Agilent
Trusted Answers

# Agilent Solutions for the Lithium-Ion Battery Industry


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# Lithium-Ion Battery Industry is Thriving

High voltage, high specific energy, long cycle life, environmental friendliness, good energy density and power density, are some advantages of lithium-ion batteries in providing the best overall performance for power batteries. Li-ion batteries are widely used in fields such as consumer electronics (for mobile phones and laptops), mobility (for rail transit and new energy vehicles), and energy storage (small-scale power supply, uninterruptible power supply (UPS), communication base station, new energy), among others.

In recent years, there is a rapid growth of product output in the Li-ion battery industry. Coupled with the surge in the new energy vehicle market, power batteries have gradually become the main applications for lithium-ion batteries. In addition, there has been increased interest in the new energy and lithium-ion battery industries. Consequently, new requirements have been set for technological breakthroughs in lithium-ion batteries in terms of high safety, long life, and high energy density.

In the upstream and midstream of the lithium-ion battery industry chain, the quality control of raw materials and products requires instrumental analysis methods to test anode and cathode materials, electrolytes, separators, and other raw materials. In the midstream of the lithium-ion battery industry chain, a comprehensive physicochemical analysis on each part of the battery using instruments (e.g., atomic spectroscopy, molecular spectroscopy, chromatography and mass spectrometry) is also necessary for research and development on the product performance and safety. When recycling and reusing waste Li-ion batteries, a quantitative analysis on valuable metallic elements must be performed using instruments like atomic spectroscopy.

As a global leader in analytical technology, Agilent has extensive experience and data on testing Li-ion battery materials. We can help you realize your objectives, whether it is for raw material tests or scientific research.

# Key Materials For Lithium-Ion Batteries

A lithium-ion battery works by repeated and reversible ingress and egress of lithium ions across anode and cathode materials to complete the conversion between chemical and electrical energies.

When charging, lithium ions run away from cathode materials, pass through electrolyte and separator, and enter into anode materials, while electrons are transferred, through external circuits, from cathode to anode materials. On the contrary, the discharge process is characterized by electrons transferring from anode to cathode materials to power external load equipment.

## Four key materials for Li-ion batteries include anode and cathode materials, electrolytes, and separators

### Cathode Materials

Cathode materials must be active materials with high oxidation-reduction potential, proneness to have chemical reactions, and structural stability, allowing batteries to achieve reversible and controllable energy storage and conversion. At present, the cathode materials commonly used in Li-ion batteries include a series of lithium-containing oxides, such as lithium cobaltate (LCO), lithium titanate (LTO), lithium iron phosphate (LFP), lithium manganese (LMO), and lithium nickel cobalt manganate (NCM; a lithium-ion ternary material).

The performance of cathode materials can affect Li-ion batteries mainly in terms of energy density, safety, and cycle life.

### Anode Materials

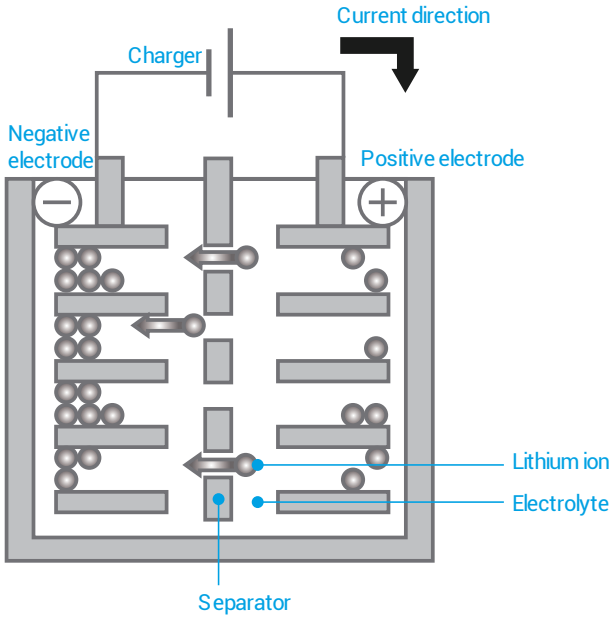
In a Li-ion battery, materials allowing reversible ingress and egress of Lithium ions are used as anode materials. Compared to cathode materials, anode materials should have lower potential to be good energy carriers with relatively high stability. Many types of anode materials are used in lithium-ion batteries, and can be divided by chemical composition into metals (including alloys), inorganic non-metals (carbon, silicon and other materials) and metal oxides. Among them, the more mature technologies are those involving carbon-containing anode materials.

The performance of Li-ion anode materials is a main contributor to energy density of a Li-ion battery.

