

Analysis of Elemental Impurities in Lithium-Ion Battery Electrolyte Solvents by ICP-MS

Direct determination of 21 elements in mixes of LIB-solvents DMC, EMC, and EC



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Introduction

The rapid increase in the use of lithium-ion batteries (LIBs) in various industries such as consumer electronics, electric vehicles (EVs), and energy storage, has driven the dramatic increase in global demand for LIBs. The current generation of LIBs demonstrates outstanding performance in terms of high energy and power density, rapid charging, multiple charge/discharge cycles, light weight, and improved safety. These performance capabilities – and future improvements – depend on the quality of the materials used for the four main battery cell components, the cathode, anode, electrolyte, and separator (1). As one of the main components in LIBs, the electrolyte plays a pivotal role in defining battery cell performance.

Current commercial LIBs use a liquid electrolyte (LE) or gel polymer electrolyte (GPE), depending on the manufacturing priorities, cost considerations, and the intended end-use application. Liquid electrolytes typically contain a carefully formulated mix of lithium electrolyte salts, such as lithium hexa-fluorophosphate (LiPF_6), combined with various additives, and dissolved in an organic solvent. The solvent makes the charge carrier Li ions mobile, so they can migrate between the cathode and anode during the battery's charge and discharge cycles. The electrolyte also ensures good electrical contact by permeating into and stabilizing the surface of the electrodes (1).

Liquid electrolyte solvents commonly use proprietary mixtures of high-purity cyclic and chain carbonates including dimethyl carbonate (DMC), ethyl methyl carbonate (EMC), and ethylene carbonate (EC). Elemental impurities and contaminants such as chromium (Cr), iron (Fe), nickel (Ni), copper (Cu), and zinc (Zn) in the electrolyte solvent (and other components of a LIB) can have a significant impact on the performance and safety of the battery. Adverse effects can include changes to ion mobility, undesired electrochemical reactions, gas evolution, and other hazards such as Li dendrite growth (2). LIB manufacturers and solvent suppliers must therefore establish a reliable and accurate analytical procedure to monitor elemental impurities at trace levels in pure solvents and electrolyte mixes.

Inorganic (metals) analysis labs in the petrochemical industry have traditionally used ICP-OES for measuring organic samples. But ICP-OES does not provide low enough detection limits for the elements of interest in LIB solvents, especially for the higher-purity solvents required for development of advanced, next generation battery technologies (3). Advanced battery manufacturers are therefore investigating the potential to use ICP-MS to give the necessary improvements in detection limit performance.

Organic solvents are often considered challenging matrices to analyze by ICP-MS. Reported issues include the rapid buildup of undissociated carbon (soot) on the interface cones, leading to signal loss and instability. Some users have also observed instability of the plasma if the RF generator and matching circuit are unable to respond quickly enough to changes associated with the organic solvent matrix. The organic matrix can also form intense, carbon-based polyatomic ions that cause spectral overlaps and inaccurate results for some analytes. For example, $^{12}\text{C}_2$ overlaps magnesium (Mg) at m/z 24, and $^{40}\text{Ar}^{12}\text{C}$ overlaps Cr at m/z 52. And the carbon matrix can cause ionization enhancement, where the sensitivity for some poorly ionized elements such as arsenic (As) and selenium (Se) increases with higher levels of carbon (4).

On Agilent ICP-MS systems, soot buildup on the interface is easily avoided by adding oxygen (as a mix of 20% O_2 in Ar, for safety reasons) via the optional fifth gas flow controller. Agilent ICP-MS systems also use a high-performance, solid-state, 27 MHz plasma RF generator with rapid frequency matching to cope with the vapor load and enable routine analysis of organic solvent matrices. Agilent ICP-MS systems include a fourth generation collision/reaction cell (CRC), the ORS⁴, that can operate in helium (He) collision mode or with a reactive cell gas to control the most intense spectral interferences. The carbon enhancement effect can be eliminated by preparing the calibration standards in a matrix that closely matches the sample composition – so-called matrix matching, or by spiking standards into a representative sample, a calibration approach known as standard addition. An Agilent ICP-MS provides high sensitivity, good selectivity, fast analysis, and broad elemental coverage for the routine determination of elemental impurities in high-purity organic solvent samples.

