Application Note

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ICP-OES Quality Control of Elements in Brines Produced by Direct Lithium Extraction (DLE)

Automated analysis of lithium brines using an Agilent ICP-OES Automation System

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Introduction

Direct Lithium Extraction (DLE) technology is a selective process designed to recover lithium (Li) from underground raw Li brine deposits while removing impurities.[1] Compared to conventional Li extraction methods, such as mining hard rock ores or using brine evaporative methods, DLE is faster, consumes less water and power, produces less waste, and requires less land. Given these process and sustainability advantages, DLE is gaining significant attention from industry, academia, investors, and environmentalists.

After multiple steps, DLE produces a high-purity Li concentrate brine that can be used to produce commercial battery-grade Li salts for use in the Lithium Ion Battery (LIB) sector. LIBs play a key role in transitioning away from fossil fuel dependency in transport and electricity production by powering electric vehicles and storing electricity generated from renewable energy sources.

Developers and users of DLE techniques require multi-elemental analytical techniques capable of assessing the profile of brines at the various stages of the process. These quality control (QC) measurements require thorough major, minor, and trace elemental composition analysis to evaluate extraction efficiency, as well as the purity of the final concentrates. ICP-OES is widely used for the simultaneous measurement of multiple elements in many types of LIB-related samples, including Li brines, and is a specified technique in various LIB-related standard methods.[2,3]

However, Li brines produced by DLE present specific analytical challenges for ICP-OES due to high levels of total dissolved solids (TDS), the high density of the solutions, and the varying concentration of analytes in the samples. Other challenges include ionization interferences arising from easily ionized elements (EIEs), sample variety throughout the DLE process, carryover and contamination between samples, and sample throughput.

Agilent ICP-OES Automation System

Robust instrumentation is therefore needed for the routine elemental analysis of large numbers of brine samples. An Agilent ICP-OES Automation System comprising the Agilent 5900 SVDV ICP-OES, Advanced Valve System (AVS 7), Advanced Dilution System (ADS 2), SPS 4 autosampler, and instrument software was used in this study. The Agilent 5800 VDV ICP-OES is also suitable for this application.

The 5900 ICP-OES uses a vertical plasma torch and a solid-state radio frequency (SSRF) generator operating at 27 MHz to create a plasma with the stability needed for the routine analysis of brines. The integrated AVS 7 switching valve ensures fast analysis times and reduces instrument exposure to solids in high TDS samples.[3] The accessory also minimizes carryover from sodium (Na) and prevents excessive torch devitrification, greatly reducing the frequency of instrument cleaning and extending the lifespan of parts.

The samples analyzed in this study were produced using a membrane-based DLE methodology and included a wide variety of sample matrices, with target analytes typically present across a broad range of concentrations. Some key analytes, such as Li, Na, and magnesium (Mg), are particularly important to quantify as they directly impact the efficiency of the DLE process.

To account for the different compositions and densities of the DLE samples, the ADS 2 was used to prepare calibration standards across a wide analytical range and to dilute the samples before analysis.[4] Both processes can be labor-intensive and time-consuming when performed by an analyst, as well as being potential sources of contamination and errors. The implementation of advanced autodilution technology for ICP-OES enhances sample turnaround time and minimizes the risk of human errors, while ensuring accurate results.

An internal standard (IS) mixture was used to correct for physical matrix interferences and non-spectral interferences, such as EIE effects, caused by the large amount of sodium chloride (NaCl) in the brine samples. Radial view mode also reduces EIE interferences so was used for the analysis of the brines.

Twelve key elements were determined in three types of real-world Li brine samples obtained from a commercial DLE process: a raw brine, a tail (Li-free) brine, and a Li concentrate brine. The elements included boron (B), barium (Ba), calcium (Ca), iron (Fe), potassium (K), Li, Mg, manganese (Mn), Na, sulfur (S), silicon (Si), and strontium (Sr).

The brine samples were auto diluted by the ADS 2 using prescriptive and reactive dilution functions and each brine presented a distinctive elemental profile. Also, 306 solutions were analyzed over seven hours to demonstrate the robustness and stability of the method. All samples were analyzed in the same run, demonstrating the suitability of the method for routine QC measurements at the various stages of the DLE process.

