KEM Application Note

№KVA-07020

Petroleum

Moisture in Bio Diesel Fuel (BDF)

Volumetric titration (Direct Method) by
Karl Fischer Moisture Titrator

Standard	JIS K 0113		ASTM E 203	
	JIS K 0068		ASTM D 1744	
	JIS K 2275-2		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.Cautons 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 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

6.Reagent

Reagent : Hydranal Composit 5(f=4.6670mg/mL) (Riedel de Haen)
Solvent : Extracting medium CM(for oils) (Hayashi Chemicals)

7.Test 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 key 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 (%) = ((Data × F − Blank) / (Wt1 − Wt2)) × 0.1

Data : Titration volume (mL)
F : Reagent factor (4.6670 mg H_2O /mL)
Blank : Blank level (0.00 mg)
Wt1 : Sample + Syringe weight (g)
Wt2 : Empty syringe (g)

9.Example of measurement

－Ambient condition－

Room temperature : 22 ℃	Humidity : 37 %	Weather : Fair

The below data were obtained from titration by MKS-500:

-Titration parameter-

[Titration]
Method Driect
Titr.Speed 3
End Time 30 S
Final Vol. 0.01 mL
Detector Mode Oil
t(stir) 0 s
t(max) 0 s
Drift Titr. On
Max.Volume 20 mL

[Calculation]
Method Driect
Calc.Formula g->%

-Titration results-

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

Sample No. 01-01

Date 07/04/20 16:08
Method Driect
Sample Name
Sample ID
Reagent Name Composit5
Reagent Factor
4.6670 mg/mL
Calc.Formula g->%
Wt1 9.4817 g
Wt2 8.5841 g
Net 0.8976 g
Blank 0.0 mg
Result 0.1040 %
0.200 mL
0.9334 mg
Titr.Time 00:02:41

Meaning of test data on printout:

≪Titration parameter≫
Method: of titration / Titr.Speed: titration speed depends on reaction of reagent and sample
End Time: waiting for endpoint / Final Vol. final dose volume / Detector Mode: detector mode
t(stir): stirring time length / t(max): maximum titration time length allowed
Drift Titr.: drift titration / Max Volume: maximum titration volume
≪Calculation parameter≫
Calc.Formula selected calculation formula

－Measurement results－

n	Sample (g)	Titrated (mL)	Water (mg)	Conc. (%)
1	0.8976	0.200	0.9334	0.1040
2	0.8823	0.200	0.9334	0.1058
3	0.8791	0.200	0.9334	0.1062

Statistics	
Mean	0.1053 %
SD	0.0012 %
RSD	1.1126 %

＊ 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 1% relative standard deviation. Stable measurement of moisture content can be expected with Karl Fischer moisture titration.

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