### Electrolyte

Electrolyte in a Li-ion battery is composed of high-purity organic solvents, electrolyte lithium salts, and additives. It is a non-aqueous medium through which lithium ions flow during charging and discharging of a lithium-ion battery. The performance of the electrolyte is critical for ensuring the safety of lithium-ion batteries.

Common electrolyte lithium salts include lithium hexafluorophosphate ($LiPF_6$), lithium perchlorate ($LiClO_4$), lithium tetrafluoroborate ($LiBF_4$), and lithium bisoxalate borate (LiBOB), among which $LiPF_6$ is now a more mature lithium salt product.




Schematic diagram of charging principle of Li-ion batteries.

Solvents commonly used in Li-ion batteries include conventional carbonate solvents such as ethylene carbonate (EC), dimethyl carbonate (DMC), diethyl carbonate (DEC), ethyl methyl carbonate (EMC), and propylene carbonate (PC), along with new organic solvents such as ethers and hydroxy acid esters.

As for electrolyte additives like vinylene carbonate (VC), fluoroethylene carbonate (FEC), vinyl ethylene carbonate (VEC) and biphenyl (BP), they can be functionally classified into SEI membrane optimizer, overcharge protection additive, flame retardant additive, additive to improve electrolyte conductivity, and additive to control water and acid content in electrolyte.

### Separator

The separator in a Li-ion battery is a microporous structure film that separates active substances of positive and negative electrodes. A separator must possess good ion permeability to allow ions in the electrolyte to pass through freely, as well as insulation for safety purposes, to prevent any short circuit caused by contact between the two electrodes. Currently, separators with bulk applications in Li-ion batteries mainly include PP, PE and multilayer composite membranes.

The performance of the separator directly determines the interface structure of the battery, thereby affecting capacity, cycle performance, current density in charge and discharge, and other key electrical properties of the battery.

# Common Analysis Items in the Lithium Battery Industry

## Lithium battery company raw material (upstream material) testing or lithium battery production management (cathode and anode materials, separator, electrolyte, etc.): including identification, and analyses on physicochemical properties, electrochemical performance, and chemical composition.

- Analysis of metal or magnetic impurities (AA, ICP-OES, and ICP-MS)
- Analysis of $SO_4^{2-}$, $Cl^-$ and other anions, and non-metal elements such as Si (UV-Vis)
- Identification of raw materials such as electrolytes (FTIR)
- Assay of organics in graphite-based anode materials (GC/MS, LC, and LC/MS)
- Composition analysis and solvent component assay (GC, GC/MS, LC, and LC/MS) of electrolyte (including additives)

## Research and development of Li-ion batteries: aims to improve key indicators of products, such as safety, cycle life, power density, and energy density

### Gas composition analysis for battery swelling (GC and micro GC)

When evaluating the aging process of lithium-ion batteries, it is necessary to analyze the gas generated during battery degradation.

In a cell cycle, chemical reactions will be caused by the contact between the electrolyte and the positive/negative electrode, resulting in a swollen battery that poses a big safety risk. The composition of the battery swelling gas is commonly analyzed using gas chromatograph (GC).

### Analysis of electrolyte and additives (GC and GC/MS)

The composition and content of ester compounds in the electrolyte are critical to the battery cycle performance.

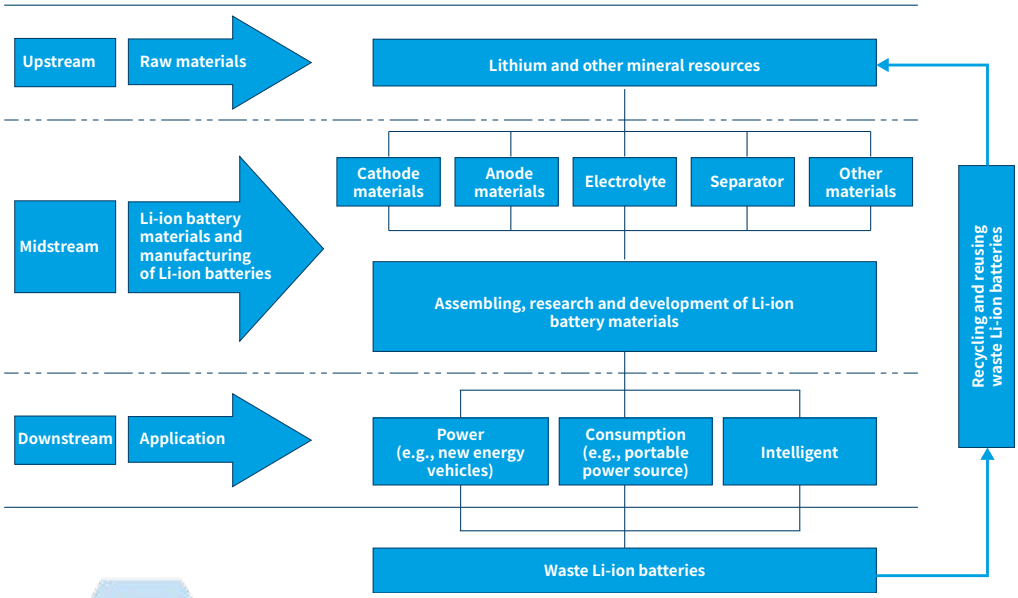
The organic electrolyte additive for Li-ion battery is low in consumption and cost, and yet able to improve performance in many aspects. To ensure a stable operating voltage and good performance of the battery at high and low temperatures, the content of each additive deserves more attention, which is usually analyzed by GC or GC/MS.

