

Robust Elemental Analysis of Sodium-Ion Battery Cathode Materials Using ICP-OES

Quality control of impurities in Prussian blue, sodium iron phosphate, and sodium manganese oxide by Agilent 5800 ICP-OES



Authors

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Introduction

Large-scale energy storage systems (ESS) and electric-powered vehicles (EVs) are vital technologies for advancing the transition from fossil fuels towards a more sustainable energy mix. Compared to other battery types, the relatively high specific capacity of lithium-ion batteries (LIBs) has led to their dominant use in these technologies. Given the huge growth potential of both these sectors, demand for LIBs is predicted to increase significantly over the next decade (1). However, the limited availability of lithium (Li) and expense of mining, extraction, and refining battery-grade Li-materials contribute to the high costs of the batteries (2). These factors have led to the development of alternative—or complimentary—battery technologies that can sustain the ever-growing demand for lower-cost battery-powered systems (3).

Sodium (Na) exhibits similar chemical properties to Li but is more abundant (23,600 ppm compared to 20 ppm in the earth's crust) and more accessible. It has therefore been of interest to battery researchers and developers for some time. Sodium-ion batteries (SIBs, also referred to as NIBs) offer a solution to many of the issues associated with LIBs, such as improved safety and reduced susceptibility to temperature changes (4-6). SIBs are considered a more cost-effective alternative to LIBs, in part due to the lower costs and environmental impact associated with the production of battery-grade Na and Na-containing cathode materials (7). SIBs can be stored and transported at 0 V, reducing shipping costs and safety concerns compared to LIBs, which require special handling (8). The performance of some SIBs compares well to lithium iron phosphate (LFP) cathode-LIBs, making them suitable for use in ESS and midrange EVs (9).

The working mechanisms of LIBs and SIBs are similar in that they both involve the movement of ions (Li $^+$ or Na $^+$) between the cathode and anode through an electrolyte. However, different materials are used for the main battery cell components. The capacity and redox potential of a SIB anode varies depending on the material used. Example anode materials include graphite, titanium oxide (TiO $_2$), antimony sulfide (Sb $_2$ S $_3$), tin phosphide (Sn $_4$ P $_3$), and phosphorus (P), among others (10).

The most common cathode materials currently used in SIBs include Layered Oxide Materials ($Na_\chi MO_2$), polyanionic compounds (phosphate-based) which are constructed by a tetrahedral XO_4 group with Na and a transition metal such as iron (Fe), vanadium (V), cobalt (Co), or manganese (Mn). Other materials include Metal-Organic compounds such as Prussian Blue Analogs (PBAs): $A_\chi MFe(CN)_6$ (A = Na K; M = Fe, Mn, Co, Ni, or Cu) (11-13). Although these cathode materials show promising electrochemical attributes, properties that are related to specific capacity, rate capability, and cycling stability can be affected by elemental impurities or contaminants.

As the SIB industry is still in its infancy, there are no set regulations or standards regarding the analysis of cathode materials, anode materials, or electrolytes. China has been working on "T/QGCML 306-2022", a group standard for the analysis of cathode material for sodium ion batteries, which requires the use of ICP-OES. Contaminating elements that are outlined in the method include calcium (Ca), magnesium (Mg), silicon (Si), zinc (Zn), and cadmium (Cd). None of these elements should exceed <0.1% of the total sample under investigation.

There are many other elements that should be analyzed in the cathode, anode, and electrolyte of SIBs. For example, elements such as arsenic (As) and lead (Pb) may negatively impact the performance of the battery as well as potentially contaminating the environment if discarded as waste rather than being recycled.

In this study, an Agilent 5800 Vertical Dual View (VDV) ICP-OES was used to measure a range of elements in three SIB cathode materials. The materials were digested using a single-step microwave digestion procedure before analysis by ICP-OES.

