Chemical Analysis for Battery Manufacturing Compendium

Improve Battery Safety, Charge Time, Power Output, and Longevity

Move forward faster with advanced battery solutions for every step from research through production to recycling

As Lithium-ion batteries play an increasing crucial role in everything from handheld electronics, electrics vehicles and electrical storage units - your lab needs automated, reliable and robust solutions with superior stability. From the extraction of processing raw materials, through quality assurance and the production line, to material recovery in recycling, as well as, assisting with the research and development of next-generation batteries, our analytical solutions can optimize your lab.

Our routine analysis and research solutions enable researchers and analytical science labs to improve battery safety and longevity, reduce charging time, improve scrap rate and enhance power output.

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Research and Development

The advancement of battery technology is central to progress in various sectors, including hybrid electric vehicles and consumer electronics. Central to improving battery performance is the continuous development of materials tailored to the various components. It is essential to understand and address anode deterioration to improve the longevity, safety, and overall performance of LiBs. To overcome these challenges and drive the evolution of energy storage solutions, ongoing research and development efforts in materials science, electrochemistry, and battery management are essential.

The criteria set by the application and the cell itself are typically complex, and battery research and development are strongly driven and evaluated against many parameters. For an EV, the requirements for energy density, chemistry, cell design, calendar, cycle life, safety, range, cost, power, and durability will greatly influence the requirements at the cell level. These specifications are determined by several criteria, including regulatory safety requirements in the target markets and the demands of a particular application. Research into electrolyte degradation and chemical analysis can help identify important electrolyte degradation products and clarify mechanistic pathways that, when inhibited, may improve degradation resistance and result in safer, longer-lasting LiBs.

Our comprehensive suite of analytical and structural characterization solutions is designed to enable you to rapidly innovate materials and products to improve battery quality, performance, and safety. By leveraging our advanced analytical techniques, including ion chromatography (IC), high-resolution mass spectrometry (HRMS), orbitrap gas chromatography-mass spectrometry (GC-MS), IC with suppressed conductivity detection (IC-CD), electron microscopy, IC coupled with high-resolution accurate mass spectrometry (IC-HRAM MS), direct infusion HRAM MS, automation, and software solutions, you can effectively address the complexities of battery research and development. Furthermore, our offerings are supported by experienced service and support to ensure you receive the assistance you need to implement these solutions seamlessly.

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POSTER NOTE | 71652

Identification of phosphorus-containing degradation products obtained from surface deposits on lithium-ion battery anodes using ion chromatography coupled to high resolution accurate mass MS/MS

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Overview

Purpose: This poster note will focus on work our group has done to identify phosphorus-containing degradation products obtained from surface deposits on lithium-ion battery (LiB) anodes showing varying degrees of capacity loss.

Methods: Ion chromatography (IC) separates analytes based on ion-exchange selectivity, which provides information on the ionizable functional groups present in each analyte. A hydroxide eluent gradient was used to separate anionic sample components on ananion-exchange column and the analytes were detected both by electrical conductivity and high resolution accurate-mass (HRAM) MS/MS.

Results: Compound classes and specific compounds that were found in these samples include methyl carbonate, ubiquitous anionic contaminants such as chloride and sulfate, electrolyte breakdown products such as fluoride, phosphate and pyrophosphate, organic acids derived from degradation of the anode as well as ionic materials derived from reactions between various ion classes found in the samples including: sulfate esters, phosphate esters and fluorophosphate esters. This poster note focuses on the phosphorus-containing species.

Conclusion

- Ion chromatography provides ion-exchange separations of anionic (or cationic) sample components
- The IC with a conductivity detector is coupled to an HRAM MS to provide information in the elucidation of unknowns
- Analytes are eluted in the order of monovalent<divalent<trivalent <higher by ion-exchange separation so information is provided on key structural features
- To date we have found components from the aging of LIB anodes in several chemical classes including carboxylic acids, esters, phosphate esters, fluorophosphate esters, sulfate esters, as well as inorganic anions
- The proposed decompositon pathway in Figure 2 is supported by our independent identification of the circled products

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POSTER NOTE | 64526

Comprehensive analysis of lithium-ion battery anode samples by ion chromatography coupled with high resolution mass spectrometry

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Overview

Purpose: To demonstrate a workflow using ion chromatography (IC) and high resolution mass spectrometry (HRMS) for lithium-ion battery (LiB) anode degradation product analysis.

Results: LiB anode degradation products were identified from four anode samples.

Conclusion

- Ion chromatography coupled with a Q Exactive mass spectrometer provides a powerful platform for LiB anode impurity and degradant analysis
- Phosphate degradation products in three aged LiB were identified
- This IC-HRMS platform can be used for QA/QC for LiB manufacturers and performance evaluations
- Further study will be conducted to investigate the relationship of the compounds identified and their intensities with the batteries performance

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CASE STUDY | MEET BATTERY RESEARCH CENTER

Orbitrap GC-MS technology provides new insight into lithium ion battery degradation

Advancing lithium-Ion battery technology

From consumer electronics to electric vehicles, the growing demand for better-performing, safer, and less costly batteries has led researchers to focus on improving several aspects of lithium ion battery technology. Münster Electrochemical Energy Technology (MEET), the battery research center at Münster University, aims to address electrolyte aging, a major factor affecting lithium ion battery life. Using the Thermo Scientific™ Q Exactive™ GC Orbitrap™ GC-MS/MS system, MEET's Analytics and Environment division gains a broader and deeper understanding of their samples that in turn provides new insight into the complex reaction mechanisms involved in electrolyte aging. Ultimately these insights will enable the research team to identify additives to curtail, or even halt, electrolyte aging.

Conclusion

The Q Exactive GC Orbitrap system brings together the power of high-resolution GC and high-resolution accurate-mass (HRAM) Orbitrap MS to provide more comprehensive characterization of samples in discovery analysis. With the Q Exactive GC-MS/MS system, MEET accesses the exact mass information of more compounds, at significantly lower levels. The result is a broader and deeper understanding of the complex reaction mechanisms involved in electrolyte aging.

View the full application note

POSTER NOTE | 71671

Analysis and identification of ionic manganese containing degradation products from the anode surface of cycle & calendar aged lithium-ion batteries

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Overview

Purpose: Identify the chemical composition of manganese degradation products (deposited on the anode surface of a cycle aged lithium-ion battery—LiB) via peaks resolved by ion chromatography (IC).

Method: Extract the electrolyte degradation products from the anode surface using deionized (DI) water. Measure the retention time of the peaks of interest from the aqueous extract using an ion chromatography system with an hydroxide-selective anion-exchange column, and correlate the results using IC with suppressed conductivity detection (IC-CD), IC coupled with high resolution accurate mass spectrometry (IC-HRAM MS), and direct infusion HRAM MS.

Results: Identification of a proposed permanganate containing degradation product by IC-HRAM MS (-ESI mode) by component identification software was consistent with the expected elution profile and supported with an *m/z* to two decimal point accuracy. However, an *m/z* ratio to four decimal point accuracy disproved the proposed product.

Conclusion

Analysis of water soluble electrolyte degradation products (deposited on the anodesurface of aged LiB samples) for Mn using anion exchange IC-ICP-MS were retained under alkaline eluent conditions suggesting an anionic Manganese oxide complex such as $Mn_xO_y^{Z-}$.

A comparison between a permanganate standard (considered a likely degradation product) and the calendar aged LiB sample by anion exchange IC shows a major peak from the calendar aged sample co-eluting with the permanganate standard. However the permanganate standard also included smaller peaks that co-elute with manganate and carbonate standards suggesting permanganate may not be stable on the column. Analysis of a permanganate standard by direct infusion -ESI HRAM MS provided a positive response, but a negative response to -ESI IC-HRAM MS. The results support the premise that permanganate may not stable on the column.

Analysis and identification of ionic manganese containing degradation products from the anode surface of cycle & calendar aged lithium-ion batteries *(continued)*

Elemental composition based on HRAM data of the cycle aged sample produced a negative response to permanganate but a positive response to a proposed Mn containing compound ($C_4H_4O_8Mn$) that corresponded to a structure containing a permanganate moiety. The proposed structure was consistent with the observed retention time, and matched a *m/z* to 2 decimal point accuracy, but, not to 4 decimal point accuracy. As a result, the proposed chemical structure was dismissed as the identified compound, saving time and resources that could have been spent pursing it further based on 2 decimal point accuracy. No other Mn containing product were proposed.

Results from the study including elution behavior by IC, possible interaction with the column, and compound identification by HRAM MS, suggest the Mn compound observed by IC-ICP-MS is not Mn oxide based but may instead be carboxylate or carbonate based, such as $MnC_xO_y^{Z-}$.

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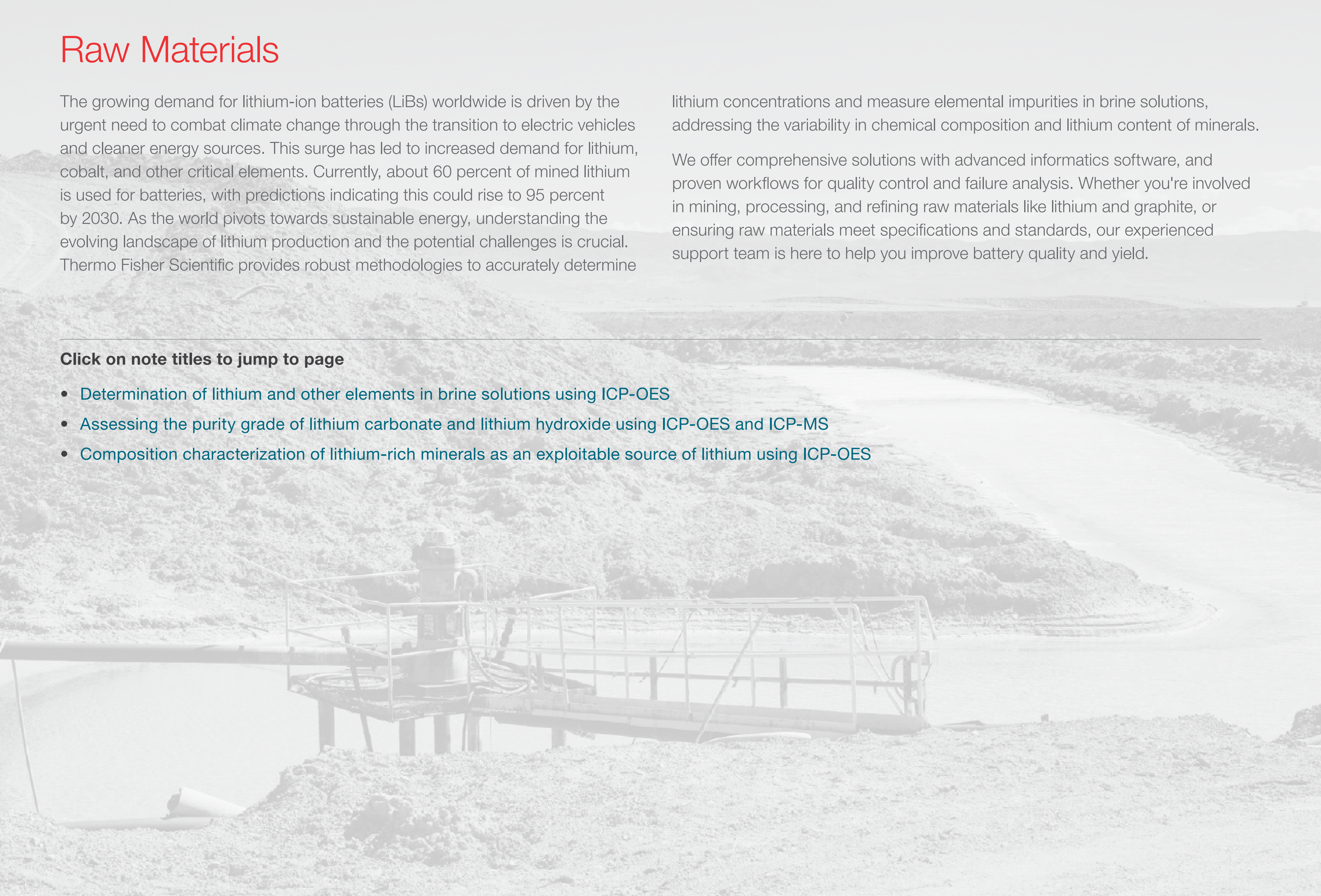
Raw Materials



The growing demand for lithium-ion batteries (LiBs) worldwide is driven by the urgent need to combat climate change through the transition to electric vehicles and cleaner energy sources. This surge has led to increased demand for lithium, cobalt, and other critical elements. Currently, about 60 percent of mined lithium is used for batteries, with predictions indicating this could rise to 95 percent by 2030. As the world pivots towards sustainable energy, understanding the evolving landscape of lithium production and the potential challenges is crucial. Thermo Fisher Scientific provides robust methodologies to accurately determine lithium concentrations and measure elemental impurities in brine solutions, addressing the variability in chemical composition and lithium content of minerals.

