

HIRANUMA APPLICATION DATA	Automatic Titrator	Data No.	L2	Jun.6. 2017
Lubricant petroleum products	Base number in lubricating oil (Hydrochloric acid method)			

1. Abstract

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

Measurement of base number is defined in several standard test methods. It is indicated by "milligrams of potassium hydroxide equivalent weight to acid required to neutralize basic components contained in 1 g of the sample". There are two methods of base number, hydrochloric acid method and perchloric acid method. In this article, hydrochloric acid method will be introduced.

The international standard methods for base number with hydrochloric acid method are shown as bellow.

- JIS K2501 2003 : Petroleum products and lubricants - Determination of neutralization number
- ASTM D4739-2011 : Standard Test Method for Base Number Determination by Potentiometric Hydrochloric Acid Titration

The potentiometric titration process is as follows:

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

Titant	:	0.1 mol/L hydrochloric acid 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 A" and 100 mL of titration solvent, "Buffer A" 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 Base 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 base 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 HCl 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

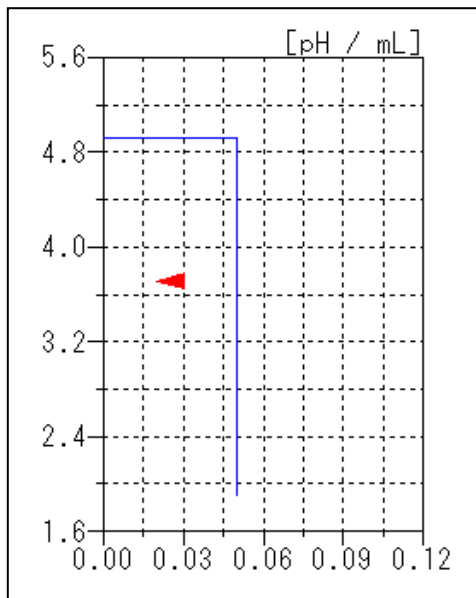
Cndt No.	47	ConstantNo.	47	Mode No.	47
Method	Set	Size	0 g	Int Time Max	0 sec
Buret No.	1	Blank	0 mL	Del K	0
Amp No.	1	Molarity	0.1 mol/L	Del Sens	0 mV
D. Unit	pH	Factor	0.9985	Int Time	60 sec
S-Timer	120 sec	K	0	Int Sens	0 mV
C.P. mL	0 mL	L	0	BrT Speed	2
Direction	↓	Unit	mL	Pulse	40
T Timer	0 sec	Formula	D		
D.P. mL	0 mL	Digits	3		
End Point pH	3.71 pH	Auto In Pram.	Non		
Over mL	0.1 mL				
Max.Vol.	20 mL				

Measurement of sample

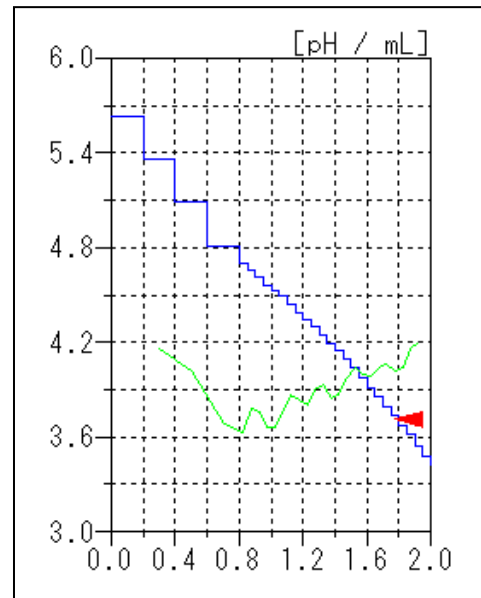
Cndt No.	48	ConstantNo.	48	Mode No.	11
Method	Oil1	Size	5.0066 g	Del mL	0.2 mL
Buret No.	1	Blank	0.018 mL	Int Time1	60 sec
Amp No.	1	Molarity	0.1 mol/L	Tran Timer	120 sec
D. Unit	pH	Factor	0.9985	Del mL2	0.05 mL
S-Timer	120 sec	K	56.1	Int Time2	60 sec
C.P. pH	6 pH	L	0	Int Time	0 sec
Direction	↓	Unit	mg/g	Int Sens	0 mV
T Timer	0 sec	Formula	(D-B)*K*F*M/S		
D.P. pH	6 pH	Digits	3		
End Point pH	3.71 pH	Auto In Pram.	Non		
Over mL	0.3 mL				
Max.Vol.	20 mL				

Measurement results

Measurement of blank			Measurement of sample			
Number of Measurement	Size (g)	Titer (mL)	Number of Measurement	Size (g)	Titer (mL)	Base number (mgKOH/g)
1	—	0.020	1	5.0066	1.786	1.978
2	—	0.015	2	5.0291	1.770	1.951
Avg. (Blank)			Avg.			1.95 mgKOH/g
			Statistic calculation			SD
						0.022 mgKOH/g
						RSD
						1.14 %



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

When you do not use the equipment for a long time, it is recommended to wash the flow channel of buret with water.

Keyword : JIS K2501, ASTM D4739, Lubricant oil, Neutralization number, Base number, Potentiometric titration, Non-aqueous neutralization titration

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