Application Note

№KVA-07026

Petroleum

Moisture in Bio Diesel Fuel (BDF)

Volumetric titration (Direct Method) by
Karl Fischer Moisture Titrator

Standard	JIS K 0113 JIS K 0068 JIS K 2275-2		ASTM E 203 ASTM D 1744 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-2275-2-2015 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 2275-2-2015 Crude petroleum and petroleum products –Determination of water – Part2: Potentiometric 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 E 203-16 Standard Test Method for Water Using Volumetric Karl Fischer Titration
7) ASTM D 1744-13 Standard Test Method for Determination of Water in Liquid Petroleum Products by Karl Fischer Reagent

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) 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 titration flask filled with extracting solvent.

5.Test equipment

Main unit : Karl Fischer moisture titration volumetric system
Electrode : Twin platinum electrode4 for KF titration

6.Reagent

Titrant : Hydranal Composit 5(f＝4.4328mg/mL) (Riedel de Haen)
Solvent : Extracting medium CM(for oil) (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) Sample approximately 1mL of test oil in a syringe.
2) Weigh the syringe on a balance of which resolution is to the nearest 0.1mg.
3) Discharge the sample into titration cell to dissolve in solvent.
4) Press Start button of titrator.
5) Weigh the syringe of above 3).
6) Enter Wt1 with weighed above 2) and Wt2 with 5).
7) The endpoint is automatically detected, from which water content can be obtained.

8.Formula

Moisture (ppm) = (Moisture / (Wt1 – Wt2)) × k
- Wt1 : Sample + Syringe weight (g)
- Wt2 : Empty syringe (g)
- k : Unit conversion coefficient (1000)
- Moisture : Water (mg) (Data × TF – Drift × t – Blank)
  - Data : Titration volume (mL)
  - TF : Factor of titrant (4.4328 mg H_2O /mL)
  - Drift : Drift level (mg/s)
  - t : Titration time (s)
  - Blank : Blank level (0.00 mg)

9.Example of measurement

－Ambient condition－

Room temperature : 24 ℃	Humidity : 67 %	Weather : Cloudy

- Titration parameter -

Model : MKA-610

Method No./Name :
08/Method08

[Titration]
Titr.mode : Normal
t(stir) : 0 s
t(wait) : 0 s
t(max) : 0 s
t(interval) : 0 s
Max.volume : 10.00 mL
Titr.bur.No. : 1
Dose mode : Off

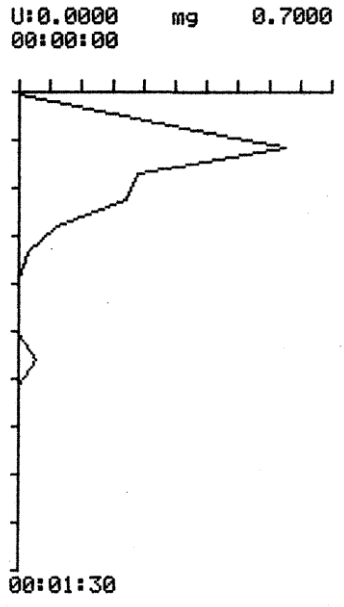
[Control]
End time : 30 s
Final vol. : 0.01 mL
Titr.speed : 3
Detect.mode : 1
Drift titr. : On
Start mode : Manual
End level : 75 mV
Samp.time : 5 s
Stir.speed : 4

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

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

[Reagent]
Reagent name :
Composite 5
Factor :4.4328 mg/mL

- Titration results -



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

Sample No. : 01-01
Date : 2007/07/19 14:51

Moisture : 1.5515 mg
Result : 1754.30 ppm
Burette 1 : 0.3500 mL
Titr.time : 00:01:19

Wt1 : 9.5054 g
Wt2 : 8.6210 g
Net : 0.8844 g

(The above printout data are obtained from titration by MKA-610 unit)

≪Titration parameter≫
Titr.mode: titration mode / t(stir): delay time before titration starts / t(wait): time without EP sense / t(max): maximum time allowed for titration/t(interval): intermittent time/ Max.volume: of titratiion/ Titr.bur.No.: buret number / Dose mode: fixed dose mode
≪Control parameter≫
End time: EP sense / Final vol.: of titration / Titr.speed: of titration/ Detect.mode: EP detection mode
Drift titr.:drifttitration / Start mode: of titration / End level: EP potential / samp.time. data sampling time
≪Calculation parameter≫
Calc.type: calculation type / Blank No. blank number/ Calc.No. : of formula
Decimal: number of digits after decimal point / Fraction: way how fraction is rounded
Drift comp.: drift compensation / Evaluation: of calculation results

－Measurement results－

N	Sample (g)	Titration (mL)	Water (mg)	Conc. (ppm)
1	0.8844	0.350	1.5515	1754.30
2	0.8538	0.340	1.5072	1765.28
3	1.0105	0.400	1.7731	1754.68

Statistics	
Mean	1758.09 ppm
SD	6.237 ppm
RSD	0.3548 %

＊ 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 heat-dehydrated waste cooking oils. Least amount of water contained in BDF is desirable for quality control.

Sample measurement in the above example shows a trace amount of water with fair repeatability of less than 0.5% relative standard deviation. Stable measurement of water content is assured by Karl Fischer moisture titration.

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