Challenge
Capability for a robust and reliable determination of trace level impurities (ppb) up to high concentration elements (up to %).

Solution
PlasmaQuant 9x00 Elite ICP-OES with high-resolution spectrometer and Dual View PLUS system.

Intended audience
Battery manufacturers and contract labs.

Determination of The Elemental Composition in Al-Air Battery Materials

Introduction

The performance and longevity of aluminium-air (Al-air) batteries are highly dependent on the purity of the aluminium anode. Impurities such as iron, silicon, and copper can significantly impact battery efficiency by promoting corrosion and passivation, which hinder electrochemical reactions [1]. To ensure optimal battery performance, high-purity aluminium (≥99.99% or 4N) is often recommended, as lower purity levels can lead to the formation of unwanted surface layers that degrade functionality [2].

Accurately determining the elemental composition of battery materials is therefore essential for ensuring quality and optimizing performance. However, aluminium alloys contain a wide range of elements, from major components like iron (Fe) and silicon (Si) to trace impurities such as lead (Pb), nickel (Ni), and zinc (Zn), with concentrations varying from percentage levels to parts per billion. This diversity presents analytical challenges, requiring a technique with high sensitivity, a broad dynamic range, and the ability to reliably differentiate trace elements from high-concentration matrix elements while minimizing spectral interferences.

Optical emission spectroscopy with inductively coupled plasma (ICP-OES) is a highly effective solution for this demanding task. The PlasmaQuant ICP-OES series delivers high spectral resolution (2 pm @ 200 nm) and advanced interference correction through its Correction of Spectral Interferences (CSI) software tool. Additionally, its Dual View PLUS technology enables optimized axial and radial plasma observation, enhancing sensitivity for trace elements while ensuring an extended dynamic range for high-concentration elements. These capabilities make the PlasmaQuant ICP-OES a powerful tool for precise and reliable elemental analysis of Al-air battery materials.

Materials and Methods

Samples and reagents

Reagents

- Nitric acid (Merck Suprapur®)
- Hydrochloric acid (Merck Suprapur®)
- Milli-Q® water
- NIST traceable standards (Inorganic ventures)

Samples

- KOH electrolyte (“KOH 01” & “KOH 02”)
- Aluminium residue (“Al res.”)

Sample preparation

The KOH electrolyte samples were diluted by weight, depending on the calibrated working range for the elements of interest. The aluminium residue was prepared by digesting a representative amount (0.5-5 g), depending on the targeted dilution and analyte concentration range, with aqua regia on a hot plate at 180 °C. Subsequently, it was filled up to a target volume with Milli-Q® water.

Instrumentation

The analysis was performed on the PlasmaQuant 9100 Elite, using the salt kit and ceramic torch parts. Representative test measurments on a PlasmaQuant 9200 Elite confirmed consistent performance under the adjusted plasma conditions. The detailed system configurations are provided in Table 1. In tables 2 and 3 the method parameters for each matrix are summarized.

Table 1: Instrumental Parameters

Parameter	Specification PlasamQuant 9100 Elite	Specification PlasamQuant 9200 Elite
Plasma power	1400 W (“KOH”) / 1350 W (“Al res.”)	1400 W („KOH“) / 1350W („Al res.“)
Plasma gas flow	15 L/min	10 L/min
Auxiliary gas flow	0.5 L/min	0.8 L/min
Nebulizer gas flow	0.6 L/min	0.6 L/min
Nebulizer	Concentric nebulizer for high salt content, borosilicate glass, 2 mL/min	Concentric nebulizer for high salt content, borosilicate glass, 2 mL/min
Spray chamber	cyclonic, 50 mL with dip tube, borosilicate glass	cyclonic, 50mL with dip tube, borosilicate glass
Outer tube / inner tube	Ceramic/ceramic*	Ceramic/ceramic*
Injector	Ceramic, 2 mm id*	Ceramic, 2 mm id*
Pump tubing	Sample: PVC (black/black); waste: PVC (red/red)	Sample: PVC (black/black); waste: PVC (red/red)
Pump rate	1.0 mL/min	1.0 mL/min
Fast pump	4.0 mL/min	4.0 mL/min
Delay time / rinse time	20 s/ 35 s	20 s/ 35 s
Torch position	0 mm	0 mm

* The salt kit used is equipped with quartz glass parts by default. As these parts can wear out quickly in the presence of high salt matrix load, the use of ceramic components can be beneficial for long-term operation.

