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# Analysis of Undiluted Seawater using ICP-MS with Ultra High Matrix Introduction and Discrete Sampling

Accurate, stable, low-level analysis using the Agilent 7850 ICP-MS with ISIS 3 DS

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## Introduction

The 17 Sustainable Development Goals (SDGs) adopted by the United Nations in 2015 aim to end poverty, protect the planet, and ensure that all people enjoy peace and prosperity by 2030 (*1*). All 17 SDGs relate to the environment either directly (13 of the 17 goals) or indirectly (the remaining four goals). Working towards the SDGs requires extensive on-going monitoring of all types of environmental samples such as air, different types of water, including seawater, and soils in all parts of the world.

According to the UN: "Our oceans—their temperature, circulation, chemistry, and ecosystems—play a fundamental role in making Earth habitable. Careful management of this essential global resource is a key feature of a sustainable future." To reflect the importance of the marine environment, the aim of Goal 14 is to 'Conserve and sustainably use the oceans, seas and marine resources.' Specifically, Target 14.1 sets out to reduce marine pollution, particularly from land-based activities, by 2025 rather than 2030. Trace elements present in industrial and domestic wastewaters are a major source of marine pollution. Therefore, determining the inorganic content of seawater samples is an important assessment method that is needed to achieve Target 14.1.

ICP-MS is a fast, multi-element technique that is often used to measure multiple analytes in a wide range of complex environmental samples that may contain high levels of total dissolved solids (TDS) (2−5). Measuring a wide range of elements in seawater by ICP-MS is especially challenging due to the very high salt content of seawater (6−8). When samples with high TDS are analyzed directly (undiluted) by ICP-MS, undissociated matrix may deposit on the interface cone orifices, impacting the sensitivity of measurements and the long-term stability of the instrument. Also, high concentrations of easily ionized elements such as Na and K in a sample reduce the ionization of other elements with higher ionization potentials, an effect known as "ionization suppression". A further challenge is that the major elements present in seawater - such as Na, Ca, Cl, and S - can form polyatomic ions in the ICP-MS mass spectrum. For example, $^{35}Cl^{16}O$ can interfere with $^{51}V$, and $^{40}Ar^{23}Na$ overlaps $^{63}Cu$. These interferences prevent the accurate determination of trace-levels of Ti, V, Cr, Ni, Cu, Zn, As, and other elements.

To compensate for these challenges, some ICP-MS methods for the analysis of seawater use high sample dilution factors or matrix removal using chelating agents. These additional sample preparation stages are time consuming and there is the risk of contamination from diluents and chelate resins.

Analysts can use Agilent ICP-MS for the direct analysis of multiple elements in high matrix samples due to a number of developments. To deal with the spectral interferences, Agilent ICP-MS systems include the ORS⁴ collision/reaction cell (CRC). The ORS⁴ is optimized for the removal of all common polyatomic overlaps using helium (He) collision mode and kinetic energy discrimination (KED). He mode allows the same cell settings to be used for most elements in many common sample types, simplifying routine analysis.

Agilent ICP-MS instruments are also equipped with the Ultra High Matrix Introduction (UHMI) aerosol dilution system for the direct introduction of high matrix samples into the ICP-MS. UHMI dilutes the sample aerosol using argon gas, which ensures that a higher temperature, more robust ICP is achieved, so stability can be maintained over long runs of high matrix samples. Plasma robustness is monitored using the CeO⁺/Ce⁺ ratio, with a lower ratio indicating a more robust plasma (9). The robust operating conditions (low CeO/Ce ratio) of Agilent ICP-MS provide effective matrix decomposition and analyte ionization in high matrix samples, minimize drift over long runs, and reduce the frequency of routine maintenance of the interface cones. As shown in this study, even undiluted seawater can be measured directly with good accuracy and long-term stability using the Agilent 7850 ICP-MS.

Agilent ICP-MS MassHunter software (version 5.1 or later) also includes a series of useful features such as autotuning, preset methods, IntelliQuant and Early Maintenance Feedback (EMF) to help laboratories run their ICP-MS analysis more efficiently. These features assist operators to optimize method development, avoid unnecessary instrument maintenance, and generate high-quality data for challenging samples, such as seawater.

