Application Note

№KVX-01517

Food

Water Content of Starch and Protein

Volumetric titration (Evaporatation 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 for measurement of water content of starch・protein processed foods. This type of food samples are often hard to dissolve in KF 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.

The test samples measured this time are as follows:
Fry oil, Corn starch, Potato starch, Corn, Gluten food, Gluten meal, Cheese

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) Handle with care when you work on test sample.
3) For high water content of sample, minimize sample size and use KF reagent of higher factor.
4) 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 titration flask with extracting medium in it.

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 5 (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 pre-titration 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 0.1～0.3g 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 20 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 20 mL Dose mode Off Oven ADP- Oven Temp. see data 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 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->%

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	%
Fry oil	0.100	ME	150	12.72	12.72
Corn starch	0.238	ME	150	30.68	12.89
Potato starch	0.183	ME	150	32.36	17.68
Corn	0.241	ME	180	33.49	13.90
Gluten food	0.282	ME	130	28.06	9.95
Gluten meal	0.234	ME	150	21.09	9.02
Cheese	0.151	ME	200	63.29	41.92

10.Summary

The purpose of food is to take in nourishment as well as enjoy taste. Starch is a Carbohydrate (Polysaccharide) of chemical formula $(C_6H_{10}O_5)n$, which is a natural polymer polymerized by glycoside linkage of a number of α-Glucose molecules.

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.

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