



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.

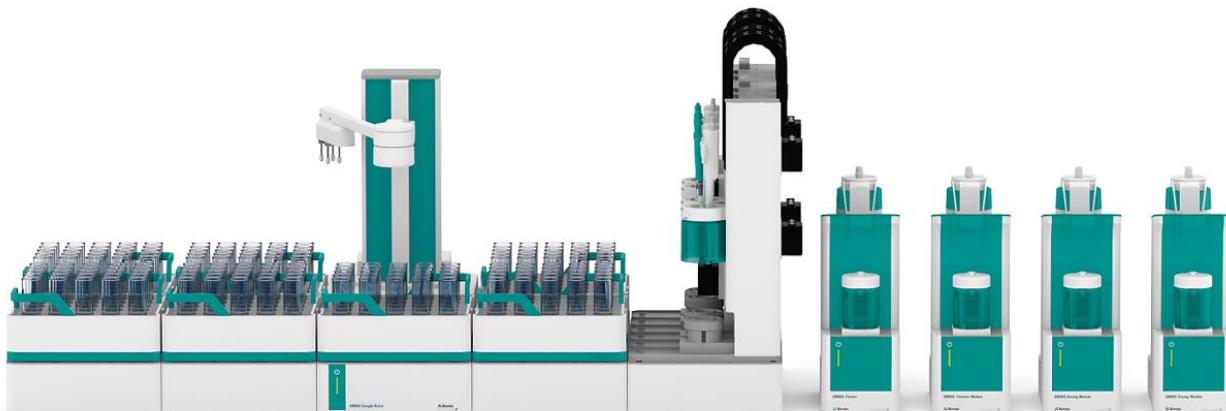


Figure 1. Exemplary OMNIS system for the fully automatic determination of NCMs in lithium-ion battery cathode materials.

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

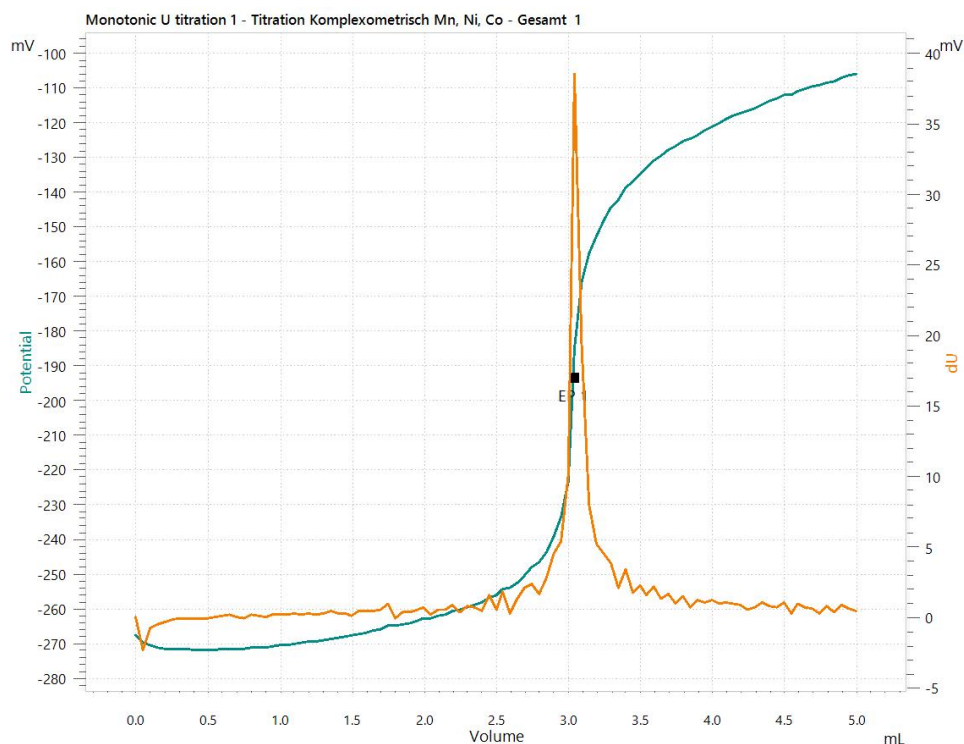


Figure 2. Example titration curve for the total metal content in cathode materials determined by complexometry.