Experimental

Instrumentation

All measurements were performed using an Agilent 5900 SVDV ICP-OES configured with an AVS 7 switching valve, ADS 2 autodilutor, and SPS 4 autosampler (Figure 1). Combined with Agilent ICP Expert Pro software instrument software, these components represent the Agilent ICP-OES Automation System. Instrument operating parameters are listed in Tables 1 to 3.

The 5900 ICP-OES was fitted with a SeaSpray nebulizer, double-pass cyclonic spray chamber, and Agilent semi-demountable VDV torch with a 1.8 mm internal diameter (id) injector.

An internal standard (IS) solution comprising 5 mg/L scandium (Sc), 100 mg/L rubidium (Rb), and 40 mg/L tellurium (Te) was prepared in 2% HNO_3 using Agilent single element standard solutions. The 7-port AVS system enables the IS solution to be directly plumbed to the valve.

Figure 1. The Agilent ICP-OES Automation System: Agilent 5900 SVDV ICP-OES with integrated AVS 7 switching valve (left), Agilent Advanced Dilution System ADS 2 (middle), and Agilent SPS 4 autosampler (right).

Table 1. Agilent 5900 SVDV ICP-OES instrument and method parameters.

Parameter	Setting
Viewing Mode	Radial
Viewing Height (mm)	11
RF Power (kW)	1.4
Nebulizer Flow (L/min)	0.9
Plasma Flow (L/min)	12
Aux Flow (L/min)	1
Replicates	3
Rinse Time (s)	25
Read Time (s)	5
Stabilization Time (s)	20
Sample Pump Tubing	White/white
Internal Standard Pump Tubing	Orange/green
Waste Pump Tubing	Blue/blue

Table 2. Agilent AVS 7 switching valve system parameters.

Parameter	Setting
Sample Loop Size (mL)	1
Pump Rate – Uptake (mL/min)	34.3
Pump Rate – Inject (mL/min)	9.9
Valve Uptake Delay (s)	9.2
Bubble Injection Time (s)	1.8
Preemptive Rinse Time (s)	1.2

Table 3. Agilent ADS 2 autodilution system parameters.

Parameter	Setting
Dilution Loop Size (mL)	1
Carrier Syringe Volume (mL)	5
Diluent Syringe Volume (mL)	10
Reactive Dilution Rinse Time (s)	25

Sample preparation

Three types of real brine solutions, obtained during the various stages of a commercial nanotechnology-based DLE process, were supplied for analysis. As shown in Figure 2, the samples included a raw brine, Li concentrate brine, and a tail (Li-free) brine. The samples were acidified to a final concentration of 2% HNO_3 before analysis.

Figure 2. Three types of DLE Li brine samples analyzed in this study.

Automated preparation of calibration standards by ADS 2

All calibration curves were prepared from the following Agilent stock solutions using the autocalibration feature of the integrated ADS 2 autodilutor:

- Agilent 1000 ppm single element stock solutions for Li and S
- Agilent 1% single element stock solutions for Ca and Na
- Agilent multi-element QC27 standard

QC solutions

For QC purposes, a blank solution of 2% HNO_3 was used as the continuing calibration blank (CCB). Three continuing calibration verification solutions (CCV-1, CCV-2, and CCV-3) were prepared from Agilent 1000 and 10,000 single element stock standard solutions.

The CCV-1 contained B, Ba, Fe, Mn, Si, and S at 10 ppm; the CCV-2 contained K, Li, Mg, and Sr at 100 ppm; and the CCV-3 contained Ca and Na at 500 ppm.

