WHITE PAPER

Quality control of analytical parameters in battery production

Replacing traditional fuel-powered vehicles with battery-powered options is essential to reduce carbon dioxide (CO_2) emissions. This greenhouse gas results from fossil fuel combustion—limiting its input into the atmosphere will also influence its effects on global warming. The demand of battery-powered electric vehicles (EVs) continues to rise in harmony with governments (e.g., the European Union, Canada) prohibiting the use of combustion engines in the future [**1**,**2**]. The International Energy Agency predicts that by 2030 60% of all new car sales will be EVs [**3**]. At the same time, renewable energy sources like wind and solar require storage capacities for electricity. Batteries are currently the most scalable materials in which to store excess electricity, and the market has been growing strongly as countries begin to invest in energy storage solutions [**4**].

Lithium-ion batteries (LIBs) are the most common rechargeable options available today. Production of LIBs needs to follow stringent quality standards. The water content, residual alkali content, or ionic impurities can have a negative impact on the safety and capacity of LIBs. Meanwhile, the composition of cathode materials or battery electrolyte can influence production costs and performance qualities of the final product. This White Paper elaborates how titration and ion chromatography can be used to monitor various battery quality parameters.

INTRODUCTION

A lithium-ion battery (LIB) consists of an anode (negative pole) and a cathode (positive pole). An electrolyte facilitates charge transfer in the form of lithium ions between these two poles. Meanwhile, a separator placed between the anode and cathode prevents short-circuits. An example cross-section can be seen in **Figure 1**.

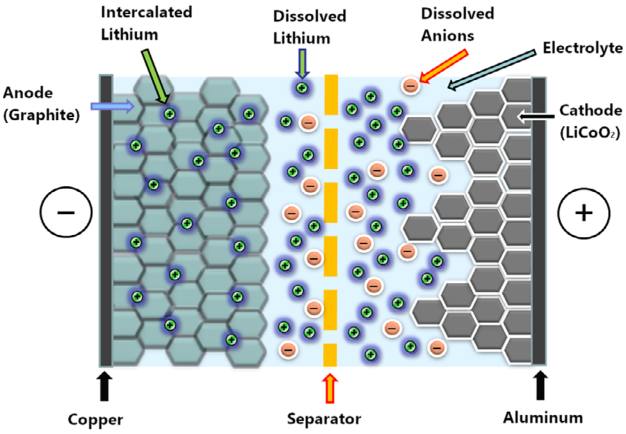

Figure 1. Illustrated cross-section of a lithium cobalt oxide (LCO) Li-ion battery.

The anode is made from graphite containing intercalated lithium applied to a copper foil, while the cathode consists of lithium metal oxides applied to an aluminum foil. Cobalt, nickel, manganese, or iron are the most commonly used transition metals in cathode materials. The electrolyte is an anhydrous aprotic solvent containing a lithium salt (e.g., lithium hexafluorophosphate) to facilitate charge transfer. The separator is an isolator made from a porous material, allowing the migration of lithium ions for charge transfer. The composition of all of these components has a significant influence on the battery characteristics.

The life cycle for LIBs can be broken down into four distinct categories: upstream, midstream, downstream, and waste. As displayed in **Figure 2**, each category can be further divided based on different materials or process steps. Upstream, the quality of the raw materials is critical as impurities can negatively affect the processing steps for midstream materials. Midstream, the correct composition of cathode material and electrolyte comes into focus as these can affect the cost and performance of Li-ion batteries significantly. Nevertheless, monitoring of impurities which may be introduced during synthesis or processing is still required as they can affect the LIB quality and

