

ICP-OES Analysis of Copper Recovered from Li-Ion Batteries for Foil Manufacturing

Automated characterization of materials for battery remanufacturing using the Agilent 5800 ICP-OES



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Introduction

The main goal of lithium-ion battery (LIB) recycling is to recover essential elements so they can be reused in the production of new batteries.¹ This cyclic process not only reduces waste and environmental impact but also helps lower manufacturing costs and supports the growing demand for battery materials.

The key elements recovered during LIB recycling include lithium (Li), manganese (Mn), cobalt (Co), and nickel (Ni), which are used in cathode production, as well as copper (Cu), which is critical for making Cu foil.

On average, LIBs contain around 10–15% Cu by weight, making it an ideal metal for recovery. During remanufacturing, the recycled Cu is dissolved in sulfuric acid (H_2SO_4) to produce an aqueous copper sulfate solution ($CuSO_4$), commonly referred to as a Cu electrolyte. Once this solution reaches the desired concentration, Cu is electroplated to produce high-purity Cu foil.² After Cu removal, the remaining electrolyte still contains valuable metals, such as Co, Ni, and Li, which can also be recovered and refined for reuse. The process is summarized in Figure 1.

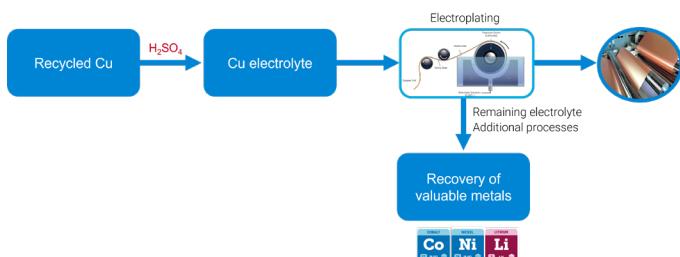


Figure 1. Schematic of the copper foil remanufacturing process using copper recovered from recycled LIBs.

These valuable streams require both macro- and trace-elemental analysis to ensure accurate quality control (QC). Agilent 5800 and 5900 ICP-OES instruments powered by Agilent ICP Expert software are central in this process. They are field-proven to provide precise, reliable detection of critical elements at every stage of LIB manufacturing, recycling, and remanufacturing processes.^{3,4}

Samples with a high total dissolved solids (TDS) content like CuSO_4 solutions can be difficult to analyze by ICP-OES due to their density, viscosity, varying analyte concentrations, and the risk of carryover. Other challenges arise from the corrosive and high-density nature of H_2SO_4 in the solution and demands for high sample throughput. Agilent ICP-OES instruments overcome these challenges through robust design of hardware components such as the vertical torch, Cool Cone Interface (CCI), and free-form optics.⁵ Also, software features such as Fitted and FACT background correction techniques, IntelliQuant Screening, Early Maintenance Feedback (EMF), and NebAlert assist the analyst with the analytical and instrumental aspects of the analysis.⁶ To ensure high throughput, an Agilent ICP-OES Automation System can be used.^{7,8}

In this study, a fully integrated system comprising the Agilent 5800 VDV ICP-OES, Advanced Valve System (AVS 7), Advanced Dilution System (ADS 2), SPS 4 autosampler, and instrument software was used to quantify 23 elements in three real-word recycled copper electrolyte samples. The elements included: aluminum (Al), antimony (Sb), arsenic (As), bismuth (Bi), cadmium (Cd), calcium (Ca), chromium (Cr), Co, Cu, iron (Fe), lead (Pb), Li, magnesium (Mg), manganese (Mn), sodium (Na), Ni, phosphorus (P), selenium (Se), silicon (Si), silver (Ag), tin (Sn), potassium (K), zinc (Zn).

The copper electrolyte samples were analyzed as received. The method's accuracy, robustness, and stability were assessed via a spike-recovery test on actual samples and a five-hour long-term stability test of 187 samples.

Experimental

Instrumentation

All measurements were performed using an Agilent 5800 VDV ICP-OES configured with an AVS 7 switching valve, ADS 2 autodilutor, and SPS 4 autosampler (Figure 2). Combined with Agilent ICP Expert Pro software, these components represent the Agilent ICP-OES Automation System. The sample introduction system consisted of a SeaSpray nebulizer, double-pass cyclonic spray chamber, argon humidifier accessory, and Agilent semi-demountable VDV torch with a 1.8 mm internal diameter (id) injector.

An internal standard (IS) solution comprising 5 mg/L scandium (Sc) and 100 mg/L rubidium (Rb) was prepared in 3% H_2SO_4 using Agilent single element standard solutions. The ISs were used to account for any matrix effects that may arise from the sample matrix. The seven-port AVS system enables the IS solution to be directly plumbed into the valve so it can be introduced with the sample.



