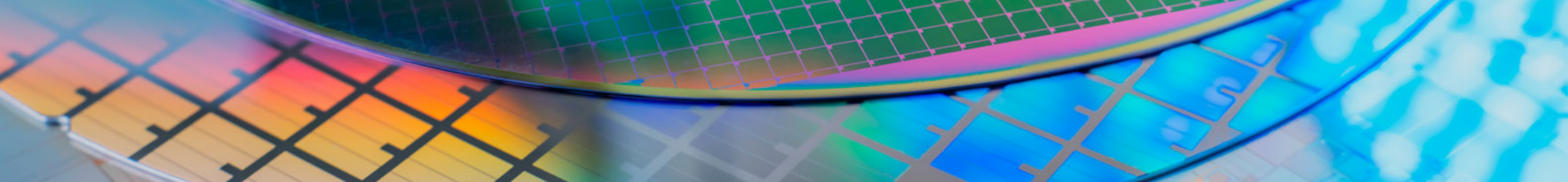


# Semiconductor workflows

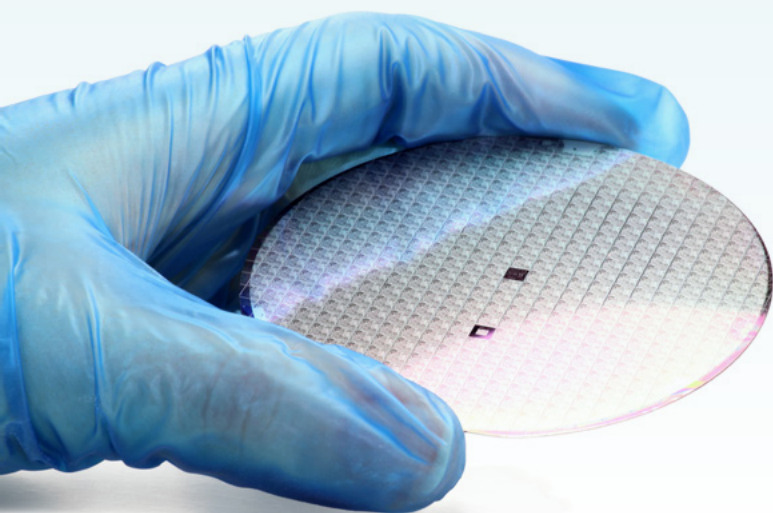
Trace contaminant analysis  
application compendium



# The role of ion chromatography in the semiconductor industry

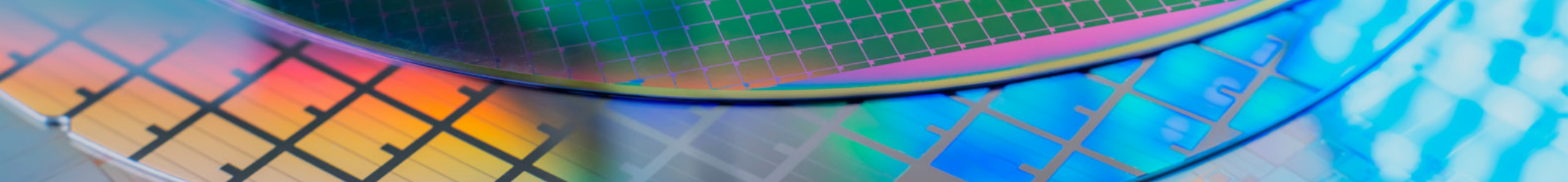
Demand for semiconductor wafers will continue its vigorous growth as the demand for mobile devices, cloud computing, Internet of Things (IoT), self-driving automobiles, artificial intelligence, and other uses increases. The complexity of the processes and costs of semiconductor fabrication (FAB) manufacturing are high, so those in the industry are always looking to improve production efficiencies.

Ionic contamination is a big concern in semiconductor manufacturing processes, in finished devices, and in consumables such as cleanroom wipes, cotton swabs, gloves, and carriers 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 to trigger immediate remediation steps.

Physical and chemical analysis of semiconductor wafer fab workflows				
	Process development	Process monitoring	QA/QC	Failure analysis
Matrices	Cleanroom air	Organics (VOC, SVOC), acids, bases, anions, metals GCMS, IC, ICP-MS, particle counters		
	Wafer/substrate	Physical/chem charac. SEM, TEM, AES, FT-IR, AFM	Contamination/environmental control and monitoring SEM, TEM, AES, FT-IR, AFM, IC, GC-MS	Physical/chem charac. SEM, TEM, AES, FT-IR, AFM
	Gases	Organic impurities dynamic and static headspace GC-MS, ICP-MS, API-MS		
	Ultra pure water	Anions, silicates, organics (VOC, SVOC) organic acids, TOC, cations, metals, transition metals IC, discrete analysis, conductivity, ICP, ICP-MS		
Products	Plating baths, chemicals, and reagents	Additives (accel., level., supp.) LC-CAD, IC	Cations and amines in H <sub>2</sub> O <sub>2</sub> IC	Amines and org acids Wafer analysis, device and board analysis, IC
		Metals in conc. HF, HCl, HNO <sub>3</sub> , H <sub>2</sub> SO <sub>4</sub> ICP-MS	Metal CN in NaCN IC-UV	Halogens Printed circuit boards, indirect materials, IC
		Cyanide in conc. alkalines IC-PAD	Metals in alcohols ICP-MS	Organics (VOC, SVOC) Wafers, boards, carriers, headspace GC-MS
		Additive byproducts in acids IC-PAD	Anions in conc. acids and solvents IC	



# Using a reagent-free ion chromatography system to monitor trace anion contamination in the extracts of electronic components

## Overview

Keeping the anionic contamination of electronic components low can reduce the incidence of component failure.