In an initial study (5), an Agilent 7900 ICP-MS was used for the quantification of 21 elements of interest in undiluted DMC/EMC and DMC/EC solvent mixes that are commonly used in LIB electrolytes. The 7900 ICP-MS was configured for organic sample analysis as outlined in the previous paragraph, and solvent resistant sample introduction and drain tubing was used. One sample of each solvent mix was used to optimize operating conditions and standard addition method settings.

In the follow-up work reported here, a more comprehensive quantitative analysis was performed using standard addition calibrations prepared in the two electrolyte solvent mixes. The analytical performance in terms of linearity, method detection limit (MDL), spike recovery, and long-term stability was evaluated for all the target elements.

In addition to the quantitative analysis results, the Agilent ICP-MS MassHunter software's QuickScan function was used to assess the levels of other, non-target analytes i.e., elements that were not in the 21-element standard mix. QuickScan adds only two seconds of additional measurement time for each sample but provides semiquantitative results for "all elements" using IntelliQuant non-specific calibration. When combined with He mode, QuickScan provides analysts with a rapid screening tool to assess the potential presence and approximate concentration of other elements in the solvent samples.

Experimental

Instrumentation

A 7900 ICP-MS fitted with organic solvent sample introduction kit (p/n G3280-60580) was used. The organics kit includes a quartz torch with 1.5 mm internal diameter (id) injector, solvent-resistant tubing, and a 0.15 mm id sample probe for the Agilent I-AS integrated autosampler. The 7900 was fitted with the optional fifth gas line for the addition of O₂/Ar (20/80 mix). Platinum (Pt) tipped interface cones (Pt-tipped, Cu base sampling cone p/n G3280-67036 and Pt-tipped Ni skimmer cone p/n G8400-67202) were fitted, as required when O₂ is introduced to the plasma. The solvent samples were introduced directly from the autosampler to the MicroMist nebulizer (p/n G3266-80004) by self-aspiration to avoid potential contamination from the peristaltic pump tubing.

For most elements, the ORS⁴ of the 7900 ICP-MS was operated using standard He collision mode conditions with kinetic energy discrimination (KED) to remove the polyatomic (molecular) ion interferences. Enhanced (high flow, high collision energy, HE) He mode or H₂ reaction cell gas were used as appropriate to ensure the most effective removal of spectral overlaps on some analytes that suffer intense argon- or carbon-based interferences.

The use of self-aspiration and the organic matrix instrument configuration (e.g. narrow id torch injector) also means that tuning conditions should be optimized while aspirating the solvent matrix, rather than using the standard autotuning approach for conventional aqueous analysis. The Agilent ICP-MS MassHunter software allows the use of "Custom Tune" for situations where non-standard operating conditions are required. The option gas flow (O₂/Ar, 20/80) was optimized to ensure complete decomposition of the carbon matrix to avoid soot deposition on the interface cones. These operating conditions allow direct, routine, long-term analysis of the undiluted organic sample matrices on the 7900 ICP-MS. The instrument operating parameters are summarized in Table 1.

Table 1. Agilent 7900 ICP-MS operating conditions and parameters.

