Direct Analysis of a 25% Sodium Chloride Sample Matrix Using the Thermo Scientific iCAP RQ ICP-MS with Argon Gas Dilution

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Key Words

25% NaCl, AGD, ICP-MS, High Matrix Samples, Sea Water

Goal

To create a method to directly analyze ultra-high matrix samples such as 25% w/w NaCl ,using the Thermo Scientific™ iCAP™ RQ ICP-MS and Argon Gas Dilution (AGD), in conjunction with a fast valve sample introduction system to improve productivity and reduce memory effects.

Application Brief

Dealing with high matrix samples is a challenge in ICP-MS. Matrix effects can significantly reduce the detection sensitivity, jeopardize the internal standard responses and lead to increased maintenance or unwanted downtime of the instrument caused by blocked cones or nebulizers.

Solutions based on automated and intelligent (liquid) dilution are available and have the capability to reduce the matrix load of a given sample by more than two orders of magnitude (e.g. from 25% to 0.25% TDS), but may not always be the best solution due to increased cost for hardware and diluents.

Using Argon Gas Dilution (AGD), the direct analysis of seawater (containing approximately 3.5% TDS) is feasible[1], but the analysis of even higher TDS samples may still have limitations in terms of the achievable dilution factors or memory effects. In this study, a combination of AGD, online dilution and the use of a segmented flow sample introduction system was used to achieve a fast, easy and low cost solution for the direct analysis of ultra-high matrix samples (25% w/w NaCl).

Method

A Thermo Scientific iCAP RQ ICP-MS was used to perform all analyses. The samples were introduced using an SC-2DX Autosampler (Elemental Scientific, Omaha, NE) with a FAST valve system. The samples were loaded into the sample loop using the instrument's peristaltic pump to reduce sample consumption and diluted 14 times through the online addition of the internal standard solution.

The iCAP RQ ICP-MS was operated using Argon Gas Dilution (AGD) and tuned daily using the dedicated autotune procedure. All analytes were analysed using a single measurement mode based on kinetic energy discrimination with pure He as the collision gas (He KED). Table 1 summarizes the instrumental parameters.

Table 1. Instrument parameters for the analysis of samples containing 25% NaCl.

Parameter	Value
RF Power (W)	1550
Nebulizer Gas Flow (L/min)	0.25
Dilution Gas Flow (L/min)	0.9
Extraction Lens (V)	-300
CCT Gas Flow (He) (mL/min)	4.2
KED Voltage (V)	3
Skimmer Cone insert	High Matrix
Nebulizer type	V-Groove

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A four point calibration was performed between 0 and 100 µg·L⁻¹, with the exception of Hg, where the calibrated range was between 0 and 4 µg·L⁻¹. The internal standard solution contained Ge, In, Tb, Ir and Bi at a concentration of 20 µg·L⁻¹ in 2% HNO_3. As a model sample matrix, high purity sodium chloride (NaCl) was dissolved in UPW to give a 25% (w/w) solution. Spike recovery for multiple elements was determined at a concentration of 25 µg·L⁻¹ (1 µg·L⁻¹ for Hg) spiked into the sample matrix.

Results

One of the main challenges of analyzing samples containing very high amounts of matrix is signal suppression caused by changing conditions in the plasma. The most common way to identify and correct for such effects is the use of internal standards.

In order to investigate the performance of the iCAP RQ ICP-MS with AGD for such samples, the internal standard response over the runtime of the analysis is shown in Figure 1.
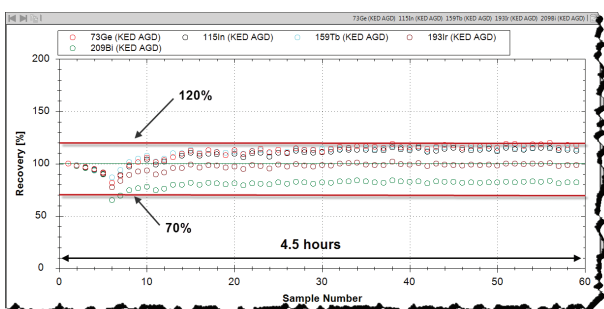

Figure 1. Internal Standard signal response for the standards and more than 50 samples in 4.5 hours.

As can be seen, the signal of the isotopes used for internal standardization do not show major suppression when switching from the initial calibration solutions prepared in 2% HNO_3 to the sample matrix. The internal standard response remains stable over the runtime of the analysis, which covered more than 50 samples in approximately 4.5 hours.

Table 2 shows the results obtained for the analysis of a sample containing 25% NaCl. As expected, the concentration of most elements was below the attainable limit of quantitation since a high purity reagent was used to prepare the brine solution. The table also includes the calculated instrumental detection limits (IDL). Detection limits determined in the sample matrix were generally found to be within the same order of magnitude.

One of the drawbacks of AGD is the decreased ionization efficiency for elements with elevated 1ˢᵗ ionization potential, such as Zn, As, Se and Cd. The use of an Argon humidifier can partially reduce this effect.[1]

In order to address the accuracy of the method, the recovery of a spiked sample was determined several times during the runtime of the LabBook. The average value is again shown in Table 2 together with the RSD value. Spike recoveries between 85 and 116% were observed and show that different trace elements can be accurately quantified at low levels in a challenging sample matrix.

Table 2. Analysis results of 25% (w/w) NaCl samples.

Isotope	Result [µg·L⁻¹]	IDL [µg·L⁻¹]	Recovery [%]	RSD [%]
9Be	<LOQ	0.56	85	5.3
51V	0.75	0.22	116	2.5
52Cr	3.25	0.61	107	2.2
55Mn	<LOQ	0.38	103	3.1
56Fe	23.5	0.42	80	2.5
59Co	<LOQ	0.12	104	2.0
60Ni	<LOQ	0.48	95	2.3
65Cu	<LOQ	0.82	94	2.1
66Zn	<LOQ	0.54	88	3.1
71Ga	<LOQ	0.16	100	2.9
75As	<LOQ	0.25	99	1.9
77Se	<LOQ	0.55	95	5.3
88Sr	83.1	0.09	101	2.2
111Cd	<LOQ	0.05	98	2.4
133Cs	<LOQ	0.05	102	2.4
137Ba	<LOQ	0.14	106	2.6
139La	<LOQ	0.02	104	2.2
140Ce	<LOQ	0.06	102	1.8
202Hg	<LOQ	0.14	110	4.2
208Pb	<LOQ	0.04	94	1.8
232Th	<LOQ	0.01	105	1.8
238U	<LOQ	0.02	105	1.7

Conclusion

The direct analysis of samples containing 25% NaCl is possible using Argon Gas Dilution in combination with a segmented flow sample introduction system and online dilution with the internal standard solution.

Due to the robustness of the iCAP RQ ICP-MS, the response of the internal standards is almost unaffected by the sample matrix, and excellent accuracy was demonstrated through the determination of the spike recovery. Although the detection sensitivity is reduced as a consequence of the use of AGD, the attainable detection limits are mostly well below 1 µg L⁻¹.

Reference

1. Technical Note 43202: Analysis of High Matrix Samples using Argon Gas Dilution with the Thermo Scientific iCAP Q ICP-MS.

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