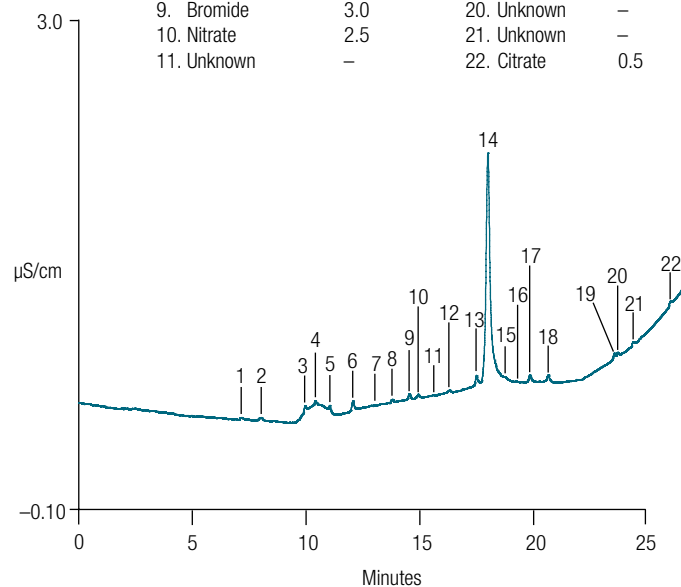
The ion chromatography method in Thermo Scientific™ Application Update 157 (AU157) demonstrated the separation of 14 inorganic anions and organic acids on a Thermo Scientific™ Dionex™ IonPac™ AS17 column set after either a 1 mL direct injection or a 5 mL sample preconcentration.

In this application update, a hydroxide gradient program similar-to, but faster than, the program used in Thermo Scientific™ Application Note 153 (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.

[View the full application update](#)

Peaks:	1. Fluoride	1.0 µg/L	12. Benzoate	5.0
	2. Acetate	5.0	13. Unknown	–
	3. Formate	3.0	14. Carbonate	n.a.
	4. Acrylate	5.0	15. Unknown	–
	5. Methacrylate	5.0	16. Unknown	–
	6. Chloride	0.75	17. Sulfate	2.5
	7. Unknown	–	18. Oxalate	2.5
	8. 2-Ethylhexanoate	10.0	19. Phosphate	5.0
	9. Bromide	3.0	20. Unknown	–
	10. Nitrate	2.5	21. Unknown	–
	11. Unknown	–	22. Citrate	0.5



# Determination of inorganic anions in alkaline solutions

## Overview

Trace anions at ppb to ppm concentrations were determined in 100  $\mu\text{L}$  of concentrated (molar to percent) amine and hydroxide reagent solutions using a single pass autoneutralization step to neutralize the sample.

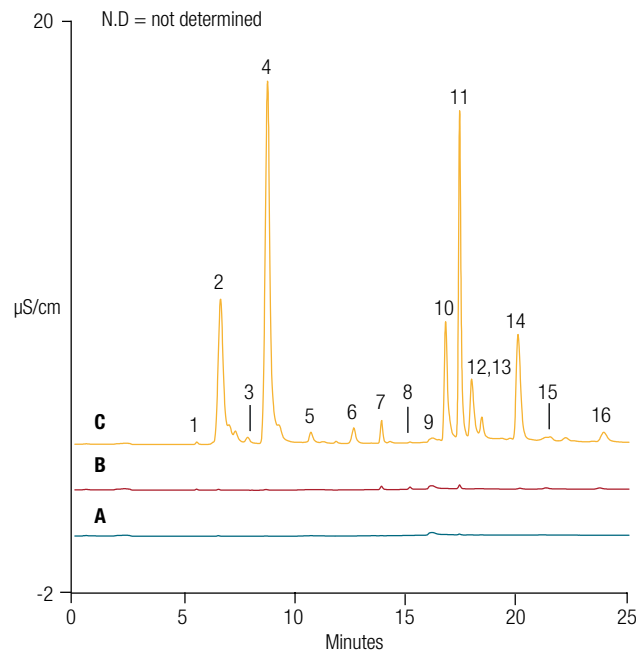
The neutralized sample was collected and concentrated onto a concentrator column, and finally analyzed by fast anion-exchange chromatography and suppressed conductivity detection.

[View the full application note](#)

Samples: A: Deionized water blank  
B: Standard (1  $\mu\text{g/L}$  fluoride, 5  $\mu\text{g/L}$  chloride, nitrite, sulfate, bromide, nitrate, 10  $\mu\text{g/L}$  phosphate)  
C: 0.1 M TBAOH reagent grade

Peaks:	B	C		B	C
1. Fluoride	1.2	2.2 $\mu\text{g/L}$	9. Carbonate	N.D.	N.D.
2. Acetate	–	–	10. Unknown	–	–
3. Organic Acid	–	–	11. Sulfate	5.1	557 $\mu\text{g/L}$
4. Formate	–	–	12. System	–	–
5. Unknown	–	–	13. Unknown	–	–
6. Unknown	–	–	14. Bromide	5.6	596
7. Chloride	5.4	29.3	15. Nitrate	N.D.	N.D.
8. Nitrite	N.D.	N.D.	16. Phosphate	10.5	98.2

N.D. = not determined




# Determination of trace cations in concentrated acids

## Overview

Determination of cations in concentrated acids (24% sulfuric acid, 25% phosphoric acid, and 10% hydrofluoric acid) 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  $\mu\text{g/L}$  and  $\text{ng/L}$  concentrations. Direct injection of concentrated acid samples overloads an IC column with the acid cation, resulting in poor chromatography.

