

Application Note AN-T-251

Carbonate in lithium carbonate by potentiometric titration

Determination of carbonate in lithium carbonate by potentiometric titration according to draft standard ISO 10662

Lithium carbonate is one of the main lithium salts in cathode materials used for lithium-ion batteries. Accurate carbonate and overall purity determination in this raw material are essential because even trace impurities can affect key physicochemical properties, ultimately influencing electrochemical behavior in downstream applications. This is especially significant in battery-grade materials, where strict compositional tolerances are required to ensure consistent manufacturing performance, long-term cell stability, and overall safety. This Application Note describes carbonate content determination in lithium carbonate on an automated OMNIS system with Dis-Cover lids. The ISO 10662 draft method quantifies the main content of lithium carbonate to enable a precise assessment of material purity.

SAMPLE AND SAMPLE PREPARATION

This method is applicable to a concentration of battery-grade lithium carbonate (Li_2CO_3) greater than 95.00%. The samples were dried at 250 °C for two hours and allowed to cool down before use.

EXPERIMENTAL

The analysis is carried out on an OMNIS Advanced Titrator with an OMNIS Sample Robot equipped with a dUnitrode and Dis-Cover lids (Figure 1). A blank analysis is performed with a start volume of hydrochloric acid and titrated until after the equivalence point.
Samples are titrated with hydrochloric acid until after the second equivalence point.



Figure 1. OMNIS Sample Robot and OMNIS Advanced Titrator equipped with a dUnitrode and Dis-Cover lids.

RESULTS

The samples were tested in duplicate on one day, and once again on the following day. Results are displayed in Table 1.

Table 1. Results of the carbonate determination of lithium carbonate samples.

Sample (n = 3)	Content mean value (%)	SD(rel) in %
A	100.37	0.18
B	98.62	0.42
C	99.87	0.96

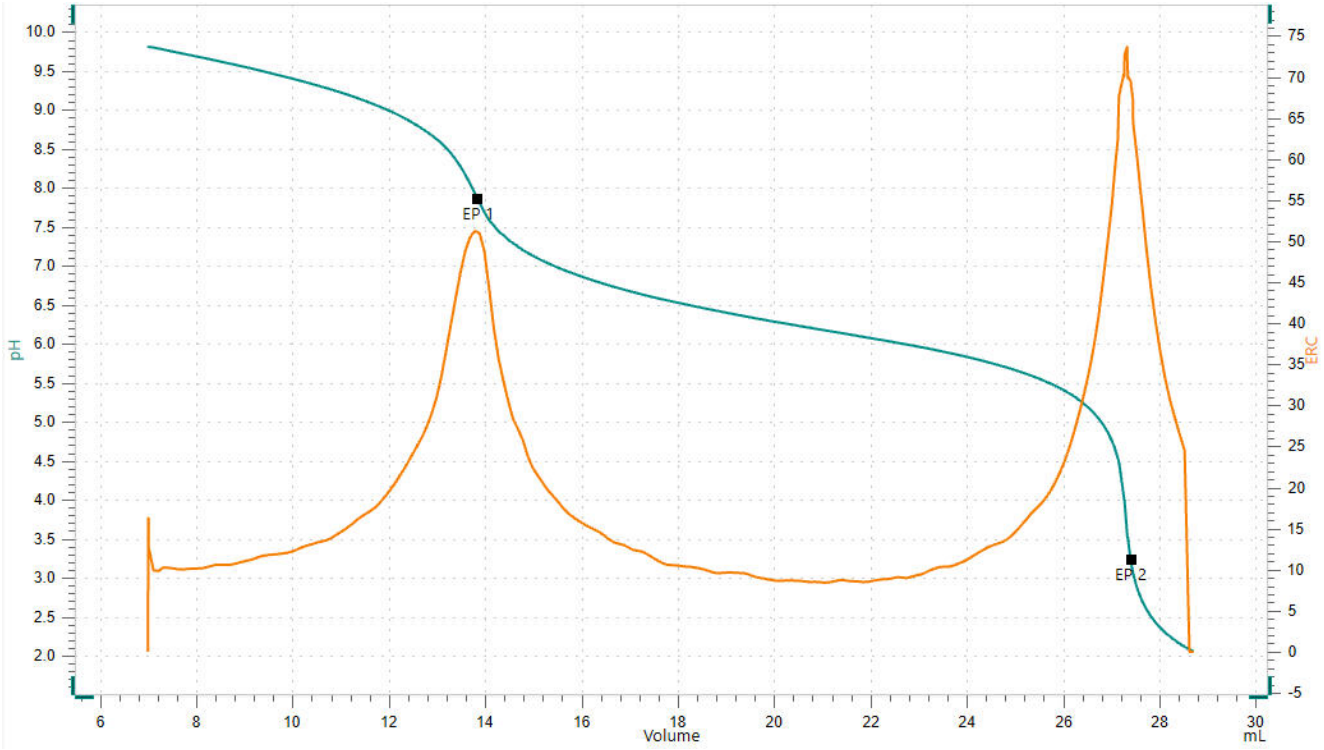


Figure 2. Titration curve of the carbonate content in lithium carbonate.

