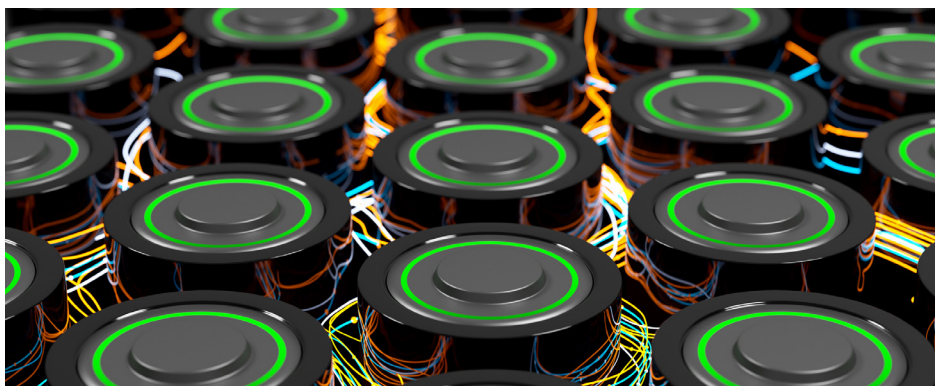**Agilent**

Trusted Answers

Determination of Elemental Impurities in Lithium Carbonate Using ICP-OES

Quality control of chemicals used in lithium ion battery components by Agilent 5800 VDV ICP-OES

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Introduction

Lithium carbonate (Li_2CO_3) is an important raw material for the lithium-ion battery (LIB) manufacturing industry. It is the precursor for many Li-containing components, such as lithium cobalt oxide and lithium iron phosphate used in cathodes, and lithium hexafluorophosphate used in electrolytes. The expanding market for LIBs is increasing demand for Li compounds, leading to a projected global market value for Li_2CO_3 of \$3.7 billion by 2030 (*1*).

LIB manufacturers require high-quality raw materials, since the purity of all battery components is critical to the performance, safety, and lifespan of the end products. So, an accurate and reliable analytical method is needed for the quality control (QC) of elemental impurities in chemicals and materials used to make LIBs.

ICP-OES is a robust, multi-elemental analytical technique that is often recommended for the elemental analysis of Li-rich compounds in industry standard methods (2). As the global leader of LIB production, China has implemented an industrial standard for battery grade Li_2CO_3, YS/T 582-2013 (3). For chemical analysis of Li_2CO_3, YS/T 582-2013 refers to the national standard GB/T 11064-2013. Part 16 of GB/T 11064-2013 relates to 'the determination of the amounts of Ca, Mg, Cu, Pb, Zn, Ni, Mn, Cd, and Al using ICP-OES' (4). An ISO method ISO/WD 11757 for the determination of Al, B, Ca, Co, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, S, and Zn in Li_2CO_3 by ICP-OES is under development (5).

The challenges of analyzing high purity Li-rich compounds using ICP-OES include the potential for high maintenance needs and compromise on sensitivity and accuracy due to the high Li concentration in the sample matrices. The high Li content can affect the analysis of easily ionized elements (EIEs) such as Na and K, leading to false-positive results (6).

To meet the challenges of the application, the advanced Agilent 5800 Vertical Dual View (VDV) ICP-OES equipped with an Agilent AVS seven-port switching valve (AVS 7) was used. The AVS reduces the amount of sample that reaches the sample introduction system, reducing sample load on the nebulizer and torch. Compared to the AVS 6, the 7 port system enables the internal standard to be directly plumbed to the valve. Also, the 5800 ICP-OES features a cooled cone interface (CCI) that improves the reliability of the results when measuring EIEs by reducing self-absorption and the formation of recombination and matrix interferences present in the LIB samples. The 5800 and Agilent ICP Expert software include smart tools to help with method development and to monitor instrument performance metrics (7−9):

– IntelliQuant uses data analytics to automatically identify spectral overlaps that can lead to false-positive results and recommends the emission wavelength that will give the most accurate result.

– Easy-to-use automated algorithms for ICP-OES background correction including Fitted Background Correction (FBC) and Fast Automated Curve-fitting Technique (FACT).

– Early Maintenance Feedback (EMF) prompts analysts to perform maintenance based on instrument usage rather than simple time-based routines.

In this study, the 5800 VDV ICP-OES with AVS 7 was used to measure 27 elements in a 99% pure Li_2CO_3 sample. The method was evaluated through sample spiking and long-term stability testing.

