APPLICATION NOTE



ICP - Mass Spectrometry

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Analysis of Trace Elements in Coastal Seawater Using the NexION 2200 ICP-MS

Introduction

Coastal oceans are among the most diverse ecosystems on the planet, housing a wealth of marine life and serving as the primary source of global seafood production. However, coastal water is exposed to various sources of

contaminants. With the increasing industrialization of both land and sea, coupled with rising global populations, there is growing concern over the contamination of coastal seawater by metals and metalloids. This contamination not only poses a threat to the biological communities that inhabit these waters but also affects human communities that rely on local seafood as a source of sustenance. Therefore, it is important to monitor and assess the presence of potentially toxic heavy metals and metalloids.

Seawater is one of the most challenging sample types to analyze due to its high total dissolved solids (TDS) content in the matrix. As a powerful technology for traceelemental analysis, inductively coupled plasma mass spectrometry (ICP-MS) has the advantages of multi-element analysis capability, high sensitivity, low detection limits, wide linear dynamic range, and easy automation. Nevertheless, ICP-MS analysis, as with all analytical techniques, is subject to interferences. The high concentrations of matrix components in seawater, such as sodium, magnesium, and chloride ions, may form polyatomic spectral interferences making the determination of trace elements, such as arsenic, cobalt, vanadium, and iron, challenging. Even for elements like cadmium, thallium, and lead, that have fewer spectral interferences, their low concentrations make them difficult to determine with accuracy and precision.



The NexION[®] 2200 ICP-MS is the ideal solution for this application. It is equipped with Universal Cell Technology (UCT) which allows for samples to be run in Standard, Collision with KED, and Reaction with dynamic bandpass tuning (DBT) modes, and three reaction gas channels allow for versatile solutions to mitigate/eliminate spectral interferences.¹ In Collision mode with KED, a chemically inert gas, usually helium, collides with analytes and interferences. Polyatomic interferences, having larger cross-sectional diameters than elemental ions of the same mass (i.e., ⁴⁰Ar³⁵Cl⁺ is larger than ⁷⁵As⁺), experience more collisions and subsequently lose more kinetic energy than the analyte ions and cannot make it past the energy barrier at the exit of the cell. In Reaction mode with DBT, a chemically active gas reacts either with the target analyte or the interferent(s), either moving the target analyte to a new mass (e.g., ⁷⁵As⁺ reacts with O_2 to form ⁷⁵As¹⁶O⁺ at mass 91) or converting the interferent to a new species that does not interfere with the target analyte (e.g., ${}^{35}\text{Cl}{}^{16}\text{O}^+$ reacts with NH₃ to create neutral ClO in a charge transfer reaction, while ⁵¹V⁺ does not react with NH₃). Following this, the analyte is mass separated in the Analyzer Quadrupole which follows the cell and is thereafter detected free of interferences.

The NexION® 2200 ICP-MS also boasts a second-generation Triple Cone Interface with OmniRing[™] technology while maintaining the highly successful three stages of mass filtration supplied by the Quadrupole Ion Deflector (QID, Q0), Universal Cell Technology (UCT, Q1) and the Analyzer Quadrupole (Q2). The new interface brings exceptional flexibility, without requiring multiple inserts and lenses. On one hand, it brings enhanced sensitivity in Extraction and Cold Plasma modes and on the other hand, it provides improved detection limits or background equivalent concentrations (BECs) in Focusing mode, thanks to the three stages of pumping and elimination of surface deposition and ion sputtering. The Quadrupole Ion Deflector (QID) acts as an electrostatic analyzer that selectively focuses ions, maximizing signal sensitivity and eliminating neutral species and photons, ensuring cleanliness and no routine maintenance beyond the cones as well as superior stability and robustness.¹

Historically, to overcome the challenges associated with the analysis of high TDS samples, several strategies have been employed, such as matrix separation, analyte preconcentration, reductive precipitation, hydride generation techniques, etc. These tactics either involve offline sample preparation and/or use of extra equipment.