The 5800 VDV ICP-OES with Agilent ICP Expert Pro software (14) includes several smart tools to ensure a robust and reliable method for the analysis of precursor chemicals for SIB production. The following software tools were used to assist method development and ensure data quality throughout the run:

- IntelliQuant Screening is a rapid screening tool that analysts can use to assess the potential presence and approximate concentration of elements in the cathode materials, making it an ideal tool for method development (15). The data also helps the analyst to identify the best element wavelengths to use for the quantitative study.
- An IntelliQuant semiquantitative analysis can also be selected to run as part of the quantitative analysis workflow to check for any unexpected spectral interferences on the analytes (16). The data serves as a QC check on the samples, ensuring the accuracy of the results throughout the analysis.
- Easy-to-use automated algorithms for ICP-OES background correction including Fitted Background Correction (FBC) and Fast Automated Curve-fitting Technique (FACT) (17–19). FBC corrects simple and complex background peaks automatically, requiring no input from the analyst. FACT is used for highly complex backgrounds.

Experimental

Instrumentation

All data was collected using the 5800 VDV ICP-OES configured with an Agilent SPS 4 autosampler. The sample introduction system consisted of a SeaSpray glass concentric nebulizer, double-pass cyclonic spray chamber, and an Agilent Easy-fit semi-demountable VDV torch with a 1.8 mm inner diameter (ID) injector.

The 5800 (and Agilent 5900) ICP-OES instruments include a well-designed echelle polychromator with a low interior volume and temperature-regulated optics, which have been optimized to provide exceptional stability over long runs (20).

The instrument and method parameters shown in Table 1 were set up, optimized, and controlled using the ICP Expert Pro software. The instrument parameters were optimized for calibration linearity, sensitivity, and dynamic range while avoiding matrix effects e.g., from Easily Ionized Elements (EIEs) such as Li, Na, K, and Ca. EIE effects can cause either an enhancement or suppression of emission signals, leading to the reporting of either false high or false low element concentrations. Internal standards were used to correct for EIE effects in the plasma.

Table 1. Agilent 5800 VDV ICP-OES instrument and method parameters.

Parameter	Setting					
Viewing Mode	Axial	Radial				
Elements	Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Ga, K, Lu, Mg, Mo, Ni, P, Pb, S, Sb, Si, Sr, Ti, V, Zn, Zr	Na, Fe, Mn				
Viewing Height (mm)	-	8				
Read Time (s)	10					
Replicates	3					
Sample Uptake Delay (s)	15					
Stabilization Time (s)	10					
Pump Speed (rpm)	12					
Fast Pump	Enabled					
RF Power (kW)	1.2					
Auxiliary Flow (L/min)	1.2					
Plasma Flow (L/min)	13.5					
Nebulizer Flow (L/min)	0.7					
Sample Pump Tubing	White-white					
Waste Pump Tubing	Blue-blue					
Internal Standard Pump Tubing	Orange/Green					
Background Correction	FBC and FACT					

Samples

Three different SIB cathode materials were bought from NEI Corporation (Somerset, NJ, USA). The samples included a Prussian Blue ($NANOMYTE\ NAB-80:\ Na_{0.61}Fe[Fe(CN)_6]_{0.94}$), a Layered Oxide Material ($NANOMYTE\ NAB-35:\ Na_{0.7}MnO_2+_x$), and a polyanionic compound ($NANOMYTE\ NAB-60:\ NaFePO_4$).







Na_{0.61}Fe[Fe(CN)₆]_{0.94}

 $Na_{0.7}MnO_2+_x$

NaFePO,

Sample preparation

The SIB cathode materials were digested in aqua regia (AR) comprising a 3 to 1 molar ratio of concentrated hydrochloric acid (30% HCl) and concentrated nitric acid (69% HNO $_3$) bought from Merck, Pty. Ltd, Australia. 50 mg of each sample was weighed accurately to \pm 0.0001 g. Each sample was added to a dry clean microwave digestion vessel and thoroughly mixed with 5 mL of AR and 5 mL of Milli-Q water. The samples were digested at a temperature of 180 °C for 10 minutes using the BLADE Microwave Digestion System (CEM Corporation, Charlotte, NC, USA) per the program outlined in Table 2. The digested solutions were diluted with Milli-Q water to a final volume of 40 mL (800 dilution factor (DF)), producing a 12.5% (v/v) AR sample matrix solution. All samples were prepared in triplicate.