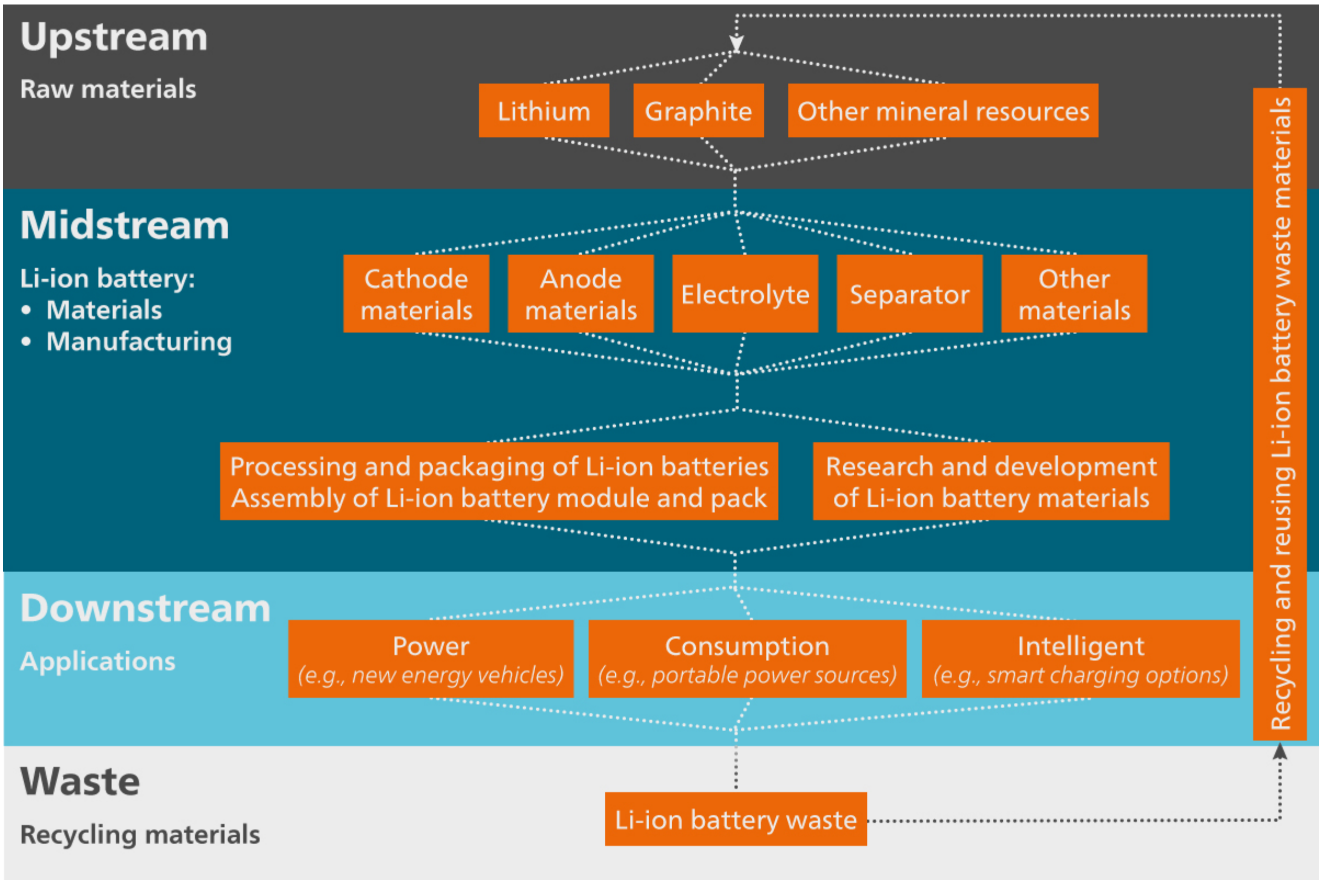

Figure 2. Schematic of the manufacturing process for LIBs.

Table 1. Overview of selected analyses offered by Metrohm and where they can be applied in the manufacturing process of LIBs. Even more analyses can be found in our flyer mentioned in the related literature section at the end of this White Paper.

Part of LIB manufacturing process	Analysis
Raw materials (Li salts, solvents, etc.)	Purity
	Moisture
	Impurities (chloride, sulfate, etc.)
Cathode materials	Moisture
	Composition of metals
	Residual alkali
	Phosphate (phosphoric acid) in lithium iron phosphate (LFP) starting material
Anode materials (graphite, graphene)	Moisture
Electrolyte	Lithium salt composition
	Moisture
	Impurities (chloride, sulfate, etc.)
Separator	Moisture
Other materials used in LIBs	Moisture
	Impurities (chloride, acids, etc.)
Processing, assembly, and packaging	Moisture
	Residual alkali

safety during use (i.e., downstream). At the end of a LIB's lifetime, recycling of the battery components is key to achieving a sustainable and circular economy. Here, testing for purity and for specific impurities of the recovered materials is again important.