In this work, we reported the direct analysis of seawater using a straightforward online dilution approach, in which the dilution takes place using a combination of online internal standard addition and online gas dilution in the form of PerkinElmer's All Matrix solution (AMS).² PerkinElmer's High Throughput System (HTS)³ is also used, which rapidly delivers the sample to the plasma and shortens the washout time. The approach has been reported in our previous work for the analysis of coastal and open-ocean seawater using the NexION 2000 ICP-MS⁴ and the NexION 5000 ICP-MS,⁵ respectively.

Experimental

Sample Preparation

All sample and calibration solution preparation was performed volumetrically. Ultrapure water (Resistivity >18.2 M Ω .cm), highpurity acids HNO₃ (55% w/w, Tama Chemicals, Moses Lake, Washington, USA) and HCI (20% w/w, Tama Chemicals) were used for all samples, blanks, standards, and wash solutions unless otherwise specified. Diluted acid solutions were prepared via the dilution of the concentrated acid(s) with ultrapure water. Isopropanol (IPA) used in internal standard and washout solutions was electronic-grade for trace metals (99.999% trace metals basis, Sigma-Aldrich, Oakville, Ontario, Canada). The concentrated HNO₃ with 70% (w/w) and concentrated HCl with 37% (w/w).

Calibration Standards

The calibration standards were prepared by diluting ICP-MS grade single-element standards and multi-element standards (see the *Consumables Used* table) in a diluent made of 2% HNO₃ and 0.5% HCl and spiked with 200 ppb of gold to aid the mobility of Hg. This diluent was also used as the calibration blank, the continuing calibration blank (CCB) and the HTS carrier solution. The concentrations of the calibration standards are shown in Table 1. Standard 3 in this standard set was used as the continuing calibration verification (CCV) standard. The CCBs and CCVs were measured periodically throughout the sequence.

Analytes	Standard 1 (µg/L)	Standard 2 (µg/L)	Standard 3 (µg/L)	Standard 4 (µg/L)	Standard 5 (µg/L)	Standard 6 (µg/L)
Li, B, Fe, Ba, Sr	1	10	100	200	500	1,000
Mg, K, P	10	100	1,000	2,000	5,000	10,000
Na, Ca	50	500	5,000	10,000	25,000	50,000
Hg	0.01	0.1	1	2	5	10
Ag, Al, As, Be, Bi, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Rb, Sb, Se, Sn, Te, Th, Tl, U, V, W and Zn	0.1	1	10	20	50	100

Table 1. Analyte Concentrations in the Calibration Standards.

Wash Solution

The wash solution consisted of 1.5% HCl (v/v) and 1.5% HNO_3 (v/v) spiked with 200 μ g/L gold and 2% IPA. IPA was used to facilitate the washout of organic materials from the sample probe and sample loop.

Internal Standard (ISTD)

The internal standard solution contained 200 μ g/L of Sc, 80 μ g/L of In, Rh and Ir and was prepared by dilution of each single-element standard (see the *Consumables Used* table) in the calibration blank plus 0.1% IPA to address the difference of carbon content between calibration standard and seawater samples.

QC Samples

Two certified reference materials (CRMs) for coastal seawater CA403 (European Reference Materials Joint Research Center) and MX014 (Australian Government National Measurement Institute, North Ryde, New South Wales, Australia) were used to validate the accuracy of the method. Two seawater samples spiked with multi-element standards to the level of Standard 3 (Table 1) were analyzed for validation of the trace elements that were not certified by the CRMs.

Instrumentation

All measurements were performed on a NexION 2200 ICP-MS (PerkinElmer Inc., Shelton, Connecticut, USA) equipped with AMS, HTS and an S20 series autosampler. The instrument components/parameters are shown in Table 2. A liquid dilution of approximately 8-fold was achieved by using a larger internal diameter peripump tubing for the internal standard than is used for the sample/carrier. AMS was optimized to achieve a further dilution of approximately 3-fold for ¹¹⁵In in Standard mode, resulting in a total 24-fold dilution for this analysis. A Peltier controlled inlet system (PC3), was incorporated with the cyclonic spray chamber, and set at 5°C to achieve a finer and consistent aerosol stream for enhanced stability and reduced oxide formation. The instrument components and operating conditions are shown in Table 2. The settings for HTS are shown in Table 3. It should be noted that a longer read delay time (60 s) than normally used (30 s) was adopted for this method. The read delay time includes the time transferring sample from the valve to the nebulizer and plasma equilibrium. Since the carrier flow is smaller (30 µL/min) than normally used, a longer transferring time is required. With the high TDS, a longer plasma equilibrium time is preferred for better repeatability.

