Application Note

Moisture of electrolyte for lithium-ion battery

Industry	:	Energy
Instrument	:	Karl Fischer Moisture Titrator
Measurement method	:	Coulometric titration / Direct method
Standards	:	

1. Scope

Lithium hexafluorophosphate ($LiPF_6$) is the most popular electrolyte in lithium-ion batteries electrolytic solution. Lithium hexafluorophosphate reacts with moisture to produce hydrofluoric acid. If moisture is present in the electrolyte, its deterioration will accelerate. Therefore, it is important to measure the moisture content of the electrolyte when considering the long life of batteries and quality control.
This application introduces an example of moisture measurement of a lithium-ion battery electrolyte containing lithium hexafluorophosphate. In this case, methanol-free reagent were used, since methanol-based reagent would cause interference due to side reactions.

2. Precautions

Test operation must be performed in the hood to eliminate the risk of organic solvents.
It is recommended to use an electronic balance that can measure up to the order of 0.1 mg.

3. Post-measurement procedure

After the measurement, hydrofluoric acid generated by the hydrolysis of lithium hexafluorophosphate causes corrosion of electrodes and flasks, and precipitation of insoluble materials. To prevent this, after the measurement, drain the reagent and wash the titration flask, twin platinum electrode and electrolytic electrode with a solvent such as toluene.

4. Apparatus

Main unit : Karl Fischer Moisture Titrator (for coulometric method)
Electrode : Twin platinum electrode, 2Component Inner Burette

5. Reagents

・KEMAQUA Anolyte AKE
・KEMAQUA Catholyte CGE

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6. Procedure

-Preparation-

1) Prepare approximately 100mL anolyte in titration cell.
2) Put approximately 5mL catholyte in the inner cell.
3) Dehydrate the measuring cell by performing pre-titration in advance.

-Measurement-

1) Take the sample using a syringe and measure the mass.
2) Inject the sample to titrating cell and measure the moisture content.
3) Measure the mass of the syringe after sample injection.
4) The difference in the mass of the syringe before and after sample injection is the sample amount.

7. Calculation

Moisture content (ppm) = (Data－Drift×t－Blank)/(Wt1－Wt2)×F×1

Data	･･･Total water content after electrolysis in the titration cell (μg)
Drift	･･･Drift level which changes by ambient moisture and carrier gas permeating into the titration cell (μg/s)
Blank	･･･Blank level (0μg)
t	･･･Titration time length from start to the end of titration after sample is discharged. (s)
Wt1	･･･The total weight of sample and sampler before sample is discharged. (g)
Wt2	･･･The total weight of sampler and sample residue after sample is discharged. (g)
F	･･･Compensation coefficient for calculation results (1.0)

8. Example

-Parameter of titration-

<Titration parameter>

Titration mode	: H2O
t(stir)	: 0s
t(wait)	: 15s
t(max)	: 0s
Drift stop	: Rel.
Rel.	: 0.1μg/s

<Control parameter>

Cell type	: 2-Comp.
Stable	: 0.1μg/s
Control gain	: 5.0
Electrolysis speed	: standard
End level	: 200mV
Start mode	: Auto
Data sampling time	: 5s
Stirrer speed	: 3

(The above condition is an example. The setting condition depends on the titrator model.)

—Titration curve—

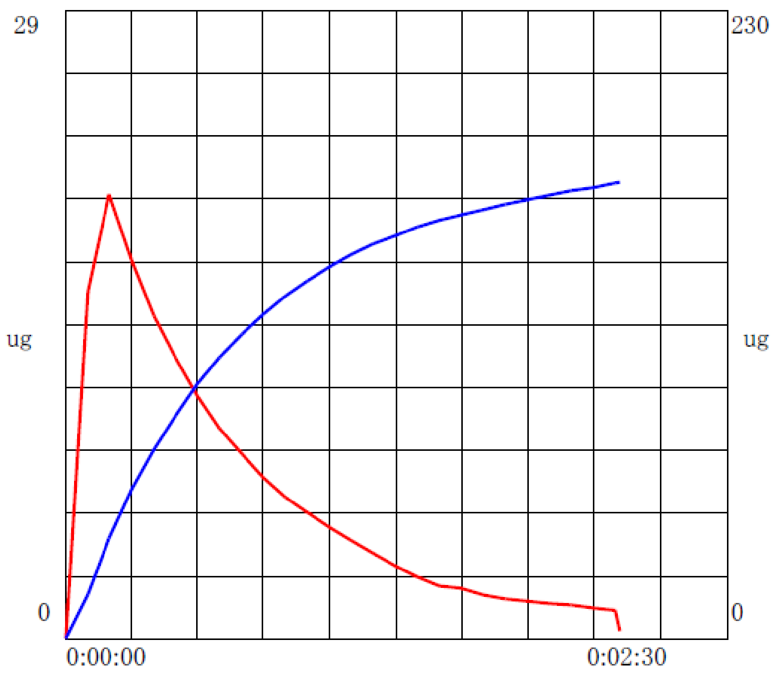

Table 1 The measurement result

	Sample(g)	Moisture(μg)	Water content(ppm)
1	3.9166	144.1	36.79
2	3.7292	133.6	35.83
3	3.9114	146.1	37.35
Mean	-	-	36.66
SD	-	-	0.77
RSD(%)	-	-	2.11

9. Summary

The RSD value (relative standard deviation) was less than 3%, and good repeatability was obtained. Karl Fischer Moisture Titrator can be used to measure the moisture content of electrolytes for lithium-ion batteries.