## Experimental

### Standard and sample preparation

The calibration standards and samples were prepared in an acid matrix of 1% $HNO_3$ and 0.5% HCl. HCl is routinely added to samples for ICP-MS analysis to ensure the stability of elements such as Sb and Hg. The standard He cell mode of the Agilent ORS⁴ CRC removes any Cl-based interferences that would be formed from the chloride matrix.

Calibration standards, spikes, and most of the quality control (QC) standards were prepared from Agilent Environmental calibration standard, part number 5183-4688. Single element standards were used for B, P, Ti, Sn, Hg (Kanto Chemicals, Japan). At high concentrations, Sn is not chemically stable for extended periods in the relatively low HCl levels used to prepare the calibration standards. To ensure calibration linearity and stability for Sn, the standards were prepared fresh prior to each batch analysis.

Six-point calibrations including the calibration blank were prepared at the concentration ranges given in Table 1. For the in-run QC, the Environmental calibration standard and single element standards for B, P, Ti, Sn, and Hg were mixed in the 1% $HNO_3$ and 0.5% HCl acid matrix. The concentrations of most of the QC samples were at the midpoint of the calibration curves.

The samples included a synthetic seawater (Marine Art, Osaka Yakken, Japan), real seawater (collected in Chiba, Japan), and NMIA MX014 Trace Elements in Sea Water certified reference material (National Measurement Institute CRM, Australia) (10). Matrix spiked samples were prepared by spiking the standards into each of the seawater samples. Spike concentrations are shown in Table 6.

The Internal Standard (ISTD) solution (Agilent part number 5183-4681) contained 2 ppm Li, Y, In, and Bi in an acid matrix of 1% $HNO_3$ and 10% HCl. 10% HCl was added to the ISTD solution to matrix match the seawater samples, which include a high concentration of chloride ions. The solution was automatically added online via the dedicated seventh port of the Agilent ISIS sampling system valve. The flow rate of the ISTD solution was about 1/15 of the sample flow rate, due to the use of smaller internal diameter pump tubing.

**Table 1.** Elements, calibration concentration range, and concentration of the QC sample.

| Elements | Calibration Range (ppb) |
|---|---|
| Na, Mg, Ca, Fe | 10 to 100,000 |
| Other trace elements | 0.1 to 100 |
| B | 10 to 5,000 |
| P | 10 to 1,000 |
| Ti, Sn | 1 to 100 |
| Hg | 0.01 to 1 |

## Instrumentation

An Agilent 7850 ICP-MS equipped with the ISIS 3 discrete sampling (DS) system was used for the analysis of the varied seawater samples. The 7850 includes a nickel-tipped copper sampling cone, nickel skimmer cone, UHMI aerosol dilution system, and ORS$^4$ CRC.

For the analysis of most typical sample types, analysts can use a preset method, automatic plasma correction by selecting the UHMI setting, lens autotune, and single He cell mode. He mode is also used as the default mode for Quick Scan data acquisition, ensuring that IntelliQuant (semiquantitative) data is free from the errors that can be caused by common polyatomic ion overlaps (*10*). However, depending on the objectives of the analysis, the analyst has the option to apply different cell gas modes and adjust the lens tuning to maximize sensitivity.

In this study, lower detection limits were required for the direct measurement of the levels of trace elements that are naturally present in seawater. To maximize the sensitivity of the measurements, the ORS$^4$ cell was operated in no gas, He, Enhanced He (HEHe) (*11*), and H$_2$ cell gas modes. To provide robust and stable plasma conditions over long analytical runs, the UHMI was set at dilution level 8 (UHMI-8). When analyzing high salt samples, it is common practice to humidify the carrier gas to prevent salt build up on the nebulizer and torch injector. In this work, both nebulizer gas and dilution gas were humidified using an Agilent argon humidifier. The CeO/Ce ratio in He mode was approximately 0.2%. The lens parameters were optimized for long-term stability.