Table 2. NexION 2200 ICP-MS Instrument Components and Operating Conditions.

Component/ Parameter	Type/Value
Nebulizer	ST-PFA MicroFlow
Spray Chamber	Quartz cyclonic with AMS matrix port
Torch	One-piece quartz torch, 2 mm injector
Cones	Nickel sampler cone with Pt tip Nickel skimmer cone with Pt tip (0.88 mm) Ni hyper-skimmer cone w/OmniRing [™] assembly
Peristaltic Pump Tubing	Carrier: orange/red (0.19 mm i.d.) Internal Standard: orange/yellow (0.51 mm i.d.) Waste: gray/gray Santoprene (1.30 mm i.d.)
Carrier Solution Probe Internal Standard Probe	0.5 mm i.d.
Autosampler Probe	1.0 mm i.d.
Sample Loop	1.5 mL; 1.0 mm i.d.
PC3	5°C
Operating Conditions	Type/Value
RF Power	1600 W
Plasma Gas Flow	15 L/min
Auxiliary Gas Flow	1.2 L/min
Nebulizer Gas Flow	0.82 L/min
AMS Gas Flow	0.25 L/min
Cell Gas	Helium; 0 ₂
Sample Uptake Rate	Carrier: 30 µL/min Internal Standard: 250 µL/min
Data Acquisition	Type/Value
Sweeps	20
Dwell Time	10 - 300 ms
Poplicator	3

Table 3. HTS Settings

Value
30
100 rpm
5
8 s
60 s
35 s

Instrument Optimization and Method Setup

Prior to sample analysis, the instrument was tuned for optimal sensitivity and oxide, as well as, doubly charged ion ratios. Using the instrument components and conditions as shown in Table 2, the oxide level as measured by CeO $^+$ /Ce $^+$ was < 1% and the doubly charged ion ratio as measured by Ce²⁺/Ce⁺ was < 1.5% in Standard mode. It should be noted that new or newly cleaned cones need to be conditioned prior to sample analysis. In this method, the cones were conditioned by aspirating diluted seawater and monitoring the internal standards until the signals stabilized. The elements analyzed, their respective isotopes and mode of analysis used for each element in this method are listed in Table 4. Arsenic (As) and selenium (Se) were measured in oxygen Reaction (DRC) Mass Shift mode, and the rest of the elements were measured in helium Collision (KED) mode. A default RPq (cell rejection parameter) was used for the analytes measured in Collision mode to simplify the method development. For the oxygen Reaction mode, RPg was optimized to maximize the sensitivity of SeO. Universal Cell Technology (UCT) enables the optimization of the RPq setting (RF voltage) on the active mass filtering guadrupole inside the reaction cell. This optimization imparts additional energy to the ions inside the cell, allowing a slightly endothermic reaction, such as the formation of SeO to be driven to completion, thus optimizing sensitivity for the selenium mass shift product ion. This is an advantage that only the UCT technology in the NexION ICP-MS series instruments can provide. As illustrated in Figure 1, the sensitivity of SeO varied greatly with the RPg and was maximized at 0.65. The optimized RPg was used for the measurement of both SeO and AsO.



Figure 1. Sensitivity changes of SeO versus RPq values. 10 ppb Se solution was used for the test.

For most applications, determination of the trace elements in the seawater samples is of predominant interest; however, it can also be useful to verify the salinity levels of the samples for some applications. PerkinElmer's proprietary Extended Dynamic Range (EDR) feature has the capability to selectively attenuate signals on individual masses by adjusting the RPa (DC voltage) on the UCT to protect the detector from being exposed to overly large number of ions.⁶ In this work, Na was measured to assess the salinity of the seawater sample using the EDR feature to reduce the signal to a reasonable range whilst calibrating up to 50,000 µg/L.

Results and Discussion

Detection Limits

The method detection limits (MDLs) were calculated as three times the standard deviation of 10 replicated measurements of the method blank. Single to double digit ppts of MDLs were achieved for most trace elements as shown in Table 3, including the challenging elements arsenic and selenium at 5 and 12 ppt, respectively.