Metrohm offers various analytical testing solutions for the quality control of LIB materials throughout the entire production process. **Table 1** summarizes applications which will be covered within this White Paper and the process area where they can be applied.

The following sections will discuss some of the main analytical parameters used for proper quality control in Li-ion battery production and how they can be solved with Metrohm solutions. The topics of **moisture**, **residual alkali or soluble base content**, **metals in cathode materials**, **Li salt composition in electrolyte**, and **ionic impurities** will be covered.

MOISTURE

Lithium-ion batteries should be virtually free of water because water reacts with the conducting salt (e.g., $LiPF_6$) to form toxic hydrofluoric acid (HF) [**5**]. Traces of water can also negatively impact the electrochemical performance of the LIB [**6**]. This is especially the case for nickel-rich cathode materials, where Ni^{3+} in the presence of water and CO_2 promotes the formation of LiOH and $LiCO_3$ (see also **Residual Alkali**) [**7**]. Sensitive coulometric Karl Fischer (KF) titration is the ideal method for determining water content at trace levels [**5,8,9**].

With coulometric KF titration, an electric current is used to generate iodine rather than adding it via buret, as is done during a classical volumetric KF titration. The current releases a stoichiometrically corresponding amount of iodine from the iodide-containing KF reagent by electrolysis. During the titration, the time and current are measured until the endpoint is reached. The amount of water within the sample is directly proportional to the product of time and current. **Table 2** summarizes how different battery

Table 2. Overview of how moisture content in different battery raw materials and final products can be analyzed.

Material	Method	Remarks
Solvents for electrolyte	Direct titration	Ketones (e.g., acetone) and aldehydes require alcohol-free reagents for titration
Electrolyte additives (e.g., VC, FEC)	Direct titration	Alcohol-free reagents needed for titration
Lithium borate salts (e.g., LiBOB, $LiBF_4$)	Direct titration	Alcohol-free reagents needed for titration
Final electrolyte	Direct titration	Alcohol-free reagents needed for titration
Carbon black, graphite	Oven method	
Anode slurries, Cathode slurries	Oven method	
Coated anode foil, coated cathode foil, separator foil	Oven method	
Final battery	Oven method	

materials can be analyzed by coulometric Karl Fischer titration.

Liquid and dissolved samples, such as solvents for the electrolyte or the final electrolyte, can be analyzed directly. The sample (liquid or dissolved) is added as-is into the closed coulometric titration cell using a syringe. However, electrolyte additives like vinylene carbonate (VC) or fluoroethylene carbonate (FEC) as well as lithium borate salts (e.g., LiBOB, $LiBF_4$, etc.) undergo side reactions with alcohol—the main solvent in standard Karl Fischer reagents. The use of alcohol-free reagents is therefore required [**10,11**]. Currently, the only commercially available alcohol-free KF reagents are Hydranal™ NEXTGEN Coulomat A-FA/C-FA from Honeywell. **Figure 3** shows two examples, each using a methanol-based KF reagent (in blue) and an alcohol-free KF reagent (in red) for the titration. In the case of the titration of 1 mL 1 mol/L $LiPF_6$ in EC/DMC with 5.5 wt% LiBOB (**Figure 3A**) both titrations give stable endpoints, but only Coulomat A-FA/C-FA provides the correct result. **Figure 3B** shows the titration of 2 mL VC, where no endpoint is reached for standard KF reagent (titration was stopped manually), which a stable endpoint is reached for the alcohol-free Coulomat A-FA/C-FA [**11**].

Due to the high sensitivity to atmospheric moisture, coulometric titration cells should not be opened when adding samples. Therefore, solid samples, such as cathode, anode, or separator materials, are best analyzed by coulometric Karl Fischer titration after gas extraction with an oven (oven method). In this method the sample is weighed under inert gas conditions into a headspace vial and closed with a septum cap. Then the vial is placed in the oven and heated to a predefined temperature, leading the sample to release its water. A dry non-reactive carrier gas transfers the vaporized water into the titration cell where the water content determination takes place. **Figure 4** depicts an automated system for the moisture determination using the oven method.