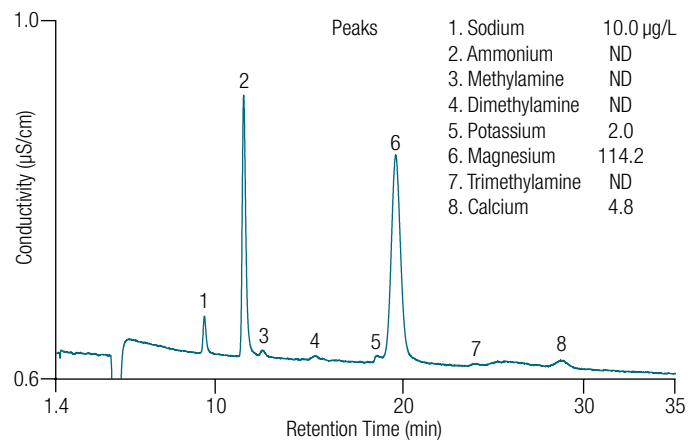
 [View the full application note](#)

## Sample Prep.

Trap Column: Dionex IonPac CTC-1,  
9 × 24 mm  
Carrier: Deionized water  
Carrier Flow Rate: 0.5 mL/min  
Inj. Vol.: 100  $\mu\text{L}$   
Temperature: 30°C  
Neutralization: AutoNeutralize,  
Dionex CSRN II,  
external water mode,  
500 mA

## Analytical

Column: Dionex IonPac CG16,  
CS16, 4 mm  
Eluent: 28 mM Methanesulfonic acid  
Eluent Source: Dionex EGC II MSA  
Flow Rate: 1.0 mL/min  
Temperature: 30°C  
Detection: Suppressed conductivity,  
Dionex CSRS ULTRA II,  
recycle mode, 82 mA  
Concentrator: Dionex IonPac TCC-UPL1



# Determination of transition metals at ppt levels in high-purity water and SC2 (D-clean) baths

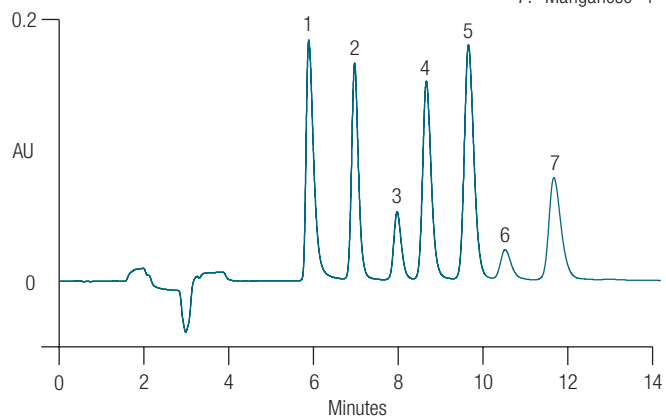
## Overview

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. To monitor metals in cleaning baths and rinse water, improved analytical methods are needed.

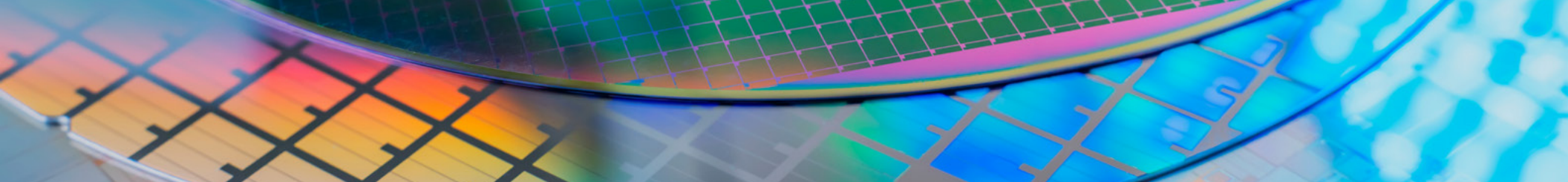
This application note describes a method for determining low ng/L amounts of transition metals in high purity water and semiconductor bath solutions. The Thermo Scientific™ Dionex™ IonPac™ CS5A Column is used for the separation of transition metals. This column has a unique bilayer latex structure consisting of both anion- and cation-exchange retention mechanisms. Transition metals can be separated using either anion or cation exchange chromatography, depending on the choice of the complexing agent used in the eluent.

The figure on the right shows the analysis of 30 mL of a 1 µg/L transition metals standard. All peaks are well separated from the void volume and from each other and are therefore easily quantified.

Analytical Columns:	Dionex IonPac CS5A, CG5A (2 mm)	Concentration	
		Time:	15 min
Eluent:	PDCA	Sample Volume:	30 mL
Eluent Flow Rate:	0.3 mL/min	Total Run Time:	30 min
Post Column Reagent:	PAR	Detection:	Vis, 530 nm
Post Column Reagent:	0.15 mL/min	Peaks:	1. Iron 1 µg/L
Concentrator Column:	Dionex IonPac TCC-2		2. Copper 1
Concentrator Pump:	DQP		3. Nickel 1
Concentrator Pump Flow:	2 mL/min		4. Zinc 1
			5. Cobalt 1
			6. Cadmium 1
			7. Manganese 1



[View the full application note](#)