We offer comprehensive solutions with advanced informatics software, and proven workflows for quality control and failure analysis. Whether you're involved in mining, processing, and refining raw materials like lithium and graphite, or ensuring raw materials meet specifications and standards, our experienced support team is here to help you improve battery quality and yield.

Click on note titles to jump to page

- Determination of lithium and other elements in brine solutions using ICP-OES
- Assessing the purity grade of lithium carbonate and lithium hydroxide using ICP-OES and ICP-MS
- Composition characterization of lithium-rich minerals as an exploitable source of lithium using ICP-OES

APPLICATION NOTE | 000602

Determination of lithium and other elements in brine solutions using ICP-OES

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Goal

To develop a robust methodology for the determination of a wide concentration range of lithium together with trace level measurement of other critical elemental contaminants in brine solutions using the Thermo Scientific™ iCAP™ PRO Series ICP-OES.

Introduction

As part of the global initiative on sustainability and green energy, battery electric vehicles (BEVs) are rapidly gaining in popularity and their share of the vehicle market is expected to increase at least 10-fold over the next decade. After a few years of rapid growth, there are now more than 10 million electric cars on the road globally, and this is anticipated to rise to about 40 million in 2030 and up to 300 million by 2050.[1]

Currently, electric vehicles most often use batteries based on lithium-ion technology because of the high energy density of such batteries relative to their weight, their ability to undergo multiple charge / discharge cycles before significantly losing performance, and their relatively low cost. Since the late 1990s, advances in the performance of lithium-ion battery technology have mainly been driven by demand from portable electronics, laptop computers, mobile phones, and power tools. However, the rapid rise of BEVs and hybrid electric vehicles (HEVs) has intensified research and development of a new generation of batteries with greater robustness and higher charge capacity to significantly increase the achievable range of these vehicles between each charging cycle.

The rapidly increasing demand for Li-ion batteries has led to a critical need for exploration and development of additional natural resources of lithium as well as other commonly used elements in battery manufacturing, such as manganese, nickel, and cobalt. In addition to mineral sources, such as spodumene ore, underground brine reserves are also rich sources of lithium. There are various methods for extracting lithium from brine, including precipitation, liquid-liquid extraction, selective membrane separation, electrodialysis, and ion exchange adsorption.[2] With cost and efficiency taken into consideration, extraction of lithium ions from solutions by ion exchange adsorption is one of the most effective methods. The abundance of lithium in brine solutions can vary significantly in the concentration range of 10 $mg \cdot L^{-1}$ or less to about 4,000 $mg \cdot L^{-1}$ or more depending upon the geography of the source.

To assess the quality of brine solutions from the perspective of their lithium content and to determine the cost-effectiveness and efficiency of the extraction process, it is important to accurately measure lithium concentrations in brine extracts prior to establishing the extraction process. The concentration of other trace impurity elements present in the original brine itself, the lithium

salts extracted from the brine, and the waste brine once the lithium has been extracted must also be determined, as these have an adverse impact on the quality of the material extracted during the process and can also have negative environmental impacts.

In this work, a comprehensive analytical method was developed and tested for analysis of brine solutions using the Thermo Scientific™ iCAP™ PRO XP ICP-OES Duo instrument, operated using the Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™ (ISDS) Software. The main objective of this application note is to present the performance of the method developed in the study for laboratories working in the lithium extraction area or otherwise involved in measuring concentrated salt solutions such as brine.

Summary and conclusions

- The quality of the analytical data observed in this study shows that the iCAP PRO XP ICP-OES equipped with a Burgener Mira Mist nebulizer and sheath gas adaptor is a reliable and robust analytical solution for the analysis of brine solutions and similar, equally challenging samples.
- The wide linear dynamic range established for lithium (up to 5,000 $mg \cdot L^{-1}$) together with the high degree of accuracy and precision across this linear range enables reliable quantification of lithium with varying concentrations, without the need for sample pre-treatments such as dilution. This enables quick sample turnaround, increasing the overall productivity of analytical laboratories involved with analysis of brine and other similar high matrix samples.
- The iCAP PRO series ICP-OES Duo instrument, with its vertical torch configuration and sheath gas adaptor, offers great flexibility for analyzing a wide range of analytes at both higher concentrations and trace levels in a single analytical measurement with robustness equivalent to that of radial ICP-OES instruments.
- The axial mode of the iCAP PRO Series ICP-OES provides significantly lower detection limits and consistently accurate analytical data while analyzing samples with high dissolved solids, enabling reliable measurement of analytes with emission wavelengths distributed across the entire wavelength range.
- The Qtegra ISDS Software simplifies method optimization through various available auto-tune options and provides useful analytical information with customizable report templates, reducing the manual effort required for data evaluation, re-processing, and results reporting.

References

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POSTER

Assessing the purity grade of lithium carbonate and lithium hydroxide using ICP-OES and ICP-MS

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Abstract

Purpose: Different approaches towards determining the purity grade of lithium salts using inductively coupled plasma optical emission spectroscopy (ICP-OES) and inductively coupled plasma mass spectrometry (ICP-MS).

Methods: Two separate methods were developed, one using ICP-OES and one using ICP-MS, for the highly sensitive analysis of elemental impurities in Li_2CO_3 and LiOH salts. Accuracy and precision measurements, and data stability checks, as well as uninterrupted sequences of circa twelve hours length were performed to validate the analytical methods.

Results: Reliable and accurate data was achieved, covering a large suite of target analytes with excellent detection limits at ppb (µg·L^{-1}), sub ppb, and ppt (ng·L^{-1}) levels. Both methods demonstrated excellent stability and robustness and data quality was not compromised by matrix effects.

Introduction

The superior energy density and high storage capacity of lithium-ion batteries has led to their rapid adoption in portable electronic devices, energy storage systems, and electric vehicle battery packs. The ongoing rapid growth of the electric vehicle market has increased the need for large quantities of high-quality lithium batteries that are safe to use and have long lifetimes. Therefore, the need for accurate, precise, and reliable battery material analysis is of utmost importance.

The two most important sources of lithium are lithium carbonate (Li_2CO_3) and lithium hydroxide (LiOH). This study describes two robust and reliable methods for the analysis of these lithium salts using either ICP-OES or ICP-MS. Modern day ICP-OES can meet the sensitivity requirements for most measured impurities for determining presently targeted purity levels. In the future it is likely that batteries with even higher efficiency and therefore, raw materials with lower impurities will be required, which will make a switch to a higher sensitivity technique like ICP-MS imperative. The targeted analytes includes common impurities, such as alkali and alkaline earth elements, transition elements, heavy metals – which may influence the performance efficiency of the final manufactured batteries. The ICP-MS method also covers the ultra-trace level lanthanide series rare earth elements (REE), which have an important use in high technology electrotechnical applications, as well as the highest concentration element in the salts – lithium (Li) itself.

Conclusions

The iCAP PRO XP ICP-OES Duo and the iCAP RQ ICP-MS with AGD instruments were used to analyze a total of 60 impurity elements in Li salt samples. The samples were prepared using a unified and simple sample preparation method which involved dissolving the samples in 2% nitric acid. The methods provided excellent results and reliable high-quality data.

- Both Li_2CO_3 and LiOH salts were analyzed applying a single method covering 45 elements simultaneously with the iCAP PRO XP ICP-OES Duo and 60 elements with the iCAP RQ ICP-MS.
- Excellent sensitivity was achieved in both methods. The ICP-MS enabled the analysis of a higher number of elements at lower detection limits.
- All selected wavelengths were covered within a single mode in ICP-OES, in this case Axial iFR – the wavelengths could all be captured simultaneously in the same exposure (10 s in this study) saving valuable analysis time and costs. This allowed measurement of all analytes together, at multiple wavelengths, without increasing the analysis time.
- A single sample preparation step was adopted, and sample extracts were fed directly into both the ICP-OES and/or ICP-MS instruments, depending on the target analytes and the sensitivity levels required. In the case of the iCAP RQ ICP-MS no additional manual dilution steps are necessary as AGD is performed at the ICP-MS instrument directly.
- Excellent spike recovery and QC results were obtained, and QC stability was demonstrated over 12 hours. The methods were found to be robust for up to a minimum of 12 hours and showed very stable internal standard responses, indicating minimum matrix effects for both the salts.
- The AGD is fully incorporated into the Qtegra ISDS software and allows for easy and simple operation. Data acquisition, visualization and reporting is straightforward and simple using the software for both methods.

View the poster note

APPLICATION NOTE | 000968

Composition characterization of lithium-rich minerals as an exploitable source of lithium using ICP-OES

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Goal

To demonstrate the suitability of the Thermo Scientific™ iCAP™ PRO X ICP-OES Duo for the elemental analysis of lithium mineral samples.

Introduction

The transition to electric vehicles (EVs) is one of the key developments of the green energy revolution, and global demand for these vehicles is soaring. Lithium-ion batteries are one of the most important power storage materials for EVs due to their power density and life cycle performance. The lithium used in the production of electric vehicle batteries and other electronic devices is obtained from sources such as underground brines[1] and lithium-rich minerals and rocks. While brine solutions can be potentially rich, relatively easy-to-access sources of lithium, consideration must be given to the potential impact of the exploitation activities themselves on climate change, related environmental risks, and raw material supply.[2] Mining lithium containing minerals is an alternative source for regions where underground brines are not existing or not possible to exploit, but developing lithium mining sites also requires environmental impact consideration.

Lithium containing minerals differ significantly in their chemical composition, characteristics, and lithium content.[3] To determine whether a given mineral or brine is worth extracting, it is important to obtain quantitative information about certain major and trace components in the mineral/brine (Table 1). Portable X-ray fluorescence (pXRF) is a technique widely used in the mining industry as it is a non-destructive analytical technique that is effective for determining the elemental composition of a sample directly in the field.

However, there are some limitations with XRF concerning the detection limits achievable. Inductively coupled plasma – optical emission spectroscopy (ICP-OES) is also widely used to screen minerals and ores for their major and trace

Table 1. Reasons for tracking specified elements in lithium mining samples[4]

Element	Information
Ca, Na, K	Indicates the amount of feldspar (one of the main rock-forming minerals) presents in the sample
K/Al ratio	Indicator of the degree of weathering (kaolinisation)
Ti	Associated with paramagnetic minerals that could dilute the final concentrate
Mn	Indicator enabling close tracking of lithium
Fe	Indication of the total amount of magnetic material not accounted for by zinnwaldite
Cr	Deleterious element for the hydrometallurgy process

elemental content. It is often the analytical technique of choice for the analysis of a large number of trace elements in a sample, as it allows simultaneous determination of major and minor elements in a single analysis due to its inherent multi-element capability and high dynamic range.

This application note describes an optimized analytical method for the analysis of a lithium containing mineral (zinnwaldite, a silicate based mineral of the approximate composition ($KLiFeAl(AlSi_3)O_{10}(OH,F)_2$) using the iCAP PRO X ICP-OES Duo. Thorough testing of the method was performed, including demonstration of accuracy and precision by certified reference material measurements, analysis of quality control standards, and the analysis of real samples as required in a mineralogical research laboratory.

Conclusions

The iCAP PRO X ICP-OES Duo instrument, equipped with a ceramic D-torch, was used to analyze 12 elements in digested rock samples containing commercially viable amounts of lithium. The results of this study showed that the analysis can be easily performed using a straightforward method to yield high accuracy and excellent analytical robustness. The method was rigorously tested, and the results obtained using a certified reference material clearly demonstrate the following analytical advantages:

- The large linear dynamic range for major and minor elements achievable with the instrument allows for precise determination of multiple elements at both low and high concentrations without further sample pre-concentration or dilution.
- Excellent accuracy was demonstrated using a certified reference material. Results of a QC (CCV) measurement performed during the analysis demonstrated the potential of the method for delivering high quality data, even for long sequences, without the need to perform maintenance.
- Robust and stable analytical performance was demonstrated over 6 hours of continuous acquisition of 120 samples, including 90 real samples, containing an average of 0.2% TDS and variable acid composition.
- The flexibility of the Qtegra ISDS Software allows customization of the analytical method by enabling selection of both plasma viewing modes (Axial and Radial) in a sample analysis, which provides a flexible range of development for analytical research.

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View the full application note

Cathode Active Materials

A cathode usually consists of a lithium transition metal oxide, such as lithium-cobalt oxide (LiCoO2, LCO) or lithium-manganese oxide (LiMnO2, LMO), which is applied as a coating to a metal foil made of copper or aluminum. The most popular cathode materials for EV batteries are nickel-cobalt-aluminum oxide (Li(NiCoAl)O2, NCA), nickel-manganese-cobalt oxide (Li(NiMnCo)O2, NMC), and lithium-iron-phosphate (LiFePO4, LFP). The development of suitable cathode materials is one of the challenges in improving the performance of LiBs to meet the increasingly demanding requirements for energy storage. Lithium ions must be able to enter and exit the cathode materials quickly for high currents and repeatedly for recharging.

Multiple challenges are associated with the cathode-active materials in LiB manufacturing. One of them is to test both trace metals and major element impurities, including magnetic particles, in the range of ppm to sub ppb in the cathode-active materials of LiB. Another challenge is to determine the exact mixture of nickel, manganese, and cobalt (NMC) in LiB.