 Table 2. Microwave digestion parameters used for all cathode samples.

Parameter	Setting
Maximum Power (W)	1800
Temperature (°C)	180
Ramp Time (min)	2
Hold Time (min)	10

Spike solutions

To evaluate the accuracy of the method, sample digests of the three cathode materials were spiked post-digest with all elements at $250 \, \mu g/L$.

Calibration standards

A 100 mg/L stock solution of Agilent Multi-element Calibration Standard QC27 was used to prepare calibration curves for Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, K, Mg, Mo, Ni, Pb, Sb, Si, Sr, Ti, V, and Zn at 0.05, 0.01, 0.25, 0.5 and 5 mg/L. 10,000 mg/L single element stocks of both Fe and P were used to prepare calibration standards at 0.5, 5, 50 and 500 mg/L. 1,000 mg/L single element stock of S was used to prepare a calibration curve at 0.05, 0.5, 5 and 50 mg/L. 1,000 and 10,000 mg/L single element stocks for Na, Mn, La, Zr, and Ga were used to prepare calibration curves at 0.05, 0.5, 5, and 50 mg/L (La, Ga, Zr) or 0.5, 5, 50, and 500 mg/L (Na, Mn).

Quality control solutions

For quality control (QC) purposes, a 200 μ g/L standard solution (prepared separately from the calibration standard) was used as a continuing calibration verification (CCV) solution. A blank 12.5% AR solution was used as the continuing calibration blank (CCB). The CCB and CCV were analyzed after every 10 samples.

Internal standards

Internal standard (IS) solutions were prepared from Agilent single element standard solutions of yttrium (Y) and rubidium (Rb) at 20 mg/L in a 12.5% AR solution.

Method development

The IntelliQuant Screening feature of ICP-Expert Pro software was used to speed up method development by recommending the best analyte wavelengths to use for the analysis of SIB cathode material digests. The results also provided estimated concentrations of the elements in the samples, providing a useful overview of the elemental content of the material and helping determine the appropriate working calibration range for each analyte.

To aid interpretation of the semiquantitative data, the IntelliQuant Screening 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 the Prussian Blue sample shows that high concentrations of Na, Fe, La, Zr, and Ca are present (Figure 1). The analyst can use this information during method development to check for any spectral interferences that may arise from these elements. Also, knowing the approximate concentration of these elements can assist in preparing calibration curves within the accurate range.

Table 3. Background correction and calibration information.

Element and Wavelength (nm)	Background Correction	Calibration Range (mg/L)	Correlation Coefficient	
Ag 328.068	Fitted	0.05-5	1.0000	
Al 396.152	Fitted	0.05-5	1.0000	
As 193.696	Fitted	0.05-5	1.0000	
B 208.956	Fitted	0.05-5	1.0000	
Ba 455.403	Fitted	0.05-5	1.0000	
Be 234.861	Fitted	0.05-5	1.0000	
Ca 396.847	Fitted	0.05-5	0.9999	
Cd 214.439	Fitted	0.05-5	1.0000	
Co 228.615	Fitted	0.05-5	1.0000	
Cr 267.716	Fitted	0.05-5	1.0000	
Cu 327.395	Fitted	0.05-5	1.0000	
Fe 238.204	Fitted	0.5-500	0.9999	
Ga 417.204	FACT	0.05-50	1.0000	
K 766.491	FACT	0.05-5	1.0000	
La 408.671	Fitted	0.05-50	1.0000	
Mg 279.553	Fitted	0.05-5	1.0000	
Mn 257.610	Fitted	0.5-500	1.0000	
Mo 202.032	Fitted	0.05-5	1.0000	
Na 588.995	Fitted	0.5-500	0.9999	
Ni 231.604	Fitted	0.05-5	1.0000	
P 213.618	FACT	0.5-500	0.9999	
Pb 220.353	Fitted	0.05-5	1.0000	
S 180.669	Fitted	0.05-50	1.0000	
Sb 217.582	Fitted	0.05-5	1.0000	
Si 251.611	Fitted	0.05-5	1.0000	
Sr 407.771	Fitted	0.05-5	0.9999	
Ti 336.122	Fitted	0.05-5	1.0000	
V 309.310	Fitted	0.05-5	1.0000	
Zn 202.548	Fitted	0.05-5	1.0000	
Zr 343.823	Fitted	0.05-50	0.9999	