Linearity

Calibration curves were plotted following internal standard correction and blank subtraction. A coefficient of correlation (R) higher than 0.9995 was obtained for all elements in the calibrated ranges. Recoveries of the calibration standards for all analytes were within \pm 10% in the calibration ranges.

Accuracy

The accuracy of the method was validated via the recoveries of the seawater CRMs and standard-spiked seawater samples. For the CRM recovery test, excellent recoveries within \pm 10% were achieved for all analytes in MX014 and CA403, except for Ni and Co, which had recoveries of 88% and 89% respectively, as shown in Table 5. The Na concentrations were measured to be 11,400 ppm and 10,700 ppm for MX014 and CA403, which fell within the normal range of salinity in coastal seawater.

The spike recovery test was performed on two seawater samples, Seawater 1 and Seawater 2 spiked with multi-element standards to the level of Calibration Standard 3 (Table 1). To perform this test, the unspiked seawater sample was measured six times, and the mean concentration of each analyte was used as the subtrahend to calculate the spiked concentration. The mean concentrations of the unspiked and the spiked samples and spike recoveries are listed in Table 6. Recoveries within ± 10% except for Ag (87%) were obtained for all matrixspiked trace-element standards, validating the accuracy of the analysis of the trace elements that were not certified by the CRMs. The lower recovery of Ag may be due to the inherent sticky effect.

Analyte	Isotope	Cell Mode	Gas	Cell Flow, mL/min	RPq	RPa	Internal Standard	MDL (µg/L)
Al	27	KED	Helium	3.4	0.25	0	Sc	0.812
Р	31	KED	Helium	3.4	0.25	0	Sc	3.982
٧	51	KED	Helium	6.0	0.25	0	Sc	0.070
Cr	52	KED	Helium	6.0	0.25	0	Sc	0.057
Mn	55	KED	Helium	5.2	0.25	0	Sc	0.038
Fe	56	KED	Helium	6.0	0.25	0	Sc	0.095
Со	59	KED	Helium	5.2	0.25	0	Sc	0.002
Ni	60	KED	Helium	5.2	0.25	0	Sc	0.029
Cu	63	KED	Helium	5.2	0.25	0	In	0.026
Zn	66	KED	Helium	5.2	0.25	0	In	0.115
As*	91	DRC	Oxygen	3.2	0.65	0	Rh	0.005
Se*	94	DRC	Oxygen	3.2	0.65	0	Rh	0.012
Мо	95	KED	Helium	5.2	0.25	0	Sc	0.045
Ag	109	KED	Helium	5.2	0.25	0	Rh	0.016
Cd	111	KED	Helium	5.2	0.25	0	Rh	0.078
Sn	118	KED	Helium	5.2	0.25	0	In	0.076
Sb	121	KED	Helium	5.2	0.25	0	In	0.070
Ва	137	KED	Helium	5.2	0.25	0	In	0.146
W	182	KED	Helium	5.2	0.25	0	In	0.004
Hg	201	KED	Helium	5.2	0.25	0	lr	0.015
TI	205	KED	Helium	5.2	0.25	0	Rh	0.002
Pb**	206+207+208	KED	Helium	5.2	0.25	0	lr	0.001
Th	232	KED	Helium	5.2	0.25	0	In	0.008
U	238	KED	Helium	5.2	0.25	0	In	0.002
Na***	23	KED	Helium	5.2	0.25	0.015	Sc	21.24

Table 4. Isotopes and mode of analysis for different elements.

* Measured as the oxide aka AsO and SeO respectively ** Sum of three most abundant isotopes to address the natural abundance variation *** EDR applied

Table 5. Recoveries for the Certified Elements in Certified Reference Materials MX014 and CA403.