### Analysis of unknown electrolyte components (GC/Q-TOF and LC/Q-TOF)

For unknown trace components produced in the circulation experiment, it is recommended to choose GC/Q-TOF or LC/Q-TOF for analysis.

## Recycling of waste Li-ion batteries: Extract and recycle valuable metallic elements in waste Li-ion batteries

Analytical assay of valuable metallic elements (Ni, Co, Mn, Li, etc.) (AA and ICP-OES).



# Agilent Atomic Spectroscopy in the Lithium-Ion Battery Industry

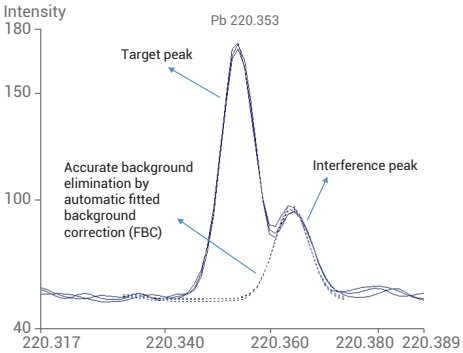
Robust matrix that can stand up to any extreme sample challenges.

## Assay demands from the industry

Monitoring and controlling metal contaminants in the raw materials used for cathodes, anodes, and electrolytes is an important quality control function within the Li-ion battery industry.

Several global standards organizations are working on regulations and standards for batteries and battery materials, including the IEC and ISO. Many countries are also developing or have implemented testing standards. For example, the SAC (China national standards) has released many standards for the lithium battery industry. There are also environment, health and safety regulations that impact battery manufacture, use, and end of life. Many of these standards specifically require the use of instrumentation such as inductively coupled plasma optical emission spectroscopy (ICP-OES).

Agilent 5800 and 5900 ICP-OES




Background elimination by unique automatic fitted background correction (FBC).

## Application of ICP-OES

### Challenges

Samples typical of the Li-ion battery industry often have complex matrices (containing high lithium salts, organics, or fluorine components). The samples may also contain fine particles that block the nebulizer and and lead to measurement problems.

### Agilent solutions

- The variable high background signal associated with battery material samples can make background correction difficult. The Agilent fitted background correction function can be used to remove the effects of a sloping baseline, or a complex background emissions. Using this function will simplify operation and improve data accuracy.
- The VistaChip III detector in the 5800 and 5900 instruments allows the measurement of both high concentration elements and low concentration contaminant elements in each sample, in the same analysis.
- Agilent offers a range of sample introduction components that are well suited to analysis of these types of samples. This includes inert components, suitable for the analysis of samples containing HF, as well as nebulizers designed for handling high dissolved solids.
- The Agilent IntelliQuant feature allows screening of samples to identify which elements are in the sample, and in what proportions. The feature also provides expert guidance on avoiding spectral interferences to get the right results.
- The Neb Alert function provides real-time warnings on unpredictable events such as nebulizer blockages or leaks. These alerts enable quick identification of problem and less wasted time when analyzing samples that may have undissolved particles.

### Typical application data

The macro and trace elements in the digestion solution of lithium nickel cobalt manganate (a ternary material used in cathodes) were analyzed simultaneously. The test results and spike recoveries are provided in the following two tables.

Analysis results of trace elements in lithium nickel cobalt manganate (NCM).

| Trace element | Al | Ba | Be | Cu | Mg | Na | Sr |
|---|---|---|---|---|---|---|---|
| Wavelength | 167.019 nm | 493.408 nm | 313.107 nm | 324.754 nm | 279.553 nm | 589.592 nm | 407.771 nm |
| Test result (mg/L) | 0.004 | 0.001 | 0.001 | 0.0003 | 0.0823 | 0.277 | 0.0003 |
| Spike amount (mg/L) | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 |
| Spike result (mg/L) | 0.097 | 0.099 | 0.09333 | 0.09933 | 0.17567 | 0.3703 | 0.09643 |
| Recovery (%) | 93% | 98% | 92% | 99% | 94% | 93% | 96% |

