

Inorganic
Chemical

Water Content of Salts

Volumetric titration (Direct 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.

The test conducted this time is an example of volumetric moisture titration according to JIS K 0113-2005 for measurement of water content in various kinds of salt, which exists as crystal water·inclusion water·adhesion water and dissolves in MI or ML (for general purpose) extracting medium.

The following salts are measured by this method:

Sodium acetate, Ammonium oxalate, Mono-ammonium phosphate,
Di-ammonium phosphate, Lithium perchlorate, Ammonium nitrate,
Calcium sulfate, Potassium sulfate, Magnesium sulfate,
p-sodium nitrophenyl phosphate

2. Reference

- 1) JIS K 0113-2005: Standard Test Method by Potentiometric, Amperometric, 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 published by 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) Water content of salt differs from each one. Change sample size accordingly.
- 3) For low water content, use Karl Fischer reagent of low factor, and for high water content, use high factor.
- 4) Obtain the factor of Karl Fischer reagent using the solvent in advance.

4. Post-measurement care

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

5. Test equipment

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

6. Reagent

Titrant : Hydranal Composit 2 and 5 (Riedel de Haen)
Solvent : Extracting medium MI (for general purpose) (Hayashi Chemicals)

7. Measurement procedure

-Pretreatment-

- 1) Prepare approximately 30mL extracting medium in the titration cell.
- 2) Dehydrate the measuring cell by performing pre-titration in advance.

-Measurement-

- 1) Prepare approximately 0.1~3g sample in sampler.
- 2) Weigh the sampler on an electronic balance of which precision is to the nearest 0.1mg.
- 3) Transfer the sample in sampler to titration cell to dissolve.
- 4) Press Start key of titration unit.
- 5) Weigh the sampler of the above 3).
- 6) Enter the weight of item 2) for Wt1, and 5) for Wt2.
- 7) Obtain water content from titration volume, of which EP is detected automatically.

8. Formula

$$\text{Moisture (\%)} = ((\text{Data} \times F - \text{Blank}) / (\text{Wt1} - \text{Wt2})) \times 0.1$$

Data : Titration volume (mL)
F : Factor of titrant (mg H₂O / mL)
Blank : Blank level (mg)
Wt1 : Sample + Sampler (g)
Wt2 : 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) 0 s t(interval) 0 s Max.volume 20 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) 0 s Drift Titr On Start Manual Max.Volume 20 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) 0 s Drift Titr. On Max.Volume 20 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->%

–Measurement results–

Sample name	Water content	
	mg	%
Sodium acetate	48.85	38.77
1-ammonium phosphate	1.32	0.41
Lithium perchlorate	2.01	0.25
Potassium sulfate	5.73	1.19
p-sodium nitrophenyl phosphate	28.58	28.30

Sample name	Water content	
	mg	%
Ammonium oxalate	12.21	12.33
2-ammonium phosphate	2.22	1.55
Ammonium nitrate	15.29	0.43
Magnesium sulfate	63.93	51.56

Solvent MI was used for extracting medium.

10.Summary

Salt is an ionic compound by neutralization of acid and chlorine, and most salts are ionic crystals soluble in water. Typical cooking salt NaCl is made by reaction of Hydrogen chloride HCl and Sodium hydroxide NaOH.

The test sample in this application dissolves in solvent MI, which makes moisture titration performed with ease.

Stable measurement of water content is assured by Karl Fischer moisture titration.