

<b>HIRANUMA APPLICATION DATA</b>	Automatic Titrator	Data No.	L1	Jun.6. 2017
<b>Lubricant petroleum products</b>	<b>Acid number in Lubricating oil</b>			

## 1. Abstract

The acid number of lubricant oil is one of the important index for judging its quality.

Measurement of acid number is defined in several standard test methods. It is indicated by "milligrams of potassium hydroxide required to neutralize acidic components contained in 1 g of the sample".

The international standard methods for acid number are shown as bellow.

- JIS K2501 2003 : Petroleum products and lubricants - Determination of neutralization number
- ASTM D664-1995 : Standard Test Method for Acid Number of Petroleum Products by Potentiometric Titration

The potentiometric titration process is as follows:

- 1) Weigh sample exactly corresponding to acid number and dissolve it in a titration solvent.
- 2) Immerse glass electrode and reference electrode.
- 3) Start titration with alcoholic potassium hydroxide solution.

Inflection point is defined as the end point if it obtained sharply. If it's not clear, the pH obtained from measurement of buffer solution is defined as the end point. The measurement with the latter end point detection will be introduced here.

## 2. Configuration of instruments and reagents

### (1) Configuration of instruments

Main unit	:	Hiranuma Automatic Titrator	COM Series
Electrode	:	Glass electrode	GE-101B
	:	Reference electrode	RE-201Z
	:	Thermistor electrode	TE-403

### (2) Reagents

Titrant	:	0.1 mol/L potassium hydroxide in 2-propanol
Titration solvent	:	Mixture of 500 mL of Toluene, 495 mL of 2-Propanol and 5 mL of water
Buffer	:	Mixture of 10 mL of "Buffer B" and 100 mL of titration solvent, "Buffer B" is regulated in "JIS K 2501"

## 3. Measurement procedure

### (1) Determination of the end point pH

- i) Put the stir bar in buffer solution and immerse electrodes.
- ii) Read the pH after the pH reading stabilizes within 5 mV or 0.1 pH per minute.

The time required for stabilization is about 5 minutes as a guide.

(2) Measurement of Acid number

- i) Take 5 g of sample into 200 mL tall beaker and weigh accurately to 0.1 mg digits.

Note that the weight of sample will be changed depending on the acid number.

- ii) Add 125 mL of titration solvent and dissolve sample by stirrer.

The stirrer speed must be adjusted to avoid the scattering of contents or taking the air into the solution.

- iii) Immerse the electrodes and titrate by alcoholic KOH titrant until the end point pH.

Also, perform the blank test with the same procedure of sample measurement.

## 4. Measurement conditions and results

### Examples of titration conditions

#### Measurement of blank

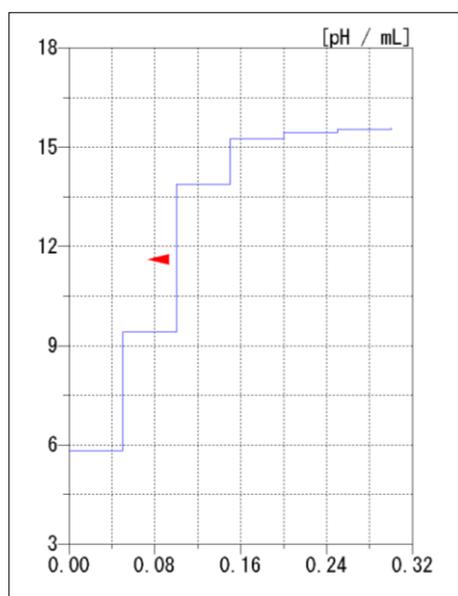
Cndt No.	1	ConstantNo.	2	Mode No.	15
Method	Set	Size	0 g	Pre Int	0 sec
Buret No.	1	Blank	0 mL	Del K	0
Amp No.	1	Molarity	0.1 mol/L	Del Sens	0 mV
D. Unit	pH	Factor	1.006	Int Time	60 sec
S-Timer	120 sec	K	56.1	Int Sens	0 mV
C.P. mL	0 mL	L	0	BrT Speed	2
Direction	↑	Unit	mL	Pulse	40
D.P. mL	0 mL	Formula	D		
End Point pH	11.6 pH	Digits	4		
Over mL	0.2 mL	Auto In Pram.	Non		
Max.Vol.	1 mL				

#### Measurement of sample

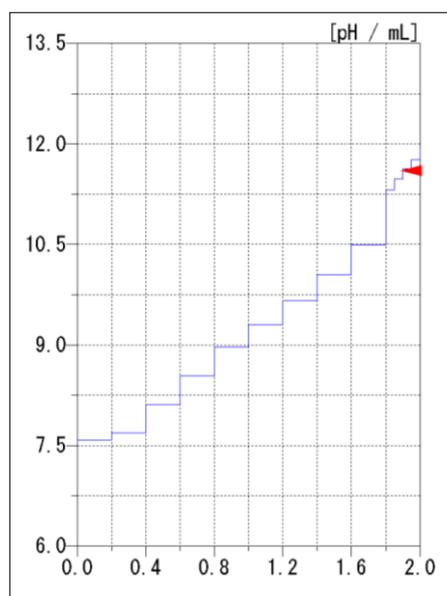
Cndt No.	2	ConstantNo.	1	Mode No.	1
Method	Oil1	Size	5.0012 g	Del mL	0.2 mL
Buret No.	1	Blank	0.075 mL	Int Time1	60 sec
Amp No.	1	Molarity	0.1 mol/L	Tran Timer	120 sec
D. Unit	pH	Factor	1.006	Del mL2	0.05 mL
S-Timer	120 sec	K	56.1	Int Time2	60 sec
C.P. pH	10.5 pH	L	0	Int Time	0 sec
Direction	↑	Unit	mg/g	Int Sens	0 mV
D.P. pH	10.5 pH	Formula	(D-B)*K*F*M/S		
End Point pH	11.6 pH	Digits	3		
Over mL	0.1 mL	Auto In Pram.	Non		
Max.Vol.	20 mL				

## Measurement results

Measurement of blank			Measurement of sample		
Number of measurement	Size (g)	Titer (mL)	Size (g)	Titer (mL)	Acid number (mg KOH/g)
1	—	0.074	4.9901	1.923	2.0900
2	—	0.076	4.9989	1.890	2.0490
3	—	—	5.0012	1.892	2.0500
Avg.		<b>0.075 mL</b>	Avg.		<b>2.06 mgKOH/g</b>
			SD		0.02 mgKOH/g
			RSD		1.13 %



Measurement of blank



Measurement of sample

## Examples of titration curves

### 5. Note

#### (1) Management of electrode

It is recommended to activate the electrodes for about 5 minutes to pure water for each measurement. This is because when glass electrode is used for a long time in a nonaqueous solvent, the response speed and electromotive force decrease. Also potassium chloride crystallizes around the liquid junction of the reference electrode, which causes pH fluctuation.

#### (2) Maintenance of buret

It is recommended to wash the flow channel of buret with water. This is because alcoholic KOH titrant have a tendency toward crystallization. When not using for a long time, please discharge titrant and then wash flow channel with water.

Keyword : JIS K2501, ASTM D664, Lubricant oil, neutralization number, Acid number, Potentiometric titration, Non-aqueous neutralization titration

\*Some measurement would not be possible depending on optional configuration of system.