Application Note

№KCA-07032

Petroleum

Moisture in Bio Diesel Fuel (BDF)

Coulometric titration (Evaporation Method) by
Karl Fischer Moisture Titrator

Standard	JIS	K 0113	ASTM	D 1533	ISO 760
	JIS	K 0068	ASTM	D 6304	
	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.

In this test, we use the indirect method with moisture evaporator (oven) for oil.

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) J 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 6304-16e1 Standard Test Method for Determination of Water in Petroleum Products, Lubricating Oils, and Additives by Coulometric Karl Fisher Titration

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

When draining out waste liquids after measurement, be aware that the sample and base oil were heated in the oven at high temperature during titration.

5.Test equipment

Main unit : Karl Fischer moisture titration coulometric system
Electrode : Electrolysis electrode
Twin platinum electrode for KF titration
Option : Evaporator (oven) for oil sample

6.Reagent

Annolyte : Hydranal Coulomat AG (Riedel de Haen)
Catholyte : Hydranal Coulomat CG (Riedel de Haen)

7.Measurement procedure

—Pretreatment—

1) Deliver base oil into the oven, and adjust flow rate of carrier gas to 200mL/min.
2) Set oven temperature to 150°C, and wait for equilibrium.
3) Connect the heater tube to vapor bottle.
4) Supply measuring cell with 150mL annolyte and the inner cell with 5mL catholyte.
5) Connect titration cell with bubbler tube and exhaust pipe.
6) Pre-titrate to dehydrate the measuring cell.

—Measurement—

1) Inject approximately 1mL of sample into vapor bottle.
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 (0.0001)
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 : 26.0 ℃	Humidity : 53 %	Weather : Fair

-Titration parameter-

Model : MKC-610

Method No./Name :
04/Evaporation(Sample)

[Titration]
Titr.mode : H2O
t(stir) : 0 s
t(wait) : 15 s
t(max) : 1200 s
Drift stop : Off

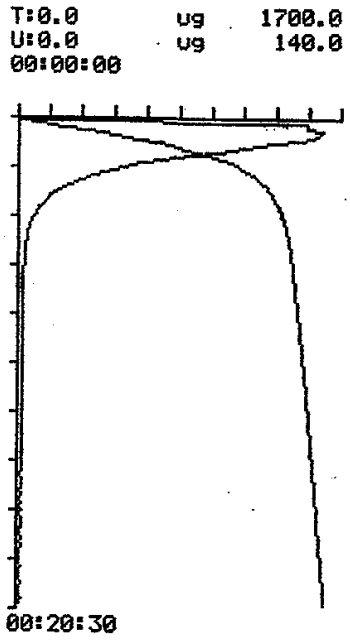
[Control]
Cell type : 2-Comp.
Stable : 0.1 ug/min
Ctrl.gain : 5.0
E.speed : Standard
Srart mode : Manual
End level : 200 mV
Samp.time : 10 s
Stir.speed : 3

[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
AG
Catholyte
CG

-Titration curve-



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

Sample No. : 01-01
Date : 2007/10/04 14:35

Drift : 0.15 ug/s
Moisture : 1444.0 ug
Result : 1680.83 ppm
Titr.time : 00:20:15
Wt1 : 9.5095 g
Wt2 : 8.6504 g
Net : 0.8591 g

(The above printout data were obtained from titration by MKC-610 + ADP-513)

≪Titration parameter≫
Titr.mode: titration mode /t(stir): stirring time before titration starts / t(wait): wait time before end sense starts
t(max): maximum time length allowed for titration / Drift stop: drift stop mode
≪Calculation parameter≫
Cell type: of titration / Stable: stability sense potential / Ctrl.gain: electrolysis speed coefficient
E.speed: of electrolysis / Start mode: of titration / End level EP potential/ samp.time.: data sampling time
Stir.speed: stirring speed
≪Calculation≫
Calc.type: of titration / Blank No.: blank level number / Calc.No.: formula number/ Unit: of result
Decimal: number of digits after decimal point / Fraction: the way how fraction is rounded
Drift comp.: drift compensation / Evaluation: of calculation results

－Measurement data－

n	Sample (g)	Moisture (μg)	Concentration (ppm)
1	0.8591	1444.0	1680.83
2	0.8732	1465.5	1678.31
3	0.8355	1389.6	1663.20
4	0.9589	1586.6	1654.60
5	0.9623	1579.8	1641.69

Statistics	
Mean	1663.73 ppm
SD	16.3874 ppm
RSD	0.98498 %

＊ The data were obtained from 5 tests of the same sample.
＊ Red underline shows the data from page 3/4.

10.Summary

Bio diesel fuel (BDF) is obtained by refining heat-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 1% relative standard deviation. Stable measurement of moisture content can be obtained by Karl Fischer moisture titration.

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