Application Note

Energy and Materials

Analysis of Elemental Impurities in Lithium Iron Phosphate Cathode Materials for LIBs by ICP-OES

Accurate, robust measurement of 31 elements in LFP using the Agilent 5800 ICP-OES

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Introduction

The global market for lithium-ion batteries (LIBs) is forecast to increase significantly by 2032 (*1*). Sectors involved in the transition towards cleaner technologies such as manufacturers of electric vehicles (EVs) and providers of grid-energy storage systems are driving demand (*2, 3*). To realize the potential of the expanding LIB market, an increasing number of LIB manufacturing facilities—and LIB recycling sites—are opening around the world. At the same time, there is ongoing investment in research and development (R&D) projects aimed at enhancing battery safety, capacity, and efficiency. These projects are focused on extending battery range and reducing production costs.

Among the components of a Li-ion battery (cathode, anode, separator, and electrolyte), the materials used for the cathode can account for a high portion of the total cost of the battery. The cathode typically consists of Li combined with a metal oxide compound that contains cobalt (Co). Examples include Co oxide (LCO), nickel (Ni) manganese (Mn) Co oxide (NMC or NCM), Ni Co aluminum (Al) oxide (NCA), or Ni Mn Co Al oxide (NMCA). However, the limited sources of Co globally affect its price, especially when demand is high, and there are ethical and environmental concerns about its extraction in the Democratic Republic of Congo. Given these concerns, there is a growing interest in reducing or eliminating the use of Co in LIBs. Therefore, Co-free cathode materials have been developed, including Li iron (Fe) phosphate (LFP) and Li Mn oxide (LMO) (*4*). LFP ($LiFePO_4$) is known for its safety, long cycle life, and thermal stability (*5*).

LIB manufacturers require high-quality cathode materials, since the purity of all battery components is critical to the performance, safety, and lifespan of the end products. So, accurate and reliable analytical methods are 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 (*6*).

In this study, commercially available high purity $LiFePO_4$ powder, which was used to represent the LFP cathode material, was prepared for analysis by the Agilent 5800 Vertical Dual View (VDV) ICP-OES.

To help with method development and sample-knowledge, the samples were first analyzed using the IntelliQuant Screening function of Agilent ICP Expert Pro instrument control software. The semiquantitative screening data provides valuable information on the identity and concentration of elements in a sample. The data can be used to establish a suitable calibration concentration range for the elements included in the quantitative method. IntelliQuant Screening data also identifies spectral interferences and recommends the best interference-free wavelengths to use in the quantitative method, ensuring the accuracy of the final results.

For the quantitative method, the 5800 VDV ICP-OES was operated in axial and radial view mode to quantify trace impurities and major elements, respectively, in the $LiFePO_4$ samples. To ensure that sufficient rinse times were used during the analysis of high matrix samples, Intelligent Rinse was selected in the ICP Expert Pro software. The Intelligent Rinse routine automatically optimizes the rinse time between samples depending on user-selected threshold settings.

Experimental

Materials, samples, and standard solutions

Trace metal (Aristar Plus) grade concentrated nitric acid (HNO_3) (67 to 70%) and hydrochloric acid (HCl) (34 to 37%) were bought from VWR International. The acids were used to prepare a 5% aqua regia (AR) solution to be used as the blank and rinse solution. The AR solution was prepared in a 1000 mL bottle by adding 37.5 mL concentrated HCl and 12.5 mL concentrated HNO_3 to 950 mL of de-ionized (DI) water.

To represent the LFP cathode material, high purity lithium iron (II) phosphate ($LiFePO_4$, 97%+ purity) was bought from Sigma Aldrich.

Calibration standards were prepared from Agilent standard solutions. The standards included: 100 mg/L multi-element standard QC27, 1,000 mg/L single element standards for Hg, Sn, W, and S; and 10,000 mg/L single element standards for Fe, Li, and P. A mixed internal standard (IS) solution of Y and Rb was prepared from two single element stock standard solutions at 10,000 mg/L.

Sample preparation

Approximately 0.1 g of the LFP powdered sample was weighed directly into a microwave digestion tube to ± 0.0001 g. As there was some static observed, 0.5 mL of DI water was added using a pipette to moisten the samples and to rinse the walls of the microwave tubes. Each sample was thoroughly mixed in 8 mL AR (6 mL HCl and 2 mL HNO_3). Samples and the spiked-sample solutions were prepared in triplicate using the same procedure. The LFP samples were spiked at 0.1 mg/L before microwave digestion.

