

Analysis of Trace Impurities in Lithium Carbonate

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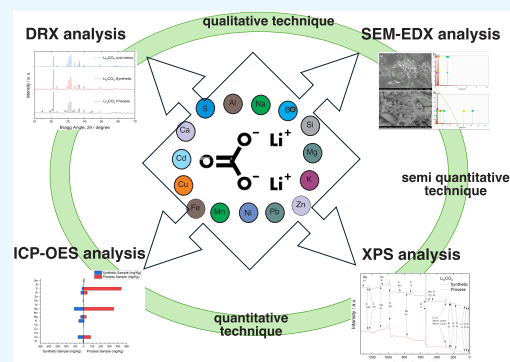


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ABSTRACT: Lithium carbonate (Li_2CO_3) is a critical raw material in cathode material production, a core of Li-ion battery manufacturing. The quality of this material significantly influences its market value, with impurities potentially affecting Li-ion battery performance and longevity. While the importance of impurity analysis is acknowledged by suppliers and manufacturers of battery materials, reports on elemental analysis of trace impurities in Li_2CO_3 salt are scarce. This study aims to establish and validate an analytical methodology for detecting and quantifying trace impurities in Li_2CO_3 salt. Various analytical techniques, including X-ray diffraction (XRD), scanning electron microscopy–energy-dispersive X-ray spectroscopy (SEM-EDX), X-ray photoelectron spectroscopy (XPS), and inductively coupled plasma optical emission spectroscopy (ICP-OES), were employed to analyze synthetic and processed lithium salt. X-ray diffraction patterns of Li_2CO_3 were collected via step-scanning mode in the $5\text{--}80^\circ 2\theta$ range. SEM-EDX was utilized for particle morphology and quantitative impurity analysis, with samples localized on copper tape. XPS equipped with a hemispherical electron analyzer was employed to analyze the surface composition of the salt. For ICP-OES analysis, a known amount of lithium salt was subjected to acid digestion and dilution with ultrapure water. Multielemental standard solutions were prepared, including elements such as Al, Cd, Cu, Fe, Mn, Ni, Pb, Si, Zn, Ca, K, Mg, Na, and S. Results confirmed the presence of the zabuyelite phase in XRD analysis, corresponding to the natural form of lithium carbonate. SEM-EDX mapping revealed impurities of Si and Al, with low relative quantification values of 0.12% and 0.14%, respectively. XPS identified eight potential impurity elements, including S, Cr, Fe, Cl, F, Zn, Mg, and Na, alongside Li, O, and C. Regarding ICP-OES analysis, performance parameters such as linearity, limit of detection (LOD), and quantification (LOQ), variance, and recovery were evaluated for analytical validation. ICP-OES results demonstrated high linearity (>0.99), with LOD and LOQ values ranging from 0.001 to 0.800 ppm and 0.003 to 1.1 ppm, respectively, for different elements. The recovery rate exceeded 90%. In conclusion, the precision of the new ICP-OES methodology renders it suitable for identifying and characterizing Li_2CO_3 impurities. It can effectively complement solid-state techniques such as XRD, SEM-EDX, and XPS.



INTRODUCTION

Lithium has traditionally been used in a variety of applications ranging from pharmaceuticals to the manufacture of air treatment systems.¹ The main sources to extract lithium are ores (spodumene, lepidolite, petalite, among others), brines, and mineral groundwater.² Due to its low atomic mass, Li has a high charge, and the power to weight ratio enables it to store and transmit energy, making it an important component for the construction of rechargeable lithium-ion batteries (LIBs).¹ With their high energy density, low self-discharge rate, and high open-circuit voltage, LIBs can be widely used in electronic devices, as well as in hybrid electric vehicles (HEVs) and electric vehicles (EV).¹ In this field, several studies have estimated that by 2025 this industry will account for almost 66% of the current Li production worldwide, and the global LIB market is going to reach a value of US\$75 billion by 2027.^{1,3,4} Typically, a LIB consists of a cathode, anode, separator, and electrolyte, LiFePO_4 , Li_2MnO_4 , and LiCoO_2 are the main materials to prepare cathodes.⁵ In order to prepare such cathode materials, large amounts of lithium carbonate