Our analytical testing solutions and methods help identify and measure trace metals and major element contaminants in cathode active materials with high precision and accuracy while enabling operational improvements. Our diverse range of solutions allows you to analyze various materials relevant to cathode manufacturing and easily expand your analytical capabilities in this area.

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- Managing the challenges of analyzing battery materials using triple quadrupole inductively coupled plasma mass spectrometry (ICP-MS) equipped with Argon Gas Dilution
- Determination of elemental impurities in lithium iron phosphate using ICP-OES
- Sensitive determination of elements in lithium batteries using the Thermo Scientific iCAP PRO XP ICP-OES

APPLICATION NOTE | 002086

Managing the challenges of analyzing battery materials using triple quadrupole inductively coupled plasma mass spectrometry (ICP-MS) equipped with Argon Gas Dilution

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Goal

To demonstrate the analysis of critical elemental impurities in highly concentrated solutions (up to 5.3% w/w) of cathode materials used in lithium-ion batteries with high sensitivity, accuracy, and robustness using triple quadrupole ICP-MS.

Introduction

The transition to electric vehicles (EVs) is one of the key developments of the green energy revolution, and the resulting demand for these vehicles shows an unprecedented speed in ramping up manufacturing capacity for both the vehicles themselves and the lithium-ion batteries that power them. Lithium-ion batteries are one of the most important power storage materials for EVs due to their power density and life cycle performance. With the high, and increasing, demand for batteries, questions arise around their fate at the end of their lifetime. Key areas of concern are recycling, to recover valuable raw materials and maintain sustainability in the supply chain, and issues surrounding environmental contamination following inappropriate battery disposal.

A typical lithium-ion battery consists of four main parts, namely the cathode, separator, anode, and electrolyte (Figure 1). To meet the required performance criteria (i.e., long battery lifetime and maximum achievable charge capacity), it is important to monitor not only the concentration ratio of the main components (typically nickel, manganese, cobalt, and lithium in nickel manganese cobalt (NMC) batteries, or iron, phosphorus, and lithium in lithium iron phosphate ($LiFePO_4$) batteries and lithium ferrophosphate (LFP) batteries), but also trace impurities in both the precursor materials and the finished products.[1]

In general, inductively coupled plasma mass spectrometry (ICP-MS) is the preferred analytical technique for the analysis of metals and related contaminants at low levels (below 1 µg·L^{-1}). However, the analysis of samples containing higher levels of total dissolved solids (TDS, typically above 0.5% w/v) is a known challenge in ICP-MS. The complexity

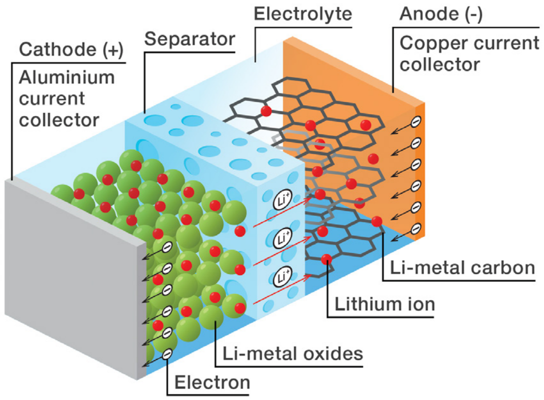

Figure 1. Lithium-ion battery diagram

of the sample matrix can significantly affect the sensitivity of the instrument, cause intensity fluctuation of the internal standard (suppression and drift), and lead to increased system maintenance with unwanted downtime due to clogging of the interface cone orifices, torch injector, or nebulizer. To analyze complex samples, dilution must be performed, either using liquid dilution or, alternatively, dilution of the sample aerosol with an inert gas such as argon. Dilution with argon gas is a particularly attractive option, as samples can be placed directly on the autosampler, without the need of a manual, time-consuming off-line manual dilution step prior to analysis.

A further challenge for the analysis of battery materials such as cathodes, when using conventional, single quadrupole ICP-MS, are polyatomic interferences generated by the principal components of the samples. For example, in addition to the wellknown argon-based interferences, all major selenium isotopes can be affected by nickel oxide-based interferences, such as $[^{60}Ni^{18}O]^+$ (on ^{78}Se) and $[^{64}Ni^{16}O]^+$ (on ^{80}Se), rendering low level analysis of this critical contaminant with a single quadrupole ICP-MS system impossible.[2]

Lithium-ion battery charge

This application note describes an optimized analytical method for the analysis of lithium battery cathode materials, as well as recycled battery materials. In this work, samples could be analyzed immediately following acid assisted dissolution, even though they contained elevated amounts of total dissolved solids (TDS), in this case up to 5.3% w/w. To overcome the analytical challenges described above, a triple quadrupole instrument, the Thermo Scientific™ iCAP™ TQe ICP-MS, was applied for the analysis. Use of triple quadrupole technology offered superior interference removal and operation using Argon Gas Dilution (AGD) facilitated direct analysis of concentrated sample solutions.

Conclusions

The iCAP TQe ICP-MS, operated using AGD, enables analysts to perform accurate and reliable elemental analysis in challenging samples such as battery cathode solutions. This was demonstrated by the analysis of 51 elements in a large number of lithium battery samples with high concentrations. The analytical method was rigorously tested for performance, and the results obtained clearly demonstrated the following analytical advantages for battery cathode sample analysis:

- AGD mode allowed the aspiration of up to 5.3% (m/v) lithium battery cathode solutions and allowed excellent MDLs to be obtained, eliminating the need for labor-intensive manual sample dilution and accelerating sample throughput.
- High sensitivity TQ-O_2 mode provided the advanced performance required for the accurate determination of As, Se, interference free in complex, lithium battery matrix samples showing outstanding analysis performance.
- Excellent CCV recovery and spike recovery results, as well as stable and consistent internal standards response, were obtained across a batch containing 202 samples of 1% (m/v) and 5.3% (m/v) lithium battery cathode solutions, demonstrating the reliability of the method.

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APPLICATION NOTE | 000245

Determination of elemental impurities in lithium iron phosphate using ICP-OES

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Goal

This application note describes the analysis of lithium iron phosphate using the Thermo Scientific™ iCAP™ PRO Series ICP-OES. The note describes the method development as well as presenting key figures of merit, such as detection limits and stability.

Introduction

Lithium iron phosphate has properties that make it an ideal cathode material for lithium-ion batteries. The material is characterized by a large discharge capacity, low toxicity, and low cost. The first large capacity lithium iron phosphate battery was produced in China in 2005, and the life cycle performance characteristics of the battery were unmatched by other batteries of a similar classification. An ideal application for batteries with a lithium iron phosphate cathode is in series in electric vehicles where frequent charging and discharging of the batteries takes place.

The purity of the cathode material is critical, and changes in the raw material processing and synthesis can cause the introduction of impurities in the final cathode material. These impurities impact the lifetime and energy storage capacity of the battery, and in extreme cases may affect the integrity of the crystal structure of the battery, causing safety issues. Therefore, accurate analysis of the cathode material is key to the performance and safety of lithium batteries made using these materials.

Conclusion

This application note demonstrates the effective application of the iCAP PRO Radial ICP-OES instrument for analysis of elemental impurities in lithium iron phosphate, a commonly used cathode material in lithium-ion batteries. A total of 23 key impurity elements were accurately and sensitively measured, as demonstrated by the results obtained for the customer supplied sample and the quantitative spike recoveries obtained for each element spiked into the sample. The instrument not only has the capability to measure elemental impurities at low concentrations, but also has the linear range to determine matrix elements accurately over a typical analysis sequence, as demonstrated by the high precision results achieved for the matrix element stability test. Use of the robust, fully demountable EMT torch design for this application ensures low operating consumable costs and this, coupled with excellent stability and sensitivity, makes the iCAP PRO ICP-OES an ideal choice for accurately measuring a range of matrix and impurity elements in materials for the lithium battery industry.

View the full application note

APPLICATION NOTE | 73872

Sensitive determination of elements in lithium batteries using the Thermo Scientific iCAP PRO XP ICP-OES

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Goal

This note demonstrates a fast analytical method for the determination of major and trace elements in the ternary cathode material of lithium batteries using the Thermo Scientific™ iCAP™ PRO Series ICP-OES.

Introduction

The continuous development of lithium-ion batteries and the research into their materials is at the forefront of the energy sector as it moves away from fossil fuels. To regulate the quality of production, the Chinese national standard method YS/T 798-2012 was established. All new lithium battery developments must meet the requirements of these standards. The ternary material of lithium batteries typically contains lithium, nickel, cobalt, and manganese, and potassium aluminate as its cathode material. In recent years, lithium batteries using ternary materials as cathode materials have gradually replaced nickel-metal hydride batteries, lithium cobalt batteries and lithium-ion phosphate batteries. This is due to the high capacity, good cycle stability (battery life), and moderate cost of the new battery type. The proportion and content of the main elements in the ternary cathode material can affect the performance and cost of the lithium battery significantly and the content of impurities in the ternary material alters the safety of the battery. Therefore, the accurate determination and quantification of the main elements, as well as trace impurities in the ternary cathode material, becomes particularly important.

Conclusion

In this application note, a Thermo Scientific iCAP PRO XP ICP-OES Radial system was used to establish a rapid detection method for the determination of major elements and trace impurities in a ternary cathode material used in lithium batteries.

For the impurity elements, recovery values of between 90% and 110% were achieved, as calculated from the spiked samples. For interferences from complex matrices, such as cobalt-nickel-manganese-lithium matrix and the spectral interference of elements, such as nickel and manganese, the iCAP PRO XP ICP-OES system uses a high-resolution optical system that can obtain accurate test results for each element.

All performance specifications meet the testing requirements specified by the national standard method YS/T 798-2012.[1] The iCAP PRO XP ICP-OES system has an ultra-high sensitivity and stability for the detection of elements with characteristic wavelengths in the far ultraviolet region (e.g., S 180.731 nm, Pb 182.205 nm). The iCAP-PRO XP ICP-OES system performance for major elements, such as nickel, cobalt, manganese, and lithium, demonstrated excellent levels of precision (with % RSD <1%) and accuracy (spike recoveries with the range 90% to 110%).

Overall, the analytical solution employed in this study with the iCAP PRO XP ICP-OES system meets all analytical requirements for routine or research laboratories that aim to analyze elements in ternary materials of lithium batteries.

Reference

1. 1. YS/T 798-2012: China National non-ferrous metal industry standards, Lithium nickel cobalt manganese oxide, Ministry of Industry and Information Technology of the People's Republic of China.

View the full application note

Electrolyte Analysis

Alongside the cathode and the anode, electrolytes are one of the key components of the battery. They play an active role in transporting the positive lithium ions between the cathode and anode and consequently in the charging and discharging performance of the battery. It is therefore crucial to check them for possible contaminants. At the same time, the electrolyte serves as a sample type that enables the examination of aging processes, as degradation products from all battery components can gradually form in it.

The LiB electrolyte solution consists of a lithium salt dissolved in an organic solvent. The most common electrolytes are lithium hexafluorophosphate ($LiPF_6$), lithium perchlorate ($LiClO_4$), lithium tetrafluoroborate ($LiBF_4$), lithium hexafluoroarsenate ($LiAsF_6$), lithium hexafluorosilicate ($LiSiF_6$), and lithium tetraphenylborate ($LiB(C_6H_5)_4$). Many organic solvents are used for the electrolyte solution, including ethylene carbonate, diethyl carbonate, dimethyl carbonate, ethyl methyl carbonate, propylene carbonate, methyl formate, methyl acrylate, methyl butyrate, and ethyl acetate. Lithium batteries typically contain a combination of lithium salts and organic solvents as electrolytes.

Assessing the quality of electrolytes for battery manufacturing is a major challenge due to the large number of chemical compounds involved, including salts, ionic species, organic solvents, and metals. To overcome these challenges, we offer comprehensive instrumentation and methods for the detection, identification and analysis of impurities in electrolytes. This includes the detection and analysis of anions of lithium salts, the determination of the qualitative and quantitative composition of organic solvents and their aging by-products as well as the measurement of impurities caused by trace elements. Our expertise ensures that you can effectively manage electrolyte quality and maintain high standards in battery manufacturing.

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APPLICATION NOTE | 70399

Determination of tetrafluoroborate, perchlorate, and hexafluorophosphate in a simulated electrolyte sample from lithium ion battery production

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Introduction

Lithium ion batteries are commonly used in portable consumer electronic devices. The electrolyte solution in these batteries consists of a lithium salt in an organic solvent. Commonly used salts are lithium hexafluorophosphate ($LiPF_6$), lithium perchlorate ($LiClO_4$), lithium tetrafluoroborate ($LiBF_4$), lithium hexafluoroarsenate ($LiAsF_6$), lithium hexafluorosilicate ($LiSiF_6$), and lithium tetraphenylborate ($LiB(C_6H_5)_4$). Some organic solvents used in the electrolyte solution are ethylene carbonate, diethyl carbonate, dimethyl carbonate, ethyl methyl carbonate, propylene carbonate, methyl formate, methyl acrylate, methyl butylate, and ethyl acetate. The electrolyte in lithium batteries may have a mixture of these lithium salts and organic solvents. The electrolyte's concentration in the solvent ranges from 0.1 to 2 mol/L, with an optimal range of 0.8–1.2 mol/L. The anions of the added lithium salts can be determined by ion chromatography (IC) to ensure that the solutions have been prepared at the proper concentrations.

