

Petroleum

Total acid number of lubricant

Acid-base titration (non aqueous) by
Automatic Potentiometric Titrator

| | | |
|----------|------|--------|
| Standard | JIS | K 2501 |
| | ASTM | D 664 |

1. Abstract

Measurement of total acid number of petroleum products and lubricant is specified in JIS K 2501-1992 Petroleum Products and Lubricant – Neutralization Test Method.

A test sample is potentiometrically titrated with 0.1mol/L potassium hydroxide 2-propanol solution, and the endpoint is determined on titration curve.

Total acid number is calculated from titration volume of potassium hydroxide 2-propanol solution reaching the endpoint.

Test samples are usually dissolved in mixture of toluene and 2-propanol with a small amount of water. Petroleum products and lubricant dissolved in the mixed solvent contain such component as organic acid, inorganic acid, ester, phenol compound, lactone, resin, heavy metal salt, ammonium salt, weak acid and polybasic acid chloride.

2. Reference

- 1) JIS K 2501-2003 Petroleum Products and Lubricant – Neutralization Test Method
- 2) ASTM D 664-95 Standard Test Method for Acid Number of Petroleum Products by Potentiometric Titration

3. Cautions in measurement

- 1) Since sensitivity of glass electrode is deteriorated during the process of titration, it is necessary to regenerate the electrode after measurement. First clean it in alcohol, and then dip it in pure water for a few minutes to one hour. (depending on how much contaminated)
- 2) Standard concentration of potassium hydroxide 2-propanol is 0.0005mol/L or less, and it is necessary to check the concentration of reagent from time to time.

4. Post-measurement care

The sensitivity of glass electrode is deteriorated after a series of measurements for the day, and the electrode needs regeneration by the method above mentioned in 3. Cautions in measurement.

5. Test equipment

Main unit: Automatic potentiometric titration system (preamplifier: STD)

Electrode: Glass electrode H-171 [№ 98100H171] option

Reference electrode R-173 [№ 98100R173] option
(junction liquid 3.33M⁻ potassium chloride)

Temperature compensation T-171 [№ 98100T171] standard equipped

6. Reagent

Reagent : 0.1mol/L potassium hydroxide 2-propanol (f=1.000)

Solvent : Mixture of toluene: pure water :2-propanol (500 : 5 : 495)

7. Test procedure

—Pretreatment—

1) Mix toluene: water: 2-propanol by ratio 500:5:495, and transfer 100mL to a 200mL beaker.

2) Perform a blank test, and obtain blank level.

—Measurement—

1) Transfer approx. 10g sample to a 200mL beaker.

2) Add the above prepared solvent, and make it 100mL.

3) Titrate with the above reagent to obtain total acid number.

8. Formula

Total acid number (mg / g) = (EPl - BLl) × TF × Cl × Kl / S

EPl : Titration volume (mL)

BLl : Blank level (0.052mL)

TF : Factor of reagent (1.000)

Cl : Concentration conversion coefficient (5.611 g/mL)
(equivalent to potassium hydroxide in 1mL of 1mol/L potassium hydroxide 2-propanol solution)

Kl : Unit conversion coefficient (1)

S : Sample size (g)

9. Example of measurement

– Ambient condition –

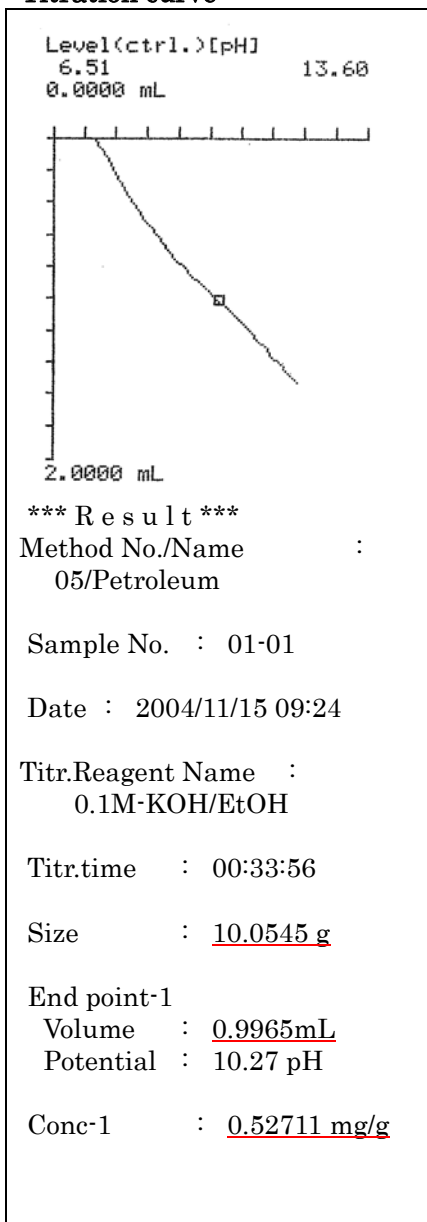
| | | |
|----------------------------|-----------------|------------------|
| Room temperature : 22.5 °C | Humidity : 47 % | Weather : Cloudy |
|----------------------------|-----------------|------------------|

The below parameters and titration curve are printed out when titrated by AT-610 unit.