Cell Mode	No Gas	He	HEHe	H ₂
RF Power (W)	1600			
Sampling Depth (mm)	9			
Nebulizer Gas Flow (L/min)	0.55			
Ar/O ₂ Option Gas (%)	20			
Ext 1 (V)	0			
Ext 2 (V)	-250			
Omega Bias (V)	-110			
Omega Lens (V)	11.1			
ORS Gas (mL/min)	-	3.5	8.5	5
Energy Discrimination (V)	5		7	3

When ICP-MS is used to analyze acidified aqueous sample types, an acidic rinse solution is run after each sample to flush the introduction system. This rinse step ensures that the following sample results are not affected by carry-over from the previous solution. The rinse would usually consist of a slightly stronger acid mix than the samples/standards, so any elemental residues are preferentially flushed by the rinse, not the next sample. When non-water-soluble organic solvents are analyzed, an acidified aqueous rinse is not appropriate, so a suitable solvent is used as the rinse solution. Often the same solvent as the samples is used, in which case, an extended rinse period may be desirable to ensure that the sample uptake tubing is completely flushed.

In this work, 100% DMC was used as the rinse solution as it is fully miscible and compatible with the electrolyte solvent samples; DMC contributes the majority (70%) of the electrolyte solvent sample matrices. Using an alternative solvent for the rinse solution could introduce additional contaminants or affect stability if not fully miscible with the samples. A 170-second rinse time was used, with the autosampler probe being moved between two different rinse containers to further reduce the chance of carry-over. The effectiveness of the two-step DMC rinse was evaluated by monitoring the concentration of analytes in the first sample blank run after the highest calibration standard (500 ppb). The levels of the target analytes measured in the first sample blank were all within 10% of the BEC, demonstrating the dual container DMC rinse was effective to clean the sample delivery path between samples.

Reagents and standards

DMC, EMC, and EC solvents (Li-ion battery grade, ≥99% purity on trace metals basis) were bought from Sigma-Aldrich or Thermo Fisher Scientific. Two different mixtures of the solvents DMC/EMC (70/30, v/v) and DMC/EC (70/30, v/v) were prepared to match the typical mixtures used by battery electrolyte manufacturers.

An Agilent 21-element organometallic standard solution in hydrocarbon oil (p/n 5190-8712) was used as the stock to prepare the spiking standards for the standard addition calibrations. The stock standard contained Ag, Al, B, Ba, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, P, Pb, Sn, Ti, V, and Zn at a concentration of 500 µg/g (ppm). For each of the electrolyte solvent mixes (DMC/EMC and DMC/EC) a 5 ppm intermediate standard was prepared by taking 20 µL of the 500 ppm stock standard solution and making up to 2 mL total volume with the solvent mix. These intermediate standard mixes were then used to prepare the spikes for the standard addition calibration standards for their respective sample matrix. For each calibration set, nine standards were prepared by spiking an appropriate amount of the intermediate (5 ppm) standard into the solvent mix to give spike levels between 1.0 and 500 ppb for all 21 elements. The unspiked solvent mix was used as the calibration blank for each sample type.

After the calibration standards were measured, it became apparent that nearly all the elements of interest were present at low or sub-ppb levels in the unspiked blank solvent mixes so only the lower standards were used to generate quantitative results for the trace elements. ICP-MS MassHunter software includes an automated function where the standard addition calibration is converted automatically to an external calibration during the batch analysis. This function means that subsequent samples of the same type can be run and quantified without needing to be individually spiked with standards. This capability removes one of the most significant barriers to analysts adopting standard addition for routine analysis.

Results and discussion

The 7900 ICP-MS uses He collision mode or a reactive cell gas mode to reduce or remove the contribution from the carbon-based polyatomic overlaps that affect analytes such as ^{24}Mg and ^{52}Cr in an organic solvent matrix. A reactive cell gas can also resolve non-polyatomic spectral overlaps such as the isobaric overlap from ^{40}Ar on ^{40}Ca . He mode was used as the default mode for most elements, as usual for Agilent single quadrupole ICP-MS systems. For a few elements with intense carbon matrix or plasma-based spectral overlaps, enhanced (HE) He mode or H_2 reaction mode was used.

