with the correct characteristics. Copper thickness, crystal orientation and grain size need to be optimized to ensure the correct conductance and to minimize degradation due to electromigration. Thermo Scientific Dionex instrumentation can determine concentrations of additives, bath components, and bath contaminants using on-line or through grab sampling to assure the quality of the plating deposition.

The applications described in this section include:

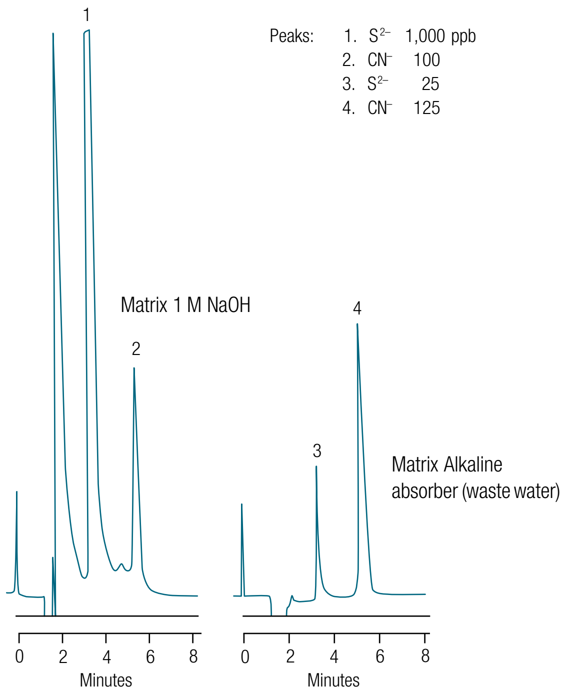
- Contaminants
- Additives

Contaminants

Determination of Cyanide in Strongly Alkaline Solutions

Determination of total cyanide in water is usually done by refluxing the sample in an acid digest and trapping the liberated HCN gas in a strongly alkaline-absorbing solution. Most methods for analyzing the trapping solution have an upper pH limit of 12.5 to 13. The direct injection method described in this application update can determine cyanide in solutions ranging up to pH 14. This allows absorbing solutions (such as the 1.25 M NaOH solution specified in U.S. EPA Method 335.2) to be quickly analyzed without dilution or other pretreatment. In addition, the technique is not subject to as many interferences as titrimetric or spectrophotometric methods, and it is well suited to automated analysis.

Conditions	
Columns:	Thermo Scientific™ Dionex™ IonPac AS7
Eluent:	0.5M Sodium acetate 0.1M Sodium hydroxid 0.5% Ethylenediamine
Flow Rate:	1 mL/min
Detector:	Thermo Scientific™ Dionex™ ED40 detector, silver working electrode, 0.00 V vs. Ag/AgCl reference



Cyanide in strongly alkaline solutions.

Download the full application update

For a more modern approach to cyanide determinations that may be applicable, download Application Note 227—Determination of Total Cyanide in Municipal Wastewater and Drinking Water Using Ion-Exclusion Chromatography with Pulsed Amperometric Detection.

Additives

Determination of Chloride in Acid Copper Plating Bath

Ion chromatography provides a convenient method for the determination of chloride in acid copper plating baths. These baths are used for deposition of copper on semiconductor wafers. Monitoring chloride concentration is of interest because it plays an important role in the quality of copper deposition. Compared to standard wet chemical techniques, IC offers improved speed and accuracy for this analysis.

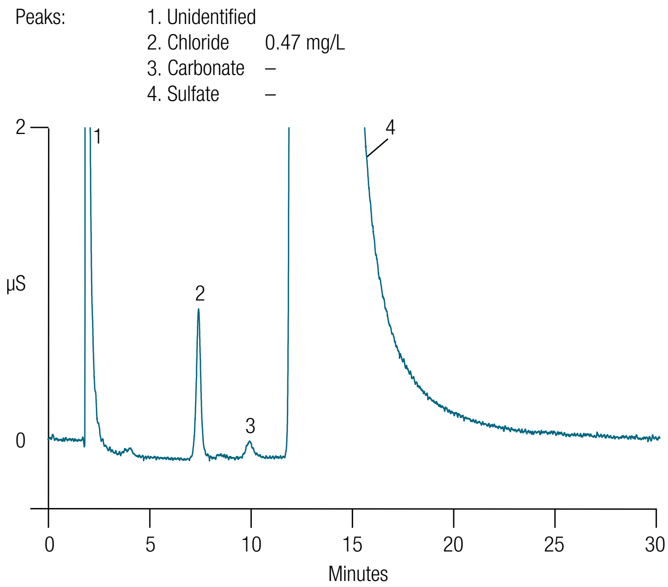
This application update describes the setup and procedure to determine chloride in a copper plating bath using the Thermo Scientific™ Dionex™ IonPac™ AS15 column.

