

HIRANUMA APPLICATION DATA	Automatic Titrator	Data No.	L15	Oct.24. 2024
Lubricant petroleum products	Base number in Petroleum products (Perchloric acid method)			

1. Abstract

The base number of petroleum products are 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. The international standard methods for base number are shown as bellow.

- ASTM D 2896 : Base Number of Petroleum Products by Potentiometric Perchloric Acid Titration
- ASTM D 4739 : Standard Test Method for Base Number Determination by Potentiometric Hydrochloric Acid Titration
- JIS K2501 : Petroleum products and lubricants - Determination of neutralization number
- ISO 3771 : Potentiometric titration method for base number (perchloric acid method)

This data sheet presents an example of how the base number of a petroleum product is measured by the perchloric acid titration method (ASTM D 2896).

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. L16".

2. Configuration of instruments and reagents

(1) Configuration of instruments

Main unit	:	Automatic Titrator	COM Series
Electrode	:	Glass electrode	GE-103B
	:	Reference electrode	RE-201Z
		(Inner solution: Saturated sodium perchlorate in glacial acetic acid)	
	*	Instead of glass and reference electrodes, the following glass-reference combination electrodes can also be used.	
	:	GR-513BZ (for non-aqueous titration, movable sleeve type)	

(2) Reagents

Titrant	:	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.

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

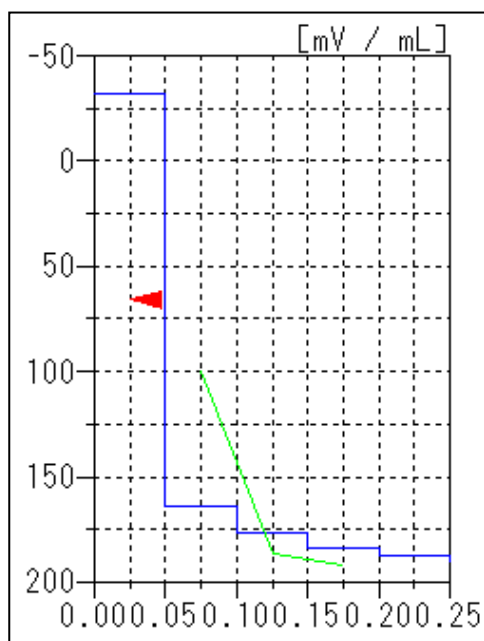
Cndt No.	1	ConstantNo.	1	Mode No.	19
Method	Auto	Size	0 g	Pre Int	0 sec
Buret No.	1	Blank	0 mL	Del K	0
Amp No.	1	Molarity	0 mol/L	Del Sens	0 mV
D. Unit	mV	Factor	0	Int Time	5 sec
S- Timer	10 sec	K	0	Int Sens	3 mV
C.P. mL	0 mL	L	0	BrT Speed	2
T- Timer	0 sec	Unit	mL	Pulse	40
D.P. mL	0 mL	Formula	D		
End Sens	300	Digits	3		
Over mL	0.2 mL				
Max. Vol.	2 mL				

Measurement of sample

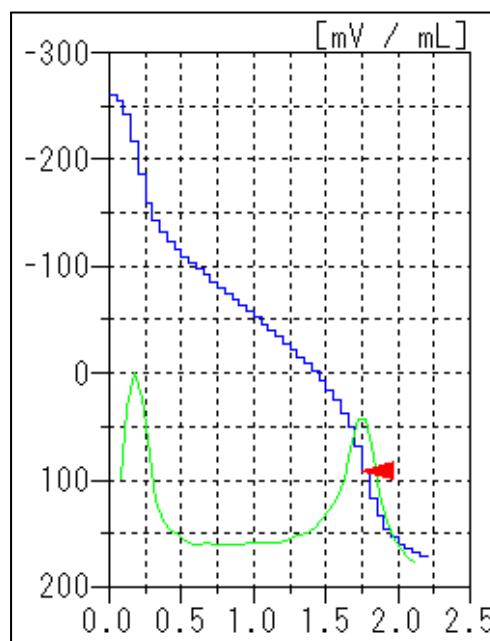
Cndt No.	2	ConstantNo.	2	Mode No.	8
Method	Auto	Size	0 g	Pre Int	0 sec
Buret No.	1	Blank	0.025 mL	Del K	5
Amp No.	1	Molarity	0.1 mol/L	Del Sens	0 mV
D. Unit	mV	Factor	1.010	Int Time	5 sec
S-Timer	10 sec	K	56.1	Int Sens	3 mV
C.P. mL	0 mL	L	0	BrT Speed	2
T- Timer	0 sec	Unit	mg/g	Pulse	40
D.P. mL	0.5 mL	Formula	(D-B)*K*F*M/S		
End Sens	300	Digits	3		
Over mL	0.2 mL				
Max. Vol.	20 mL				

Measurement results

Measurement of blank			Measurement of sample			
Number of measurement	Size (g)	Titrant volume (mL)	Number of measurement	Size (g)	Titrant volume (mL)	Base number (mgKOH/g)
1	—	0.025	1	0.9590	1.747	10.174
2	—	0.025	2	0.9708	1.764	10.150
Avg.		0.025	平均值			10.162
Difference between repetitive results					:	0.024
Repeatability limit					:	0.508
Reproducibility (Inflection)= $X \times 0.05$ (Within 5% of average)						
X : The average of the two test results						



Measurement of blank



Measurement of sample

Examples of titration curves

5. Note

(1) Electrode

In this titration, a glass electrode (GE-103B) with a low-resistance glass membrane was used. This electrode has improved response due to its reduced internal resistance, and is expected to provide more stable results, especially for non-aqueous neutralization titrations.

In addition, a movable sleeve type glass-reference combination electrode (GR-513B) can also be used in this titration instead of the glass electrode and reference electrode. Note that the internal solution must be replaced with a saturated sodium perchlorate glacial acetic acid as described in ASTM D2896, and it is recommended to leave it to stand overnight after replacement.

Repeated titrations over a long period of time reduce the response and electromotive force to the glass electrode, so the electrode should be periodically immersed in water to activate it.

(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 and the composite electrode GR-513BZ are 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 (or GR-513BZ) 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 : ASTM D 2896, Petroleum products, Base number, Potentiometric titration, Perchloric acid titration