Here, the authors prepare simulated samples that contain either lithium tetrafluoroborate, lithium perchlorate, or lithium hexafluorophosphate in an equal mixture of ethylene carbonate, diethyl carbonate, and propylene carbonate, and demonstrate that the anionic content can be determined accurately using a Reagent-Free™ IC (RFIC™) system. The RFIC system allows the analyst to avoid the problems encountered in eluent preparation. The RFIC system also delivers excellent retention time reproducibility for easy and reproducible quantification in the analysis of lithium ion battery electrolyte solutions.

Conclusion

This application demonstrates an IC method that uses an RFIC system to easily assay the anions in the simulated lithium ion battery electrolyte samples. The results show that this method is both accurate and reproducible.

View the full application note

APPLICATION NOTE | 1053

Determination of dissolved manganese in lithium/manganese oxide battery electrolyte

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Goal

To develop an IC method that accurately determines dissolved manganese in the electrolyte of a Li/LiMn$_2$O$_4$ battery using an RFIC system.

Introduction

Lithium-ion batteries are widely used in products such as portable consumer electronic devices and electric vehicles. Many different materials are used to make the cathode in lithium batteries, including those that are manganese-, cobalt-, and nickel-based. Lithium-ion batteries that are manganese-based are more environmentally friendly, have a good safety record, and can be made at a lower cost; however, they have a shorter lifetime than other types of lithium-ion batteries. One of the reported causes of lifetime loss is the dissolution of manganese from the cathode into the electrolyte during cycling (i.e., charging/discharging). Lithium/lithium manganese oxide (Li/LiMn$_2$O$_4$) is a type of battery that has a manganese-based cathode.

In Thermo Scientific Application Note (AN) 258, ion chromatography (IC) was applied to determine the anions tetrafluoroborate, perchlorate, and hexafluorophosphate in a simulated electrolyte solution for lithium-ion batteries.[1] AN 258 used a Reagent-Free™ IC (RFIC™) system, and it is also possible to use an RFIC system to determine manganese in a simulated electrolyte solution for lithium-ion batteries. There is one report of an IC method that uses manually prepared eluents and direct conductivity detection to determine dissolved manganese in the electrolyte of a Li/LiMn$_2$O$_4$ battery.[2] However, that method has poor sensitivity, which is inherent with direct conductivity detection. Even with a three-component (tartaric acid, dipicolinic acid, and ascorbic acid) mobile phase, the manganese peak exhibits extreme tailing on the chosen column. A better IC cation column will improve peak shape and therefore improve integration precision and method accuracy, while suppressed conductivity detection will improve method sensitivity.

The work shown here uses an RFIC system with suppressed conductivity detection to quantify dissolved manganese in the simulated electrolyte of a Li/LiMn$_2$O$_4$ battery. The method uses the Thermo Scientific™ Dionex™ IonPac™ CS12A column set, which was designed to deliver good peak shapes for cations such as manganese(II) (Mn^{2+}), with a simple methanesulfonic acid (MSA) eluent produced by an eluent generator. The combination of the RFIC system and a quality IC column yields a method that is sensitive, accurate, reproducible, and easy to execute, requiring only the addition of deionized water to the RFIC system.

Conclusion

This study demonstrates an accurate and reproducible IC method that uses suppressed conductivity detection to determine manganese in the simulated electrolyte of a Li/LiMn$_2$O$_4$ battery. The method uses an RFIC system and requires only 15 min per analysis with a simple isocratic separation using an MSA eluent. The eluent is produced by an eluent generator to preclude the labor and potential error associated with eluent preparation.

References

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APPLICATION NOTE | 002026

Determination of tetrafluoroborate, perchlorate, and hexafluorophosphate in an electrolyte sample for lithium-ion battery production

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Goal

To update the application that determined tetrafluoroborate, perchlorate, and hexafluorophosphate in an electrolyte sample that was designed to simulate a lithium-ion battery production sample with new equipment and a current generation electrolytic suppressor.

Introduction

Rechargeable batteries are an increasing part of our daily life as we use more portable electronic devices, including mobile phones. These batteries are also important for the electric car industry. Lithium-ion batteries are the most commonly used rechargeable batteries because of their high volumetric energy density.[1] The electrolyte in these batteries are lithium salts in non-aqueous solutions. Commonly used lithium salts are lithium hexafluorophosphate ($LiPF_6$), lithium perchlorate ($LiClO_4$), lithium tetrafluoroborate ($LiBF_4$), lithium hexafluoroarsenate ($LiAsF_6$), lithium hexafluorosilicate ($LiSiF_6$), and lithium tetraphenylborate ($LiB(C_6H_5)_4$). Commonly used organic solvents are ethylene carbonate, diethyl carbonate, dimethyl carbonate, ethyl methyl carbonate, propylene carbonate, methyl formate, methyl acrylate, methyl butylate, and ethyl acetate.[1] The electrolyte in lithium batteries may have a mixture of these lithium salts and organic solvents. The electrolyte's concentration in the solvent ranges from 0.1 to 2 M, with an optimal range of 0.8 to 1.2 M. The anions of the added lithium salts can be determined by ion chromatography (IC) to ensure that the solutions have been prepared at the proper concentrations. Thermo Scientific™ Application Note 258 (AN258)[2] demonstrated that the anionic content can be determined accurately using a Thermo Scientific™ Reagent-Free™ Ion Chromatography (RFIC™) System with 4 mm versions of Thermo Scientific™ Dionex™ IonPac™ AS20 Columns. Here we demonstrate the same application using updated equipment and suppressor with 2 mm versions of Dionex IonPac AS20 columns and an improved method for the determination of the lithium salt anions in simulated battery electrolyte solutions.

Conclusion

This application demonstrated an improved method for the determination of tetrafluoroborate, perchlorate, and hexafluorophosphate in simulated lithium-ion battery production samples using a RFIC system with suppressed conductivity detection, and 2 mm versions of Dionex IonPac AS20 columns. This method significantly reduces eluent consumption and thus prolongs the life of the EGC as flow rate is reduced from 1.2 to 0.3 mL/min. The method has a linear calibration over the concentration range of 1 to 40 mg/L and was shown to be precise and accurate.

References

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APPLICATION NOTE | 001124

Comprehensive analysis of electrolyte solutions for lithium-ion batteries using gas chromatography-mass spectrometry

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Goal

The goal of this application note is to demonstrate the Thermo Scientific™ ISQ™ 7610 Single Quadrupole GC-MS capabilities for accurate quantitation and characterization of lithium battery electrolyte components.

Introduction

The development of the lithium-ion battery (LIB) over the past three decades has led to groundbreaking advancements in energy storage. This comes at an opportune time as the world's current and ever-growing energy demands have reached a critical point. Traditional petroleum-based energy sources (i.e., oil, natural gas) are dwindling, while strict mandates have been initiated by governments to reduce petroleum-based carbon emissions under the Paris Agreement by 2030. In addition, recent geopolitical factors have made access to traditional energy sources in Europe highly volatile and unstable, further driving the need for new sources of energy. While renewable energy sources (i.e., solar, hydro, wind) could be a viable alternative, storage of energy is a limiting factor for larger scale use.

The biggest impact of LIBs can be seen in the transport sector with their implementation in electric vehicles. However, the longevity of LIB operation is a crucial factor to become a suitable energy replacement to petroleum in motor vehicles. The durability of LIBs is attributed to the thermodynamics of the battery electrolyte. The most common electrolyte used in LIBs is lithium hexfluorophosphate ($LiPF_6$) salt mixed with organic carbonate solvents (i.e., dimethyl carbonate, ethyl methyl carbonate, diethyl carbonate). Upon the first cycling of the LIB, reduction of the electrolyte at the anode surface produces a conductive film known as the solid electrolyte interface (SEI), where current (i.e., Li^+) can pass through while simultaneously preventing further reduction of the electrolyte. However, the LiPF6 is thermodynamically unstable at elevated operating temperatures (>60 °C) causing the organic carbonate solvents to be further reduced. As a result, a thicker and more resistive SEI is formed, limiting current flow and LIB charging capacity. In addition, thermal dissociation of the $LiPF_6$ to PF_5 causes deterioration of the SEI through side reactions, resulting in continued electrolyte degradation as it comes in contact again with the anode surface.[1]

The addition of electrolyte additives can help reduce electrolyte degradation in LIB batteries. These chemicals are preferentially reduced at higher potentials, helping prevent further electrolyte degradation under such operating conditions. Differences in additive functional groups will alter the properties of the SEI. Thus, knowledge of electrolyte composition and byproducts formed during battery operation is key for future development of more efficient, stable, and high performing LIBs.

In this application note, an analytical method for the compositional analysis of LIB electrolytes is demonstrated. Extraction and analysis of LIB electrolytes for 16 different electrolyte solvent, additive, and degradation compounds were performed using the Thermo Scientific™ TRACE™ 1610 gas chromatograph coupled to the Thermo Scientific™ ISQ™ 7610 single quadrupole GC-MS system. High linearity was observed over an extended calibration range with trace level detection possible at μg·L^{-1} levels. Quantification, confirmation, and identification were achieved using full scan together with selected ion monitoring (SIM) acquisition for accurate determination of LIB electrolyte composition.

Conclusion

This application demonstrates the ISQ 7610 MS equipped with the new XLXR detector provides a high linear dynamic range for quantification of LIB electrolyte components over varying concentration ranges.

- Efficient separation of various electrolyte components was achieved within 16 min.
- Full scan analysis provided characterization of unknown additives present in electrolyte samples while t-SIM acquisition provided confirmation of electrolyte additives not achievable with full scan acquisition.
- Linear dynamic range of over 4 orders of magnitude was achieved using the XLXR detector.
- Detection of LIB electrolyte components were achievable at μg·L^{-1} levels with IDLs ranging from 0.003 to 0.021 μg·L^{-1}.

References

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APPLICATION NOTE | 000394

Analysis of electrolyte components of lithium-ion batteries using gas chromatography-mass spectrometry

Thermo Fisher Scientific Inc.

Goal

At present, methods and literature references for determining electrolyte composition are limited. The purpose of this work is to establish a simple and efficient gas chromatography–mass spectrometry detection method for a sensitive and fast determination of lithium-ion battery electrolyte composition and content.

Introduction

The electrolyte in a lithium-ion battery is the carrier of positive lithium ions between the cathode and anode. It is generally made of lithium salts dissolved in organic solvents. Electrolyte solutions must enable the Li-ions to transport freely, requiring both high dielectric constant and low viscosity. For this reason, suitable electrolyte solutions are a mixture of cyclic and linear carbonate esters, in which the exact composition plays a key role in the performance of lithium-ion batteries.

Gas chromatography–mass spectrometry can be used to provide the qualitative and quantitative composition of the Li-battery electrolyte, with a direct injection of diluted samples. In this study, a simple GC-MS method has been developed to detect ester compounds in a concentration range of 3.0 µg/L to 30.0 µg/L.

Conclusion

The GC-MS method is a simple, reliable, and accurate approach for lithium-ion battery electrolyte composition analysis and quality control requirements. The samples can be simply diluted with a suitable solvent and directly injected into the GC-MS system.

The results show very good recovery for the target ester compounds of 92.4.3–105.3%, demonstrating the high accuracy of the method. The excellent precision of the method was also demonstrated, with an RSD value ≤4.16% over six repeated injections.

Full scan acquisition can be applied with good sensitivity. SIM acquisition can be utilized to lower the limits of detection for the method.

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APPLICATION NOTE | 002666

Determination of impurities in lithium salts used in battery manufacturing by ion chromatography and ICP-MS

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Goal

Quantification of ionic impurities present in lithium-based raw materials used in batteries by Thermo Scientific™ ion chromatography and ICP-MS systems.

Introduction

Lithium-ion batteries (LIBs) have revolutionized the modern electronic industry by providing high energy density, longer lifespan, and a more environmentally friendly alternative to traditional lead-acid and nickel-cadmium batteries. These rechargeable batteries are widely used in portable electronic devices, electric vehicles, and energy storage systems.[1]

The key advantages of LIBs over other battery chemistries are their high energy density, which allows them to store more energy in a smaller size and weight, and their low self-discharge rate, which means that they can hold their charge for a longer time when not in use. Additionally, LIBs have a longer lifespan compared to other rechargeable batteries, which makes them more cost-effective.[1,2]

However, despite their numerous advantages, there are still challenges associated with the production and use of LIBs. These challenges include the high cost of raw materials, concerns regarding their safety and stability, and the environmental impact of battery disposal. To overcome these challenges, there is ongoing research to improve the performance and sustainability of LIBs.

