Quantifying Metal Impurities in Li-Ion Battery Raw Materials by ICP-MS/MS

Sensitive, robust analysis of 64 elements in lithium carbonate using the Agilent 8900 ICP-QQQ

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Figure 1. Lithium-ion battery pack.

Increasing need for battery power and storage

Demand for high-capacity, high-density lithium-ion (Li-ion) batteries is increasing rapidly, especially for use in consumer electronics, electric vehicles (EVs), and for storage associated with renewable electricity generation. The purity of the materials, such as lithium carbonate (Li_2CO_3) used in the battery electrolyte and cathode, has a major impact on the performance of the final device. To control the level of inorganic impurities in battery components, raw material suppliers and manufactures analyze the level of trace metals in the chemicals, typically using a sensitive, multi-element technique such as ICP-MS.

An Agilent 8900 Triple Quadrupole ICP-MS (ICP-QQQ) with m-lens provides the robustness, sensitivity, and interference removal capabilities for this application. The 8900's robust plasma and m-lens allow high matrix lithium salts to be measured after a single dilution step, minimizing the risk of contamination through multiple dilutions. Trace-level analysis of 64 elements was achieved using a single multi-tune method. The MS/MS configuration of the 8900 allows controllable reaction chemistry to be used for analytes that suffer spectral overlaps from polyatomic, isobaric, or doubly charged interferences (*1*).

Quantitative analysis of Li_2CO_3 using ICP-MS/MS

High purity Li_2CO_3 was bought from two manufactures. 0.25 g of Li_2CO_3 was dissolved in 250 g of high purity 1% HNO_3 (TAMAPURE-AA-100) and the solution was stabilized for 15 min to degas CO_2. Three sigma method detection limits (MDLs) were calculated from three measurements of the unspiked Li_2CO_3 sample, multiplied by the dilution factor (1000). The concentrations of all analytes were determined by method of standard addition (MSA) by spiking one of the Li_2CO_3 samples. Table 1 shows ng/kg to µg/kg level MDLs and low concentrations for most of the 64 analytes in the two Li_2CO_3 samples.

MDLs and quantitative impurity results

Table 1. Analytes, MDLs, and concentration of impurities in two Li_2CO_3 samples, A and B, corrected for the dilution factor (x1000). All units, mg/kg.