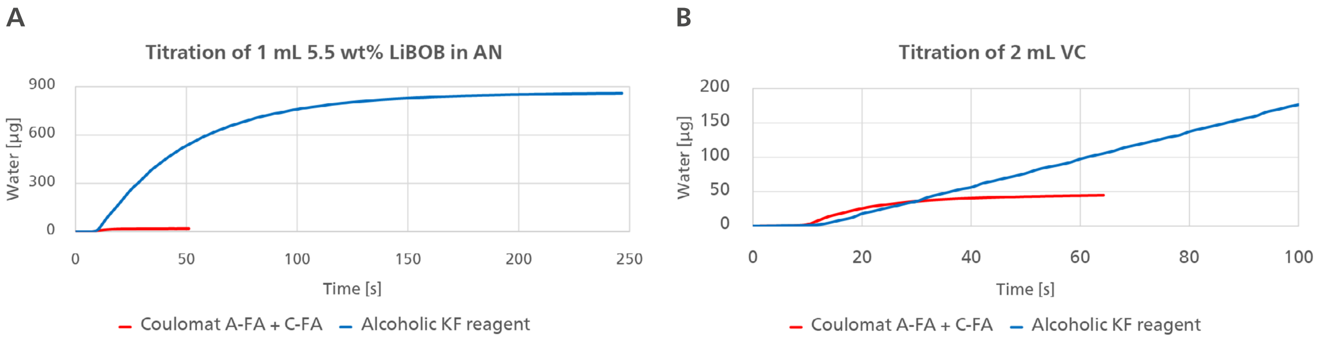

Figure 3. Comparison of the KF titration with alcoholic KF reagents (blue curves) and alcohol-free Hydranal NEXTGEN Coulomat A-FA/C-FA (red curves). A) Titration of 1 mL 1 mol/L $LiPF_6$ in EC/DMC with 5.5 wt% LiBOB and B) titration of 2 mL VC. All data provided by Honeywell.

Figure 4. A Metrohm coulometric Karl Fischer titration system consisting of an 851 Titrando and an 874 Oven Sample Processor for the automatic analysis of solid battery samples.

RESIDUAL ALKALI OR SOLUBLE BASE CONTENT

Metal carbonates (e.g., lithium carbonate) are formed during the reaction between surface hydroxides and CO_2 when cathode materials are exposed to ambient air. These surface carbonates, also referred to as residual alkali or soluble base content, can lead to the out-gassing of CO_2 and CO. This can cause swelling in polymer batteries [**12,13**]. Surface metal carbonates can also negatively impact the slurry preparation of cathode materials. Gelation can occur, causing processing problems during the battery production [**13,14**]. However, the residual alkali content is not just seen as an impurity, but rather as a surface property used for quality control [**15**].

The residual alkali content or soluble base content can be determined with high reproducibility using pH titration. Hydrochloric acid (HCl) is commonly used as the titrant [**14**]. The same titration is also proposed by the International Organization for Standardization (ISO) for the quantitative determination of lithium hydroxide (LiOH) and lithium carbonate (Li_2CO_3) [**16,17**]. For the analysis, a defined amount of sample (e.g., 5 g of cathode powder) is added to 100 mL of degassed ultrapure water. The dispersion is filtered after a defined reaction time and an aliquot is then titrated. Two equivalence points are observed at approximately at pH 8.5 and 4.5. **Figure 5** displays an example titration curve for pure lithium hydroxide. The second equivalence point (EP 2) corresponds to the total residual alkali content, the difference between the two equivalence points corresponds to the residual carbonate content (Li_2CO_3), while the residual hydroxide (LiOH) content can be calculated from the difference between the residual alkali and residual carbonate content.