Lithium-ion batteries are now ubiquitous in modern-day technology, powering everything from smartphones and laptops to electric vehicles and grid-scale energy storage. Central to the performance of LIBs are the salts that make up their electrolytes which facilitate the movement of lithium ions between the battery's electrodes. As such, the choice of electrolyte salt plays a critical role in determining the battery's overall performance, including its energy density, power output, safety, and lifespan. For instance, impurities can interfere with the electrochemical process, leading to reduced battery capacity, and certain elements can react with battery components causing instability or potential hazards, such as overheating or even battery failure. In recent years, there has

been considerable research aimed at developing new electrolyte salts and improving existing ones to enhance the performance of LIBs and enable their broader adoption.[6]

In this Application Note, various electrolyte salts (raw materials) such as lithium carbonate, lithium fluoride, lithium hydroxide, and phosphorus pentachloride were tested for their ionic and elemental impurities content using ion chromatography (IC) and ICP-MS separately. The percentage assay of these salts was also checked using an IC system.

Conclusion

This study demonstrated the accurate quantification of anionic impurities and cations in lithium salts by IC. This method uses a Dionex IonPac AS11-HC column with a hydroxide eluent, which is ideal for separating a wide range of inorganic anions with high sensitivity. The high capacity Dionex IonPac CS16 column is ideal for the analysis of a wide range of cations with high sensitivity and resolution. Eluent generation technology is used for both anionic and cationic methods and provides uniform, highly stable eluent concentrations, which helps to achieve highly reproducible results.

One of the main advantages of using ICP-MS for elemental impurities determinations is its ability to detect low concentration levels (sub-ppb) routinely and with ease. The fast scanning speed, robustness, and selectivity of ICP-MS allow for rapid, accurate, and efficient analysis of elemental content in salts.

Since regulations are becoming tougher, fast and accurate techniques are the solution for the present and future. In this regard, IC and ICP-MS have proven to be the best solutions in terms of productivity and performance for the analysis of ions and elements in lithium-ion battery electrolyte salts.

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APPLICATION NOTE | 000243

Robust and sensitive measurement of trace element impurities in $LiPF_6$ electrolyte solutions using ICP-OES

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Goal

To demonstrate the capabilities of the Thermo Scientific™ iCAP™ PRO XP ICP-OES Duo for sensitive, robust, fast, and straightforward analysis of trace elements in lithium hexafluorophosphate electrolyte samples.

Introduction

Lithium ion (Li-ion) batteries are increasingly being used in electronic devices and electric vehicles (EV). The demand for electric vehicles is expected to grow significantly in the next years, as they can be a viable alternative to fossil fuel driven vehicles and help to rapidly decrease CO_2 emission from traffic. These developments have led to a growing industry demand for Li-ion batteries globally that is complemented by significant efforts in research and development, dedicated to providing efficient and cost-effective solutions[1]. For production, especially in the ramp-up phase, regular and rigorous quality control of all components, including the lithium salt, anode and cathode material, and electrolyte, of an Li-ion battery is crucial. The electrolyte plays an important role in the charging and discharging performance of the battery, and hence needs to be checked for potential impurities. At the same time, the electrolyte is also a sample type that allows the investigation of ageing processes, as degradation products from all components of the battery can accumulate within it over time. Finally, once the battery is at the end of its life, all components must be thoroughly screened to ensure that potential environmental contamination and injury risks to personnel disassembling the batteries are minimized.

One of the most common electrolytes in Li-ion batteries is lithium hexafluorophosphate ($LiPF_6$) dissolved in a binary or ternary mixture of ethylene carbonate (EC) and linear carbonates, such as diethyl carbonate (DEC) and ethyl-methyl carbonate (EMC). It is a popular electrolyte material in the industry due to its high energy density and power properties.[2,3] At the moment, there is only one standard method available for the analysis of electrolytes for lithium-ion batteries, based on the Chinese Standard HGT/ 4067-2015,[4] which requires method detection limits of 1 $mg·L^{-1}$ in the final $LiPF_6$ electrolyte samples. The Thermo Scientific™ iCAP™ PRO ICP-OES Series can be the instrument of choice for this analysis, delivering detection limit performance well within the required range and providing a robust setup that can accurately characterize $LiPF_6$ electrolyte sample materials.

Conclusions

This study has demonstrated the performance of the iCAP PRO XP ICP-OES Duo system for highly sensitive and accurate analysis of impurities in electrolyte solutions containing LiPF6 and organic carbonates, such as ethyl carbonate and ethyl methyl carbonate. The proposed method allowed for fast sample turnaround times combined with high reproducibility and robustness, allowing reliable analysis of long sequences over multiple days of analysis.

A summary of the main results and conclusions is presented below:

- Excellent sensitivity, sufficient for detecting impurities in $LiPF_6$ electrolyte samples (in the low $\mu g \cdot L^{-1}$ (ppb) range), was achieved for all the target elements using the Axial iFR mode of the instrument. A large linear dynamic calibration range of up to 1,000 $\mu g \cdot L^{-1}$ was also obtained. The linear dynamic range could be further expanded, if required, by adding standard solutions with higher concentrations. This would be beneficial when analyzing, for example, used electrolyte samples for battery ageing studies or failure diagnostic purposes.
- The accuracy of the method was verified by successfully recovering known spiked concentrations in fresh electrolyte samples in spite of the challenges arising from the sample matrix composition.
- Excellent system robustness and reproducibility was demonstrated over multiple days, proving the reliability of the method, with accuracy over long measurement runs validated by analysis of QC solutions, spiked samples, and internal standards.
- Daily continuous measurements of more than 6 hours are possible with the described method. Using a short exposure time of 10 s ensured not only fast analysis (3 min 6 s/sample), but also high sensitivity. A total of more than 100 samples could be measured daily following this approach. This high sample turnaround with consumption of low sample volumes, minimal downtime, and no need for extensive sample preparation or user interaction positions the iCAP PRO XP ICP-OES Duo system as an effective choice of instrument for quality control and characterization of trace elements in fresh electrolyte samples.

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Anode Analysis

Graphite powder is mainly used as an anode material in LiBs due to its numerous advantageous properties. Its ease of molding, chemical stability, and non-metallic nature make it particularly suitable for this application. In addition, graphite powder has excellent electrical conductivity and robust resistance to high temperatures, oxidation and corrosion. One of the most important properties of graphite powder is its considerable lithium ion diffusion coefficient and lithium storage capacity. In particular, the volume of graphite remains unchanged when the lithium ions are inserted, which is critical for maintaining structural integrity and consistent performance during battery operation. In addition, advanced oxidation and pyrolysis processes can be used to create a core-shell configuration in the graphite powder. This modification significantly improves charge and discharge capability and extends the life of the anode.

The service life of a battery is crucial to its functionality, as LiBs are generally designed for up to 1000 charge/discharge cycles. However, the anode degrades over time, reducing charge capacity and discharge efficiency. Therefore, monitoring and understanding the chemical and physical changes that occur in the battery system is of utmost importance.

We offer comprehensive solutions to assess and understand anode degradation and the presence of metallic impurities in the anode. Our advanced instruments such as GC, liquid chromatography (LC), ICP-MS and IC-HRAM MS can identify the degradation products that build up over time and reduce battery performance. In addition, our ICP-OES and ICP-MS technologies can determine metallic impurities in the anode powder, ensuring thorough analysis and improved battery maintenance.

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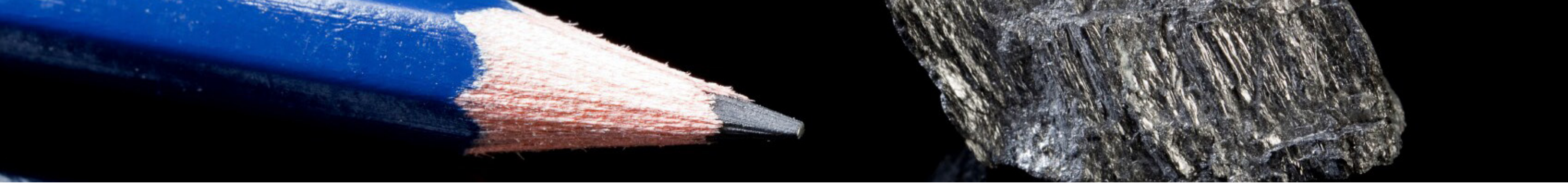
- Determination of elemental impurities in graphite powder for lithium-ion battery anodes



APPLICATION NOTE | 000466

Determination of elemental impurities in graphite powder for lithium-ion battery anodes

Thermo Fisher Scientific Inc.

Goal

To develop a robust and reliable method for the determination of elemental impurities in graphite powder samples for lithium-ion battery anodes, using the Thermo Scientific™ iCAP™ PRO X ICP-OES Duo instrument. This note will demonstrate the performance of the method with respect to calibration linearity for 13 trace element impurities (target $R^2 > 0.999$) and quantification of these elements. It will also illustrate the robustness of the method developed through spike recovery analysis of the microwave digested samples.

Introduction

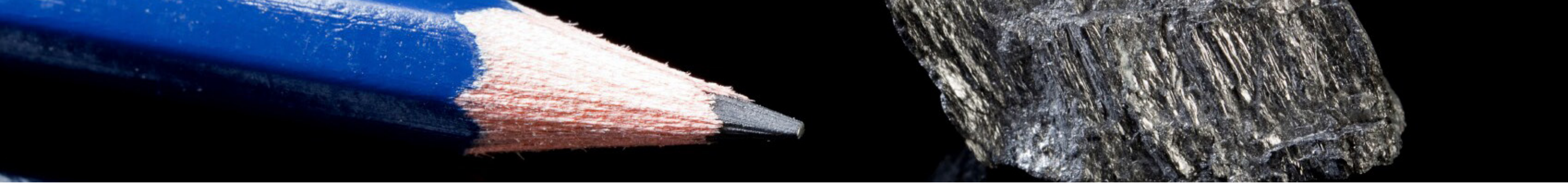
For lithium-ion batteries, the negative electrode (anode) material is generally made from graphite powder. Graphite powder is suitable for this application primarily because it is an easily molded, chemically stable, and non-metallic material with good electrical conductivity and high temperature, oxidation, and corrosion resistance. It also has a large lithium-ion diffusion coefficient with a high lithium insertion capacity and does not change volume with insertion of lithium ions. In addition, graphite powder can be modified through various oxidation and pyrolysis processes to generate a core-shell structure that can improve its charging/discharging performance and increase the anode lifetime.

Graphite powder has become the main lithium-ion battery anode material in use today in smaller consumer goods, such as mobile phones, as well as in electrical vehicles.

Through optimization of the sample pre-treatment process and plasma parameters, together with rigorous selection of the most suitable analyte emission lines, a robust, accurate and reliable ICP-OES analytical method for the determination of 13 trace elements in graphite powder materials for lithium ion batteries was developed.

Conclusion

This application note has demonstrated the performance of the iCAP PRO X ICP-OES Duo instrument for quantitative trace element impurity analysis in graphite powder samples (derived from briquetting coal) used for lithium-ion battery anode production. Using the ASTM D6357-2004 and GB/T24533-2009 (Appendix H) standards as a guide, a robust sample preparation method based on microwave digestion of the samples was developed. Concentrations were determined for all 13 trace elements in the three raw material briquetting charcoal samples, and the robustness of the analysis was confirmed by the achievement of quantitative spike recoveries of the measured elements (in the range 93 to 104%).



Determination of elemental impurities in graphite powder for lithium-ion battery anodes *(continued)*

The iCAP PRO X ICP-OES Duo provides the advantages of high sensitivity, good stability, fast analysis speed and low operating costs required for all aspects of elemental analysis in the lithium ion battery workflow. From quantifying lithium concentrations in ores and brine to routinely confirming the composition of cathode active materials in a QA/QC environment, the iCAP PRO X ICP-OES Duo offers the performance and flexibility required to meet the analytical demands of this rapidly growing sector.

➡ View the full application note

Manufacturing

A lithium-ion battery consists of electrodes (anode and cathode), an electrolyte, and a separator. The cathode consists primarily of active materials, usually binary or ternary alloys, including nickel, cobalt, manganese and a conductive framework. During manufacture, N-methyl-2-pyrrolidone (NMP) is used to dissolve the cathode binder, polyvinylidene fluoride (PVDF), so that it can be applied evenly to both sides of the collector. PVDF, a type of fluoropolymer, is known for its exceptional thermal stability and excellent chemical resistance, making it well suited to the specific chemical conditions in Li-ion cells. It is therefore primarily used as an adhesive and insulating layer in lithium batteries.

Improving battery performance requires the development of novel battery components and a thorough understanding of the factors that lead to performance degradation during repeated charge and discharge cycles. Ensuring quality at every stage, from raw materials to cell assembly, is critical to efficient production and minimizing waste. Even when developing new battery materials, all factors that could affect battery performance must be carefully considered throughout the production process.

One of the key challenges in battery manufacturing is to ensure that every component in LiBs is free from contamination and that the highest quality raw materials are used. A comprehensive range of analytical solutions is required to ensure the consistency and integrity of these materials. Manufacturers must use these techniques to perform quality control checks and ensure that there are no variations in chemical composition or impurities in electrode materials and electrolytes, as these can significantly affect battery performance.

Our solutions comprehensively cover all your requirements for the analysis of raw materials. We confirm the purity of both raw materials and processed raw materials and manage the composition of electrode and electrolyte materials. Our range of analytical instruments includes ICP-OES, ICP-MS, XRF, GC-MS, reagent-free ion chromatography (RFIC) and IC-HRAM MS. These tools help effectively address the challenges in battery manufacturing.