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

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CONFIGURATION



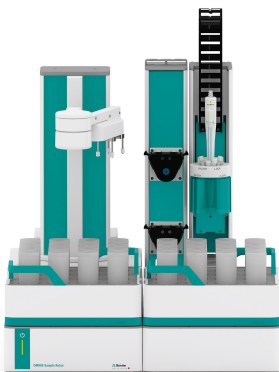
OMNIS Professional Titrator

新型、模式位分析 OMNIS Titrator 滴定,于独立行或作 OMNIS 滴定系的核心元件行,用于使用 OMNIS Sample Robot 行点和等当点滴定(一/)。由于采用 3S 瓶配器技,理化学品从未像在一安全。可以使用量模和量管元自由配置滴定,并在需要展一台螺旋拌器。包括用于使用其他滴定或加液模平行滴定的“Professional”功能可。

- 通算机或本地网控制
- 可以其他用或助溶液外接最多四个滴定模或加液模
- 螺旋拌器的接方式
- 可提供不同大小的量管:5、10、20 或 50 mL
- 采用 3S 技的瓶配器:安全理化学品,自生商的原始数据

量模式和件:

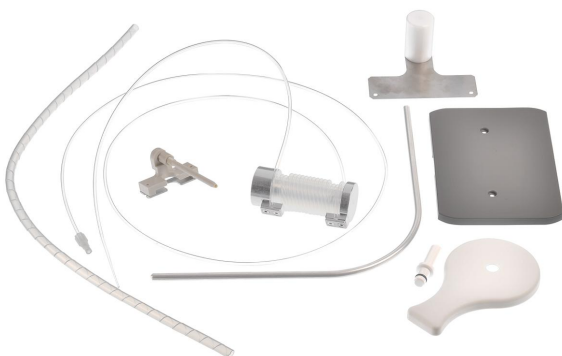
- 点定滴定:“Basic” 功能可
- 点和等当点滴定(一/):“Advanced” 功能可
- 点和等当点滴定(一/),包括平行滴定:“Professional” 功能可



OMNIS Sample Robot S Pick and Place

OMNIS Sample Robot S 具有一个“蠕”模(2 通道)和一个 Pick&Place 模以及大量附件,可直接入全自滴定。此系具有个品位置,可用于 32 个 120 mL 的品。此模化系供已完全安装完,因此可在短内投入行。

系也可根据需要展配外台蠕以及多加一个 Pick&Place 模,由此使通量翻倍。如果需要更多工作台,可将此 Sample Robot 展 L 格款型的 OMNIS Sample Robot,由此可使七个品的品在多四个 Pick&Place 模上并行理,将品通量大四倍。



OMNIS

完整附件,用于将 OMNIS Sample Robot Pick&Place 改装可行移液的器型号。套件可安装在所有版本 OMNIS Sample Robot(大号、中号和小号)上。



Cu

具有晶体膜的特性。

ISE 必搭配参比使用,并且用于:

- Cu^{2+} 的子量(10^{-8} 至 0.1 mol/L)
- 小品体的子量(最小浸没深度 1 mm)
- 用 CuEDTA 行合滴定

得益于 EP 材的固/不易破碎的塑料杆,感器可以承受高的机械荷。

使用随供的抛光套件,可以松清和更新表面。



Pt

陶瓷柱隔膜的合式形。

用于 pH 可的化原滴定,例如:

- 按照温克勒法的含量
- 用 KMnO_4 定化
- 重化滴定

作参考解液并行制使用了 $c(\text{KCl}) = 3 \text{ mol/L}$ 。