

Application Note AN-T-248

Lithium hydroxide and lithium carbonate in lithium hydroxide according to ISO/AWI 11045-1

Two-step lithium hydroxide titration with OMNIS

Lithium hydroxide monohydrate (LHM) is a preferred source of lithium for some of the high energy density cathode materials available on the market. Lithium carbonate impurities in LHM can lead to handling issues, inefficient processes during battery production, off-gassing effects, and other potential issues. As a result, the expectations and requirements for analytical test methods used to certify the quality of upstream materials have also increased. Titration test methods for lithium hydroxide and lithium carbonate impurity are key for monitoring and characterizing LHM product quality.

A two-step titration for lithium hydroxide and lithium carbonate according to ISO/AWI 11045-1 was performed in this study, resulting in fast, accurate determination of both lithium hydroxide and lithium carbonate in lithium hydroxide samples.

INTRODUCTION

As innovation drives increasing battery performance, purity requirements for upstream materials (such as lithium hydroxide monohydrate) have become more stringent. Lithium carbonate impurities in lithium hydroxide monohydrate can lead to issues with battery production, handling issues, and off-gassing effects. The expectations and requirements of analytical testing methods have increased in recent years. Accurate test methods for lithium hydroxide content and lithium carbonate impurity are essential for characterizing and monitoring the quality of lithium hydroxide monohydrate.

SAMPLE AND SAMPLE PREPARATION

This application is demonstrated on solid lithium hydroxide samples.

No sample preparation is required.

EXPERIMENTAL

The determinations are carried out on an OMNIS Sample Robot S with Dis-Cover lids (Figure 1), a nitrogen sparge source, a 907 Titrando, two 800 Dosinos, and an Ecotrode.

A 120 mL OMNIS beaker is sparged manually with nitrogen, then 1 g of sample is weighed into the titration beaker. The beaker is covered with a Dis-Cover cap and placed on the OMNIS Sample Robot S. The system adds 50 mL of nitrogen-sparged ultrapure water and titrates, under nitrogen, to pH 10.5 using 2 mol/L hydrochloric acid. Following this, the system titrates potentiometrically to two endpoints using 0.01 mol/L HCl.

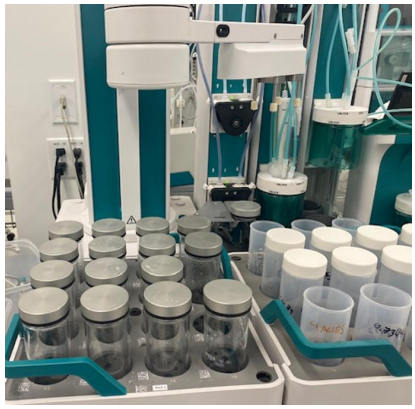


Figure 1. An OMNIS Sample Robot S with Dis-Cover caps and nitrogen sparging helps to protect the lithium hydroxide samples from carbon dioxide absorption prior to analysis.

RESULTS

Repeatable results within the expected ranges were obtained for all the lithium hydroxide samples analyzed using this method (Table 1). The first titration was a SET pH titration to pH 10.5 using 2 mol/L HCl. The second titration was a DET pH titration using 0.01 mol/L HCl. The two-step titration approach allows for efficient titration of the high lithium hydroxide content while still allowing for speciation of two endpoints (Figure 2). The first endpoint, plus the endpoint from the SET pH titration, corresponds to the lithium hydroxide content, while the second endpoint corresponds to the lithium carbonate content.

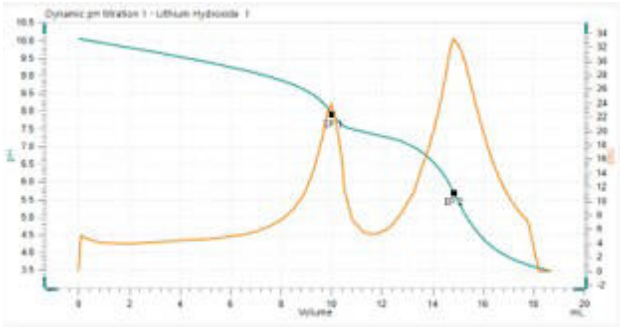


Figure 2. Lithium hydroxide and lithium carbonate endpoints from the second titration.

Table 1. Results of the determination of lithium hydroxide and lithium carbonate concentration in lithium hydroxide samples (n = 15).

Sample (n = 15)	Lithium hydroxide (%)	Lithium carbonate (%)
Mean value	58.36	0.27
SD(abs)	0.10	0.02
SD(rel) in %	0.17	6.15

CONCLUSION

The two-step titration approach using an OMNIS robot with Dis-Cover caps and nitrogen sparging allows for fast and accurate determination of both lithium hydroxide and lithium carbonate in lithium hydroxide samples. The initial SET pH titration with 2 mol/L HCl to reach pH 10.5 saves significant analysis time. The use of 0.01 mol/L HCl for the second titration allows for the speciation needed between the hydroxide and carbonate endpoints.