In addition, by using various advanced analytical techniques, researchers can obtain important structural and chemical information that is necessary for the improvement of batteries. These include micro-computed tomography (microCT), scanning and transmission electron microscopy (SEM and TEM), DualBeam (focused ion beam SEM; FIB-SEM), Raman spectroscopy, X-ray photoelectron spectroscopy (XPS) and advanced 3D visualization and analysis software. By integrating information from different modes and scales, researchers can gain comprehensive insights into the essential properties of batteries that change over time. This multi-layered approach can lead to significant advances in battery development and performance.

Click on note titles to jump to page

- Determination of inorganic anions in saturated lithium carbonate solution
- Determination of chloride and sulfate in saturated lithium hydroxide solution
- Rapid determination of residual *N*-methyl-2-pyrrolidone (NMP) in lithium battery electrodes by headspace gas chromatography
- Characterization of battery samples by the FlashSmart Elemental Analyzer

APPLICATION NOTE | 001967

Determination of inorganic anions in saturated lithium carbonate solution

Hua Yang, Jeffrey Rohrer
Thermo Fisher Scientific, Sunnyvale, CA, USA

Goal

To demonstrate the determination of inorganic anions in saturated lithium carbonate solution.

Introduction

Lithium carbonate is an important compound for industry. It is used in many applications including lithium-ion and lithium polymer batteries. Rechargeable batteries are an increasing part of our daily life as we use more portable electronic devices, including mobile phones. These batteries are also important for the electric car industry. Lithiumion batteries are the most commonly used rechargeable batteries because of their high volumetric energy density.[1] Lithium carbonate is a precursor to make lithium compounds used in lithium-ion batteries. Determination of inorganic anions in saturated lithium carbonate solution and using those values to determine the amounts in the solid, is desired by both battery recyclers and chemical suppliers.

This application note demonstrates the determination of inorganic anions in saturated lithium carbonate solution using the Thermo Scientific™ Dionex™ ICS-5000$^+$ Reagent-Free™ Ion Chromatography (RFIC™) system[2] with a Thermo Scientific™ Dionex™ IonPac™ AS23 anion-exchange column.[3]

Conclusion

This application note described a method for the determination of inorganic anions (fluoride, chloride, nitrite, bromide, nitrate, phosphate, and sulfate) in saturated lithium carbonate solution using a RFIC system with a Dionex IonPac AS23 column, carbonate eluent suppressor, and carbonate removal device. The method is sensitive (MDL 0.02–0.22 mg/L in saturated lithium carbonate solution), precise (RSD range 1–6%, and accurate (recovery range 95–107%).

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APPLICATION NOTE | 002343

Determination of chloride and sulfate in saturated lithium hydroxide solution

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Goal

To demonstrate the determination of chloride and sulfate in saturated lithium hydroxide solution.

Introduction

Lithium hydroxide is an important compound for industry. It is used in many applications, such as battery manufacturing (lithium-ion and lithium polymer batteries), welding, electroplating painting (pigments, binders, and biocides), cleaning products, and household care.[1]

Rechargeable batteries are an increasing part of our daily life as we use more portable electronic devices, including mobile phones. These batteries are also important for the electric car industry. Lithium-ion batteries are the most commonly used rechargeable batteries because of their high volumetric energy density.[2] Lithium hydroxide and lithium carbonate are precursors to make lithium compounds used in lithium-ion batteries. Determination of chloride and sulfate in saturated lithium hydroxide and lithium carbonate solutions, and using those values to determine the amounts in the solid, is desired by both battery recyclers and chemical suppliers.

A method was developed for lithium carbonate.[3] This application note demonstrates the determination of chloride and sulfate in saturated lithium hydroxide solution using a Thermo Scientific™ Dionex™ Reagent-Free Ion Chromatography™ (RFIC™) system[4] with a Thermo Scientific™ Dionex™ IonPac™ AS29-Fast-4µm anion-exchange column.[5] The method includes sample preparation using Thermo Scientific™ Dionex™ OnGuard™ II H cartridges.[6]

Conclusion

This application note describes a method for the determination of chloride and sulfate in saturated lithium hydroxide solution using an RFIC system with a 2 mm version of a Dionex IonPac AS29-Fast-4µm column. The method is sensitive (MDL = 0.09 mg/L for chloride and 0.13 mg/L for sulfate in saturated lithium hydroxide solution), precise (RSD range = 1–6%), and accurate (recovery range = 91–102%).

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APPLICATION NOTE | 000712

Rapid determination of residual *N*-methyl-2-pyrrolidone (NMP) in lithium battery electrodes by headspace gas chromatography

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[1]Thermo Fisher Scientific, Shanghai, China; [2]Thermo Fisher Scientific, Milan, Italy

Goal

The aim of this application note is to demonstrate the performance of a simple and robust method for the determination of residual *N*-methyl-2-pyrrolidone in electrode sheets using gas chromatography coupled to static headspace sampling.

Introduction

Lithium-ion batteries have been integrated into a wide range of consumer products due to their small size, utility, and affordability.[1] Because of their high energy density, they also offer a suitable solution for the currently growing number of electric vehicles. A lithium-ion battery consists primarily of the electrodes (anode and cathode), an electrolyte, and a separator. The cathode is mainly composed of active material (often binary or ternary alloys containing nickel, cobalt, and manganese) and a conductive skeleton. In the manufacturing process, *N*-methyl-2-pyrrolidone (NMP) is used to dissolve the cathode binder, polyvinylidene fluoride (PVDF), allowing for uniform coating of both sides of the collector. PVDF is a fluoropolymer characterized by high thermal stability and excellent chemical resistance, which makes it suitable to the specific chemical environment of Li-ion cells. Thus, it is mainly used as a positive binder and separator coating in lithium batteries.

After the battery electrode coating is completed, NMP needs to be removed to avoid residual amounts of it leading to the deterioration of the battery cycle performance and safety. Therefore, effective monitoring of residual NMP content in the electrode skeleton is key to ensure battery quality and electrode performance. To date, the standard procedure for extracting residual NMP from electrode sheets involves using a large quantity of a different and more volatile organic solvent in an ultrasonic bath. This is a time-consuming procedure that produces a considerable amount of organic waste.

An alternative, more convenient method for the determination of residual NMP on lithium battery electrodes was developed by using headspace sampling coupled to gas chromatography with flame ionization detection (FID). The headspace sampling technique allows for the extraction of NMP from the solid matrix in a fast and simple way by heating the sample in a closed vial, thus eliminating time-consuming sample preparation and organic solvent consumption. This method meets the sample throughput and productivity requirements of contract testing laboratories offering this service. A few microliters of water are added to the sample to keep consistency with the external standards preparation. The addition of such a low amount of water combined with the total vaporization technique[2] allows for quick, safe, and easy sample preparation in compliance with the ever-growing Green Chemistry concept.[3]

Conclusion

The results obtained in these experiments demonstrate that the TRACE 1600 Series GC in combination with flame ionization detection and the TriPlus 500 headspace autosampler is a reliable analytical tool for determination of residual NMP in Li-ion battery electrodes, with no time-consuming sample preparation required. The use of nitrogen as a carrier gas offers an efficient chromatographic process with reduced costs of operation.

- Headspace sampling allowed for the extraction of NMP from the solid matrix in a fast, simple, and safe way, removing time-consuming sample preparation and solvent waste.
- The direct column connection to the valve manifold with a short sample path ensured efficient analyte extraction and transfer resulting in excellent linearity (R^2=0.9996, AvCF %RSD=2.6) and % recovery between 98 and 115 with the calculated amount falling within 15% of the expected values.
- The highly efficient pneumatic control of the TRACE 1610 GC and the sample path inertness of the TriPlus 500 HS ensured reliable and reproducible analyte transfer to the column, offering outstanding repeatability and precision for everyday analysis of residual NMP, with absolute peak area %RSDs (n=6) of 1.02 in n=6 samples spiked 10 µg/g.
- Efficient purging of the pneumatic circuit of the TriPlus 500 HS and uniform heating of the sample path prevents the risk of carryover of high boilers. No NMP could be detected when analyzing a blank after the highest standard.
- Chromeleon CDS (compliant with the FDA Title 21 CFR part 11 requirements) ensures data integrity, traceability, and effective data management, allowing for easy and fast data processing, quantitation, and reporting.

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APPLICATION NOTE | 001152

Characterization of battery samples by the Flash*Smart* Elemental Analyzer

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Goal

Demonstrate accurate and precise CHNS determination in battery samples using the Thermo Scientific™ FlashSmart™ Elemental Analyzer.

Introduction

During the development and production of batteries, quality control (QC) of raw, intermediate and finished products is required to assure high quality and performance, and also safety. A simple and fast way to monitor elemental composition of these products for QC is by using the Flash*Smart* Elemental Analyzer (EA). Elemental characterization by Flash*Smart* EA mainly focuses on two elements: **carbon and sulfur**. Carbon and sulfur elemental determination allows accessing information on the purity of material which is critical as changes in production can cause the introduction of impurities and ultimately impact the lifetime and energy storage capacity of the battery.

The electrochemical properties of $LiFePO_4$ cathodes with different carbon contents are analyzed to determine the role of carbon as conductive additive, whereas as lead paste exhausted batteries are analyzed for sulfur content. Carbon and sulfur determination are also performed on graphite material used in negative electrode production.

Conclusions

The Flash*Smart* Elemental Analyzer, based on the combustion (modified Dumas method), determines CHNS of battery material in a wide range from low to high content and without the use of sample digestion or toxic chemicals, which is normally required by traditional methods. Simultaneous CHNS or NCS determination can be easily done in a single run, with support of dedicated Eager*Smart* Data Handling Software that provides you with customized reports of your data. The Flash*Smart* Elemental Analyzer demonstrates excellent repeatability, reproducibility, accuracy, and precision in the analysis of different battery material in a cost-efficient and simple way.

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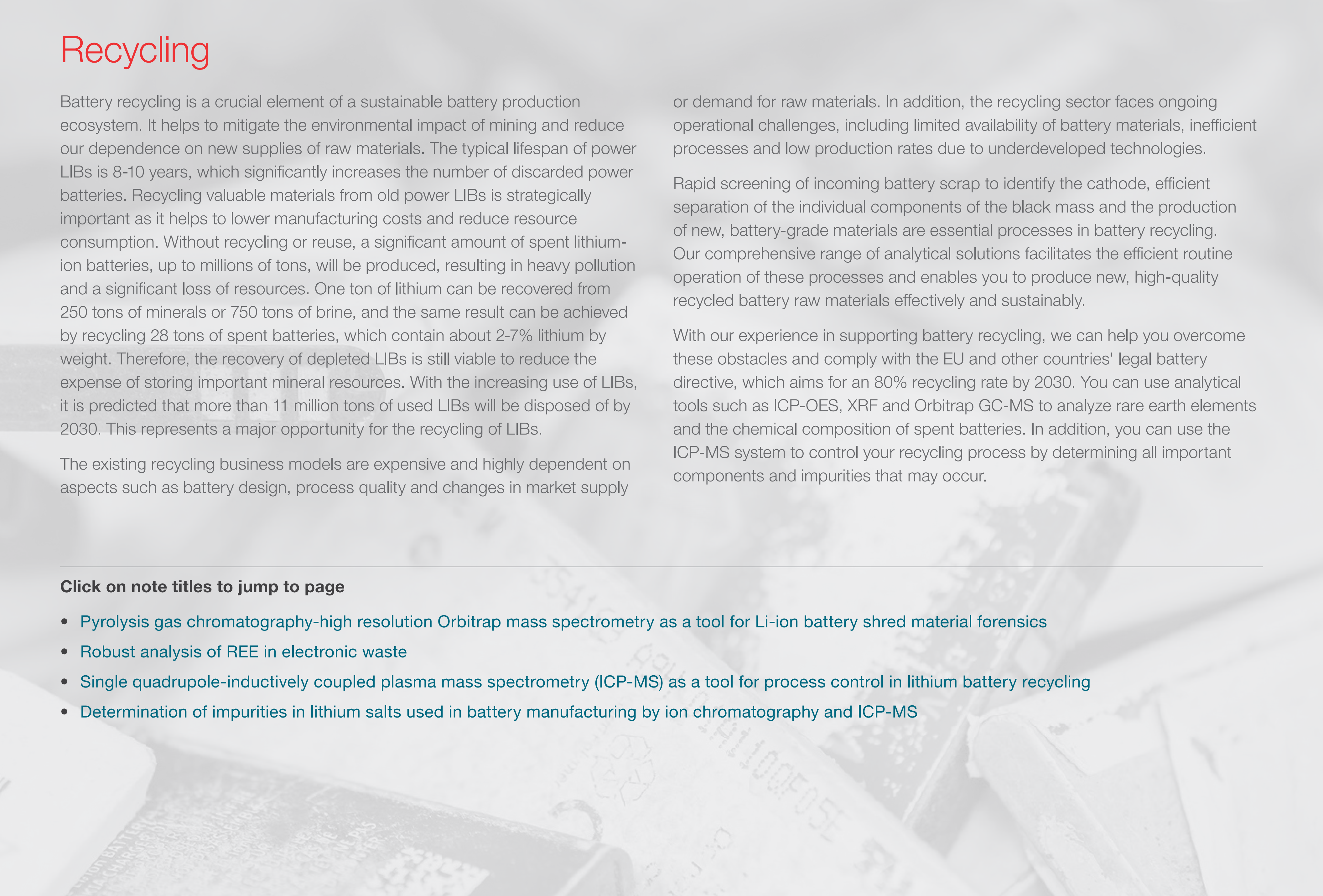
Recycling



Battery recycling is a crucial element of a sustainable battery production ecosystem. It helps to mitigate the environmental impact of mining and reduce our dependence on new supplies of raw materials. The typical lifespan of power LIBs is 8-10 years, which significantly increases the number of discarded power batteries. Recycling valuable materials from old power LIBs is strategically important as it helps to lower manufacturing costs and reduce resource consumption. Without recycling or reuse, a significant amount of spent lithium-ion batteries, up to millions of tons, will be produced, resulting in heavy pollution and a significant loss of resources. One ton of lithium can be recovered from 250 tons of minerals or 750 tons of brine, and the same result can be achieved by recycling 28 tons of spent batteries, which contain about 2-7% lithium by weight. Therefore, the recovery of depleted LIBs is still viable to reduce the expense of storing important mineral resources. With the increasing use of LIBs, it is predicted that more than 11 million tons of used LIBs will be disposed of by 2030. This represents a major opportunity for the recycling of LIBs.

