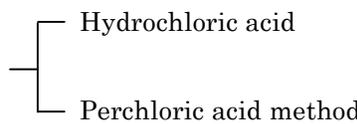


AQUACOUNTER Application Sheet	COM series	DATA No. L3	1st edition
Petroleum Products	Measurement of total base value in lubricants (perchloric acid method)		

1. Measurement outline

The total base value of lubricants is one of the important indices for evaluating the quality of lubricants. The method for the total base value is stipulated in JIS K 2501, and it is expressed as milligrams (mg) of potassium hydroxide (KOH) equivalent for the hydrochloric acid or perchloric acid required for neutralizing the total basic components contained in 1g of sample. The components for total base value are organic and inorganic bases, amino compounds, salts of weak acids (soaps), basic salts of polyacidic bases, and salts of heavy metals. The methods for total base value are hydrochloric acid titration method and base value-perchloric acid titration method (hereinafter called perchloric acid method). The following lists the related measurement method:

- JIS K 2501: Total base value 
 - Hydrochloric acid
 - Perchloric acid method
- ISO 3771-1994: Base value test method by potentiometric titration method (perchloric acid method)
- ASTM D2896-1998: Base value test method by potentiometric titration method (perchloric acid method)
- ASTM D4739-1996: Base value test method by potentiometric titration method (hydrochloric acid method)

This section introduces an example in which the total base value of diesel engine oil (used) was measured by perchloric acid method.

In this method, the sample volume corresponding to total base value is weighed precisely, and is added and dissolved in a mixture of chlorobenzene and acetic acid. Then electrodes are immersed for titration by perchloric acid titrant (acetic acid solution). For the samples which have well-defined inflection points, end points are taken at well-defined inflections. When the inflection points are not clear, it is measured by the back titration method.

2. Reagents and Electrodes

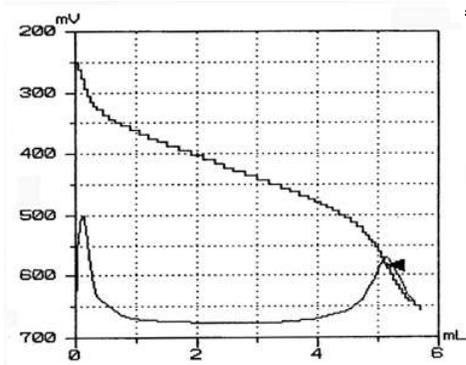
(1) Reagents	Titrant	0.1mol/L perchloric acid titrant (acetic acid solution)
	Titration solvent	Chlorobenzene : acetic acid (2 : 1) mixture solution, 120mL used for 1 measurement Note) Toluene can also be used instead of chlorobenzene.
(2) Electrodes	Indicator electrode	*Glass electrode GE-101B to IE-1 jack
	Reference electrode	*Reference electrode RE-201 (with saturated sodium perchlorate in acetic acid) to RE-1 jack
*standard accessories		

3. Measurement conditions example (for COM-1600S)

Master File No.1	
Condition file: 1	
Parameters for Condition file 1	
Method	AUTO
Buret No.	1
Meas Unit	mV
S-Timer	10 sec
CP mL	0 mL
DP mL	1.00 mL
Direction	N/A
End Sens	100
Over mL	0.50 mL
Max Vol	20 mL
Mode No.	4
Unit	mg/g
Formula	$(D-B) \times K \times F \times M/S$
Blank	BLANK measurement value 0
Molarity	0.1
Factor	Titre of the titrant
K	56.1 (as KOH)

Mode No.22	
Pre Int	0 sec
Del K	9
Del Sens	0 mV
Int Time	5 sec
Int Sens	5 mV
Brst Speed	2
Pulse	40

4. Measurement example



Measurement results on total base value by perchloric acid method

Sample No.	Sample volume (g)	Titration value (mL)	Total base value (mg/g)
1	1.6242	5.119	17.717
2	1.5647	4.978	17.883
3	1.6149	5.123	17.833
Avg.			17.81 mg/g
Std. Dev.			0.09 mg/g
C.V.			0.48 %

]

5. Outline

(1) Electrode activation and maintenance

After repeating several measurements, the response of the glass electrode may deteriorate or the electromotive force may be reduced. Thus, electrodes shall be immersed in water for 5 minutes to activate after each measurement is completed.

(2) The effect of temperature on titer of the titrant

Organic solvent (acetic acid) is used in the titrant of this measurement, and caution is required that its change in volume in concurrence to temperature change (approximately 0.11% change at 1°C) is larger compared to normal titrants with aqueous solution. It is important that measurement is taken at a temperature as constant as possible.

If the temperature for titer evaluation of the titrant and the temperature for titration vary, it can be corrected by substituting the following titer correction formula into concentration calculation formula.

$$F = \frac{F_0}{1 + \alpha(t - t_0)}$$

F : Titer for sample titration (corrected)
F₀ : Titer at the time of titer evaluation
α : Volumetric expansion coefficient for the titrant
 (acetic acid = 1.07 × 10⁻³)
T : Temperature for sample titration
t₀ : Temperature for titer evaluation

(3) Method of preparing reference electrode

For this measurement, reference electrode RE-201 is to be changed into the reference electrode for non-aqueous titration.

- Drain out saturated KCl solution in RE-201, and wash well with acetic acid. Then prepare an acetate solution saturated with sodium perchlorate (special class reagent) and inject it into the filling port. Since the electrode potential may not stabilize immediately, it is recommended that it is used after leaving it standing for 1 entire day.

Key words

Lubricants, total base value, perchloric acid method, neutralization value test, JIS K 2501

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