Typically, before the measurement of high matrix samples by ICP-MS, the instrument's sample introduction system is preconditioned. In this study, a 500 ppm Ca standard solution was aspirated for 15 min using General Plasma operating conditions. After a quick rinse using a blank solution, the undiluted seawater sample was then aspirated for 15 min. Finally, the blank sample was introduced to the instrument for 30 min to rinse the sample introduction system.

Sample delivery was performed using the Agilent SPS 4 autosampler. The ISIS 3 DS system speeds up the overall analysis by starting the rinse program while acquiring data for the previous sample. ISIS 3 DS shortened the analysis time by two minutes compared with conventional sample introduction, while minimizing carry over from previous samples. Details of the ISIS 3 configuration and functionality are given elsewhere (*4, 5*).

Instrument operating conditions are shown in Table 2 and ISIS 3 operating parameters are shown in Table 3.

**Table 2.** Agilent 7850 ICP-MS operating parameters.

| Parameter | Setting | | | |
|---|---|---|---|---|
| Plasma Mode | UHMI-8 | | | |
| RF Power (W) | 1600 | | | |
| Sampling Depth (mm) | 10 | | | |
| Nebulizer Gas (L/min) | 0.70 | | | |
| Dilution Gas (L/min) | 0.28 | | | |
| Extract 1 (V) | −4 | | | |
| Extract 2 (V) | −250 | | | |
| Omega Bias (V) | −100 | | | |
| Omega Lens (V) | 9 | | | |
| Gas Mode | No gas | H$_2$ | He | HEHe |
| Deflect (V) | 13.2 | 1.2 | 1.4 | −74 |
| Gas Flow Rate (mL/min) | - | 4.5 | 4.5 | 10 |
| Energy Discrimination (V) | 5 | 5 | 4 | 7 |

*The shaded parameters were set automatically by the ICP-MS MassHunter software when UHMI-8 was selected.*

**Table 3.** Agilent ISIS 3 parameters.

| Parameter | Setting | |
|---|---|---|
| Loop Volume (mL) | 1.4 | |
| | Time (s) | Pump Speed (%) |
| Sample Load | 9 | 35 |
| Stabilize | 23 | 5 |
| Probe Rinse | 25 | 8 |
| Probe Rinse 2 | 10 | 60 |
| Probe Rinse 3 | 10 | 5 |
| Optional Loop Probe Wash | 13 | 60 |
| Optional Loop Wash | 3 | 5 |

**Table 4.** Gas mode, integration time, ISTD, and MDLs.

| Analyte | Gas Mode | Integration Time (s) | ISTD | MDL (ppb) |
|---|---|---|---|---|
| 9 Be | No gas | 0.3 | 6 Li | 0.003 |
| 11 B | No gas | 0.3 | 6 Li | 9.67 |
| 23 Na | He | 0.1 | 45 Sc | 98.7 |
| 24 Mg | He | 0.1 | 45 Sc | 0.874 |
| 31 P | HEHe | 0.3 | 45 Sc | 5.59 |
| 44 Ca | $H_2$ | 0.3 | 45 Sc | 3.08 |
| 47 Ti | He | 0.3 | 45 Sc | 0.218 |
| 51 V | He | 0.3 | 45 Sc | 0.033 |
| 52 Cr | He | 1.0 | 45 Sc | 0.022 |
| 55 Mn | He | 0.3 | 45 Sc | 0.070 |
| 56 Fe | $H_2$ | 1.0 | 45 Sc | 0.058 |
| 59 Co | He | 0.3 | 103 Rh | 0.006 |
| 60 Ni | He | 0.5 | 103 Rh | 0.022 |
| 63 Cu | He | 0.5 | 103 Rh | 0.025 |
| 66 Zn | He | 0.5 | 103 Rh | 0.043 |
| 75 As | He | 1.0 | 89 Y | 0.076 |
| 78 Se | $H_2$ | 1.0 | 89 Y | 0.010 |
| 95 Mo | He | 0.3 | 103 Rh | 0.008 |
| 111 Cd | He | 1.0 | 103 Rh | 0.004 |
| 118 Sn | He | 0.3 | 115 In | 0.054 |
| 121 Sb | He | 0.3 | 115 In | 0.009 |
| 201 Hg | He | 3.0 | 209 Bi | 0.005 |
| Pb* | He | 0.33 x 3 | 209 Bi | 0.004 |
| 238 U | He | 0.3 | 209 Bi | 0.003 |