The effectiveness of these CRC approaches is illustrated in Figure 1, which shows the standard addition calibrations for several interfered elements in the DMC/EMC solvent matrix. The analytes and cell gas modes shown in Figure 1 are ^{24}Mg and ^{40}Ca (H_2 cell gas), ^{52}Cr and ^{53}Cr (HEHe mode), and ^{63}Cu and ^{111}Cd (He mode). The nearly identical result obtained for ^{52}Cr and ^{53}Cr in He KED mode illustrates the effectiveness of the Agilent ORS⁴ in He collision mode to remove the intense ArC^+ polyatomic ion overlap at mass 52. The Cr comparison also demonstrates the potential use of multiple isotopes (where available) to provide an internal qualifier measurement to confirm the results. Alternative cell gas modes can be used in a similar way. The good linearity and accurate analysis at low, sub-ppb levels for Cu and Cd demonstrate the benefit of ICP-MS for trace level contamination control of high purity LIB electrolyte solvents.

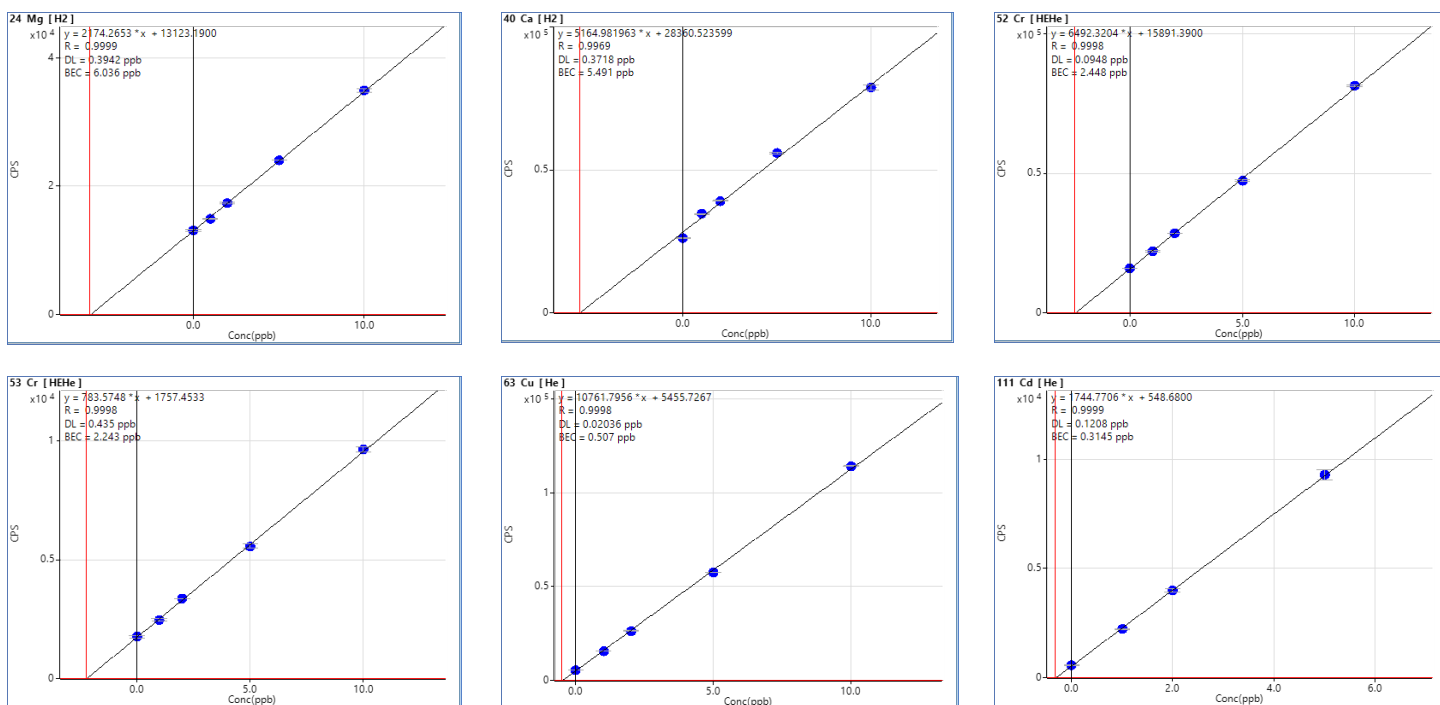


Figure 1. Standard addition calibration plots for ²⁴Mg and ⁴⁰Ca (H₂ mode), ⁵²Cr and ⁵³Cr (HEHe mode), ⁶³Cu and ¹¹¹Cd (He mode) in DMC/EMC. Standard addition plots zoomed to show spike levels up to 10 µg/L (ppb) for all elements except Cd (5 ppb).

As well as polyatomic and isobaric overlaps, the extremely high intensity of the carbon signal (major isotope at m/z 12) causes a peak-tail overlap at the adjacent mass, affecting the major isotope of boron, ¹¹B. A triple quadrupole, "tandem" ICP-MS such as the Agilent 8900 ICP-QQQ can use double mass filtering (MS/MS) to eliminate this peak tail overlap. But laboratories performing routine analysis of LIB electrolyte solvents using a single quadrupole ICP-MS require a different approach to avoid the carbon peak tail.

