

BRUKER NANO ANALYTICS

Ba La Hi

Rapid and cost-effective metal analysis in material sciences and battery research

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Overview

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Analysis of Li-ion battery degradation





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Introduction – Ionic liquids

Introduction What are Ionic liquids?

- Ionic liquids (ILs) are materials composed entirely of ions that can form stable liquids under operating conditions
- In many chemical reaction processes, ILs are applied as solvents, catalysts, reagents, or combinations of these
- One important application of ILs is the extraction of rare earth elements (REE) – over 2000 scientific publications about this topic since 2022
- Analysis of ionic liquids by ICP-OES and ICP-MS is difficult and demands careful sample preparation and adjustment of the instrumental conditions (spray chambers, matrix correction etc.)
- With TXRF ILs can be analyzed directly or after simple dilution with methanol or water





 $0.57 \le x \ge 0.84$

The structure of binary alkoxide ionic liquids ([Pyrr1,4][NTf2]x[OiPr]1-x).





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Recycling of Rare Earth Elements (REE)

Ionic liquids and magnetic materials The growing demand for rare earth metals (REE)

Usage of rare earth elements (REE)

- REE are currently used in numerous products: batteries, ceramics, catalysts etc.
- REE are of substantial need in rapid growth areas
 - wind turbines
 - permanent magnet synchronous motors ("PMSMs") of electric vehicles (EV)
- forecast 2040: about 7 times more REE processed*



Link to <u>article</u>: "The Role of Critical Minerals in Clean Energy Transitions"

RAPID AND COST-EFFECTIVE METAL ANALYSIS IN MATERIAL SCIENCES AND BATTERY RESEARCH

Ionic liquids and magnetic materials The growing demand for rare earth metals (REE)

Issues of rare earth element (REE) analysis by ICP*

- Double charges of REE are easily formed causes inferences with elements from Ga to Sr
 → reaction/collision gas strictly needed (He, O₂, NH₄)
- Additionally, rather complicated corrections for oxide interferences must be applied
- REE are often concentrated in mineral phases, which are highly resistant to acids. Thus, very aggressive acid mixtures (HCl, HNO₃, HF and HClO₄) must be used for the sample digestion

*) Source: Marc Ulrich, Sarah Bureau, Catherine Chauvel, Christian Picard, Geostandards and Geoanalytical Research, 2012, 36 (1)



Link to <u>article</u>: "Accurate Measurement of Rare Earth Elements by ICP-MS after Ion-Exchange Separation: Application to Ultra-Depleted Samples"





Applications of REEs and ionic liquids

Applications

- Analysis of REE concentrations in powders
- Extraction of REEs from electronic waste using ionic liquids
- Characterization of new magnetic compounds

Tasks

- Screening of powders; quantification possible, if oxygen content is known
- Development of efficient and rapid extraction methods
- For process control and optimization, REE concentrations in the leaching and leached solution must be analyzed on a regular basis



Imidazolium-based ionic liquid (PD)



Issues of REE and ionic liquid analysis

Issues of ICP analysis

- Installation of an ICP-OES in process buildings not possible in most cases due to missing laboratory infrastructure
- Limited budget for daily operation
- In research different types of samples and concentrations ranges require laborious setup of an ICP

Challenges for TXRF

- Powders of high density, preparation of a suspension is not possible
- Concentrated solutions require high dilution factors (up to 1 : 10.000)
- Critical line overlaps, if the sample contains many different REE compounds and/or transition metals



REEs and ionic liquid samples Qualitative screening

Samples

- Iron-rich powders containing dysprosium, neodymium, praseodymium and more
- Extraction solutions containing many different REEs
- Magnetic compounds containing other metals like Al, Fe, Co

Screening

- Fine ground powders can be applied directly on discs prepared with grease
- S2 PICOFOX is suitable for the detection of most REE (Mo-K excitation)
- S4 T-STAR[®] is recommended in case of additional elements like Al, Cr (W-L excitation) or Pd (W-Brems excitation)





