

HIRANUMA APPLICATION DATA	Automatic Titrator	Data No.	L3	Jun.6. 2017
Lubricant petroleum products	Base number in lubricating oil (Perchloric 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, perchloric acid method 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) Start titration with perchloric acid in acetic acid solution.

There are two procedures for perchloric acid 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.

Inflection point is defined as the end point if it obtained sharply. If it's not clear, back-titration method could be applied. Back-titration method is mentioned in "HIRANUMA APPLICATION DATA No. L11".

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

Inner solution should be changed; it is described below. to below described

(2) Reagents

Titant	: 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 titrate by 0.1 mol/L Perchloric acid-Acetic acid standard solution.

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.	42	ConstantNo.	42	Mode No.	19
Method	Auto	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	mV	Factor	1.003	Int Time	5 sec
S-Timer	10 sec	K	0	Int Sens	3 mV
C.P. mL	0 mL	L	0	BrT Speed	2
T Timer	0 sec	Unit	mL	Pulse	40
D.P. mL	0 mL	Formula	D		
End Sens	500	Digits	4		
Over mL	0.1 mL	Auto In Pram.	Non		
Max.Vol.	2 mL				

Measurement of sample

Cndt No.	41	ConstantNo.	41	Mode No.	8
Method	Auto	Size	1.0126 g	Pre Int	0 sec
Buret No.	1	Blank	0.025 mL	Del K	5
Amp No.	1	Molarity	0.1 mol/L	Del Sens	0 mV
D. Unit	mV	Factor	1.003	Int Time	5 sec
S-Timer	30 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	(D-B)*K*F*M/S		
End Sens	200	Digits	4		
Over mL	0.3 mL	Auto In Pram.	Non		
Max.Vol.	20 mL				

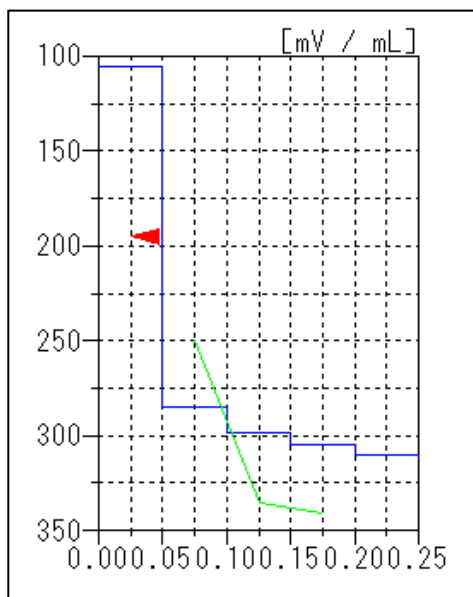
Measurement results

Measurement of blank

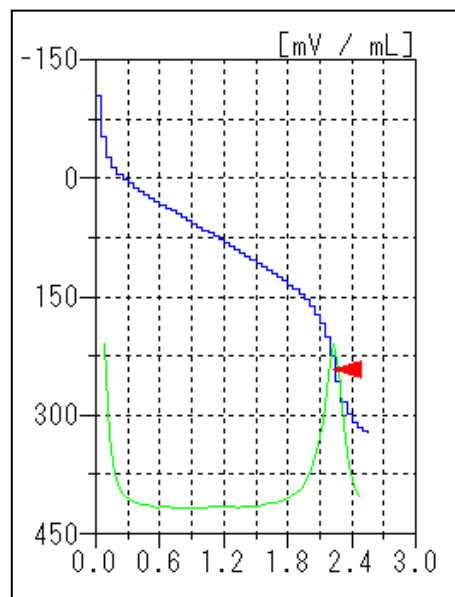
Number of Measurement	Size (g)	Titer (mL)
1	—	0.025
2	—	0.025
Avg. (Blank)		0.025 mL

Measurement of sample

Number of Measurement	Size (g)	Titer (mL)	Base number (mgKOH/g)
1	1.0126	2.227	12.236
2	0.9114	1.998	12.181
3	1.0073	2.218	12.250
		Avg.	12.2 mgKOH/g
		SD	0.0366 mgKOH/g
		RSD	0.30 %



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 and cause 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, and when the temperature changes by 1 ° C, the titrant causes a 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 acid titration