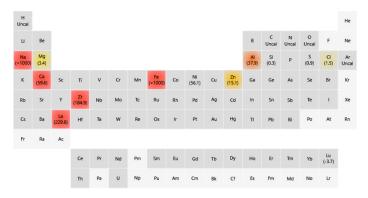
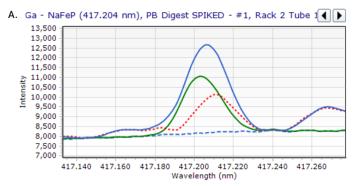


Figure 1. IntelliQuant Screening periodic table 'heat map' view showing which elements are present in the Prussian Blue sample. All values are semiquantitative and are reported in ppm.

Background correction

The ICP Expert software includes smart background correction techniques including Fitted Background Correction (FBC) and Fast Automated Curve-fitting Technique (FACT). 18, 19 For elements where there were no direct overlaps, FBC was used. FACT was used when interferences were closer to, or overlapped, the analyte of interest.

An example of a FACT model for the correction of the Fe interference on Ga 417.204 nm is shown in Figure 2. FACT subtracts the contribution to the analyte signal from the interference and the blank, providing an accurate result for Ga (green line).



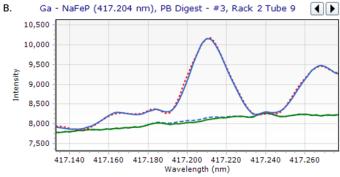


Figure 2. FACT model for correction of a Fe interference on Ga (total signal shown by the solid blue line). The Fe (red dashed) interference line is overlapping the Ga 417.204 nm (solid green) analyte line. The signal from the blank is shown by the light blue dashed line. **A:** A Prussian Blue sample spiked with 250 μ g/L Ga. **B:** An unspiked sample of Prussian Blue containing no Ga, showing the signal for Fe.

Results and discussion

Calibration

Representative calibration curves for As, Cd, Co (0.05 to 5 mg/L), and P (0.5 to 500 mg/L) are shown in Figure 3. Calibration curves for all elements were linear with a correlation coefficient greater than 0.999 (Table 3) and less than 10% calibration error on each calibration point.

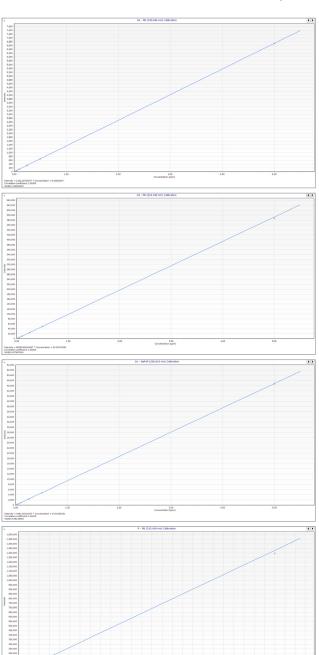


Figure 3. Calibration curves for As, Cd, Co, and P.

Method Detection Limits (MDLs)

To measure MDLs of low concentrations of trace elements in the presence of high concentrations of Na, Mn, Fe, and P, DLs were determined in the three cathode materials spiked with 10 μ g/L of each element. DLs were also determined in the method blank (12.5% AR). This method was found to be more representative of the complexity of real-life samples than using a method blank matrix only spiked with representative elements. The MDLs reported in Table 4 were calculated based on 3.14 x standard deviation (SD) of three measurements of the trace elements in each of the spiked samples. The MDLs for the primary elements (Na, Mn, Fe, and P) were calculated using the 12.5% AR acid solution spiked at 10 μ g/L.

Table 4. MDLs for impurity elements in SIB cathode materials and for Na, Mn, Fe, and P in 12.5% AR. All elements were spiked at 10 μ g/L. Units: μ g/L (ppb).