# Determination of trace anions in high purity waters using a large-volume direct injection

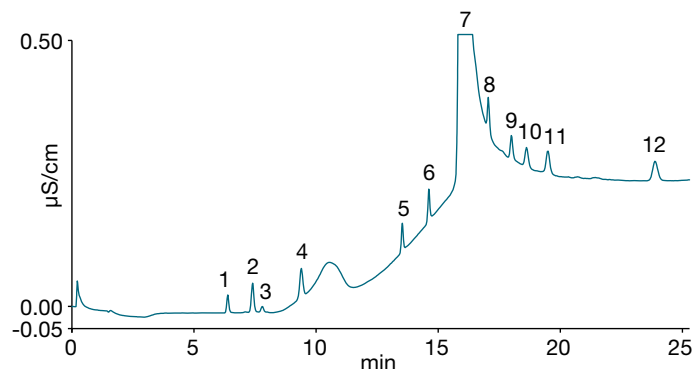
## Overview

Trace anion analysis in high purity water is an important application for the semiconductor, disk drive, electronic components, and nuclear power industries. The measurement of trace levels of ionic impurities throughout the power generation process is critical for the identification and prevention of corrosive conditions in many power plant components.

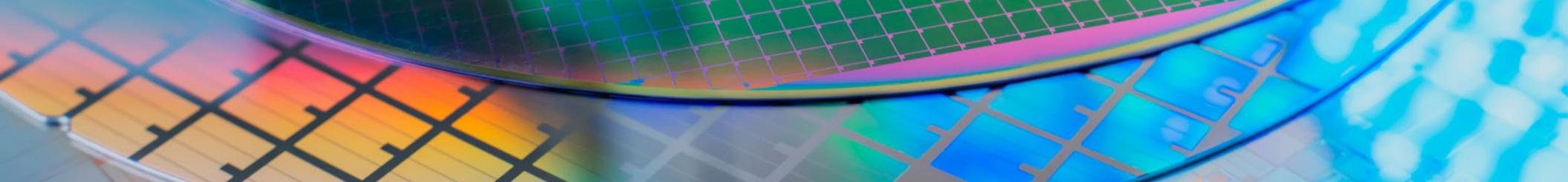
The Thermo Scientific™ Dionex™ IonPac™ AS28-Fast-4 $\mu$ m (2 × 150 mm) Column is well suited for trace-level analysis of common inorganic anions and low molecular weight organic acids. For trace analysis, a large-loop injection combined with gradient elution was used. The column provides improved peak efficiencies compared to the Thermo Scientific™ Dionex™ IonPac™ AS15-5 $\mu$ m (3 × 150 mm) Column, which results in lower detection limits. The Thermo Scientific™ Dionex™ ADRS 600 Suppressor delivers low background and noise for sensitivity at trace levels.

The figure on the right shows a typical chromatogram for a standard 11 anions at trace levels. The large peak at ~17 min is carbonate, the size of which will vary with the amount of dissolved carbon dioxide in the sample. The chromatographic baseline shift is approximately 200–300 nS/cm. The separation is performed at 30°C to provide consistent retention times during analysis.

1. Fluoride	0.125 $\mu$ g/L (ppb)	7. Carbonate	-
2. Acetate	0.313	8. Sulfate	0.625
3. Glycolate	0.313	9. Oxalate	0.313
4. Formate	0.313	10. Bromide	0.625
5. Chloride	0.313	11. Nitrate	0.625
6. Nitrite	0.625	12. Phosphate	1.250



[View the full application note](#)



# Determination of silicate in high-purity water using ion chromatography and online sample preparation


## Overview

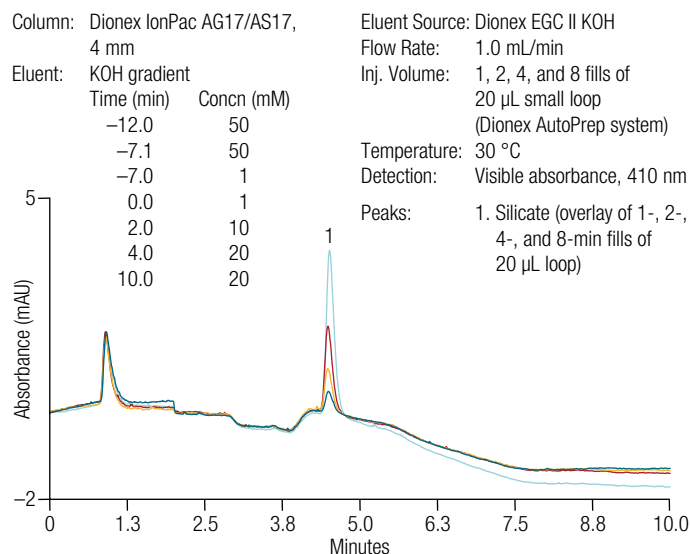
The water used in the manufacture of semiconductors and other modern electronic components must be extremely pure. The production of ultrahigh-purity water (UHPW) includes deionization to remove corrosive strong acid anions, e.g., chloride and sulfate. Deionization cartridges exhaust their capacity over 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.

While silicate can be separated at high pH, it has very little dissociation from its acid form at neutral pH, and therefore cannot be detected sensitively using suppressed conductivity. However, silicate will react with molybdate to form a complex that can be detected with high sensitivity at 410 nm.