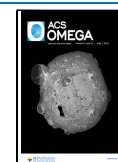
(Li_2CO_3) are required, followed by lithium hydroxide ($\text{LiOH}\cdot\text{H}_2\text{O}$) with a very high chemical purity, and battery-grade compounds (over 99.5%).⁶ Lithium carbonate and hydroxide impurities classify the final product as battery or technical grade, for instance, technical grade lithium carbonate is generally about 99%, which is slightly lower than battery grade lithium carbonate ($>99.5\%$) (SQM). In the case of lithium hydroxide, battery grade of monohydrated salt is at least 99.3%.⁷ Technical grade lithium salt is often used in fiberglass and general glass manufacturing, ceramics, and enamel products, as a catalyst for esterification processes, and as an additive in aluminum electrolysis melts, among others, while battery grade lithium is used for cathode

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preparation. Moreover, the quality of lithium salts determines the performance of the battery, since high-purity chemical products can avoid short circuits because of some deformation occurring in the electronic wafers, resulting in performance loss.⁸ In this context, impurity analysis of lithium carbonate and hydroxide are crucial for suppliers of this raw material and the users.^{1,3,4} In Chile, both lithium salts are obtained from Brines of Salar de Atacama, which are evaporated and processed in order to eliminate the maximum of several ions present in these kinds of salts. Concerning lithium carbonate, the main impurities that might be present are Ca, Cd, Cu, Fe, Mn, Ni, Pb, Zn, K, Mg, Si, B, Na, Al, and S. The concentrations of these ions in the final product are at the ppm level, since lithium carbonate battery grade has a purity of 99.5 wt %, while technical grade salt is 99.3 wt %. In this context and considering the relevance in the quantitative identification of impurities in lithium salts, the aim of this work is to setup an analytical methodology to quantify lithium carbonate impurities, such as Ca, Cd, Cu, Fe, Mn, Ni, Pb, Zn, K, Mg, Si, B, Na, Al, and S, by ICP-OES and to characterize such impurities by solid state techniques (X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), and scanning electron microscopy–energy-dispersive X-ray spectroscopy (SEM-EDX)).

EXPERIMENTAL SECTION

Reagents. High-purity HNO₃ (Sigma-Aldrich), ultrapure water, and calibration standards for ICP (Merck) were used, considering the following elements: Ca, Cd, Cu, Fe, Mn, Ni, Pb, Zn, K, Mg, Si, B, Na, Al, and S. Additionally, a commercial Li₂CO₃ (99% purity) was used as a synthetic sample, and Li₂CO₃ from local industry (process sample) was kindly donated for this study.

Preparation of Standard Solutions and Sample. A matrix solution 1% (w/v) of Li₂CO₃ was prepared for calibration curve standards. Briefly, 5 g of Li₂CO₃ was digested with 50 mL of HNO₃ during 120 min at 120 °C. Samples were cool-down for 15 min, ultrapure water was added to complete 500 mL. This matrix solution was used to prepare multielement standard solutions.

Three multielement standard solutions were prepared in a matrix solution of 1% Li₂CO₃ for calibration curves: Multielement solution 1 (20 mg/L) contained the following analytes: Al, Cd, Cu, Fe, Mn, Ni, Pb, Si, and Zn. Multielement solution 2 (20 mg/L) contained Ca, K, Mg, and Na. The third standard solution contained 20 mg/L S. From multielement solutions 1–3, calibration curves were prepared as shown in Tables S1–S3 for inductively coupled plasma optical emission spectrometry (ICP-OES) analysis.

Three independent replicates of synthetic samples were prepared as follows: 0.5 g of 99% Li₂CO₃ was digested with 5 mL of concentrated HNO₃ for 120 min at 120 °C in a hot plate. Then, samples were cooled down for 15 min and filtered through 0.45 μm filter paper. Finally, the digested samples were completed to 50 mL with ultrapure water and stored for ICP-OES analysis. All details can be found in Tables S1–S3.

Analytical Parameters of ICP-OES Instrument. In order to select the optimal parameters for ICP-OES analysis, selected wavelengths, plasma view and purge gas flow were defined according to the previous report (Referencial). Every analysis was carried out by ICP-OES (optima DV7000, PerkinElmer, USA). Detailed information about final instrument parameters can be found in Table S4, and wavelengths, plasma views, and

other conditions are shown in Table S5. Particularly, auxiliary gas flow was adjusted to 0.5 L/min and the RF power was modified to 1200 W for sulfur (S) analysis.

