RADOM™

Reimagine Plasma

MICAP-OES 1000

DETERMINATION OF TRACE ELEMENTS IN UNDILUTED 30% BRINE SOLUTIONS



- Lowest Operating Cost
- Simultaneous Measurement
- Lowest Carbon Footprint
- Smallest Laboratory Footprint

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Introduction

The analysis of brine solutions for elemental composition by ICP is complicated by the high probability of sample introduction failure by clogging of the nebulizer, deposits in the torch injector and/or plasma failure due to matrix overloading. A common first step is sample dilution which solves all the aforementioned problems, but raises the detection limits of the analytes. Given the quest for ever lower levels of detection, dilutions can miss levels deemed important. To overcome these issues, the MICAP-OES 1000 was fitted with a Jet Vortex Interface (21-809-4819), a Semi-Demountable Torch with a 2.4 mm quartz injector (30-808-4306), an Elegra Dual-Channel Argon Humidifier (70-803-1273), a Tracey DC Spray Chamber (20-809-4845) and a Slurry DC Nebulizer(A13-07-US6), all from Glass Expansion. For the Jet Vortex Interface, a programmable Mass Flow Controller (Bronkhorst Mass-View 402) was used for precise control of the additional sheath gas flow to the Jet Vortex Interface accessory.

This application brief outlines the process of preparing and analyzing Calcium (Ca) and Magnesium (Mg) in 30 % NaCl with no dilution at limits of detection (LOD) of < 20 ppb. Also the capabilities of the MICAP across a wider range of analytes is demonstrated.

MICAP-OES

MICAP-OES 1000 is a Microwave Indicatively Coupled Atmospheric Plasma – Optical Emission Spectrometer with 1 kW power. This innovative nitrogen-based plasma source replaces the traditional argon generated plasma technology. This is only possible by incorporating Radom's CERAWAVE™ technology which replaces the metallic water-cooled coil found in commercially available ICP-OES instruments today.



The independent plasma source is coupled to an echelle based, research-grade spectrometer via a fiber optic connection. The plasma is viewed axially with auto-optimized plasma tail removal. The optimized viewing position is ensured with the torch alignment system. The entire echelle spectrum (194nm to 625nm + 766nm) is simultaneously captured with each replicate measurement.

MICAP-OES 1000 is a powerful instrumental technique for metals analysis. This instrumentation provides fast, simultaneous sample measurements eliminating multiple preparation steps. In addition, the analysis is performed using instrumentation with low carbon footprint by eliminating the need for combustible gas, sustained usage of argon gas, and chillers. The benefits are low-cost, ease of use analysis while reducing chemical waste and carbon emissions.

Experimental Design

The Dual-Channel Elegra humidifier to was used to humidify both the Jet Vortex interface gas and the nebulizer gas to help prevent salt deposition in both the injector and the nebulizer tip. The Jet Vortex Interface accessory couples securely to the torch and spray chamber (DC Direct Coupled). This additional gas flow swirls around the aerosol, which delivers a sheath gas that surrounds the aerosol - eliminating salt deposition on the injector walls.

A simulated brine solution was prepared with 30% NaCl w/v by dissolving 15 g of 99.999% sodium chloride (Sigma Aldrich 204439-VAR) in 50 mL Type 1 water and 2% nitric acid. Four standards were prepared at 0, 0.025, 0.1, and 0.5 ppm with the same NaCl and acid concentrations. Yttrium (Y) was used as the internal standard at 10 ppm in all solutions. These MICAP sample introduction components are summarized in Table 1, and displayed installed on the MICAP in Figure 1.

Component	Description
Torch	Semi-demountable Glass Expansion torch with 2.4 mm Quartz Injector
Spray Chamber	Tracey DC
Nebulizer	Slurry DC
Injector Gas	Jet Vortex with MFC
N2 Humidifier	Dual Channel Elegra
Sample Tubing	Black/Black PVC (0.76mm ID)
Drain Tubing	Blue/Yellow PVC (1.52mm ID)

Table 1. MICAP-OES 1000 Sample Introduction Assembly

The Plasma and Measurement Conditions are presented in Figure 2. The total sample measurement time is 2 minutes and 50 seconds. Nitrogen flow to Jet Vortex is 0.15 L/min.