REEs and ionic liquid samples Qualitative screening

Qualitative results

- Typical spectra of magnetic materials containing La, Lu and Fe (left fig., dark blue) or Nd and Dy (left figure, light blue)
- A challenging extraction sample containing numerous REE (right figure)





Typical TXRF spectrum of a magnetic material

TXRF spectrum of a sample containing several Rare Earth Elements



REEs and ionic liquid samples Sample preparation for quantitative analysis



Typical preparation of REE extract solution

Typical preparation of ionic liquids





RAPID AND COST-EFFECTIVE METAL ANALYSIS IN MATERIAL SCIENCES AND BATTERY RESEARCH

REEs and ionic liquid samples Research for process optimization

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Accurate results after process optimization

 Ionic liquids were analyzed after 1:10000 dilution with methanol.

Element (mg/l)	Sample 1	Sample 2	Sample 3		
La measured	996				
La expected	900				
Ce measured		61	847		
Ce expected		50	950		

TXRF spectrum of an ionic liquid extraction solution



REEs and ionic liquid samples Research for process optimization

Accurate results after process optimization

- For transition metals the extraction procedure can be applied, too.
- The homogenous preparation of water solutions was optimized by addition of polyvinyl alcohol solution.

Element (mg/l)	Sample 4	Sample 5	Sample 6	
Со	1.2	1.9	4.7	
Fe	3.9	17	12	
Co/Fe meas.	0.305	0.116	0.399	
Co / Fe expect.	0.294	0.150	0.400	

TXRF spectrum of transition metals









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Batteries Analysis of graphite for anode production



Graphite sample

- Leading European company for the production of synthetic graphite
- For anode production the graphite must be free of metallic impurities
- In production /process control WD-XRF (main elements) and ICP-OES (traces) is used for analysis. This was increasingly found to be too complicated and time consuming (difficult sample preparation)
- TXRF was chosen as an alternative analytical technique



Current state of electric vehicle (EV) battery technology. The majority of lithium-ion battery technology consists of four main components: an anode, a cathode, an electrolyte and a separator.



Qualitative analysis with S2 PICOFOX

- Ti, V, Cr, Mn, Fe, Co, Ni, Cu and Zn were detectable
- Na, Mg, Si, P, K and Ca were below the detection limit
- Decision to perform quantitative analysis with the S4 T-STAR[®] and W-L excitation for light elements



TXRF spectrum of graphite powder



Quantitative analysis with S4 T-STAR®



- Elements analyzed with W-L excitation
- Elements analyzed with Mo-K excitation
- Internal standard Sc (W-L) and Ga (Mo-K)





Sample preparation

- Weighing of 100 ± 10 mg sample into plastic vials
- Suspension in 1 ml ethylene glycol
- Addition of 5 µl Sc and Ga standard solution (100 mg/l) for internal standardization
- Homogenization in an automatic sample shaker for 30 s
- Transfer of 2 µl suspension onto quartz glass or sapphire (for Si analysis) sample carriers
- Drying on a hot plate at 110 °C

All samples plus a blank ethylene glycol sample were prepared and measured as triplicates





Quantitative analysis

- Ca, V, Ti, Mn can be analyzed with S2 PICOFOX and S4 T-STAR[®]
- For quantification of the light elements Mg, Al, Si, P and K the S4 T-STAR[®] applying the W-L excitation is strictly recommended
- The additional element of interest Na cannot be analyzed with both systems





TXRF spectrum of graphite powder showing the light element range

Quantitative analysis

- The elements for customer test requirements (Fe, Cr, Ni, Cu, Zn and Co) can be analyzed with both systems, applying Mo-K excitation
- The detection limits for the S4 T-STAR[®] are slightly better compared to the S2 PICOFOX



TXRF detection limits of contaminants in graphite powder (Mo-K excitation)





Quantitative analysis

 Detection limits for S4 T-STAR[®] applying Mo-K and W-L excitation are typically in the ppb range for all elements of interest











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Batteries Analysis of Li-ion battery degradation

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Mechanisms of Li battery aging

- Battery aging is caused by different mechanisms, e.g., dissolution of transition metals from the cathode
- A solid electrolyte interphase (SEI) protects the graphite anode
- Transition metals that have electro-deposited on a graphite anode lead to cracking of the SEI
- A rapid analytical technique to examine deposited metals on the anode is desired