		MX014		CA403			
Element	Certified Value (µg/L)	Measured Value (µg/L)	Recovery (%)	Certified Value (µg/L)	Measured Value (µg/L)	Recovery (%)	
V	4.93	5.17	105				
Cr	2.701	2.55	94				
Mn	1.53	1.39	91	2.47	2.3	93	
Fe	22.43	21.17	94				
Со	2.961	2.62	88	0.074	0.077	103	
Ni	3.78	3.37	89	1.04	1.13	109	
Cu	2.99	2.83	95	0.87	0.9	103	
As	3.06	3.05	100	1.9	2	104	
Se	3.17	3.45	109				
Мо		9.27		12	10.78	90	
Cd	1.363	1.25	92	0.094	0.1	106	
Hg	0.448	0.44	99				
Pb	2.55	2.46	96	0.098	0.092	94	

Analyte	Spiked		Seawater 1		Seawater 2			
	Conc. (µg/L)	Unspiked (µg/L)	With Spike (µg/L)	Spike Recovery (%)	Unspiked (µg/L)	With Spike (µg/L)	Spike Recovery (%)	
Al	10	1.529	11.96	104	4.154	14.18	100	
Р	1000	19.08	965.1	95	20.65	989.2	97	
V	10	1.366	11.39	100	0.776	11.10	103	
Cr	10	0.127	9.686	96	0.120	9.926	98	
Mn	10	0.705	9.764	91	1.770	10.89	91	
Fe	100	0.731	94.86	94	1.669	98.93	97	
Со	10	0.027	9.185	92	0.069	9.127	91	
Ni	10	0.264	9.371	91	0.416	9.752	93	
Cu	10	0.206	9.638	94	0.530	9.868	93	
Zn	10	0.548	9.601	91	1.114	10.30	92	
As*	10	1.106	11.67	106	0.989	11.86	109	
Se*	10	0.212	10.22	100	0.345	10.87	105	
Мо	10	7.622	16.57	90	7.340	17.50	102	
Ag	10	0.002	8.723	87	0.003	8.919	89	
Cd	10	0.042	9.636	96	0.035	9.900	99	
Sn	10	0.019	9.988	100	0.011	10.24	102	
Sb	10	0.196	9.902	97	0.193	10.38	102	
Ва	100	4.363	103.4	99	6.491	111.6	105	
W	10	0.012	10.13	101	0.015	10.69	107	
Hg	1	0.007	1.021	101	0.006	1.074	107	
TI	10	0.010	9.683	97	0.010	10.49	105	
Pb**	10	0.002	9.861	99	0.010	10.09	101	
Th	10	0.006	10.08	101	0.005	10.61	106	
U	10	2.573	12.21	96	2.497	12.68	102	

Table 6. Spike Recovery Results

* Measured as the oxide aka AsO and SeO respectively

** Sum of three most abundant isotopes to address the natural abundance variation

Stability

To assess the long-term stability, seawater samples were analyzed repeatedly over an extended period of 12 hours and the CCV recoveries and the internal standards were monitored.

CCV Recovery: The CCV recoveries were normalized to Standard 3 (Table 1). As shown in Figure 2, for all the analytes,

the recoveries were within 90% - 115% of the original reading and there was no apparent trending through the runs, demonstrating the validity of the calibration over the 12-hour sample run. This performance is important for high-throughput laboratories in terms of overall efficiency and productivity by avoiding the frequent reruns of the calibration standards.





Internal Standard Recovery: The internal standards were normalized to the calibration blank, and the time resolved plot is shown in Figure 3(a). The higher recoveries were from the CCVs and the blanks while lower recoveries were from the seawater samples, which is not surprising considering the suppression effect from the high concentrations of easily ionized elements (EIEs) in seawater samples. The overall IS recoveries were largely within the 60% - 120 % range, and the stability is displayed more impressively by the plot of seawater samples only, as shown in Figure 3(b). These findings demonstrate the outstanding stability and robustness of this method and system and their suitability for the extended sample runs.

Conclusion

This application note reported a procedure for the direct analysis of trace elements in coastal seawater using the NexION 2200 ICP-MS equipped with AMS, HTS and PC3. Online dilution was realized by a combination of online internal standard addition and online gas dilution provided by AMS. The overall matrix load is further reduced by using the HTS sample introduction system. A Peltier-controlled inlet system (PC3) was used and set to 5 °C to achieve a finer and consistent aerosol stream to enhance stability and oxide performance.

The analyses were performed using two cell gas modes, the helium Collision (KED) mode and the oxygen Reaction (DRC) mode. As

and Se were measured using oxygen Mass Shift Reaction mode for best analytical performance, and the rest of the elements were measured in helium Collision mode.

