



# **Quantitative Measurement in Lithium Manganate**

### **Background / Challenges**

#### Background

With the increasing demand for lithium-ion batteries, various compounds have been investigated as cathode materials. Typical examples include lithium cobaltite and lithium manganate, and the amount of oxygen deficiency is important as one of the items to evaluate the material properties. This is because deficient oxygen has an important influence on battery performance.

#### Challenges

A very high quantitative system is required to see oxygen deficiency in actual samples. Also, since the sample is a powder, the effect of atmospheric oxygen can cause a large measurement error. But there are a few instruments in the market that can do quantitative analysis as oxygen atoms with high accuracy.

> EMGA-Pro/Expert O/N/H Analvzer



Solution from HORIBA

#### **Testimonial from a** material manufacturer

"We do not want to require special pretreatment or advanced analytical skills. We also need to measure a large number of samples, so we want to analyze them easily and accurately .. "

By sealing the sample in a Ni capsule using a special jig and using a transfer vessel to reduce atmospheric effects, a very high accuracy quantitative analysis was possible, and the difference in valence could be confirmed from the analysis results.

Capsule press jig



	Oxygen(%)	Oxygen Valence
Sample A	35.29	3.99
Sample B	35.94	3.95
Theoretical value	35.39	4.00

Transfer vessel

Result of oxygen

### Application: The oxygen concentration in the cathode materials

As one of the parameters to evaluate the material properties of lithium cobaltite and lithium manganate, it is important to understand the oxygen content and the oxygen release characteristics under temperature conditions.

Composition	LiCoO <sub>2</sub>	LiMn <sub>2</sub> O <sub>4</sub>	Li(Ni-Mn-Co)O <sub>2</sub>	
Theoretical value (mass%)	32.7	35.4	32.9	
Oxygen value (mass%)	32.4	35.6	32.5	
	32.3	35.2	32.5	
	32.4	35.4	32.7	
	32.4	35.2	32.9	
	32.2	35.6	32.8	
Average value	32.3	35.4	32.7	

Result of oxygen content in each material

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0.2 LiMn204 Concentration of Oxygen (mass%) 0.10 0.00 0 .... Li(Ni,Co,Mn)O2 LiFePO4 500 1500 2000 1000 Temperature (℃) Relationship between extraction curve and temperature for each material

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### Application: Carbon analysis in cathode material

The EMIA-Expert can help to measure the carbon concentration in the cathode materials at ppm level. It can also accurately measure sulfur concentration, which affects battery lifetime and capacity degradation.

Analysis time is as short as approximately 1 minute, enabling highly accurate quantitative analysis with a small sample volume.



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(%)	0.7900	

Result of carbon content in LiCoO2

### Application: Purity analysis in material

It helps to control the concentration of carbon/sulfur/oxygen as an impurity in cathode materials at the ppm level. Analysis time is as short as approximately 1 minute, enabling highly accurate quantitative analysis with a small sample volume.



EMGA-Pro/Expert		EMIA-Pro/Expert		
Sample: Copper		Sample: Copper		
Sample weight (g)	Oxygen (ppm)	Sample weight (g)	Carbon (weight %)	Sulfur (weight %)
1.0190	0.00047	0.7127	0.00842	0.00351
1.0230	0.00048	0.7248	0.00880	0.00364
1.0220	0.00051	0.6932	0.00820	0.00352
Average	0.00049	Average	0.00847	0.00356

This data is an image only, as actual data cannot be released due to confidentiality obligations.

## Application: Ultra trace sulfur analysis

With the demand for materials of high purity, the analysis of sulfur in ultra-trace amounts is a very difficult analysis. The ability to trap (collect) and extract gases makes analysis of ultra-trace amounts of sulfur possible.





### The Auto sampler for the EMIA/EMGA series

#### ✓ Maximized accuracy and reproducibility

Minimizes human errors and ambient dust contamination, resulting in precise and consistent results.

#### ✓ Enhanced safety

Standard equipment with safety covers to reduce risk of injury and sample contamination

# ✓ Improved productivity and reduction of operator workload

It facilitates more efficient operation and sample handling, resulting in reduced processing time and increased sample throughput.



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