Element and	SIB Cathode Materials								
Wavelength	12.5% AR Prussian Blue Sodium Iron Sodium								
(nm)	Blank	i raddian Biae	Phosphate						
	MDL	MDL	MDL	MDL					
Ag 328.068	1.00	1.00	0.5	5.00					
Al 396.152	0.7	1.3	10.0	1.00					
As 193.696	5.00	6.00	7.00	13.0					
B 208.956	1.00	1.00	1.00	1.00					
Ba 455.403	0.10	0.10	0.10	0.10					
Be 234.861	0.10	0.40	0.30	0.10					
Ca 396.847	0.30	3.10	4.90	2.10					
Cd 214.439	0.10	1.60	0.40	0.20					
Co 228.615	1.00	1.00	1.00	1.00					
Cr 267.716	0.40	0.40	2.30	1.50					
Cu 327.395	0.50	0.40 0.50		0.50					
Fe 238.204	1.00	NA	NA	3.00					
Ga 417.204	3.00	5.00	4.00	4.00					
K 766.491	2.00	16.0	3.00	2.00					
La 408.671	0.40	10.8	0.50	0.30					
Mg 279.553	0.10	0.10	0.40	0.20					
Mn 257.610	1.00	1.00	7.00	NA					
Mo 202.032	0.50	0.40	0.60	0.70					
Na 588.995	20.0	NA	NA	NA					
Ni 231.604	1.00	1.00	2.00	1.00					
P 213.618	14.0	14.0	NA	16.0					
Pb 220.353	2.00	2.00	3.00	2.00					
S 180.669	4.00	4.00	11.0	12.0					
Sb 217.582	3.00	3.00							
Si 251.611	1.00	4.00	3.00	5.00					
Sr 407.771	0.20	0.10	0.10	0.10					
Ti 336.122	0.20	0.30	0.50	0.30					
V 309.310	0.20	0.30	0.20	1.60					
Zn 202.548	0.20	0.60	2.40	0.70					
Zr 343.823	0.30	2.60	9.60	1.10					

Quantitative data and spike recoveries

In the absence of suitable certified reference materials (CRMs) for the application, spike recovery tests were used to check the accuracy of the method. Prussian Blue (PB), Sodium Iron Phosphate (SIP or NIP), Sodium Manganese Oxide (SMO or NMO), and a method blank were spiked with 250 mg/L. Each spike solution was prepared in triplicate and each digest was also analyzed in triplicate.

Recoveries for all reported analytes fell within 100 \pm 10% of the expected value, as shown in Table 5. No recovery results were obtained for Na in all three materials, Fe in the PB and SIP samples, Mn in SMO, and P in SIP. These elements were present in the sample at a much higher concentration than the 250 mg/L spike level. The excellent spike recovery data confirms the suitability of the 5800 VDV ICP-OES method for the accurate analysis of trace elemental impurities in all three SIB cathode materials.

Stability test

If an analytical instrument can remain stable over long runs, there is less need to recalibrate, which improves the productivity and cost-effectiveness of the method. To check the stability of the 5800 VDV ICP-OES, all elements were measured in actual sample digests. The sample digests, which contained 0.250 mg/L (250 mg/L) of all analyte elements, were measured throughout the 10 hour run against the same calibration curve. Overall, 410 measurements were completed over 10 h at an average rate of 87 s per sample, without recalibration. The sequence included 260 measurements of spiked and unspiked SIB cathode materials, two CCV solutions (at 100 and 200 $\mu g/L$), and finally a CCB solution of 12.5% AR.

The concentrations of 32 of the CCV measurements were normalized as percentage recoveries of the expected value (200 $\mu g/L$) and plotted against time (Figure 4). Accurate measurements within ±10% of the expected value (indicated by the dotted lines) were obtained, with %RSDs for all wavelengths below 3%. The excellent QC stability shows the robustness of the 5800 ICP-OES for routine analysis of all target analytes in the three SIB cathode materials.

