Application note

Quality Control of Li_2CO_3 from Salars by XRD analysis using ARL X'TRA Companion benchtop XRD

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Figure 1. ARL X'TRA Companion diffraction system

Introduction

This application note presents the X-ray diffraction (XRD) analysis of lithium carbonate (Li_2CO_3) from salars. Salars are salt flats or salt pans where lithium is extracted as Li_2CO_3 or LiOH from brines or saltwater through a series of evaporation steps. Lithium carbonate is widely used in the production of batteries, ceramics, and glass, among others. Its purity and crystallinity are crucial to ensure the quality of the final product.

XRD is a non-destructive technique that provides structural information about crystalline materials. It is commonly used to identify and quantify the phases present in a sample and to determine their crystalline structure.

The results of XRD analysis provide valuable information about the quality and purity of Li_2CO_3 from salars, which is essential for its application in various industries.

Instrument

The Thermo Scientific™ ARL™ X'TRA Companion (c.f. Figure 1) is a simple, easy-to-use bench top XRD system for process control and more advanced applications.

The ARL X'TRA Companion uses a θ/θ goniometer (160 mm radius) in Bragg-Brentano geometry and a 600 W X-ray source (Cu or Co). The radial and axial collimation of the beam is controlled by divergence and Soller slits, while air scattering is reduced by a variable beam knife. An integrated water chiller is available as an option.

Due to the state-of-the art solid state pixel detector (55x55µm pitch) the ARL X'TRA Companion provides very fast data collection and comes with one-click Rietveld quantification capabilities and automated result transmission to a LIMS.

Experimental

Two powdered samples of Li_2CO_3 from salars were measured in reflection geometry using an ARL X'TRA Companion with Cu Kα radiation (Ni filter). Each sample was measured for 10 minutes. Phase quantification was performed with Profex[1] (BGMN algorithm) using a fundamental parameters approach.

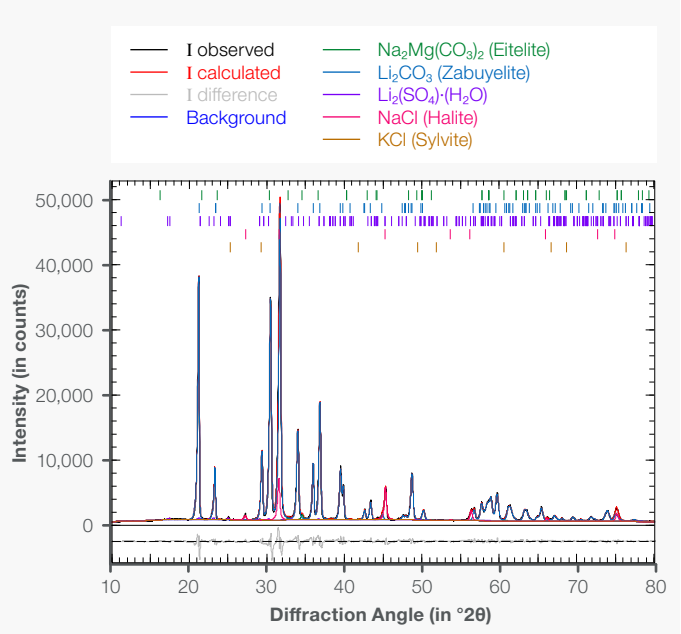

Figure 2. Rietveld fit of Li_2CO_3 sample A (10 minutes)

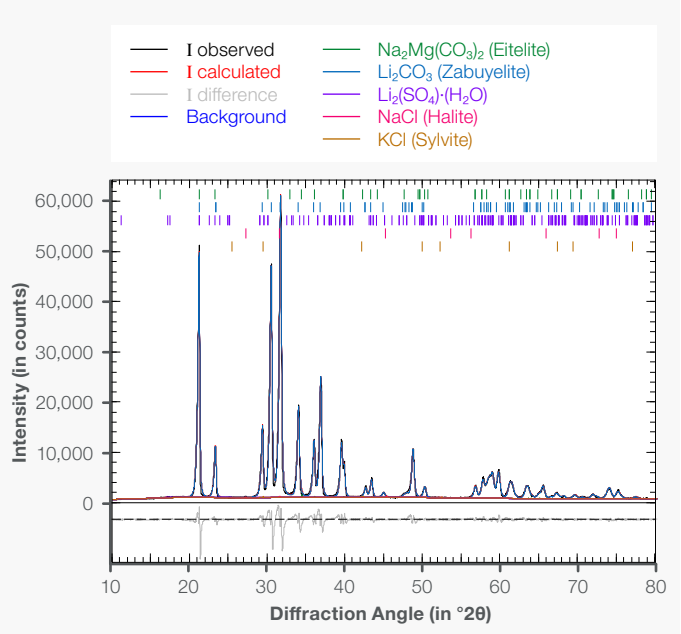

Figure 3. Rietveld fit of Li_2CO_3 sample B (10 minutes)

Phase	Sample A (in wt%)	Sample B (in wt%)
$Na_2Mg(CO_3)_2$ (Eitelite)	0.7 (1)	0.2 (1)
Li_2CO_3 (Zabuyelite)	95.3 (2)	99.5 (2)
$Li_2(SO_4)\cdot H_2O$	0.7(1)	0.2 (1)
NaCl (Halite)	3.3(1)	0.1 (1)
KCl (Sylvite)	0	0
Total Li (XRD/Reference)	18.0 / 17.2	18.7 / 18.5

Table 1. Results of Rietveld refinement on Li_2CO_3 samples (3σ in brackets)

Results

In Figure 2 and 3 the refinements of two Li_2CO_3 samples are shown.

Table 1 shows results from the refinements. The two samples are of different quality. Ascertainment of quality is important because phase-pure material with high total Li content is essential for further processing. The Li content can be indirectly ascertained from Rietveld refinements assuming stochiometric phases with known Li content. Sample A is contaminated with 3.3 wt% NaCl and other minor phases, which results in a total Li amount of 18.0 wt%;this is in good agreement with the reference value of 17.2 wt% from chemical analysis. Sample B is of better quality and contains minor contaminations, which results in a total Li amount of 18.7 wt% (reference: 18.5 wt%). Both samples are completely crystalline.

Conclusion

XRD is a powerful technique to assess the quality and purity of Li_2CO_3 because it can indirectly measure the total Li content of a sample as well as contaminants which could impact the price and further processing of the material. The ARL X'TRA Companion XRD can collect data on Li_2CO_3 in only 10 minutes, which allows the identification of contaminants and the quantification of the whole phase content. The one-click Rietveld refinement based on a fundamental parameter approach is an extremely robust method with high reproducibility. Thanks to these qualities, the ARL X'TRA Companion is the perfect solution for any process control or quality control task.

Reference

1. N. Döbelin, R. Kleeberg, *J. Appl. Crystallogr.* **2015**, *48*, 1573-1580.

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