Figure 2. The Agilent ICP-OES Automation System: Agilent 5800 VDV ICP-OES with integrated AVS 7 switching valve (left), Agilent Advanced Dilution System ADS 2 (middle), and Agilent SPS 4 autosampler (right).

The method was optimized using both axial and radial views. For the axial view, optimization focused on increasing the residence time of the sample in the plasma by raising the RF power and reducing the nebulizer flow from the default settings. The RF power remained unchanged in radial view, while the viewing height and nebulizer flow were optimized to reduce background for emission lines above 400 nm, including those of Li, K, and Na.

The operating conditions of the integrated ICP-OES automation system are listed in Tables 1 and 2, respectively.

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

Parameter	Setting	
Viewing Mode	Axial	Radial
Viewing Height (mm)	–	11
RF Power (kW)	1.4	
Nebulizer Flow (L/min)	0.65	0.9
Plasma Flow (L/min)	12	
Aux Flow (L/min)	1	
Replicates	3	
Rinse Time (s)	20	
Read Time (s)	10	5
Stabilization Time (s)	10	5
Sample Pump Tubing	Solvaflex white/white	
Internal Standard Pump Tubing	Solvaflex orange/green	
Waste Pump Tubing	Solvaflex blue/blue	

Table 2. Agilent AVS 7 and ADS 2 operating parameters.

Parameter	Setting
Sample Loop Size (mL)*	1.5
Pump Rate – Uptake (mL/min)	31.9
Pump Rate – Inject (mL/min)	3.6
Valve Uptake Delay (s)	12.4
Bubble Injection Time (s)	1.8
Preemptive Rinse Time (s)	1.2
Reactive Dilution Rinse (s)	10

*Both the AVS 7 and ADS 2 loops are the same size.

Sample preparation

Three Cu electrolyte solutions were supplied by a US-based foil remanufacturing company. The samples contained 3.5–5% CuSO₄ in 3% H₂SO₄, with varying levels of trace elements. The samples were designated as Cu electrolyte 1, Cu electrolyte 2, and Cu electrolyte 3 (Figure 3). Each sample was tested as received—no manual handling or preparation was performed before analysis.



Figure 3. Three copper electrolyte samples in a 3% sulfuric acid matrix. These samples were analyzed as received using the Agilent 5800 VDV ICP-OES with ADS 2 autodilutor.

Automated preparation of calibration standards by ADS 2

A 3% H₂SO₄ diluent was prepared by diluting 93–98% ultrapure H₂SO₄ (ARISTAR ULTRA, VWR Chemicals, BDH, Avantor, USA). Calibration curves were prepared automatically, using the autocalibration feature of the integrated ADS 2. The standards were prepared from the following Agilent stock solutions using the 3% H₂SO₄ diluent:

- 1000 ppm single-element stock solutions for Bi, Li, P, Si, and Sn
- 1% single element stock solution for Cu
- Multi-element Environmental Spike Mix standard

Only Pb was not prepared using the ADS 2; instead, it was prepared as a single-point standard at 1 ppm due to stability considerations in the 3% H₂SO₄ matrix. The Pb calibration standard was prepared by diluting an Agilent single element standard with the 3% H₂SO₄ diluent.

Quality Control solutions

For quality control purposes, a blank solution of 3% H₂SO₄ was used as the continuing calibration blank (CCB). Three continuing calibration verification solutions (CCV-1, CCV-2, and CCV-3) were prepared from Agilent standards, including the Environmental Spike Mix standard and single-element stock solutions at 1000 and 10,000 ppm.

- CCV-1 contained 0.5 ppm of Ag, Al, As, Bi, Cd, Co, Cr, Li, Mn, Ni, P, Pb, Sb, Se, Si, Sn, and Zn, and 5 ppm of Ca, Fe, K, Mg, Na
- CCV-2 contained Pb at 0.5 ppm
- CCV-3 contained Cu at 100 ppm

Method development

IntelliQuant Screening

As part of the ICP Expert Pro software, the IntelliQuant Screening routine can be used to collect full-spectrum data of a sample, requiring only a few seconds analysis time and with little input from the analyst.⁹ The IntelliQuant algorithm then processes the full-spectrum data against premeasured calibrations, generating a semiquantitative reading for every element present in the sample.

IntelliQuant Screening was used in this study during method development to determine the approximate concentration of elements in the three Cu electrolyte samples. The data informed the selection of the calibration range, internal standards, and emission lines best suited for accurate quantitative analysis.

The software can display the data in various ways, including as a periodic table heatmap or pie chart. Figure 4A shows a pie chart of the elemental composition of the Cu electrolyte 3 sample. As expected, Cu and S were the predominant elements. To better understand the trace components of the sample, Cu and S were excluded from the graphic, revealing the presence of additional elements such as Na, Ca, Fe, Li, Si and Ni, among others (Figure 4B).