Analysis results of macro elements in lithium nickel cobalt manganate.

| Macro element | Co | Li | Mn | Ni |
|---|---|---|---|---|
| Wavelength | 236.379 nm | 670.783 nm | 280.108 nm | 222.486 nm |
| Test result | 19.7% | 7.7% | 18.6% | 20.5% |
| RSD%, n = 6 | 0.21% | 0.51% | 0.27% | 0.25% |
| Labeled content (%) | 20 ± 2 | 7.6 ± 0.5 | 18.5 ± 2 | 20 ± 2 |

The control of impurities in lithium hexafluorophosphate, used in the electrolyte of lithium batteries, is important for performance and safety. Samples were diluted with organic solvent.The results and recoveries of the samples are provided in the following table.

Analysis results of impurity elements in lithium hexafluorophosphate electrolyte.

| Impurity element | Ca | Cd | Cr | Fe | Hg | K | Mg | Na | Pb |
|---|---|---|---|---|---|---|---|---|---|
| Wavelength | 396.847 nm | 226.502 nm | 267.716 nm | 259.940 nm | 253.652 nm | 766.491 nm | 279.553 nm | 588.995 nm | 283.305 nm |
| Test result (mg/L) | 0.0016 | N.D.* | N.D. | N.D. | N.D. | N.D. | N.D. | N.D. | N.D. |
| Spike amount (mg/L) | 0.02 | 0.02 | 0.02 | 0.02 | 0.05 | 0.1 | 0.02 | 0.02 | 0.05 |
| Spike result (mg/L) | 0.021 | 0.019 | 0.020 | 0.018 | 0.056 | 0.104 | 0.019 | 0.020 | 0.045 |
| Spike recovery | 98% | 97% | 98% | 91% | 111% | 104% | 97% | 98% | 90% |

** N.D.: Not detected*

7850 ICP-MS

7900 ICP-MS

## Applications of ICP-MS

### Challenges

The high salt levels in Li-ion battery materials result in complex matrices. Analysis via ICP-MS often requires multiple dilutions before sample measurement. This is time-consuming and can easily introduce contamination.

### Agilent solutions

- The ultra-high matrix introduction (UHMI) system uses clean argon gas to dilute samples as they are introduced. UHMI enhances the instrument's ability to directly analyze complex matrix samples and improves productivity by avoiding manual dilutions. UHMI can handle samples with up to 25% total dissolved solids (TDS). This compares to the less than 0.2% TDS that conventional instruments can handle.
- The octopole reaction system (ORS[4]) with helium collision mode removes polyatomic interferences from lithium samples whilst maintaining analyte sensitivity.
- Agilent ICP-MS instruments have up to 11 orders of magnitude dynamic range, from sub-ppt to percent-level concentrations. This capability allows the measurement of trace elements and majors in the same run.

### Typical application data

An 7900 ICP-MS was used to analyze three types of cathode materials: lithium nickel cobalt manganate (NCM; ternary material); lithium nickel cobalt aluminate (NCA; ternary material), and lithium iron phosphate (LFP). The test results and spike recoveries are provided in the table below.

Test results and spike recoveries of three types of cathode materials.

| Sample | $^{52}$Cr [He] | $^{63}$Cu [He] | $^{66}$Zn [He] | $^{75}$As [He] | $^{78}$Se [He] | $^{95}$Mo [He] | $^{111}$Cd [He] | $^{208}$Pb [He] |
|---|---|---|---|---|---|---|---|---|
| NCA result (ng/mL) | 0.368 | 0.299 | 2.243 | 2.532 | 1.341 | N.D. | 0.019 | 0.297 |
| NCA+5 result (ng/mL) | 5.259 | 5.36 | 6.613 | 7.018 | 5.886 | 4.94 | 4.999 | 5.464 |
| Recovery (%) | 97.8% | 101.2% | 87.4% | 89.7% | 90.9% | 98.8% | 99.6% | 103.3% |
| NCM result (ng/mL) | 2.186 | 1.123 | 1.512 | 3.81 | 0.626 | 0.164 | 0.551 | 0.355 |
| NCM+5 result (ng/mL) | 7.514 | 6.427 | 7.224 | 9.092 | 5.459 | 5.668 | 6.098 | 5.917 |
| Recovery (%) | 106.6% | 106.1% | 114.2% | 105.6% | 96.7% | 110.1% | 110.9% | 111.2% |
| LFP result (ng/mL) | 69.41 | 0.119 | 0.764 | 0.577 | 0.125 | 1.377 | 0.02 | 0.135 |
| LFP+5 result (ng/mL) | 74.133 | 4.782 | 5.975 | 5.478 | 4.47 | 6.531 | 5.461 | 5.461 |
| Recovery (%) | 94.5% | 93.3% | 104.2% | 98.0% | 86.9% | 103.1% | 108.8% | 106.5% |

# Agilent Molecular Spectroscopy in the Lithium-Ion Battery Industry

Efficiency. Accuracy. Robustness.