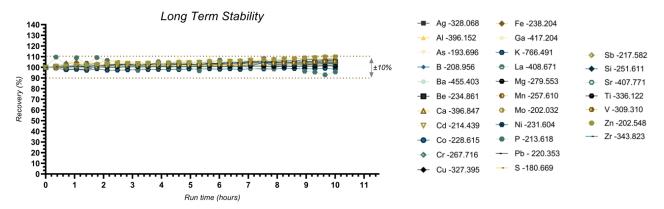


Figure 4. Long-term stability showing recoveries of the 200 μ g/L CCV solution measured over 10 hours.

Table 5. Quantitative results for the three SIB cathode materials of Prussian Blue (PB), Sodium Iron Phosphate (SIP), and Sodium Manganese Oxide (SMO) at 600x dilution using the Agilent 5800 VDV ICP-OES and 250 μg/L spike recovery data.

Element and Blank			Prussian Blue			Sodium Iron Phosphate			Sodium Manganese Oxide			
Wavelength (nm)	Sample Concentration µg/L	Concentration in Spiked Sample		Sample Concentration µg/L	Concentration in Spiked Sample	Spike Recovery %	Sample Concentration µg/L	Concentration in Spiked Sample	Spike Recovery %	Sample Concentration µg/L	Concentration in Spiked Sample	Spike Recovery %
Ag 328.068	8.1	255	99	0.6	248	99	0.8	248	99	38.9	298	104
Al 396.152	9.7	266	103	27.8	291	105	70.6	336	106	31.5	298	107
As 193.696	<mdl< td=""><td>256</td><td>102</td><td><mdl< td=""><td>248</td><td>99</td><td><mdl< td=""><td>247</td><td>99</td><td><mdl< td=""><td>246</td><td>98</td></mdl<></td></mdl<></td></mdl<></td></mdl<>	256	102	<mdl< td=""><td>248</td><td>99</td><td><mdl< td=""><td>247</td><td>99</td><td><mdl< td=""><td>246</td><td>98</td></mdl<></td></mdl<></td></mdl<>	248	99	<mdl< td=""><td>247</td><td>99</td><td><mdl< td=""><td>246</td><td>98</td></mdl<></td></mdl<>	247	99	<mdl< td=""><td>246</td><td>98</td></mdl<>	246	98
B 208.956	<mdl< td=""><td>274</td><td>110</td><td><mdl< td=""><td>265</td><td>106</td><td><mdl< td=""><td>267</td><td>107</td><td>14.4</td><td>278</td><td>105</td></mdl<></td></mdl<></td></mdl<>	274	110	<mdl< td=""><td>265</td><td>106</td><td><mdl< td=""><td>267</td><td>107</td><td>14.4</td><td>278</td><td>105</td></mdl<></td></mdl<>	265	106	<mdl< td=""><td>267</td><td>107</td><td>14.4</td><td>278</td><td>105</td></mdl<>	267	107	14.4	278	105
Ba 455.403	<mdl< td=""><td>248</td><td>99</td><td><mdl< td=""><td>238</td><td>95</td><td><mdl< td=""><td>237</td><td>95</td><td>4.4</td><td>239</td><td>94</td></mdl<></td></mdl<></td></mdl<>	248	99	<mdl< td=""><td>238</td><td>95</td><td><mdl< td=""><td>237</td><td>95</td><td>4.4</td><td>239</td><td>94</td></mdl<></td></mdl<>	238	95	<mdl< td=""><td>237</td><td>95</td><td>4.4</td><td>239</td><td>94</td></mdl<>	237	95	4.4	239	94
Be 234.861	<mdl< td=""><td>260</td><td>104</td><td><mdl< td=""><td>241</td><td>96</td><td><mdl< td=""><td>244</td><td>98</td><td><mdl< td=""><td>248</td><td>99</td></mdl<></td></mdl<></td></mdl<></td></mdl<>	260	104	<mdl< td=""><td>241</td><td>96</td><td><mdl< td=""><td>244</td><td>98</td><td><mdl< td=""><td>248</td><td>99</td></mdl<></td></mdl<></td></mdl<>	241	96	<mdl< td=""><td>244</td><td>98</td><td><mdl< td=""><td>248</td><td>99</td></mdl<></td></mdl<>	244	98	<mdl< td=""><td>248</td><td>99</td></mdl<>	248	99
Ca 396.847	75	336	105	98	347	100	145	402	103	142	386	98
Cd 214.439	<mdl< td=""><td>255</td><td>102</td><td>5.3</td><td>251</td><td>98</td><td>5.6</td><td>250</td><td>98</td><td><mdl< td=""><td>235</td><td>94</td></mdl<></td></mdl<>	255	102	5.3	251	98	5.6	250	98	<mdl< td=""><td>235</td><td>94</td></mdl<>	235	94