The existing recycling business models are expensive and highly dependent on aspects such as battery design, process quality and changes in market supply or demand for raw materials. In addition, the recycling sector faces ongoing operational challenges, including limited availability of battery materials, inefficient processes and low production rates due to underdeveloped technologies.

Rapid screening of incoming battery scrap to identify the cathode, efficient separation of the individual components of the black mass and the production of new, battery-grade materials are essential processes in battery recycling. Our comprehensive range of analytical solutions facilitates the efficient routine operation of these processes and enables you to produce new, high-quality recycled battery raw materials effectively and sustainably.

With our experience in supporting battery recycling, we can help you overcome these obstacles and comply with the EU and other countries' legal battery directive, which aims for an 80% recycling rate by 2030. You can use analytical tools such as ICP-OES, XRF and Orbitrap GC-MS to analyze rare earth elements and the chemical composition of spent batteries. In addition, you can use the ICP-MS system to control your recycling process by determining all important components and impurities that may occur.

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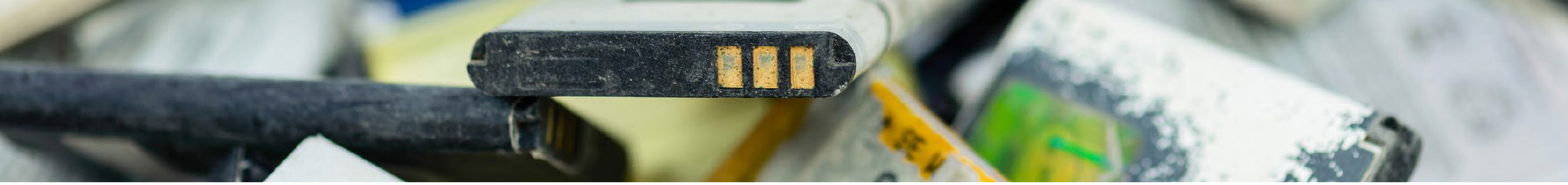
- Pyrolysis gas chromatography-high resolution Orbitrap mass spectrometry as a tool for Li-ion battery shred material forensics
- Robust analysis of REE in electronic waste
- Single quadrupole-inductively coupled plasma mass spectrometry (ICP-MS) as a tool for process control in lithium battery recycling
- Determination of impurities in lithium salts used in battery manufacturing by ion chromatography and ICP-MS



APPLICATION NOTE | 002297

Pyrolysis gas chromatography – high resolution Orbitrap mass spectrometry as a tool for Li-ion battery shred material forensics

Nicholas A. Warner[1], Dennis Kessen[2], Christoph Peschel[2], Sascha Nowak[2], Dominic Roberts[3],
Simon Nelms[3], and Daniel Kutscher[1]
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[3]Thermo Fisher Scientific, Hemel Hempstead, United Kingdom

Goal

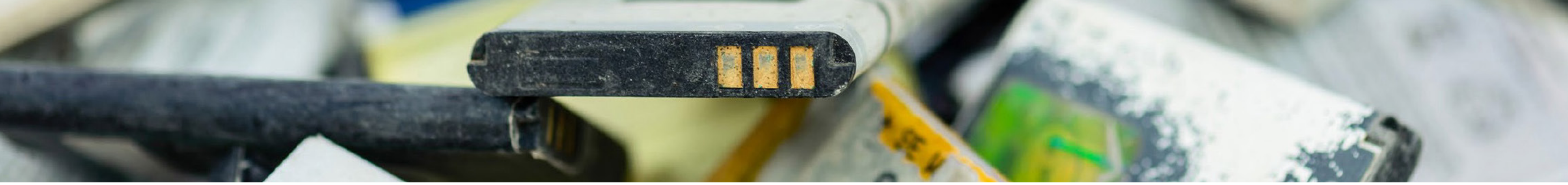
The goal of this application note is to demonstrate the identification of organic binders in lithium-ion battery shred material using pyrolysis as a sample introduction technique together with high mass resolution analysis with the Thermo Scientific™ Orbitrap Exploris™ GC 240 mass spectrometer.

Introduction

The invention of the lithium-ion battery (LIB) has had an unprecedented impact on our society, leading to the development of powerful and versatile portable electronics (e.g., smart phones, laptop computers). However, with the exponential increase in demand for LIBs to drive electrification of the automotive sector, greater stress is being placed on the finite resources of metals. Thus, recycling of LIB material will be critical to maintain the supply of essential minerals while reducing both economic and environmental costs associated with battery production.

Due to the complexity of LIBs, efficient separation of the various components is critical to maximize recovery of essential metals needed for battery production. This is of particular importance for organic binders, as their presence in LIB shred material can reduce the extraction efficiency of essential metal components during the recycling process.[1] This highlights the need for binder identification in LIB material to optimize recycling processing.

The complexity of the aged electrode matrix in which the binders are present requires selective separation and detection techniques for accurate identification. The combination of pyrolysis (Py) with gas chromatography-mass spectrometry (GC-MS) facilitates polymer analysis by overcoming the limitations of high molecular mass and low volatility while allowing for direct material analysis without pretreatment. Compositions of polymers can be thermally decomposed with subsequent analysis of their fragments by Py-GC-MS to provide reproducible chromatograms (pyrograms) as a recognizable fingerprint.[2,3]



In this study, the high selectivity of the Orbitrap Exploris GC 240 mass spectrometer in combination with pyrolysis gas chromatography is demonstrated for the direct analysis of LIB shred material for organic binder identification. Minimal sample material was needed due to the high selectivity and sensitivity of the Orbitrap Exploris GC 240 mass spectrometer, helping instrument performance remain robust during repetitive analyses. Specific binder marker molecules could be easily identified with the high selectivity of the instrument, enabling reliable binder identification.

Conclusion

This application note demonstrates the advantage of using the high mass resolution and accuracy power of the Orbitrap Exploris GC 240 system for fast and accurate binder identification.

- Direct sample introduction of LIB shred material using pyrolysis to overcome challenges in sample matrix volatility
- Rapid switching between EI and CI ionization techniques with NeverVent technology for binder identification and degradation mechanisms
- Fingerprint matching of binder materials in LIB shred material facilitated by selective removal of isobaric interferences/ distortions at sub ppm mass accuracy

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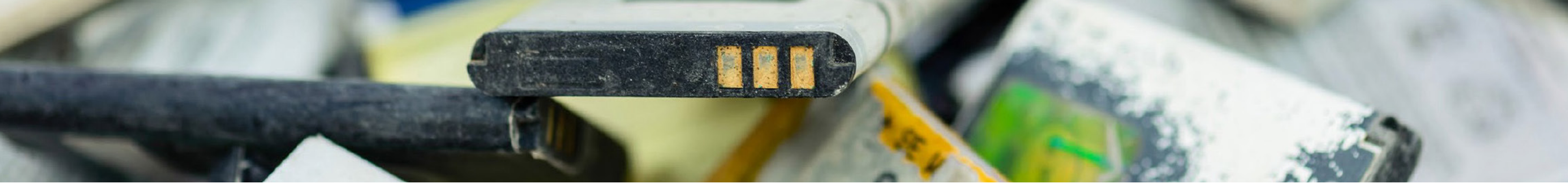

APPLICATION NOTE | 44466

Robust analysis of REE in electronic waste: High sample throughput with analysis times of less than 2 minutes per sample with the iCAP PRO Series ICP-OES

Sukanya Sengupta[1], Cristian Cojocariu[2] and Bhagyesh Surekar[1]
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Goal

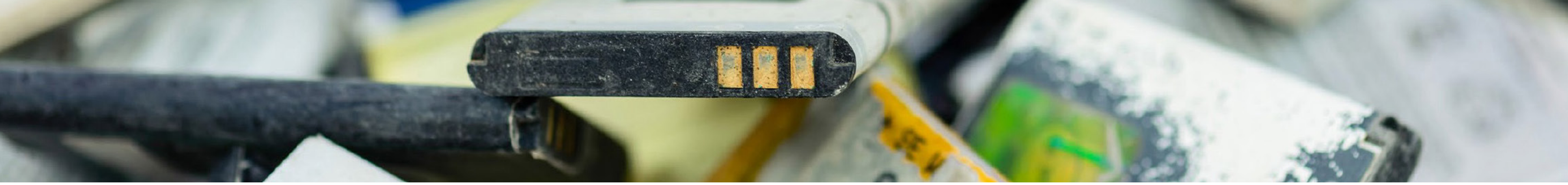
To demonstrate the capability of the Thermo Scientific™ iCAP™ PRO Series ICP-OES in axial viewing mode for high throughput, robustness, and highly sensitive analysis of trace and ultra-trace amounts of rare earth elements (REE) and metals in electronic waste such as mobile phones.

Introduction

Rare earth elements (REEs) are 17 elements in the periodic table, out of which 15 belong to the lanthanide series, found in the second last row of the periodic table – lanthanum (La) to lutecium (Lu). The remaining two elements are scandium (Sc) and yttrium (Y), commonly included in REEs because of their close geological occurrence in ores along with the lanthanides. In nature, REEs can often also be found associated with thorium (Th), an element commonly included and reported in REE studies. REEs, unlike their name might suggest, are not rare in nature but the ores from which they can be mined in an economically viable fashion are often hard to find. Nevertheless, REEs have important industrial applications: Ce, La, and Y in glass and metallurgy industries, Nd in permanent magnets, and Gd in medical image visualization, to name just a few, and this contributes to their high market demand and price. REEs are also used in various components of electronic devices such as in magnets, screens, etc.

High demand and limited resources of REEs in the world and the environmental impact of electronic waste (e-waste) enforce the development of sensitive, selective, and robust analytical technology to detect and characterize these elements from e-waste and environmental samples, such as wastewaters. The amount of electronics production as well as generation of e-waste has increased significantly in the last decades due to accelerating technological advances. Thus, the recycling and management of waste electrical and electronic equipment (WEEE) is a growing problem. Old and discarded electronic waste is emerging as an important source for recovery of valuable REEs and other metals like Fe, Ni, Ag, and Au.

REEs are analyzed using techniques like inductively coupled plasma mass spectrometry (ICP-MS), ICP optical emission spectrometry (ICP-OES) and X-ray fluorescence.1 ICP-MS is often the technique of choice for such analyses because of the high sensitivity and dynamic range of linearity achievable. ICP-OES is a suitable, low initial cost alternative for this kind of analyses. ICP-OES instruments are easy to operate, require low maintenance, and can provide



high sample throughputs. With new age technology it is possible for ICP-OES techniques to deliver similar detection limits, high linearity range, and very good spectral resolution for REEs at much lower investments.

In the current application note, WEEEs in the form of mobile phone wastes have been inspected and analyzed for REEs and other metals using the robust and highly sensitive iCAP PRO series ICP-OES system, demonstrating the analytical capabilities of the instrument for this kind of important application.

Conclusions

This application note describes an analytical solution that can be used for the determination of REEs, precious metals, and toxic elements in electronic waste using the iCAP PRO XP ICP-OES Duo system. The analytical procedure outlined in this note includes a simplified sample preparation technique to handle mobile phone waste and ensure that such complex samples are suitably prepared for analysis using ICP-OES.

