

Petroleum

Acid number of Bio Diesel (BDF)

Automatic Potentiometric Titrator
Acid-base titration (non aqueous)

Standards	JIS	K 2501	IP	177
	ASTM	D 664	GOST	29255
	ISO	6619		

1. Abstract

Quality control of bio diesel fuel (BDF) on acid number is important since the number increases when the fuel itself deteriorates or BDF in production in line is not conforming.

The acid number of BDF is measured according to JIS, ASTM or ISO standards by potentiometric titration with 0.1mol/L potassium hydroxide 2-propanol solution until the endpoint is found on titration curve.

The acid number of bio diesel fuel is calculated from the titration volume of potassium hydroxide 2-propanol solution consumed in reaching the endpoint.

2. Reference

- 1) JIS K 2501-2003 Test Method for Neutralization number – Petroleum products and lubricants
- 2) ASTM D 664-11a Standard Test Method for Acid Number of Petroleum Products by Potentiometric Titration
- 3) ISO 6619:1988 Petroleum products and lubricants – Neutralization number – Potentiometric Titration method

3. Cautions in measurement

- 1) Repeated measurement lowers sensitivity of glass electrode, and thus requires regeneration process for the electrode each time a measurement is finished. To regenerate the electrode, dip it in an alcohol solution once for cleaning, and then dip it in pure water for 10 ~ 60 min. (depends on how much contaminated)
- 2) The standard concentration of potassium hydroxide 2-propanol solution is 0.0005mol/L or less. Adjust it to the standard level from time to time.

4. Post-measurement care

After a series of measurements for the day is over, be sure to regenerate the electrode once for all according to the preceding "3. Cautions in measurement".

5. System configuration

Main unit : Automatic potentiometric titrator (preamplifier: STD)

Electrodes : (* mark shows standard supplied item)

* Glass electrode

* Sleeve type Reference electrode

(Junction liquid: 3.33M-potassium chloride solution)

* Temperature compensation electrode

6. Reagent

Reagent : 0.1mol/L Potassium hydroxide 2-propanol solution (f=1.000)

Mixed solvent : Toluene : Water : 2-propanol (500 : 5 : 495)

7. Measurement procedure

—Pretreatment—

- 1) Transfer 120mL mixed solvent to a 200mL beaker.
- 2) Perform a blank test, and obtain the blank level.

—Measurement—

- 1) Transfer approx.20g sample liquid to a 200mL beaker.
- 2) Add 120mL mixed solvent.
- 3) Titrate with 0.1mol/L potassium hydroxide 2-propanol to obtain acid number.

8. Formula

Total oxidation (mg / g) = (EPl - BLl) × TF × Cl × K1 / SIZE

EPl : Titration volume (mL)

BLl : Blank level (0.00mL)

TF : Reagent factor (1.00)

Cl : Concentration conversion coefficient (56.1 g/mL)

(equivalent to potassium hydroxide in 1mL of 1mol/L potassium hydroxide 2-propanol solution)

K1 : Unit conversion coefficient (0.1)

SIZE : Sample size (g)

9. Example of measurement

—Ambient condition—

Room temperature : 24 °C	Humidity : 78 %	Weather : Rain
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(The below data and titration curve are printed out when titrated by AT-510 unit.)

-Titration parameter-

Model : AT-510	
Serial No. : NZB52534	
Method No. : 01	
Titr.mode : Auto Intermit	
Titr.form : EP Stop	
[Titration parameter]	[Result parameter]
Form : EP Stop	<Calculation>
APB No. : 1	Calc.Type : Sample
Unit No. : 1	Conc.1 : Set
Detector No. : 1	C01=
Unit : pH	(EP1-BL1)*TF*K1*C1/SIZE
Max. Volume : 20.0mL	Unit : mg/g
Wait Time : 0s	EP No. : 1
Direction : Auto	Temp. Comp. : Off
[Control parameter]	<Constant>
End Point No. : 1	C1 (mg/mL) : 56.1
End sense : Auto	K1 : 0.1
End Point Area : Off	[Titr. constant]
Separation : Off	TF : 1.00
Over Titr. Vol. : 0mL	[Blank list]
Gain : 1	Blank1 : 0.00
Data samp. Pot. : 4.0mV	
Data samp. Vol. : 0.5mL	
Stability : 0.5mV/s	
Delay Time : 0s	
Limit Time : 30s	

-Titration curve-

*** Result ***
Sample No. : 01-01
Date : 2007/06/25 14:34
Sample ID : Sample
Method No. : 01
< Auto Intermit >
Method Name : Auto Intermit
Titr. Reagent Name : 0.1M-KOH
Titr. time : 00:12:24
Size : <u>19.994 g</u>
Conc-1 : <u>0.6301mg/g</u>
End point-1
Volume : <u>2.2456mL</u>
Potential : 12.94pH
3.00 [pH] 16.00
0.000
4.000
[mL]

Meaning of printout data:

<p><<Titration parameter>></p> <p>Form: titration form / APB No.: number of power burette / Unit No.: APB Unit File number</p> <p>Detector No.: detector number / Max Volume.: of titration / Wait Time: before titration starts</p> <p>Direction.: of titration</p> <p><<Control parameter>></p> <p>End Point No.: total EPs / End sense: of EP detection method/ End Point Area: EP detection area</p> <p>Separation: separated potential / Over Titr.Vol.: over titration / Gain: sensitivity of signal</p> <p>Data samp.Pot.: potential change for data sampling / Data samp.Vol.: titration change for data sampling</p> <p>Stability: stability level / Delay Time: before stability check / Limit Time: for stability check</p> <p><<Result parameter>></p> <p>Calc.Type: calculation type / Conc.1: concentration formula 1 / Unit: of calculated results</p> <p>EP No. EP number / Temp.Comp.: temperature compensation / C1(mg/mL): concentration conversion coefficient</p> <p>K1: unit conversion coefficient / TF: factor of reagent / Blank1: blank level 1</p>

—Measurement results—

n	Sample (g)	Titrated (mL)	Acid number (mg/g)
1	<u>19.994</u>	<u>2.246</u>	<u>0.6301</u>
2	19.994	2.239	0.6281
3	19.996	2.232	0.6261
4	19.991	2.224	0.6240
5	19.995	2.243	0.6292

Statistics of acid number		
Mean	0.6275	mg/g
SD	0.0025	mg/g
RSD	0.3921	%

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

10. Summary

The acid number is potassium hydroxide in mg consumed in neutralizing one (1) gram of free fatty acid.

The above example shows a good result of repeatability with less than 0.4% RSD (relative standard deviation) by the automatic potentiometric titrator.