Scheme of the transition metal dissolution in batteries by the example of $LiMn_2O_4$ spinel electrodes

(Evertz, M. et al, Spectrochimica Acta Part B 112 (2015) 34–39; Fig. 1)

Link to article: "Development of a method for direct elemental analysis of lithium ion battery degradation products by TXRF"

Experimental

- Charging and discharging cycles at different charging voltages (4.3 V and 4.6 V) and "C-rates"* were performed
- After cycling the electrodes were washed
- An uncycled electrode was prepared in the same manner as reference cell

*) C-rate defines (dis-)charging time A battery with 1.000 mAh capacity provides 1.000 mA at 1C for 1h or 2.000 mA at 2C for 0.5 h

Sample preparation

- Cutting the electrodes to 1 x 1 cm pieces
- Addition of 1 ml Triton X-100 and glass grinding balls
- Milling for 90 min at 30 Hz to remove the graphite from the copper collector (reduce Cu signal), graphite agglomerates about 15 µm
- 1:10 dilution with Triton X-100 and standardization with 1 mg/l As
- Homogenization, pipetting 5 µl on quartz carrier measurement of each sample in triplicates



Results

- Metal amount is extremely high, especially for Mn
- Recovery rates were in the range of ±10% compared to ICP-MS as shown in the figure
- LOD were slightly higher than ICP-OES values, but still sufficient

Element	LOD (µg/l)
Mn	4.8 ± 0.9
Со	3.5 ± 0.6
Ni	2.9 ± 0.5



Comparison of the deposited elemental amount on graphite anodes after charge/discharge rate test (mass per anode area in cm²)

(Evertz, M. et al, Spectrochimica Acta Part B 112 (2015) 34–39; Fig. 4)



Results

- Drastically increasing amounts of transition metals at high cut-off voltage
- Recovery rates show good applicability of TXRF
- TXRF has large potential also for Si-based electrodes, which avoids digestion with HF prior to ICP analysis

Sample	Recovery rate (%)					
(validating method)	Mn	Со	Ni			
4.6 V (ICP-0ES)	102	87	94			
4.3 V (ICP-MS)	93	94	102			
Reference (ICP-MS)	114	100	94			



Amounts of the investigated elements deposited on the anode for different cut-off voltages (mass per anode area in cm²)

(Evertz, M. et al, Spectrochimica Acta Part B 112 (2015) 34–39; Fig. 5d)



Batteries Analysis of Lithium-ion Battery Electrolytes

Background

- Li-ion batteries contain electrolyte solutions working as ion carrier for lithium
- Battery aging is caused by particle cracking, phase transformation, transition metal dissolution and more
- High salt content and very low electrolyte volume is challenging for ICP analysis
- TXRF spectrometry is most suitable for low volumes, but is also disturbed by the matrix leading to "doughnut effect" (ring-shaped cristallization during drying)
- The objective was the development of an optimized sample preparation method









Batteries Analysis of Lithium-ion Battery Electrolytes

Experimental

- Li-ion batteries were cycled and the separators containing the electrolytes were removed
- The separator was centrifuged and the residue analyzed by TXRF
- The separator was dissolved and a suspension prepared as shown in the example before
- To minimize the "doughnut effect" a special dispensing device providing a pattern of nanoliter droplets was applied
- A centered pattern of the sample was deposited followed by deposition of the internal standard inbetween the electrolyte spots

Centered pattern

- 28 electrolyte droplets $(414 \pm 27 \text{ nl})$, red arrow
- 13 standard droplets,
 10 mg/l Ga
 (200 ± 4 nl), blue arrow

(Evertz, M. et al, Spectrochimica Acta Part B 149 (2018) 118–123; Fig. 1)

Link to article: "TXRF in the field of Li-ion batteries –Elemental detection in Lithium containing electrolytes using nanoliter droplets"





Batteries Analysis of Lithium-ion Battery Electrolytes

Results

- While conventional µl droplet deposition lead to salt crusts with a height of 100 µm, the nl deposits reduced the height of the sample spots below 60 µm
- The recovery was improved from below 90% to a range of 98% to 105%