Table 2: Method Settings and Evaluation Parameters – „KOH“

Element	Wavelength [nm]	Plasma view	Integration mode	Read time [s]	Evaluation		
					No. of pixels	Baseline fit	Poly. deg.
As	189.979	axial	spectrum	3.0	3	ABC	auto
Be	234.861	axial	spectrum	3.0	3	ABC	auto
Bi	223.061	axial	spectrum	3.0	3	ABC	auto
Cd	214.441	axial	spectrum	3.0	3	ABC	auto
Cr	284.325	axial	spectrum	3.0	3	ABC	auto

Element	Wavelength [nm]	Plasma view	Integration mode	Read time [s]	Evaluation		
					No. of pixels	Baseline fit	Poly. deg.
Cu	213.598	axial	spectrum	3.0	3	ABC	auto
Fe	238.204	axial	spectrum	3.0	3	ABC	auto
Na	589.592	atten. radial	spectrum	3.0	3	ABC	auto
Ni	221.648	axial	spectrum	3.0	3	ABC	auto
Pb	220.353	axial	spectrum	3.0	3	ABC	auto
Sb	217.581	axial	spectrum	3.0	3	ABC	auto
Se	196.028	axial	spectrum	3.0	3	ABC	auto
Sn	189.927	axial	spectrum	3.0	3	ABC	auto
V	309.311	axial	spectrum	3.0	3	ABC	auto
Zn	206.200	axial	spectrum	3.0	3	ABC	auto

ABC: Automatic Baseline Correction

Table 3: Method Settings and Evaluation Parameters – „Al res."

Element	Wavelength [nm]	Plasma view	Integration mode	Read time [s]	Evaluation		
					No. of pixels	Baseline fit	Poly. deg.
Al	396.152	atten. radial	spectrum	3.0	3	ABC	auto
Fe	259.940	atten. axial	spectrum	3.0	3	ABC	auto
K	766.491	atten. radial	spectrum	3.0	3	ABC	auto
Mg	279.078	atten. radial	spectrum	3.0	3	ABC	auto
Sn	189.927	radial	spectrum	3.0	3	ABC	auto

ABC: Automatic Baseline Correction

Calibration

For the KOH samples a matrix-matched calibration was performed by standard addition procedure. The digested aluminium residue was analyzed by using an aqueous standard calibration. Multi-element stock solutions of 1000 mg/kg and 10,000 mg/kg were used for preparation of the calibration standards and sample additions by appropriate dilution. The concentrations of standards for both calibration strategies are listed in table 4. The obtained calibration plots and corresponding coefficients of determination (R^2(adj.)) are displayed in figure 1 and 2.

Table 4: Standard concentrations

Sample "KOH"			Sample "Al res."					
Standard	As, Be, Bi, Cd, Cr, Cu, Fe, Ni, Pb, Sb, Se, Sn, V, Zn [µg/L]	Na [mg/L]	Standard	Al [mg/kg]	Fe [mg/kg]	K [mg/kg]	Mg [mg/kg]	Sn [mg/kg]
Cal. 0	0	0	Cal. 0	0	0	0	0	0
Std.Add. 1	5	10	Std. 1	20	0.1	0.5	2	0.5
Std.Add. 2	10	20	Std. 2	50	0.2	1	5	1
Std.Add. 3	25	30	Std. 3	100	0.5	2	10	2
Std.Add. 4	50	40	Std. 4	150	1	3	25	5