Method development

IntelliQuant Screening

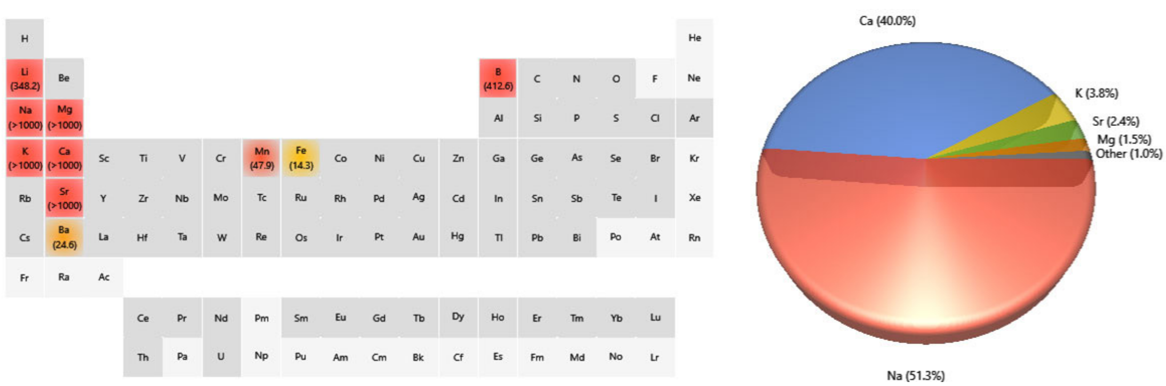
As part of the ICP Expert Pro software, IntelliQuant Screening collects full-spectrum data of a sample, requiring only a few seconds analysis time and with little input by the analyst.[5] The IntelliQuant algorithm then processes the full-spectrum data against premeasured calibrations, generating a semiquantitative reading for every element present in the sample.[6]

IntelliQuant Screening was used in this study during method development to determine the approximate concentration of elements in the three types of brine samples. This information was used to determine the calibration range, dilution factors, and the best emission lines for the quantitative analysis. All samples were analyzed using a single quantitative method.

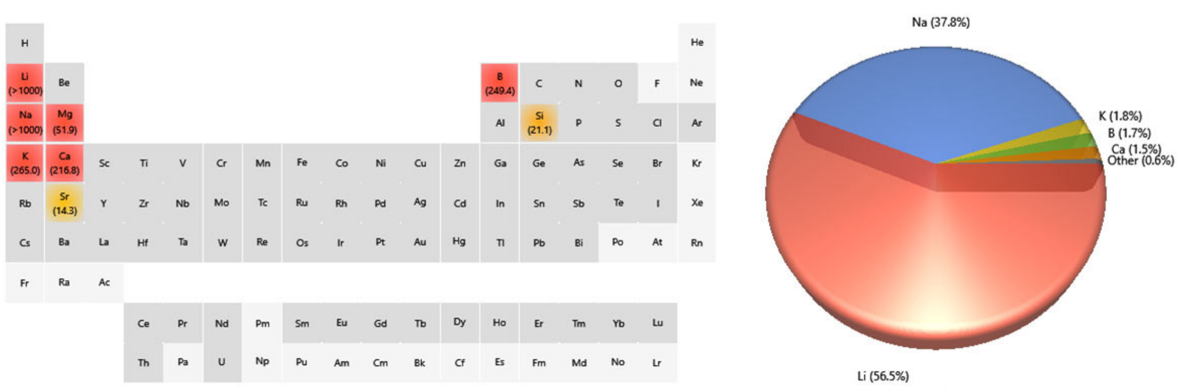
The software generates a periodic table heat map to visually represent the relative concentration of elements present in a sample. Elements present at low concentrations are shown in yellow, medium concentrations are shown in orange, and high concentrations are shown in red.

The IntelliQuant heat map and pie graph displays of the data obtained for the three brine samples (Figure 3) show the relative concentrations of elements in each sample type. Figure 4 also shows the easy-to-interpret star ranking system for selecting the most suitable analyte wavelengths for the quantitative method, using Li as an example. Li 670.783 received a five star rating, suggesting that the wavelength is free of spectral interferences and background shifts and therefore the best choice for the quantitative method.