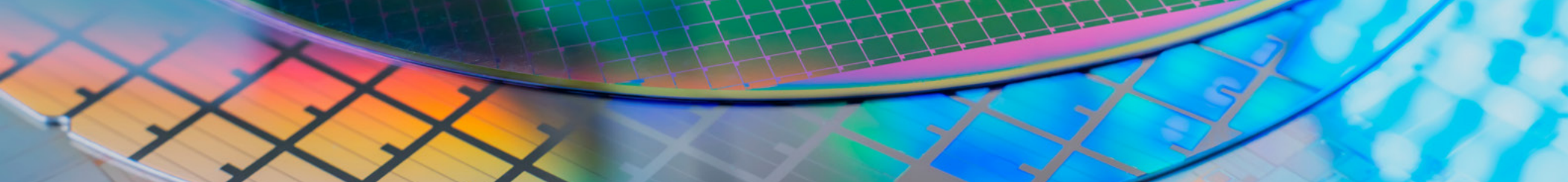
To confirm that the silicate-free water is indeed silicate-free and can be used to prepare the standards the large loop was filled with the silicate-free water and a blank analysis run. The Thermo Scientific™ Dionex™ AutoPrep™ System was used to calibrate this method. The four standards used by the system for method calibration were 200, 400, 800, and 1,600 ng/L. The figure on the right shows an overlay of these four standards.

This application note shows how the combination of Thermo Scientific™ Reagent-Free™ ion chromatography (RFIC™) and the Dionex AutoPrep system minimizes the time and labor needed for sensitive and reproducible determinations of silicate in UHPW.

 [View the full application note](#)








## Trace borate determination in high-purity waters

### Overview

Borate is one of the most weakly retained anions on anion-exchange materials and is the first to break through many water purification systems. In the power generation and semiconductor industries, monitoring trace levels of boron as borate in high-purity waters is a measure of the efficiency of the water purification systems. The presence of borate is a sign that the water purification system requires service.

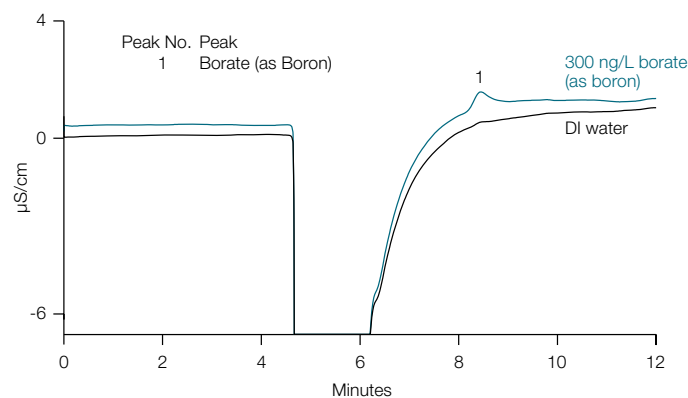
In this application note, we demonstrate and evaluate an ion-exclusion method for the determination of trace-level borate in high-purity water. This ion-exclusion method coupled with suppressed conductivity detection offers an integrated solution for the determination of trace level boron as borate.

We used a Thermo Scientific™ Dionex™ IonPac™ ICE-Borate analytical column and a Thermo Scientific™ Dionex™ IonPac™ TBC-1 Trace Borate concentrator column, specifically designed for this application. The experimental approach shown combines large sample volume/preconcentration with ion exclusion chromatography. This approach achieves sensitive detection at low to sub- $\mu\text{g/L}$  levels.

 [View the full application note](#)

The figure below shows a typical chromatogram for a borate standard at trace levels. The typical background conductance ranges from  $\sim 140$  to  $160 \mu\text{S/cm}$  depending on the quality of the mannitol used in eluent preparation. The separation is performed at  $30^\circ\text{C}$  to provide consistent retention times during analysis specifically (no other anions are concentrated) and can be eluted with an acid/mannitol eluent for the ion exclusion separation step.

This study demonstrated the determination of trace borate in high-purity waters using a large sample volume/preconcentration method with ion-exclusion chromatography and suppressed conductivity detection. The method was validated with calibration and precision studies of deionized (DI) water spiked with trace levels of borate.



# Determination of chlorine, bromine, and sulfur in polyethylene materials using combustion ion chromatography

## Overview

Polyethylene (PE) is a thermoplastic created from the polymerization of ethylene, a process that produces long, straight chains of hydrocarbon monomers.

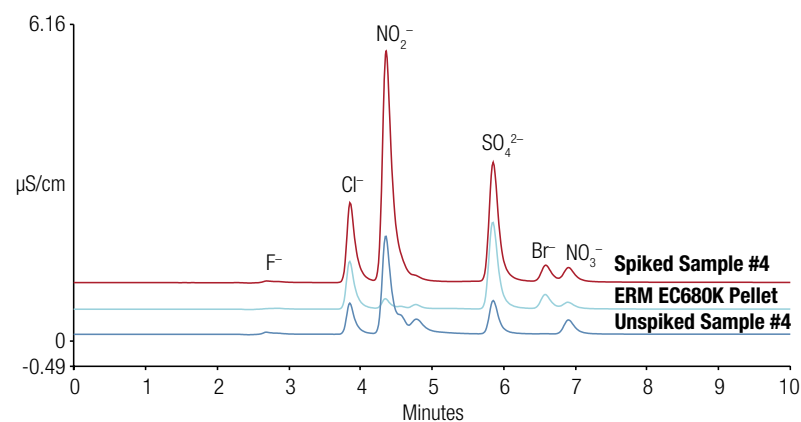
Polyethylene is widely used in packaging (plastic bags, plastic films, containers including bottles, etc.).

