



## Lithium Ion Battery Analysis

PerkinElmer offers application solutions for lithium-ion secondary battery analysis that combine analytical instruments such as FTIR, ICP-OES, GC/MS, and DSC. Demand for lithium ion secondary batteries with high energy density is increasing as new energy. Materials Fee Analysis of battery contents such as positive electrode material, negative electrode material, separator, electrolyte, etc. are regarded as very important in research development and quality control. In addition, in order to improve battery characteristics and safety, it is also necessary to understand the state inside the battery from multiple analysis results.

Atomic absorption spectroscopy and ICP emission spectroscopic analysis are used for the principal component analysis of positive and negative electrodes, qualitative determination of additives etc in R & D and quality control process.

Fourier transform infrared spectroscopic analysis is used to perform impurity management, oxidative degradation analysis, etc. in the constituent materials used for the positive electrode and the negative electrode. For example, it is possible to evaluate the degradation of the separator surface by imaging.

Gas chromatograph mass (AA) spectrometry is used to analyze composition of electrolyte and others. In addition to managing the composition of the electrolyte solution, we also grasp the change in composition after charging and discharging, so that it can be used for deterioration analysis and improvement of safety.

Thermal analysis can evaluate the thermal stability of each material indispensable for battery design. Also, by analyzing the composition of the constituent materials, it is also possible to grasp the state of deterioration.

## Fourier Transform Infrared Analysis (FTIR)

We can qualify organic compound materials, such as binder and separator, used for positive and negative electrodes. The far-infrared region can be used for the qualification of inorganic oxides which are positive electrode active materials. Furthermore, with the IR imaging method, advanced analysis, such as deterioration analysis of the separator surface, is possible.



Frontier Series



Spotlight 400

## Atomic Spectroscopy (ICP-OES, AA)



Avio<sup>™</sup> ICP-OES Series



PinAAcle™ AA Series

From the main component of the positive electrode active material to fine components, such as additives and impurities, it is possible to qualitatively quantify multiple elements in a short time. In addition, direct introduction of organic solvents for analysis enable the checking of the components of positive electrode active materials that have eluted into the electrolyte.

## Gas Chromatography Mass Spectrometry (GC/MS)

It is possible to determine the composition ratio of electrolyte. Changes in composition ratio and decomposition components can be confirmed from the electrolyte solution after charging and discharging. This allow us to study the deterioration of electrolyte. It is also possible to estimate the behavior inside the battery by measuring the gas generated within the battery.



Clarus™ 680 GC



Clarus SQ 8 GC/MS

## Thermal Analysis (TGA, DSC) & Hyphenation



TGA 8000™



DSC 8500™

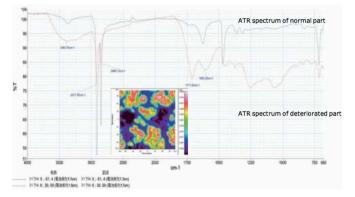
PerkinElmer offers a broad range of thermal analysis techniques for the research, material optimization and quality control of lithium-ion rechargeable batteries.

For example, Thermogravimetric analysis (TGA) instruments determine the thermal decomposition profile of the battery materials under controlled heating. Differential Scanning Calorimetry (DSC) systems allow the study of the melting profile of the battery separator. In addition, PerkinElmer's hyphenated solutions that couple two or more instruments, (for example, TG-IR or TG-IR-GC/MS) greatly enhance the analysis profile and productivity by acquiring information from a single run.



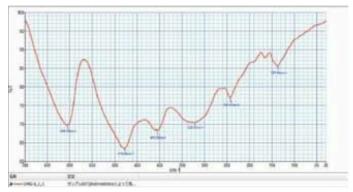
# The Right Solutions For Any Application

Evaluation of separator by FTIR imaging



*Fig1:* Application example of ATR imaging of separator (PE). Evaluation of oxidation degradation can be easily done.

#### Example of positive electrode active material



*Fig2:* Infrared spectrum of the positive electrode material in the far infrared region is shown. By using a single reflection ATR accessory using diamond crystal, inorganic oxide information of positive electrode material can be obtained. It is possible to investigate the qualitative and deteriorated state of the cathode material.

### Application Example: Analysis of the process of cathode materials "out of control heating"



*Fig3:* Thermalgravimetric data of the sample (red is absolute weight loss curve; blue is derivative weight loss curve)

#### Inorganic elemental analysis

From upstream raw materials in the lithium industry to electrode materials in the mid-stream, and the electrolytes use, all require the quality control of impurities to ensure that the metal impurities and main component are within the production control range and do not affect the performance of the battery. PerkinElmer complete inorganic elemental analysis solutions to protect battery quality right from the source.

#### Instrument: Avio 500

Plasma Condition		Eleme	nt wt%
Power	1300 W	Li	7.09
Plasma Gas	10L / min	Co	20.1
Makeup Gas	0.2L / min	Ni	19.8
Nebulizer Gas	0.7L / min	Mn	19.1
Mode	Axial View	Al	0.66
		В	0.077

Example of analysis of element contained in positive electrode active material using ICP emission spectroscopy analyzer. By measuring with both axial and radial view, it is possible to simultaneously measure the main component and minor additives contained in the positive electrode material at the same time in a short time.