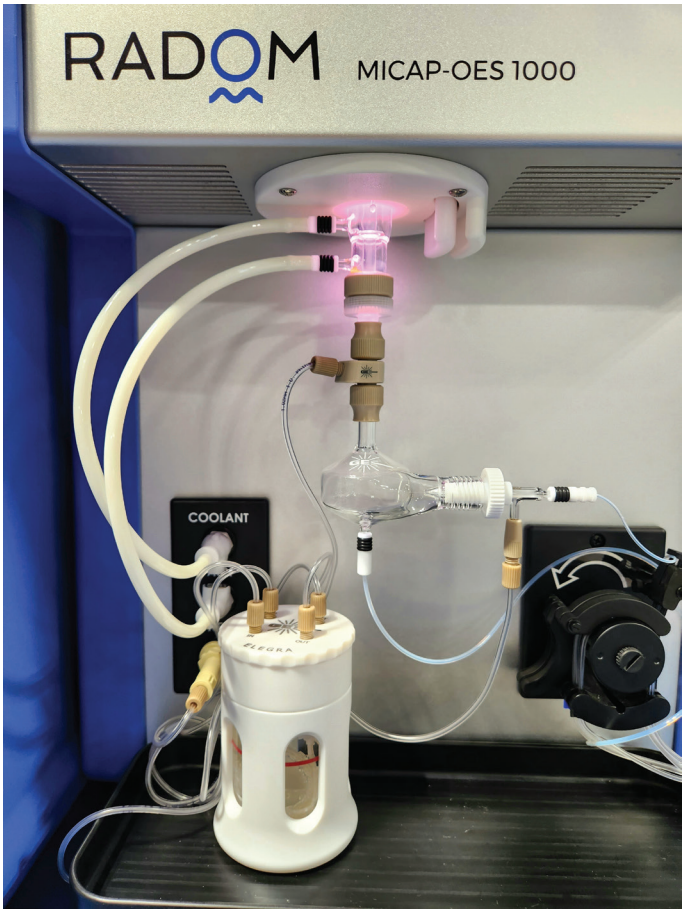


Figure 1. MICAP sample introduction area with Glass Expansion accessories utilized in this brine analysis

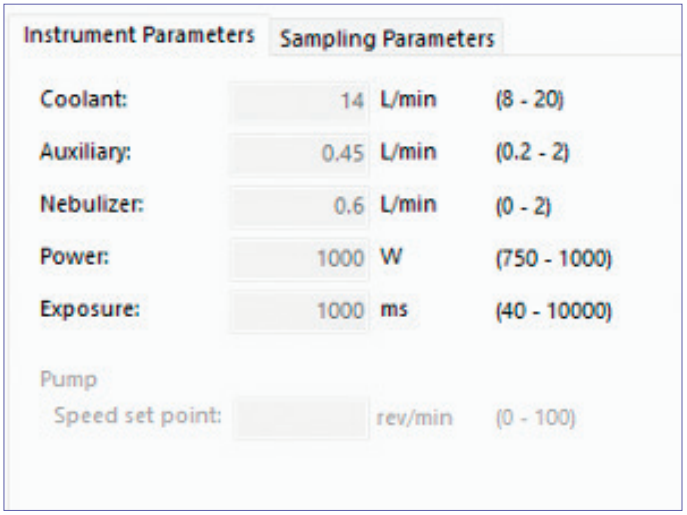


Figure 2. Plasma and Measurement Conditions

Representative calibration curves with figures of merit for each wavelength are presented below. The graphs Included represent the appropriate concentration range per wavelength.