Sample "KOH"			Sample "Al res."					
Standard	As, Be, Bi, Cd, Cr, Cu, Fe, Ni, Pb, Sb, Se, Sn, V, Zn [µg/L]	Na [mg/L]	Standard	Al [mg/kg]	Fe [mg/kg]	K [mg/kg]	Mg [mg/kg]	Sn [mg/kg]
Std.Add. 5	100	50	Std. 5	200	2	4	50	10
Std.Add. 6	150	-	Std. 6	-	5	5	100	20
Std.Add. 7	200	-	-	-	-	-	-	-

ABC: Automatic Baseline Correction

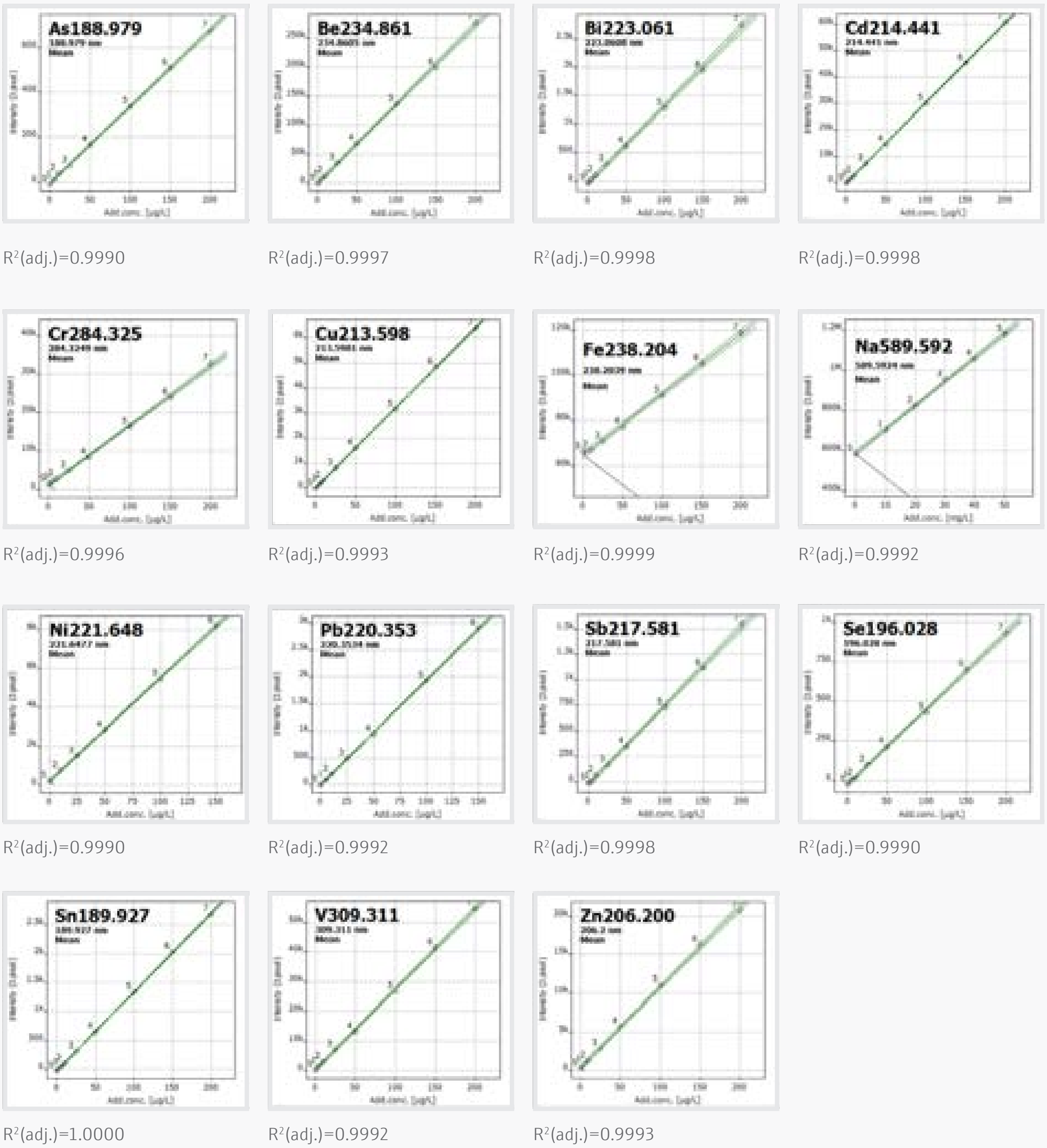
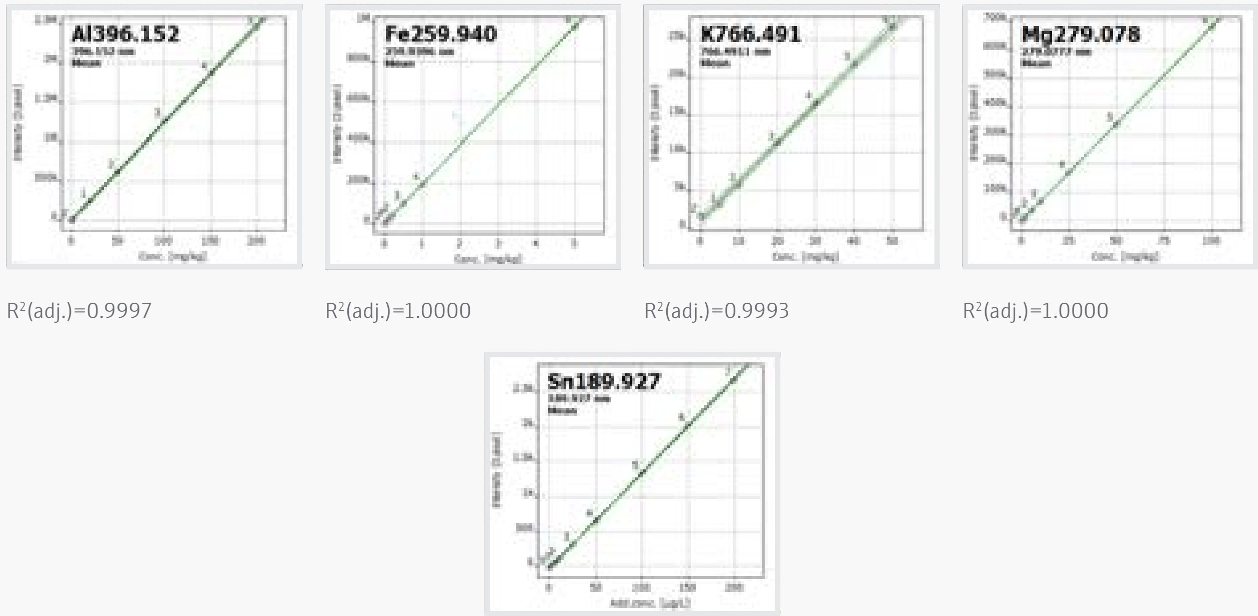


