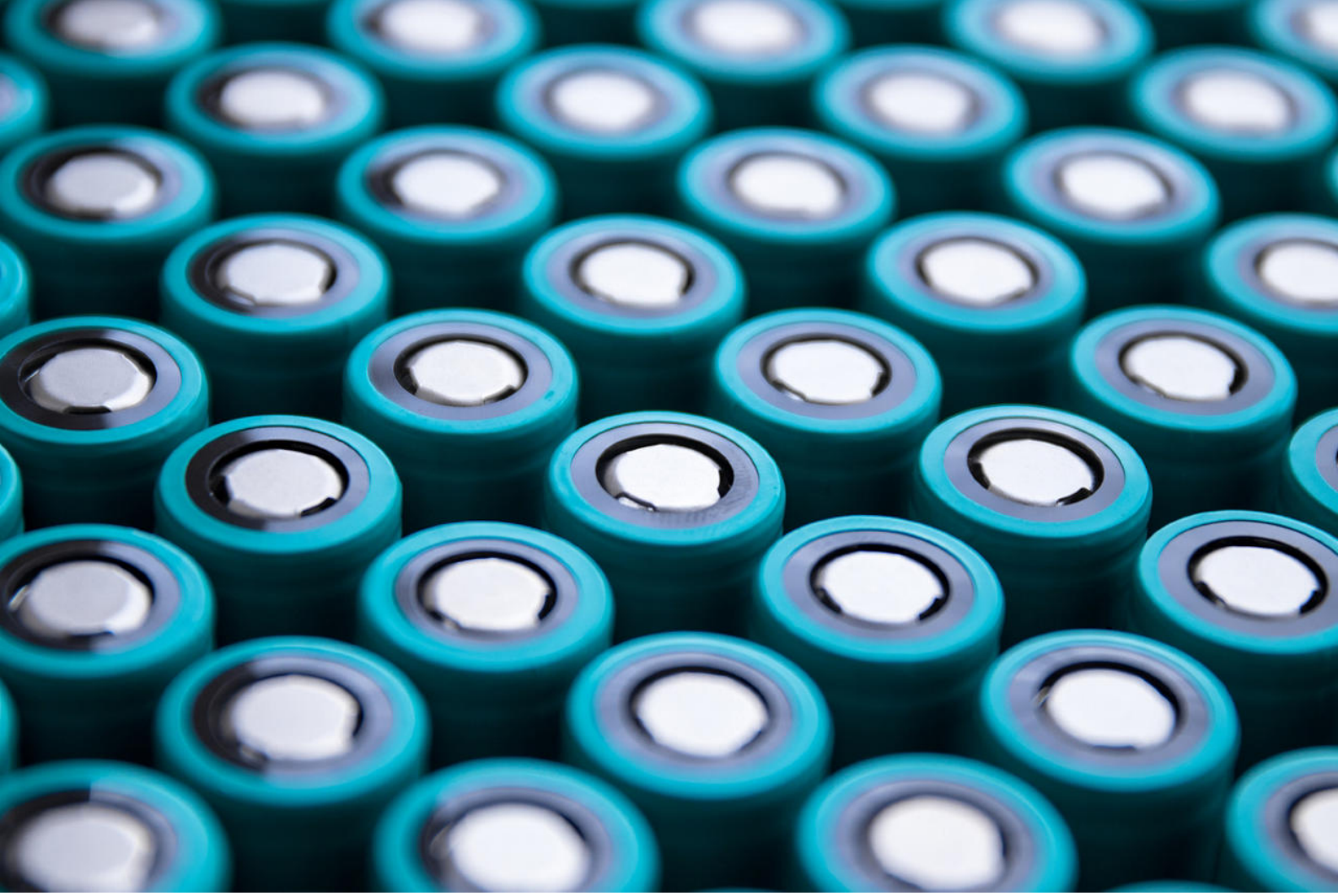


Application Note AN-T-252

Chloride in lithium carbonate by potentiometric titration

Determination of chloride in lithium carbonate by potentiometric titration according to draft standard ISO 12403

Accurate quantification of chloride impurities in lithium carbonate is essential to ensure the material's suitability for high-performance applications, particularly within the battery industry. During lithium-ion battery production, even trace levels of contamination in the raw materials can influence cathode quality, manufacturing consistency, and overall electrochemical behavior.

This Application Note describes a method for determining chloride content in lithium carbonate by potentiometric back-titration according to ISO 12403. Excess silver nitrate (AgNO3) was added to the acid-dissolved sample, and the remaining silver was titrated with sodium chloride (NaCl) to the first equivalence point.

SAMPLE AND SAMPLE PREPARATION

This method is applicable to determine chloride content in lithium carbonate (Li_2CO_3) in the range of 10 mg/kg to 650 mg/kg. The samples are 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 (Figure 1) equipped with a dAg-ring electrode. Nitric acid is added to acidify the sample, and a known amount of silver nitrate is added prior to analysis.