Conditions	
Columns:	Dionex IonPac AG15, AS15, 4 mm
Eluent:	30 mM Potassium hydroxide
Eluent Source:	Dionex EG40
Flow Rate:	1.2 mL/min
Sample Volume:	25 µL of copper plating bath diluted 1:100
Detection:	Suppressed conductivity, Recycle mode
Dionex EG40 Offset Volume:	0 µL

Pump Program:

Time (min)	*Flow (mL/min)*	*%A*	*Valve*	*EG40 Conc (mM)*	*Comments*
Initial	1.2	100	Load	30	Load sample loop
0.00	1.2	100	Inject	30	Inject
30.00	1.2	100	Load	30	End acquisition

Dionex ASRS Current Setting:	100 mA
Expected Background Conductivity:	1 µS
Expected System Backpressure:	16.5 MPa (2,400 psi)



Determination of chloride in acid copper plating bath.

Determination of Anionic Fluorochemical Surfactant in Semiconductor Etch Bath

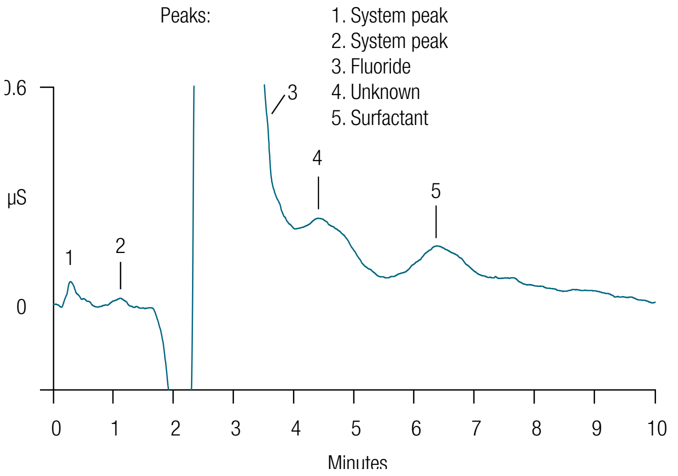
Perfluorinated surfactants are used as wetting agents in semiconductor acid etching solutions. Acid etching engraves fine patterns in silicon dioxide. Poor wetting of the wafer surface by the acid etchant during semiconductor device fabrication can result in air entrapment through the formation of small bubbles. These bubbles can mask a portion of the area to be etched, which can result in bridging over of the fine openings in the resistor surface and electrical shorts in the device. The addition of a small amount of surfactant can eliminate air entrapment by improving the wetting properties of the solution.

This application note describes the techniques, instrumentation, and method for determining low mg/L (ppm) amounts of the anionic fluorochemical surfactant FC-93 in an etch bath (1 part hydrofluoric acid/6 parts ammonium fluoride) by ion chromatography.

Conditions	
Concentrator Column:	Dionex IonPac NG1 Guard, 4 × 50 mm (P/N 39567)
Analytical Columns:	OmniPac PAX-500 Analytical, 4 × 250 mm (P/N 042152) OmniPac PAX-500 Guard, 4 × 50 mm (P/N 042153)
Eluents:	A: 20 mM Sodium hydroxide B: Acetonitrile
Eluent Composition:	55% A/45% B
Eluent Flow Rate:	1 mL/min
Rinsing Reagent:	Deionized water
Rinsing Flow Rate:	2 mL/min
Rinse Time:	20 min
Total Run Time:	30 min
Sample Volume:	100 µL
Detection:	Suppressed conductivity, Thermo Scientific™ Dionex™ ASRS™ (4 mm), AutoSuppression™ external water mode
System Backpressure:	1500–2000 psi (10.3–13.8 MPa)
Conductivity Background:	0.3–3 µS

Pump Program:

Time	*%A*	*%B*	*Valve 1*	*Valve 2*	*Remarks*
Initial	55	45	load	inject	
0.0	55	45	load	load	Fill sample loop
1.1	55	45	inject	load	Sample to NG1
20.5	55	45	load	inject	Begin sampling*
30.0	55	45	load	inject	Finish sampling



5 mg/L (ppm) surfactant in an etch bath.