Experimental

Materials and standard solutions

A commercial Li_2CO_3 product with 99% purity and a commercial lithium hydroxide (LiOH) product with 98% purity were used to represent the LIB raw materials. LiOH was used for the long-term stability test only. High purity concentrated nitric acid (HNO_3) (69%) was bought from Merck Pty. Ltd, Australia. The calibration standards were prepared using Agilent 1,000 or 10,000 mg/L single element standard solutions of Al, As, B, Ba, Be Ca, Cd, Co, Cr, Cu, Fe, Ga, K, Li, Mg, Mn, Mo, Na, Ni, Pb, S, Sb, Si, Sr, Ti, V, Zn, and Zr. Internal standard (IS) solutions were prepared using Agilent 1,000 or 10,000 mg/L single element standard solutions of Bi, In, and Rb.

Calibration standards and internal standards

A 10 mg/L stock solution containing Al, As, B, Ba, Be Ca, Cd, Co, Cr, Cu, Fe, Ga, K, Mg, Mn, Mo, Na, Ni, Pb, S, Sb, Si, Sr, Ti, V, Zn, and Zr was prepared by diluting each of the 1,000 or 10,000 mg/L single element standard solutions in 5% HNO_3. To prepare calibration standard solutions of all elements, except for Na, the stock solution was diluted using 10% HNO_3 to the final concentrations of 0, 0.010, 0.050, 0.100, 0.200, and 0.500 mg/L. For Na, calibration standards were prepared at 0, 0.020, 0.050, 0.100, 0.200, and 0.500 mg/L. To evaluate the effects of the sample matrix, Li was analyzed using calibration standard solutions of 100, 1,000, and 3,000 mg/L prepared from an Agilent 10,000 mg/L Li standard solution.

For quality control (QC) purposes, a blank 10% HNO_3 solution was used as the continuing calibration blank (CCB). A standard solution containing 0.1 mg/L of the 27 elements, prepared separately from the calibration standards, was used as the continuing calibration verification (CCV) solution. The CCB and CCVs were analyzed every 10 to 12 samples. An IS solution containing 20 mg/L Bi and In and 100 mg/L Rb was prepared in 10% HNO_3.

Sample preparation

The sample preparation method for ICP-OES analysis was based on the method outlined in GB/T 11064.16-2013 (*3*). Around 1 g of Li_2CO_3, accurately weighed to ±0.0001 g, was dissolved in 10% HNO_3 solution to a final volume of 100 mL. The solution was allowed to degas for a minimum of 1 hour in a fume hood. Spike experiments were carried out by spiking an aliquot of the Li_2CO_3 solution with the 27 elements at 0.05 and 0.1 mg/L. A blank 10% HNO_3 solution was also spiked as a control. All solutions were prepared in triplicate. To assess the long-term stability of the method for repeated measurements of Li salts, 1% LiOH solutions were also included in the test. The 1% LiOH solutions were prepared following the same procedures as for the 1% Li_2CO_3 solution. The solutions were allowed to cool to room temperature before being spiked with the 27 elements at 0.1 mg/L.

Instrumentation

Elemental analysis of the samples was carried out using the 5800 VDV ICP-OES fitted with a SeaSpray glass concentric nebulizer, double-pass cyclonic spray chamber, an Easy-fit fully demountable torch with 1.8 mm quartz injector, and an argon humidifier. The humidifier enables the accurate analysis of challenging sample types that may potentially cause particulates to build up within the sample introduction system, especially the nebulizer.

The instrument and method were controlled and optimized using the ICP Expert software with the Pro feature pack, which was needed to control the AVS 7. The high level of Li in the Li_2CO_3 samples and the need to analyze trace impurities using axial view of the plasma cause ionization interferences that affect the measurement of EIEs. The instrument operating conditions shown in Table 1 were therefore optimized for the analysis of both trace elements and EIEs.

Sample introduction was performed using the AVS 7 and Agilent SPS 4 autosampler. Control of the AVS 7 and SPS 4 are fully integrated and controlled through the ICP Expert Pro pack software. The software includes an AVS parameter calculator that facilitates the setup and method development of the AVS. The parameters of the AVS 7 are presented in Table 2.

The 5800 ICP-OES features a resilient, vertical, plug-and-play torch configuration. The vertical orientation of the torch ensures uncompromised, robust measurements of the most challenging samples. Also, compared to an axially oriented torch, the vertical torch requires less cleaning, less-frequent torch replacement, and less downtime. The solid-state RF (SSRF) system of the 5800, operating at 27 MHz, produces a reliable, robust, and maintenance-free plasma suitable for high matrix samples, such as Li_2CO_3 samples.

Table 1. Agilent 5800 VDV ICP-OES operating conditions.