Yttrium (Y), a widely used internal standard, was detected in the sample (Figure 4C). This guided the selection of Sc and Rb as internal standards.

The IntelliQuant star rating system further supported method development by recommending optimal emission lines and identifying potential spectral interferences. For instance, in this sample, the Zn 213 and Zn 202 nm lines were affected by interferences from Fe and Cu, and Cu, respectively (Figure 4D). Based on this valuable information, Zn 206 nm was selected as the most suitable line for the quantitative method.

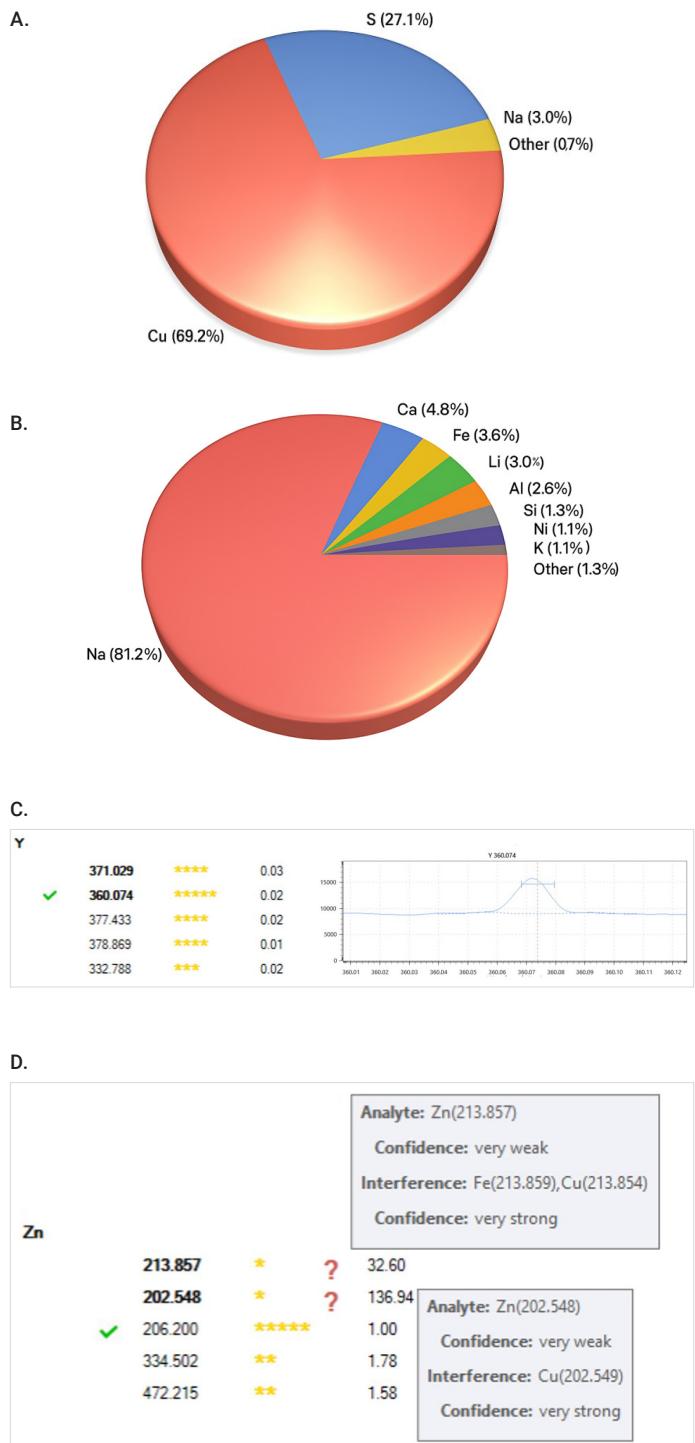


Figure 4. IntelliQuant results, including A: elemental composition of Cu electrolyte sample 1; B: trace profiles after excluding Cu and S from the pie chart; C: detection of Y; D: star rating guidance for the selection of the best Zn line.

Autocalibration and linearity

Details of the calibration standards for each element are given in Table 3. Each calibration point was created by auto diluting the stock standard solutions at multiple factors with 3% H₂SO₄ using the ADS 2.

All calibration curves were linear over the range, as indicated by correlation coefficients between 0.99999 and 1.0000 (Table 3). Representative calibration curves for Ag, Fe, and Li are shown in Figure 5.

Table 3. Concentration of calibration standards mg/L, background correction, calibration correlation coefficients, and the internal standard used for each element.