Agilent Cary 60 UV-Vis with Fiber Optics

## Assay demands from the industry

According to YS/T 582-2013 "Battery Grade Lithium Carbonate" and GB/T 26008-2010 "Battery Grade Lithium Hydorxide Monohydrate", it is required to detect substances like $SO_4^{2-}$, $Cl^-$ and Si by spectrophotometry.

In GB/T 19282-2014 "Analytic Method For Lithium Hexafluorophosphate" and other relevant standards, it is required to perform a product identification assay by infrared spectroscopy (or equivalent).

## Cary 60 UV-Vis

- A unique pulsed xenon flashlamp with an exceptionally long lifetime covers wavelengths in the ultraviolet-visible region, and can be used to directly replace 2 light sources (deuterium and tungsten lamps) of conventional UV-visible light spectrophotometer.
- Instant high energy output for more stable and accurate results.
- The measurement is not affected by the indoor light, so no need to close the sample chamber cover, which is more convenient for adding reagents or configuring different accessories.
- One-of-a-kind fiber optics eliminate the need to frequently change samples in the pool, delivering much higher productivity.

Agilent Cary 630 FTIR

## Cary 630 FTIR spectrometer

- With compact design and ease of use, Cary 630 represents the world's smallest benchtop FTIR.
- Graphical working interface enables the simplest operation.
- Moisture and shock resistance, robustness, and operational reliability.
- Short optical path design to minimize interferences from water vapor and carbon dioxide in the air.
- Short time to results, with a detection speed of above 2 times that of a conventional system.

# Agilent Micro GC in the Lithium-Ion Battery Industry

Measure anytime, anywhere. Get results you need in seconds.

Agilent 990 Micro GC

Agilent 990 Micro GC Field Case

Quantitation results of Li-ion battery swelling gas

| Ingredients | Peak Area | Concentration (%) (external standard method) | Concentration (%) (normalization) |
|---|---|---|---|
| $H_2$ | 716.3 | 23.6108 | 21.7778 |
| $O_2$ | 15.68 | 4.2114 | 3.8844 |
| $N_2$ | 78.42 | 21.1097 | 19.4708 |
| $CH_4$ | 4.309 | 0.5974 | 0.5510 |
| CO | 105.2 | 41.6783 | 38.4425 |
| $CO_2$ | 169.6 | 16.7355 | 15.4362 |
| $C_2H_4/C_2H_2$ | 2.247 | 0.2355 | 0.2172 |
| $C_2H_6$ | 2.437 | 0.2203 | 0.2032 |
| $C_3H_6$ | 0.1158 | 0.0088 | 0.0081 |
| $C_3H_8$ | 0.1252 | 0.0092 | 0.0085 |

## Assay demands from the industry

During recycling or storage of lithium-ion batteries, the SEI membrane may be decomposed to produce gases due to film formation and oxidation of the electrolyte components, overcharge and overdischarge of the battery, internal micro short circuit or others. Also, a battery swelling may be caused by gases from electrolysis at a high content of water, thereby posing high safety risks. Common gas-producing components include permanent gases (e.g., $H_2$, CO, and $CO_2$) and alkanes (e.g., $CH_4$, $C_2H_4$, and $C_2H_6$).

## Application of Micro GC

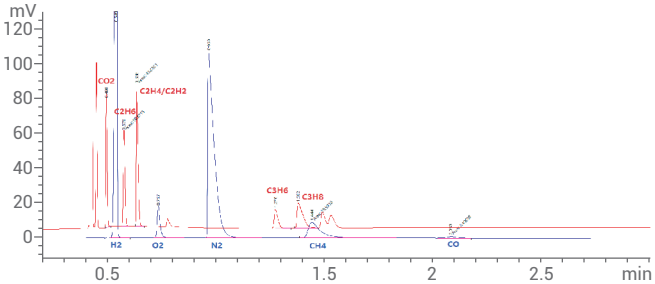
### Agilent solutions

Agilent recommends the Agilent 990 Micro GC for analysis of composition of battery swelling gases.