#### Application example 1: Determination of impurities in high purity metal raw materials

#### **Avio Series ICP-OES Application Advantages:**

- Avio Series ICP-OES capable of selecting interference-free spectral lines from tens of thousands of spectral lines
- FlatPlatel<sup>™</sup> plasma technology with solid-state RF generator can effectively overcome matrix effect
- High sensitivity to meet the determination of high purity metals
- Standard sampling system with high salt resistance, hydrofluoric acid resistance, and resistance to strong corrosive samples

Cobalt carbonate Unit: mg/kg				
Al	As	Ca	Cd	
76.44	6.07	4.49	2.9	
Cr	Cu	Fe	Li	
7.57	3.34	12.41	0.01	
Mg	Mn	Na	Ni	
1.47	4.93	15.52	1.4	
Pb	Si	Zn		
<2	<1	0.28		

#### Lithium carbonate Unit: mg/kg

		0, 0	
Na	K	Cu	Ni
2.05	0.64	<0.2	<0.2
Mn	Zn	Al	Ca
<0.1	0.3	4.51	1
Fe	Mg	Cr	Pb
<0.2	0.13	<0.2	<0.3
Rb			
<0.2			

С	Cobalt metal Unit: mg/kg				
	Al	As	Ca	Cd	
	20.58	13.74	2.36	0.8	
	Cr	Cu	Fe	Li	
	0.64	5.29	32.68	<0.5	
	Mg	Mn	Na	Ni	
	0.24	0.49	30.19	26.1	
	Pb	Si	Zn		
	<2	<1	1.49		

#### Lithium fluoride Unit: mg/kg

Na	K	Cu	Ni	
6.00	1.40	<0.2	<0.2	
Mn	Zn	Al	Ca	
1.4	0.33	1.74	32	
Fe	Mg	Cr	Pb	
21.4	0.62	<0.2	<0.3	
Rb				
<0.2				

Cobalt oxide Unit: mg/kg				
Al	As	Ca	Cd	
1.17	69.91	7.45	58.01	
Cr	Cu	Fe	Li	
0.64	1140	6151	<0.5	
Mg	Mn	Na	Ni	
2.31	2448	10.53	5827	
Pb	Si	Zn		
58.1	30.15	197.4		

#### Application example 2: Three elemental material (lithium, iron, phosphate) main component composition and impurities

#### **Avio Series ICP-OES Application Advantages:**

- Avio series ICP has 106 dynamic range for accurate determination of high concentration samples
- Excellent stability ensures precision and data stability for the determination of high concentration elements
- High sensitivity to determine the lower level impurity in the sample
- Dual view capability to simultaneously fulfill analytical need for high concentration element as well as low concentration determination of trace impurities

#### Lithium Iron Phosphate Unit: %

Ni	Со	Mn	Li	Al
30.01	12.33	17.2	7.584	0.014
Ti	Zr	Zn	В	Ba
0.172	<0.001	0.052	0.005	< 0.0005

#### Analysis of lithium iron phosphate battery materials

Sample	Fe 238.204	Fe 239.562	Fe 259.939	Average
LiFePO <sub>4</sub> 1	34.314	34.349	34.242	34.302
LiFePO <sub>4</sub> 2	34.753	34.765	34.68	34.733
LiFePO 3	31.115	34.086	34.007	33.069

Sample	P 213.617	P214.914	P178.221	Average
LiFePO <sub>4</sub> 1	19.51	19.393	18.869	19.257
LiFePO <sub>4</sub> 2	19.677	19.54	19.215	19.477
LiFePO <sub>4</sub> 3	19.248	19.738	19.716	19.567

Sample	Li 670.784	Li 610.362	Average
LiFePO₄ 1	4.295	4.449	4.372
LiFePO <sub>4</sub> 2	4.331	4.441	4.386
LiFePO <sub>4</sub> 3	4.276	4.357	4.317

#### **Avio Series ICP-OES Application Advantages:**

- PerkinElmer offers a unique organic sample ICP-OES system for direct injection of various electrolytes
- The organic sample introduction systems can also withstand strong corrosion from fluoride ions and lithium salts

	-				
DMC Elec	DMC Electrolyte: ug/L (ppb)				
Mg	K	Ca	Cr		
<2	<5	54	<5		
Mn	Fe	Cu	Zn		
<5	<5	<5	<5		
Al	Cd	Pb	Ni		
322	<5	<10	6		
Na					
89					

#### Avio Series ICP-OES Application Advantages:

- The high sensitivity of the Avio series ICP-OES ensures a lower detection limit
- PerkinElmer provides a unique pretreatment method for rapid processing of cathode material samples
- Granular nebulizer can be used to analyze samples containing particulate matter

#### Determination of impurities in graphite

20.97	0.22	<0.5	1.12
Al	Мо	Fe	
1.76	<0.5	3.35	



# Technology List by Type of Analysis

### **Research and Development**

	Cathode				Anode			Separator	Electrolyte	
	Cathode Composition			Parts	Anode		Part		Composition	
	Positive	Conductive	Binder	Electrode Sheet	Negative	Binder	Electrode Sheet	Qualitative	Solvent	Electrolyte
FTIR			*			*		*		
AA	•			•			•			
ICP-OES	•			•			•			•
GC/MS									•	
DSC, TG, TG-DTA / DSC	•	•	•		•	•		•		

\* Qualitative in Standard Conditions

### **Quality Control**

		Battery		Raw Materials	Processing		
	Storage Management	Degradation Analysis	Investigation of Impurity	Composition and Impurity Control	Investigation of Impurity Composition	Contamination Study	
FTIR		*	*	*		*	
AA				•	•		
ICP-OES			•	•	•		
GC/MS	•	•		•			
DSC, TG, TG-DTA / DSC		•					

\* Qualitative in Standard Conditions

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