Agilent ICP-MS systems use a high-frequency (3 MHz) quadrupole mass filter with true hyperbolic profile rods to provide the most effective rejection of non-target masses while maintaining the highest possible ion transmission. Even with this high-performance mass filter, the low-mass peak tail from ¹²C still makes some contribution to ¹¹B. However, B has a secondary isotope, ¹⁰B, that is practically free of overlap from the ¹²C peak tail, so boron can be analyzed at low ppb levels in organic solvents by measuring the ¹⁰B isotope. The isotopic abundance of ¹⁰B (19.9%) is about four times lower than ¹¹B (80.1%), so detection limits are slightly compromised, but the 7900 ICP-MS is still easily able to meet the current method requirements for B in LIB electrolyte solvents.

Concentration results, detection limits, and spike recoveries

Table 2 includes the method performance and quantitative results for all 21 elements measured in the two unspiked electrolyte solvent mixes, DMC/EMC and DMC/EC. Detection limits (DLs) were calculated from multiple repeats of the blank (unspiked) samples. The samples were analyzed without dilution, so the DLs indicate the true measurement limit in the electrolyte solvents. Single- or sub-ppb DLs were achieved for all analytes, which demonstrates the excellent performance of the 7900 ICP-MS configured for direct analysis of undiluted electrolyte solvents. The low DLs also confirm that any potential carbon-based interferences were removed effectively by the cell gas modes. The blank solvent matrices contained a relatively high level of a few of the analytes of interest, which led to higher standard deviation and therefore increased DL for those elements.

To evaluate the accuracy and precision of the analysis, three separate aliquots from each sample were prepared and spiked at 25 ppb. Table 2 shows that the 7900 method achieved spike recoveries of between 88 to 107% with precision (%RSD of the three separate recoveries) of <8% for all analytes except Zn. No recovery result was obtained for Zn in the DMC/EC sample, as this element was present in the sample at much higher concentration than the 25 ppb spike level.

Table 2. Analyte masses, cell modes, DLs, measured concentrations, and spike recovery results for trace elements in electrolyte solvent mixes DMC/EMC and DMC/EC.