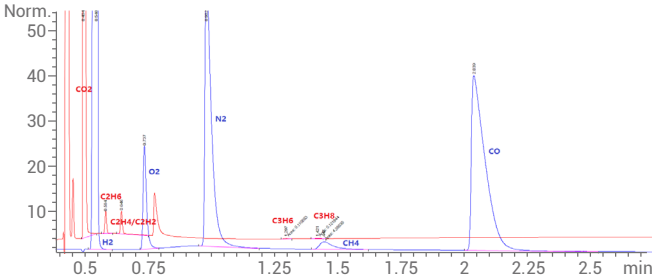
- The 990 Micro GC is an analytical instrumentation specially designed for gas composition analysis, and in standard version, it is combined with a micro-mechanical sampler and a highly sensitive TCD detector, which is suitable for the analysis of low-content components of battery swelling gases. Up to 4 independent analysis channels can be selected to simultaneously analyze different swelling gases. Each channel is a separate GC with pneumatics, sampler, column, and detector. The channel module is easy to configure, and ready to use.
- For lithium-ion battery swelling gases, 10–20 mL syringe for manual sampling is a common choice when using 990 micro GC, and the results can be obtained in only a few minutes after injection, delivering much higher efficiency in research, development, and test of Li-ion batteries.
- Compared to an ordinary benchtop GC, the 990 is more robust and compact, and also lower in consumption. This equipment is suitable for gas analysis in the laboratory, online and on-site, and can also be easily transferred between different test points. The optional portable field case, equipped with carrier gas supply and rechargeable batteries, increases the system's flexibility even more.

### Typical application data

In this experiment, use the 990 micro GC to analyze the composition of Li-ion battery swelling gases, while selecting 2 channels, PPQ and MS5A. The table below provides the quantitative analysis results of the battery swelling gases; the following figures are the mixed standard gas spectrum and the swelling gas analysis spectrum for actual battery sample, respectively.




Mixed standard gas spectrum




Swelling gas analysis spectrum for actual battery sample

# Agilent GC and GC/MS in the Lithium-Ion Battery Industry

Reliable, proven performance. A faster route to insight.

Agilent 8890 GC

## Assay demands from the industry

- When testing and developing Li-ion electrolyte raw materials, GC/MS is a typical option for qualitative and quantitative analysis of Li-ion battery solvents (formulation ingredients) and additives.
- According to GB/T 24533-2009 "Graphite Negative Electrode Materials for Lithium Ion Battery" and other relevant standards for lithium-ion batteries, it is required to use GC/MS to detect organics such as polychlorinated biphenyls (PCBs), polybrominated biphenyl (PBBs), and acetone.

## Application of GC and GC/MS

### Agilent solutions

Agilent recommends GC-FID or GC/MS for accurate and quantitative analysis, and GC/MS for qualitative analysis of formulation ingredients. The Agilent MassHunter toolkit effectively transforms data into scientific insights to help you quickly interpret data by complex sample matrix and obtain results.

- MassHunter Unknowns Analysis Software: Analyze spectra quickly and smartly. With a built-in automatic deconvolution software, the compound peaks can be purified faultlessly to improve the matching of detected compounds and the detection rate of low-content compounds. Also, the equipment will effectively eliminate the matrix interference in the background and enable automatic library matching during analysis of compounds in samples, allowing easy and intelligent acquisition of qualitative results of target substances in complex samples.
- MassHunter Library Editor: When it comes to electrolyte analysis, some compounds, due to advanced technologies or innovative compounds, are not included in the NIST library so that the corresponding qualitative analysis can not be managed by conventional ICP-MS. With the easy-to-use library editor in MassHunter software, the standard profiles and information for typical Li-ion battery electrolyte components on the market are now compiled into a special database for a later qualitative analysis of organics in the electrolyte.