All samples were digested using a Mars 6 Microwave Digestion System (CEM Corporation) at 200 °C, holding for 20 mins. After digestion, each solution was made up to 50 mL using DI water.

For the long-term stability study, an LFP sample was digested using 0.2 g of sample in a final volume of 100 mL and spiked at 0.10 mg/L.

Calibration standards and internal standards

Agilent standards were used to prepare the calibration standards and sample spikes. Stock solutions were prepared at 10 mg/L by diluting the 100 mg/L QC27 standard solution and the 1,000 and 10,000 mg/L single element standards in 5% HNO_3. To prepare calibration standard solutions in the analytical working range, the 10 mg/L stock solutions were diluted to the desired concentrations using the 5% AR matrix solution. All the trace elements were calibrated using standards at 0, 0.05, 0.150, 0.250, and 0.500 mg/L. Sulfur was also calibrated 0 to 0.500 mg/L, but separately, using a single-element standard, to avoid reaction with some of the trace elements such as Ba, Pb, etc. The matrix elements were calibrated as follows: Fe at 0, 50, 150, 250, 500, and 1000 mg/L; Li at 0, 5, 25, 50, and 100 mg/L; and P at 0, 25, 125, 250, and 500 mg/L.

For QC, a blank 5% AR solution was used as the continuing calibration blank (CCB) and a 0.1 mg/L standard solution was used as a continuing calibration verification (CCV) solution. The CCV was prepared separately from the calibration standards and included all elements apart from Li, Fe, and P. The CCB and CCV were analyzed after every 10 samples.

An IS solution containing 5 mg/L Y and 75 mg/L Rb was prepared in 5% AR.

Instrumentation

The 5800 VDV ICP-OES was fitted with a standard (default) sample introduction system comprising a SeaSpray nebulizer, double-pass glass cyclonic spray chamber, and a one-piece 1.8 mm i.d injector Easy-fit torch. An argon humidifier was used to reduce desolvation at the nebulizer caused by samples with a high total dissolved solids (TDS) content and to improve the stability of the instrument over long runs. The instrument and method were controlled and optimized using ICP Expert Pro software, which was also used to process the analytical data.

To provide the robustness and stability of the plasma over long analytical runs of complex LIB samples, the 5800 VDV ICP-OES uses a vertical plasma, a solid-state radio frequency (SSRF) generator operating at 27 MHz, and a Cooled Cone Interface (CCI). The CCI deflects the plasma's cooler tail, avoiding interferences that form in the cooler region. So, when reading the vertical plasma in axial view mode, elements at trace concentration levels can be measured with minimal interfering effects.

The instrument operating conditions were optimized to take account of easily ionizable elements (EIEs), such as Li, Na, K, and Ca. These elements have low ionization energies and are easily ionized in the plasma. If EIEs are present in a sample at a high enough concentration, 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. Internal standards can be used to correct for these effects in the plasma.

The Intelligent Rinse function within ICP Expert Pro software was used to monitor the intensities of nominated element wavelengths during the rinse period and to control the Agilent SPS 4 autosampler rinse times. The software automatically ends the rinse when element intensities reach a user-specified threshold. There are three defined washout thresholds within Intelligent Rinse: quick, moderate, and thorough. Monitoring signal intensities means that rinse periods vary, depending on the time taken to wash out each sample. In this study, *thorough* mode was used and rinse times varied from 3 s for blanks to 45 s for the LFP samples, based on the concentration (threshold setting) for Li, Fe, P, B, Na, and Si. Intelligent Rinse is a simple way to achieve effective and accurate washout, while optimizing sample throughput and argon usage.

Instrument operating parameters are given in Table 1.