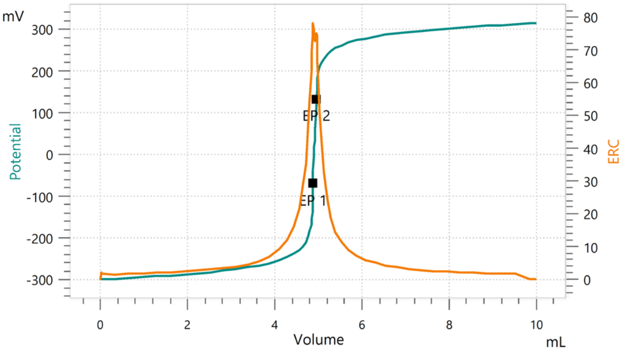

Figure 5. Titration of LiOH (purity >99%) with HCl. EP 1 corresponds to the lithium hydroxide content, while EP 2 corresponds to the lithium carbonate impurity.

It is crucial that the sample, filtrate, and titration aliquot are protected from the uptake of CO_2 from the environment. **Table 3** displays the results of residual alkali titration on an automated titration system as depicted in **Figure 6**. The sample beakers were left uncovered in the first series (**Table 3**, left side). In the second series (**Table 3**, right side), the samples remained covered up just prior to the titration. The results demonstrate that without a beaker cover, the carbonate content increased over the titration series, while when covered the carbonate values remained stable.

Figure 6. Metrohm offers solutions for the automated titration of the residual alkali content in cathode materials. This system is equipped with the Dis-Cover functionality to prevent the uptake of CO_2 from the environment. Before the titration, the system automatically removes the cover for the most accurate results.

Table 3. Comparison of the automated assay of residual alkali content. Without a sample beaker cover, the carbonate content increases over time due to uptake of CO_2 from the ambient air. By using the OMNIS Dis-Cover system, the sample is protected and the carbonate content remains stable.

Sample without cover for beaker			Sample with Dis-Cover		
Sample size (g)	**Residual alkali expressed as LiOH (%)**	**Residual carbonate expressed as CO_3^{2-} (%)**	**Sample size (g)**	**Residual alkali expressed as LiOH (%)**	**Residual carbonate expressed as CO_3^{2-} (%)**
0.1227	56.24	0.86	0.1227	55.92	0.84
0.1227	55.02	2.11	0.1227	55.83	0.85
0.1227	54.90	2.24	0.1227	56.20	0.90
0.1227	54.48	2.52	0.1227	56.00	1.04
0.1227	54.26	2.97	0.1227	55.91	0.95
0.1227	54.18	3.03	0.1227	55.96	0.93
0.1227	53.94	3.37			
Mean	**54.46**	**2.71**		**55.97**	**0.92**

METALS IN CATHODE MATERIALS

Cathode materials are usually either layered metal oxides like lithium cobalt oxide (LCO), or ternary metal oxides like lithium nickel manganese cobalt oxide (NMC). During the synthesis of cathode materials used for batteries, the composition of the starting solutions and of the final cathode material is essential for optimizing production costs. Potentiometric titration is a proven analysis technology to test the metal composition of cathode materials [**18–20**]. Titration has several advantages over other analysis methods like ICP-OES (inductively coupled plasma - optical emission spectrometry). Compared to ICP-OES, titration can handle a much higher metal concentration – dilution of cathode materials before analysis is therefore unnecessary, improving the accuracy and precision of the results. In addition, titration has lower investment and running costs compared to other sophisticated methods and requires hardly any training to perform the analysis.

Titration of layered metal oxides is straightforward with a single titration. Ternary metal oxides require more than one titration to differentiate between the different metal species. **Table 4** summarizes how the different metals within various cathode materials are determined by titration.

Table 4. Overview of the different kinds of titration methods used to analyze several metals in various cathode materials.