Parameter	Setting	
Viewing Mode	Axial	Radial
Viewing Height (mm)	-	8
RF Power (kW)	1.2	
Nebulizer Flow (L/min)	0.70	
Plasma Flow (L/min)	13.5	
Aux Flow (L/min)	1.2	
Pump Speed (rpm)	12	
Replicates	3	
Rinse Time (s)	10	
Read Time (s)	10	5
Stabilization Time (s)	10	0
Sample Pump Tubing	White/white	
Internal Standard Pump Tubing	Black/black	
Waste Pump Tubing	Blue/blue	

Table 2. AVS 7 operating parameters.

Parameter	Setting
Sample Loop Size (mL)	1.0*
Pump Rate - Uptake (mL/min)	35
Pump Rate - Inject (mL/min)	4
Valve Uptake Delay (s)	6
Bubble Inject Time (s)	2
Pre-emptive Rinse Time (s)	1

If IntelliQuant analysis is used as part of the quantitative method, a 1.5 mL sample loop is required.

IntelliQuant Screening for simplified method development and sample insight

The IntelliQuant Screening feature in the ICP Expert Pro pack software allows users to run a quick, 15 s, semiquantitative analysis of a sample making it an ideal tool for method development (*10*). IntelliQuant collects data from the entire spectral range from 167 to 785 nm, providing the semiquantitative concentrations of up to 70 elements. By displaying the results in a color-coded periodic table 'heat map', analysts can see which elements are present in the sample at what concentration-level. The Smart Views function of IntelliQuant provides a simple way for users to filter their screening data to show only those results that are of interest. When Li was chosen as the element of interest in order to determine Li concentration in the 1% Li_2CO_3 sample matrix, the element in the IntelliQuant 'heat map' was highlighted in red as > 1000 ppm, while the smart view window displayed the concentration of Li as 2061 mg/L, as shown in Figure 1.
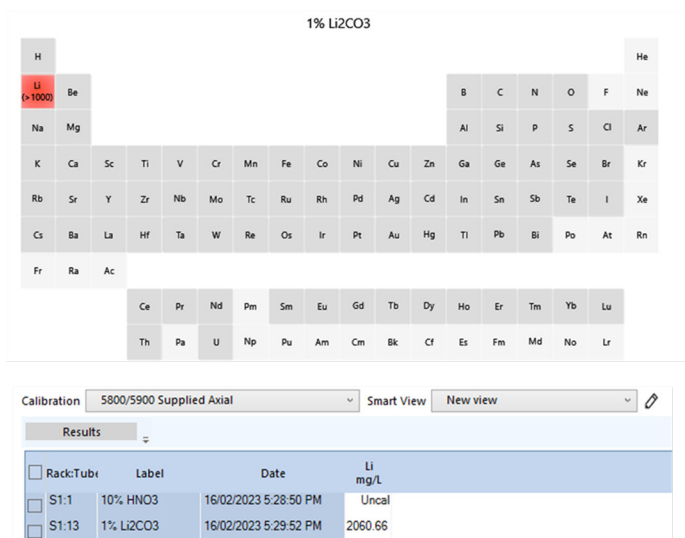
Figure 1. A periodic table 'heat map' (top) and smart view window (bottom) generated by IntelliQuant, showing the concentration of Li present in a 1% Li_2CO_3 solution.

IntelliQuant identifies any potential interferences on element wavelengths and recommends the best lines to use in the quantitative method using a star ranking system. As shown in Figure 2, the wavelengths of Mg 279.553 nm, Na 589.592 nm, and Si 251.611 nm receive a green tick as the recommended lines for Mg, Na, and Si, respectively. The red question mark next to Si 288.158 nm gives information on a spectral interference. Based on the IntelliQuant recommendations, Mg 279.553 nm, Na 589.592 nm, and Si 251.611 nm were used in the quantitative method.

Figure 2. IntelliQuant star ranking for Mg, Na, and Si helped with the selection of which analyte wavelength to use in the quantitative method.

IntelliQuant Screening is an ideal tool for method development as it requires no prior knowledge of the samples or advanced knowledge of spectroscopy. With minimal setup required, IntelliQuant Screening helps users create a customized quantitative method for the sample analysis, saving method development time, and providing confidence in the method.

AVS for reduced maintenance and high sample throughput

The use of the AVS is helpful when dealing with challenging samples such as compounds that contain high levels of EIEs (Li in the case of Li_2CO_3). The challenges of using a conventional sample introduction system without AVS include low productivity due to the long rinse time required to minimize sample carryover and the high maintenance needs due to Li deposits on the torch.

The AVS rinses the sample introduction system while the next sample is delivered to the instrument, virtually eliminating the delay-times of a conventional ICP-OES analysis. Shorter analysis times mean that argon consumption can be reduced by at least 50% per sample, reducing running costs. More efficient analyses and less sample passing through the torch minimize the exposure of the torch, nebulizer, and pump tubing to complex samples, increasing the lifetime of consumables and further reducing costs. Using argon bubble injection between the sample and rinse solution reduces carry over in the spray chamber of the 5800 and maximizes measurement time for a given sample loop, leading to a more stable plasma and better analytical precision.

