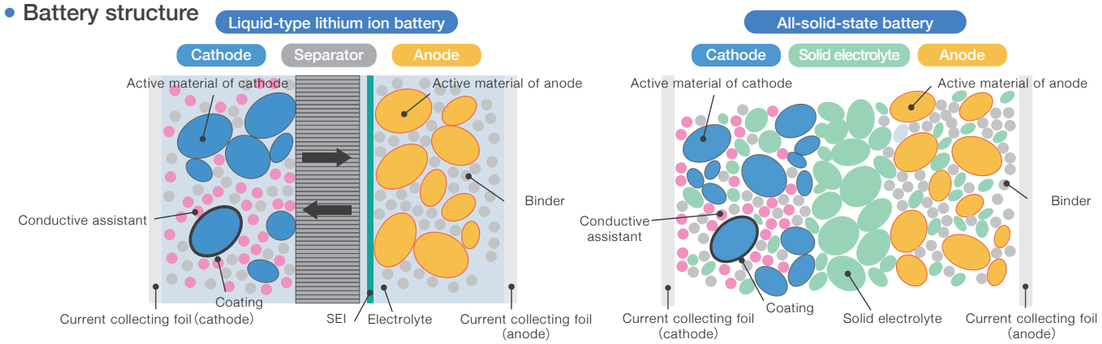
Application Navi

Battery

Lithium ion batteries are widely used in various fields near to our daily lives, and all-solid-state batteries show promise as the next generation of batteries. The research and development of these batteries calls for new analysis and evaluation technologies as scientists set their sights on higher quality and performance. HORIBA provides total analysis and measurement solutions for all processes involved in the production of such battery materials, from research and development, production control, and battery performance evaluation through to support for electric vehicle development. HORIBA can evaluate the performance of a wide variety of batteries, from compact batteries (single cell) to large batteries (battery modules/battery packs).

Research and development support

• Battery structure

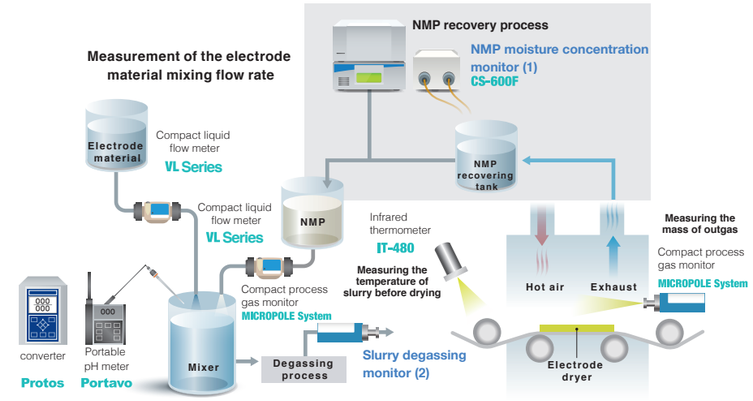


• Battery material evaluation/Causes of deterioration and analysis methods

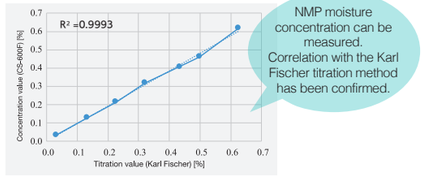
Target			HORIBA analytical systems
Active materials	Cathode	Material distribution evaluation	Raman Spectroscopy, Laser Scattering Particle Size Distribution Analyzer
		Measurement of carbon concentration in materials	Combustion-infrared absorption method
		Crystallinity evaluation of active materials	Raman Spectroscopy
		Observation of active material coating state	Raman Spectroscopy, Particle image analysis
		Elution of manganese ions	ICP-OES
		Stoichiometric abnormality in lithium	Glow Discharge Optical Emission Spectrometry
		Localization and cavities in active materials	Combustion-infrared absorption method, X-ray Fluorescence Analyzer
		Evaluation of carbon crystallinity due to insertion and separation of lithium ions	Raman Spectroscopy
		Foreign material contamination	X-ray Fluorescence Analyzer
		Oxygen deficiency	Inert gas fusion-infrared absorption method
	Anode	Elemental analysis of anode surface (effect of cathode active materials)	X-ray Fluorescence Analyzer
		Elemental analysis of anode depth profile (surface to current collecting foil)	Glow Discharge Optical Emission Spectrometry
		Localization and cavities in active materials and foreign material contamination	X-ray Fluorescence Analyzer
		Decrease in reactivity due to SEI formation	Glow Discharge Optical Emission Spectrometry
		Evaluation of carbon crystallinity due to insertion and separation of lithium ions	Raman Spectroscopy
		Atomization affecting reactivity	Laser Scattering Particle Size Distribution Analyzer
Binder	PVDF	Moisture mixed into NMP	Absorption spectroscopy
	SBR	pH control in the anode slurry adjustment process, Outgas monitoring	pH meter, quadrupole mass spectrometry
Current collecting foil	Cathode：Al	Surface oxidation, corrosion	Glow Discharge Optical Emission Spectrometry ,Raman Spectroscopy
	Anode：Cu	Surface oxidation, corrosion	Glow Discharge Optical Emission Spectrometry, Raman Spectroscopy
Separator	Polyolefin, etc.	Metal foreign material	X-ray Fluorescence Analyzer
		Separator damage caused by particle detachment and burrs	X-ray Fluorescence Analyzer
Solid electrolyte		Lithium distribution in solid electrolytes	Glow Discharge Optical Emission Spectrometry
		Structural changes in solid electrolytes	Raman Spectroscopy