Element and Wavelength (nm)	Background Correction	Calibration Range (ppm)	Correlation Coefficient	Internal Standard
Ag 328.068	FACT	0.025–1	1.00000	Sc 357.634
Al R 396.152	FACT	0.025–1	1.00000	Sc 357.634
As 188.980	FACT	0.025–1	1.00000	Sc 357.634
Bi 190.171	FACT	0.1–1	1.00000	Sc 357.634
Ca R 315.887	Fitted	1–100	1.00000	Sc 357.634
Cd 214.439	Fitted	0.025–1	1.00000	Sc 357.634
Co 228.615	Fitted	0.025–1	1.00000	Sc 357.634
Cr 267.716	Fitted	0.025–1	1.00000	Sc 357.634
Cu R 327.395	Fitted	100–200	0.99999	Sc 357.634
Fe R 234.350	Fitted	1–100	1.00000	Sc 357.634
K R 766.491	FACT	1–100	1.00000	Rb 780.026
Li R 670.783	FACT	0.1–1	1.00000	Rb 780.026
Mg R 285.213	Fitted	0.25–10	1.00000	Sc 357.634
Mn 257.610	Fitted	0.25–1	1.00000	Sc 357.634
Na R 589.592	FACT	1–100	1.00000	Sc 424.682
Ni 231.604	Fitted	0.025–10	1.00000	Sc 357.634
P 178.222	FACT	0.1–1	1.00000	Sc 357.634
Pb 220.353	FACT	1	1.00000	Sc 357.634
Sb 206.834	FACT	0.025–1	0.99999	Sc 357.634
Se 196.026	Fitted	0.025–1	1.00000	Sc 357.634
Si R 251.611	Fitted	0.1–1	1.00000	Sc 357.634
Sn 189.925	Fitted	0.1–1	1.00000	Sc 357.634
Zn 206.200	Fitted	0.1–1	1.00000	Sc 357.634

R: Analyzed in radial view.



Figure 5. Representative linear calibration curves for Ag, Fe, and Li with a correlation coefficient of 1.0000 and a Relative Standard Error (%RSE) <1.9%.

ADS 2 dilution features

The ADS 2 enables both prescriptive and reactive dilutions. Prescriptive dilution applies a user-defined dilution factor (up to 400x) before analysis, replacing manual preparation. Reactive dilution (up to 400x) is triggered automatically if an analyte exceeds the calibration (or detector) range or IS recovery limits. In these cases, the system calculates and applies the necessary dilution factor in real time.

For greater flexibility when analyzing different sample types in the same run, the Dilution Lists software function allows the user to select specific elements to trigger automatic dilution only when concentrations exceed the calibration range (Figure 6).

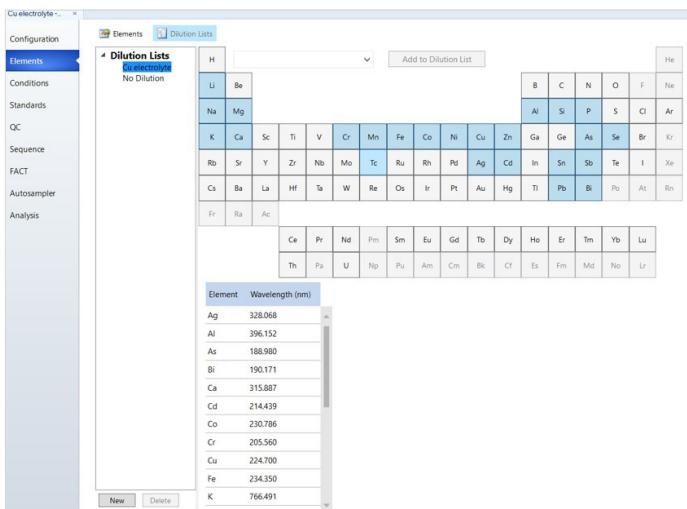


Figure 6. Selection of elements in Dilution Lists for “Cu electrolyte”.

Only the selected elements will trigger an automatic reactive dilution by the Agilent ADS 2 autodilutor when the Cu electrolyte dilution list is selected – see Figure 7.

Dilution Lists can also be used to exclude certain sample types from reactive-dilution triggers, avoiding unnecessary dilutions. For example, matrix spikes, which are used to verify analyte recovery in real samples, can be excluded from reactive dilution, as shown for Cu electrolyte 1 spike (prepared from Cu electrolyte 1 20x) in Figure 7. In this case, a dilution list removing Cu named “No Dilution” was selected for either Cu electrolyte 1 spike or Cu electrolyte 1 20x, ensuring both samples were analyzed as prepared thereby supporting accurate validation without unnecessary dilution cycles.

For the analysis of the neat (undiluted) Cu electrolyte samples, the dilution list “Cu electrolyte” was selected. This list specifies the elements that can trigger a reactive dilution, as shown in Figure 6. With this setup, reactive dilutions are automatically initiated when required.