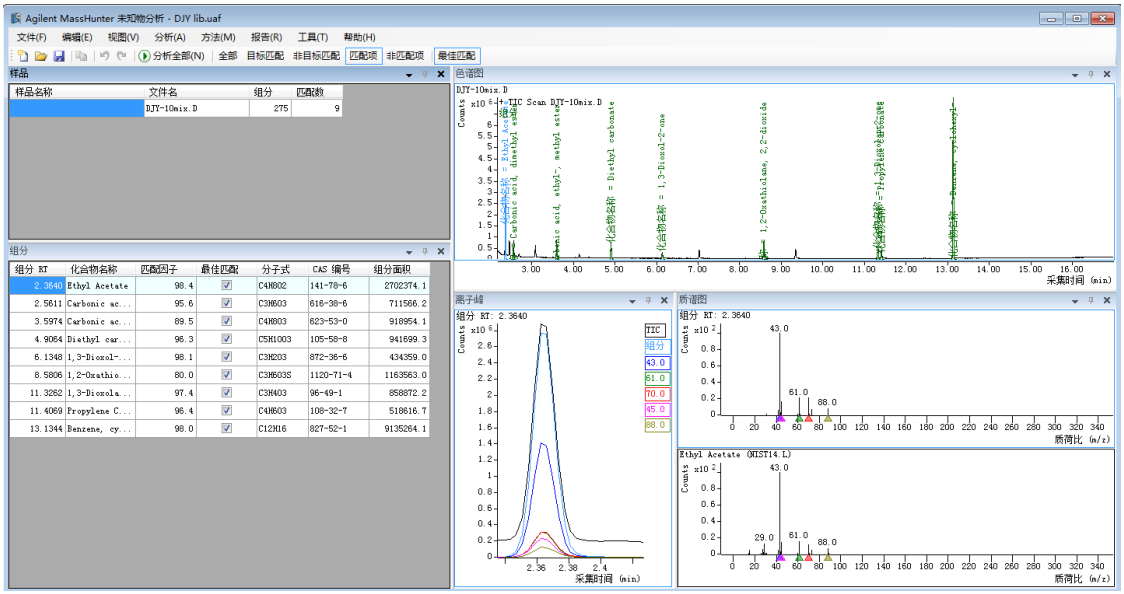

Agilent Intuvo 9000 GC and 5977B MS

### Typical application data

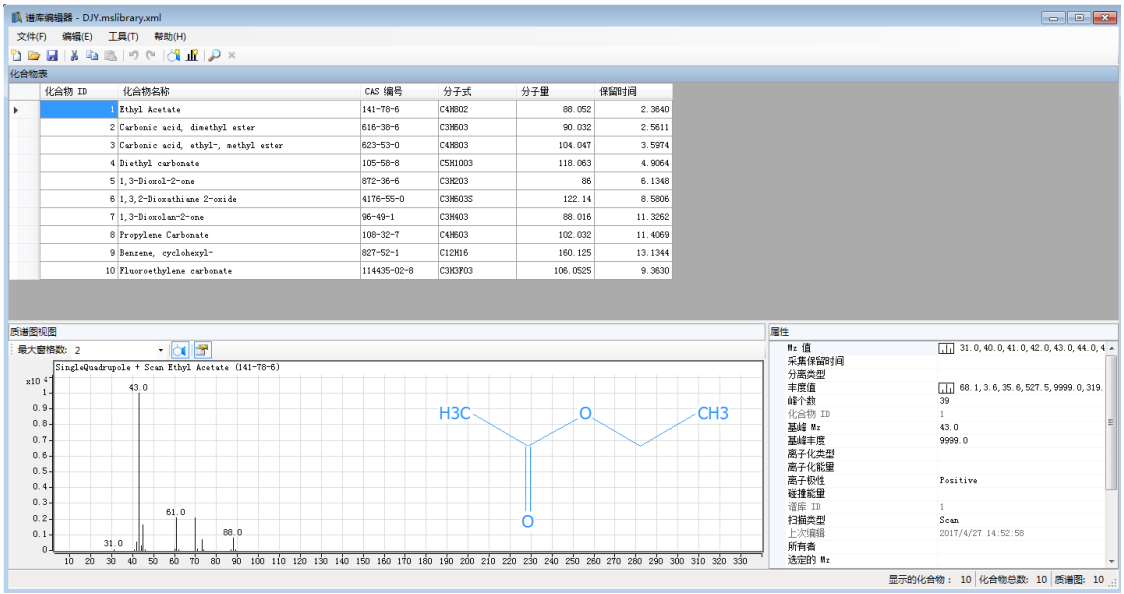
In this experiment, the Agilent GC/MS platform are used in combination with MassHunter software to analyze 10 organic electrolyte components. The table and figure below respectively provide the selection of ion monitoring mode parameters and software operation interface during the analysis.

Selection of ion monitoring mode parameters for 10 organic electrolyte components

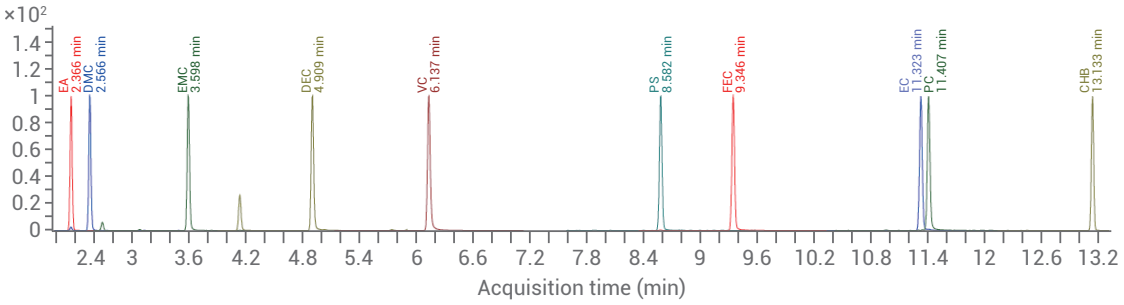
| Compound name | Retention time | Quantifier | Qualifier 1 | Qualifier 2 |
|---|---|---|---|---|
| EA | 2.329 | 88 | 70 | 61 |
| DMC | 2.526 | 90 | 62 | 59 |
| EMC | 3.601 | 77 | 45 | 59 |
| DEC | 4.928 | 91 | 45 | 63.31 |
| VC | 6.141 | 86 | 58 | 42.87 |
| PS | 8.593 | 92 | 58 | 65.57 |
| FEC | 9.363 | 62 | 106 | 43.29 |
| EC | 11.453 | 88 | 58 | 43.29 |
| PC | 11.503 | 87 | 102 | 57.43 |
| CHB | 13.151 | 160 | 117 | 104.91 |