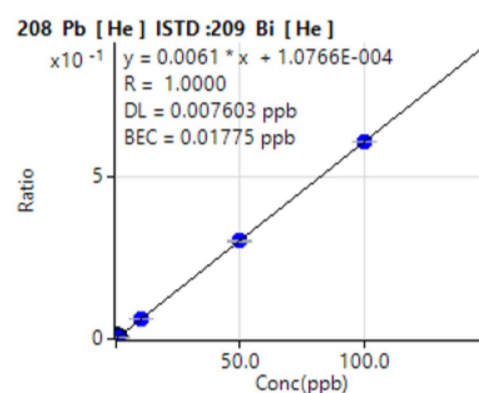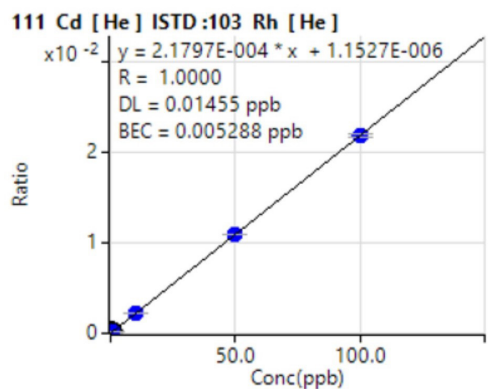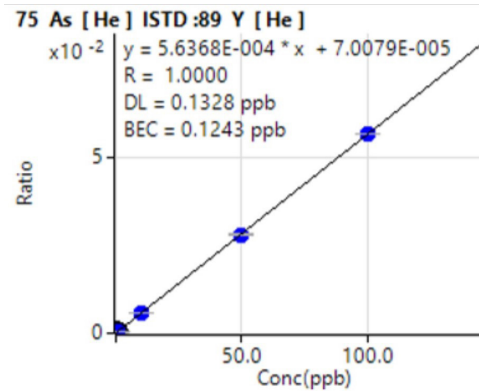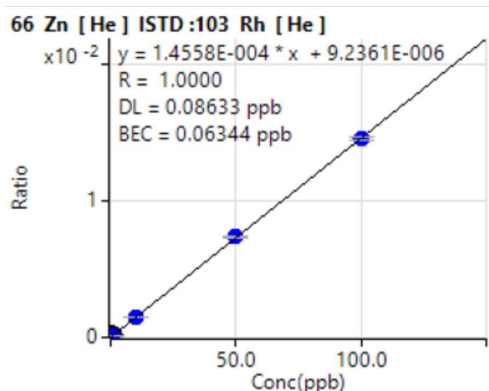*Pb was measured as the sum of the three most abundant isotopes, 206, 207 and 208.*