Matrix Effect Assay. An additional assay was done by using a carbonate lithium matrix of 2% in order to verify whether matrix produces effect in the sensitivity of the method.

CHARACTERIZATION OF LITHIUM CARBONATE IMPURITIES

Analytical Parameters X-ray Diffraction (XRD). The crystalline phase of sample was identified with X-ray diffraction (XRD, D8 Advance Bruker). XRD patterns were collected by step-scanning mode in the range of 5–80° 2θ with a step of 0.020° 2θ sec⁻¹. The X-ray generator worked at 40 kV and 30 mA. All the data were processed by EVA software using the database of the ICDD (International Center for Diffraction Data)

Analytical Parameters X-ray Photoelectron Spectroscopy (XPS). The surface composition of lithium carbonate was analyzed by X-ray photoelectron spectroscopy (XPS) using a Surface Analysis Station 1 electron spectrometer (XPS RQ300/2, Staib Instrumente GmbH, Langenbach, Germany) equipped with a hemispherical electron analyzer DESA 150 detector/2700 V and nonmonochromatic Al Kα (1486.6 eV) X-ray source. XPS spectra were collected by scan range of 1350–0° eV with a step size of 0.7 eV and step time of 0.2 s.

Analytical Parameters Scanning Electron Microscopy (SEM-EDX). Particle morphology and quantitative analysis for the determination of impurities in lithium carbonate were obtained from a scanning electron microscope (SEM) (Hitachi, model SU5000 (ZrO/W Schottky emit electron gun), equipped with XFlash 6I30 detectors (Bruker) and a STEM detector EDX for relative quantification.

RESULTS AND DISCUSSION

Characterization of Lithium Carbonate Impurities by XRD. Samples were characterized first by X-ray powder diffraction. According to this analysis, both samples are crystalline and composed of Li₂CO₃, as shown in Figure 1. Moreover, synthetic sample seems to be a single phase while the process sample contains minor impurities identified as a:

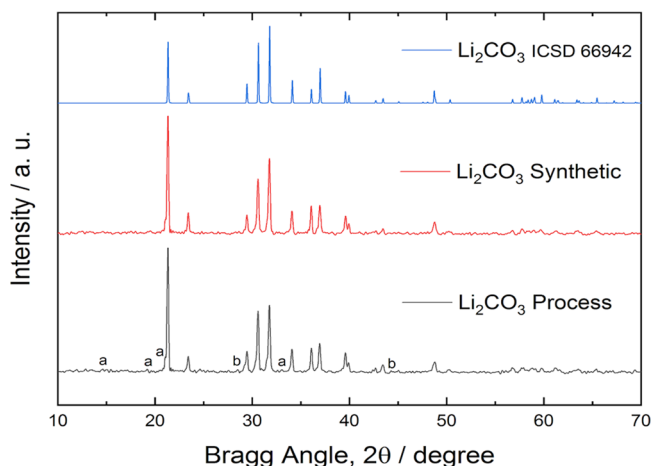


Figure 1. Powder X-ray diffraction patterns of Li₂CO₃ from process (black), synthetic (red) and reference from ICSD (blue). Characters *a* and *b* indicate the reflections associated with MgSO₄·7H₂O and MgO, respectively.

MgSO₄·7H₂O and *b*: MgO. Such impurities are not surprising, since Mg is associated with Li in brines. It is well-known that the detection limit of this technique is over 5% w/w for side phases; therefore, different solid-state characterization techniques are employed in order to determine the impurities in Li₂CO₃ samples.

Both samples, synthetic and processed, were characterized by powder XRD. The powder diffraction pattern of the synthetic sample is shown in Figure 1. Single crystalline phase is observed and thus far another impurity was not detected. For the processed sample, shown in the same figure, several crystalline phases were detected by the match algorithm employed in the EVA software. These minority phases might be associated with impurities that can be quantitatively determined by another technique.

Based on the findings, among all anticipated impurities, X-ray diffraction analysis revealed the presence of the Li₂CO₃ pattern, along with magnesium sulfate and oxide exclusively. The identification of Mg was somewhat challenging, which is not unexpected given the sensitivity of this technique. As mentioned before, the limit of detection of this technique is over 5% w/w; therefore, it is expected to identify only one or even no impurities.⁹

Considering the XRD pattern, it aligns well with previous reports where it was able to discern the lithium carbonate successfully.¹⁰ In this context, an additional mineralogical technique was employed to enhance the detection of impurities or identify new elements.