MassHunter Unknowns Analysis Software: result interface of electrolyte sample analysis.




MassHunter Library Editor: information interface of electrolyte analysis database.




TIC chromatograms of 10 organic electrolyte components in "Scan" mode.

# Agilent LC/Q-TOF and GC/Q-TOF in the Lithium-Ion Battery Industry

High quality, high accuracy, and high resolution. Ideal tool to interpret unknowns.

Agilent 6545 LC/Q-TOF

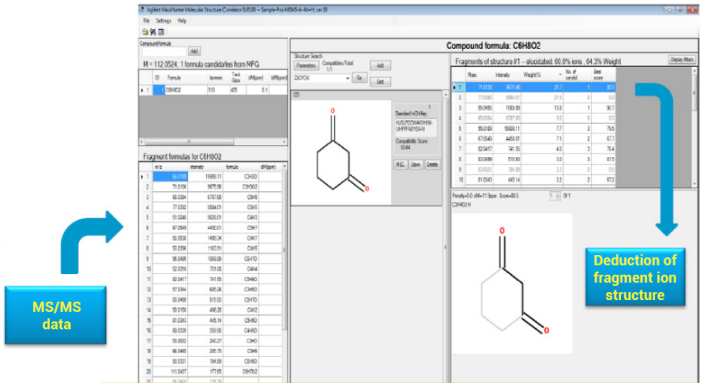
Agilent 7250 GC/Q-TOF

## Assay demands from the industry

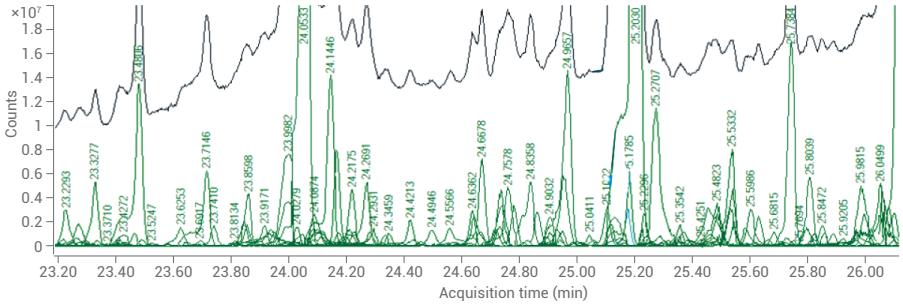
When developing a Li-ion battery, it is necessary to perform a qualitative analysis on unknown organics. For example, in the study of cycle performance, unknown electrolyte compounds introduced by a cell cycle is analyzed given that they may affect the battery's performance. Agilent recommends LC/Q-TOF or GC/Q-TOF for precise qualitative analysis of unknown compounds.

## Application of LC/Q-TOF and GC/Q-TOF

– MassHunter MSC (MS/MS Structure Correlation; software for unknown structure derivation and analysis): For compounds with complex fragment ions yet without secondary mass spectrometry in the database, the software for unknown structure deduction and analysis can be used to infer the structure of unknown compounds.




– MassHunter MFE (molecular feature extraction) for molecular information extraction: A molecular feature extraction function (MFE) specially developed by LC/Q-TOF data features; can automatically and quickly sort out all compounds from the spectrum, and identify them by precise mass, isotope information, accurate secondary mass spectrometry and structure-assisted interpretation software.




– Mass Profiler Professional (MPP; statistical analysis software for MS data): Being compatible with GC/Q-TOF, LC /Q-TOF, ICP-MS and other mass spectrometry product data, the software can analyze all components in the sample, along with the significance of the difference using principal component analysis (PCA), unsupervised cluster analysis, analysis of variance, Venn diagram and other statistical analysis algorithms.

– Q-TOF Database and Library: Use a personal compound database (PCD) and a personal compound database and library (PCDL) for accurate mass search, providing the most comprehensive database and library in the industry.

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