Figure 1: Calibration functions – "KOH"



R^2(adj.)=0.9997 R^2(adj.)=1.0000 R^2(adj.)=0.9993 R^2(adj.)=1.0000

R^2(adj.)=0.9998

Figure 2: Calibration functions – “Al res.”

Results and Discussion

The following table 5 shows the measurement results for both analyzed sample matrices. To check for non-spectral interferences, the KOH solutions were additionally spiked with known concentrations of the analytes (“QC spike”: 20 µg/kg, and 30 mg/kg for Na). The recovery of this QC spike experiment is listed as well.
The argon line at 420.068 nm was used as an internal standard to monitor the plasma conditions during the analysis. Figure 3 shows the recovery of the argon intensity during the analysis. Examples for analyte spectra in the observed sample solutions are shown in table 6.

Table 5: Measurement results and QC spike recovery

Element	Sample “KOH 01”		Sample “KOH 02”		Sample “Al res.”
	Meas. value [mg/kg]	QC spike recovery [%]	Meas. value [mg/kg]	QC spike recovery [%]	Meas. value [mg/kg]
Al	-	-	-	-	89,030
As	n.d.	92.0	n.d.	92.8	-
Be	n.d.	106	N.D.	102	-
Bi	n.d.	87.6	0.002	84.9	-
Cd	n.d.	101	n.d.	94.1	-
Cr	0.030	103	n.d.	102	-
Cu	n.d.	102	n.d.	101	-
Fe	0.972	-	0.071	98.9	n.d.
K	-	-	-	-	179,900
Mg	-	-	-	-	1,890

Element	Sample "KOH 01"		Sample "KOH 02"		Sample "Al res."
	Meas. value [mg/kg]	QC spike recovery [%]	Meas. value [mg/kg]	QC spike recovery [%]	Meas. value [mg/kg]
Na	1 144	105	1 962	91.2	-
Ni	0.016	110	0.126	104	-
Pb	0.015	103	n.d.	102	-
Sb	n.d.	80.4	n.d.	95.8	-
Se	n.d.	95.3	n.d.	98.6	-
Sn	n.d.	99.6	n.d.	96.5	352.4
V	0.004	108	0.010	100	-
Zn	0.014	105	n.d.	104	-

n.d.: not detected (<LOD)

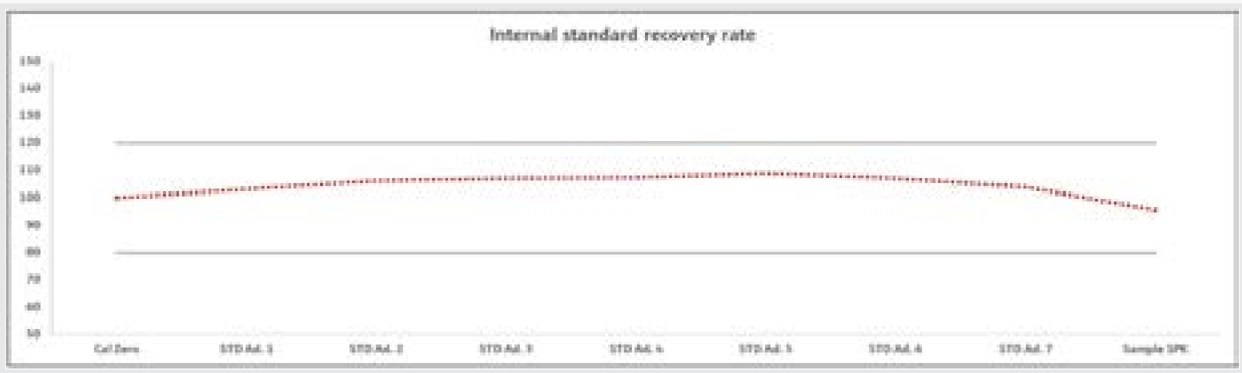
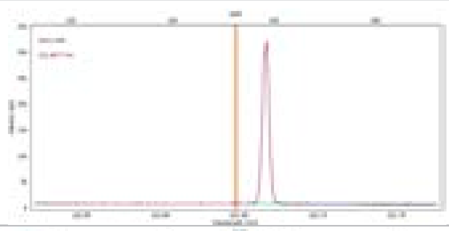
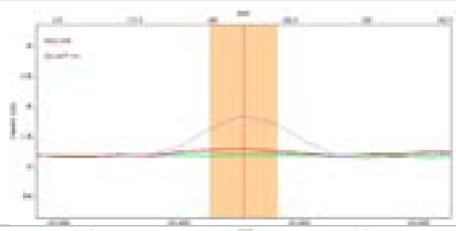
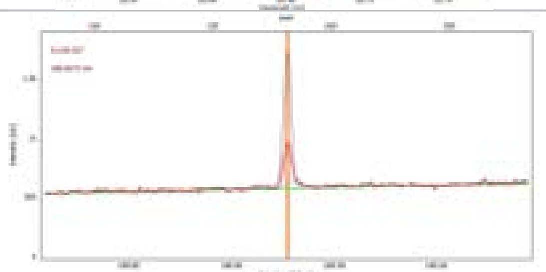
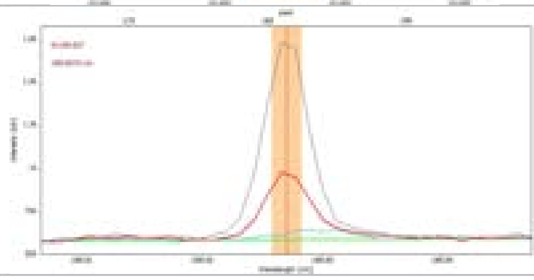
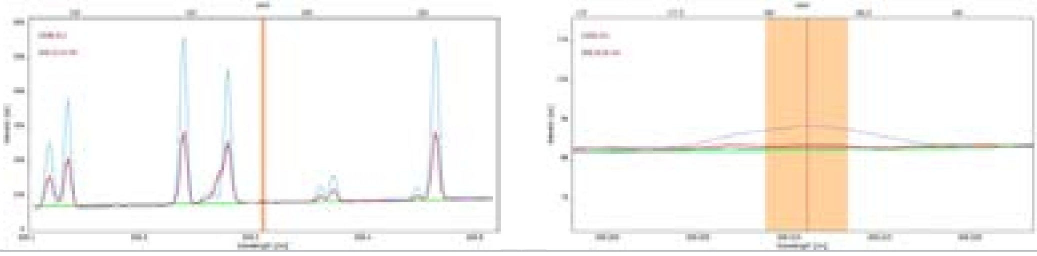