Aqueous calibration standards were used for quantification to simplify the standard preparation and provide an option when a high-purity matrix standard is not available.^{4,5}

The data quality and instrument performance were evaluated by the method detection limits (MDLs), the linearity, the accuracy and long-term stability. Single- to double-digit ppt of MDLs were achieved for most trace elements. The accuracy of the method was validated via the recoveries of the seawater CRMs and standard-spiked seawater samples. For the CRM recovery test, excellent recoveries within ± 10% were achieved for most analytes in MX014 and CA403, except for Co and Ni that had recoveries of 88% and 89% respectively. The accuracy was further evaluated by the spike test. Recoveries within ± 10% except for Ag (87%) were obtained for all matrix-spiked traceelement standards, validating the accuracy of the analysis of the trace elements that were not certified by the CRMs.

Excellent consistency of the internal standards and CCV recoveries over an impressive 12-hour period demonstrated the suitability, robustness and stability of the method and system used for the analysis of seawater.



Figure 3. Internal standard recoveries during a 12-hour sequence of analysis of seawater samples and CCVs and blanks. The numbers inside the parentheses are the gas flows used in this work.

References

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- 3. High Throughput System for ICP-MS/OES, Technical Note, PerkinElmer, 2020.
- 4. Liyan Xing and Chady Stephan; Direct Analysis of Trace Element in Coastal Seawater Using the NexION 2000 ICP-MS. PerkinElmer Application Note, 2021.
- 5. Liyan Xing and Chady Stephan; Direct Analysis of Trace Elements in Open-Ocean Seawater Using the NexION 5000 ICP-MS; PerkinElmer Application Note, 2022.
- 6. https://www.perkinelmer.com/library/extended-dynamicrange-feature-on-nexion-icp-ms-series-instruments.html, PerkinElmer, 2022.

Consumables Used

Component	Description	Part Number
Nebulizer	PFA-ST3	N8152378
Nebulizer Line	ST Nebulizer line, 0.25 mm i.d.	N0812026
Spray Chamber	SilQ Cyclonic High Sensitivity Spray Chamber with Matrix Gas Port	N8152424
Torch	One Piece Ultrapure Quartz Torch with 2.0 mm Injector	N8152428
Sampler	Sampler with Pt Tip	N8161140
Skimmer	Skimmer with Pt Tip (0.88 mm)	N8161041
Peripump Tubing	ISTD: Orange/Yellow (0.51 mm i.d.) Carrier: Orange/Red (0.19 mm i.d.) Waste: Gray/Gray Santoprene (1.30 mm i.d.)	N8152404 N8152401 N8152415
Internal Standard Probe Carrier Sample Probe	Carbon Fiber Autosampler Probe for ST Nebulizers	N0777225
Autosampler Probe used with HTS	Carbon Probe 1.0 mm i.d. 2 Blue Bands	N0811956
HTS Sample Loop	Sample Loop 1.0 mL; 1.0 mm i.d.	N8152929
Multi-Element Standard 1*	10 $\mu g/mL$ of Ag, Al, As, Be, Bi, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Rb, Sb, Se, Sn, Te, Tl, V, $$ W and Zn in 5% HNO_3/Trace Tartaric Acid/Trace HF	N8145600
Multi-Element Standard 2*	5000 μg/mL of Ca and Na 1000 μg/mL of K and Mg 100 μg/mL of Ba, Fe, Li, and Sr 10 μg/mL of Th in 5% HNO ₃	N8145601
	1000 μ g/mL P in H ₂ O	N9303788
	1000 μg/mL B in H ₂ 0	N9303760
	1000 μg/mL Au in 10% HCl	N9303759
	1000 μg/mL Ir in 10% HCl	N9303778
Single-Element Standard	1000 μ g/mL Hg in 10% HNO $_3$	N9303740
	10 μ g/mL Sc in 2% HNO ₃	N9303741
	10 μ g/mL in in 2% HNO ₃	N9303738
	10 μg/mL Rh in 2% HCl	N9303749
	1000 μ g/mL Sr in 2% HNO ₃	N9303802

* Subset of PerkinElmer's True MS Calibration Std Kit ISO 17294

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