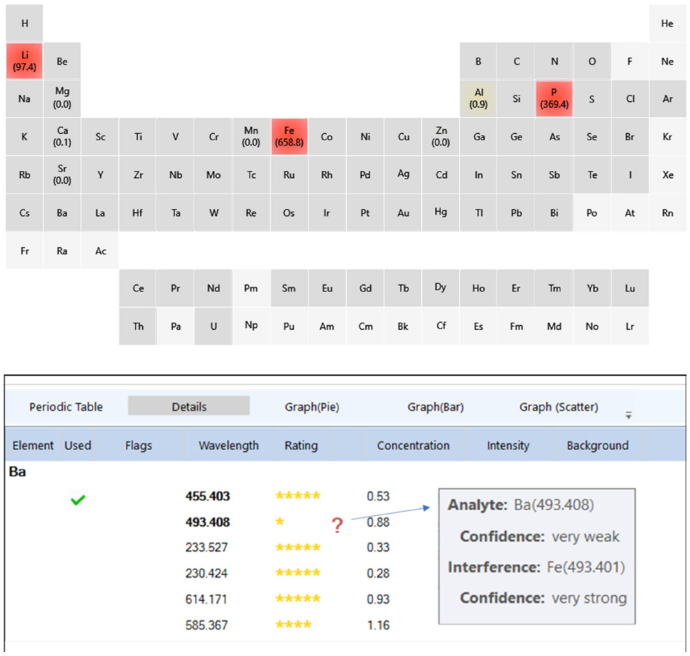
Table 1. Agilent 5800 VDV ICP-OES operating conditions.

Parameter	Setting	
Viewing Mode	Axial (Trace elements)	Radial (Fe, Li, P)
RF Power (kW)	1.3	1.2
Nebulizer Flow (L/min)	0.70	
Plasma Flow (L/min)	12	
Aux Flow (L/min)	1.0	
Pump Speed (rpm)	12	
Viewing Height (mm)	NA	10
Replicates	3	
Intelligent Rinse	Enabled (*Thorough*)	
Read Time (s)	10	7
Stabilization Time (s)	15	10
Sample Pump Tubing	White/white	
Internal Standard Pump Tubing	Orange/green	
Waste Pump Tubing	Blue/blue	

Method development

IntelliQuant Screening was used to confirm or recommend the best analyte wavelengths to use for the quantitative analysis of the $LiFePO_4$ sample digests. The results also provided estimated concentrations of the elements in the samples, helping to determine the appropriate working calibration range for each analyte.

To aid interpretation of the semiquantitative data, the IntelliQuant results can be presented visually, including as a periodic table 'heat map', a pie chart, or a bar chart. The periodic table heat map of a digested LFP sample (Figure 1, top) shows a high concentration of Fe (not corrected for dilution). Fe can cause interferences on analytes such as Ba, potentially leading to false positive readings, as flagged by ICP Experts' star ranking on the Ba 493.408 nm analyte wavelength (Figure 1, bottom). The analyst can use the insights provided by the IntelliQuant Screening data when considering background correction, as shown in Figure 2.



H																	He
Li (97.4)	Be											B	C	N	O	F	Ne
Na	Mg (0.0)											Al (0.9)	Si	P (369.4)	S	Cl	Ar
K	Ca (0.1)	Sc	Ti	V	Cr	Mn (0.0)	Fe (658.8)	Co	Ni	Cu	Zn (0.0)	Ga	Ge	As	Se	Br	Kr
Rb	Sr (0.0)	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe
Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn
Fr	Ra	Ac															
			Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu	
			Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr	

Element	Used	Flags	Wavelength	Rating		Concentration	Intensity	Background
Ba								
	✓		**455.403**	★★★★★		0.53		
			493.408	★	?	0.88		
			233.527	★★★★★		0.33		
			230.424	★★★★★		0.28		
			614.171	★★★★★		0.93		
			585.367	★★★★		1.16		

Analyte: Ba(493.408)
Confidence: very weak
Interference: Fe(493.401)
Confidence: very strong

Figure 1. Top: IntelliQuant Screening periodic table 'heat map' view showing which elements are present in of a digested LFP sample. All values are semiquantitative and are reported in ppm. Bottom: IntelliQuant star ranking information for Ba. The information helps with selection of the best analyte wavelength to use in the quantitative method.

Background correction

The fitted background correction (FBC) mode was selected for most elements in the ICP Expert software (Table 2). FBC automatically corrects the contribution from the background and interfering peaks that do not directly overlap the analyte peak. No manual background marker selection is needed when using FBC.

The Agilent VistaChip III charge-coupled device (CCD) detector of the 5800 provides high-speed continuous wavelength coverage from 167 to 785 nm. The extensive wavelength range often ensures that a suitable, interference-free line can be found for most analytes, although complex samples often generate many emission lines. In some cases, these lines can be located too closely to the analyte line of choice. When an interfering peak overlaps the spectral peak of an analyte, the Fast Automated Curve-fitting Technique (FACT) can be used to effectively resolve the interference. FACT provides real-time spectral correction using an advanced spectral modeling technique to mathematically separate the analyte signal from the raw spectrum, greatly improving detection limits of any interfered analytes.