Rack:Tube	Solution Label	Solution Type	Dilution List
1:1	Cu electrolyte 1 20x	Sample	No Dilution
1:2	Cu electrolyte 1 20x spike	Sample	No Dilution
2:2	Rinse 3% H ₂ SO ₄	Sample	No Dilution
1:3	Cu electrolyte 1 Neat	Sample	Cu electrolyte
2:2	Rinse 3% H ₂ SO ₄	Sample	No Dilution
1:4	Cu electrolyte 2 Neat	Sample	Cu electrolyte

Figure 7. Screenshot of the Agilent ICP Expert Pro software showing the Dilution List function, which allows analysts to select which elements will trigger reactive dilutions to samples on an individual basis by the Agilent ADS 2. In the orange-highlighted dilution list “Cu electrolyte”, Cu is included along with other elements, meaning samples containing these elements will undergo reactive dilution. In the blue highlighted “No dilution” list, Cu has been removed, so samples will not be diluted reactively.

Results and discussion

Method detection limits

Method Detection Limits (MDLs) were determined by calculating three times the standard deviation from ten replicate measurements of the 3% H₂SO₄ blank. To represent the sample matrix, MDLs were also determined in a 1% Cu solution (Agilent standard solution).

For some of the elements listed in Table 4, the selected wavelengths were not the most sensitive ones available. However, IntelliQuant identified these lines as being free from spectral interferences, making them more suitable for accurate quantification in high-Cu matrix samples. MDLs were in the sub-ppb to low-ppb range even in the high-Cu matrix (Table 4), demonstrating the excellent sensitivity of the 5800 ICP-OES method.

Table 4. Method detection limits for elements in 3% H₂SO₄ and 1% Cu solution.

Element and Wavelength (nm)	MDL (mg/L)	
	3% H ₂ SO ₄	1% Cu
Ag 328.068	0.00047	0.00044
Al R 396.152	0.00430	0.00567
As 188.980	0.00160	0.00348
Bi 190.171	0.00525	0.01261
Ca R 315.887	0.00382	0.00996
Cd 214.439	0.00012	0.00022
Co 228.615	0.00051	0.00088
Cr 267.716	0.00041	0.00053
Cu R 327.395	0.00286	NA
Fe R 234.350	0.00319	0.00513
K R 766.491	0.03718	0.17615
Li R 670.783	0.00239	0.00074
Mg R 285.213	0.00164	0.00169
Mn 257.610	0.00007	0.00005
Na R 589.592	0.00270	0.10244
Ni 231.604	0.00112	0.00118
P 178.222	0.00433	0.00486
Pb 220.353	0.00232	0.00463
Sb 206.834	0.00212	0.00577
Se 196.026	0.00608	0.00586
Si R 251.611	0.00790	0.01305
Sn 189.925	0.00290	0.00936
Zn 206.200	0.00031	0.00049

R: Analyzed in radial view.

Quantitative analysis

Three Cu electrolyte samples obtained from the lithium recycling and foil remanufacturing process were analyzed using the 5800 ICP-OES with ADS 2.

As shown in Figure 8, Cu electrolyte 1 was first analyzed without dilution, and only Cd was within the calibration range at 0.086 ppm. The ADS system then automatically performed a 5x reactive dilution of the sample, which brought Al, Co, and Fe into range. Since Na remained above the calibration limit, a 25x dilution was applied. Both dilution steps were conducted automatically, without any input from the analyst.

The results 'Summary' report selects the best measurement result for each element from the available iterations, without

overwriting any existing data: undiluted for Cd, 5x dilution for Al, Co, and Fe, and 25x dilution for Na. Despite the high-TDS content of the sample and the use of 3% H₂SO₄ as the diluent, excellent precision was maintained. The respective RSDs were <0.5% for Cd (undiluted), <0.4% for Co (5x dilution), and <0.7% for Na (25x dilution).

Despite the use of different dilution factors, the results for each analyte were consistent after applying the dilution factors, demonstrating the reliability of the ADS 2 system. For complex matrices such as Cu electrolyte, where analyte concentrations can vary widely, the ADS 2 streamlines the dilution process and ensures accurate, efficient, and productive analysis, thereby supporting LIB recycling and remanufacturing.