Determination of Additives and Byproducts in an Acid Copper Plating Bath by Liquid Chromatography

Copper electroplating systems are used for the deposition of copper on semiconductor wafers. The primary components of an acid copper plating bath are copper sulfate, sulfuric acid, and hydrochloric acid. A variety of proprietary additives are used to influence the quality of copper deposition. As the bath ages, certain byproducts are formed as a result of the plating process. Chromatography can be used to quantitatively measure individual additives and byproducts. Tracking the levels of these components ensures the quality of the fill. This application note describes the use of the Thermo Scientific™ Dionex™ IonPac™ NS1 column with absorbance detection to determine additives and byproducts in acid copper plating baths.

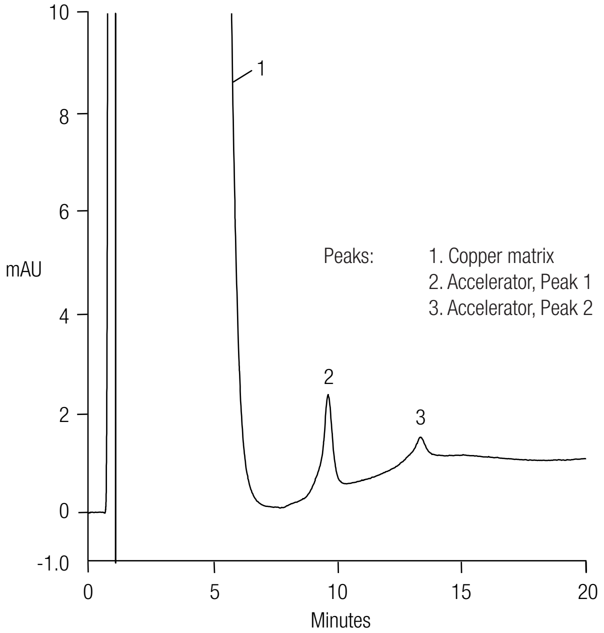
Chromatographic conditions for shallow gradient

Columns:	Dionex IonPac NS1 Analytical, 10 µm, 4 × 250 mm Dionex IonPac NG1 Guard, 10 µm, 4 × 35 mm
Eluent Flow Rate:	2.0 mL/min
Detection:	UV, 246 nm or 200 to 325 nm with PDA
Sample Volume:	10 or 500 µL
Expected System Backpressure:	13.1 MPa (1900 psi)

Shallow gradient program

V = Injection valve
E1 = 100 mN Sulfuric acid
E2 = Acetonitrile

Time	*V*	*% E1*	*% E2*	*Description*
0 min	Load	97.5	2.5	Initial
0.1	Inject	97.5	2.5	Inject sample, start gradient
2.5	Inject	95	5	
18	Inject	10	90	
20	Inject	97.5	2.5	End gradient
22	Inject	97.5	2.5	Return to initial conditions



Analysis of acid copper plating bath before electrolysis (shallow gradient).

Download the full application note

Determination of the Suppressor Additive in Acid Copper Plating Bath

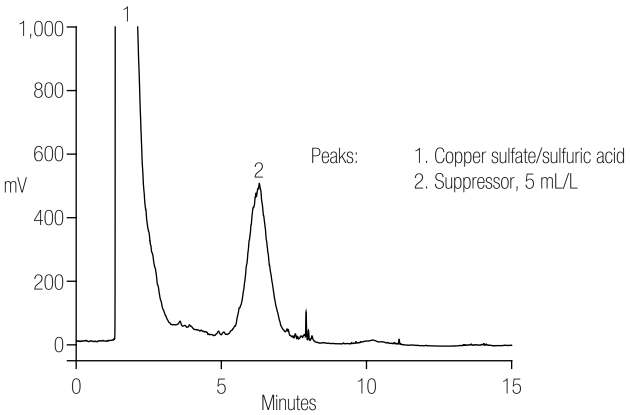
Copper electroplating systems are used for the deposition of copper on semiconductor wafers. The primary components of an acid copper plating bath are copper sulfate, sulfuric acid, and hydrochloric acid. A proprietary suppressor additive is used to influence the quality of copper deposition.

This application note describes the use of the Thermo Scientific™ Dionex™ IonPac™ NS1 column with evaporative light-scattering detection to determine two different proprietary suppressors in acid copper plating baths.