In this study, the average measuring time per sample was reduced from 137 to 84 s using the AVS 7. After almost 400 high concentration Li (>1000 mg/L) samples were measured without any maintenance, no deterioration of instrument performance was detected. When an Agilent semi- or fully demountable torch is used, the outer tube set can be replaced less frequently than using conventional sample introduction. The AVS 6/7 significantly reduces the total running costs and improves productivity.

EMF for proactive maintenance

The 5800 ICP-OES has over 100 sensors that monitor instrument performance. The ICP Expert software includes an EMF function that uses these sensors and other counters to alert the analyst when maintenance is required. Traffic light color-coding of the counters show which maintenance activities should be done immediately (red), which are due soon (amber), and which can wait (green). EMF reduces downtime and repair costs by scheduling routine maintenance of components based on actual use (e.g., days elapsed, number of samples run, plasma on hours), rather than at set time intervals (e.g., daily, weekly, or monthly). Analysts can set the counter limits or add new counters to suit their specific needs. As shown in Figure 3, when the counter for wavelength calibration has elapsed, a pop-up window alerts the user to perform the maintenance task. This ensures routine maintenance is not missed and keeps the instrument running at its optimum level of performance.
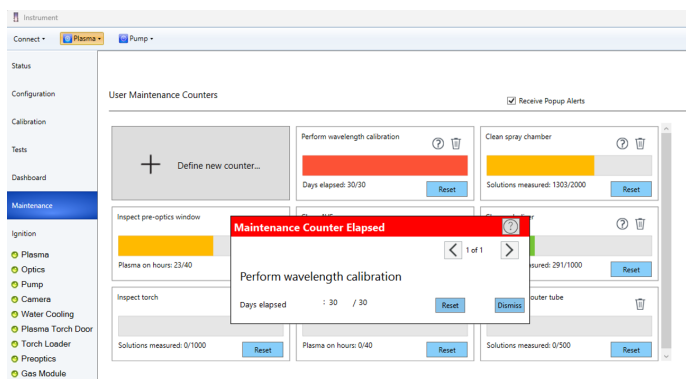

Figure 3. The EMF function in Agilent ICP Exert software uses traffic light color-coded counters for proactive maintenance, for example with a pop-up alert for wavelength calibration.

Results and discussion

Calibration linearity and internal standardization

The calibration correlation coefficients (R) of the 27 elements at selected wavelengths are summarized in Table 3. Good linearity (R>0.999) was achieved for all elements in their respective working ranges. Representative calibration curves for K 766.491 and Na 589.592 nm are shown in Figure 4.
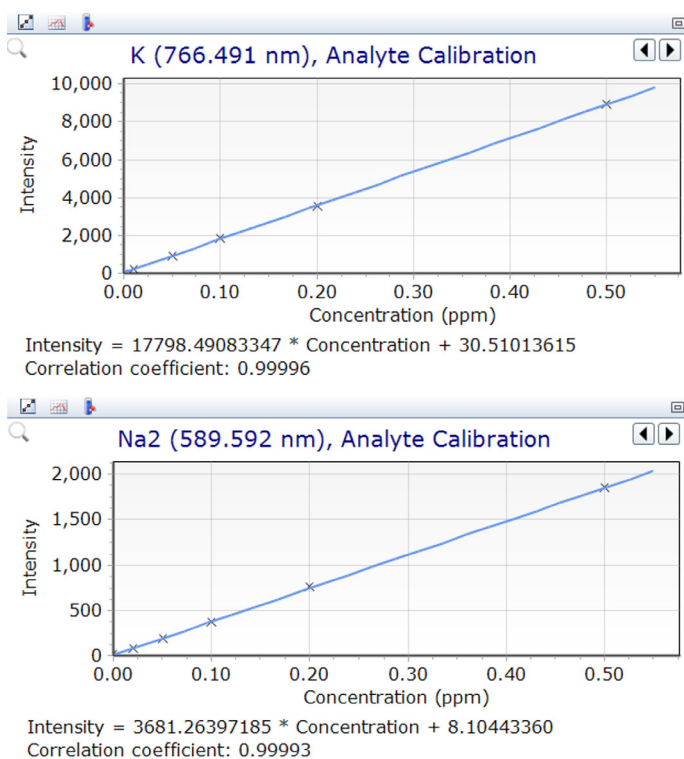

Intensity = 17798.49083347 * Concentration + 30.51013615
Correlation coefficient: 0.99996

Intensity = 3681.26397185 * Concentration + 8.10443360
Correlation coefficient: 0.99993

Figure 4. Calibration curves of K 766.491 nm (axial) and Na 589.592 nm (radial).