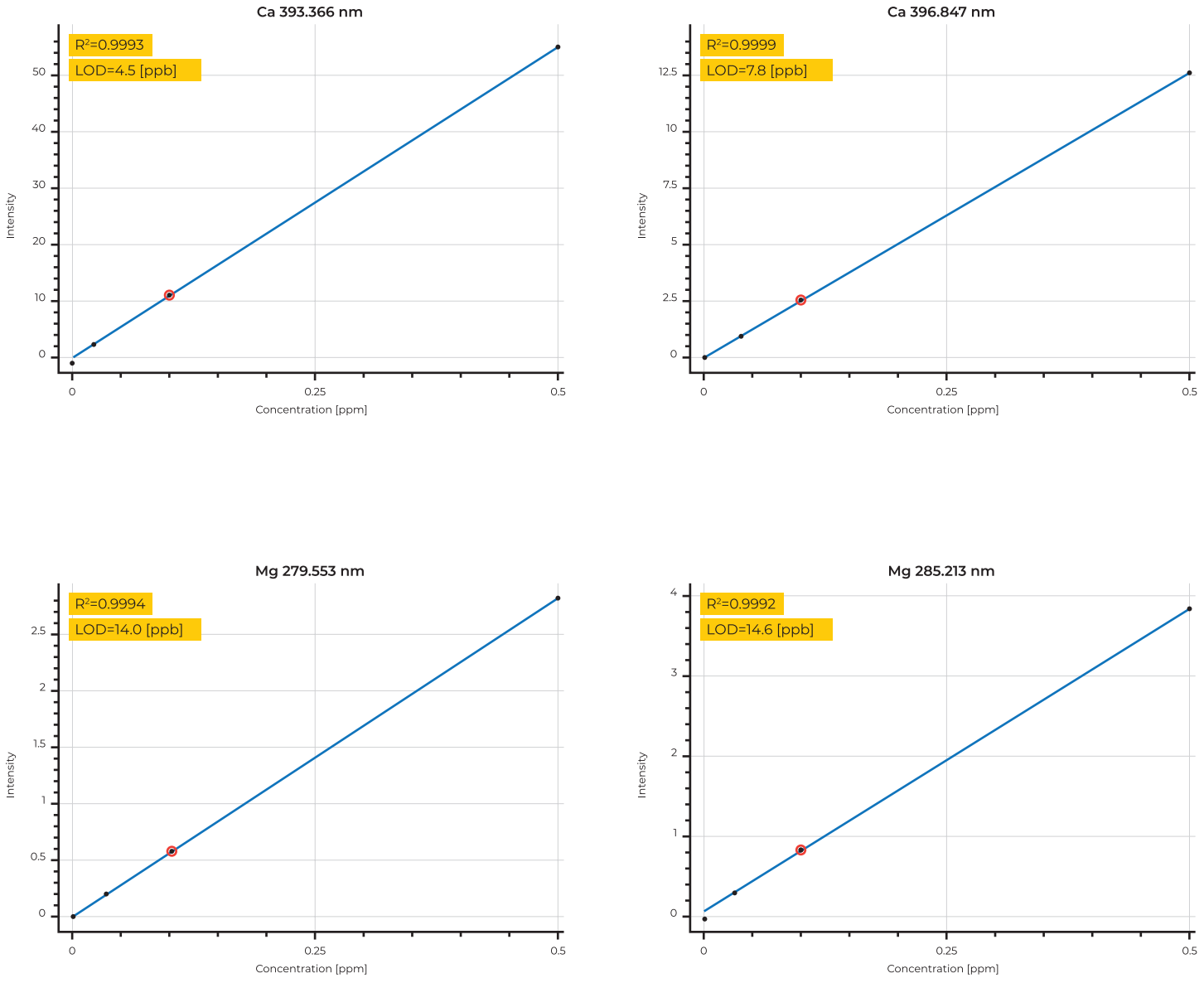


Figure 3. Representative Calibration Curves

The stability of the system while introducing this NaCl brine sample was evaluated by analyzing samples containing 30% NaCl for 4 hours, with each sample analysis separated with a 60 second rinse in 2% nitric acid. Figure 4 displays the excellent stability observed across the 4 hour analysis time.