# Results and discussion

## MDLs and calibration curves

All analytes were measured using the acquisition parameters listed in Table 2. Method Detection Limits (MDLs) were calculated from three times the standard deviation of 10 measurements of the calibration blank in the acid diluent matrix (Table 4). The MDLs for the trace analytes were mostly in the low ng/L (ppt) level, confirming the high sensitivity of the 7850 ICP-MS for the application. [201]Hg is the preferred (default) isotope for varied sample types, although [202]Hg could also be measured and may give a slightly lower DL due to its higher abundance. Figure 1 shows the calibration curves for Mg, P, Cr, Ni, Zn, As, Cd, and Pb. The calibrations demonstrate good linearity over a wide dynamic range from 0.1 ppb to 100 ppm, as indicated by the calibration coefficients, R, which were greater than 0.9999.
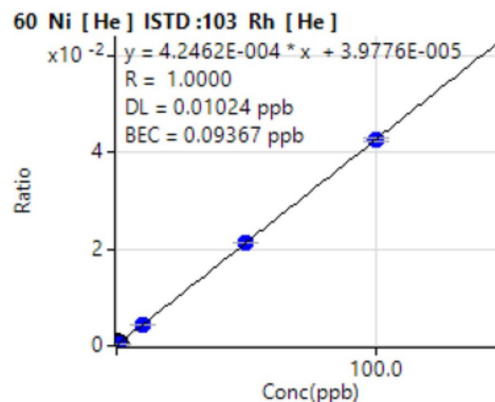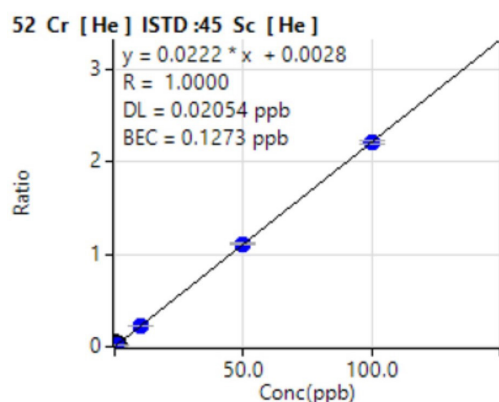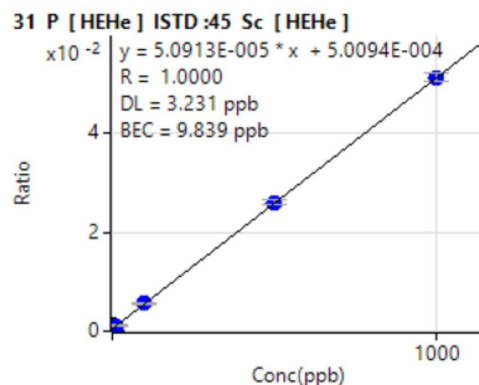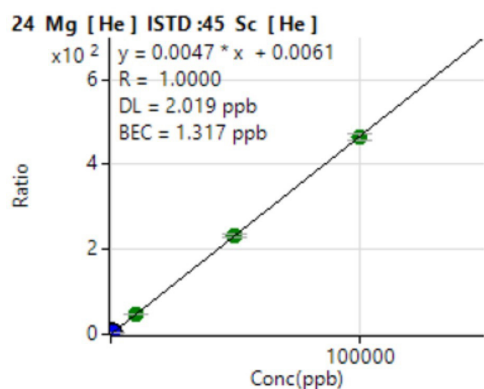
**Figure 1.** Calibration plots for Mg, P, Cr, Ni, Zn, As, Cd, and Pb.

## IntelliQuant determination of the level of solids in a sample

IntelliQuant Quick Scan data was collected as part of the quantitative method. The IntelliQuant results provide semiquantitative concentrations for up to 78 elements without needing specific standards for each element. IntelliQuant also supports identification and confirmation of unexpected elements by comparison with isotopic abundance templates. The periodic table "heat map" view of the results provides a quick and simple overview of the concentration of all elements within the sample. IntelliQuant can also calculate and display the total matrix solids (TMS) content of each sample (12). TMS is a powerful tool for method development for new or unusual sample types, enabling representative samples to be quickly assessed for total matrix level. TMS can also be used to track the sample load on the interface cones, helping to plan routine maintenance. The TMS results for a seawater sample, QC, and blank are shown in Figure 2.

| Sample Name | TMS (ppm) | 23 Na Conc. | 23 Na SQ Unit | 24 Mg Conc. | 24 Mg SQ Unit |
|---|---|---|---|---|---|
| seawaterB | 11161.849 | 9615.383 | mg/l | 1095.728 | mg/l |
| QC | 106.559 | 91.496 | mg/l | 4.327 | mg/l |
| blank | 2.703 | 2.353 | mg/l | 0.093 | mg/l |

**Figure 2.** IntelliQuant TMS data and semiquantitative concentrations of Na and Mg in three samples. Accurate QC recoveries were obtained for Na (91%) and Mg (86%) from the Quick Scan data.