The highlights are summarized below:

- Various REEs and other valuable or toxic metals were found in the analyzed e-waste samples, displaying both the potential value for such samples in the recycling industry as well as the need for responsible waste management.
- Excellent large linear dynamic range was obtained for REE (1 to 10,000 μg·L-1) and basic metals (1 to 100,000 μg·L-1), making the iCAP PRO XP ICP-OES Duo system an ideal analytical tool for multi-elemental analysis of samples such as e-waste, which have large variations in concentrations of constituent elements. The large dynamic range enables users to reliably analyze trace to ultra-trace levels at low concentration (Eu, Pb, etc.) as well as accurately quantify elements like Fe, Cu, etc. present in very high concentration.
- Highest sensitivity (in the low ppb range) was achieved for all targeted analytes in the Axial viewing mode. This is essential for detecting REEs, precious and poisonous elements, which often are present in very small amounts in e-waste samples.
- Recovery values within the expected (±15%) range were obtained for both real as well as laboratory simulated samples, demonstrating the accuracy of the technique.
- Excellent system robustness was demonstrated with no significant bias in the analytical results as observed for two consecutive days of continuous data acquisition. The vertical torch design in the Duo instruments enables plasma stability even with complicated samples matrices.
- Fast analysis time with less than 2 min/sample allows for high sample throughput and minimal downtime, making it ideal for contract testing laboratories or for users engaged with analyzing a high number of samples daily.
- For applications where sample throughput is not critical, the iCAP PRO X ICP-OES Duo system with slightly longer integration times, translated to more time per sample, can be used without compromising on analytical performance.
- Using analytical advances available on the iCAP PRO XP ICP-OES system, in combination with the Qtegra ISDS Software tools, suitable wavelength selection and effective interference removal of target elements in e-waste sample matrix is obtained with ease.

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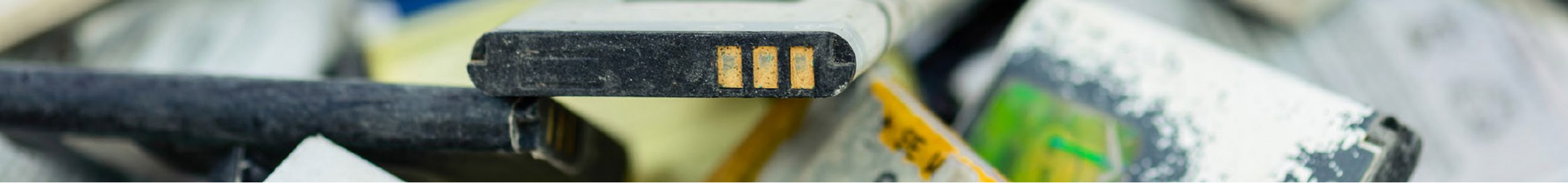

APPLICATION NOTE | 002085

Single quadrupole-inductively coupled plasma mass spectrometry (ICP-MS) as a tool for process control in lithium battery recycling

Tomoko Vincent[1], Keith Brent[2], Bruno Thompson[2], Simon Nelms[3]
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[3]Thermo Fisher Scientific, Hemel Hempstead, United Kingdom

Goal

To demonstrate the analysis of critical impurities in anode materials from recycled lithium-ion batteries with high sensitivity, accuracy, and robustness using single quadrupole ICP-MS.

Introduction

The transition to electric vehicles (EVs) is one of the key developments of the green energy revolution. As a result, the demand for fully electric vehicles has seen an acceleration in manufacturing capacity for both the vehicles themselves and the associated lithium-ion batteries as one of the core components. However, lithium-ion batteries are also used as important power storage materials in other areas, such as electricity storage from renewable energy sources, due to their power density and life cycle performance. In many cases, batteries that have reached their end of life in one application (i.e., vehicles) can still be efficiently and safely used for power storage in buildings. Nonetheless, the unprecedented ramp up in production also means that strategies and technology for complete and efficient recycling of used battery cells will have to be developed to fulfill the promise of a more sustainable future. Ultimately, the batteries used today should be considered under the principles of zero waste production and zero additional carbon footprint.

A typical lithium-ion battery consists of four main parts—a cathode, separator, anode, and electrolyte (Figure 1). The main components that can be easily recovered for recycling purposes are the cathode and anode materials. However, due to the high purity requirements in the production of new battery cells, any recycling process must be able to deliver new raw materials with sufficient purity to meet the required material quality.

This is particularly true for impurities, like trace elements. For the anode, which is produced from graphite powder, the purity level determines the product performance characteristics of thermal conductivity, strength, stability, and resistance.[1]

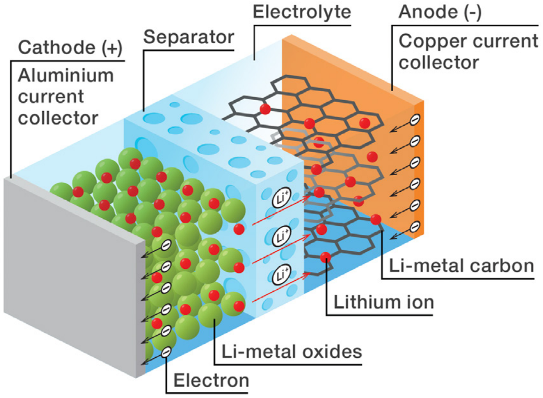
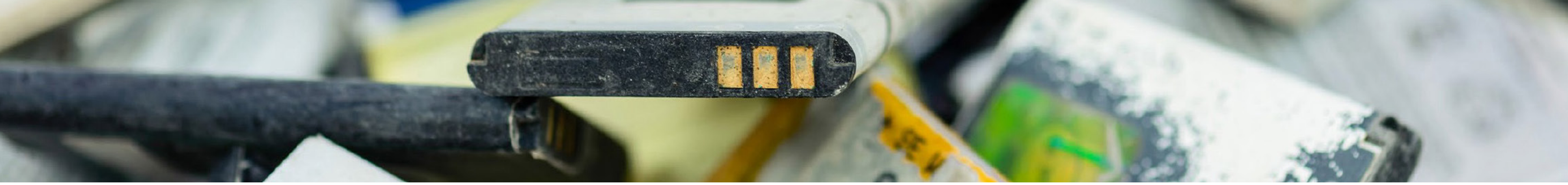


Figure 1. Lithium-ion battery diagram

In general, inductively coupled plasma mass spectrometry (ICP-MS)



Single quadrupole-inductively coupled plasma mass spectrometry (ICP-MS) as a tool for process control in lithium battery recycling *(continued)*

is the preferred analytical technique for the analysis of metals and related contaminants at low levels. However, the analysis of samples containing higher levels of total dissolved solids (TDS, typically above 0.5% w/v) or high acid concentration are a known challenge in ICP-MS. The complexity of the sample matrix can significantly affect the sensitivity of the instrument or cause problematic polyatomic interferences.

This application note describes an optimized analytical method for the analysis of recycled lithium battery anodes. The anode material (typically graphite powder) needs to be separated from the battery structure and any remaining binder materials. Finally, the recycled material needs to be dissolved to be accessible to ICP-MS based analysis. In this case, this is accomplished by microwave assisted digestion, providing a solution containing a high concentration of aqua regia. To overcome the analytical challenges described above, a Thermo Scientific™ iCAP™ RQplus ICP-MS equipped with the novel PLUS torch[2] was applied for the analysis, with superior interference removal achieved using kinetic energy discrimination (KED) with pure helium as the collision gas. The instrument was operated using Argon Gas Dilution (AGD)[3] to facilitate the direct analysis of a highly concentrated digestion solution.

Conclusions

The iCAP RQplus ICP-MS, operated using AGD, enables analysts to perform accurate and reliable elemental analysis in challenging samples. This was demonstrated for the analysis of 44 elements in a large number of samples, covering a wide range of target concentrations. The analytical method was rigorously tested for performance, and the results obtained clearly demonstrated the following analytical advantages for anode solution analysis:

- AGD-low mode with excellent sensitivity allowed the aspiration of anode recycling with 16% (m/m) aqua regia samples and allowed excellent MDLs to be obtained, eliminating the need for labor-intensive further manual sample dilution, and accelerating sample throughput using a Step Ahead feature for minimizing total analysis time.
- High performance He KED mode provided the advanced performance required for the accurate determination of elements typically suffering from polyatomic interferences in the presence of higher amounts of hydrochloric acid.
- Excellent CCV recovery and spike recovery results, as well as stable and consistent internal standards response, were obtained across a batch containing 189 samples of a 1% w/w TDS anode solution, demonstrating the reliability of the method.

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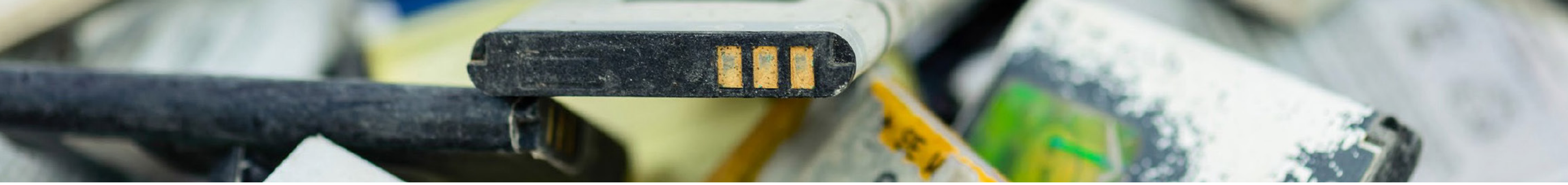

APPLICATION NOTE | 002666

Determination of impurities in lithium salts used in battery manufacturing by ion chromatography and ICP-MS

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Goal

Quantification of ionic impurities present in lithium-based raw materials used in batteries by Thermo Scientific™ ion chromatography and ICP-MS systems.

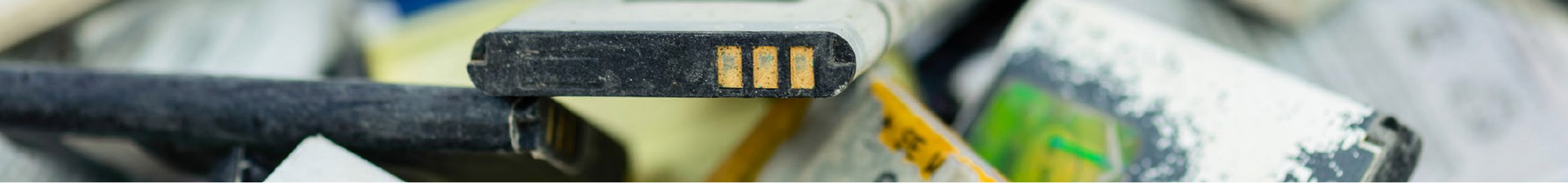
Introduction

Lithium-ion batteries (LIBs) have revolutionized the modern electronic industry by providing high energy density, longer lifespan, and a more environmentally friendly alternative to traditional lead-acid and nickel-cadmium batteries. These rechargeable batteries are widely used in portable electronic devices, electric vehicles, and energy storage systems.[1]

The key advantages of LIBs over other battery chemistries are their high energy density, which allows them to store more energy in a smaller size and weight, and their low self-discharge rate, which means that they can hold their charge for a longer time when not in use. Additionally, LIBs have a longer lifespan compared to other rechargeable batteries, which makes them more cost-effective.[1,2]

However, despite their numerous advantages, there are still challenges associated with the production and use of LIBs. These challenges include the high cost of raw materials, concerns regarding their safety and stability, and the environmental impact of battery disposal. To overcome these challenges, there is ongoing research to improve the performance and sustainability of LIBs.

Lithium-ion batteries are now ubiquitous in modern-day technology, powering everything from smartphones and laptops to electric vehicles and grid-scale energy storage. Central to the performance of LIBs are the salts that make up their electrolytes which facilitate the movement of lithium ions between the battery's electrodes. As such, the choice of electrolyte salt plays a critical role in determining the battery's overall performance, including its energy density, power output, safety, and lifespan. For instance, impurities can interfere with the electrochemical process, leading to reduced battery capacity, and certain elements can react with battery components causing instability or potential hazards, such as overheating or even battery failure. In recent years, there has been considerable research aimed at developing new electrolyte salts and



improving existing ones to enhance the performance of LIBs and enable their broader adoption.[3]

In this Application Note, various electrolyte salts (raw materials) such as lithium carbonate, lithium fluoride, lithium hydroxide, and phosphorus pentachloride were tested for their ionic and elemental impurities content using ion chromatography (IC) and ICP-MS separately. The percentage assay of these salts was also checked using an IC system.

Conclusion

This study demonstrated the accurate quantification of anionic impurities and cations in lithium salts by IC. This method uses a Dionex IonPac AS11-HC column with a hydroxide eluent, which is ideal for separating a wide range of inorganic anions with high sensitivity. The high capacity Dionex IonPac CS16 column is ideal for the analysis of a wide range of cations with high sensitivity and resolution. Eluent generation technology is used for both anionic and cationic methods and provides uniform, highly stable eluent concentrations, which helps to achieve highly reproducible results.

One of the main advantages of using ICP-MS for elemental impurities determinations is its ability to detect low concentration levels (sub-ppb) routinely and with ease. The fast scanning speed, robustness, and selectivity of ICP-MS allow for rapid, accurate, and efficient analysis of elemental content in salts.

Since regulations are becoming tougher, fast and accurate techniques are the solution for the present and future. In this regard, IC and ICP-MS have proven to be the best solutions in terms of productivity and performance for the analysis of ions and elements in lithium-ion battery electrolyte salts.

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