Manufacturing, process analysis, control

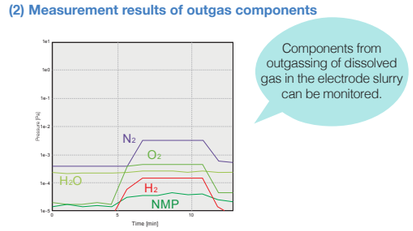
• Measurement of the electrode material mixing flow rate



(1) Measurement results of NMP moisture concentration



(2) Measurement results of outgas components



Battery and electric vehicle development solutions

HORIBA provides a wide range of development support solutions, from evaluation of on-board batteries themselves and vehicle evaluation based on the simulation of actual use of on-board batteries through to battery model development, optimization design, prototyping, and evaluation.

Model development/prototyping

Battery module/pack development support

HORIBA MIRA provides support for the development of efficient, high energy density battery modules and packs according to customers' requests.

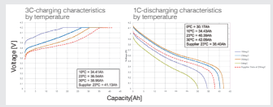
Determining requirements — We hear your requests and determine the requirements of the battery pack to be prototyped.

Battery module/pack design

BMS development and design

Designing a more efficient and optimal battery management system (BMS)

- Modeling and simulation analysis of the battery system
- BMS software design and optimization
- Verification by hardware-in-the-loop simulation (HILS)



Thermal management system design

Developing and designing of integrated thermal management

- Heat load, battery cooling/heating performance
- Analysis of electric power characteristics in the battery drive cycle
- Synthetic analysis of cooling and air conditioning equipment

We also carry out various designing services, including architecture design and battery pack structure design.

Prototyping and evaluation

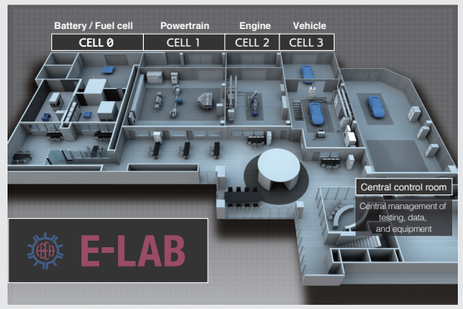
Using advanced engineering skills to create a prototype according to the requirements and design

- Joining of cells/modules
- Assembly of electrical harnesses and temperature sensors
- Assembly of power electronics devices (fuses, connectors)
- Assembly of water cooling/air cooling units, etc.

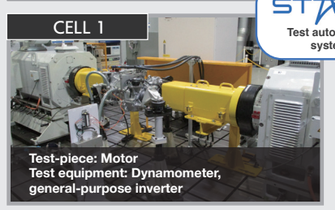
Design Optimization

Evaluation method that contributes to electrified vehicles power train optimization: Test in the Loop™

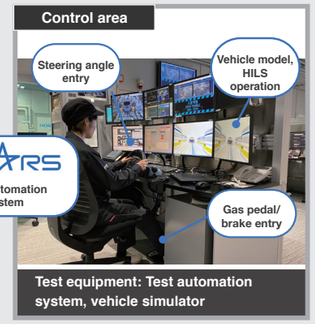
By simulating the vehicle environment using Test in the Loop™, battery performance such as battery charging and discharging characteristics and thermal management can be evaluated in an environment incorporated into the vehicle system.You can evaluate the charging and discharging behavior of an actual battery under the condition in which it is connected to the powertrain while a running road load is imposed upon it as well as evaluate battery performance by changing the ambient temperature.