Cathode name	Metal	Titration	Remarks
NMC Lithium nickel manganese cobalt oxide	Total metal content	Complexometric titration with EDTA	Titration at pH 10 **YS/T 1006.1** describes this analysis
	Nickel	—	Value calculated from total metal content and manganese and cobalt content
	Manganese	Redox titration with $KMnO_4$ after addition of pyrophosphate	**YS/T 1472.1** describes this analysis
	Cobalt	Redox titration with ferricyanide $[Fe(CN)_6]^{3-}$	Back titration of excess $[Fe(CN)_6]^{3-}$ with cobalt standard solution **YS/T 1472.2** describes this analysis
LFP Lithium iron phosphate	(Total) Iron	Redox titration with potassium dichromate $K_2Cr_2O_7$	Iron(III) is reduced to iron(II) with $TiCl_3$ before the titration **YS/T 1028.1** describes this analysis
	Phosphate / phosphoric acid (as starting material)	Acid-base titration with NaOH	—
LCO Lithium cobalt oxide	Cobalt	Complexometric titration with EDTA	Titration at pH 10 **GB/T 23367.1** describes this analysis
LMO Lithium manganese oxide	Manganese	Redox titration with ferrous ammonium sulfate (FAS) $(NH_4)_2Fe(SO_4)_2$	—
NCA Lithium nickel cobalt aluminum oxide	Cobalt	Redox titration with ferricyanide $[Fe(CN)_6]^{3-}$	Back titration of excess $[Fe(CN)_6]^{3-}$ with cobalt standard solution **YS/T 1263.2** describes this analysis

LITHIUM SALT COMPOSITION IN ELECTROLYTE

Lithium hexafluorophosphate ($LiPF_6$) is the main conductive salt used in commercially available LIBs. It has a good conductivity, chemical inertness, and most importantly, the ability to passivate the aluminum current collector [**21**]. However, $LiPF_6$ tends to decompose into LiF and PF_5 at elevated temperatures. It can also react with traces of water to form toxic HF. Because of these safety issues, alternative lithium salts are being investigated for use in production of LIBs. Currently, lithium borate salts (e.g., LiBOB, LiODFB, or $LiBF_4$) or imide-based lithium salts (e.g., LiFSI, LITFSI) are often used as additives to the electrolyte to mitigate some of the safety issues and improve the high-temperature performance of LIBs.

With ion chromatography (IC), it is possible to quantify the amount of different lithium salts in an electrolyte by determining the anion composition of the different salts. To determine the different counter anions, approximately half a gram of electrolyte is dissolved in ultrapure water. After thoroughly mixing, an aliquot of the sample solution is directly injected into the ion chromatograph (**Figure 7**).

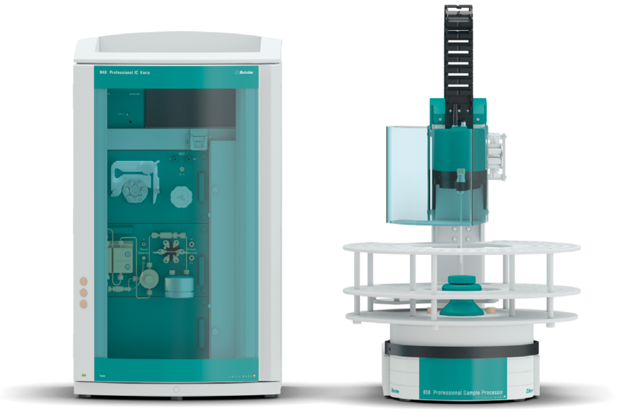

Figure 7. Ion chromatography system consisting of a 940 Professional IC Vario ONE/SeS/PP, an 850 Conductivity detector, and an 858 Professional Sample Processor.

Table 5. Results for the analysis of different lithium salt counter anions in a simulated LIB electrolyte.

Sample size (g)	ODFB (%)	PO_2F_2 (%)	BF_4 (%)	BOB (%)	PF_6 (%)	TFSI (%)	FSI (%)
0.4120	0.57	0.59	0.56	0.52	12.4	0.48	0.50
0.4032	0.54	0.53	0.54	0.51	12.1	0.48	0.49
Mean	**0.56**	**0.56**	**0.55**	**0.52**	**12.3**	**0.48**	**0.50**

Separation occurs on an anion exchange column, and suppressed conductivity detection is used to quantify the individual anions. **Figure 8** shows a chromatogram of a simulated LIB electrolyte analyzed on an IC system as depicted in **Figure 7**. The results are displayed in **Table 5**.

