Sheet No.
GT-310-PE-045E Energy & Petroleum Products

Base Number Analysis of Petroleum Products - Perchloric Acid Titration (ASTM D2896) Direct Titration - Alternative Solvent

Related standard: ASTM D2896-15 Standard Test Method for Base Number of Petroleum Products by Potentiometric Perchloric Acid Titration

Outline

ASTM D2896 is a standard for determination of base number by dissolving a sample in an anhydrous mixture of chlorobenzene and glacial acetic acid. The appendix states that xylene can be used instead chlorobenzene. It also notes that the addition of approximately 10 % acetone to the alternative titration solvent reduces electrode noise.
Testing can be performed as manual titration using a buret or automatic recording titration using an automatic buret in accordance with Procedure A (120 mL).
For this application sheet, commercial lubricating oil was measured using automatic titration with an alternative solvent.

Principle

The basic constituents in the sample are neutralized using perchloric acid. Titration is performed at a speed of 1.0 mL/min maximum while recording the potential difference between a glass electrode and the reference electrode.
Then the inflection point is detected as the end point. When there is no inflection point or only a very poor one, perform back-titration to detect the end point (see application sheet no. GT-310-PE-046E).

Apparatus

Automatic titrator:	GT-310
Electrodes:	GLASS ELECTRODE, L=105 (GTPH1B), REFERENCE ELECTRODE SLEEVE L=105 (SLEEVE TYPE) (GTRS10B) (Inner solution: sodium perchlorate electrolyte)
Buret cassette:	BURET CASSETTE UNIT WITH TEMPERATURE SENSOR, 20mL (GTECST)

Reagents

[Titrant]
[Reagents]

- 0.1 mol/L Perchloric acid standard solution in acetic acid
- Titration solvent: Add one volume of glacial acetic acid to two volumes of xylene.
- Acetone (special grade)
- Sodium perchlorate electrolyte: saturated solution of sodium perchlorate ($NaClO_4$) in glacial acetic acid
- Potassium hydrogen phthalate (certified reference material)

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Analytical Procedure

[Testing of electrodes]

1. A well-stirred mixture of 100 mL of glacial acetic acid plus 0.2 g of potassium hydrogen phthalate was prepared. The screen of multi controller was switched to the potential monitor. The electrodes were dipped into the solution, and the potential was recorded (the potential was recorded when it changed by less than 5 mV/min).
2. The electrode was rinsed with xylene. 1.5 mL of 0.1 mol/L perchloric acid solution was added in 100 mL of acetic acid using the GT-310BRT. The electrode was immersed in the mixture and the potential was then recorded (the potential was recorded when it changed less than 5 mV/min).
3. It was confirmed that the potential difference between steps 1 and 2 was at least 300 mV.

[Blank titration]

1. 120 mL of titration solvent was added into a 250 mL beaker, and 10 mL of acetone was added to it.
2. It was titrated with 0.1 mol/L solution of perchloric acid in acetic acid.*1

[Sample titration]

1. A sample was weighed into a 150 mL beaker, referring to Table 1, and 120 mL of titration solvent was added to dissolve it. 10 mL of acetone was also added.*2
2. It was titrated with 0.1 mol/L solution of perchloric acid in acetic acid.*1

Table 1 Sample weighing (Procedure A)*3

Approximate weight of sample (g)	Sample weight (g)	Precision of weighing (g)
28/Expected base number	10 to 20	0.05
	5 to 10	0.02
	1 to 5	0.005
	0.25 to 1.0	0.001
	0.1 to 0.25	0.0005

*1: The electrode was rinsed and immersed in purified water for at least 5 minutes before each titration.
*2: If the solution of the sample proves difficult, dissolve it in 80 mL of xylene in the titration beaker, then add 40 mL of glacial acetic acid..
*3: For procedure A, maximum of 20 g should be taken for analysis.