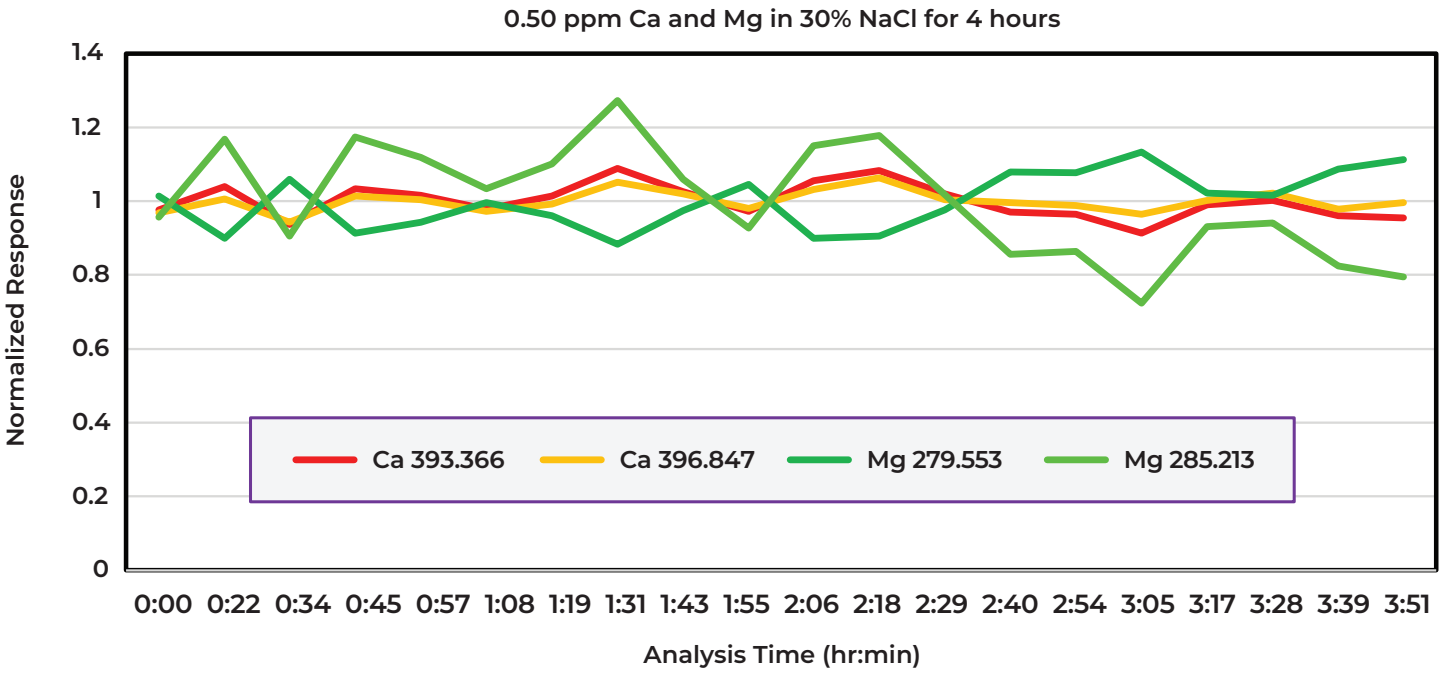


Figure 4. Long Term Stability for Brine Analysis

Result Summary and Discussion

MICAP-OES 1000 is a simultaneous, echelle polychromator with CMOS detector. As a result, multiple wavelengths can be selected with no disadvantage to the required sample volume or time of analysis. Two wavelengths were selected for each target element. The LOD determined for calcium and magnesium were reported from the primary and secondary wavelengths as an example of performance.