Unadjusted													
Track Analysis < MDL Flagging Sort Results... Hide Columns... Column Properties... Delete Results													
	Rack:Tube		Solution Label		Timestamp	Al R 396.152 nm ppm	Cd 214.439 nm ppm	Co 228.615 nm ppm	Fe R 234.350 nm ppm	Na R 589.592 nm ppm	Rb R 780.026 nm Ratio	Sc 357.634 nm Ratio	Sc R 357.634 nm Ratio
		1:3	Cu electrolyte 1 Neat		1/20/2025 5:42:57 PM	0.574	0.086	0.577	40.778	39.979	--	--	--
<input checked="" type="checkbox"/>	1:3		Summary ✓		1/20/2025 5:42:57 PM	0.574	0.086	0.577	40.778	39.979	--	--	--
<input type="checkbox"/>	Neat		Original		1/20/2025 5:35:17 PM	2.954 o	0.086	2.809 o	198.161 o	1015.259 o	0.90	0.89	0.90
<input type="checkbox"/>	Reactive		Dilution - 5		1/20/2025 5:37:43 PM	0.574	0.018	0.577	40.778	200.136 o	0.98	0.97	0.97
<input type="checkbox"/>	Reactive		Dilution - 25		1/20/2025 5:40:15 PM	0.113	0.003	0.116	8.140	39.979	1.01	1.01	1.00
Precision:													
 Cadmium Original: Concentration 0.086 !, Intensity 9608.117 ! Average: 0.086 !, SD: 0.000 % RSD: 0.49, Background: N/A			 Cobalt Original: Concentration 0.577 !, Intensity 14405.082 ! Average: 0.577 !, SD: 0.010 % RSD: 0.36, Background: N/A			 Sodium Original: Concentration 39.979 !, Intensity 373693.944 ! Average: 39.979 !, SD: 6.741 % RSD: 0.67, Background: N/A			Undiluted <input checked="" type="checkbox"/> Replicate Intensity Concentration 1 9582.250 0.086 2 9662.236 0.086 3 9579.866 0.086				
5x													
 Cadmium Original: Concentration 0.086 !, Intensity 9608.117 ! Average: 0.086 !, SD: 0.000 % RSD: 0.49, Background: N/A			 Cobalt Original: Concentration 0.577 !, Intensity 14405.082 ! Average: 0.577 !, SD: 0.010 % RSD: 0.36, Background: N/A			 Sodium Original: Concentration 39.979 !, Intensity 373693.944 ! Average: 39.979 !, SD: 6.741 % RSD: 0.67, Background: N/A			<input checked="" type="checkbox"/> Replicate Intensity Concentration 1 14346.390 0.574 2 14442.496 0.578 3 14426.358 0.578				
25x													
 Cadmium Original: Concentration 0.086 !, Intensity 9608.117 ! Average: 0.086 !, SD: 0.000 % RSD: 0.49, Background: N/A			 Cobalt Original: Concentration 0.577 !, Intensity 14405.082 ! Average: 0.577 !, SD: 0.010 % RSD: 0.36, Background: N/A			 Sodium Original: Concentration 39.979 !, Intensity 373693.944 ! Average: 39.979 !, SD: 6.741 % RSD: 0.67, Background: N/A			<input checked="" type="checkbox"/> Replicate Intensity Concentration 1 37425.886 40.039 2 375886.775 40.214 3 370940.171 39.685				
Concentration													
	Rack:Tube		Solution Label		Timestamp	Al R 396.152 nm ppm	Cd 214.439 nm ppm	Co 228.615 nm ppm	Fe R 234.350 nm ppm	Na R 589.592 nm ppm	Rb R 780.026 nm Ratio	Sc 357.634 nm Ratio	Sc R 357.634 nm Ratio
		1:3	Cu electrolyte 1 Neat		1/20/2025 5:42:57 PM	2.868	0.086	2.884	203.890	999.486	--	--	--
<input checked="" type="checkbox"/>	1:3		Summary ✓		1/20/2025 5:42:57 PM	2.868	0.086	2.884	203.890	999.486	--	--	--
<input type="checkbox"/>	Neat		Original		1/20/2025 5:35:17 PM	2.954 o	0.086	2.809 o	198.161 o	1015.259 o	0.90	0.89	0.90
<input type="checkbox"/>	Reactive		Dilution - 5		1/20/2025 5:37:43 PM	2.868	0.089	2.884	203.890	1000.679 o	0.98	0.97	0.97
<input type="checkbox"/>	Reactive		Dilution - 25		1/20/2025 5:40:15 PM	2.832	0.085	2.889	203.508	999.486	1.01	1.01	1.00

Figure 8. Example of the Agilent ADS 2 performing 5x and 25x reactive dilutions of Cu electrolyte sample 1. Top: Unadjusted concentration data for Al, Cd, Co, Fe, and Na. Middle: Precision for Cd, Co, and Na in the undiluted (original) and diluted sample analyses. Bottom: Data corrected for dilution factors, collated in the Summary row.

The quantitative data for the three Cu electrolyte samples are reported in Table 5.

Table 5. Quantitative results for elements measured in each of the three Cu electrolyte samples analyzed by the Agilent 5800 VDV ICP-OES with ADS 2. The results have been adjusted for the dilution factor.