A blank measurement is performed and titrated with sodium chloride solution until after the equivalence point. An appropriate mass of sample is weighed and titrated with sodium chloride solution until after the equivalence point.



Figure 1. OMNIS Advanced Titrator equipped with a dAg-ring electrode.

RESULTS

The samples were tested in duplicate on one day, and once again on the following day. Results are displayed in Table 1. Figure 2 shows an example titration curve for the analysis.

Table 1. Results of the chloride determination in lithium carbonate samples. Sample ID number relates to the expected chloride impurity in mg/kg.

Sample ID (n = 3)	Content mean value Cl (mg/kg)	SD(rel) in %
10	0.00	0.00
50	60.04	3.97
115	141.36	1.93

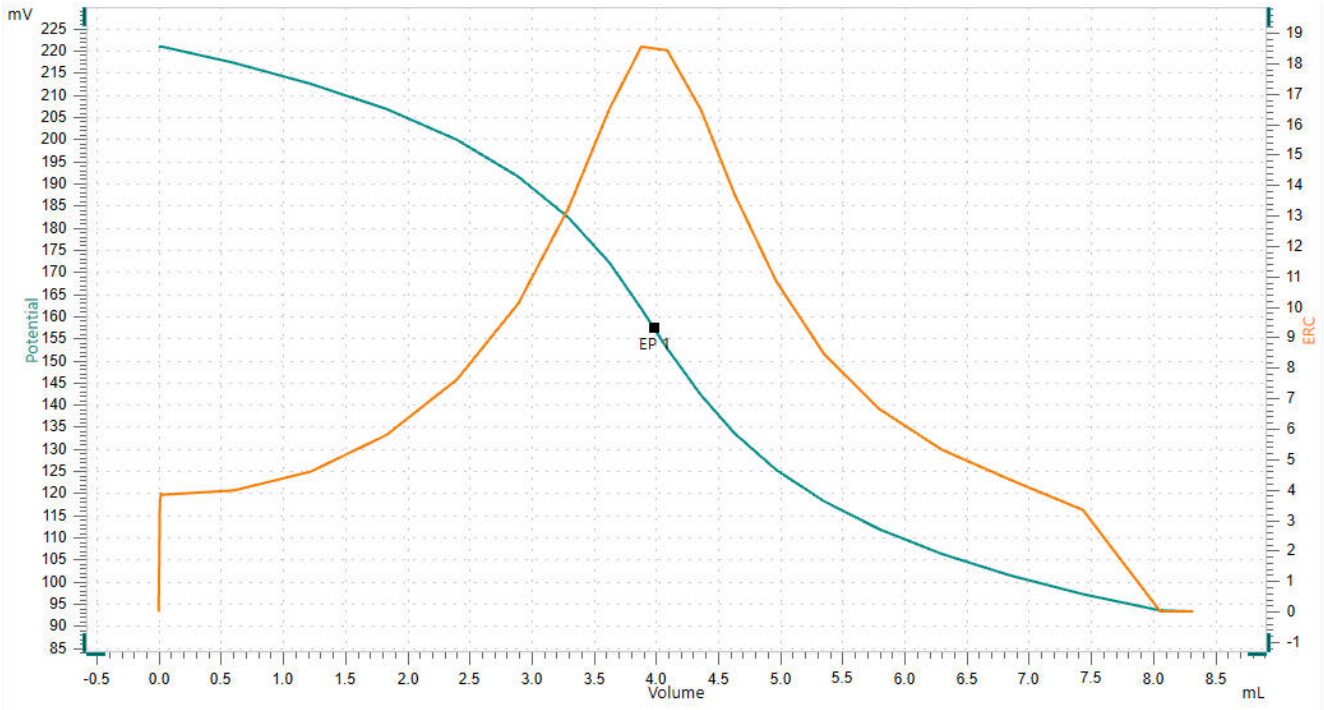


Figure 2. Titration curve of the chloride content in lithium carbonate.

CONCLUSION

The OMNIS Titrator provides a fast and reliable determination of chloride in lithium carbonate according to draft standard ISO 12403. The OMNIS system can be expanded to other titration applications as required.

CONTACT

Metrohm SA
1 Bridle Close
2052 Woodmead
sales@metrohm.co.za

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



Cylinder unit OMNIS 20 mL

Intelligent 20 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.



dAg ring electrode

Digital, combined silver ring electrode for OMNIS with fixed ground-joint diaphragm.
This electrode is suitable for precipitation titrations where the pH value varies (titrant silver nitrate) of, e.g.

- Chloride, Bromide, Iodide
- Sulfides
- Hydrogen sulfide
- Mercaptans
- Cyanides

The fixed ground-joint diaphragm is insensitive to contamination. c(KNO_3) = 1 mol/ L is used as reference electrolyte and for storage.
dTrodes can be used on OMNIS Titrators.