## Analysis of seawater samples and seawater CRM

The two seawater samples and the seawater CRM were each analyzed three times by the 7850 ICP-MS. The mean concentrations and CRM recoveries are shown in Table 5. All the certified elements gave recoveries within ±10%, demonstrating the accuracy of the method. Not all the analytes have certified values in NMIA MX014, so blank cells in Table 5 indicate the absence of a certified (or reference) value.

**Table 5.** Measured concentrations in synthetic seawater, local seawater, and recoveries of certified elements in the NMIA MX014 seawater CRM. Concentration units µg/L except for elements in shaded rows that are in mg/L.

|  | Synthetic Seawater | Seawater Sample | NMIA MX014 | NMIA MX014 Certified Value | Recovery (%) |
|---|---|---|---|---|---|
| 9 Be | 0.114 | 0.003 | <MDL | - | - |
| 11 B | 5.18 | 5.58 | 6.20 | - | - |
| 23 Na | 8,960 | 9,070 | 9,580 | - | - |
| 24 Mg | 1,240 | 1,090 | 1,160 | - | - |
| 31 P | 10.9 | 59.1 | 26.0 | - | - |
| 44 Ca | 377 | 381 | 415 | - | - |
| 47 Ti | <MDL | <MDL | <MDL | - | - |
| 51 V | 0.057 | 1.77 | 4.80 | 4.76 ± 0.12 | 101 |
| 52 Cr | 0.630 | 0.129 | 2.35 | 2.613 ±0.075 | 90 |
| 55 Mn | 1.07 | 0.154 | 1.37 | 1.48 ± 0.16 | 93 |
| 56 Fe | 6.52 | 0.327 | 20.5 | 21.70 ± 0.32 | 94 |
| 59 Co | 0.428 | 0.033 | 2.98 | 2.864 ± 0.068 | 104 |
| 60 Ni | 0.246 | 0.548 | 3.61 | 3.66 ± 0.10 | 98 |
| 63 Cu | 0.232 | 0.643 | 2.94 | 2.90 ± 0.25 | 101 |
| 66 Zn | 0.142 | 1.38 | 9.88 | - | - |
| 75 As | 0.133 | 1.52 | 3.01 | 2.96 ± 0.26 | 102 |
| 78 Se | 0.130 | 0.092 | 2.75 | 3.06 ± 0.26 | 90 |
| 95 Mo | 9.84 | 11.7 | 11.2 | - | - |
| 111 Cd | 0.022 | 0.017 | 1.21 | 1.318 ± 0.034 | 92 |
| 118 Sn | 0.029 | 0.022 | 0.043 | - | - |
| 121 Sb | 0.043 | 0.210 | 0.185 | - | - |
| 201 Hg | 0.017 | 0.016 | 0.404 | 0.433 ± 0.010 | 93 |
| Pb* | 0.162 | 0.008 | 2.51 | 2.467 ± 0.065 | 102 |
| 238 U | 0.022 | 3.14 | 3.24 | - | - |

*Pb was measured as the sum of the three most abundant isotopes, 206, 207 and 208.

## Spike recovery

Matrix Spike (MS) results for the two seawater samples and the seawater CRM are given in Table 6. Since seawater contains B, Na, Mg, and Ca at a higher concentration than the 1000 µg/L spike, no recovery results are given for these elements. Recoveries for the other elements were mostly within ±10% of the spike level, further demonstrating the accuracy of the method.