-Titration parameter-

| | |
|----------------------------|-------------------------|
| Model : AT-610 | [Calculation parameter] |
| Method No. : 01 | Calc. Type : Sample |
| Titration mode : Petroleum | CO1 : On |
| Titration form : EP Stop | Unit : mg/g |
| [Titration parameter] | Formula : |
| Burette No. : 1 | (EP1-BL1)*TF*K1*C1/S |
| Max volume : 10.00 mL | EP position : EP1 |
| Channel, Unit | Decimal : 5 |
| Ctrl. : Ch1, pH | Fraction : Half adjust |
| Ref. : Off | Evaluation : Off |
| pH polarity : | Active constant |
| Standard | C1 : 5.61100 |
| Direction : Auto | K1 : 1.00000 |
| Wait time : 0 s | Temp.comp. : Off |
| Dose mode : None | [Titration constant] |
| [Control parameter] | TF : 1.00000 |
| Number of EP : 1 | [Blank list] |
| End sense : Auto | Blank1 : 0.05200 |
| Gain : 1 | |
| Data sampling : Std. | |
| Ctl. speed mode : Set | |
| Limit time : 0 s | |
| Integral time : 60 s | |
| Start Limit : 0 s | |
| 1st Dose vol. : 0.10 mL | |
| 2nd Dose vol. : 0.05 mL | |
| Stability : 5.0 | |
| mV/min | |
| dE(1st-2nd) : 7.00 pH | |
| Other control : Std. | |
| Stir. Speed : 4 | |

-Titration curve-



Meaning of parameters on printout:

« Titration parameter »

Burette No.: burette number used in titration / Max Volume: total titration volume

Channel, Unit: selection of detector number and potential unit for detection

pH polarity: direction of pH potential / Direction: of titration / Wait Time: waiting time before titration starts

Dose mode: the way of fixed dose

« Control parameter »

Number of EP: the number of endpoints / End sense: EP sense direction / Gain: sensitivity of detection signal

Data sampling: condition / Ctl. speed mode: control speed mode

Other control: other controls / Stir. Speed: of stirrer

« Calculation parameter »

Calc. Type: calculation type / CO1: concentration formula 1 / Unit: unit of results

BL1: blank level / EP1: first endpoint / TF: reagent factor / C1(mg/mL): concentration conversion coefficient

K1: unit conversion coefficient

S: sample size / EP position: for calculation / Decimal: number of digits after decimal point in calculation

Fraction: rounding fraction / Evaluation: of calculated results

–Measurement results–

| n | Sample (g) | Titrated (mL) | Total acid number (mg/g) | Statistics | |
|---|----------------|---------------|--------------------------|--------------|--------------|
| | | | | Mean | SD |
| 1 | <u>10.0545</u> | <u>0.9965</u> | <u>0.52711</u> | 0.53572 mg/g | 0.02641 mg/g |
| 2 | 10.0259 | 0.9516 | 0.50347 | | |
| 3 | 10.0058 | 1.0090 | 0.53669 | | |
| 4 | 10.0270 | 1.0824 | 0.57663 | | |
| 5 | 10.0247 | 1.0073 | 0.53472 | | |
| | | | | RSD | 4.92981 % |

* The data were obtained from 5 tests of the same sample.

* Red underline shows the data from page 3/4.

10. Summary

Total acid number means potassium hydroxide in mg required to neutralize total acid components in 1g of lubricating oil.

In spite of the total acid number of this sample as small as 0.5mg/g, it showed a good repeatability with less than 5% relative standard deviation.

The automatic potentiometric titration system provides stable and reliable measurements. Total acid number of lubricant is measured by any of the models shown below.

【AT-610】



Awarded Product of Supreme Technology from Kyoto City

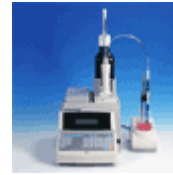
- Easy key entry by touch panel of large color LCD (8-inch wide)
- Simultaneous titration in parallel
- Both potentiometric and Karl Fischer moisture titration (coulometric·volumetric) can be performed at a time.

【AT-510】



- Compact and cost performance model
- PC card expands data memory for convenience and versatility.

【AT-500N-1】



- Low cost and high performance
- Easy view with back light LCD
- GLP/GMP conformed model

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