KEM **Application Note** №KVX-01551

Mineral Ore

Moisture of Mineral Ore

Volumetric titration (Evaporation Method) by
Karl Fischer Moisture Titrator

Standard	JIS	M 8211	ISO	7335	
	JIS	K 0113	ISO	760	
	ASTM	E 203			

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.

Mineral ores are hard to dissolve in Karl Fischer solvent, 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 measuring cell by carrier gas where moisture titration is performed according to JIS M 8211-1995 Standard Test Method by Karl Fischer titration.

The combine water termed in JIS means water evaporation from iron ore between 105°C and 950°C, however, the oven temperature is adjusted appropriate to the sample in present test.

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

Test samples measured this time are as follows:

Iron ore, Manganese ore, Rock, Kaolin, Hornblende, Black mica

2.Reference

1) JIS M 8211-1995 Test Method for Iron ore －Combine water
2) JIS K 0113-2005: Standard Test Method by Potentiometric, Amperometrtic, Coulometric and Karl Fischer Titration
3) ASTM E 203-16 Standard Test Method for Water Using Volumetric Karl Fischer Titration
4) ISO 760:1978 Determination of Water-Karl Fischer method (General method)
5) ISO 7335:1987 Iron ores -- Determination of combined water content -- Karl Fischer titrimetric method
6) 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 (for high heat, mineral ore)

6.Reagent

Reagent : Hydranal Composit 5 (Riedel de Haen)
Solvent : Extracting medium ME (for gas) (Hayashi Chemicals)

7.Measurement procedure

The following test proceeds regardless of water of adhesion or combine water in sample.

-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) Start back purge for 180 seconds and cell purge for 120 seconds manually.
2) Take approximately 0.1～1g sample with sampler.
3) Weigh the sampler on balance of which resolution is to the nearest 0.1mg.
4) After cell purge, press Start key of titrator.
5) Transfer the sample onto the boat in heating unit through sample inlet.
6) Press Start key to move the boat into the oven. Again press Start key of titrator to start titration.
7) Weigh the sampler after the sample is discharged.
8) Enter Wt1 with the weight of above 3),and Wt2 with 7).
9) 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) 1800 s t(interval) 0 s Max.volume 10 mL Titr.bur.No. 1 Dose mode Off [Control] End time 30 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	[Titration] Method 1 Titr Mode Normal Titr Buret No. 1 End Time 30 s Final Vol. 0.01 mL Titr.Speed 3 Detector Mode 1 t(stir) 0 s t(wait) 10 s t(max) 1800 s Drift Titr On Start Manual Max.Volume 10 mL Dose mode Off Oven Off	[Titration] Method Direct Titr.Speed 3 End Time 30 s Final Vol. 0.01 mL Detector Mode Normal t(stir) 0 s t(max) 1800 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 Evaluation Off	[Calculation] Calc. 2 Unit % Weight Variable	[Calculation] g->%

Evaporator(for high heat, mineral ore)

Flow rate 200mL/min Temp. (high temp furnace): See attached Temp. (Low temp furnace): 105°C	Flow rate 200mL/min Temp. (high temp furnace): See attached Temp. (Low temp furnace): 105°C	Flow rate 200mL/min Temp. (high temp furnace): See attached Temp. (Low temp furnace): 105°C

－Measurement results－

Sample name	Sample (g)	Solvent	Oven (℃)	Water content	
				mg	%
Iron ore (Indian ore)	0.5638	ME	750	13.58	2.41
Manganese ore	0.2313	ME	750	24.29	10.50
Rock	0.4112	ME	700	22.04	5.36
Kaolin	0.1414	ME	800	19.94	14.10
Hornblende	1.0283	ME	1000	16.04	1.56
Black mica	1.1005	ME	1000	25.75	2.34

10.Summary

Mineral ores are rocks and sands which contain natural resources useful for human economic activities. The sample test has been successfully conducted by using the evaporator. The moisture from the 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. In order to separate water of adhesion and combine water, it is recommended to use an oven with two heat ranges: 50～130°C heat zone, and 50～1000°C overheat zone.

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