Conditions	
Eluent Flow Rate:	1.0 mL/min
Detection:	Evaporative Light Scattering
Evaporator Temperature:	80°C
Nebulizer Temperature:	75°C
Nitrogen Flow Rate:	1.0 L/min
Detector Output:	10 volt full scale
Sample Volume:	100 µL

Time (min)	V	% E1	% E2	Description
-5	Load	60	40	Load Sample
0	Inject			Begin data acquisition
2	Inject	60	40	Gradient from 40–90% acetonitrile
7	Inject	10	90	End gradient. Hold at 90% acetonitrile
10	Inject	10	90	Gradient from 90–40% acetonitrile
15	Inject	60	40	End data acquisition

V = Injection valve
E1 = Deionized water
E2 = Acetonitrile



Determination of enthone suppressor in acid copper plating bath before electrolysis.

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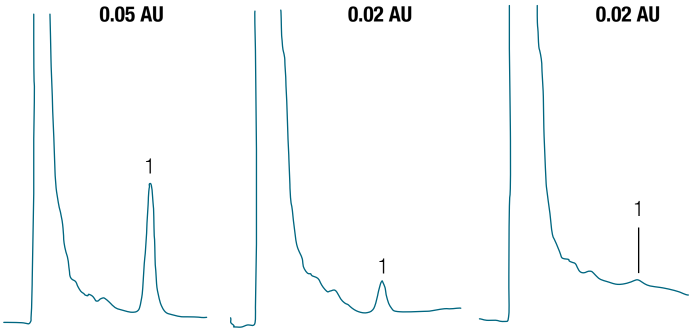
Copper Gleam PCM or PC in Acid Copper Baths

The major active component in Copper Gleam™ PCM or Copper Gleam PC can be determined down to 0.01% in acid copper baths. The manufacturer's suggested concentration range is 0.4% to 1.0%. This application update describes this determination.

Conditions	
Column:	Thermo Scientific™ Dionex™ IonPac™ NS1
Eluent:	10 mN H_2SO_4 10% CH_3CN
Flow Rate:	1 mL/min
Sample Volume:	50 µL
Detection:	UV/Vis, 254 nm



1% Brightener Level (High Level)

0.1% Brightener Level (Low Level)

Depleted Bath (No Brightener)

Determination of brightener in acid copper baths by ion chromatography.

Download the full application update

Saccharin in Electrolytic Nickel Sulfate Baths

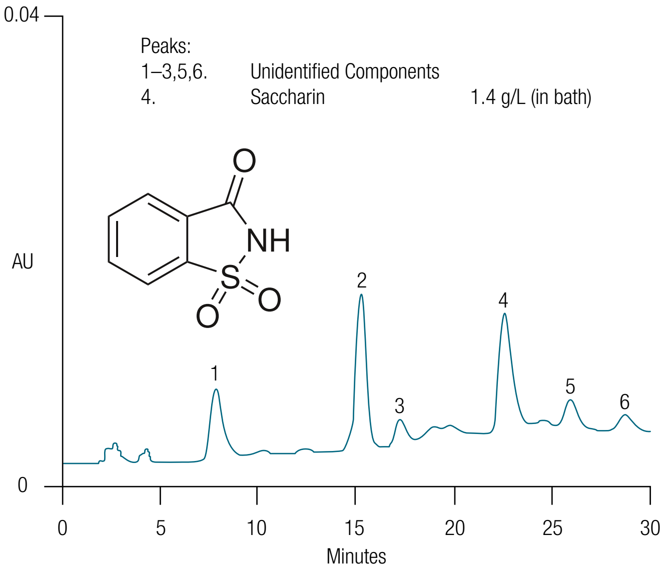
Saccharin decreases the tensile strength of electrolytic nickel deposits and can, with increasing concentrations, cause compressive stress. Sulfur can be introduced into the plate from the saccharin when class II nickel brighteners are not present, thus producing deposits with higher tensile strength, higher hardness, and relatively lower ductility. Saccharin, without other nickel brighteners present, produces a cloudy bright plate with relatively low luster.

This application update describes the determination of saccharin in a nickel sulfate bath solution using direct injection into an ion chromatography system.