Characterization of Lithium Carbonate Impurities by XPS. Survey spectra were collected for both samples as shown in Figure 2. As expected from the detection limit of this

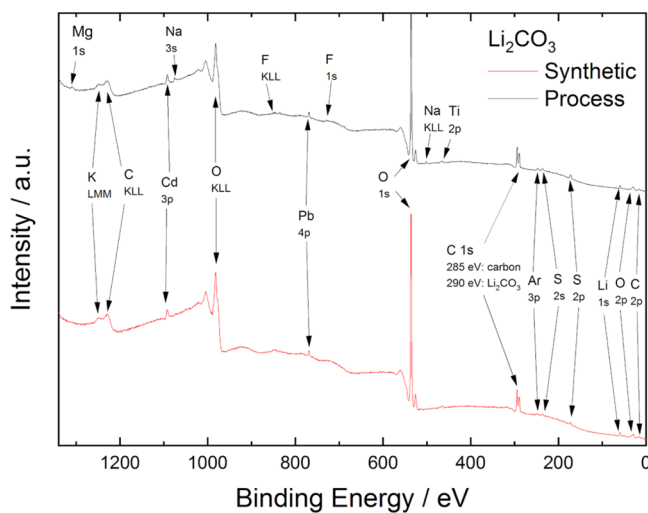


Figure 2. XPS survey spectra of Li₂CO₃ samples. The process sample (black line) reveals additional signals that are not present in the synthetic sample (red line).

technique (from 0.1 at%),¹¹ several signals, besides the expected ones from Li, O, and C, are present in both spectra. The association of these signals to photoelectrons emitted by elements was carried out by using the database in the CasaXPS software. According to this analysis, some impurities are common to both samples (K, Cd, and Pb), while additional impurities were found in the process sample (Mg, Na, and F).

Consequently, X-ray photoelectron spectroscopy (XPS) was utilized, leading to improved results, as shown before. Both

XPS spectra of Li₂CO₃ are consistent with previous reports.¹² For instance, the C 1s spectrum contains a main narrow component at 290.1 eV which corresponds to carbon in the carbonate ion. The O 1s spectrum consists of two lines showed to be at 531.8 eV, and at 533.2 eV. The low intensity of this second line of O 1s, is due to sample warming.¹²

Characterization of Lithium Carbonate Impurities by SEM-EDX. SEM images of the surfaces of Li₂CO₃ samples are shown in Figure 3. Synthetic and process samples crystallize

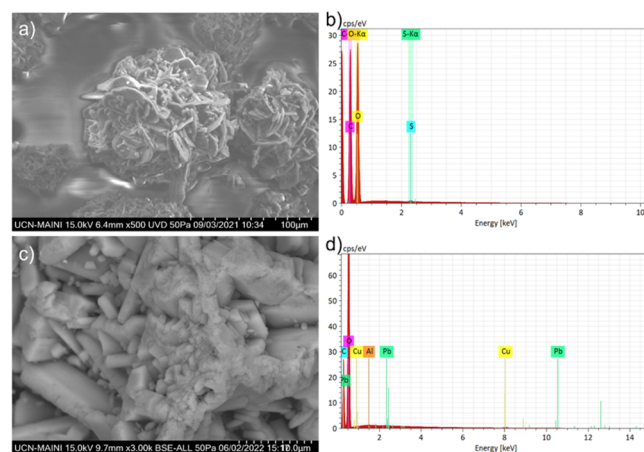


Figure 3. SEM images of Li₂CO₃ surfaces of (a) synthetic sample and (c) process sample. EDS mapping of Li₂CO₃ samples: (b) synthetic and (d) process.

with very different morphology: The synthetic sample consists of agglomerate of large crystallites forming a kind of nest (Figure 3a), while the process sample shows well-shaped prismatic crystallites (Figure 3c). To confirm the stoichiometry of the samples and aiming to find additional impurities, EDS mapping was performed on both samples. The EDS spectra of synthetic and process samples, shown in Figure 3b,d, respectively, is in good agreement with the qualitative analysis from XPS. According to previous studies,¹³ the typical detection limit for this technique is around 0.08 wt %. Consequently, the relative abundance considered in this analysis ranged from 0.1 to 5%, taking into account impurities such as Si, Na, Pb, S (in the case of synthetic samples), Ag, and Cu. The obtained results are promising, as it was previously reported that the same technique did not detect impurities. The authors noted that SEM/EDX only identified carbon and oxygen within the captured SEM segment, with no traces of other metals detected.¹⁰ It is important to bear in mind that this is a punctual technique and should not be considered as representative of the whole sample. Therefore, ICP-OES was also employed for impurities detection and quantification. All details about SEM-EDX analysis can be found in Table S6.