Detailed view of the centered nl pattern with respect to the validating ICP-OES technique and the conventional sample application technique (microliter droplet)

(Evertz, M. et al, Spectrochimica Acta Part B 149 (2018) 118-123; Fig. 3)







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Summary Part I

Summary Part I

Rare Earth Elements (REE)

- Due to the wide concentration range of Rare Earth Elements in powders and leaching solutions TXRF is the method of choice
- During recycling REE are typically extracted with ionic liquids, which can be directly measured by TXRF
- REE can be measured with the S2 PICOFOX and the S4 T-STAR[®], line overlaps must be considered carefully for quantification

Battery research and production

- TXRF allows a rapid control of impurities of graphite powder used for battery electrodes
- The S4 T-STAR[®] and S2 PICOFOX provide sufficient detection limits for the quantification of degradation products in battery electrodes and electrolytes
- TXRF spectrometry is the method of choice due to the rapid sample preparation and the minute amount of sample required







Münster Electrochemical Energy Technology

Investigation of Transition Metal Dissolution of Lithium Ion Batteries by TXRF

Münster Electrochemical Energy Technology











Investigation of Transition Metal Dissolution of Lithium Ion Batteries by Total Reflection X-ray Fluorescence

Sascha Nowak*

Bruker Webinar, 12.07.2023

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Overview



Lithium Ion Batteries and Background







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Nowak et al., The Role of Sub-and Supercritical CO2 as "Processing Solvent" for the Recycling and Sample Preparation of 38 Lithium Ion Battery Electrolytes. Molecules 2017, 22 (3), 403.

Nowak et al., Elemental analysis of lithium ion batteries. Journal of Analytical Atomic Spectrometry 2017, 32 (10), 1833-1847.





Transition metal dissolution in LIBs



Two-phase structure:

Manganese dissolves from <u>unstable delithiated structure</u> accompanied with a structural change to form a stable single phase (MnO, Mn₂O₃)



Mn³⁺-content at maximum for fully discharged LiMn₂O₄

Jahn-Teller distortion:

- High rates → Li⁺-ions accumulate at the surface and lower the average Mn valence below 3.5
- Jahn-Teller distortion leads to an increase of the unit cell by 16%
- In Li-rich surface is the Mn³⁺-content at maximum (Li₂Mn₂O₄) and more likely to dissolve via disproportion

Overview



Experimental Setup





TXRF Sample Preparation

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Evertz et al., Development of a Method for Direct Elemental Analysis of Lithium Ion Battery Degradation Products by Means of Total Reflection X-Ray Fluorescence, Spectrochimica Acta Part B: Atomic Spectroscopy, 2015, 112, 34-39.

Overview



Results



Charge/Discharge Aging Criteria



- Aging criteria:
 - Charge cut-off: 4.25 V / 4.55 V
 - Discharge cut-off: 3.0 V
- Electrolyte: 1 M LiPF6 in EC/EMC (50:50 wt%)
- After charge/discharge aging the electrodes are with 1 mL EMC

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2	3	3	2	1
5	6	6	5	4
8	9	9	8	7
ara	ntor	anode		





Method Validation for TXRF Sample Preparation

- Alternative sample preparation: Recovery rates of 90 to 110 %
- The LOD of the TXRF method were in the range of 4.8 ± 0.9 ppb, 3.5 ± 0.6 ppb and 2.9 ± 0.5 ppb for manganese, cobalt and nickel (0.5 ng/cm²)
- Low LOD plus minimum sample preparation
 - TXRF method on ICP-MS level
- Only drawback, is the lack of lithium detection





Evertz et al., Development of a Method for Direct Elemental Analysis of Lithium Ion Battery Degradation Products by Means of Total Reflection X-Ray Fluorescence, Spectrochimica Acta Part B: Atomic Spectroscopy, 2015, 112, 34-39.

