

Plastic, Rubber

Moisture of Resin Pellet

Coulometric Titration (Evaporation Method) by
Karl Fischer Moisture Titrator

Standard	JIS	K 0113	ASTM	D 1533
	JIS	K 0068	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.

Pellet is generally hard to dissolve in KF solvent, and therefore, the 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 the cell by carrier gas where moisture titration is performed.

2. Reference

- 1) JIS K 0113-2005 Standard Test Method by Potentiometric, Amperometric, Coulometric and Karl Fischer Titration
- 2) JIS K 0068-2001 Test Method for Water Content in Chemical Products
- 3) ISO 760:1978 Determination of Water-Karl Fischer method (General method)
- 4) ASTM D 1533-12 Standard Test Method for Water in Insulating Liquids by Coulometric Karl Fischer Titration

3. Cautions in measurement

- 1) The test must be conducted in an air conditioned room to avoid permeation of ambient moisture.
- 2) Since water factor differs from sample type, select sample size and heating temperature appropriate to the test sample.
- 3) The resolution of mass balance is desirable to the nearest 0.01mg.
- 4) Use nitrogen gas for carrier gas with minimum water content.

4. Post-measurement care

Drain out residual reagent in titration cell and inner cell, and rinse them with alcohol. The electrode and sample boat also need cleaning with alcohol.

5. Test equipment

Main unit : Karl Fischer moisture titration coulometric system
Electrode : Electrolysis electrode
 Twin platinum electrode for KF titration
Option : KF oven

6. Reagent

Annolyte : KEMAQUA AGE
Catholyte : KEMAQUA CGE
Carrier gas : Nitrogen gas (99.99%)

7. Test procedure

-Pretreatment-

- 1) Prepare approximately 150mL annolyte in titration cell.
- 2) Inject approximately 5mL catholyte in the inner cell.
- 3) Dehydrate the measuring cell by performing pre-titration in advance.
- 4) Connect the oven and set heating temperature 220°C.
- 5) Purge dry tube lines and heating unit using nitrogen gas.

-Measurement-

- 1) Set "Pre treat 2" at [Option parameter] in APD-611, and then perform back purge for 3 minutes and cell purge for 2 minutes.
- 2) Put 1g of sample with finger type sampler onto sample boat, insert sample boat to heating unit, and start titration to measure moisture of pellet.
- 3) Weigh mass of finger type sampler before and after putting sample on, and mass difference is assumed to be sample size.

8. Formula

$$\text{Moisture (ppm)} = F \times (\text{Moisture} / (\text{Wt1} - \text{Wt2}))$$

F : Factor (1)

Wt1 : Sample + sampler (g)

Wt2 : Mass of empty sampler (g)

Moisture : Water content (Data – Drift × t – Blank) (μg)

Data : Total moisture (μg)

Drift : Drift level (μg/s)

t : Measuring time (s)

Blank : Blank level (0.00 μg)

9. Example of measurement

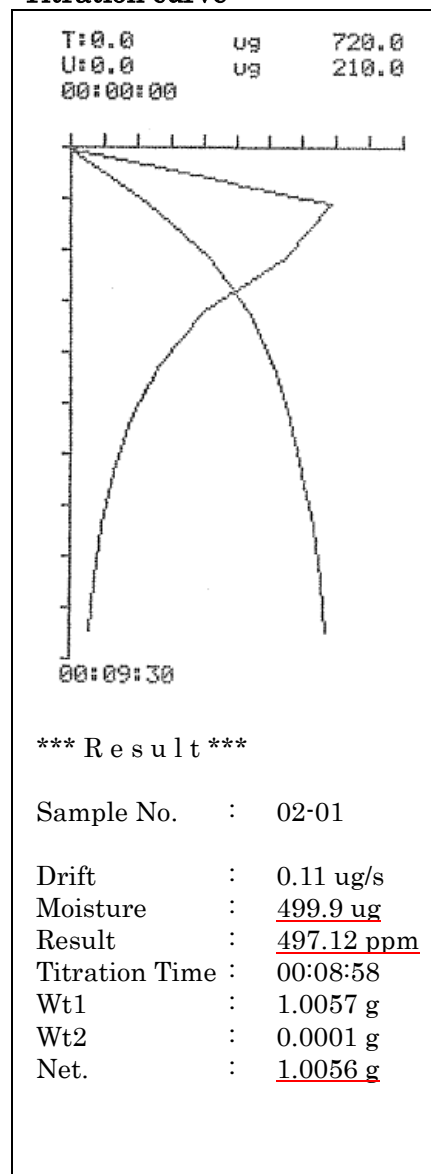
— Ambient condition —

Room temperature : 19 °C	Humidity : 39 %	Weather : Fair
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-Titration parameter-

Method No. : 06	
[Titration]	[Calculation]
Titration Mode : H2O	Calc.Type : Sample
t(stir) : 0 s	Blank No. : 1
t(wait) : 15 s	Calc.No. : 2
t(max) : 1185 s	Unit : ppm
Drift stop : Rel.	Decimal : 2
Drift : 0.10 ug/s	Fraction : Half adjust
	Drift Comp. : Auto
	Evaluation : Off
[Control]	[Option]
Cell Type : 2-comp	Pre Treat : 2
Stable : 0.1 ug/min	Cell Purge : 120 s
Ctrl.Gain : 5.0	Back Purge : 180 s
E.Mode : Standard	Heat.Mode : Set
Start Mode : Auto	Oven Temp. : 220°C
End Level : 200 mV	
Samp.Time : 60 s	
Stirrer : 3	

-Titration curve-



(Printout data from titration by MKC-610 and ADP-611)

«Titration parameter»

Titration Mode: titration mode / t(stir): wait time before titration / t(wait): EP sense inhibit time
t(max): titration limit time / Drift Stop: stop drift mode / Drift: drift level

«Control parameter»

Cell Type: of titration / Stable: stability potential / Ctrl.Gain: electrolysis speed coefficient
E.Mode: electrolysis mode / Start Mode: titration start mode / End Level: EP potential
Samp.Time: data sampling time / Stirrer: speed of stirrer

«Calculation parameter»

Calc.Type: of titration / Blank No: order of blank / Calc.No.: of formula
Unit: dimension / Decimal: number of digit after decimal point / Fraction: rounding fraction
Drift Comp.: compensation / Evaluation: of calculation results

«Option parameter»

Pre Treat: sample charge method onto oven / Cell Purge: purge time of heating unit
Back Purge: purge time of sample boat in heating unit
Heat.Mode: heating temperature setting / Oven Temp.: heating temperature

—Measurement results—

n	Sample (g)	Moisture (µg)	Concentration (ppm)
1	<u>1.0056</u>	<u>499.9</u>	<u>497.12</u>
2	1.0037	528.9	526.95
3	1.0093	501.9	497.28

Statistics	
Mean	507.12 ppm
SD	17.18 ppm
RSD	3.39 %

* The data were obtained from 3 tests of the same sample.

* Red underline shows the data from page 3/4.

10. Summary

Pellet means generally small bullet from materials. The pellet sample in this application is 3 to 5 mm in size popularly used for material of plastic products.

Sample measurement in the above example shows fair repeatability of 3.4 % relative standard deviation.