Analyte	Gas Mode	DMC/EMC Sample					DMC/EC Sample				
		R	DL (ppb)	Measured Concentration in Sample (ppb)	Spike Recovery (%)	%RSD of Recovery (n=3)	R	DL (ppb)	Measured Concentration in Sample (ppb)	Spike Recovery (%)	%RSD of Recovery (n=3)
10 B	No gas	0.9994	2.395	8.78	97	3.2	0.9999	0.342	31.40	100	7.8
23 Na	No gas	0.9995	0.162	2.60	89	5.8	0.9992	2.265	3.16	98	3.6
24 Mg	H ₂	0.9999	0.394	6.04	97	3.8	0.9997	1.472	36.40	103	3.7
27 Al	No gas	0.9997	0.895	3.00	100	1.2	0.9995	2.810	22.70	95	6.4
31 P	HEHe	1.0000	2.861	52.00	100	5.4	0.9991	3.336	61.50	99	3.0
39 K	H ₂	0.9996	0.172	4.63	107	0.9	0.9994	2.452	7.17	100	3.8
40 Ca	H ₂	0.9969	0.372	5.49	102	0.8	0.9994	1.434	64.50	103	4.7
47 Ti	He	1.0000	0.261	4.77	90	0.9	0.9996	0.919	12.90	93	2.2
51 V	He	0.9996	0.006	0.04	101	2.5	0.9999	0.032	0.40	100	3.5
52 Cr	HEHe	0.9998	0.095	2.45	95	0.4	0.9999	0.438	7.78	98	1.3
55 Mn	He	0.9997	0.007	0.14	104	2.3	0.9995	0.016	0.40	92	1.6
56 Fe	H ₂	0.9995	0.041	0.49	93	1.5	0.9999	0.307	6.82	95	0.5
60 Ni	He	1.0000	0.014	0.09	101	0.6	1.0000	0.085	0.57	96	1.1
63 Cu	He	0.9998	0.020	0.51	90	2.6	0.9994	0.048	1.95	104	1.3
66 Zn	He	0.9998	0.051	3.14	88	1.4	0.9968	8.098	61.30	ND*	ND*
95 Mo	He	0.9999	0.026	0.37	105	1.0	0.9996	0.448	4.79	97	6.4
107 Ag	He	0.9992	0.009	0.10	93	3.3	0.9999	0.059	1.03	88	2.7
111 Cd	He	0.9999	0.121	0.31	92	3.4	0.9999	0.049	0.22	99	1.5
118 Sn	He	0.9996	0.005	0.12	107	2.9	0.9998	0.004	0.09	88	0.4
137 Ba	He	1.0000	0.026	0.07	102	0.4	0.9992	0.132	0.61	96	5.5
208 Pb**	He	0.9998	<0.001	0.17	98	0.9	0.9999	<0.001	0.57	102	2.1

*Not determined as the unspiked concentration was much higher than the spiking level. **Pb was measured as the sum of the three most abundant isotopes, 206, 207, and 208.

Long-term stability study

The long-term stability of the 7900 ICP-MS for the routine analysis of LIB electrolyte solvent matrices was evaluated based on a six-hour continuous analytical run. A quality control (QC) was prepared from a sample of DMC/EMC spiked at a concentration of 20 ppb for the 21 target elements. The QC sample was run after every 10 DMC/EMC samples. Figure 2 shows the normalized QC recoveries (%) for all the analytes over six hours of analysis, using the initial 20 ppb calibration spike as the reference. Recoveries were all within $\pm 20\%$ (between 80 and 115%) throughout the six-hour run, with precision less than 10% RSD. The excellent QC stability shows the suitability of the 7900 ICP-MS for routine analysis of all target analytes in LIB electrolyte solvents. As well as demonstrating the good matrix tolerance of the system, the QC results confirm the ability of the ORS⁴ cell to control the matrix-based interferences on analytes such as Mg, P, and Cr.

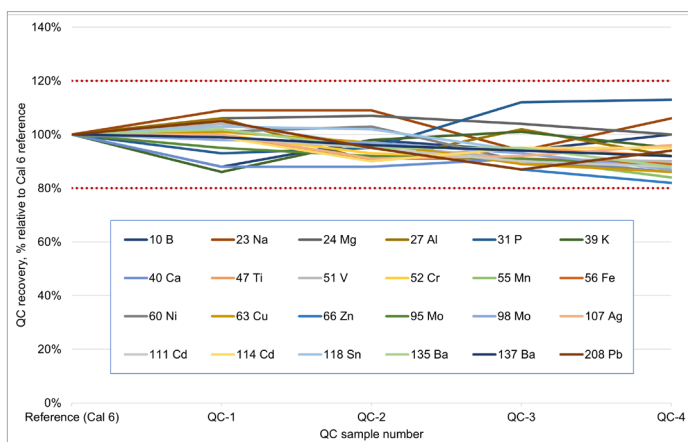


Figure 2. QC recoveries for a 20 ppb spike in the DMC/EMC sample run after every 10 unknown samples during a six hour analysis sequence.