Figure 2 shows the uncorrected spectrum of Cr 267.716 nm (blue solid line) that is a combination of the analyte signal and the interfering peak from P 267.711 nm (red dotted line). FACT was used to model the interfering peak from P 267.711 nm and resolve the analyte signal, providing accurate results for Cr 267.716 nm (green solid line in Figure 2).

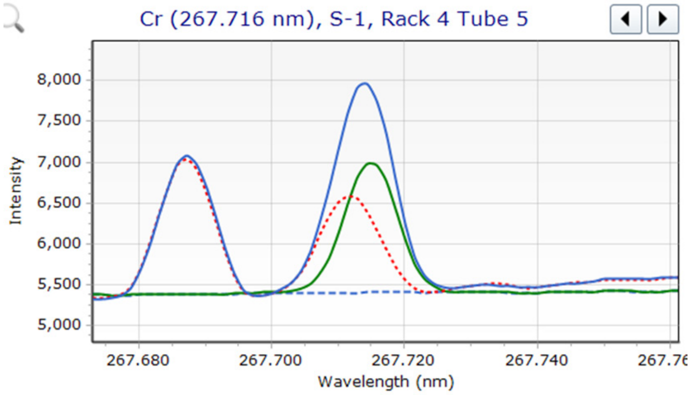
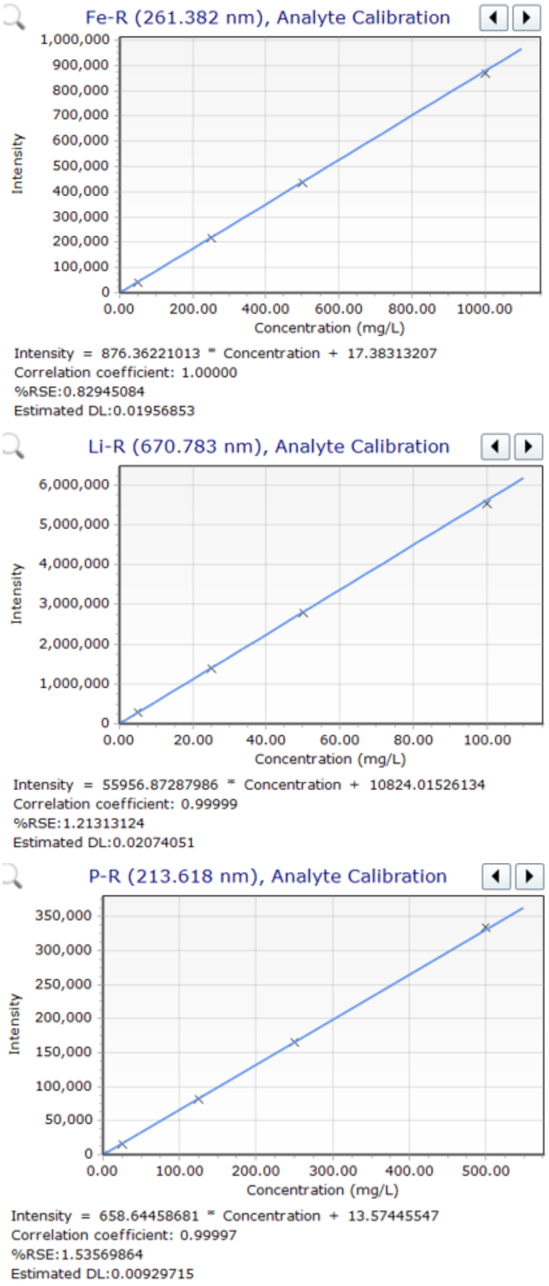


Figure 2. FACT background correction used to resolve the spectral interference of P on Cr (total signal shown by the solid blue line). The P 267.711 nm (red dashed) interference line is overlapping the Cr 267.716 nm (solid green) analyte line. The signal from the blank is shown by the light blue dashed line.

Results and discussion

Calibration linearity

The calibration correlation coefficients of the 31 elements at selected wavelengths are summarized in Table 2. Good linearity was achieved for all elements in their respective working ranges, as shown by R values close to 1. Representative calibration curves for the three matrix elements and three trace elements and are shown in Figures 3 and 4, respectively.