Influence of Different Charging Cut-Off Voltages on the TMD (Anodes)



The active TM loss from the cathode detected on the separator is $0.03 \% \pm 0.01 \%$, $0.02 \% \pm 0.01 \%$ and $0.04 \% \pm 0.01 \%$, for nickel, cobalt and manganese, respectively.

WWU Evertz et al, Unraveling Transition Metal Dissolution of Li_{1.04}Ni_{1/3}Co_{1/3}Mn_{1/3}O₂ (NCM 111) in Lithium Ion Full Cells by Using the Total Reflection X-ray Fluorescence Technique, Journal of Power Sources, 2016, 329, 364-371.

meet

Influence of Different Charging Cut-Off Voltages on the TMD (4.6 V)





The total active mass lost from cells cycled to a cut-off voltage of 4.6 V, with respect to TM deposition on the anode and on the separator is 1.48 $\% \pm 0.17 \%$, 1.33 $\% \pm 0.27 \%$ and 1.65 $\% \pm 0.24 \%$ for nickel, cobalt and manganese, respectively.

WWU Evertz et al, Unraveling Transition Metal Dissolution of Li_{1.04}Ni_{1/3}Co_{1/3}Mn_{1/3}O₂ (NCM 111) in Lithium Ion Full 47 ^{MÜNSTER} Cells by Using the Total Reflection X-ray Fluorescence Technique, Journal of Power Sources, 2016, 329, 364-371.



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Is the TMD affected by HF in the Electrolyte?



WWU Evertz et al, Unraveling Transition Metal Dissolution of Li_{1.04}Ni_{1/3}Co_{1/3}Mn_{1/3}O₂ (NCM 111) in Lithium Ion Full Cells by Using the Total Reflection X-ray Fluorescence Technique, Journal of Power Sources, 2016, 329, 364-371.

Influence of the Stoichiometry of Layered Cathode Materials on the TMD

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Kasnatscheew et al. Improving cycle life of layered lithium transition metal oxide (LiMO₂) based positive electrodes for Li ion batteries by smart selection of the electrochemical charge conditions, Journal of Power Sources, 2017 359, 458-467.

Influence of the Transition Metal Dissolution





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- TXRF analysis of all cell components revealed that the dissolution of transition metals is uniform
- Materials do not behave significantly different
- Highest active material found on the cathode/separator interface
- Results are calculated with respect to the active mass of the positive electrode
- No pronounced manganese dissolution is observable
- In electrolyte soaked separators, always higher contents of nickel are found due to the Li/Ni mixing

Kasnatscheew et al. Improving cycle life of layered lithium transition metal oxide (LiMO₂) based positive electrodes for Li ion batteries by smart selection of the electrochemical charge conditions, Journal of Power Sources, 2017 359, 458-467.

Obtained electrolyte samples from EVs



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- The field-tested (street driven) prismatic 5 Ah cells were based on NMC/graphite cell chemistry
- The discharged LIB packs of EVs were disassembled into single cells
- No information about electrochemical cell performance nor about the used specific battery materials were available
- Cells were opened in a glove box (O_2 , $H_2O < 0.1$ ppm)
- Collected in aluminum vials
- Stored in a deep freezer until analysis



Overview of electrolyte samples investigated from EV cells of five global OEMs. The assumed application in EV is indicated, namely hybrid electric vehicle (HEV) and battery-powered electric vehicle (BEV).



Electrolyte degradation – elemental analysis



- TXRF was applied in 6 samples for the identification and quantification of a possible metal dissolution from cathode materials (Fe, Ni, Mn, Co), copper dissolution from the anode current collector or battery materials and housing originated contamination (Ni, Fe, Zn, Cl, K, Ca)
- High copper concentrations (4-105 μg L⁻¹) indicate an anode current collector degradation for all EV samples
- The detected sulfur (27 mg L⁻¹) in sample A₂ could indicate the application of sulfur-based film-forming additives
- The detection of chlorine, potassium and calcium in all samples point to impurities of the applied cell materials