He mode QuickScan and IntelliQuant screening

Among all commercial ICP-MS systems, Agilent instruments provide by far the best He collision mode performance for controlling polyatomic ion interferences. Agilent ICP-MS users can therefore use He mode to collect full mass range, "all-element" data that is practically free from common spectral overlaps. ICP-MS MassHunter includes a "QuickScan" function, which can be acquired as part of any spectrum analysis, adding only two seconds to the total acquisition time for each sample. QuickScan data acquired in He collision mode gives access to practically every isotope of nearly every element, making the Agilent ICP-MS ideally suited for screening unknown samples.

The spectra in Figure 3 illustrate the additional information available with He mode QuickScan. The three spectra on the left show the main, high intensity peaks in the mass range from 53 to 118 for the DMC blank, a DMC/EMC sample, and a DMC/EC sample. The spectra on the right show the same comparison, but with the intensity (Y-axis) scale zoomed by 100x to show the lower intensity peaks from trace contaminants. Since the DMC solvent is the major component of all the samples (DMC makes up 70% of DMC/EMC and DMC/EC), the differences between the spectra can mostly be attributed to the different contaminants present in the EMC and EC solvents.

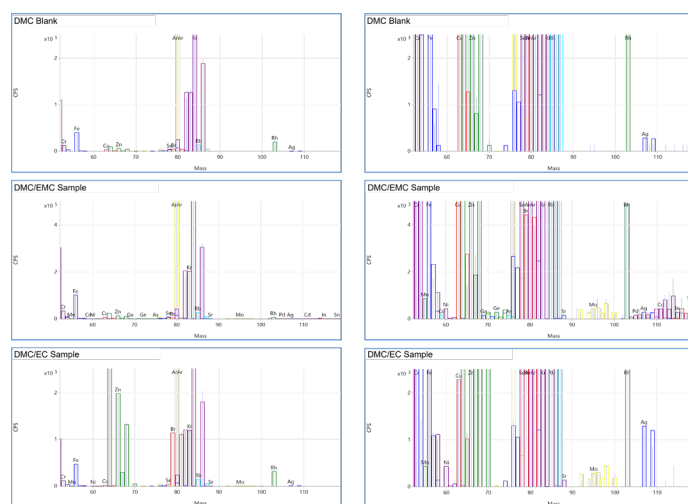


Figure 3. Left: QuickScan spectra of DMC blank (top), DMC/EMC sample (middle), and DMC/EC sample (bottom) showing comparison of high-level elements between mass 53 and 118. Right: Same comparison with intensity scale zoomed 100x to show differences in trace element content.

The spectra on the left-side of Figure 3 clearly show that the DMC/EC sample contained much higher levels of Zn (green outlined peaks between mass 64 and 70) and Br (red outlined peaks at mass 79 and 81). These major components were not present in the DMC blank, so must have come from the EC solvent. Rh (mass 103) is also visible in the DMC/EC sample and not apparent at the same level in the DMC/EMC. The high intensity purple outlined peaks between mass 78 and 86 are due to krypton (Kr) a trace contaminant in high purity argon.