Conditions	
Column:	Thermo Scientific™ OmniPac™ PAX-500
Eluents:	A. 30 mM Sulfuric acid; 5% acetonitrile
	B. 30 mM Sulfuric acid; 25% acetonitrile
Flow Rate:	1 mL/min
Inj. Volume:	25 µL
Detection:	UV, 225 nm

Gradient Program:

Time	*%A*	*%B*
0.0	100	0
20.0	0	100
30.0	0	100



Saccharin in a working O electrolytic nickel sulfate bath.

thermo scientific

Contaminants from Processes and the Environment

The manufacture of electronic devices is typically a series of chemical and mechanical operations that involve plating, masking, soldering, rinsing, etching, and cleaning. Each operation, along with the environment in which it occurs, has some effect on the device or assembly. The processes and manufacturing environment leave chemical fingerprints on the devices that are unique to the manufacturing process. In the same way that a forensic scientist would use fingerprints to trace a criminal, analytical techniques can be used to troubleshoot a manufacturing process or field failure to understand and correct the root cause. Ionic contamination can also be found on materials that come in contact with electrical devices during manufacture, e.g., gloves. These manufacturing consumables can transfer contamination to the product and can therefore require examination for contamination as well.

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Failure Analysis Applications

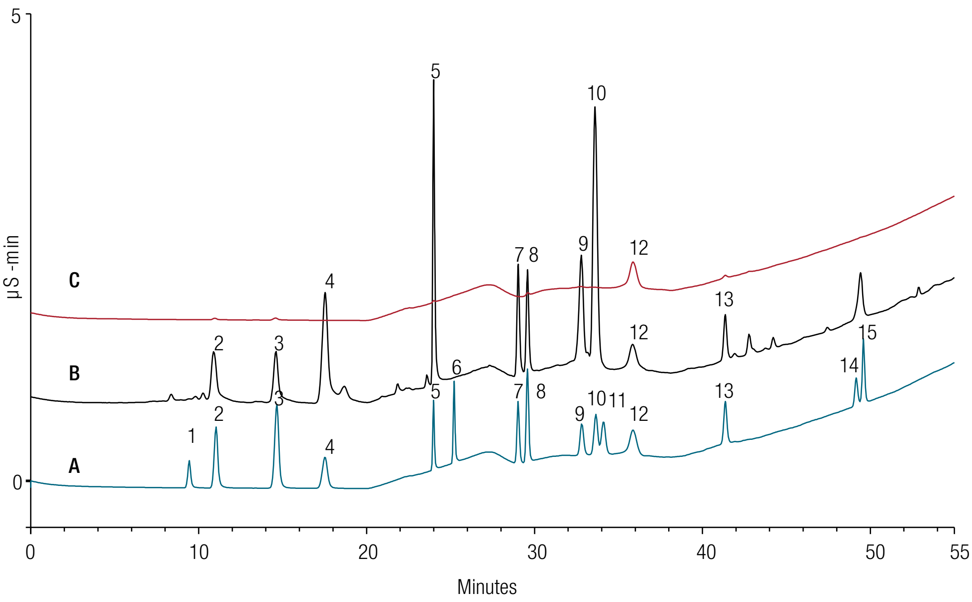
Determination of Anions on the Surface of Printed Circuit Boards by IPC-TM-650 Method 2.3.28 using HPIC

The printed circuit board industry has long been interested in the ionic cleanliness of printed board surfaces and its correlation with corrosion, electrochemical migration, dendritic growth, and subsequent opens, leakage current, or shorting during testing or in the field. Initial methods for cleanliness evaluation included resistivity of solvent extract (ROSE), which measured the conductivity of a solution after flowing it over a surface of interest. A major disadvantage of this technique was its inability to detect the specific ionic species generating the measured conductivity.

Ion chromatography (IC) has become an important technique for the evaluation of ionic cleanliness. This technique, which detects individual ions, allows quicker troubleshooting of contamination sources and better predictions about the detrimental effects of each ionic species.

This application note describes an IC method using a Thermo Scientific™ Dionex™ IonPac™ AS11-HC-4µm column and a Thermo Scientific™ Dionex™ ICS-5000⁺ HPIC™ system* to determine extractable anionic contaminants on the surface of PCBs, including inorganic anions and weak organic acids, following the extraction procedure described in IPC-TM-650 Method 2.3.28.

*Equivalent or improved results can be achieved using a Thermo Scientific™ Dionex™ ICS-6000 HPIC™ system.



Separation of PCB assembly B and PCB blank B on the Dionex IonPac AS11-HC-4 µm column in (A) a mix of 14 anion standards, (B) PCB assembly B, and (C) PCB blank B.