The E-LAB automobile measurement and test center where HORIBA's advanced solutions can be seen, used, and experienced.



* Test in the Loop is a registered trademark or trademark of HORIBA, Ltd.



Evaluation

Charging and discharging characteristics evaluation

Features

- Support for a power range of up to 1 MW
- Electrical efficiency: 95% or above
- Response speed (t90): Less than 3 ms

The charging and discharging characteristics of driving batteries and stationary storage batteries can be evaluated.We have a lineup of battery test stations that support everything from driving cells, modules, and packs to large stationary storage batteries.

Automotive | Housing | Factory | Power system

Battery test station
Evaluator B

Your Partner in Science

HORIBA Scientific www.horiba.com/scientific

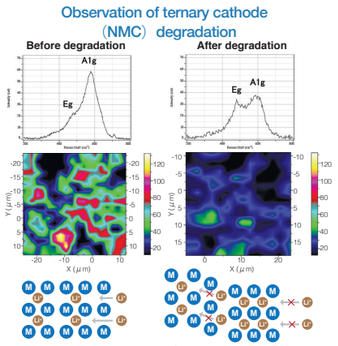
Bulletin: HRE-3699A

Material analysis, deterioration evaluation, quality control

Deterioration analysis

Observation of ternary cathode(NMC) degradation

Raman spectroscopy makes it possible to determine differences between the molecular structures and crystallinity of organic and inorganic compounds and to analyze the crystal structure and molecular structure by a non-destructive, non-contact method while observing the materials through an optical microscope. By Raman spectroscopy imaging, the crystalline structure can be visualized to capture the changes in electrolyte due to deterioration.

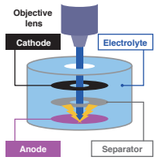


Li-ions are easy to move and diffuse without crystal structural disorder.

Crystal structural disorder prevents Li-ions from moving.

Ultimate Raman Microscope

LabRAM HR Evolution

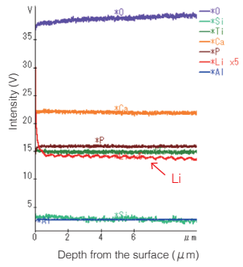


- Cell for *in-situ* analysis

This system can analyze the anode surface of a Li-ion battery in a closed state while charging.

Depth profiling of solid electrolyte in all-solid-state batteries

Depth elemental profiling of lithium distribution in the solid electrolyte revealed segregation of lithium on the solid electrolyte surface. With GD-Profiler 2, you can carry out stable analysis of even easily ionized elements such as lithium with its function for reducing heat damage during measurement.



Pulsed RF Glow Discharce Optical Emission Spectrometer (GD-OES)

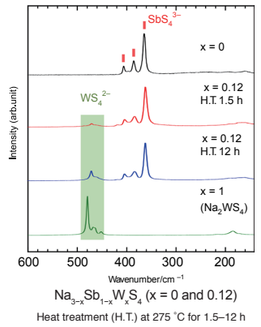
GD-Profiler 2

- Transfer vessel

Our transfer vessel enables the sample to be transferred from the glove box to the device without air exposure.

Analysis of solid electrolyte powder (sulfide-based)

Based on changes in the Raman spectrum of sulfide-based ($Na_{2.88}Sb_{0.88}W_{0.12}S_4$) solid electrolyte, the changes in the structure of electrolyte after 1.5 to 12 hours from applying heat and that WS_4 increases as time progresses could be confirmed.



*The data was provided by Professor Akitoshi Hayashi of Osaka Prefecture University.

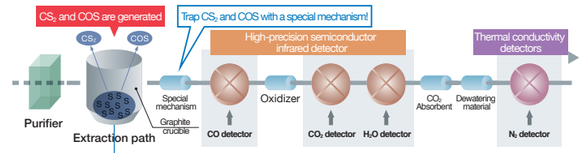
Ultimate Raman Microscope

LabRAM HR Evolution

Material composition analysis

Measurement of oxygen concentration in a solid sample containing sulfur

It was difficult to determine the quantity of oxygen in oxygen analysis of a solid sample containing sulfur due to the reaction between sulfur in the sample and carbon in the graphite crucible. Using EMGA-930 with a sulfur-removal trap, oxygen in a sulfide can be measured.