Fe-R (261.382 nm), Analyte Calibration

```
Intensity = 876.36221013 * Concentration + 17.38313207
Correlation coefficient: 1.00000
%RSE:0.82945084
Estimated DL:0.01956853
```

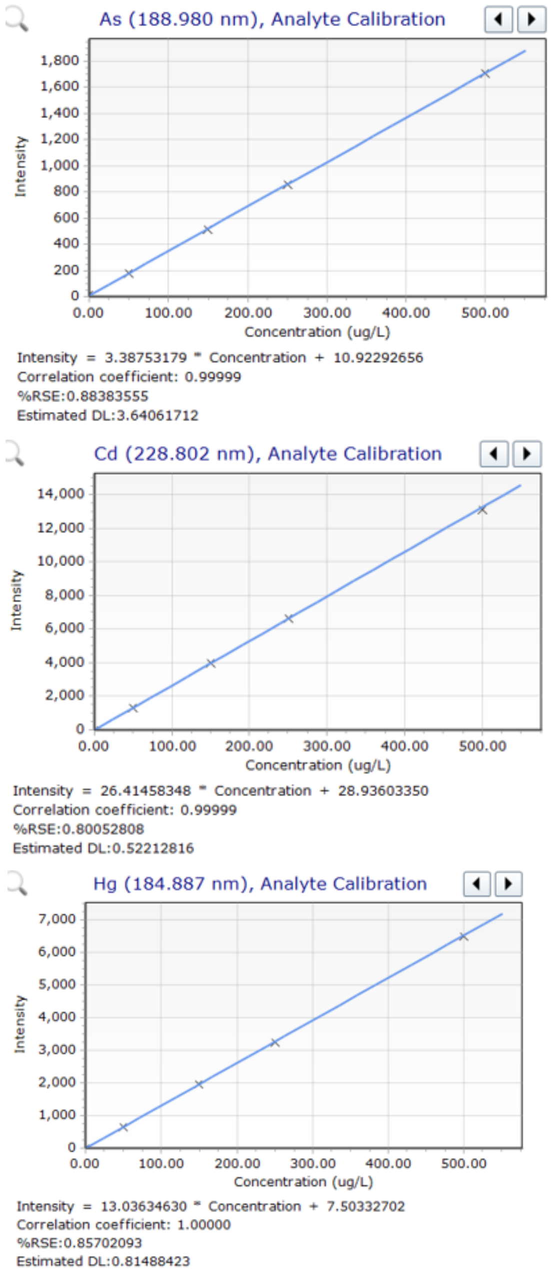
Li-R (670.783 nm), Analyte Calibration

```
Intensity = 55956.87287986 * Concentration + 10824.01526134
Correlation coefficient: 0.99999
%RSE:1.21313124
Estimated DL:0.02074051
```

P-R (213.618 nm), Analyte Calibration

```
Intensity = 658.64458681 * Concentration + 13.57445547
Correlation coefficient: 0.99997
%RSE:1.53569864
Estimated DL:0.00929715
```

Figure 3. Calibration curves of matrix elements: Fe 261.382, Li 670.783, and P 213.618 nm.


As (188.980 nm), Analyte Calibration

```
Intensity = 3.38753179 * Concentration + 10.92292656
Correlation coefficient: 0.99999
%RSE:0.88383555
Estimated DL:3.64061712
```

Cd (228.802 nm), Analyte Calibration

```
Intensity = 26.41458348 * Concentration + 28.93603350
Correlation coefficient: 0.99999
%RSE:0.80052808
Estimated DL:0.52212816
```

Hg (184.887 nm), Analyte Calibration

```
Intensity = 13.03634630 * Concentration + 7.50332702
Correlation coefficient: 1.00000
%RSE:0.85702093
Estimated DL:0.81488423
```

Figure 4. Calibration curves of representative trace elements: As 188.980, Cd 228.802, and Hg 184.887 nm.

Method detection limits

Two sets of method detection limits (MDLs) were determined in this study based on 3 x standard deviation (SD) of 10 measurements of the elements in the blank and in a salt solution (Table 2). A blank AR solution was spiked with all elements at 5 µg/L (ppb).