Peaks	Standard	PCB Assembly A mg/L	PCB Blank A
1. Fluoride	0.250	—	—
2. Acetate	2.50	3.08	0.0993
3. Formate	2.50	1.99	0.112
4. Methanesulfonate	2.50	8.42	—
5. Chloride	0.750	2.76	0.123
6. Nitrite	1.25	—	—
7. Bromide	2.50	4.43	—
8. Nitrate	2.50	2.83	0.147
9. Adlpate	2.50	9.64	—
10. Succinate	2.50	19.2	—
11. Malate	2.50	—	—
12. Carbonate	n.a.	n.a.	n.a.
13. Sulfate	1.25	1.27	0.118
14. Phthalate	2.50	—	—
15. Phosphate	3.75	—	—

“-"= < LOQ
n.a. = not applicable

Conditions

Columns:	Dionex IonPac AG11-HC-4-µm Guard, 2 × 50 mm (P/N 078036)
	Dionex IonPac AS11-HC-4-µm Analytical, 2 × 250 mm (P/N 078035)
Eluent Source:	Dionex EGC 500 KOH Eluent Generator Cartridge with CR-ATC 500
Eluent A:	DI Water
Eluent B:	Methanol (CH_3OH)
Gradient:	

Time (min)	KOH (mM)
-5.00	1
0.00	1
17.00	1
24.00	15
35.30	15
54.60	60
54.61	1
55.00	1

Pump_1: Multi-Step Gradient

Time (min)	B (%)	Curve
-5.00	0	5
0.00	0	5
22.00	0	5
24.00	10	5
39.30	12	5
51.00	10	5
54.60	0	5
55.00	0	5

Pump_1 Flow Rate:	0.38 mL/min
Pump_2:	Isocratic (delivers external water for the Dionex AERS 500e suppressor)
Pump_2 Flow Rate:	0.76 mL/min
Injection Vol.:	5 µL
Temperature:	40 °C (column compartment), 25 °C (detector compartment)
System Backpressure:	~4000 psi (1 mM KOH/0% CH_3OH), ~4900 psi (22 mM KOH/12% CH_3OH)
Detection:	Suppressed Conductivity, Dionex AERS 500e Electrolytically Regenerated Suppressor (2 mm), AutoSuppression, 57 mA, external water mode
Background Conductance:	~ 0.5 µS
Run Time:	60 min

Download the full application note

Monitoring for Trace Anion Contamination in the Extracts of Electronic Components

Ion chromatography is the method of choice for the analysis of ionic contamination on electronic components. When present with moisture, some anions—particularly chloride and sulfate—form dilute concentrations of mineral acids and cause corrosion. Ion chromatography has been successfully applied to the determination of ionic contamination on printed circuit boards, semiconductor wafers, and disk drive components.

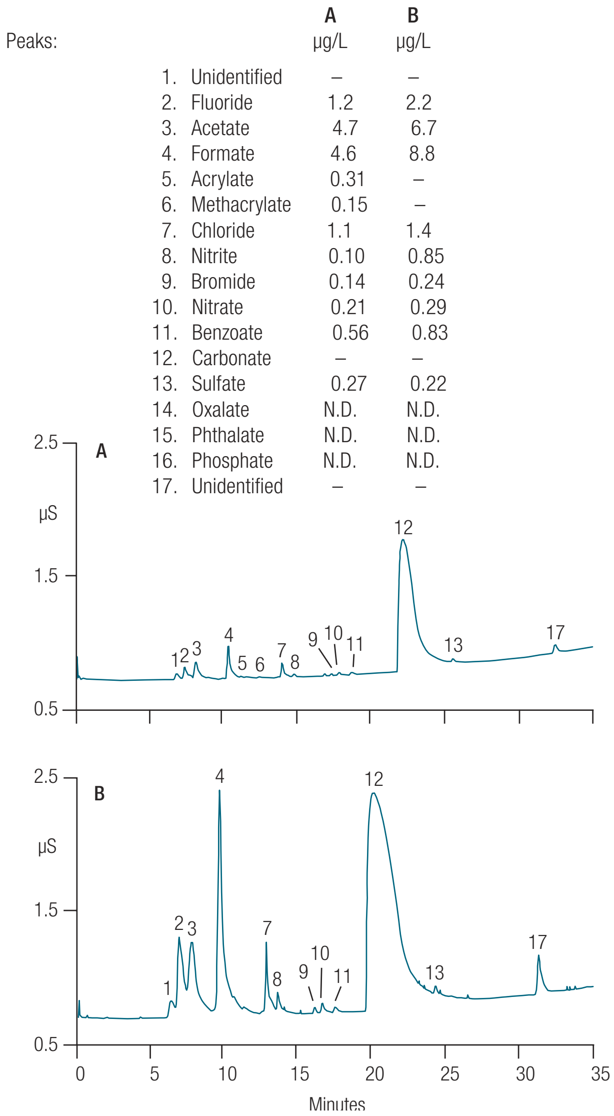
Modern disk drives have very close head-to-disk interfaces, making component cleanliness critical to drive performance and reliability. Anions are one of the possible contaminants of drive components. A comprehensive anion analysis of drive components prior to manufacturing can significantly reduce the incidence of corrosion and head-to-disk interface failures.