(A) Raw brine



(B) Li concentrate brine



(C) Tail brine/Li-free brine

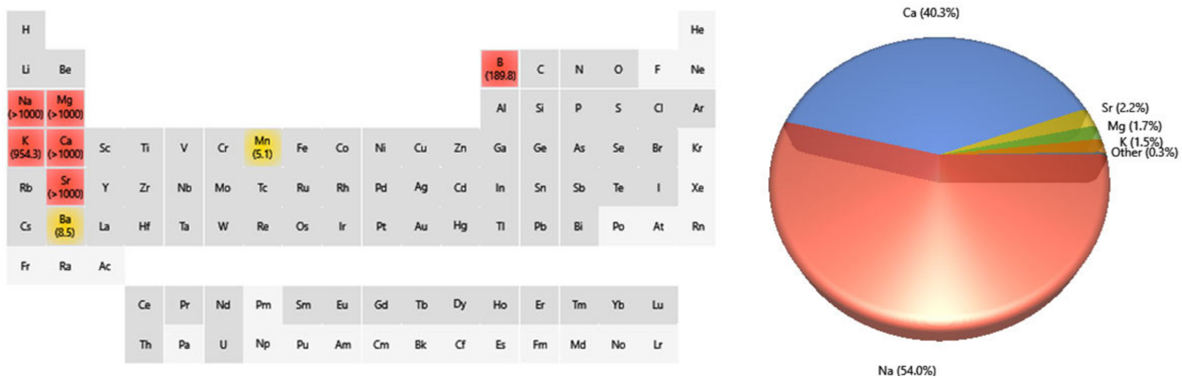


Figure 3. IntelliQuant periodic table heatmaps and pie charts of (A) raw brine, (B) Li concentrate brine, and (C) tail brine, displaying the distinctive elemental profile of each sample. (B) IntelliQuant star ranking information for Li wavelengths in the Li concentrate brine sample. The information helps with selection of the best analyte wavelengths to use in the quantitative method for the analysis of brines.

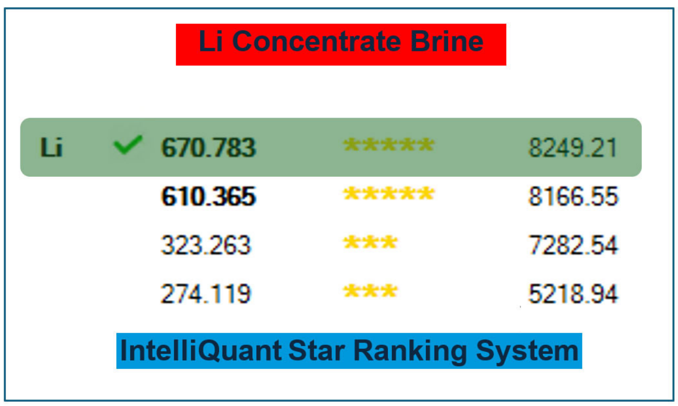


Figure 4. Star rating system for Li showing 670.783 recieved a five star rating.

Autocalibration and linearity

Details of the calibration standard range for each element are given in Table 4. Each calibration point was created by auto diluting the stock standard solutions at multiple factors using the ADS 2.

All calibration curves were linear over the range, as indicated by correlation coefficients between 0.99993 and 1.0000 (Table 4). Representative calibration curves for Ca and Li are shown in Figure 5.

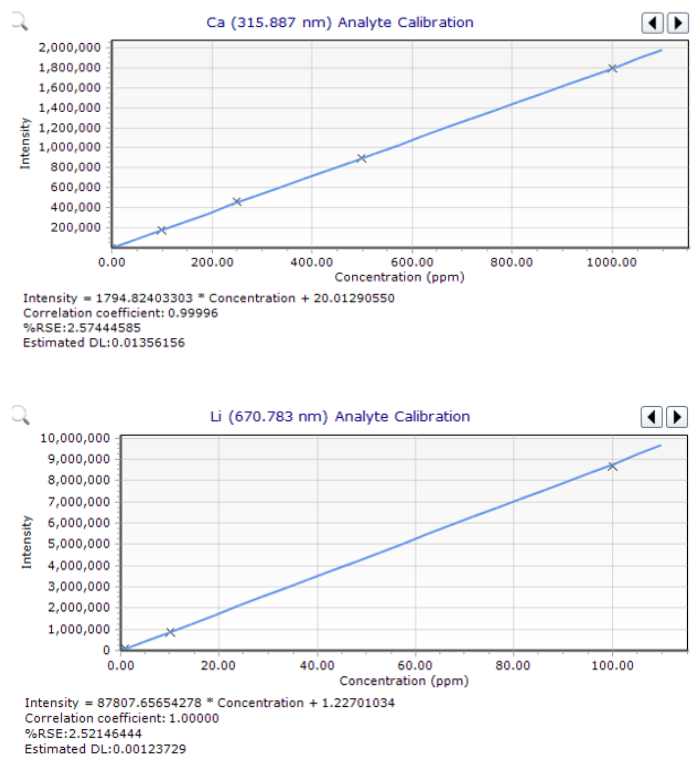


Figure 5. Representative linear calibration curves for Ca and Li, both with a correlation coefficient greater than 0.9999 and a Relative Standard Error (%RSE) <2.6%.