Internal standardization is an important tool for sample matrix correction. In general, the IS element and analyte are matched in terms of atomic or ionic state wavelengths (Table 3). The use of ISs is critical to this application as the concentration of Li, an EIE, is as high as 2000 mg/L in the sample matrix. EIEs have low ionization energies and are easily ionized in the plasma. When EIEs are present at a very high level, the plasma electron density and the atomization-ionization equilibria are affected. These effects cause either an enhancement or suppression of the emission signals, leading to the reporting of either false high or false low element concentrations. The ionization interference is especially significant in axial view and when both the analyte (e.g. K and Na) and the co-existing element (e.g. Li) have low ionization potentials (*6*). In this study, due to the very high ionization interferences on Na from Li in axial view mode, Na was measured in radial view without the need for IS correction.

Table 3. Calibration range, viewing mode, linear correlation coefficient, background correction mode, and internal standard of 27 elements at selected wavelengths.

Element and Wavelength (nm)	Working Range (mg/L)	Viewing Mode	Correlation Coefficient	Background Correction Mode	IS and Wavelength (nm)
Al 396.152	0.010 – 0.500	Axial	0.9994	FACT	In 325.609
As 193.696	0.010 – 0.500	Axial	0.9997	Fitted	Bi 223.061
B 249.772	0.010 – 0.500	Axial	1.0000	Fitted	Bi 223.061
Ba 455.403	0.010 – 0.500	Axial	1.0000	Fitted	Bi 190.171
Be 234.861	0.010 – 0.500	Axial	1.0000	Fitted	Bi 223.061
Ca 396.847	0.010 – 0.500	Axial	0.9999	Fitted	Bi 190.171
Cd 214.439	0.010 – 0.500	Axial	1.0000	Fitted	Bi 190.171
Co 238.892	0.010 – 0.500	Axial	1.0000	Fitted	Bi 190.171
Cr 267.716	0.010 – 0.500	Axial	1.0000	Fitted	Bi 190.171
Cu 327.395	0.010 – 0.500	Axial	0.9999	Fitted	Bi 306.771
Fe 238.204	0.010 – 0.500	Axial	0.9999	Fitted	Bi 190.171
Ga 294.363	0.010 – 0.500	Axial	0.9998	Fitted	Bi 306.771
K 766.491	0.010 – 0.500	Axial	0.9999	Fitted	Rb 780.026
Mg 279.553	0.010 – 0.500	Axial	0.9995	Fitted	Bi 190.171
Mn 257.610	0.010 – 0.500	Axial	1.0000	Fitted	Bi 190.171
Mo 202.032	0.010 – 0.500	Axial	1.0000	Fitted	Bi 190.171
Na 589.592	0.020 – 0.500	Radial	0.9999	FACT	-
Ni 231.604	0.010 – 0.500	Axial	1.0000	FACT	Bi 190.171
Pb 220.353	0.010 – 0.500	Axial	1.0000	FACT	Bi 190.171
S 180.669	0.010 – 0.500	Axial	0.9999	Fitted	In 325.609
Sb 206.834	0.010 – 0.500	Axial	0.9999	Fitted	Bi 223.061
Si 251.611	0.010 – 0.500	Axial	1.0000	Fitted	Bi 223.061
Sr 407.771	0.010 – 0.500	Axial	1.0000	Fitted	Bi 190.171
Ti 336.122	0.010 – 0.500	Axial	1.0000	Fitted	Bi 190.171
V 292.401	0.010 – 0.500	Axial	1.0000	Fitted	Bi 190.171
Zn 213.857	0.010 – 0.500	Axial	1.0000	Fitted	Bi 223.061
Zr 343.823	0.010 – 0.500	Axial	1.0000	Fitted	Bi 190.171

Background correction

FBC was used for background correction of most of the elements. FBC uses an advanced algorithm to model the background signal under the analyte peak. FBC is applied simultaneously upon measurement of the analyte peak, providing fast, easy, and accurate background correction. Eliminating the need for the user to manually determine off-peak background correction points, FBC provides accurate and repeatable correction of both simple and complex background structures. As the example in Figure 5 shows, FBC accurately models and corrects OH 327.421 nm interference on Cu 327.395 nm.
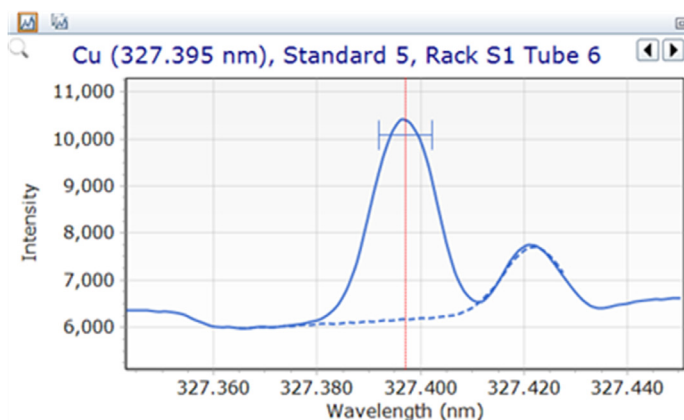

Figure 5. The spectrum of Cu 327.395 nm corrected using FBC.