This application note describes the use of high-volume direct injection and preconcentration to determine trace anion contaminants in extracts of disk drive components.

Conditions

Eluent:	Potassium hydroxide (Dionex EG50 Eluent Generator as the source)
Temperature:	30 °C
Dionex EG50 Offset Volume:	0 mL
Eluent Flow Rate:	0.5 mL/min
Detection:	Suppressed conductivity, Dionex ASRS ULTRA recycle mode
Dionex ASRS Current Setting:	50–100* mA
Expected Background Conductivity:	1 µS (40 mM KOH)
Expected System Backpressure:	15.2–16.6 MPa (2200–2400 psi)
Sample Volume:	1 mL for direct injection method or 5 mL for preconcentration method

* 50 mA is the low range for these conditions. The data shown in this application note was run at 100 mA.



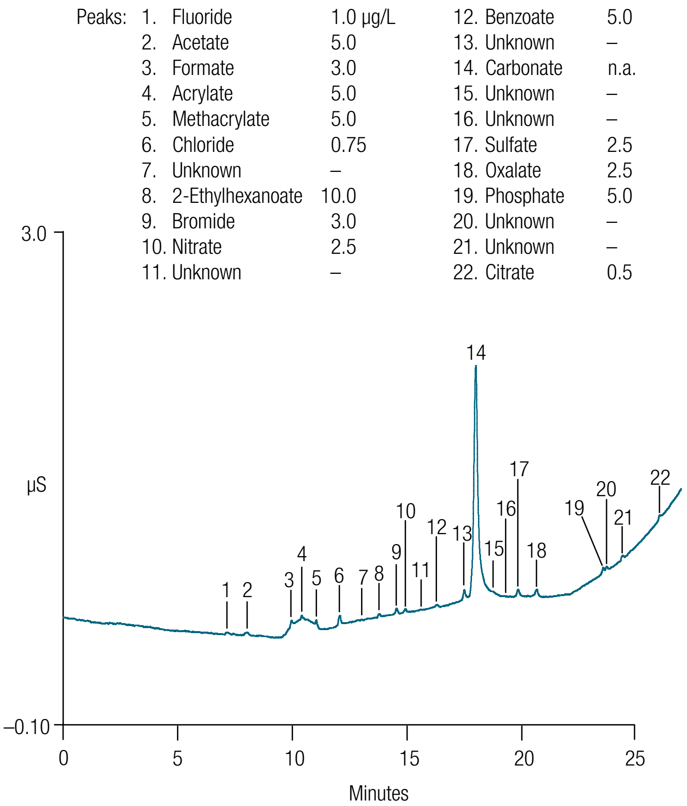
Peaks:	A µg/L	B µg/L
1. Unidentified	–	–
2. Fluoride	1.2	2.2
3. Acetate	4.7	6.7
4. Formate	4.6	8.8
5. Acrylate	0.31	–
6. Methacrylate	0.15	–
7. Chloride	1.1	1.4
8. Nitrite	0.10	0.85
9. Bromide	0.14	0.24
10. Nitrate	0.21	0.29
11. Benzoate	0.56	0.83
12. Carbonate	–	–
13. Sulfate	0.27	0.22
14. Oxalate	N.D.	N.D.
15. Phthalate	N.D.	N.D.
16. Phosphate	N.D.	N.D.
17. Unidentified	–	–

Analysis of deionized water extract from a disk drive spacer (A) 1 mL direct injection, (B) 5 mL preconcentration.

Using a Reagent-Free Ion Chromatography System to Monitor Trace Anion Contamination in the Extracts of Electronic Components

Keeping the anionic contamination of electronic components low can reduce the incidence of component failure. The IC method in AN153 demonstrated the separation of 14 inorganic anions and organic acids on a Thermo Scientific™ Dionex™ IonPac™ AS17 column set after either a 1 mL direct injection or a 5 mL sample preconcentration. In this application update, a hydroxide gradient program similar to, but faster than, the program used in AN153 separates a new set of 14 anions. This set of anions differed from the original set by the replacement of nitrite and phthalate with 2-ethylhexanoate and citrate. 2-Ethylhexanoate is the byproduct of an adhesive sometimes used in the disk drive industry.