Co 228.615	<mdl< td=""><td>251</td><td>100</td><td>4.3</td><td>245</td><td>96</td><td>9.4</td><td>249</td><td>96</td><td>76</td><td>304</td><td>91</td></mdl<>	251	100	4.3	245	96	9.4	249	96	76	304	91
Cr 267.716	<mdl< td=""><td>247</td><td>99</td><td>1.2</td><td>237</td><td>94</td><td>38</td><td>274</td><td>94</td><td>64</td><td>310</td><td>98</td></mdl<>	247	99	1.2	237	94	38	274	94	64	310	98
Cu 327.395	<mdl< td=""><td>244</td><td>98</td><td>5.2</td><td>248</td><td>97</td><td>5.5</td><td>249</td><td>97</td><td>10.5</td><td>251</td><td>96</td></mdl<>	244	98	5.2	248	97	5.5	249	97	10.5	251	96
Fe 238.204	5.5	262	103	365000	NA	NA	387000	NA	NA	174.6	412	95
Ga 417.204	1	275	109	145	419	110	2.3	275	109	14	268	102
K 766.491	19	258	96	584	846	105	48	314	107	37	301	106
La 408.671	<mdl< td=""><td>259</td><td>104</td><td>410</td><td>666</td><td>102</td><td>1.0</td><td>252</td><td>100</td><td>1.7</td><td>254</td><td>101</td></mdl<>	259	104	410	666	102	1.0	252	100	1.7	254	101
Mg 279.553	3.0	268	106	6.4	253	99	22	270	99	21	262	96
Mn 257.610	5	254	100	56	302	98	612	866	102	503820	NA	NA
Mo 202.032	<mdl< td=""><td>247</td><td>99</td><td>0.4</td><td>239</td><td>95</td><td>1.4</td><td>240</td><td>95</td><td><mdl< td=""><td>236</td><td>94</td></mdl<></td></mdl<>	247	99	0.4	239	95	1.4	240	95	<mdl< td=""><td>236</td><td>94</td></mdl<>	236	94
Na 588.995	114	349	94	112000	NA	NA	187000	NA	NA	184000	NA	NA
Ni 231.604	<mdl< td=""><td>254</td><td>102</td><td>30</td><td>268</td><td>95</td><td>117</td><td>356</td><td>96</td><td>10</td><td>240</td><td>92</td></mdl<>	254	102	30	268	95	117	356	96	10	240	92
P 213.618	<mdl< td=""><td>256</td><td>102</td><td>57</td><td>320</td><td>105</td><td>237345</td><td>NA</td><td>NA</td><td>98</td><td>352</td><td>102</td></mdl<>	256	102	57	320	105	237345	NA	NA	98	352	102
Pb 220.353	<mdl< td=""><td>251</td><td>100</td><td>9</td><td>249</td><td>96</td><td>10</td><td>247</td><td>95</td><td>68</td><td>297</td><td>91</td></mdl<>	251	100	9	249	96	10	247	95	68	297	91
S 180.669	5.0	262	103	15	269	102	334	587	101	1100	1354	102
Sb 217.582	2.3	259	103	1.1	236	94	1.4	236	94	11	254	97
Si 251.611	266	515	100	245	490	98	252	501	100	406	650	98
Sr 407.771	0.2	251	100	0.2	239	96	0.3	238	95	0.6	235	94
Ti 336.122	0.3	246	98	5.4	246	96	19	258	96	20	257	95
V 309.310	<mdl< td=""><td>244</td><td>98</td><td><mdl< td=""><td>237</td><td>95</td><td>1.5</td><td>236</td><td>94</td><td>2.3</td><td>231</td><td>91</td></mdl<></td></mdl<>	244	98	<mdl< td=""><td>237</td><td>95</td><td>1.5</td><td>236</td><td>94</td><td>2.3</td><td>231</td><td>91</td></mdl<>	237	95	1.5	236	94	2.3	231	91
Zn 202.548	138	397	103	149	399	100	119	368	100	99	339	96
Zr 343.823	<mdl< td=""><td>255</td><td>102</td><td>217</td><td>474</td><td>103</td><td>358</td><td>596</td><td>95</td><td>24</td><td>277</td><td>101</td></mdl<>	255	102	217	474	103	358	596	95	24	277	101