Limit of detection (LOD) and limit of quantitation (LOQ)

To obtain LODs and LOQs, 10 solutions of 1% Li_2CO_3 in 10% HNO_3 were analyzed. The LODs and LOQs were calculated as 3 and 10 times the standard deviation, respectively, of the 10 measurements. The values presented in Table 4 are the average of three LOD and LOQ results from separate tests carried out on three nonconsecutive days, calculated as 1 g Li_2CO_3 in 100 mL solution. The high sensitivity of the 5800 ICP-OES is demonstrated by the low LODs and LOQs, which meet the specification requirement for elemental impurities in the Chinese industrial standard YS/T 582-2013 for battery grade Li_2CO_3. For example, Cu, Mn, Pb, and Zn ≤ 3 mg/kg, and Na ≤ 250 mg/kg.

Elemental impurities in Li_2CO_3 and spike recoveries

The 1% Li_2CO_3 solution and the solutions spiked at 0.05 and 0.1 mg/L were analyzed using the 5800 VDV ICP-OES. The quantitative results for the 99% pure Li_2CO_3 sample and the respective spike recoveries for 27 elements are shown in Table 4.

Most of the impurity elements present in the Li_2CO_3 sample were measured below 1 mg/kg, while the concentration of the major elements, Si, Ca, Na, S, K, Mg, and Zn, in the solid Li_2CO_3 were between 1 and 20 mg/kg. Li was also quantified in the 1% Li_2CO_3 solution. The measured concentration of 2061 mg/L confirmed the semiquantitative result obtained by IntelliQuant (Figure 1).

Spike recovery tests are an effective way to evaluate the reliability of the sample preparation method and/or the analytical method, especially when no certified reference materials (CRMs) are available. As shown in Table 4, excellent spike recoveries within ±10% of the expected value were achieved for all 27 elements at both 0.05 and 0.1 mg/L spike levels. These results confirm the accuracy of the 5800 ICP-OES method for the analysis of low-level impurities, despite the very high Li level in the sample matrix.

Table 4. LOD and LOQ (calculated as 1 g sample in 100 mL solution), concentrations of elemental impurities in 99% Li_2CO_3, and spike recovery data at 5 and 10 mg/kg spike concentrations. Concentration units: mg/kg, n=3.

Element, Wavelength (nm)	LOD	LOQ	Li_2CO_3	Li_2CO_3 Spiked at 5 mg/kg*		Li_2CO_3 Spiked at 10 mg/kg*	
			Measured Concentration	Measured Concentration	Spike Recovery %	Measured Concentration	Spike Recovery %
Al 396.152	0.19	0.64	0.328	5.63	106	11.0	107
As 193.696	0.57	1.9	0.595	5.90	106	11.1	105
B 249.772	0.12	0.41	0.891	5.94	101	11.5	106
Ba 455.403	0.013	0.044	0.101	5.33	105	10.6	105
Be 234.861	0.0075	0.025	< LOD	5.31	106	10.7	107
Ca 396.847	0.32	1.1	15.1	20.1	100	25.0	99
Cd 214.439	0.026	0.087	< LOD	5.03	101	10.0	100
Co 238.892	0.078	0.26	< LOD	4.95	99	9.94	99
Cr 267.716	0.064	0.21	< LOD	5.02	100	9.97	100
Cu 327.395	0.085	0.28	0.614	5.66	101	11.2	106
Fe 238.204	0.100	0.33	0.799	5.66	97	10.7	99
Ga 294.363	0.48	1.6	0.832	5.72	98	11.3	105
K 766.491	0.13	0.42	1.95	7.29	107	12.9	110
Mg 279.553	0.11	0.36	1.73	6.61	98	11.5	98
Mn 257.610	0.027	0.088	0.0721	4.90	97	9.77	97
Mo 202.032	0.22	0.73	< LOD	5.22	104	10.3	103
Na 589.592	1.9	6.2	6.74	11.7	99	16.5	98
Ni 231.604	0.14	0.48	0.305	5.06	95	9.82	95
Pb 220.353	0.44	1.5	< LOD	5.24	105	10.9	109
S 180.669	0.64	2.1	2.64	7.43	96	11.7	91
Sb 206.834	0.70	2.3	< LOD	5.31	106	10.4	104
Si 251.611	0.40	1.3	19.3	24.5	104	30.0	107
Sr 407.771	0.0045	0.015	< LOD	5.09	102	10.2	102
Ti 336.122	0.024	0.081	< LOD	5.04	101	10.4	104
V 292.401	0.14	0.47	< LOD	5.28	106	10.5	105
Zn 213.857	0.090	0.30	1.29	6.36	101	11.8	105
Zr 343.823	0.093	0.31	< LOD	4.96	99	10.1	101