To ensure the detection of low concentrations of trace elements in the presence of high concentrations of Li, Fe, and P, a salt blank solution was prepared that contained Li at 100 ppm, Fe at 1000 ppm, and P at 500 ppm. The salt solution was spiked at 5 ppb with a multi-element standard. The "salt" MDL data was converted to mg/kg, by applying the dilution factor of 500.

Table 2. Calibration range, correlation coefficient (R), background correction mode, and method detection limits of spiked blank and spiked LFP salt samples (calculated as 0.1 g sample in 50 mL solution) at selected wavelengths.

Element	Wavelength (nm)	Calibration Range, (mg/L)	Correlation Coefficient	Background Correction Mode	Clean MDL (µg/L)	Salt MDL (µg/L)	Salt MDL In Solid (mg/kg)	Sample Conc. (mg/kg)
Al	396.152	0.05–0.500	0.99996	Fitted	0.82	0.73	0.36	16.27
As	188.98	0.05–0.500	0.99992	Fitted	2.52	2.37	1.19	<MDL
B	182.577	0.05–0.500	0.99997	Fitted	0.67	0.89	0.44	0.76
Ba	455.403	0.05–0.500	0.99994	Fitted	0.09	0.03	0.02	0.15
Be	313.107	0.05–0.500	0.9999	Fitted	0.09	0.06	0.03	<MDL
Ca	396.847	0.05–0.500	0.99994	Fitted	0.15	0.10	0.05	24.88
Cd	228.802	0.05–0.500	0.99997	Fitted	0.35	0.57	0.28	<MDL
Co	228.615	0.05–0.500	0.99992	Fitted	0.57	1.50	0.75	4.92
Cr	267.716	0.05–0.500	0.99998	FACT	0.22	0.42	0.21	7.89
Cu	327.395	0.05–0.500	0.99999	Fitted	0.37	0.36	0.18	2.25
Fe-R	261.382	50–1000	0.99997	Fitted	11.51	NA	NA	364893
Hg	184.887	0.05–0.500	0.99996	Fitted	0.50	1.36	0.68	<MDL
K	766.491	0.05–0.500	0.99994	FACT	1.33	0.77	0.38	6.56
Li-R	670.783	5.0–100	0.99998	Fitted	5.89	NA	NA	41645
Mg	279.553	0.05–0.500	0.99994	Fitted	0.08	0.08	0.04	3.16
Mn	257.61	0.05–0.500	0.99993	Fitted	0.10	0.16	0.08	24.39
Mo	202.032	0.05–0.500	0.99993	Fitted	0.32	0.44	0.22	<MDL
Na	589.592	0.05–0.500	0.99999	FACT	0.29	0.18	0.09	11.11
Ni	231.604	0.05–0.500	0.99997	Fitted	0.43	0.61	0.31	8.00
P-R	213.618	25–500	1	Fitted	5.74	NA	NA	179665
Pb	220.353	0.05–0.500	0.99997	FACT	1.43	1.82	0.91	<MDL
S	180.669	0.05–0.500	1	Fitted	2.89	3.68	1.84	3.99
Sb	206.834	0.05–0.500	0.99995	FACT	2.93	3.30	1.65	<MDL
Se	196.026	0.05–0.500	0.99994	FACT	3.00	5.52	2.76	5.18
Si	251.611	0.05–0.500	0.99991	Fitted	1.19	2.40	1.20	49.32
Sn	189.925	0.05–0.500	0.99996	Fitted	0.87	0.81	0.40	2.63
Sr	407.771	0.05–0.500	0.99993	Fitted	0.11	0.04	0.02	2.77
Ti	334.941	0.05–0.500	0.99992	Fitted	0.08	0.10	0.05	3.03
V	311.837	0.05–0.500	0.99992	Fitted	0.72	0.98	0.49	<MDL
W	207.912	0.05–0.500	0.99993	Fitted	1.43	0.78	0.39	0.42
Zn	206.2	0.05–0.500	0.99996	FACT	0.16	2.44	1.22	16.12

Quantitative data and spike recoveries

The $LiFePO_4$ sample digests and spiked-samples were analyzed using the 5800 VDV ICP-OES. The average measured results and the respective spike recoveries for all 31 elements are shown in Table 3. The "in solid" sample results, which had the 500x dilution applied, are listed in the last column of Table 2.

The quantitative results show the ability of the 5800 method to measure both percentage (%) level concentrations and trace level contaminants all in the same analysis, using the same sample preparation and analytical conditions.