Q1/Q2, Analyte	Cell Gas	MDL	Li_2CO_3 Sample A	Li_2CO_3 Sample B
11 -> 11 B	No gas	0.032	0.089	0.049
23 -> 23 Na	O_2	0.055	0.599	2.29
24 -> 24 Mg	O_2	0.025	0.130	0.254
27 -> 27 Al	O_2	0.060	0.264	0.687
28 -> 28 Si	H_2	0.050	32.3	28.4
31 -> 47 P	O_2	0.016	<MDL	<MDL
32 -> 48 S	O_2	1.11	8.64	3.19
39 -> 39 K	O_2	0.020	0.306	0.195
40 -> 40 Ca	H_2	0.411	14.2	29.0
45 -> 61 Sc	O_2	0.001	0.001	<MDL
48 -> 64 Ti	O_2	0.010	<MDL	<MDL
51 -> 67 V	O_2	0.004	0.043	0.016
52 -> 52 Cr	O_2	0.001	0.029	0.020
55 -> 55 Mn	O_2	0.003	0.030	0.044
56 -> 56 Fe	O_2	0.014	0.207	0.187
59 -> 59 Co	H_2	0.007	0.042	0.260
60 -> 60 Ni	O_2	0.010	0.023	0.012
63 -> 63 Cu	O_2	0.006	0.054	0.037
66 -> 66 Zn	O_2	0.078	1.12	0.650
71 -> 71 Ga	O_2	0.003	<MDL	<MDL
72 -> 72 Ge	O_2	0.001	0.001	0.001
75 -> 91 As	O_2	0.021	0.225	0.227
78 -> 78 Se	H_2	0.009	0.022	0.014
85 -> 85 Rb	No gas	0.0003	0.001	0.0004
88 -> 88 Sr	No gas	0.002	0.014	0.043
89 -> 105 Y	O_2	0.008	0.173	0.533
90 -> 106 Zr	O_2	0.007	0.011	<MDL
93 -> 93 Nb	No gas	0.0001	0.0001	0.0001
95 -> 95 Mo	H_2	0.001	<MDL	<MDL
101 -> 101 Ru	O_2	0.0003	<MDL	<MDL
103 -> 103 Rh	No gas	0.0001	<MDL	<MDL
105 -> 105 Pd	H_2	0.002	<MDL	0.008
107 -> 107 Ag	O_2	0.001	<MDL	<MDL
111 -> 111 Cd	H_2	0.002	<MDL	<MDL
118 -> 118 Sn	O_2	0.001	0.003	0.002
121 -> 121 Sb	No gas	0.001	<MDL	0.001
125 -> 125 Te	No gas	0.004	<MDL	<MDL
133 -> 133 Cs	H_2	0.002	0.007	0.003
137 -> 137 Ba	O_2	0.011	0.165	0.349
139 -> 139 La	No gas	0.002	0.013	0.006
146 -> 146 Nd	No gas	0.001	0.001	<MDL
147 -> 147 Sm	No gas	0.001	<MDL	<MDL
153 -> 153 Eu	No gas	0.0002	<MDL	<MDL
157 -> 173 Gd	O_2	0.001	0.001	0.003
159 -> 159 Tb	No gas	0.0002	<MDL	<MDL
163 -> 163 Dy	No gas	0.001	<MDL	<MDL
165 -> 165 Ho	No gas	0.0001	<MDL	<MDL
166 -> 166 Er	No gas	0.0003	<MDL	<MDL
169 -> 169 Tm	No gas	0.0001	<MDL	<MDL
172 -> 172 Yb	No gas	0.0002	<MDL	<MDL
175 -> 175 Lu	No gas	0.0001	<MDL	<MDL
178 -> 178 Hf	H_2	0.001	<MDL	<MDL
181 -> 181 Ta	No gas	0.0003	<MDL	<MDL
182 -> 182 W	No gas	0.002	0.009	0.011
185 -> 185 Re	No gas	0.0003	<MDL	<MDL
193 -> 193 Ir	No gas	0.001	<MDL	<MDL
195 -> 195 Pt	No gas	0.001	<MDL	<MDL
197 -> 197 Au	No gas	0.001	<MDL	<MDL
208 -> 208 Pb	No gas	0.005	0.093	0.075
209 -> 209 Bi	No gas	0.0004	<MDL	<MDL
232 -> 232 Th	No gas	0.0001	<MDL	<MDL
238 -> 238 U	No gas	0.001	<MDL	<MDL

Table 1 continues...

Table 1 continued

Checking the purity-level of Li_2CO_3

According to the suppliers of Li_2CO_3 samples A and B, the material purity was 99.99 and 99.9%, respectively. To check the manufacturers' purity claims, the concentrations of all 64 elements measured in each sample were summed to give a total impurity level, as shown in Table 2. By subtracting the total impurities, purity levels of 99.994% (sample A) and 99.993% (sample B) were obtained, confirming the manufacturers' claimed purities. The study demonstrates the suitability of the 8900 ICP-QQQ for the analysis of a wide range of potential analytes of interest in the complex, high matrix Li_2CO_3 samples, run using a single multi-tune method.

Table 2. Calculated purity level of Li_2CO_3 samples A and B.

	Li_2CO_3 Sample A	Li_2CO_3 Sample B
Impurity (mg/kg)	58.9	66.5
Purity (%)	99.994	99.993

1. Handbook of ICP-QQQ Applications using the Agilent 8800 and 8900, Agilent publication 5991-2802EN

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