Gas types

N_2, H_2, CO, CO_2, CS_2, COS

Oxygen/Nitrogen/Hydrogen Analyzer

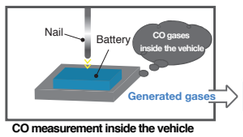
EMGA-930

- Transfer vessel

Our transfer vessel enables measurement without air exposure of samples that react with water, oxygen, and CO_2 in the air and then deteriorate without air exposurer.

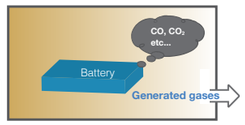
Analysis of gases generated during safety testing

Lithium-ion batteries require a high level of safety, and there are various kinds of safety tests. For example, our analyzer is used during the development stage of vehicles to measure any inflow of CO gases generated from the nail penetration test of lithium ion battery inside the vehicle, thus contributing to safety evaluation. Our analyzer can also measure in real time the concentration of CO and CO_2 gases generated during heat tests of batteries under development.



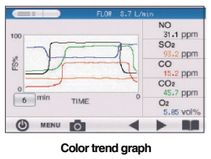
Gas analysis

CO measurement inside the vehicle during the battery nail penetration test



Gas analysis

Battery heat test



Color trend graph

Portable Gas Analyzer

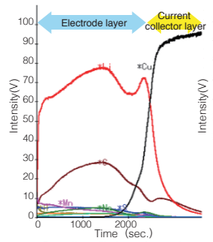
PG-300 Series

Simultaneous measurement of up to five components with one unit

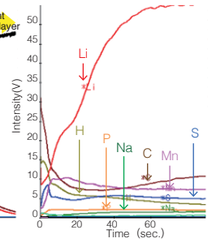
NOx SOx CO CO_2 O_2 CH_4

Analysis of the effect of the eluted active materials of the cathode on the anode

The effect of the eluted active materials of the cathode is considered to be one of the causes of deterioration of the anode. Based on the obtained overall profile from the surface to the current collecting foil and an enlarged profile of elements near the surface, a large quantity of manganese (Mn), an eluted material of the cathode that is a cause of anode surface deterioration, and sulfur (S) estimated to have dissolved from the additive components were detected. On the anode surface, in addition to carbon (C) and lithium (Li), the major components of the solid electrolyte interphase (SEI), a large quantity of Mn that is estimated to have eluted from the cathode and S, which is considered to be an adhesion of the dissolved additive, were detected.



Overall profile from the surface to the current collecting foil

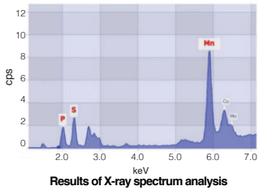


Enlarged profile near the surface

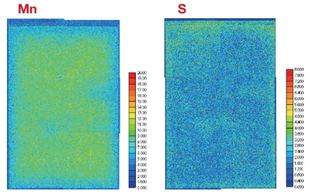
Pulsed RF Glow Discharce Optical Emission Spectrometer (GD-OES)

GD-Profiler 2

Based on the results of X-ray spectrum analysis, Mn, which is considered to have eluted from the cathode, and S, which is considered to be a dissolved component of the additive, were also detected on the anode surface.



Results of X-ray spectrum analysis



Elemental image of the anode surface

Left: It was observed that Mn, which is considered to have eluted from the active materials of the cathode due to battery deterioration, had attached to the entire anode.
Right: It was observed that S, which is predicted to have dissolved from the additive components, had unevenly attached to the electrode terminal section and edge section, which are heated to relatively high temperatures.

X-ray Analytical Microscope

XGT-9000

Controlling the concentrations of carbon and sulfur in the cathode materials

The carbon/sulfur analyzer EMIA-Expert can control the carbon concentration in the cathode materials at the ppm level. It can also accurately control the sulfur concentration, which affects battery life and capacity degradation.

Carbon/Sulfur Analyzer
(Tubular electric resistance heating furnace type)

EMIA-Expert

Sample weight	Carbon(mass %)
0.3059	0.0200
0.3061	0.0201
0.3055	0.0199
0.3057	0.0198
0.3059	0.0197
Average	0.0199
Standard deviation	0.0002
Coefficient of variation(%)	0.79

Reference) Concentration analysis of carbon in $LiCoO_2$

Carbon/Sulfur Analyzer
(High-frequency induction furnace type)

EMIA-Step

The variable heating temperature function makes it possible to perform quantitative analysis by isolating carbon and sulfur by temperature or state in addition to total analysis.