Figure 3: Calibration functions – "Al res."

Table 6: Examples for analyte spectra (left: original, right: magnification)

Element	Spectra	
Ni 221 nm --- Blank --- KOH 02 --- KOH 02 + 5 µg/L Ni -- Base line (ABC)		
Sn 189 nm --- Blank --- Al. res. --- Cal. std. 1 --- Base line (ABC)		

Element	Spectra
V 309 nm --- Blank --- KOH 01 --- KOH 01 + 5 µg/L Ni --- Base line (ABC)	

Summary

The sample analysis study confirms the suitability of the PlasmaQuant 9x00 Elite for determining the elemental composition of Al-air battery materials. The combination of Dual View PLUS technology and high spectral resolution allows for precise quantification of element concentrations ranging from µg/L to percentage levels within a single measurement run. Additionally, the robust plasma generation of the PlasmaQuant 9x00 series ensures reliable analysis across a wide range of complex sample matrices.

Figure 4: PlasmaQuant 9200 Elite

Recommended device configuration

Table 7: Overview of devices, accessories, and consumables

Article	Article number	Description
PlasmaQuant 9x00 Elite	818-09101-2	High resolution ICP-OES
Teledyne Cetac ASX 560	810-88015-0	Teledyne-Cetac ASX-560 autosampler for ICP-OES and ICP-MS
Salt kit	810-88009-0	SALT KIT for PlasmaQuant 9x00 series
Consumable set Salt Kit	810-88046-0	Consumables Set Salt Kit for PlasmaQuant 9x00 series
Outer tube ceramic	418-13-410-491	Outer tube (Syalon) for V Shuttle Torch
Inner tube ceramic	418-13-410-492	Inner tube (Alumina ceramic) for V Shuttle Torch
Injector ceramic	418-13-410-412	Injector 2.0mm, Alumina for V Shuttle Torch

References

[1] Wikipedia - Aluminium–air battery (https://en.wikipedia.org/wiki/Aluminium%E2%80%93air_battery)

[2] Jianming Ren, Chaopeng Fu, Qing Dong, Min Jiang, Anping Dong, Guoliang Zhu, Jiao Zhang, and Baode Sun, Correction to Evaluation of Impurities in Aluminum Anodes for Al-Air Batteries (https://pubs.acs.org/doi/pdf/10.1021/acssuschemeng.1c07387)

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