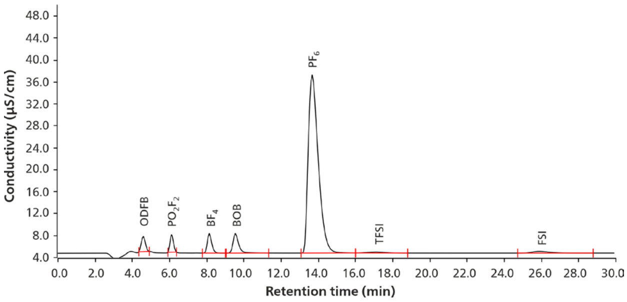

Figure 8. Chromatogram of a simulated LIB electrolyte consisting of seven commonly used lithium salts.

IONIC IMPURITIES

During the incorporation of lithium ions (intercalation) into the anode, the electrolyte is reduced at the anode surface, forming the solid electrolyte interphase (SEI). The SEI layer acts as an insulator yet at the same time being conductive for lithium ions, and the stability of the SEI directly influences the lifetime of LIBs [**22**]. If the SEI is not stable, it will continue to grow after each charging-discharging cycle and the cell will ultimately fail [**7**]. Impurities within the electrolyte can have a negative impact on the SEI, leading to a decreased lifetime and lower capacity of the LIB.

IC is ideally suited to detect ionic impurities at trace levels in the raw materials used to produce the electrolyte, cathode, or anode materials. For the analysis of cationic impurities in lithium salts, the pure salt (e.g., $LiPF_6$) is dissolved in ultrapure water. The sample is then diluted further before being injected into the ion chromatograph. Separation occurs on a cation exchange column, and suppressed conductivity detection is used to quantify the individual cations.

In the case of fluoroethylene carbonate (FEC), high anion concentrations (mainly fluoride) can disturb the analysis of trace anions like chloride. With 2D separation, the analysis becomes possible. For the analysis, the ions are extracted from FEC with ultrapure water. The aqueous extract is then diluted further and injected into the ion chromatograph. First, the interfering ions are pre-eluted with an ion-exclusion column (first step). The chloride is held on a preconcentration column and is then injected onto an ion-exchange column (second step). **Figure 9** shows a chromatogram of a simulated LIB electrolyte. The results of this example are displayed in **Table 6**.

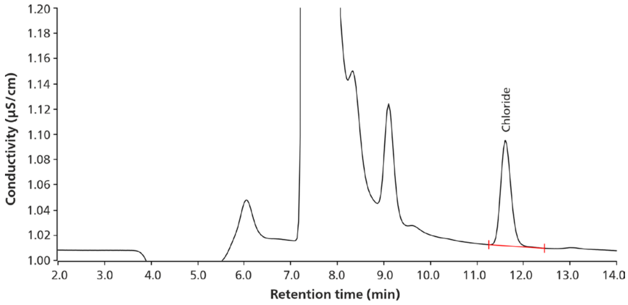

Figure 9. Chromatogram of the analysis of chloride in FEC after elimination of other anionic matrix constituents using two-dimensional ion chromatography.

Table 6. Results for the determination of chloride in FEC after two-dimensional IC separation.

Sample	Chloride (mg/L)
FEC-1	4.447
FEC-2	4.274

CONCLUSION

Analytical testing of various quality parameters like moisture content or material composition is necessary over the entire production process of Li-ion batteries. Moisture content and ionic impurities which negatively impact the safety, lifetime, or performance of LIBs can be analyzed using Karl Fischer titration or ion chromatography. Titration and IC can also be used to monitor the composition of final cathode materials and electrolytes ensuring a cost-efficient production of these materials. For more information about Li-ion battery analysis, read the materials gathered in the «**Further related Metrohm literature**» section at the end of the White Paper.

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Assay of lithium hydroxide and lithium carbonate. **AN-T-215**

Analysis of Li-ion battery cathode materials made from Co, Ni, and Mn. **AN-T-218**

Composition of lithium salts in battery electrolyte. **AN-S-372**

Trace cations in lithium hexafluorophosphate. **AN-CS-011**

Battery research and production – Materials, parameters, and analytical methods. **8.000.5429**

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