In run QC using IntelliQuant

IntelliQuant was used as an extra in-run QC step throughout the quantitative analysis of the cathode samples (Figure 5). Acquiring IntelliQuant data provides extra confidence and assurance that there are no unexpected elements in the samples at high enough concentrations to impede the

accurate analysis of the analyte wavelengths selected for the quantitative method. The semiquantitative results generated by IntelliQuant are often within about ±20% of the quantitative values, which may be sufficient to flag a production or manufacturing problem or contamination issue.

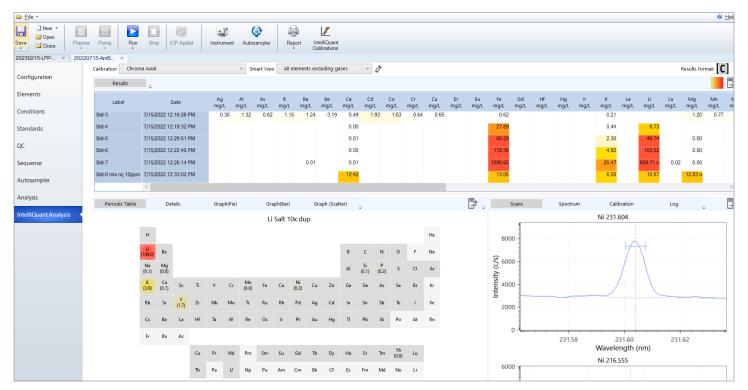


Figure 5. Screenshot showing an in-run IntelliQuant QC report.

Top: The concentration thresholds in the table can be adjusted.

Bottom left: The periodic table heat map shows relative concentrations of the elements in a sample.

Bottom right: The "scans" view can be used to check the signal for individual analytes and to check for any potential interferants.

Conclusion

The accurate measurement of SIB cathode materials, Prussian Blue (PB), Sodium Iron Phosphate (SIP), and Sodium Manganese Oxide (SMO), was carried out using the Agilent 5800 VDV ICP-OES with Agilent SPS 4 autosampler.

The smart tools included in the Agilent ICP Expert Pro software added an extra layer of quality assurance to the analysis. The IntelliQuant Screening and IntelliQuant features assisted with method development and result validation, respectively, reducing the need to re-analyze any samples, and increasing confidence in the quantitative results. FBC automatically corrected the background structures that arise when analyzing highly complex samples for most elements and FACT deconvoluted analyte peaks from more complex spectra to ensure accurate results for Ga, K, and P.

Excellent calibration linearity and low detection limits were obtained for all 30 analyte elements. The high performance of the $5800 \, \text{VDV}$ ICP-OES was demonstrated by the recovery data for all three type samples. All recoveries were within $100 \pm 10\%$, showing that the $5800 \, \text{can}$ tolerate the varied high matrix samples. The $5800 \, \text{also}$ maintained excellent stability over the 10-hour QC recovery test, demonstrating the suitability of the method for the routine measurement of SIB precursor materials.

By avoiding matrix effects and prioritizing the highest precision and accuracy over the highest sensitivity, the study demonstrated the suitability of the 5800 VDV ICP-OES for the multi-element analysis of SIB cathode materials.

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