Table 4. Concentration of calibration standards mg/L, background correction, the calibration correlation coefficients, and the internal standard used for each element.

Element and Wavelength (nm)	Background Correction	Calibration Range (ppm)	Correlation Coefficient	Internal Standard
B 249.678	Fitted	0.10–10	1.00000	Te 214.282
Ba 493.408	Fitted	0.10–10	1.00000	Sc 361.383
Ca 315.887	Fitted	100–1000	0.99996	Sc 361.383
Fe 259.940	Fitted	0.10–10	1.00000	Sc 255.235
K 766.491	Fitted	1–100	1.00000	Rb 780.026
Li 670.783	FACT	1–100	1.00000	Rb 780.026
Mg 279.800	Fitted	1–100	1.00000	Sc 255.235
Mn 257.610	Fitted	0.1–10	1.00000	Sc 255.235
Na 330.237	Fitted	100–500	0.99993	Rb 780.026
S 181.972	Fitted	0.5–5	0.99999	Te 214.282
Si 251.611	Fitted	0.1–1	1.00000	Te 214.282
Sr 216.596	Fitted	1–100	1.00000	Sc 255.235

Prescriptive and reactive autodilution

Dilution helps to minimize viscosity and matrix effects when analyzing samples such as brines that contain high levels of TDSs by ICP-OES. The prescriptive dilution feature of the ADS 2 is therefore useful for samples that require a routine dilution step. The autodilutor can perform precise, prescriptive dilutions by specifying a dilution factor up to 400x in the ICP Expert software, effectively removing a time-consuming manual dilution step from the sample preparation procedure.

The ADS 2 can also dilute solutions in response to the sample measurement using a process known as reactive dilution. Where a sample measurement exceeds the maximum calibration range or an IS exceeds the recovery range, the ICP Expert software automatically calculates the appropriate dilution factor needed to bring the out-of-range analytes within user-identified limits.

Results and discussion

Method detection limits

Na was quantified in all brine samples. The highest Na concentration in the raw brine was estimated to be equivalent to 18% NaCl concentration. Therefore, a 1% NaCl solution is representative of the brine sample matrix following a ~20-fold dilution in 2% HNO_3.

Method detection limits (MDLs) were determined by ten measurements of the 1% NaCl sample, spiked with the analytes at 20 ppb. The MDLs were calculated as three times the standard deviation of the concentration determined for each element, multiplied by the dilution factor (Table 5).

For many of the elements listed in Table 5, the wavelengths used were not the most sensitive lines, but they were identified by IntelliQuant as being interference-free within the brine sample matrices.

Table 5. Method detection limits for elements in a synthetic 1% NaCl solution spiked at 20 ppb.

Element and Wavelength (nm)	MDL (mg/L)
B 249.678	0.043
Ba 493.408	0.004
Ca 315.887	0.248
Fe 259.94	0.047
K 766.491	1.39
Li 670.783	0.164
Mg 279.800	0.208
Mn 257.61	0.008
S 181.972	0.166
Si 251.611	0.127
Sr 216.596	0.055

Quantitative analysis

The three real brine samples obtained from the DLE process were analyzed using the 5900 ICP-OES with ADS 2. Given the high TDS content of the samples, they were all auto diluted using the prescriptive dilution function of the ADS.

Using the Li concentrate brine sample as an example, Figure 6 shows that the sample was diluted using both the prescriptive and reactive dilution functions of the ADS 2. First, the sample was automatically diluted 50x by the ADS 2. Since the measured concentration for the Li 670.783 line of 192 ppm was above the upper calibration point for Li of 100 ppm (indicated by 'o'), the ADS 2 performed a reactive dilution of 200x to bring it within range. Both dilution steps were conducted automatically, without any input from the analyst.