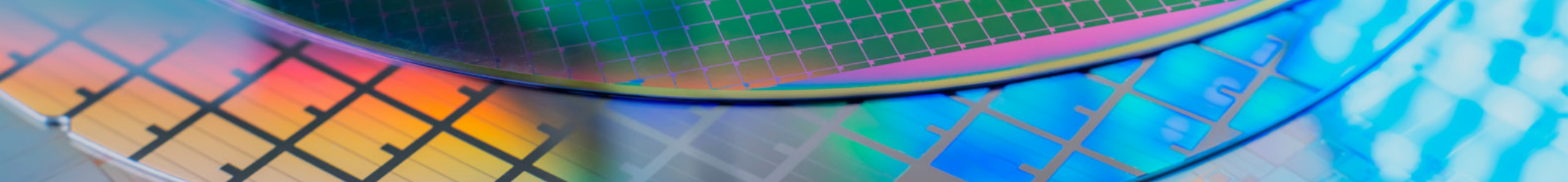
Plastic materials are not only polymers. Halogen and sulfur containing compounds are often added as plasticizers, flame retardants, and heat stabilizers.

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 allows the analyst to determine the amounts of sulfur and halogens in PE. From the semiconductor and the Institute of Printed Circuits (IPC) industry, halides and sulfur are potentially corrosive and therefore, polymers containing halides and sulfur are avoided by both industries.

Method accuracy was evaluated using a polymer certified reference material (ERM EC680k) for Cl, Br, and S and spike recovery experiments for each analyte.

[View the application note](#)






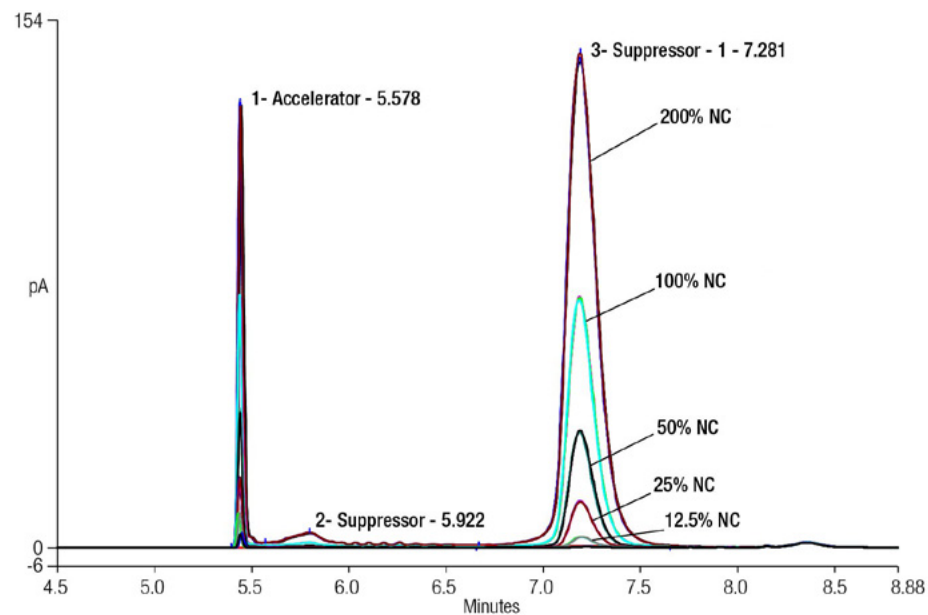
# Measurement and control of copper additives in electroplating baths using high performance liquid chromatography

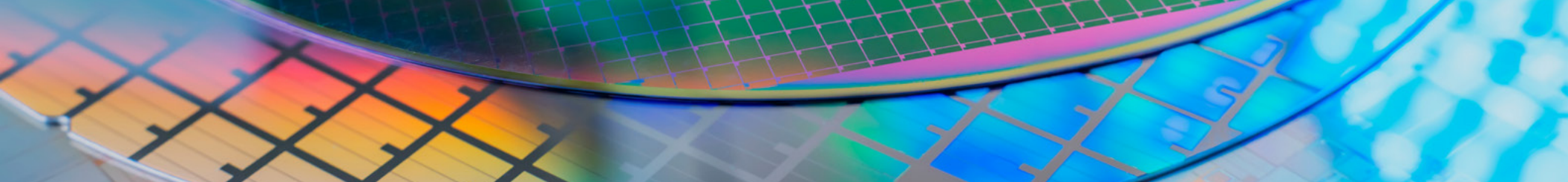
## Overview

The HPLC Corona CAD (Charged Aerosol Detection) methods for the determination of accelerator, leveler, and suppressor organic additives in acid copper plating baths provide reliable and fast determinations of the levels of these organic additives.

Unlike cyclic voltammetric stripping (CVS) analysis that shows a cumulative signal related to all the additives and their impurities, actual quantities of additives can be reliably obtained.

 [View the full white paper](#)





# Airborne molecular contamination clean-room monitoring

## Overview

Airborne Molecular Contamination (AMC) is a serious issue in the semiconductor industry as the presence of volatile and semi-volatile organic vapors adversely affects the growth and performance of silicon wafers which in turn affects the performance of microprocessors, data storage devices, and other key electronic components. As such, air cleanliness by chemical concentration (ACC) should be assessed (ISO 14644-8). The contaminants to be monitored as part of ACC are segmented into the following groups:

- Acids
- Bases
- Biotoxics
- Condensables (organic compounds with boiling point (bp) >150°C)
- Corrosives
- Dopants
- Organics (carbon containing compounds)

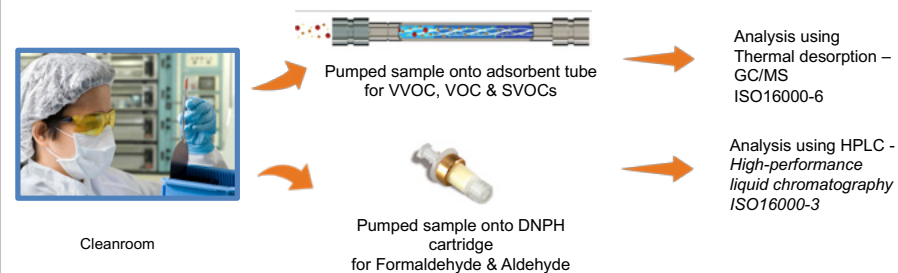
Thermal desorption coupled to GC or GC-MS is the technique of choice for both online monitoring, with a direct sample analysis after sample collection, and offline monitoring of cleanrooms or material which could outgas and cause AMC, with sample collection separated from sample analysis. For cleanroom monitoring the grade of cleanroom will often determine which type of monitoring is desired, with online measurements being preferred for cleanrooms of a high grade. TD-GC(-MS) can be applied to the measurement of organics, biotoxics, condensables, and dopants from the cleanrooms and from materials.



