



Application Note AN-T-218

分析由 Co、Ni 和 Mn 制成的锂离子电池正极材料

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.

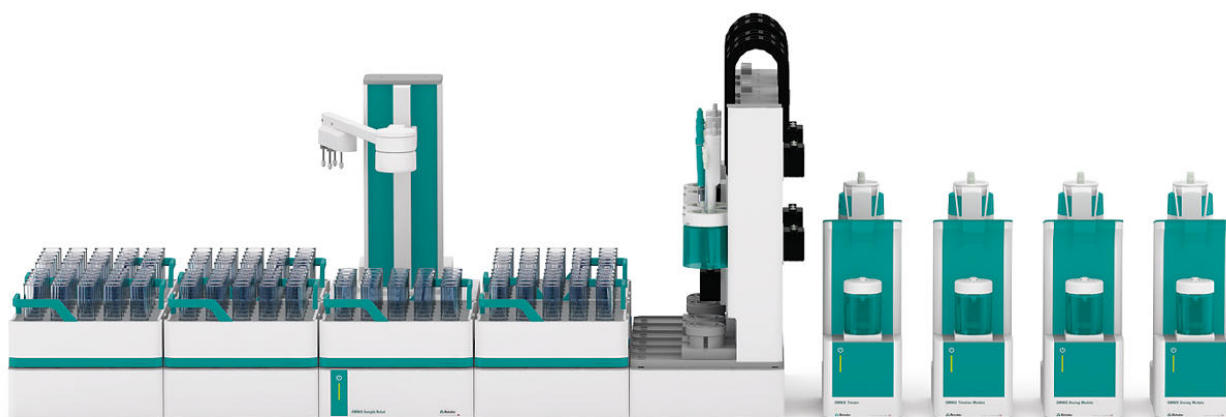


Figure 1. Exemplary OMNIS system for the fully automatic determination of NCMs in lithium-ion battery cathode materials.

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

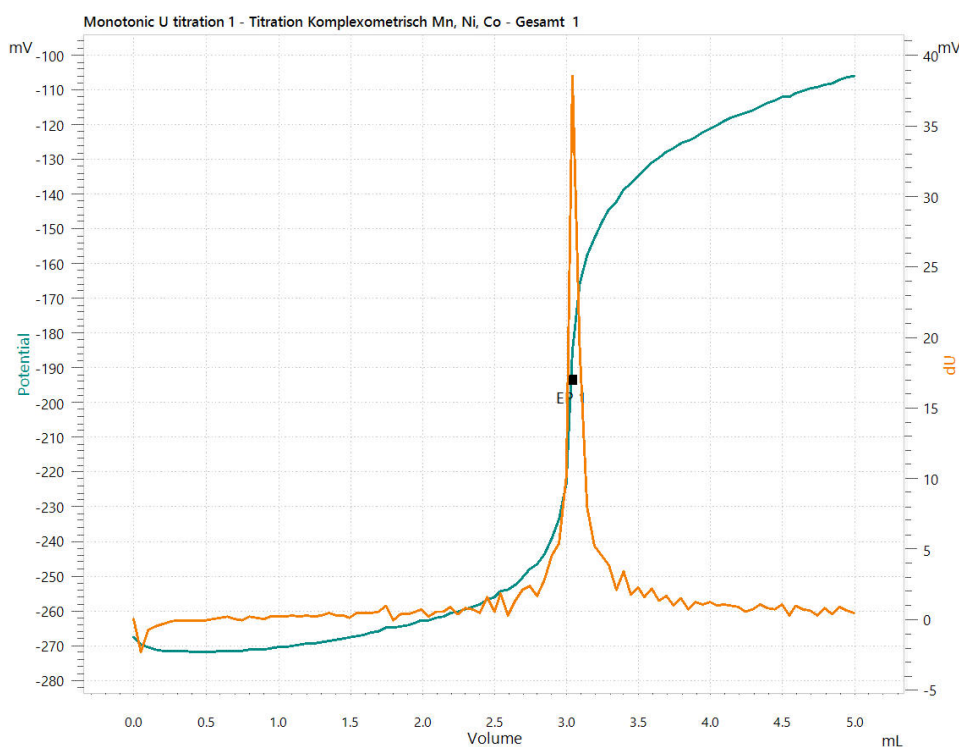


Figure 2. Example titration curve for the total metal content in cathode materials determined by complexometry.