Analytical Performance of ICP-OES for Quantification of Lithium Carbonate Impurities. Calibration curves were constructed following the methodology outlined in a previous report,¹⁴ with certain parameters, such as the viewing mode of the plasma and wavelength, adjusted to optimize performance. The linearity of the calibration curves for each analyte was assessed, yielding values exceeding >0.99, and the sensitivity for each analyte is presented in Table S7.

Limits of detection (LOD) and quantification (LOQ) were calculated using eqs 1 and 2, respectively. The results for each analyzed element (in μg/L) are summarized in Table 1.

$$\text{LOD} = \frac{3S_y/x}{\text{slope}} \quad (1)$$

$$\text{LOQ} = \frac{10S_y/x}{\text{slope}} \quad (2)$$

Table 1. Limit of Detection and Quantification of Lithium Carbonate Impurities by ICP-OES

analyte	LOD (mg/L)	LOQ (mg/L)	analyte	LOD (mg/L)	LOQ (mg/L)
Al	0.0054	0.0180	Mn	0.0014	0.0047
Ca	0.0200	0.0660	Na	0.0820	0.2750
Cd	0.0015	0.0051	Ni	0.0052	0.0180
Cu	0.0016	0.0054	Pb	0.0068	0.0230
Fe	0.0150	0.0490	Zn	0.0016	0.0054
K	0.0430	0.1440	Si	0.0160	0.0520
Mg	0.0110	0.0380	S	0.0990	0.3300

Limited published work has been done regarding optimization and validation of methodologies for quantification of impurities in a matrix of lithium carbonate. In this investigation, values of LOD and LOQ are slightly higher than a previous report;¹ nevertheless, results are still favorable considering that measurements were performed using lithium carbonate as matrix.

Along with already discussed validation parameters, spike recovery was performed to evaluate accuracy and precision of this method by repeatability in synthetic and processed samples. %RSD results are summarized in Table 2 in addition to spike recovery mean in percentages.

Table 2. Spike Recovery and Precision of Methodology Used for Quantification of Lithium Carbonate Impurities

element and wavelength (nm)	synthetic Li ₂ CO ₃		processed Li ₂ CO ₃	
	recovery (%)	RSD (%) ^a	recovery (%)	RSD (%) ^a
Al 396.153	115	3.45	102	0.41
Ca 422.673	97	4.34	104	0.70
Cd 226.502	80	0.22	103	1.22
Cu 327.393	90	0.17	105	1.44
Fe 238.204	98	4.79	104	0.81
K 766.490	106	2.20	97	1.04
Mg 279.077	107	0.44	98	1.21
Mn 257.610	85	0.44	103	1.32
Na 589.592	99	0.89	104	2.06
Ni 227.022	78	0.15	103	0.48
Pb 220.353	89	0.30	103	0.87
Zn 213.857	90	0.44	99	1.16
Si 251.611	92	3.41	97	0.99
S 181.975	102	7.22	103	0.88

^aRSD corresponds to three independent replicates.

Observing Table 2, it is evident that the precision, expressed as %RSD in recovery assays, consistently falls below 10%, with the majority even below 5%, for both types of samples. These findings indicate that our methodology demonstrates high reproducibility with minimal random errors. In comparison to the anticipated precision, as outlined in the AOAC Official Methods of Analysis "Guidelines for Standard Method Performance Requirements", where %RSD is expected to be

within the range of 15–30%, our achieved precision levels are notably favorable.¹⁵

Furthermore, accuracy was assessed by examining spike recoveries for both synthetic and process samples. The results, presented in Table 1, indicated recoveries ranging from 80 to 115%. According to the AOAC Official Methods of Analysis,¹⁵ the expected recovery as a function of the analyte concentration should fall within the range of 40–120%. In the case of synthetic samples, certain analytes such as Cd, Cu, Mg, Mn, Ni, and S fell within these specified percentages, ranging from 60 to 115% for Pb and Zn recovery and from 80 to 110% for Al, Fe, K, Na, Ca, and Si recovery. While Al recovery slightly exceeded the anticipated value, the %RSD value ensures that the spike recovery remains within the expected range. For processed samples, recoveries ranged from 97 to 104%, aligning with the suggested range in the AOAC Official Methods of Analysis.¹⁵