Spike concentrations, 5 and 10 mg/kg, are equivalent to 0.05 and 0.1 mg/L, respectively, of 1 g Li_2CO_3 in 100 mL.

Long-term stability

To check the stability of the instrument and long-term validity of the calibration, 290 measurements were completed over 6.8 hours at an average rate of 84 s per sample, without recalibration. The sequence included 120 measurements of 1% Li_2CO_3 and 120 of 1% LiOH, both spiked at 0.1 mg/L and also 25 pairs of CCBs and CCVs (0.1 mg/L). 10 solutions of spiked 1% Li_2CO_3 alternated with 10 solutions of spiked 1% LiOH with a CCB and a CCV in between.

The concentrations of the CCV measurements were normalized as percentage recoveries of the expected value (0.1 mg/L) and plotted against time (Figure 6). Accurate measurements within ±10% of the expected value (indicated by the dotted lines) were obtained, with %RSDs for all wavelengths below 3%, except for S 180.669 (3.5%) and Na 589.592 (4.3%). The precision of the 240 measurements of the spiked 1% Li_2CO_3 and 1% LiOH samples was excellent over 6.8 hours, with %RSDs below 5%, as shown in Table 5. More information on the analysis of LiOH can be found in reference 11.

The vertical torch position and the SSRF system of the 5800 ICP-OES provides excellent plasma robustness and long-term stability, especially for high Li samples. The outcome is a reduced number of reruns of samples and QC solutions and higher sample throughput.
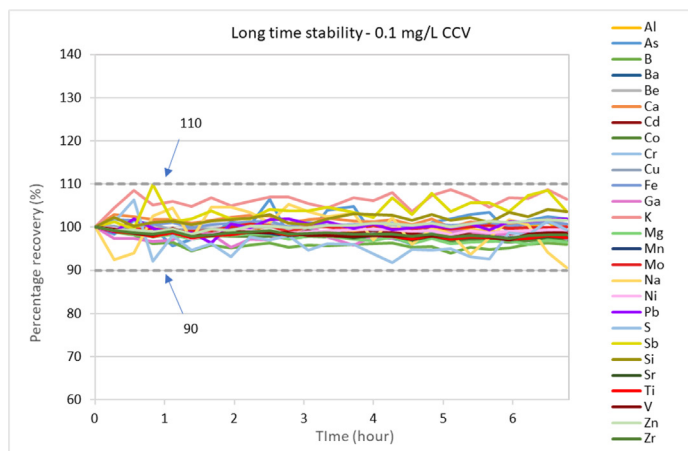

Figure 6. Long-term stability showing percentage recoveries of a 0.1 mg/L CCV solution measured over 6.8 hours.

Table 5. Long-term stability of the Agilent 5800 ICP-OES over 6.8 h. %RSD of measurements of spiked 1% Li_2CO_3 and 1% LiOH samples.

Element and Wavelength (nm)	RSD %	Element and Wavelength (nm)	RSD %
Al 396.152	1.3	Mn 257.610	1.4
As 193.696	4.0	Mo 202.032	0.82
B 249.772	1.9	Na 589.592	4.5
Ba 455.403	1.6	Ni 231.604	1.0
Be 234.861	0.52	Pb 220.353	1.5
Ca 396.847	1.5	S 180.669	4.0
Cd 214.439	1.1	Sb 206.834	3.7
Co 238.892	1.3	Si 251.611	0.65
Cr 267.716	1.3	Sr 407.771	1.5
Cu 327.395	1.2	Ti 336.122	1.6
Fe 238.204	2.2	V 292.401	1.4
Ga 294.363	1.4	Zn 213.857	0.56
K 766.491	0.77	Zr 343.823	1.4
Mg 279.553	1.8		

Conclusion

The Agilent 5800 VDV ICP-OES was used to quantify 27 elemental impurities in a 99% pure Li_2CO_3 solid sample, a precursor chemical that is used in the production of LIB cathodes and electrolytes. IntelliQuant Screening helped with method development and provided additional sample information to improve data quality.

The use of the Agilent AVS switching valve improved sample throughput by reducing run time per sample by over 50 s compared to using conventional sample introduction, without any compromise in performance. The use of a fully demountable Easy-fit torch further lowered maintenance and running costs. The resilient vertical torch used in the 5800 ICP-OES provided the best configuration for handling high Li samples.

