

HIRANUMA APPLICATION DATA	Automatic Titrator	Data. No	L11	Jun.6. 2017
Lubricant petroleum products	Base number in Lubricant oil (Perchloric acid / Back-titration)			

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 addition, there are two types of perchloric acid titration, forward-titration and back-titration. In this article, perchloric acid method with back-titration will be introduced.

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

- JIS K2501 2003 : Petroleum products and lubricants - Determination of neutralization number
- ASTM D2896-06 : Standard Test Method for Base Number of Petroleum Products by Potentiometric Perchloric 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) Add fixed amount of perchloric acid in acetic acid solution to the sample.
- 4) Start titration with sodium acetate in acetic acid solution.

There are two procedures for perchloric acid titration with back-titration, A and B on ASTM D2896. Procedure A and B use different titration solvent volume and sample weight. In this article, measurement with procedure B will be applied.

2. Configuration of instruments and reagents

(1) Configuration of instruments.

Main unit	: Hiranuma Automatic Titrator	COM Series
	One buret	Option
Electrode	: Glass electrode	GE-101B
	: Reference electrode	RE-201Z
	Inner solution should be changed; it is described below.	

(2) Reagents

Titrant	: 0.1 mol/L sodium acetate in glacial acetic acid standard solution
Additive	: 0.1 mol/L perchloric acid in glacial acetic acid standard solution
Titration solvent	: Mixture of 500 mL of glacial acetic acid and 1 L of chlorobenzene Refer; Toluene can be used instead of chlorobenzene.
Inner solution	: Saturated sodium perchlorate in glacial acetic acid

3. Measurement procedure

(1) Take 1 g of sample into 100 mL beaker and weigh accurately to 0.1 mg digits.

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

(2) Add 60 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.

(3) Immerse the electrode and start titration. 4 mL of 0.1 mol/L perchloric acid-Acetic acid standard solution is added with buret, and the contents of beaker is stirred for 2 min.

(4) Titrate by 0.1 mol/L sodium acetate in acetic acid standard solution.

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

4. Measurement conditions and results

Example of titration conditions

(1) Measurement of blank

i) Dispense 0.1 mol/L perchloric acid in acetic acid standard solution.

Cndt No.	43
Method	Disp
Buret No.	1
S-Timer	0 sec
Disp Vol.	4 mL

ii) Titration with 0.1 mol/L sodium acetate in acetic acid standard solution.

Cndt No.	45	ConstantNo.	45	Mode No.	8
Method	Auto	Size	0 g	Pre Int	0 sec
Buret No.	2	Blank	0 mL	Del K	5
Amp No.	1	Molarity	0.1 mol/L	Del Sens	0 mV
D. Unit	mV	Factor	1.006	Int Time	5 sec
S-Timer	20 sec	K	0	Int Sens	3 mV
C.P. mL	0 mL	L	0	Brst Speed	2
T Timer	0 sec	Unit	mL	Pulse	40
D.P. mL	0 mL	Formula	D		
End Sens	300	Digits	4		
Over mL	0.3 mL	Auto In Pram.	Non		
Max.Vol.	20 mL				

(2) Measurement of sample

i) Dispense 0.1 mol/L perchloric acid in acetic acid standard solution.

Cndt No.	43
Method	Disp
Buret No.	1
S-Timer	0 Sec
Disp Vol.	4 mL

ii) Titration with 0.1 mol/L sodium acetate in acetic acid standard solution.