As shown in Table 3, the recoveries for the elements in the $LiFePO_4$ sample that was spiked before microwave digestion were ±10% from the expected value. The results demonstrate the effectiveness of the microwave digestion sample preparation procedure for the LFP cathode materials and confirm the accuracy of the 5800 ICP-OES method for the analysis of complex samples.

Spike recovery tests are an effective way to evaluate the reliability of the sample preparation method and/or the analytical method, especially when no suitable certified reference materials (CRMs) are available.

To confirm instrument-performance and absence of matrix effects, a lab fortified blank (LFB) was prepared by spiking the method blank at 100 mg/L (ppb) with all elements apart from Li, Fe, and P before microwave digestion. The recoveries for the elements in the LFB were ±10% from the expected value, as shown in Table 3, confirming the performance of the 5800 ICP-OES.

Table 3. Agilent 5800 VDV ICP-OES measured concentrations of elemental impurities in $LiFePO_4$ samples, and spike recovery data for $LiFePO_4$ samples, n=3.

Element and Wavelength nm	Sample Concentration µg/L	Concentration in Spiked Sample µg/L	Sample Spike Recovery %	Method Blank (MB) µg/L	Concentration of Lab Fortified Blank (LFB) µg/L	LFB Spike Recovery %
Al 396.152	33.3	142.3	109	0.00	97.4	97
As 188.980	<MDL	96.9	96	0.77	96.5	96
B 182.577	1.6	98.2	97	0.04	96.7	97
Ba 455.403	0.3	99.6	99	0.00	98.8	99
Be 313.107	<MDL	96.7	97	0.04	97.5	97
Ca 396.847	50.9	145.1	94	0.03	96.6	97
Cd 228.802	<MDL	97.9	98	0.00	97.5	98
Co 228.615	10.1	107.1	97	0.02	97.3	97
Cr 267.716	16.1	114.2	98	0.01	98.5	99
Cu 327.395	4.6	105.0	100	0.20	98.3	98
Fe-R 261.382	746570	-	-	5.00	-	-
Hg 184.887	<MDL	96.9	97	0.00	96.7	97
K 766.491	13.4	121.6	108	1.89	98.8	97
Li-R 670.783	85205	-	-	0.00	-	-
Mg 279.553	6.5	102.8	96	0.00	97.3	97
Mn 257.610	49.9	145.7	96	0.04	98.6	99
Mo 202.032	<MDL	98.3	98	0.00	99.3	99
Na 589.592	22.7	114.2	92	0.19	99.5	99
Ni 231.604	16.4	110.0	94	0.00	97.6	98
Pb 220.353	<MDL	96.2	95	0.00	98.3	98

Table 3 continues on next page

Table 3 continued...

P-R 213.618	367595	-	-	0.00	-	-
S 180.669	8.2	103.2	95	0.00	96.4	96
Sb 206.834	<MDL	95.2	95	0.07	98.0	98
Se 196.026	10.6	103.2	93	2.20	94.9	93
Si 251.611	100.9	194.8	94	0.00	99.7	100
Sn 189.925	5.4	101.9	97	0.39	98.7	98
Sr 407.771	5.7	103.8	98	0.02	97.0	97
Ti 334.941	6.2	105.6	99	0.22	98.7	99
V 311.837	<MDL	98.3	98	0.49	97.7	97
W 207.912	0.9	96.9	96	0.37	94.9	95
Zn 206.200	33.0	123.3	90.	0.10	96.8	97

Long-term stability (LTS)

To check the long-term validity of the calibration, 20 measurements of a CCV (0.1 mg/L) were completed over a nine-hour period without recalibration. The CCV was measured after every 10 measurements of the samples. The concentrations of the 20 CCV measurements were plotted against time, as shown in Figure 5. Accurate measurements within ±10% of the expected value (indicated by the dotted lines) were obtained.