Quantification of Lithium Carbonate Impurities by ICP-OES. The analysis encompassed both synthetic and process samples of Li₂CO₃. A minimum of three independent replicates, coupled with three analytical replicates, were conducted. The findings are succinctly presented in Figure 4,

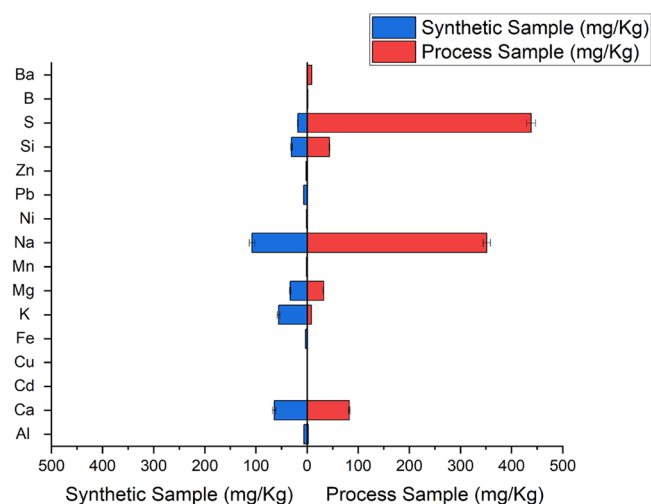


Figure 4. Quantification of lithium carbonate impurities by ICP-OES. Error bars correspond to standard deviation of three replicates.

indicating that sulfur (S), sodium (Na), and calcium (Ca) emerge as the predominant impurities in the process sample, followed by silicon (Si), magnesium (Mg), barium (Ba), and potassium (K). In the case of the synthetic sample, sodium (Na), calcium (Ca), and potassium (K) take precedence, succeeded by silicon (Si) and magnesium (Mg), as depicted in Figure 4.

During the ICP-OES analysis, it was observed that only Ba, Cd, and B were below the limit of detection (LOD) in the synthetic sample. In the case of the process sample, Pb and Zn were not detected using the current methodology. Cu and Fe were present as impurities in lower concentrations in the synthetic and process samples, respectively. While Na was the most abundant impurity in both samples, S was consistently higher in the process sample. All details about concentrations can be found in Table S8.

CONCLUSION

This study marks the inaugural exploration of lithium carbonate impurity analysis, utilizing ICP-OES alongside solid-state techniques such as XPS, XRD, and SEM. Solid-state methodologies have proven instrumental in providing initial insights into potential impurities within lithium salt samples. It is important to note that the relative abundances obtained via SEM-EDX lack a direct correlation with ICP-OES quantification, as SEM merely offers preliminary data in this investigation.

Regarding ICP-OES methodology, it has exhibited exceptional accuracy and precision in quantifying approximately 14 trace elements, considered impurities in Li_2CO_3 . These findings are evidently indicative of the entire sample, especially when compared with solid-state techniques. This is primarily due to the pretreatment of the sample, such as acid digestion, which ensures complete dissolution and yields more precise and accurate results.

Although the ICP-OES technique possesses certain limitations compared to more precise methods like ICP-MS, its accessibility makes it a favorable analytical approach for laboratories. This accessibility allows laboratories to adopt a contemporary and essential method, particularly given the widespread applications of lithium.

In conclusion, this study represents an initial step toward understanding the analytical challenges associated with assessing lithium carbonate purity. Accurate quantification of lithium impurities is crucial for categorizing the salt as technical or battery grade, directly impacting its industry pricing.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acsomega.4c00085>.

Additional experimental details, concerning ICP-OES analysis. SEM-EDX relative abundances, linearity and impurities quantification by ICP-OES (PDF)

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Author Contributions

A.S. and A.J.: conceptualization, methodology, investigation, and data curation, and formal analysis. R.C.: investigation, writing—review and editing. K.G.: conceptualization, methodology, investigation, and data curation, formal analysis, writing—original draft and review/editing, visualization, supervision, project administration, funding acquisition. All authors have given approval to the final version of the manuscript.

Notes

The authors declare no competing financial interest.

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