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

117702
100085

marketing@metrohm.com.cn

CONFIGURATION



OMNIS Professional Titrator

创新型、模块式电位分析 OMNIS Titrator,适于独立运行或作为 OMNIS 滴定系统的核心元件运行,用于终点和等当点滴定(单一/动态)。由于采用 3S OMNIS Liquid Adapter 技术,处理化学品从未像现在一样安全。可以使用测量模块和计量管单元自由配置滴定仪,并在需要时扩展一台棒式搅拌器。包括用于使用其他滴定或加液模块平行滴定的“Professional”功能许可证。

- 通过计算机或本地网络控制
- 可以为其他应用或辅助溶液另外连接最多四个滴定模块或加液模块
- 棒式搅拌器的连接方式
- 可提供不同大小的计量管:5、10、20 或 50 mL
- 采用 3S 技术的 OMNIS Liquid Adapter:安全处理化学品,自动传输生产厂家的原始试剂数据

测量模式和软件选项:

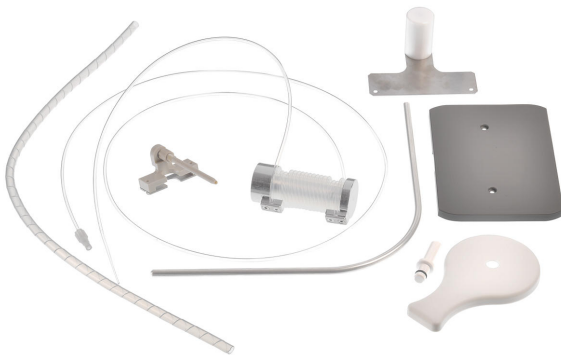
- 终点设定滴定:“Basic” 功能许可证
- 终点和等当点滴定(单一/动态):“Advanced” 功能许可证
- 终点和等当点滴定(单一/动态),包括 5 路平行滴定:“Professional” 功能许可证



OMNIS Sample Robot S Pick and Place

OMNIS Sample Robot S 具有一个“蠕动”泵模块(2通道)和一个 Pick&Place 模块以及大量附件,可直接进入全自动滴定。此系统具有两个样品盘位置,可用于 32 个 120 mL 的样品盘。此模块化系统供货时已完全安装完毕,因此可在极短时间内投入运行。

该系统也可根据需要扩展配备另外两台蠕动泵以及多加一个 Pick&Place 模块,由此使通量翻倍。如果需要更多工作台,则可将此 Sample Robot 扩展为 L 规格款型的 OMNIS Sample Robot,由此可使七个样品盘的样品在多达四个 Pick&Place 模块上并行处理,将样品通量扩大四倍。



OMNIS

完整附件组,用于将 OMNIS Sample Robot Pick&Place 改装为可进行移液的仪器型号。该套件可安装在所有版本 OMNIS Sample Robot(大号、中号和小号)上。



Cu

具有晶体膜的铜选择性电极。

这种 ISE 必须搭配参比电极使用,并且适用于:

- Cu^{2+} 的离子测量(10^{-8} 至 0.1 mol/L)
- 小样品体积的离子测量(最小浸没深度为 1 mm)
- 用 CuEDTA 进行络合滴定

得益于 EP 材质的坚固/不易破碎的塑料杆,该传感器可以承受较高的机械载荷。

使用随供的抛光套件,可以轻松清洁和更新电极表面。



Pt

带陶瓷柱隔膜的组合式铂环形电极。

该电极适用于 pH 值可变的氧化还原滴定,例如:

- 按照温克勒法的氧含量
- 用 KMnO_4 测定过氧化氢
- 重氮化滴定

作为参考电解液并进行制备时使用了 $c(\text{KCl}) = 3 \text{ mol/L}$ 。