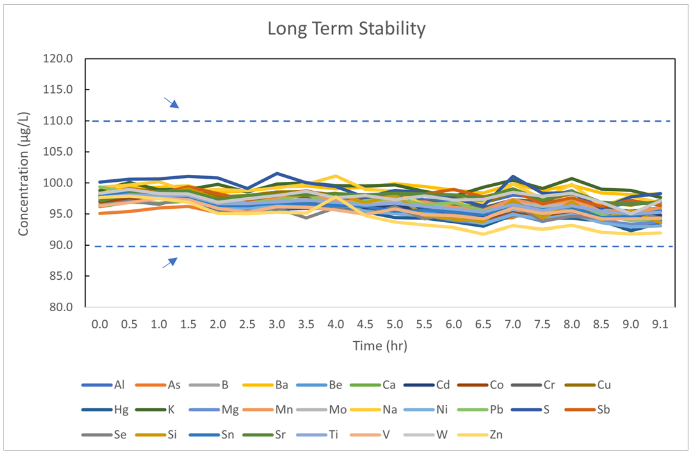


Figure 5. Long-term stability graph showing recoveries of the 100 µg/L CCV solution measured over 9-hours.

To further check the stability of the 5800 ICP-OES, the spiked (0.1 mg/L) $LiFePO_4$ sample was analyzed as a QC after every 10 samples. A total of 236 solutions were measured over nine hours without recalibration. The precision of the 20 measurements of the spiked $LiFePO_4$ sample was excellent over nine hours, with %RSDs below 2.2%, as shown in Table 4. The results show that the 5800 VDV ICP-OES can analyze high salt matrix samples with no instrument drift over a long run.

Table 4. Long-term stability of Agilent 5800 ICP-OES over 9 h. %RSD of measurements of $LiFePO_4$ samples spiked at 0.1 mg/L, n=20.

Element and Wavelength (nm)	RSD %	Element and Wavelength (nm)	RSD %
Al 396.152	0.6	Mo 202.032	1.0
As 188.980	1.3	Na 589.592	0.6
B 182.577	0.8	Ni 231.604	1.6
Ba 455.403	0.5	Pb 220.353	1.2
Be 313.107	1.3	P-R 213.618	1.5
Ca 396.847	0.7	S 180.669	1.3
Cd 228.802	1.2	Sb 206.834	2.0
Co 228.615	1.4	Se 196.026	2.1
Cr 267.716	0.7	Si 251.611	1.1
Cu 327.395	0.6	Sn 189.925	1.3
Fe-R 261.382	1.2	Sr 407.771	0.6
Hg 184.887	1.8	Ti 334.941	0.9
K 766.491	0.4	V 311.837	0.9
Li-R 670.783	1.0	W 207.912	1.1
Mg 279.553	1.5	Zn 206.200	2.1
Mn 257.610	0.9		

Conclusion

Lithium iron phosphate ($LiFePO_4$), also known as LFP, is a novel, cobalt-free cathode material, which is increasingly used in lithium ion batteries (LIBs). To test the elemental purity of an LFP cathode matrix, a high purity $LiFePO_4$ sample was prepared for analysis using microwave digestion.

The Agilent 5800 VDV ICP-OES was used to quantify 28 trace elements plus Li, Fe, and P in the sample digests. Excellent calibration linearity was obtained for major and minor elements over a wide linear working range and the method detection limits for most elements in the solid were below 1 mg/kg. Recoveries of all trace elements spiked in the $LiFePO_4$ sample before digestion were between 90 and 110%, confirming the accuracy of the sample preparation procedure and the ICP-OES method. The instrument was stable over 9 h, as shown by %RSDs of < 2.2% for all trace elements measured in the spiked LFP sample. The results confirmed the robustness of the 5800 ICP-OES for the routine analysis of high salt matrix samples.

The study has shown that the 5800 VDV ICP-OES can provide the accuracy, precision, and stability needed for the analysis of trace elements in new cathode materials.

References

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Agilent part numbers

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
1610132400	Y-piece connector for online addition of internal standard/ionization buffer
5190-9418	Quality Control Standard 27, multi-element calibration standard solution for 27 elements at 100 ppm
5190-8485	Agilent 1000 ppm single element stock solution for Hg
5190-8543	Agilent 1000 ppm single element stock solution for Sn
5190-8547	Agilent 1000 ppm single element stock solution for W
5190-8403	Agilent 10,000 ppm single element stock solution for Fe
5190-8409	Agilent 10,000 ppm single element stock solution for Li
5190-8429	Agilent 10,000 ppm single element stock solution for P
5190-8529	Agilent 1000 ppm single element stock solution for S
5190-8248	Agilent 1000 ppm single element stock solution for Ba
5190-8233	Agilent 10,000 ppm single element stock solution for Y
5190-8441	Agilent 10,000 ppm single element stock solution for Rb

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