**Table 6.** Spike recoveries (%) of synthetic seawater, local seawater, and NMIA MX014 seawater CRM.

| Element | Spike Conc (ppb) | Synthetic Seawater (%) | Seawater Sample (%) | NMIA MX014 (%) |
|---|---|---|---|---|
| 9 Be | 10 | 109 | 110 | 104 |
| 31 P | 500 | 115 | 111 | 118 |
| 47 Ti | 10 | 111 | 101 | 107 |
| 51 V | 10 | 102 | 99 | 99 |
| 52 Cr | 10 | 96 | 94 | 93 |
| 55 Mn | 10 | 98 | 99 | 97 |
| 56 Fe | 1000 | 100 | 101 | 100 |
| 59 Co | 10 | 106 | 108 | 106 |
| 60 Ni | 10 | 103 | 102 | 100 |
| 63 Cu | 10 | 100 | 101 | 98 |
| 66 Zn | 10 | 103 | 101 | 95 |
| 75 As | 10 | 104 | 103 | 100 |
| 78 Se | 10 | 96 | 97 | 89 |
| 95 Mo | 10 | 107 | 108 | 107 |
| 111 Cd | 10 | 100 | 100 | 99 |
| 118 Sn | 10 | 102 | 99 | 98 |
| 121 Sb | 10 | 106 | 105 | 101 |
| 201 Hg | 0.1 | 107 | 103 | 109 |
| Pb* | 10 | 105 | 105 | 103 |
| 238 U | 10 | 108 | 106 | 103 |

*Pb was measured as the sum of the three most abundant isotopes, 206, 207 and 208.

## Long-term stability

To demonstrate the robustness of the 7850 ICP-MS for the analysis of undiluted seawater, 120 samples and 30 standards and QC samples were analyzed repeatedly for seven hours. Using the combination of ISIS discrete sampling and UHMI aerosol dilution, the 7850 can tolerate such long-term, routine analysis of high matrix samples. Nevertheless, high matrix levels do lead to signal variation due to changes in the physical processes of sample transport, nebulization, and aerosol transfer.

Also, even with a well-optimized and robust plasma, some ionization suppression will occur when high salt samples are measured. To correct for these physical and ionization effects, analysts add internal standards automatically online to each sample solution.

Figure 3 shows that the ISTD recoveries remained within 70–120% throughout most of the run, with no mass-dependent drift. The ISTD recovery data confirms the robustness and matrix tolerance of the plasma and UHMI for minimizing matrix deposition on the instrument's interface. The ISTD plot also shows that the 7850 provides excellent control of signal suppression, with ISTD signals being consistent for elements covering a range of masses and ionization potentials. A QC test was also performed. The recoveries of all elements in the QC sample were within 90–110% throughout the run, showing the stability of the method (Figure 4).

Both sets of recovery results demonstrate the long-term robustness and high matrix tolerance of the 7850 ICP-MS with UHMI.
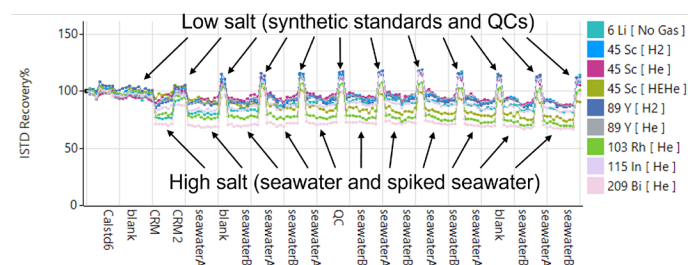


**Figure 3.** ISTD stability for 150 sequences (120 samples, 7 hours). Due to the space limitation, not all sample names are shown. The alternating high and low ISTD signals are due to changes in transport and nebulization of the low and high matrix sample types respectively. Low matrix samples include the synthetic calibration standards and QC solutions, while high matrix samples are the undiluted seawater and spiked seawater solutions.
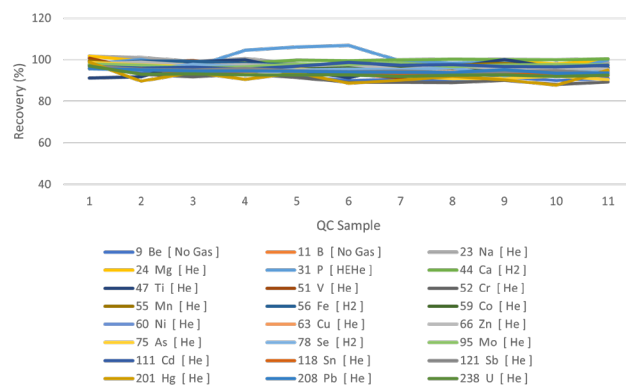


