

Application Note

Base number of lubricant

Industry : Petroleum

Instrument : Automatic potentiometric titrator

Measurement method : Potentiometric titration / Neutralization titration

Standards : ASTM D2896, JIS K2501

1. Scope

Base number (*BN*) of a lubricant was measured based on "ASTM D2896 Standard Test Method for Base Number of Petroleum Products by Potentiometric Perchloric Acid Titration".

A sample dissolved in mixed solvent of glacial acetic acid and chlorobenzene was potentiometrically titrated with 0.1mol/L perchloric acid acetic acid solution. An inflection point on the titration curve was regarded as the end point. *BN* of the sample was calculated from the volume of perchloric acid acetic acid solution used to titrate sample to the endpoint.

2. Precautions

- 1) When measurement of *BN* is repeated, regenerating the sensitivity of the electrodes is required for each measurement. After measurement, wash the electrodes with titration solvent and then immerse them in pure water until the indicated potential becomes stable. Before next measurement, wash the electrodes with titration solvent again.
- 2) Do not leave the electrodes to remain immersed in titration solvent.
- 3) When the glass electrode is contaminated, wash it by immersing in cold chromic acid or an alternative cleaning solution for 5 minutes.
- 4) Standardize the 0.1mol/L perchloric acid acetic acid solution more than once a week.
- 5) Because of the relatively large coefficient of volumetric expansion of organic liquids, the 0.1 mol/L perchloric acid acetic acid solution should be used within $\pm 5^{\circ}\text{C}$ of the temperature at which it was standardized.
- 6) Handle the reagents in a well ventilated room or a draft chamber.

3. Post-measurement procedure

Because the sensitivity of the electrodes is deteriorated after a series of measurements, regenerate the sensitivity of them by the method described in "2. Precautions, 1)".

4. Apparatus

Main unit : Automatic potentiometric titrator (preamplifier : STD)

Electrode : pH glass electrode

Double junction reference electrode

(inner solution : saturated NaClO₄ acetic acid solution)

Temperature compensation electrode

5. Reagents

Reagent : 0.1mol/L perchloric acid acetic acid solution

Titration solvent : acetic acid : chlorobenzene = 1 : 2

6. Procedure

- -Blank test-
- 1) Add 60mL of the titration solvent into a 200mL tall beaker.
- 2) Titrate with 0.1mol/L perchloric acid acetic acid solution to measure blank level.
- -Measurement-
- 1) Weigh 1g of the sample into a 200mL tall beaker.
- 2) Add 60mL of the titration solvent.
- 3) Titrate with 0.1mol/L perchloric acid acetic acid solution to measure BN.

7. Calculation

 $BN \text{ (mgKOH/g)} = (EP1 - BL1) \times TF \times C1 \times K1 / S$

Titer (mL) EP1

BL1 Blank level = 0.0037mL TF Factor of titrant = 0.9483

C1 Concentration conversion coefficient = 5.611mg/mL

K1 Unit conversion coefficient = 1

S Sample (g)

8. Example

-Titration parameter-

<Titr. Mode> : Auto Int. <Ctrl. Para.>

: EP Stop Number of EP <Titr. Form>

> End Sense : Auto

Gain : 1 <Titr. Para.>

Max. Volume : 0.2 (mL) (Blank test) Data Sampling : Auto

: 20 (mL) (Sample) Ctrl. Speed : Standard Channel/Unit(Ctrl.) : Ch1, mV Other Ctrl. : Standard

Channel/Unit(Ref.) : Off Auto Int. Mode : Blank (Blank test)

pH Polarity : Standard : Standard (Sample)

Titr. Type Check : No Check Stirrer Speed : 3

Direction : Auto Wait Time : 10 (s) Dose Mode : None

(The measurement parameter and the titration curve are an example of our automatic potentiometric titrator. In some titrators, parameter item may be different or another parameter item may be added.)



Sample -Titration curve--Measurement results-731 BNSample Titer (g) (mL)(mgKOH/g) 1 1.0054 1.7402 9.19 Level(Ctrl.)[mV] 185 0 mL 2 1.0213 1.7813 9.26 3 1.0379 1.8116 9.27 Mean 9.24 SD 0.04 RSD (%) 0.47 Repeatability limit* 0.46

X = Mean of the results

9. Summary

In this measurement, the results showed a good repeatability with 0.47% RSD (Relative standard deviation), and the differences of the results were within the repeatability limit regulated by ASTM D2896 and JIS K2501.

In some samples, verification of the measurement capability is required. In such case, please contact us.

10. References

- 1) ASTM D2896-11 Standard Test Method for Base Number of Petroleum Products by Potentiometric Perchloric Acid Titration
- 2) JIS K2501 : 2003 Petroleum products and lubricants Determination of neutralization number



^{*}Repeatability limit = 0.05X