Element and Wavelength (nm)	Cu Electrolyte Samples Concentration (mg/L)		
	1	2	3
Ag 328.068	0.198	0.065	0.016
Al R 396.152	2.87	2.89	37.0
As 188.980	0.014	0.006	0.005
Bi 190.171	<MDL	<MDL	<MDL
Ca R 315.887	151	153	60.7
Cd 214.439	0.086	0.086	<MDL
Co 228.615	2.88	2.92	1.21
Cr 267.716	0.837	0.839	3.32
Cu R 327.395	14089	14194	19508
Fe R 234.350	204	206	57.1
K R 766.491	6.124	5.40	24.6
Li R 670.783	0.766	0.756	36.9
Mg R 285.213	6.15	6.18	11.3
Mn 257.610	0.893	0.896	1.95
Na R 589.592	999	87.7	1583
Ni 231.604	262	264	20.4
P 178.222	0.197	0.138	2.88
Pb 220.353	3.02	3.07	0.108
Sb 206.834	0.165	<MDL	0.026
Se 196.026	<MDL	<MDL	<MDL
Si R 251.611	14.3	14.1	27.5
Sn 189.925	2.15	<MDL	<MDL
Zn 206.200	21.1	21.4	0.911

R: Analyzed in radial view.

Spike recovery test

To further evaluate the 5800 VDV ICP-OES method, a spike recovery test was conducted on Cu electrolyte sample 1. The sample was diluted 20x and spiked as follows: 5.5 ppm for Ni; 5 ppm for Ca, Fe, K, Mg, Na; and 0.5 ppm for the rest of the elements.

As shown in Table 6, recoveries for all elements were within $100 \pm 5\%$, confirming accuracy across the concentration range. All spike information is displayed before the dilution factor has been applied.

Table 6. Spike recovery data for the Cu electrolyte sample 1 diluted 20x by the ADS 2.

Element and Wavelength (nm)	Conc in Solution (ppm)	Cu Sample + Spike (ppm)	Recovery (%)
Ag 328.068	0.011	0.531	104
Al R 396.152	0.141	0.638	101
As 188.980	<MDL	0.506	100.8
Bi 190.171	<MDL	0.485	96.6
Ca R 315.887	7.6	12.7	98.3
Cd 214.439	0.004	0.494	98
Co 228.615	0.147	0.638	98.2
Cr 267.716	0.042	0.536	98.8
Fe R 234.350	10.3	15.3	99.2
K R 766.491	0.187	5.13	100.3
Li R 670.783	0.039	0.548	101.2
Mg R 285.213	0.307	5.19	97.7
Mn 257.610	0.045	0.528	96.6
Na R 589.592	50.4	55.6	104.1
Ni 231.604	13.5	18.9	99.2
P 178.222	0.010	0.518	101
Pb 220.353	0.154	0.677	104.6
Sb 206.834	0.008	0.517	102
Se 196.026	<MDL	0.501	100
Si R 251.611	0.744	1.26	103.2
Sn 189.925	0.108	0.594	97.2
Zn 206.200	1.08	1.57	97.6

R: Analyzed in radial view.

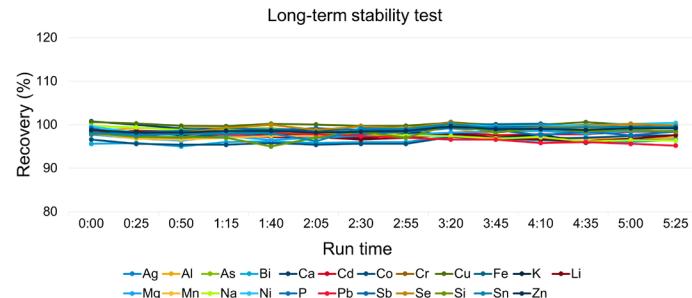


Figure 9. Normalized recovery of QC solutions analyzed over five hours by the Agilent ICP-OES Automation System.

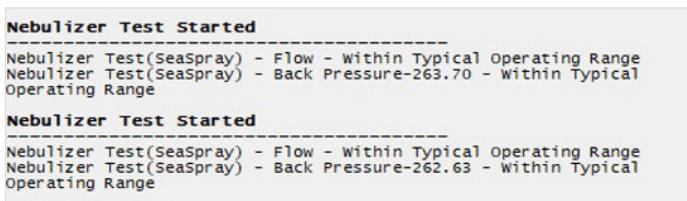


Figure 10. Nebulizer test before and after the stability test showing a back pressure reading of 263.70 and 262.63 kPa, respectively.

Stability test

To demonstrate the robustness of the ICP-OES Automation System, 187 solution measurements were collected over five hours without recalibration. The test sequence consisted of a 1% CuSO_4 solution in 3% H_2SO_4 , interspersed with a QC block after every 10 runs. Each QC block included a CCB and three CCV solutions.