CONTACT

Metrohm Taiwan Ltd.
開封街二段 46 號 5 樓
108 台北市
info@metrohm.com.tw

CONFIGURATION



Pick and Place S

用于构建 S 规格款型 OMNIS Sample Robot Pick&Place 的主模块。该模块包括模块支架和样品盘架。已配备了主升降台和夹爪。若要扩展为功能就绪的 Sample Robot 则除了样品盘和抓取夹指之外还需要工作台,例如 Pick&Place 取放模块或泵模块。这些部件的选择可通过应用程序来完成。



Pick and Place

用于安装到 OMNIS Sample Robots Pick&Place 模块支架中的模块。该工作台可置放分析用的样品杯。两次分析之间会将所使用的传感器在 Pick&Place 模块存储杯中进行清洁或置放。如果分析时需要搅拌,则必须在该工作台上使用独立的棒式搅拌器。



4

用于安装到 OMNIS Sample Robots Pick&Place 模块支架中的模块。此工作站配备一台冲洗和抽液泵。可用于清洁 Pick&Place 模块中的传感器并在分析完成之后、重新将样品杯放回样品盘之前将其清空。



OMNIS Rod Stirrer Sample Robot

棒式搅拌器,可直接连接至 OMNIS Sample Robot,带有固定安装的电缆(2.5米)。



20 mm ETFE

搅拌螺旋桨 20 mm,高度可调,用于棒式搅拌仪“Titrator”(滴定)或棒式搅拌仪“Sample Robot”



4365 mm

OMNIS Sample Robot Pick&Place 的抓手夹指,用于抓取外直径 43-65 mm 的样品杯。



3 NS14/ 4 6.4mmP and P

具有 3 个 NS14 和 4 个 6.4 mm 开孔的滴定头,可用于 OMNIS Sample Robot Pick&Place(配备 2 个收回的加液器尖管)的 Pick&Place 取放模块中的 120 mL 样品杯。已准备好装备冲洗和抽吸管。



OMNIS 16 x 120 mLPP

OMNIS 样品架,用于 OMNIS Sample Robot Pick&Place,适配 16 个样品杯。以下样品杯均可使用:6.01400.200、6.01400.300、6.01400.303。
塑料:聚丙烯 (PP)



OMNIS 16 x 120 mLPP

OMNIS 样品架,用于 OMNIS Sample Robot Pick&Place,适配 16 个样品杯。以下样品杯均可使用:6.01400.200、6.01400.300、6.01400.303。

塑料:聚丙烯 (PP)



120 mL20

透明玻璃材质的样品杯 120 mL,适用于 OMNIS 样品架 602041030,20 个。



120 mL20

透明玻璃材质的样品杯 120 mL,适用于 OMNIS 样品架 602041030,20 个。



FEP / M6 / 150

带光照保护和扭折保护



FEP / M6 / 150
带光照保护和扭折保护



FEP / M6 / 150
带光照保护和扭折保护

OMNIS

A WHOLE NEW LEVEL OF PERFORMANCE

OMNIS 2
允许单机版 OMNIS Software 在一台 WindowsTM 计算机上运行。
特性:

- 该许可证已含有两份 OMNIS 设备许可证
- 须通过万通许可授权平台进行激活。



907 Titrando

用于使用一个测量接口和 Dosino 加液单元电位分析和容量卡尔·费休滴定法滴定的电位滴定仪。

- 多达四套 800 Dosino 加液系统
- 动态(DET)、等量(MET)和终点滴定(SET),酶和 pH-STAT 滴定(STAT)、卡尔·费休容量滴定(KFT)
- 使用离子选择性电极测量(MEAS CONC)
- 智能电极“iTrode”
- 带监控的加液功能,LQH
- 用于其他搅拌器或加液器系统的四个 MSB 接口
- USB 接口
- 使用 OMNIS-Software、*tiamo*-软件或 Touch Control
- 如果需要,满足 GMP/GLP 和 FDA 要求,比如 21 CFR 第 11 部分



800 Dosino (1.5 m)

800 Dosino 是一种带有智能型配液单元读写硬件的驱动装置。配有固定安装的电缆(长度 1.5 m)。



800 Dosino (1.5 m)

800 Dosino 是一种带有智能型配液单元读写硬件的驱动装置。配有固定安装的电缆(长度 1.5 m)。



807 Dosing Unit 20 mL

807 Dosing Unit,带内置的数据芯片,20 mL 玻璃计量筒和遮光罩,可安装在有 ISO/ DIN 玻璃螺纹 GL 45 的试剂瓶上。FEP 管路连接、防扩散滴管头。



807 Dosing Unit 20 mL

807 Dosing Unit,带内置的数据芯片,20 mL 玻璃计量筒和遮光罩,可安装在有 ISO/ DIN 玻璃螺纹 GL 45 的试剂瓶上。FEP 管路连接、防扩散滴管头。



2 / F

用于将带瑞士万通G型插入式连接头的电极连接到瑞士万通测试仪(F型插座)。



Ecotrode plus

适用于含水的酸/碱滴定的组合 pH 电极。固定磨口隔膜对污染不敏感。参比电解质:c(KCl) = 3 mol/L,储存在储存溶液中。



OMNIS Sample Robot S

用于在 OMNIS Sample Robot S 系统中安装 OMNIS 泵模块或盲板结构 6.02007.000 的带盖储物盒。



Dis-Cover 120 mL OMNIS 16

OMNIS Sample Robot Pick&Place 系统中 120 mL 样品杯 6.01400.200、6.01400.300 和 6.01400.303 用 Dis-Cover 盖子。



Dis-Cover 120 mL OMNIS 16

OMNIS Sample Robot Pick&Place 系统中 120 mL 样品杯 6.01400.200、6.01400.300 和 6.01400.303 用 Dis-Cover 盖子。