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 664-95 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 H-171 [№ 98100H171]
   * Reference electrode R-173 [№ 98100R173]
     (Junction liquid: 3.33M-potassium chloride solution)
   * Temperature compensation electrode T-171 [№ 98100T171]

6. Reagent

Reagent: 0.1mo1/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 0.1mo1/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 1mo1/L potassium hydroxide 2-propanol solution)
   K1 : Unit conversion coefficient ( 0.1 )
   SIZE : Sample size ( g )
9. Example of measurement

--- Ambient condition ---

<table>
<thead>
<tr>
<th>Room temperature</th>
<th>Humidity</th>
<th>Weather</th>
</tr>
</thead>
<tbody>
<tr>
<td>24 °C</td>
<td>78 %</td>
<td>Rain</td>
</tr>
</tbody>
</table>

(The below data and titration curve are printed out when titrated by AT-510 unit.)

**Titration parameter**

- **Model**: AT-510
- **Serial No.**: NZBS2534
- **Method No.**: 01
- **Titr. mode**: Auto Intermit
- **Titr. form**: EP Stop

**[Result parameter]**

- **Form**: EP Stop
- **APB No.**: 1
- **Unit No.**: 1
- **Detector No.**: 1
- **Unit**: pH
- **Max. Volume**: 20.0mL
- **Wait Time**: 0s
- **Direction**: Auto

**[Calc. Type]**

- **Calc. Type**: Sample
- **Conc. 1**: Set
- **CO1**: (EP1-BL1) * TF * K1 * C1 / SIZE

**[Control parameter]**

- **End Point No.**: 1
- **End sense**: Auto
- **End Point Area**: Off
- **Separation**: Off
- **Over Titr. Vol.**: 0mL
- **Gain**: 1
- **Data samp. Pot.**: 4.0mV
- **Data samp. Vol.**: 0.5mL
- **Stability**: 0.5mV/s
- **Delay Time**: 0s
- **Limit Time**: 30s

**[Blank list]**

- **Blank1**: 0.00

**Meaning of printout data:**

**<Titration parameter>**

- **Form**: titration form / **APB No.**: number of power burette / **Unit No.**: APB Unit File number
- **Detector No.**: detector number / **Max Volume.**: of titration / **Wait Time**: before titration starts
- **Direction.**: of titration

**<Control parameter>**

- **End Point No.**: total EPs / **End sense**: direction / **End Point Area**: EP detection area
- **Separation**: separated potential / **Over Titr. Vol.**: over titration / **Gain**: sensitivity of signal
- **Data samp. Pot.**: potential change for data sampling / **Data samp. Vol.**: titration change for data sampling
- **Stability**: stability level / **Delay Time**: before stability check / **Limit Time**: for stability check

**<Result parameter>**

- **Calc. Type**: calculation type / **Conc. 1**: concentration formula 1 / **Unit**: of calculated results
- **EP No.**: EP number / **Temp. Comp.**: temperature compensation / **C1(mg/mL)**: concentration conversion coefficient
- **K1**: unit conversion coefficient / **TF**: factor of reagent / **Blank1**: blank level 1

--- Result ---

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Date</th>
<th>Sample ID</th>
</tr>
</thead>
<tbody>
<tr>
<td>01-01</td>
<td>2007/06/25 14:34</td>
<td>Sample</td>
</tr>
</tbody>
</table>

**Method No.**: 01

- **Method Name**: Auto Intermit

- **Titr. Reagent Name**: 0.1M-KOH
- **Titr. time**: 00:12:24
- **Size**: 19.994 g
- **Conc-1**: 0.6301mg/g
- **End point-1**: Volume : 2.2456mL

**Potential**: 12.94pH

**Result parameter**

- **Calc. Type**: calculation type / **Conc. 1**: concentration formula 1 / **Unit**: of calculated results
- **EP No.**: EP number / **Temp. Comp.**: temperature compensation / **C1(mg/mL)**: concentration conversion coefficient
- **K1**: unit conversion coefficient / **TF**: factor of reagent / **Blank1**: blank level 1

**TIA-07025 Ver.01 3/4**
### Measurement results

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

<table>
<thead>
<tr>
<th>Statistics of acid number</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean</td>
</tr>
<tr>
<td>SD</td>
</tr>
<tr>
<td>RSD</td>
</tr>
</tbody>
</table>

* 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. The acid number of BDF can be analyzed by any of the following titration systems.

- **[AT-610]**
  - Awarded Supreme Technology from Kyoto City
  - Easy key entry by touch panel of large color LCD (8-inch wide)
  - Simultaneous titration in parallel
  - Both potentiometric and Karl Fischer moisture titration (coulometric-volumetric) can be performed at a time.

- **[AT-510]**
  - Compact and cost performance model
  - PC card expands data memory for convenience and versatility.

- **[AT-500N-1]**
  - Low cost and high performance
  - Easy view with back light LCD
  - GLP/GMP conformed model

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![Logo](logo.png)

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