Matrix-Matched Standard Calibration Approach in Glow Discharge Sector Field-Mass Spectrometry (GD-SF-MS) for Lithium Ion Battery Electrodes

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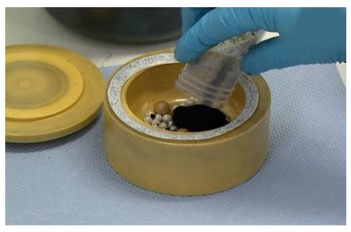
Introduction

The aging of lithium ion batteries (LIBs) is an unwanted but still present phenomenon which can be observed in every LIB, no matter which chemistries are involved. [1,2]

The focus of this work is on calibration techniques which allow the quantification of distributed elements in aged graphite electrodes. $Li_1Ni_{1/3}Co_{1/3}Mn_{1/3}O_2$ (NMC) as a commonly used cathode material was added to graphite electrodes in a solid doping approach to obtain an elemental calibration. Takahara *et. al.* already showed the accessibility of sintered lithium sources for the quantification of cycled electrodes via GD-OES. [3]

Methods

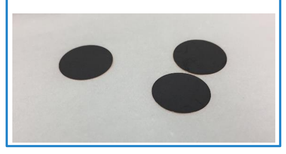


- $Li_1Ni_{1/3}Co_{1/3}Mn_{1/3}O_2$ acts as source for the corresponding element
- Composite graphite (MesoCarbon Microbeads / Hard Carbon) acts as carbonaceous source

Homogenization in ball-mill using NMP as solvent

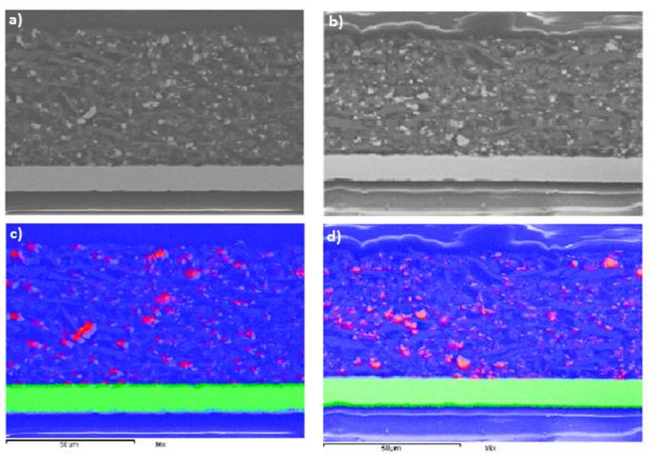
Drying @ 80 ° C

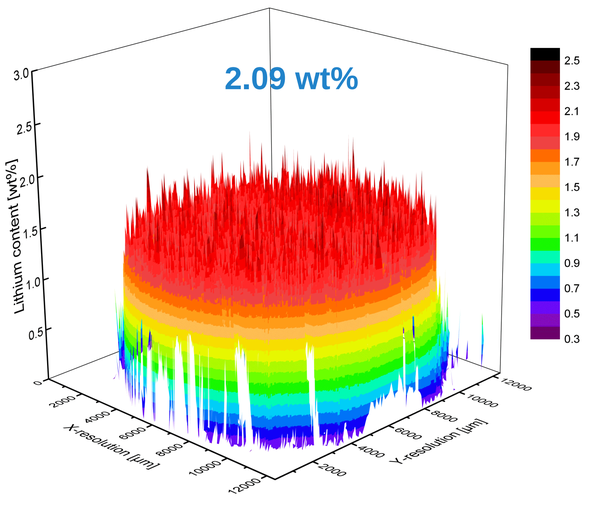
Punch out electrodes with 12 mm in diameter