CONCLUSION

The OMNIS Titrator and OMNIS Sample Robot with Dis-Cover lids provide a fast and reliable determination of carbonate in lithium carbonate according to draft standard ISO 10662. The OMNIS system can be expanded to other titration applications as required.

CONTACT

Metrohm AG
Ionenstrasse
9100 Herisau
info@metrohm.com

CONFIGURATION



OMNIS Professional Titrator with magnetic stirrer

Innovative, modular potentiometric OMNIS Titrator for stand-alone operation or as the core of an OMNIS titration system for endpoint titration and equivalence point titration (monotonic/dynamic). Thanks to 3S Liquid Adapter technology, handling chemicals is more secure than ever before. The titrator can be freely configured with measuring modules and cylinder units and can have a rod stirrer added as needed. Including "Professional" function license for parallel titration with additional titration or dosing modules.

- Actuation via PC or local network
- Connection option for up to four additional titration or dosing modules for additional applications or auxiliary solutions
- Connection option for one rod stirrer
- Various cylinder sizes available: 5, 10, 20 or 50 mL
- Liquid Adapter with 3S technology: Safe handling of chemicals, automatic transfer of the original reagent data from the manufacturer

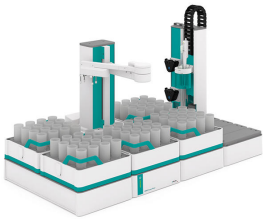
Measuring modes and software options:

- Endpoint titration: "Basic" function license
- Endpoint and equivalence point titration (monotonic/dynamic): "Advanced" function license
- Endpoint and equivalence point titration (monotonic/dynamic) with 5-way parallel titration: "Professional" function license



OMNIS Titration Module with magnetic stirrer

Titration module with magnetic stirrer for connection to an OMNIS Titrator for extending the system to include an additional titration stand, a buret for titration/dosing and up to two measuring modules (analog or digital). Freely selectable cylinder unit with 5, 10, 20 or 50 mL.



OMNIS Sample Robot M Pick and Place

OMNIS Sample Robot M with a "Peristaltic" (4-channel) pump module and a Pick&Place module in addition to extensive accessories for the direct transition to fully automatic titration. The system provides space in five sample racks for 80 sample beakers of 120 mL each.

The system can be extended upon request to include up to four peristaltic pumps and three additional Pick&Place modules, thus quadrupling the throughput. If even more samples are to be placed on the Sample Robot, then this Sample Robot can be expanded to become an L-sized OMNIS Sample Robot, thus enabling 112 samples to be processed in parallel on up to four Pick&Place modules and thus guaranteeing long and interruption-free operation.



OMNIS Rod Stirrer Sample Robot

Rod stirrer with permanently mounted cable (2.5 m) for direct connection to the OMNIS Sample Robot.



Cylinder unit OMNIS 50 mL

Intelligent 50 mL cylinder unit for an OMNIS Titrator, Titration Module or Dosing Module. Includes dosing tubing and antidiffusion tip.



Cylinder unit OMNIS 50 mL

Intelligent 50 mL cylinder unit for an OMNIS Titrator, Titration Module or Dosing Module. Includes dosing tubing and antidiffusion tip.



Measuring module digital

Digital measurement channel for one OMNIS Titrator or Titration Module for the connection of digital "dTrodes" electrodes.



Sample beakers, plastic (PP), 120 mL, 20 pieces

Sample beakers, plastic (polypropylene, PP), 120 mL, suitable for 602041030 OMNIS sample rack, 20 pieces.



Dis-Cover lid for OMNIS 120 mL sample beaker, 16 pieces

Dis-Cover lid for 120 mL 6.01400.200, 6.01400.300 and 6.01400.303 sample beakers in the OMNIS Sample Robot Pick&Place system.



Stirring propeller 20 mm ETFE

Stirring propeller, 20 mm, with adjustable height for use with Rod Stirrer "Titrator" or Rod Stirrer "Sample Robot"



dUnitrode with Pt1000

Digital, combined pH electrode for OMNIS with integrated Pt1000 temperature sensor. This electrode is particularly suitable for :

- pH measurements and titrations in difficult, viscous, or alkaline samples
- at elevated temperatures
- long-term measurements

The fixed ground-joint diaphragm is insensitive to contamination.

Reference electrolyte: c(KCl) = 3 mol/L, storage in storage solution.

Alternatively: Reference electrolyte for measurements at T>80°C: Idrolyte, storage in Idrolyte.

dTrodes can be used on OMNIS Titrators.