The results tables in Figure 6 (unadjusted (top) and adjusted for the dilution factor (bottom)) provide a clear overview of the data for Li. The results 'summary' report selects the best measurement result for each element from the available iterations, without overwriting any existing data.

The quantitative data reported in Table 6, acquired in a single analysis using the 5900 ICP-OES, presents a distinctive elemental profile for each brine sample.

Table 6. Quantitative results for elements measured in each of the three types of brine samples analyzed by the Agilent 5900 SVDV ICP-OES. The data has been adjusted for the dilution factor.

Element and Wavelength (nm)	Brine Sample Concentration (mg/L)		
	Raw Brine	Li Concentrate Brine	Li Free Brine
B 249.678	333	200	151
Ba 493.408	30.4	0.0290	10.5
Ca 315.887	39013	202	28369
Fe 259.94	15.7	<MDL	<MDL
K 766.491	6071	515	1776
Li 670.783	392	10626	8.83
Mg 279.800	2976	114	2267
Mn 257.610	52.5	0.105	5.70
Na 330.237	70505	8407	54616
S 181.972	58.4	3.02	90.3
Si 251.611	14.4	35.3	3.21
Sr 216.596	2525	17.6	1643

Unadjusted ▾ | Track Analysis | < MDL Flagging | Sort Results... ▾ | Hide Columns... ▾ | Column Properties... | Delete Results

☐	Rack:Tube	Solution Label	Timestamp	Li 460.289 nm ppm	Li 610.365 nm ppm	Li 670.783 nm ppm	Rb-I 780.026 nm Ratio	Sc 255.235 nm Ratio	Sc 361.383 nm Ratio	Te-I 214.282 nm Ratio
☐	▾ 1:15	Li Concentrate Brine	8/23/2024 5:34:25 PM	53.23	53.30	53.13	--	--	--	--
	☑ 1:15	*Summary*	*8/23/2024 5:34:25 PM*	*53.23*	*53.30*	*53.13*	--	--	--	--
	Prescriptive	*Original - 50*	*8/23/2024 5:32:03 PM*	*200.12 o*	*198.24 o*	*192.03 o*	*1.00*	*0.97*	*0.97*	*0.96*
	Reactive	*Dilution - 200*	*8/23/2024 5:34:25 PM*	*53.23*	*53.30*	*53.13*	*1.01*	*0.99*	*0.99*	*0.99*

Concentration ▾ | Track Analysis | < MDL Flagging | Sort Results... ▾ | Hide Columns... ▾ | Column Properties... | Delete Results

☐	Rack:Tube	Solution Label	Timestamp	Li 460.289 nm ppm	Li 610.365 nm ppm	Li 670.783 nm ppm	Rb-I 780.026 nm Ratio	Sc 255.235 nm Ratio	Sc 361.383 nm Ratio	Te-I 214.282 nm Ratio
☐	▾ 1:15	Li Concentrate Brine	8/23/2024 5:34:25 PM	10646.51	10660.21	10625.84	--	--	--	--
	☑ 1:15	*Summary*	*8/23/2024 5:34:25 PM*	*10646.51*	*10660.21*	*10625.84*	--	--	--	--
	Prescriptive	*Original - 50*	*8/23/2024 5:32:03 PM*	*10006.21 o*	*9911.89 o*	*9601.47 o*	*1.00*	*0.97*	*0.97*	*0.96*
	Reactive	*Dilution - 200*	*8/23/2024 5:34:25 PM*	*10646.51*	*10660.21*	*10625.84*	*1.01*	*0.99*	*0.99*	*0.99*

Figure 6. An example of the Agilent ADS 2 performing a prescriptive dilution of 50x of the Li concentrate brine sample and a secondary reactive dilution to ensure that the measured Li concentration was within the calibration range. Top: Unadjusted concentration data for Li. Bottom: Data corrected for the dilution factor.

It can be assumed that Ca, K, Li, Mg, and Na are present as chloride salts ($CaCl_2$, KCl, LiCl, $MgCl_2$, and NaCl) in the raw brine sample. As shown in Table 7, these concentrations equate to a TDS content of more than 31%, before dilution, making this sample the most challenging of the set. NaCl was present at ~18%, followed by $CaCl_2$ at ~11%. Despite the variation in TDS, all these brines were autodiluted and analyzed in the same run in a simple workflow using the ICP-OES Automation System.