View the full presentation by Markes International

## Airborne molecular contamination (AMC) Clean-Room Monitoring

Off-line monitoring of volatile organic contamination in clean-room environments



## Pin pointing contaminant sources

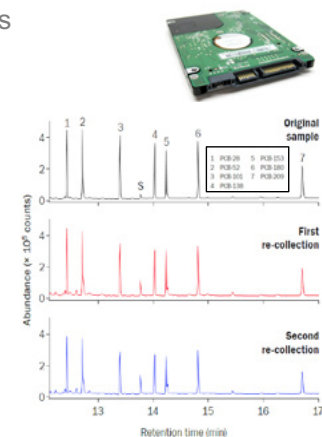
Condensable and organic contaminants - PCBs

PCBs are emitted from electrical equipment and construction materials.

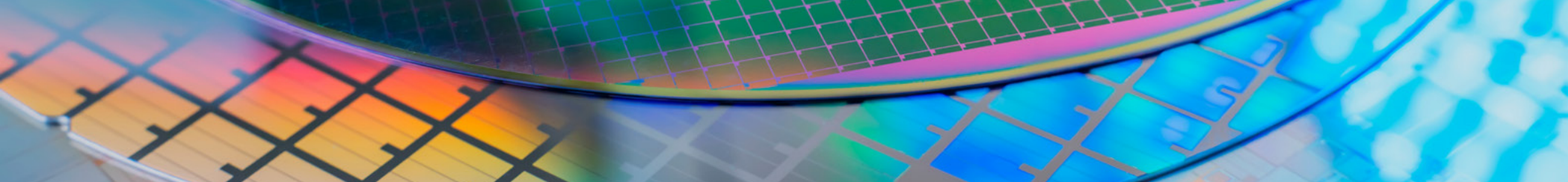
Organic contamination is important to understand and control because the molecules can adhere onto the surface of silicon wafers leading to irregularities (hydrophobization and haze generation).

This effects clean up of metallic contaminants from the silicon surface and prevents uniform etching of silicon substrates and insulator films during aqueous chemical treatments.

- Sample: 10 ng/μL standard solution of PCBs in isoocane
- Tube and trap: 'SVOC' tube and 'General-purpose' trap



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# Automated industrial manufacturing processed water and wastewater analysis using Thermo Scientific™ Gallery™ and Gallery™ Plus Discrete Analyzer methods

## Overview

Most industrial manufacturing processes, such as crude oil distillation, paper production, power generation, and high-speed microprocessor production, require large quantities of high-purity water to avoid scaling and corrosion, and to maintain component functionality and reliability.

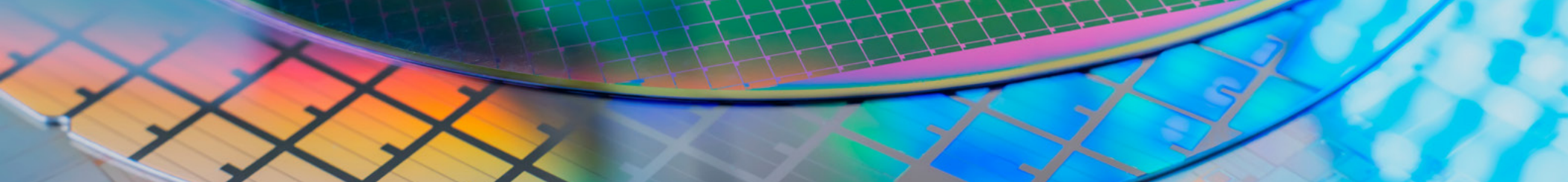
For these industries, accurate monitoring of the quality of raw water intake, process waters, and wastewater are a necessary everyday task.

Gallery and Gallery Plus discrete analyzers are easy-to-use, high-throughput, automated systems that allow laboratories to simplify their testing with dual benefits: time and cost savings. All necessary analysis steps are automated, providing true walkaway time for the operator. Both Gallery and Gallery Plus instruments provide an integrated platform for two measurement techniques, photometric and electrochemical (pH and conductivity), which can be run concurrently. Parallel determination of multiple analytes from a single sample as well as the presence of several automated features ensure analytical efficiency and increase laboratory productivity.