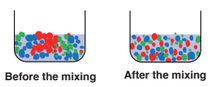
- Auto sampler

Up to 20 samples can be set; automatic setting of the crucible inside the furnace to disposal of the crucible after measurement.

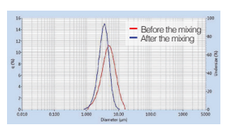
Dispersion

Combined evaluation of cathode materials

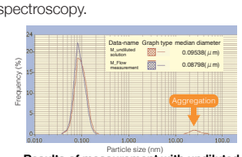
The dispersion state of a slurry sample before and after mixing were compared by particle size distribution measurement using high concentration cells and Raman spectroscopy. With high concentration cells, changes in overall particle size can be observed, while the behavior of each component can be observed on the μm order with Raman spectroscopy.



Before the mixing **After the mixing**



Results of particle size distribution measurement using high concentration cells



Results of measurement with undiluted solution and flow measurement

It was found that there is a difference in particle size distribution depending on the kneading. By using high concentration cells, the transmittance, which has thus far been adjusted by the sample concentration based on the dilution of solvent, can be adjusted by the optical path length, which allows even a high concentration sample to be measured at the minimum dilution rate.

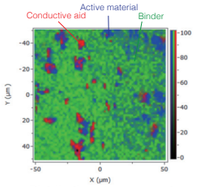
- High concentration cell unit

This unit enables measuring at the original liquid concentration without diluting the sample. Cell units for various sample viscosities are available.

Laser Scattering Particle Size Distribution Analyzer

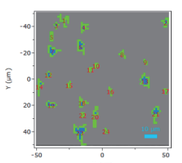
Partica LA-960V2

With Raman spectroscopy, the dispersion state of each component can be visualized. Using the particle analyzing software ParticleFinder enables the particle size to be automatically measured per particle. Thus, comparison of figures before and after mixing becomes possible with this method and software.

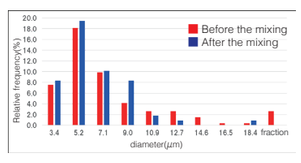


Distribution of components with Raman imaging using the multivariate analysis CLS*

* Classical least square



Example of detecting particles using ParticleFinder



Comparison by a histogram

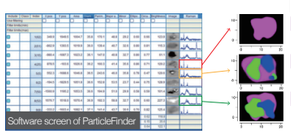
NEW

Raman Imaging System

LabRAM Soleil

Evaluation of battery active material coating

It has become clear that evenly coating electrolyte on the surface of active material particles is effective for improving battery capacity. The resulting coating film is thin and its state differs depending on each particle, so observing such a state is difficult. However, by using Raman spectroscopy and particle analyzing software, Raman measurement for each particle becomes possible. In addition to visualizing each particle's coating state, analysis using the automatic particle recognition function and another type of analysis on the correlation between particle size and shape information can also be conducted.

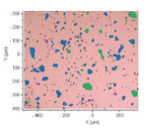


Area occupancy ratios

Base particle	Primary coating	Secondary coating
100%	0%	0%

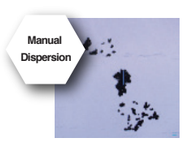
Base particle	Primary coating	Secondary coating
40%	32%	28%

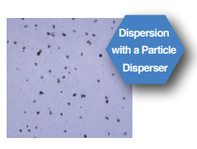
Base particle	Primary coating	Secondary coating
24%	41%	35%



Powder dispersion

The particle disperser makes powder dispersion on the sample stage easy and helps to improve analysis efficiency. Instant powder dispersion is possible by simple operations not subject to human error. By using the particle disperser in conjunction with Raman spectroscopy or X-ray analysis, analysis of the molecular structure and components of each particle becomes possible.



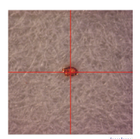


Particle dispersion unit

Particle Disperser

Contamination inspection

Analysis of metal foreign material on separators



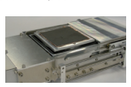
Use of an optical image to set the analysis area

With the X-ray Analytical Microscope XGT-9000, you can detect micro conductive foreign material (metal foreign material) on the separator, which may cause a malfunction due to short circuiting, and carry out an analysis of its elemental composition. Foreign material can be quickly detected by optical image observation, high-speed screening, and highlighting through image processing.

Expanded mapping analysis

- Transfer vessel

Our transfer vessel controls the environment between the glove box and the Instrument, enabling analysis without air exposure.



Point analysis by Ø10 μm X-ray

X-ray Analytical Microscope

XGT-9000