Table 7. Calculation of percentage of TDS in the raw brine sample.

Element	Raw Brine Sample		
	ppm	Salt	%
Ca	39013	$CaCl_2$	10.8
K	6071	KCl	1.2
Li	391.8	LiCl	0.2
Mg	2976	$MgCl_2$	1.2
Na	70505	NaCl	17.9
		Total	31.3

Spike recovery test

To further evaluate the 5900 SVDV ICP-OES method, a spike recovery test was conducted on the raw Li brine sample. The sample was diluted 250x and spiked at 1 ppm for B and S; 10 ppm for K, Li, Mg; and 100 ppm for Ca and Na.

As shown in Table 8, all recoveries were within 100 ±5% of the expected values, demonstrating the accuracy of the method for the determination of the target elements in brine. All spike information is displayed before the dilution factor has been applied.

Table 8. Spike recovery data for the raw Li brine sample diluted 250x.

Element and Wavelength (nm)	Conc in Li Brine Solution (ppm)	Li Brine + Spike (ppm)	Recovery (%)
B 249.678	1.32	2.33	101
Ba 493.408	0.124	1.11	98.8
Ca 315.887	156	256	100
Fe 259.94	0.0650	1.02	95.3
K 766.491	24.3	34	97.8
Li 670.783	1.6	11.7	101
Mg 279.800	11.8	21.8	100
Mn 257.610	0.216	1.20	98.7
Na 330.237	288	386	98.1
S 181.972	0.229	1.26	103
Si 251.611	0.050	1.089	104
Sr 216.596	10.2	20.2	100

Stability test

To demonstrate the robustness of the ICP-OES Automation System, 306 solution measurements were taken over seven hours without recalibration. The solutions consisted of a raw brine diluted 150x and a QC block that comprised the CCB and the three CCV solutions. The QC block was measured after every 10 measurements of the raw brine samples. The ADS 2 performed a prescriptive 10x dilution on the CCV-1 and CCV-2 solutions, resulting in final concentrations of 1 ppm for B, Ba, Fe, Mn, Si, and S, and 10 ppm for K, Li, Mg, and Sr. The CCV-3 consisted of 500 ppm for Ca and Na.

The plot of the CCV recoveries shows the stability of all elements to be within 100 ±5% over the entire analytical run that included autodilution before every measurement (Figure 7). The recovery data and precision (%RSDs of less than 2%) of the measurements demonstrate the excellent robustness of the 5900 SVDV ICP-OES and ADS 2 method for the routine analysis of Li brines over seven hours.

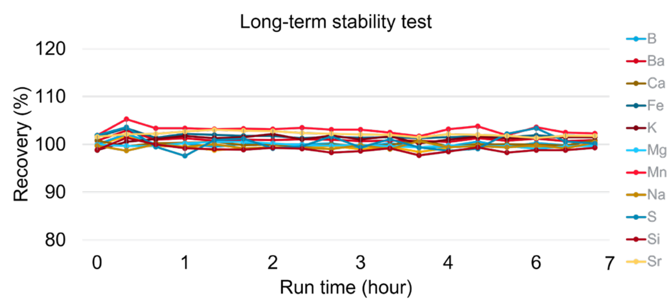


Figure 7. Percent recovery of QC solutions analyzed over seven hours by the Agilent ICP-OES Automation System.

Table 9. Long-term stability of the Agilent 5900 ICP-OES during the analysis of the raw Li brine over seven hours. %RSD of measurements.

Element and Wavelength (nm)	RSD %
B 249.678	0.57
Ba 493.408	0.58
Ca 315.887	0.57
Fe 259.94	0.97
K 766.491	0.80
Li 670.783	0.67
Mg 279.800	0.59
Mn 257.61	0.60
Na 330.237	0.63
S 181.972	1.93
Si 251.611	3.06
Sr 216.596	0.66

The precision of the repeated concentration measurements of the 12 analytes in the raw brine samples was excellent over seven hours, with %RSDs below 3.1% (Table 9).