 [View the full brochure](#)

Gallery discrete analyzer — U.S. EPA and international reference methods					
Regulatory methods	Analytes	Regulatory methods	Analytes	Regulatory methods	Analytes
EPA 310.2 ISBN 0117516015 SMWW 2320 B	Alkalinity*	ISO 11083 DIN EN ISO 23913:2009 ASTM D1687-12(A)	Hexavalent chromium	EPA 420.1 ISBN 0117516171	Phenols (Total)
SM 3500-Al B	Aluminium*	SM 3500 Cr-B SW 7196 A		EPA 365.1 EN ISO 6878 ISO 15923-1 ISBN 0117515825 SM 4500 P-E	Phosphate
EPA 350.1 ISO 7150 ISO 15923-1 DIN 38406 ISBN 0117516139	Ammonia	ISO 6332-1988 SM 3500 F-B	Iron	EPA 365.4 ISBN 0117518883	Phosphorus (Total)
ISBN 0117515833	Boron*	SM 3500-Fe B	Iron (Ferrous), Iron (Total)		Silica
EPA 410.4	COD*	ASTM D7781-14 (NEC) Nitrate	Nitrate (TON ENz)	EPA 370.1 ISO 15923-1 SM 4500 SiO2-C USGS I-2700-85	
EPA 325.2 EN ISO 15682 ISO 15923-1 ISBN 0117516260 SM 4500-Cl-E	Chloride	Reductase method for drinking water (USGS I-2547-11 (USGS I-2548-11 (NEC) Method N07-0003		EPA 375.4 ISO 15923-1 DIN 38405-D 5-2 ASTM D516-11 ISBN 0117533406 SM 4500 SO4-E	Sulfate
EPA 330.5 SM4500-Cl G	Chlorine*	EPA 353.1 ISO 15923-1 ISBN 0117515930 SM 4500-NO3 H	Nitrate (TON Hyd)	EPA 376.2 ISBN 011751718 SM 4500-S2 D HMSSO SCA Blue Book	Sulfide*
EPA 120.1	Conductivity	NEMI (Nitrate via manual Vanadium (III) reduction)	Nitrate (TON Vanadium)	SMWW 4500-CN-M	Thiocyanate*
ASTM D2036-09(B)	Copper	EPA 354.1 ISO 13395:1996 ISO 15923-1 DIN EN 26777 ISBN 0117515930 SM 4500 NO2-B	Nitrite	EPA 351.2 ASTM D3590-11 (B) ISBN 0117521299 SMWW 4500-N(Org)	Total Kjeldahl Nitrogen (TKN)
EPA 335.4	Cyanide (Total)				
EPA 340.3 SM 3500-F D	Fluoride				
EPA 130.1	Hardness (Total)				
		EPA 150.2	pH		

\*Third party reagents; # after digestion



# Determination of ultratrace elements in semiconductor grade sulfuric acid using the Thermo Scientific™ iCAP RQ ICP-MS System

## Overview

Concentrated sulfuric acid ( $H_2SO_4$ ) is used in the semiconductor industry to remove organic substances from the surface of silicon wafers. Because  $H_2SO_4$  comes into contact with wafer surfaces, it is necessary to monitor its trace elemental impurities.

High sulfur matrices are problematic in ICP-MS analysis due to the formation of spectral interferences that are preferentially created due to their low ionization energies in the ICP ion source. The most challenging elements in the trace determination of sulfuric acid are Ti, V, Cr, Zn, and Ge. All major isotopes of these metals are severely interfered by polyatomic species generated by the sample matrix.

The iCAP RQ ICP-MS system provides excellent performance in the ultratrace determination of metals in semiconductor grade acid  $H_2SO_4$ . The flexible combination of different analysis modes has been shown to be ideally suited for ultratrace metal determination in advanced semiconductor applications.

[View the full application note](#)

Analyte	Mode	LoD (ng·L <sup>-1</sup> )	BEC (ng·L <sup>-1</sup> )	Recovery (%)
<sup>7</sup> Li	CP	0.3	0.9	99
<sup>23</sup> Na	CP	0.7	2.6	107
<sup>24</sup> Mg	CP-NH <sub>3</sub>	0.9	0.8	102
<sup>27</sup> Al	CP	0.9	1.1	99
<sup>39</sup> K	CP	2.2	8.5	102
<sup>40</sup> Ca	CP	5.9	17.0	103
<sup>48</sup> TiNH(NH <sub>3</sub> ) <sub>3</sub>	CCT-Cluster	2.7	21.7	97
<sup>51</sup> V	CCT-NH <sub>3</sub>	0.6	0.2	100
<sup>53</sup> Cr	CP-NH <sub>3</sub>	2.7	1.7	103
<sup>55</sup> Mn	CP	0.3	0.2	102
<sup>56</sup> Fe	CP	1.0	1.8	100
<sup>58</sup> Ni	CP	1.5	2.1	98
<sup>59</sup> Co	CP	0.3	0.1	99
<sup>65</sup> Cu	CP	1.9	1.1	103
<sup>68</sup> Zn	KED-NH <sub>3</sub>	0.5	2.9	99
<sup>71</sup> Ga	CP	0.1	0.1	101
<sup>74</sup> Ge	KED-NH <sub>3</sub>	0.7	0.1	108
<sup>85</sup> Rb	KED-NH <sub>3</sub>	1.0	0.8	100
<sup>88</sup> Sr	KED-NH <sub>3</sub>	0.5	0.6	105
<sup>111</sup> Cd	KED-He	1.2	0.4	107
<sup>115</sup> In	KED-He	0.3	0.5	107
<sup>138</sup> Ba	KED-NH <sub>3</sub>	1.1	0.1	96
<sup>205</sup> Tl	KED-He	0.6	0.5	92
<sup>208</sup> Pb	KED-He	1.5	1.4	97
<sup>209</sup> Bi	KED-He	0.5	3.1	98
<sup>238</sup> U	KED-He	0.4	0.4	101

## Systems solutions

Control of the process steps and wafer environment to meet the daily challenges of routine wafer compliance requires the use of many diverse characterization techniques, including many employing analytical instrumentation.

Learn about semiconductor fabrication and how Thermo Fisher Scientific electron microscopes and analytical instrumentation can help you control process steps and analyze the wafer environment throughout semiconductor manufacturing to ensure you are reaching the highest yield possible.



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