The spectra on the right side of Figure 3 show the same samples and mass range, but with the Y-axis scale zoomed by a factor of 100 to show lower intensity peaks. Note that, with the full mass QuickScan spectrum acquisition taking only two seconds, low concentration elements only give a few raw counts. This means that the precision of measurements at low concentrations is not as good as it would be with the longer acquisition times used for conventional quantitative analysis. However, the right-side spectra indicate that the DMC/EMC sample contained significantly higher levels of several trace elements, Pd (mass 102–110), Cd (106 to 116), and Sn (red peaks between mass 112 and 118) than DMC/EC. In contrast, the DMC/EC sample contained a higher level of Ag (blue outlined peaks at mass 107 and 109).

QuickScan data is processed using IntelliQuant, a non-specific calibration approach that provides semiquantitative results for elements that were not included in the calibration standards. The IntelliQuant calibration uses the measured signal for a few reference elements to calculate semiquantitative concentrations for the remaining elements, based on the stored mass response curve of the ICP-MS. In this study, IntelliQuant results were calculated for the blank (unspiked) DMC/EMC and DMC/EC solvent mixes.

The semiquantitative concentrations can be reported in a conventional “all-element” concentration report, or displayed in a periodic table heat map view, as shown in Figure 4. The periodic table heat map uses different color intensities to represent the concentrations of the elements, with a darker color indicating a higher concentration. The periodic table view gives a quick visual indication of unexpected high concentration elements and variations between samples.

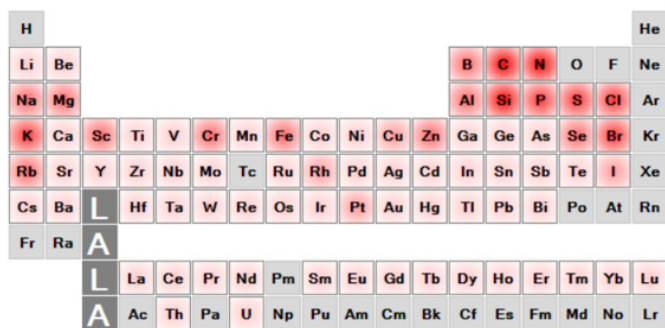


Figure 4. IntelliQuant periodic table heat map for DMC/EC sample showing darker shading (higher concentrations) for elements including Zn, Br, and Rh, highlighted in the QuickScan spectrum in Figure 3.

IntelliQuant results are often within about $\pm 20\%$ of the quantitative value, which is typically enough to identify a production or manufacturing problem or troubleshoot a contamination issue.

Conclusion

An Agilent 7900 ICP-MS method has been developed for the quantitative determination of 21 trace elements in the organic solvents commonly used in LIB electrolytes. The 7900 was fitted with the option gas (O_2/Ar) line to decompose the carbon matrix, and the sample introduction system was configured for organic sample analysis. Samples were delivered from an Agilent I-AS autosampler and the solvents were run directly, without dilution. The use of self-aspiration rather than the peristaltic pump for sample uptake provided a constant flow rate for the organic solvents, while avoiding the possibility of elemental contamination from the pump tubing material.

The 7900 method was based on standard addition calibrations in each solvent mix, and the analytical performance was evaluated in terms of linearity, sensitivity, precision, and spike recovery. Sub-ppb DLs were obtained for most of the analytes, and spike recoveries of 88–107% with excellent precision were achieved for all analytes where the spike amount could be measured reliably. The results demonstrate that the 7900 system can tolerate the organic solvent matrices and control carbon-based polyatomic spectral interferences, allowing for the direct determination of problematic elements in organic matrices. He mode QuickScan allowed additional, non-target elements to be measured semiquantitatively as part of the acquisition, providing a useful screening tool to monitor unexpected contaminants.

Excellent long-term stability for a six-hour sample analysis run showed that the 7900 system provides outstanding tolerance to organic sample matrices, allowing routine analysis of large batches of undiluted organic solvents. In addition, the 7900 ICP-MS provides high sensitivity, excellent control of spectral overlaps, and low DLs for all target elements in complex organic matrices. The Agilent ICP-MS method therefore offers improved performance compared to the ICP-OES methods traditionally used for organic solvent analysis in the LIB industry.

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