Conditions	
Column:	Thermo Scientific™ Dionex™ IonPac™ AS17, 4 × 250 mm
Guard:	Thermo Scientific™ Dionex™ IonPac™ AG17, 4 × 50 mm
Eluent:	Dionex EGC-KOH cartridge
Gradient:	0.3 mM isocratic to 7 min; gradient to 15 mM at 20 min; gradient to 40 mM at 25 min; isocratic to 26.9 min; step change to 0.3 mM at 27 min
Time (min)	*Concentration (mM)*
-7.0	0.3
0.0	0.3
7.0	0.3
20.0	15
25.0	40
26.9	40
27.0	0.3
Flow Rate:	1 mL/min
Temperature:	30ºC
Sample Loop:	25 µL
Detection:	Suppressed conductivity, Thermo Scientific™ Dionex™ ASRS™ ULTRA II in external water mode with CRD



Separation of a low concentration mixed anion and organic acid standard.

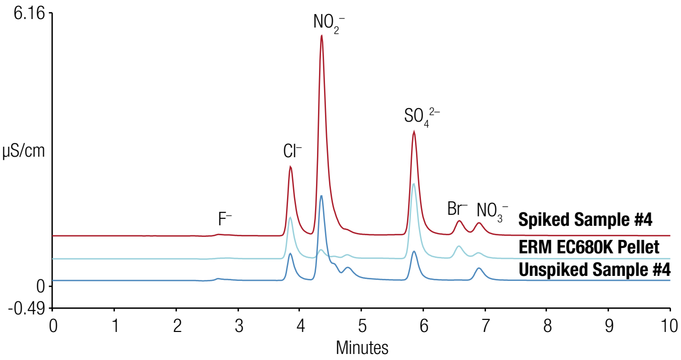
Download the full application update

Determination of Chlorine, Bromine, and Sulfur in Polyethylene Materials using Combustion Ion Chromatography

Polyethylene (PE) is a thermoplastic created from the polymerization of ethylene, a process that produces long, straight chains of hydrocarbon monomers. Polyethylene is widely used in packaging (plastic bags, plastic films, containers including bottles, etc.). Plastic materials do not consist only of plastic polymers. Halogen and sulfur-containing compounds are often added as plasticizers, flame retardants, and heat stabilizers. Due to the widespread use of plastic/polymers and their subsequent impact on the environment, it is important to know the content of the halogens and sulfur when polyethylene materials are disposed of or recycled.

Combustion ion chromatography (CIC) has been demonstrated for determination of halogens and sulfur in a variety of difficult samples. In this application note, it is demonstrated that a CIC system can be used for automated qualitative and quantitative analysis of halogens and sulfur in plastic samples. A similar method can also be used for determination of halogens in printed circuit boards to establish compliance with restrictions dictated by regulations such as Restriction of Hazardous Substances (RoHS) and WEEE (Waste from Electrical and Electronic Equipment).

Conditions	
Columns:	Dionex IonPac AS11-HC-4µm, Analytical, 2 × 250 mm Dionex IonPac AG11-HC-4µm, Guard, 2 × 50 mm
Eluent:	25 mM KOH
Eluent Source:	Dionex EGC 500 KOH cartridge with high pressure CR-ATC
Flow Rate:	0.38 mL/min
Sample Loop:	100 µL
Column Temp.:	30 °C
Compartment Temp.:	15 °C
Detection:	Suppressed conductivity with Dionex ERS 500 Electrolytically Regenerated Suppressor, recycle mode
Suppressor Current:	24 mA
System Backpressure:	~4050 psi
Background Conductance:	~0.1–0.2 µS/cm
Noise:	0.2–0.4 nS/cm peak-to-peak
Run Time:	10 min



Chromatogram of unspiked sample 4, ERM EC680k pellet, and sample 4 spiked with ERM pellet.

Download the full application note

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Thermo Scientific™ Dionex™ ICS-4000 Integrated Capillary IC System

Thermo Scientific™ Dionex™ ICS-6000 HPIC™ System

Combustion Ion Chromatography System

Thermo Scientific™ Dionex™ Integral Process Analytical System

Find out more at **thermofisher.com/IC**

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