Limits of Detection (LOD)					
Analyte	Wavelength (nm)	LOD (ppb)	Analyte	Wavelength (nm)	LOD (ppb)
Ca	393.366	4	Mg	279.553	14
Ca	396.847	8	Mg	285.213	15

Table 2. Ca and Mg Limits of Detection

Additional Elemental Detection Limits

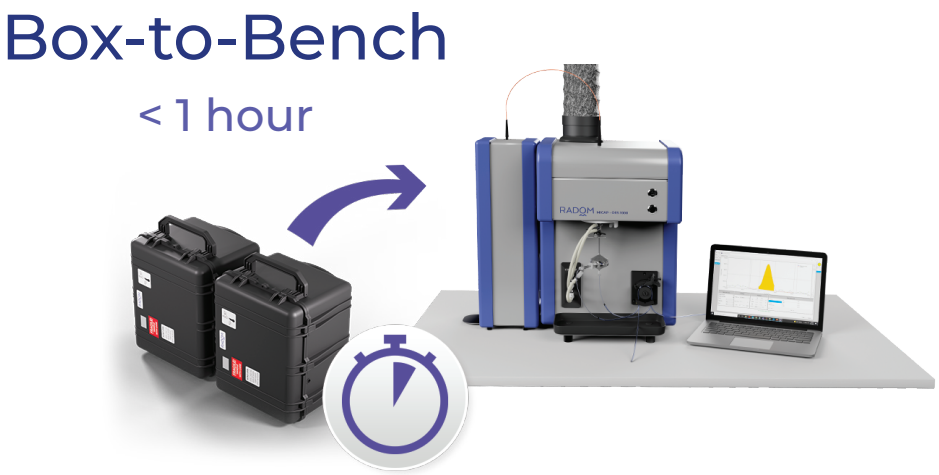
Following the Ca and Mg analysis, testing was performed on a wider range of analytes using the same sample introduction and method parameters. Differences were wavelengths analyzed and Calibration Standard concentrations (0, 0.01, 0.05, 0.25, 1.0, 5.0, 10). The analytes of interest were as follows: Ag, Al, Au, B, Ba, Bi, Cd, Co, Cr, Cu, Fe, Ga, In, Li, Mn, Ni, Pb, Pd, Pt, Sr, Tl, and Zn. As with the Ca and Mg testing, multiple wavelengths were analyzed for mulitple analyte elements. See Table 3. for Limit of Detection (LOD) results.

Limits of Detection (LOD)					
Analyte	Wavelength (nm)	LOD (ppb)	Analyte	Wavelength (nm)	LOD (ppb)
Ag	328.068	19	Fe	259.940	762
Ag	338.289	35	Ga	403.298	22
Al	394.401	47	In	410.175	192
Al	396.152	24	In	451.130	84
Au	242.795	215	Li	460.289	2628
B	249.677	331	Mn	257.610	161
B	249.772	418	Mn	259.372	288
Ba	455.403	2	Ni	341.476	48
Ba	493.408	5	Pb	405.781	238
Bi	223.061	398	Pd	363.469	37
Cd	228.802	154	Pt	299.796	442
Co	345.350	78	Pt	306.471	667
Cr	425.435	33	Sr	407.771	1
Cr	428.973	60	Sr	421.552	6
Cu	324.754	8	Tl	377.572	379
Cu	327.396	22	Zn	213.857	57

Table 3. Additional Limits of Detection

Conclusions

MICAP-OES 1000 robust nitrogen plasma is customer installable with its “Box to Bench” design. The addition of the humidified gas flows and the sheath gas accessory allowed the MICAP to demonstrate excellent performance and long term stability in this saturated brine sample matrix. The optimized method for the determination of Ag, Al, Au, B, Ba, Bi, Ca, Cd, Co, Cr, Cu, Fe, Ga, In, Li, Mg, Mn, Ni, Pb, Pd, Pt, Sr, Tl, and Zn in 30% NaCl has already been created. The only component missing is you.



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