**Figure 4.** Recoveries of the elements in the QC sample over seven hours.

### Automatic maintenance alerts

Analyzing high matrix samples such as seawater can be tough on the sample introduction system of an ICP-MS. To maximize analytical performance and minimize unplanned instrument downtime, it can be beneficial to schedule routine maintenance tasks based on the type of sample and workload. With ICP-MS MassHunter, an analyst can set sample-type specific counters based on sample number or elapsed time to monitor instrument use and trigger EMF alerts (13). The ISIS 3 discrete sampling system reduces the sample matrix reaching the cones, allowing those counters to be set to higher values.

As well as the normal instrument prerun performance check, a postrun check can be scheduled to run after the sample sequence has completed. The postrun check gives analysts a clear indication if they need to perform routine maintenance tasks such as cleaning the cones before the next morning's run, making scheduling maintenance easier.

## Conclusion

The study has shown the suitability of the Agilent 7850 ICP-MS fitted with the Agilent ISIS 3 discrete sampling system for the long-term analysis of multiple elements in undiluted seawater samples.

Analyzing samples with high matrix levels by ICP-MS can lead to signal drift, suppression, and polyatomic ion interferences. The 7850 analysis was successful due to the robustness of the plasma with UHMI-8, interference removal capability of the ORS[4], and ICP-MS MassHunter software functions that provide an optimized analytical workflow.

The stability of the internal standard measurements made during the analysis of undiluted seawater samples over seven hours demonstrated the matrix tolerance and stability of the 7850 with UHMI aerosol dilution technology. Instrument stability is important for the productivity of routine, high throughput applications, as it reduces drift, QC failures, sample reanalysis, and maintenance.

The accuracy of the method was evaluated by analyzing a seawater CRM and conducting a spike recovery test for 24 elements in two seawater samples and the seawater CRM. All certified elements measured in the CRM were within ±10% and matrix spike results were also mostly within ±10%. The accuracy of the results was due to the effective control of polyatomic interferences of the ORS[4] in helium and hydrogen mode. Getting the right answer first time avoids the need to remeasure samples, which can be a significant burden for routine laboratories that use analytical techniques with less reliable control of spectral overlaps.

The EMF routine and postrun performance check were used to alert the analyst when maintenance was needed. IntelliQuant also provided an estimate of the total matrix content of the seawater digests, which can also help with scheduling of routine maintenance and avoiding any unplanned instrument downtime.

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11. Enhanced Helium Collision Mode with Agilent ORS[4] Cell, Agilent publication, 5994-1171EN

12. Agilent IntelliQuant for ICP-MS, Agilent publication, 5994-2796EN

13. Smart Self-Health Checks for ICP-MS Instruments, Agilent publication, 5994-2780EN

# Consumables list

| Consumable Description | Agilent Part Number |
|---|---|
| Sampler cone: nickel-tip with copper base. Standard cone used with x-lens for most routine applications | G3280-67040 |
| ICP-MS skimmer cone: nickel. Standard cone used with x-lens for most common applications | G3280-67041 |
| Easy-fit peristaltic pump tubing, standard for sample uptake, recommended for aqueous, acid, or alkaline matrix, 1 pack of 12 pcs | 5005-0020 |
| Easy-fit peristaltic pump tubing, standard for Internal Standard uptake, recommended for aqueous, acid, or alkaline matrix, 1 pack of 12 pcs | 5005-0021 |
| Easy-fit peristaltic pump tubing, long-life, standard for spray chamber drain, 1 pack of 12 pcs | 5005-0022 |
| Argon humidifier, two gas lines | G8400-60700 |
| Tubing set for argon humidifier | G8400-60710 |
| Environmental calibration standard, 100 mL | 5183-4688 |
| Internal standard mix, 100 mL | 5183-4681 |

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