



Application Note AN-T-218

Analysis of Li-ion battery cathode materials made from Co, Ni, and Mn

Fully automated determination including sample preparation using the OMNIS pipetting equipment

The lithium-ion battery market is continuously growing due to the tremendous demand for items like handheld electronics, electric vehicles, and other battery powered consumer products. The quality of these batteries improves continuously by modifying and improving the main components (e.g., cathode, anode, slurry, and separators).

So-called «NCMs», a mixture of nickel, cobalt, and manganese oxides, have been gathering interest as cathode materials. These materials replace the cobalt

oxides traditionally used in Li-ion batteries. Quality analysis of the post-sintered materials or recycled batteries can be performed by titration, as demonstrated in this Application Note. A fully automated analysis of the corresponding metals can be performed with OMNIS and its pipetting equipment. For this purpose, only small sample amounts are needed which are accurately transferred to titration beakers for analysis.

SAMPLE AND SAMPLE PREPARATION

The method is demonstrated using dissolved mixtures

of Ni, Co, and Mn ion standards.

EXPERIMENTAL

For the metal determination, three titrations are performed. In the first titration, the total metal content is determined with a complexometric titration in an alkaline buffered solution using an excess of EDTA as a ligand for the metal ions, and copper(II) sulfate as a titrant. A Cu ISE was used as a potentiometric sensor. Nickel and cobalt can be determined with the same complexometric titration, but in slightly acidic environments.

The determination of Mn and Co is performed in alkaline conditions with a combined Pt ring electrode and potassium hexacyanoferrate ($K_3[Fe(CN)_6]$) as the titrant. With these determinations the individual metal content of Ni, Co, and Mn can be calculated. While it is possible to work on two Pick&Place modules, the sample throughput can be increased if a system with three Pick&Place modules is used.

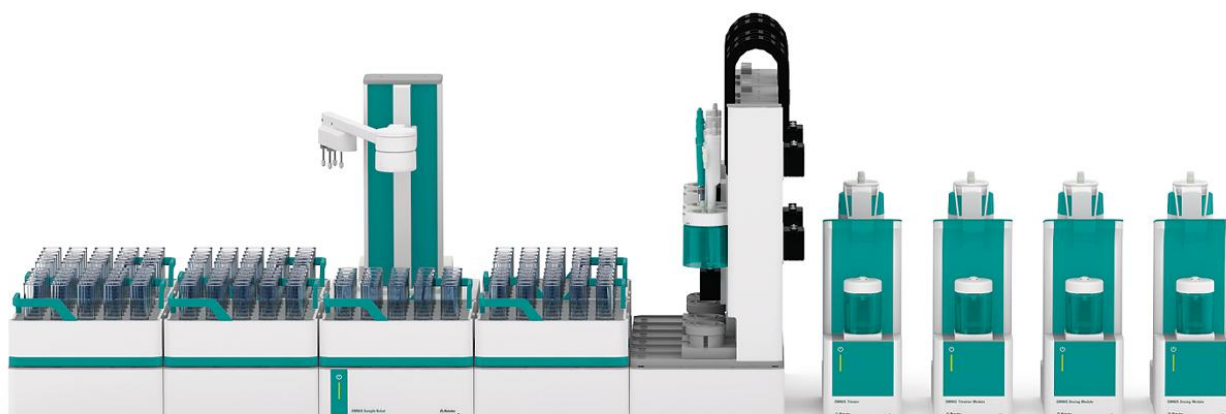


Figure 1. Exemplary OMNIS system for the fully automatic determination of NCMs in lithium-ion battery cathode materials.

RESULTS

The analysis demonstrates acceptable results and well-defined titration curves. The results and an

example titration curve are displayed in **Table 1** and **Figure 2**, respectively.

Table 1. Determined metal content of Ni, Co, and Mn in Li-ion battery cathode materials.

Analyte	Recovery in %	SD(rel) in %
Ni	100.66	0.38
Co	101.56	1.10
Mn	97.68	2.29

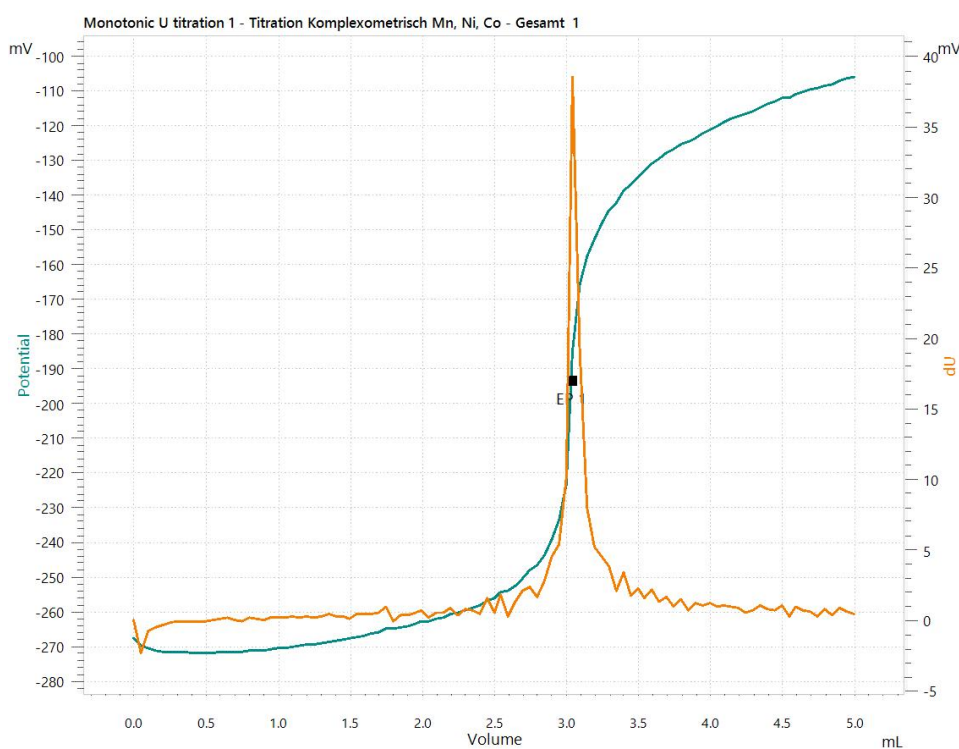


Figure 2. Example titration curve for the total metal content in cathode materials determined by complexometry.

CONCLUSION

With the OMNIS automated pipetting system, NCMs can be determined both quickly and accurately with little sample consumption (< 1 mL). The method is

accurate and can not only be used for single elements, but also for sample mixtures consisting of nickel, cobalt, and manganese.

Internal reference: AW TI-CH1-1313-082020

CONTACT

Metrohm Brasil
Rua Minerva, 161
05007-030 São Paulo

metrohm@metrohm.com.br