Also, during the stability test, the IS recovery plots acquired for all samples were within 100 ±6%, with no IS failures throughout the run, as shown in Figure 8. The results demonstrate the robustness of the plasma and the tolerance of the ICP-OES Automation System for handling high matrix samples.

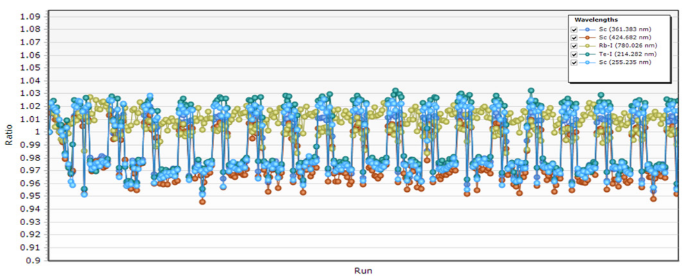


Figure 8. Stability of IS measurements over seven hours. The IS recoveries have been normalized to the calibration blank for all samples.

Conclusion

An Agilent ICP-OES Automation System comprising the Agilent 5900 SVDV ICP-OES, AVS 7, ADS 2, SPS 4 autosampler, and ICP Expert Pro instrument software was used to analyze 12 elements in real brine samples. The raw, tail (Li-free), and Li concentrate brine samples were sourced from before and after processing using a membrane-based Direct Lithium Extraction (DLE) technique. All samples were analyzed using a single method in a single run.

The IntelliQuant Screening method development tool within the software was used to set the calibration range for the analytes and to select the best analyte wavelengths.

The use of the ICP-OES Automation System provided productivity and performance benefits for the brine application including:

- Automatic calibration. Completely automated calibration from three stock solutions removed the need to manually prepare standards to cover the wide and varying concentration range of analytes in the brines.
- Prescriptive dilution. The ADS 2 was used to automatically dilute all three of the Li brine samples 50 times, so that the samples could be measured in a single run.
- Reactive dilution. The ADS 2 automatically diluted the Li concentrate brine sample 200x in response to the concentration of Li exceeding the calibration range before remeasurement.
- The ADS 2 also performed reactive dilutions on the raw brine (150x) and the tail brine (250x) to quantify Na.
- Results summary report. The software selected the best measurement results to display from the available reactive dilution remeasurement results. It allowed for straightforward data viewing and reduced the amount of time required for manual data processing.
- The AVS 7 minimized the exposure of the torch, nebulizer, and pump tubes to the high matrix samples. It also worked seamlessly with the ADS 2 to improve sample turnaround times.

The matrix tolerance, robustness, and accuracy of the method was demonstrated by the excellent recovery data of 100 ±5% for all elements spiked in the raw brine sample, and by the long-term stability data.

As DLE technologies continue to develop, the sector can rely on the ICP-OES Automation System for the efficient and accurate analysis of complex brine matrices, paving the way for more effective Li extraction processes.

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Agilent part numbers

Part number	Description
G8020-68005	Easy-fit fully demountable torch with 1.8 mm quartz injector for Agilent 5000 series ICP-OES
G8010-60256	Double-pass spray chamber, glass cyclonic design with ball joint socket and UniFit drain outlet, for Agilent 5000 series ICP-OES
G8010-60255	SeaSpray concentric glass nebulizer for Agilent 5000 series ICP-OES
G8010-60346	Nebulizer gas humidifier for 5000 series ICP-OES
3710034400	Peristaltic pump tubing, PVC, white/white, 12/pk
3710068300	Peristaltic pump tubing, PVC, orange/green, 12/pk
3710034600	Peristaltic pump tubing, PVC, blue/blue, 12/pk
5190-9418	Quality Control Standard 27, multi-element calibration standard solution for 27 elements at 100 ppm
5190-8529	Sulfur (S) standard, 1,000 µg/mL, in H_2O, 100 mL
5190-8477	Lithium (Li) standard, 1,000 µg/mL, in 5% HNO_3, 100 mL
5190-8368	Calcium (Ca) standard, 10,000 µg/mL, in 5% HNO_3, 100 mL
5190-8454	Sodium (Na) standard, 10,000 µg/mL, in 5% HNO_3, 100 mL

www.agilent.com/chem/5900icpoes

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Published in the USA, January 20, 2025
5994-8041EN

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