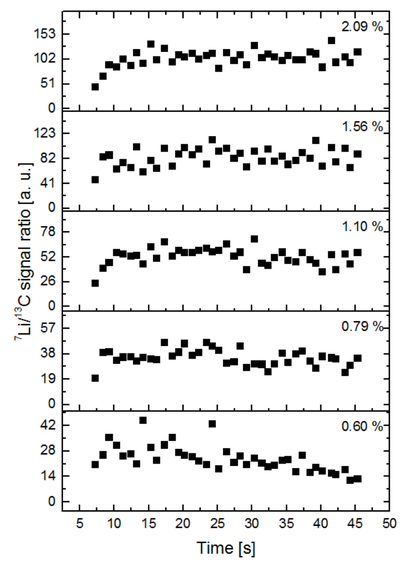


Results & Discussion

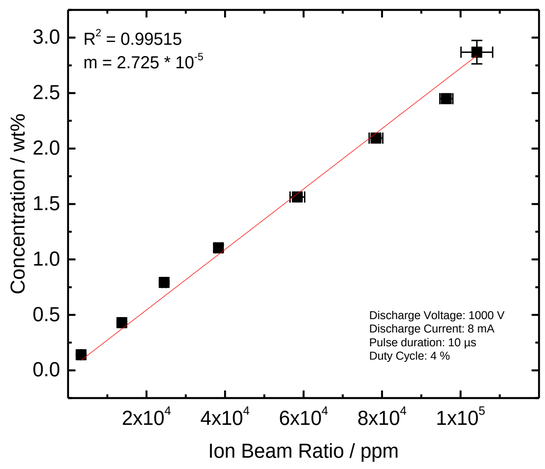
Homogeneity Check

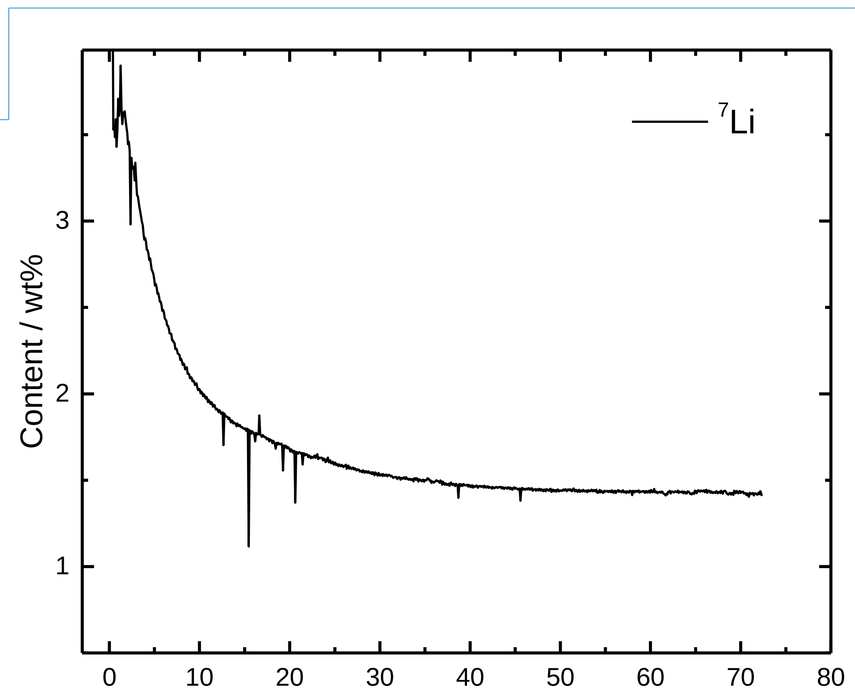






Quantification of cyclic aged Electrodes





	LOD / ppm	LOQ / ppm
^7Li @ R=400	80	240
^7Li @ R>4000	162	486
^{55}Mn @ R>4000	265	797
^{59}Co @ R>4000	393	1180
^{58}Ni @ R>4000	333	1001

*Calculated using 3- and 9-sigma criteria

- Good linearity using the custom-made matrix-matched standards could be observed
- The homogeneity of the standards was checked via SEM/EDX cross-sections as well as LA-ICP-MS depth profiling
- The LODs and LOQs are low for quantification purposes in the field of lithium ion battery research

Literature/References

[1] J. Vetter, P. Novák, M. R. Wagner, C. Veit, K. C. Möller, J. O. Besenhard, M. Winter, M. Wohlfahrt-Mehrens, C. Vogler and A. Hammouche, *J. Power Sources*, 2005, **147**, 269-281.
[2] M. Evertz, F. Horsthemke, J. Kasnatscheew, M. Börner, M. Winter, S. Nowak, *J Power Sources*, 2016, **329,** 364-371.
[3] H. Takahara, M. Shikano and H. Kobayashi, *J. Power Sources*, 2013, **244**, 252-258.

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