Excellent calibration linearity and low detection limits were obtained for both major and minor elements. Recoveries of all 27 elements spiked at two concentrations in the sample solutions were between 90 and 110%, confirming the accuracy of the ICP-OES method. The instrument was stable over 6.8 h.

The results have shown that the 5800 ICP-OES with AVS 7 can provide accurate, precise, and stable measurements of trace elements in Li_2CO_3 over long runs with low maintenance needs. The robust method is suitable for the routine QC of chemicals used in LIB manufacturing.

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8. Agilent IntelliQuant Software: For greater sample insight and simplified method development, Agilent publication, 5994-1516EN

9. ICP-OES Background and Interference Removal Accurate. Easy. Reliable. Agilent publication, 5991-8452EN

10. Agilent IntelliQuant Screening: Smarter and quicker semiquantitative ICP-OES analysis, Agilent publication, 5994-1518EN

11. Qi, Y., and Drvodelic, N., Determination of Elemental Impurities in Lithium Hydroxide Using ICP-OES, Agilent publication, 5994-6172EN

Note: If the AVS 6/7 switching valve is bought for use with the Agilent 5800 VDV ICP-OES, ICP Expert Pro pack software is included. The software is needed to control the AVS 6/7 and to access IntelliQuant Screening—a useful tool for method development. The AVS 6/7 and ICP Expert Pro pack software are standard with the Agilent 5900 SVDV ICP-OES.

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
G8010-60263	Quartz outer tube set for semi- and fully-demountable torches for Agilent 5000 series VDV/SVDV 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, white/white, 12/pk
3710027200	Peristaltic pump tubing, black/black, 12/pk
3710034600	Peristaltic pump tubing, blue/blue, 12/pk.
G8495-60000	Spare valve, 7 port, 2 position, for AVS 7 switching valve
G8495-60003	Stator and rotor, for 7-port valve, for AVS7 switching valve
G8010-60306	Autosampler connection kit for AVS switching valve
G8010-60300	Check valve outlet for AVS switching valve, non-metallic
G8010-60307	Internal standard connection kit, for AVS 7 switching valve
G8010-60304	Nebulizer connection kit for AVS switching valve
G8010-60308	Pump outlet connection kit, for AVS switching valve
G8010-60303	Sample inlet connection kit for AVS switching valve
G8010-60335	Sample/Rinse connection kit, for AVS switching valve, 2/pk
5190-8243	Agilent 1000 ppm single element stock solution for Al
5190-8249	Agilent 1000 ppm single element stock solution for Ba
5190-8328	Agilent 1000 ppm single element stock solution for Cd
5190-8347	Agilent 1000 ppm single element stock solution for Co
5190-8349	Agilent 1000 ppm single element stock solution for Cu
5190-8472	Agilent 1000 ppm single element stock solution for Fe
5190-8468	Agilent 1000 ppm single element stock solution for In
5190-8504	Agilent 1000 ppm single element stock solution for K
5190-8482	Agilent 1000 ppm single element stock solution for Mg
5190-8484	Agilent 1000 ppm single element stock solution for Mn
5190-8487	Agilent 1000 ppm single element stock solution for Mo
5190-8492	Agilent 1000 ppm single element stock solution for Ni
5190-8476	Agilent 1000 ppm single element stock solution for Pb
5190-8527	Agilent 1000 ppm single element stock solution for Sr
5190-8546	Agilent 1000 ppm single element stock solution for Ti
5190-8559	Agilent 1000 ppm single element stock solution for Zr
5190-8356	Agilent 10,000 ppm single element stock solution for As
5190-8365	Agilent 10,000 ppm single element stock solution for B
5190-8362	Agilent 10,000 ppm single element stock solution for Bi
5190-8360	Agilent 10,000 ppm single element stock solution for Be
5190-8369	Agilent 10,000 ppm single element stock solution for Ca
5190-8375	Agilent 10,000 ppm single element stock solution for Cr
5190-8389	Agilent 10,000 ppm single element stock solution for Ga
5190-8408	Agilent 10,000 ppm single element stock solution for Li

Part numbers continue on next page

5190-8206	Agilent 10,000 ppm single element stock solution for Na
5190-8441	Agilent 10,000 ppm single element stock solution for Rb
5190-8210	Agilent 10,000 ppm single element stock solution for S
5190-8354	Agilent 10,000 ppm single element stock solution for Sb
5190-8451	Agilent 10,000 ppm single element stock solution for Si
5190-8229	Agilent 10,000 ppm single element stock solution for V
5190-8235	Agilent 10,000 ppm single element stock solution for Zn

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