The ADS 2 performed a prescriptive 2x dilution on CCV-1 and CCV-3, resulting in final concentrations of 0.5 ppm for Ag, Al, As, Bi, Cd, Co, Cr, Li, Mn, Ni, P, Pb, Sb, Se, Si, Sn, and Zn; 5 ppm for Ca, Fe, K, Mg, and Na; and 100 ppm for Cu. CCV-2 consisted of a 0.5 ppm Pb solution.

As shown in Figure 9, recoveries for all elements remained within $100 \pm 5\%$ across the full analytical run, which included autodilution before each measurement of CCV-1 and CCV-3. Precision was excellent, with %RSD values below 1.3%, confirming the stability and reproducibility of the 5800 VDV ICP-OES with ADS 2 for routine, high-throughput analysis of high-Cu solutions.

The screenshot from the ICP Expert software in Figure 10 shows near-identical nebulizer pressure before and after the five-hour test, further confirming instrument stability over long runs of complex samples.

Conclusion

The Agilent ICP-OES Automation System—a combination of the Agilent 5800 VDV ICP-OES with the AVS 7 switching valve, ADS 2 autodilutor, SPS 4 autosampler, and ICP Expert Pro software—provides a powerful solution for elemental analysis in lithium battery recycling and copper foil remanufacturing. The system delivered accurate, precise, and reproducible results across a wide concentration range, even in challenging high-matrix copper electrolyte samples. Automated prescriptive and reactive dilutions minimized manual sample handling, reduced errors, and ensured that all analytes were reliably quantified within calibration limits.

Spike recovery and long-term stability tests further demonstrated the robustness of the method, with recoveries within $100 \pm 5\%$ and precision better than 1.3% RSD over

five hours of continuous operation. These results confirm that the ICP-OES Automation System not only streamlines sample preparation and analysis but also enhances laboratory productivity, data quality, and confidence in results.

By simplifying the analysis of complex matrices while maintaining accuracy and throughput, the system enables laboratories in the energy and chemicals sector to efficiently support the recovery and reuse of critical elements from lithium-ion batteries, contributing to sustainable battery manufacturing.

References

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More information

View the on-demand webinar: [Eco-Friendly Extraction Methods for Lithium-Ion Battery Recycling and Elemental Analysis of Impurities in Recovered Metal Sulphates | Separation Science](#)

Products used in this application

Agilent products

Description	Part Number
Easy-fit 1.8 mm semi-demountable torch for 5000 series VDV/SVDV ICP-OES	G8020-68005
Double-pass spray chamber, glass cyclonic design with ball joint socket and UniFit drain outlet, for Agilent 5000 series ICP-OES	G8010-60256
SeaSpray concentric glass nebulizer for 5000 series ICP-OES	G8010-60255
Peristaltic pump tubing, white/white, 12/pk	3710034400
Peristaltic pump tubing, orange/white, 12/pk	3710046900
Peristaltic pump tubing, blue/blue, 12/pk	3710034600
Diluent/carrier bottle kit for ADS 2 and Autosampler (6L HDPE)	5005-0435
Waste container kit, 10L with Stay Safe cap and filter	5005-0437
Agilent Multi-element Quality Control Standard 27	5190-9418
Agilent 1000 ppm single element stock solution for Al, 500 mL	5190-8243
Agilent 1000 ppm single element stock solution for Ca, 500 mL	5190-8330
Agilent 1000 ppm single element stock solution for Cd, 500 mL	5190-8328
Agilent 1000 ppm single element stock solution for Co, 500 mL	5190-8347
Agilent 1000 ppm single element stock solution for Cr, 500 mL	5190-8345
Agilent 1000 ppm single element stock solution for Cu, 500 mL	5190-8349
Agilent 1000 ppm single element stock solution for Fe, 500 mL	5190-8472
Agilent 1000 ppm single element stock solution for K, 500 mL	5190-8504
Agilent 10,000 ppm single element stock solution for Li, 500 mL	5190-8409
Agilent 1000 ppm single element stock solution for Mg, 500 mL	5190-8482
Agilent 1000 ppm single element stock solution for Mn, 500 mL	5190-8484
Agilent 1000 ppm single element stock solution for Na, 500 mL	5190-8526
Agilent 1000 ppm single element stock solution for Ni, 500 mL	5190-8492
Agilent 10,000 ppm single element stock solution for P, 500 mL	5190-8429
Agilent 1000 ppm single element stock solution for Pb, 500 mL	5190-8476
Agilent 1000 ppm single element stock solution for Rb, 100 mL	5190-8511
Agilent 1000 ppm single element stock solution for Sb, 500 mL	5190-8245
Agilent 1000 ppm single element stock solution for Si, 500 mL	5190-8522
Agilent 1000 ppm single element stock solution for Y, 500 mL	5190-8556
Agilent 1000 ppm single element stock solution for Zn, 500 mL	5190-8558

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