

Water content measurement using Karl Fischer method, TPD-MS and GC

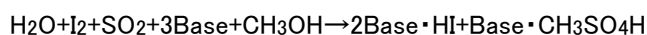
To obtain a reliable water content, Karl Fischer(KF) method, Temperature Programmed Desorption – Mass Spectroscopy(TPD-MS) and Gas chromatography(GC) can be applied. It is important to select an appropriate method depending on sample features (e.g. size, composition or state).

Comparison between representative methods

Method	Range of concentration	Appropriate sample/ Feature	Unsuitable sample/ Restriction
KF	•wtppm~wt%	•Liquid and solid (e.g. solvent, oil) / •Selectivity for water	•Samples producing side reactions(e.g. aldehyde)/ •Several grams of sample is necessary for a microdetermination
TPD-MS	•Hundreds of wtppb ~wt%	•Solid (e.g. Thin-layer of solid) / •Monitorable for other components simultaneously •Monitorable for a gas evolution behavior	•Liquid •Resin causing a generation of multiple organic gases during thermal decomposition
GC	•Tens of wtppm ~wt%	•Liquid and solid / •Substitute method for KF (also applicable to unsuitable samples for KF) •Monitorable for other components simultaneously	•Multiple components inseparable by GC •Insoluble resin

KF method

Water reacts with I₂ and SO₂ quantitatively, under the presence of alcohol and base. The water content is calculated from the loss of the I₂ in the reaction below.



- ✓ Absolute content determination (μg level~)
- ✓ Suitability for use as a reference method



[Vaporizing method]
Insoluble sample is injected into vaporizer. Generated water is blown into anolyte with dry N₂ gas.

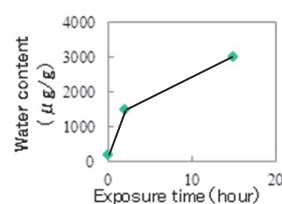
Soluble in anolyte ?	Yes	Side reactions occur ?
No	No	Yes
Vaporizing method	Direct injection	Several solution
		(Use of a special reagent/ Neutralization)

【Applicable samples】

Industrial product/ Food/ Drug/ Mineral

Measurement of electrolyte for Li-ion battery

7mL of Electrolyte (Diethylcarbonate/Storage under an inert gas) was put into a 10mL vial. After 2 or 15 hours of air exposure at Lab. (Temp.25°C/ RH50%), the KF titration was performed for samples.

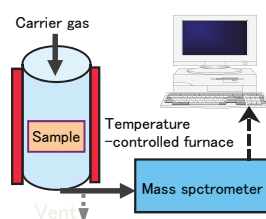


Exposure time	N	Determined water(μg/g)	
		Mesurement	Mean
0 hours	1	186	190
	2	192	
2 hours	1	1522	1500
	2	1528	
15hours	1	3019	3000
	2	3037	

with high accuracy :

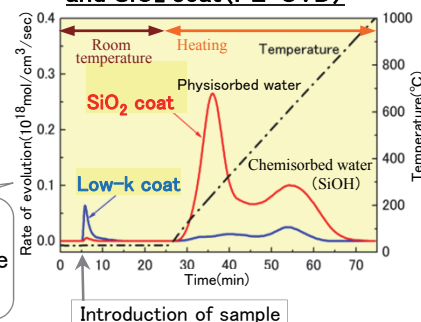
TPD-MS

The TPD-MS is used to determine the amount of gas desorbed from the sample as a function of heating time or temperature.



Physisorbed water and chemisorbed water were observed, respectively.

Measurement of Low-k coat and SiO₂ coat (PE-CVD)

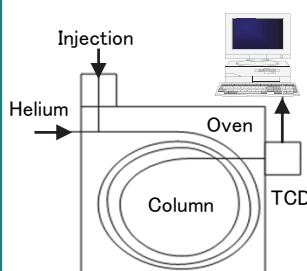


【Applicable samples】

PI coat on Si Substrate/ Ceramic insulator/Color filter/ Resin/ Optical material/ Catalyst

GC

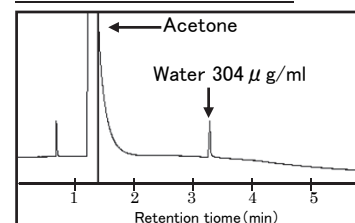
Water is separated through a column and detected with TCD (Thermal Conductivity Detector). Water content is estimated from the peak area.



【Applicable samples】

Solvent/ Resin

Measurement of acetone



The peak of water is clearly distinguished from that of acetone.