Supplementary Information

Automated on-line monitoring of a lithium hydroxide production process using Micro-Discharge OES

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The following supplementary information provides additional data, diagrams and experimental details to support the presented results of the corresponding paper.

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Additional results of the minor components Na/K and the trace elements Ca/Mg

Further results of the minor components Na/K and the trace elements Ca/Mg are presented below in comparison with the reference methods ICP-OES and IC (Fig. A1-A3). The agreement between the Micro-Discharge OES and the reference methods is also good for these elements for many samples, although larger deviations occur for some samples, especially for Ca and Mg, which are low in concentration. This can be explained by the fact that high dilution ratios were used during the measurement campaign, resulting in concentrations of trace elements close to their respective detection limits. Furthermore, the samples were filtered for the ICP-OES and IC measurements, but not for the μ DOES analyses.



Figure A1 Comparison of the Ca and Mg Micro-discharge OES results with laboratory ICP-OES based on selected samples of the ore leaching process step (on-line μ DOES: n=1 continuous process, ICP OES: n=5).



Figure A2 Comparison of the concentrations measured by Micro-discharge OES and ion chromatography using selected samples of the lithium carbonate precipitation process step (batch-wise µDOES: n=3, IC: n=1).



Figure A3 Na, K and Ca concentrations of the obtained LiOH solution comparing μDOES and ion chromatography (batch-wise μDOES: n=3, IC: n=1).

Batch-wise analysis of the solid LiOH·H₂O samples

Process step IV, the purification of lithium hydroxide monohydrate, was carried out batch-wise to obtain the solid end product. Prior to analysis with the Micro-Discharge OES, the samples were dissolved completely and diluted using ultrapure water (Merck Milli-Q[®], 18.2 M Ω /cm), whereby a few drops of concentrated nitric acid (Merck, analytical grade, 65%) using an acid dispenser were added to

improve the solubility. The samples were then diluted 100x with ultrapure water and analysed. Based on the weighted masses (Tab. A1) and the measured lithium concentrations, the lithium content in the salt could be determined.

Sample	$m_{LiOH \cdot H_2^0}$ in g	$m_{HNO}^{}_{3}$ in g*	$m_{H_2^0}$ in g	$m_{_{Total}}$ in g
1	1.0020	0.3480	500	501.3500
2	1.0003	0.3480	500	501.3483
3	0.5000	0.1390	250	250.6390
4	0.4998	0.1390	250	250.6388

Tab. A1 Preparation of the LiOH·H₂O samples.

 ${}_{*}^{\rho}{}_{HNO_{3},\,65\%,\,20^{\circ}C} = 1.39 \frac{g}{mL} = 1390 \frac{mg}{mL}$, 1 dispenser drop ≈ 0.05 mL