KEM **Application Note** №KVX-01352

Inorganic Chemical

Water Content of Metal Oxides

Volumetric titration (Evaporation Method) by
Karl Fischer Moisture Titrator

Standard	JIS	K 0113		
	ASTM	E 203		
	ISO	760		

1.Abstract

Moisture titration using Karl Fischer reagent is popularly practiced water determination worldwide as the most reliable method. The procedure is adopted in many official standards as test method specified in ISO, ASTM, DIN, BS and JIS.

Metal oxides are hard to dissolve in Karl Fischer solvent with side reaction, and therefore, the indirect method using an oven to evaporate moisture in sample is generally practiced. The test sample is first heated in the oven, and the evaporated moisture is transferred to solvent by carrier gas where moisture titration is performed according to JIS K 0113-2005 Standard Test Method by Potentiometric, Amperometrtic, Coulometric and Karl Fischer Titration

For indirect method, the extracting solvent ME from Riedel de Haen is used.

Test samples measured this time are as follows:
Titanium dioxide, Ferrite oxide (Colcothar)

2.Reference

1) JIS K 0113-2005: Standard Test Method by Potentiometric, Amperometrtic, Coulometric and Karl Fischer Titration
2) ASTM E 203-16 Standard Test Method for Water Using Volumetric Karl Fischer Titration
3) ISO 760:1978 Determination of Water-Karl Fischer method (General method)
4) Hydranal manual from Riedel de Haen

3.Cautions in measurement

1) In order to refrain from the effect of ambient humidity, the test must be conducted in a well air-conditioned room.
2) Since water coexistence varies from test sample, select sample size and oven temperature appropriate to each sample piece.
3) Obtain the factor of Karl Fischer reagent using the solvent in advance.

4. Post-measurement care

After the reagent in flask is drained out and the electrode is cleaned, keep the electrode in titration flask filled with extracting medium.

5.Test equipment

Main unit : Karl Fischer moisture titration volumetric system
Electrode : Twin platinum electrode for KF titration
Option : Water evaporator

6.Reagent

Reagent : Hydranal Composit 2 (Riedel de Haen)
Solvent : Extracting medium ME (for gas) (Hayashi Chemicals)

7.Measurement procedure

-Preparation-

1) Prepare approximately 50mL ME solvent in the titration cell.
2) Dehydrate the measuring cell by performing pretitration in advance.
3) Set the oven to a temperature appropriate to the sample and maintain the temperature.
4) Purge the evaporating system with carrier gas.

—Measurement—

1) Press Start key of oven. (Start back purge and cell purge)
2) Take approximately 1g sample with sampler.
3) Weigh the sampler on balance of which resolution is to the nearest 0.1mg.
4) After cell purge, discharge the sampler with sample in it onto the sample boat through sample inlet of heating unit.
5) Press Start key to move the boat into the oven. Again press Start key of titrator to start titration.
6) Weigh the sampler after the sample is discharged.
7) Enter Wt1 with the weight of above 3),and Wt2 with 6).
8) After titration, obtain water content from titration volume.

8.Formula

Water content (%) = ((Data × F − Blank) / (Wt1 − Wt2)) × 0.1

Data : Titration volume (mL)
F : Factor of titrant(mg H_2O / mL)
Blank : Blank level (mg)
Wt1 : Sample + sampler (g)
Wt2 : Weight of empty sampler (g)

9. Example of measurement

-Titration parameter-

MKV-710M/S,MKA-610	MKA-520	MKS-500
Method No. 1 [Titration] Titr.mode Normal t(stir) 0 s t(wait) 10 s t(max) 1200 s t(interval) 0 s Max.volume 10 mL Titr.bur.No. 1 Dose mode Off [Control] End time 0 s Final vol. 0.01 mL Titr.speed 3 Detect.mode 1 Drift titr. On Start mode Manual End level 75 mV Samp.time 5 s Stir.speed 4 [Option] Pre treat 2 Cell purge 120 s Back purge 180 s Heat.mode Set Oven temp. See attached	[Titration] Method 1 Titr Mode Normal Titr Buret No. 1 End Time 0 s Final Vol. 0.01 mL Titr.Speed 3 Detector Mode 1 t(stir) 0 s t(wait) 10 s t(max) 1200 s Drift Titr On Start Manual Max.Volume 10 mL Dose mode Off Oven ADP- Oven Temp. refer to other paper Pre Treat 2 Back Purge 180 s Cell Purge 120 s	[Titration] Method Direct Titr.Speed 3 End Time 0 s Final Vol. 0.01 mL Detector Mode Normal t(stir) 0 s t(max) 1200 s Drift Titr. On Max.Volume 10 mL

-Calculation parameter-

MKV-710M/S,MKA-610	MKA-520	MKS-500
[Calculation] Calc.type Sample Blank No. 1 Calc.No. 2 Unit % Decimal 2 Fraction Round (Half adjust) Drift comp. Off Evalution Off	[Calculation] Calc. 2 Unit % Weight Variable	[Calculation] g->%

Evaporator

Flow rate 200mL/min	Flow rate 200mL/min	Flow rate 200mL/min Oven temp. See attached

－Measurement results－

Sample name	Sample (g)	Solvent	Oven (℃)	Water content	
				mg	%
Titanium dioxide	0.997	ME	300	0.76	0.08
Ferrite Colcothar)	0.997	ME	300	1.55	0.16

10.Summary

Metal oxides change in variety by elementary composition and crystal structure, and its features are revealed in usefulness of insulation, semi-conductor, electric conductivity like metal, ferroelectricity, piezo and ferromagnetism.

The sample test has been successfully conducted by using the evaporator. Moisture from sample is delivered to solvent ME in titration cell by carrier gas. Precise and reliable water content can be obtained by Karl Fischer moisture titration system.

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