Sample	Mn	Fe	Со	Ni	Cu	Zn	CI	K	Са	S
	μg L ⁻¹	μg L ⁻¹	μg L ⁻¹	μg L ⁻¹	µg L⁻¹	µg L⁻¹	μg L-1	μg L ⁻¹	μg L-1	mg L ⁻¹
A ₁	3.8	104.9	3.6	45.6	210.4	<lod< th=""><th>55.1</th><th>28.2</th><th>16.7</th><th><lod< th=""></lod<></th></lod<>	55.1	28.2	16.7	<lod< th=""></lod<>
A ₂	1.3	3.9	<lod< th=""><th>0.9</th><th>5.4</th><th><lod< th=""><th>41.4</th><th>146.1</th><th>9.3</th><th>27.3</th></lod<></th></lod<>	0.9	5.4	<lod< th=""><th>41.4</th><th>146.1</th><th>9.3</th><th>27.3</th></lod<>	41.4	146.1	9.3	27.3
A ₄	<lod< th=""><th>14.7</th><th><lod< th=""><th>26.3</th><th>109.8</th><th>2.6</th><th>104.3</th><th>44.5</th><th>33.6</th><th><lod< th=""></lod<></th></lod<></th></lod<>	14.7	<lod< th=""><th>26.3</th><th>109.8</th><th>2.6</th><th>104.3</th><th>44.5</th><th>33.6</th><th><lod< th=""></lod<></th></lod<>	26.3	109.8	2.6	104.3	44.5	33.6	<lod< th=""></lod<>
B ₅	n.d.	6.1	<loq< th=""><th>88.9</th><th>609.5</th><th><lod< th=""><th>86.3</th><th>60</th><th>13.6</th><th><lod< th=""></lod<></th></lod<></th></loq<>	88.9	609.5	<lod< th=""><th>86.3</th><th>60</th><th>13.6</th><th><lod< th=""></lod<></th></lod<>	86.3	60	13.6	<lod< th=""></lod<>
D ₂	<loq< th=""><th>15.8</th><th><lod< th=""><th>0.7</th><th>163.4</th><th><lod< th=""><th>88.9</th><th><lod< th=""><th>14.6</th><th><lod< th=""></lod<></th></lod<></th></lod<></th></lod<></th></loq<>	15.8	<lod< th=""><th>0.7</th><th>163.4</th><th><lod< th=""><th>88.9</th><th><lod< th=""><th>14.6</th><th><lod< th=""></lod<></th></lod<></th></lod<></th></lod<>	0.7	163.4	<lod< th=""><th>88.9</th><th><lod< th=""><th>14.6</th><th><lod< th=""></lod<></th></lod<></th></lod<>	88.9	<lod< th=""><th>14.6</th><th><lod< th=""></lod<></th></lod<>	14.6	<lod< th=""></lod<>
E ₁	23.9	5.1	<lod< th=""><th>2390.9</th><th>90.2</th><th><lod< th=""><th>92.1</th><th>172.7</th><th>14.4</th><th><lod< th=""></lod<></th></lod<></th></lod<>	2390.9	90.2	<lod< th=""><th>92.1</th><th>172.7</th><th>14.4</th><th><lod< th=""></lod<></th></lod<>	92.1	172.7	14.4	<lod< th=""></lod<>



Overview



Conclusion and Outlook





Conclusion: HF-free Metal Migration Mechanisms

Schematic consideration of the three postulated reasons for HF-free TMD:

I) impurities within the NCM host lattice from synthesis, like oxygen vacancy,

II) structural deformation of NCM particles on the atomic scale leading to particle cracking or

III) by phase transformation from layered to spinel structures on the nanoscale level





Evertz et al, Unraveling Transition Metal Dissolution of Li₁₀₄Ni_{1/3}Co_{1/3}Mn_{1/3}O₂ (NCM 111) in Lithium Ion Full Cells by Using the Total Reflection Xray Fluorescence Technique, Journal of Power Sources, 2016, 329, 364-371. Evertz et al., Development of a Method for Direct Elemental Analysis of Lithium Ion Battery Degradation Products by Means of Total Reflection X-Ray Fluorescence, Spectrochimica Acta Part B: Atomic Spectroscopy, 2015, 112, 34-39.







Q&A

Any Questions?

Please **type in** the questions you may have for our speakers in the **Questions Box** and click **Submit**







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