Cndt No.	44	ConstantNo.	44	Mode No.	8
Method	Auto	Size	1.0307 g	Pre Int	0 sec
Buret No.	2	Blank	3.979 mL	Del K	5
Amp No.	1	Molarity	0.1 mol/L	Del Sens	0 mV
D. Unit	mV	Factor	1.006	Int Time	5 sec
S-Timer	120 sec	K	56.1	Int Sens	3 mV
C.P. mL	0 mL	L	0	Brt Speed	2
T Timer	0 sec	Unit	mg/g	Pulse	40
D.P. mL	0 mL	Formula	$((B-D)*K*F*M)/S$		
End Sens	300	Digits	4		
Over mL	0.3 mL	Auto In Pram.	Non		
Max.Vol.	20 mL				

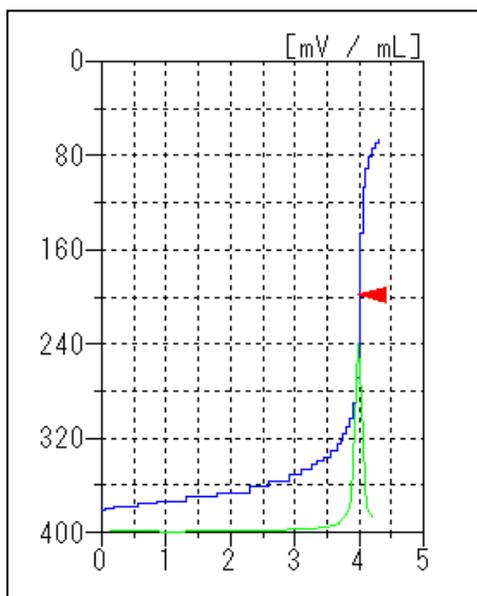
Measurement results

Measurement of blank

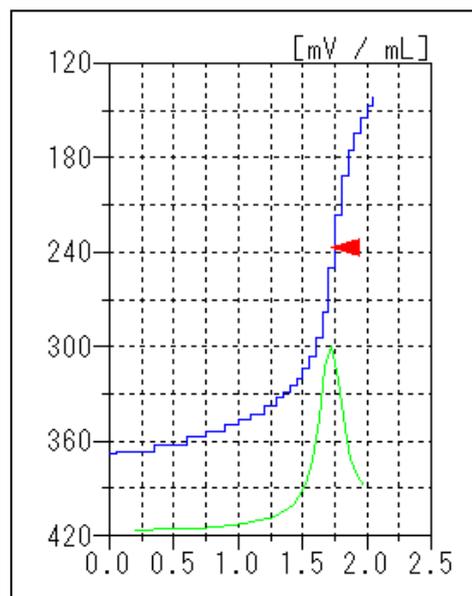
Measurement number	Size (g)	Titet (mL)
1	—	3.979
2	—	3.978
Avg. (Blank)		3.979 mL

Measurement of sample

Measurement number	Size (g)	Titer (mL)	Base number (mgKOH/g)
1	1.0307	1.719	12.375
2	1.0015	1.779	12.398
3	1.0108	1.763	12.373
Statistic calculation		Avg.	12.4 mgKOH/g
		SD	0.0138 mgKOH/g
		RSD	0.11 %



Measurement of blank



Measurement of sample

Examples of titration curves

5. Note

(1) Management of the 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. Since the electrolyte of the inner solution and the oil sample may adhere around the liquid junction of reference electrode, it causes the fluctuation of the potential. Therefore it is recommended to clean the liquid junction periodically.

(2) Experiment temperature

Acetic acid used as a solvent for the titrant has a relatively large thermal expansion coefficient. Temperature change of 1 ° C causes the titrant volume change of 0.1 %. For accurate measurement, factor titration and sample measurement should be performed at the same room temperature as much as possible.

(3) Preparation of inner solution for reference electrode

The inner solution of the reference electrode RE-201Z is filled with saturated KCl aqueous solution when purchased. For this measurement, it is necessary to replace inner solution to saturated sodium perchlorate in acetic acid solution. Replacement procedure is described below.

- i) Prepare the saturated solution of sodium perchlorate in acetic acid with reagent grade of these.
- ii) Discharge inner solution from reference electrode RE-201Z and wash inside it with water and then acetic acid.
- iii) Fill the prepared inner solution into reference electrode from the supply port.
- iv) Leave the electrode for one day before use.

Keyword : JIS K2501, ASTM D2896, Lubricant oil, Neutralization number, Base number, Potentiometric titration, Perchloric titration, Back-titration

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