Application Note

№KCA-07027

Petroleum

Moisture in Bio Diesel Fuel (BDF)

Coulometric titration (Direct Method) by
Karl Fischer Moisture Titrator

Standard	JIS	K 0113	ASTM	D 1533	ISO	760
	JIS	K 0068	ASTM	D 4928	ISO	12937
	JIS	K 2275-3				

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 coulometric moisture titration according to JIS K-2275-3-2015 for measurement of water content in bio diesel fuel.

2.Reference

1) JIS K 0113-2005 Standard Test Method by Potentiometric, Amperometrtic, Coulometric and Karl Fischer Titration
2) JIS K 0068-2001 Test Method for Water Content in Chemical Products
3) JIS K 2275-3-2015 Crude petroleum and petroleum products –Determination of water – Part3: Coulometric Karl Fischer titration method
4) Hydranal manual published by Riedel de Haen
5) ISO 760:1978 Determination of Water-Karl Fischer method (General method)
6) ASTM D 1533-12 Standard Test Method for Water in Insulating Liquids by Coulometric Karl Fischer Titration
7) ASTM D 4928-12 Standard Test Methods for Water in Crude Oils by Coulometric Karl Fischer Titration
8) ISO 12937 2000 Petroleum products – Determination of water - Coulometric Karl Fischer Titration method

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) The resolution of mass balance is desirable to the nearest 0.01mg.
3) Utmost care must be taken in handling a sample with trace amount of water in it.

4. Post-measurement care

Drain out reagent in titration cell and inner cell, and rinse them with alcohol.
The electrode also needs cleaning with alcohol.

5.Test equipment

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

6.Reagent

Annolyte : Hydranal Coulomat A (Riedel de Haen)
Catholyte : Hydranal Coulomat CG (Riedel de Haen)
Additive : benzoic acid

7.Measurement procedure

－Pretreatment－

1) Put approx. 20g benzoic acid and 100mL annolyte in the titration cell.
2) Prepare about 5mL catholyte in the inner cell.
3) Dissolve benzoic acid running the stirrer.
4) Dehydrate the titration cell by performing pre-titration in advance.

－Measurement－

1) Sample the test liquid in a syringe, and discharge approx.1mL into the cell.
2) Start measurement.

8.Formula

Moisture (ppm) = F × (Moisture / (Wt1 － Wt2)) × k

F : Compensation coefficient (1)
Wt1 : Sample + Syringe weight (g)
Wt2 : Empty syringe (g)
k : Unit conversion coefficient (1)
Moisture: (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 : 24 ℃	Humidity : 67 %	Weather : Fair

The below data were obtained from titration by MKC-610:

-Titration parameter-

Model : MKC-610

Method No./Name :
09/Method09

[Titration]
Titr.mode : H2O
t(stir) : 0 s
t(wait) : 15 s
t(max) : 1800 s
Drift stop : Rel.
Drift : 0.10 ug/s

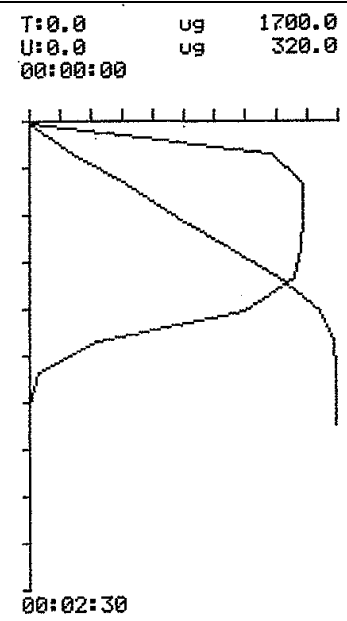
[Control]
Cell type : 2-Comp.
Stable : 0.1 ug/min
Ctrl.gain : 5.0
E.speed : Standard
Srart mode : Manual
End level : 200 mV
Stir.speed : 3

[Calculation]
Calc.type : Sample
Blank No. : 1
Calc.No. : 2
Unit : ppm
Decimal : 2
Fraction : Half adjust
Drift comp. : Auto
Evaluation : Off

[Report]
Report format : Short
Graph : On
Data list : Off

[Reagent]
Anolyte
A
Catholyte
CG

-Titration curve-



*** R e s u l t ***

Sample No. : 01-01
Date : 2007/07/06 13:55

Drift : 0.04 ug/s
Moisture : 1688.3 ug
Result : 1742.49 ppm
Titr.time : 00:01:37
Wt1 : 9.4660 g
Wt2 : 8.4971 g
Net : 0.9689 g

Meaning of test data on printout:

≪Titration parameter≫
Titr.mode: titration mode / t(stir): stirring time before titration starts / t(wait) wait time before EP detection
t(max): maximum time allowed for titration / Drift stop: drift stop mode / Drift: relative drift level
≪Control parameter≫
Cell type: of titration cell / Stable: stability potential level / Ctrl.gain: electrolytic speed coefficient
E.speed: electrolytic mode / Start mode: of titration / End level : potential level of endpoint
Stir.speed: of stirrer
≪Calculation parameter≫
Calc.type: calculation of titration / Blank No.: blank number / Calc.No. calculation formula number
Unit: of value / Decimal: number of digits after decimal point / Fraction: way of rounding fraction
Drift comp.: drift compensation / Evaluation: of calculation results

－Measurement resuls－

n	Sample (g)	Drift (μg/s)	Water (μg)	Conc. (ppm)
1	0.9689	0.04	1688.3	1742.49
2	0.7546	0.05	1324.7	1755.50
3	0.9050	0.06	1593.5	1760.77

Statistics	
Mean	1752.92 ppm
SD	9.4097 ppm
RSD	0.53680 %

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

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

10.Summary

Bio diesel fuel (BDF) is obtained by refining dehydrated waste cooking oils. Least amount of water contained in BDF is desirable for quality control.

Sample measurement in the above example showed a trace amount of water with fair repeatability of 0.5% relative standard deviation. Stable measurement of moisture content can be expected with Karl Fischer moisture titration.

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