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[Calculation]

Base number (mgKOH/g) = (A1 − B) × Q × f × FW / W

With temperature correction*1

Base number (mgKOH/g) = [A1 × {1 ＋ 0.001 × (X1 − t)} − B × {1＋0.001 × (X1 − X2)}] × Q × f × FW / W

A1: Perchloric acid solution used to titrate the sample to the end point: inflection point on the titration curve (mL)
B: Volume of 0.1 mol/L perchloric acid solution used to titrate the solvent to the end point for blank titration (= 0.0117 mL)
Q: Concentration of 0.1 mol/L perchloric acid solution (= 0.1 mol/L)
f: Factor of 0.1 mol/L perchloric acid solution (= 0.992)
FW: Molar mass of potassium hydroxide (= 56.1 g/mol)
W: Sample weight (g)
X1: Temperature of 0.1 mol/L perchloric acid solution at standardization (°C)
t: Temperature of 0.1 mol/L perchloric acid solution at sample titration (°C)
X2: Temperature of 0.1 mol/L perchloric acid solution at blank titration (°C)

*1: Use this formula when the temperature difference of the titrant exceeds 5 °C between the time of standardization and use. The calculation formula with temperature correction was not used in this application sheet, as the temperature difference was within 5 °C.

*2: Standardization was performed in accordance with ASTM D2896 (see application sheet no. GT-310-PE-047E).

Other Requirements

- Confirm reagent labels and safety data sheets for safety.
- Wear safety goggles, gloves, and/or other safety equipment when handling reagents.
- Replace the reference electrode inner solution at regular intervals.
- It was confirmed that the buret with temperature sensor had an accuracy of ± 0.02 mL.
- The standard states that precision of the alternative solvent is worse than the original. However, it does not mention the precission when acetone is added.

Measurement Results

Sample	Sample amount (g)	Titration volume (mL)	Base number (mgKOH/g)	Average (mgKOH/g)	RSD (%)
Automotive engine oil	2.725	5.1092	10.4	**10.3**	**1.2**
	2.745	5.0858	10.3		
	2.730	5.0014	10.2		

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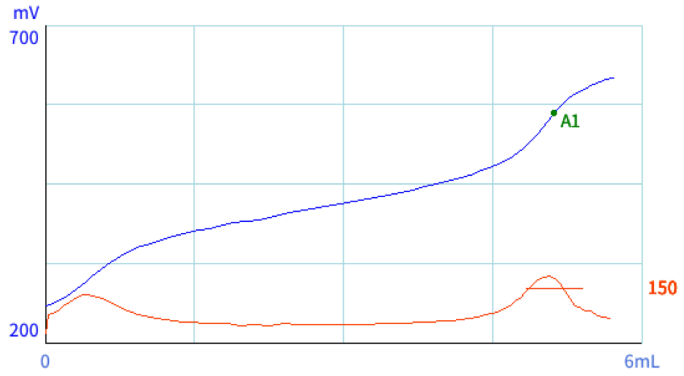

Sample name:	Automotive engine oil	
End point:	5.1092 mL	561.4 mV
Start of measurement:	0.000 mL	259.1 mV
End of measurement:	5.709 mL	617.8 mV
Measurement time:	24 min 26 s	
Start temperature:	25.6 °C	

■ Default values were used for parameters not listed below.

	Blank titration	Sample titration
Stirrer speed:	2.5	2.5
Titration mode:	TAN/TBN standard method: OIL-A	TAN/TBN standard method: OIL-A
Detector:	mV1	mV1
Initial wait time:	60 s	90 s
Drop volume control:	Individual	Individual
Max. drop volume:	50 µL	200 µL
Min. drop volume:	10 µL	50 µL
Stability criteria:	Individual	Individual
Delta potential:	1.0 mV	1.0 mV
Delta time:	12 s	12 s
E1:	Inflection/Set-Potential	Inflection/Set-Potential *2
E1 potential:	End point potential of sample titration*1	490 mV
E1 potential width:	0 mV	140 mV
E1 derivative threshold:	2,000 mV/mL	150 mV/mL*3
E1 evaluation points:	3	5
Max. titration volume:	10 mL	20 mL
End derivative:	50 mV/mL	50 mL/mV

*1: Because the blank titration volume is defined as “the volume corresponding to E ($HClO_4$ solution used to titrate the sample to the inflection point on the titration curve) for blank titration at same potential as sample.”
*2: If no inflection point is detected, the E1 potential is used to detect the end point. If the potential for the End point 1 (A1) is the same as the E1 potential, the titration amount should be treated as 0.
*3: Adjust as necessary.

* This application sheet is provided as reference, and does not assure the measurement results. Please consider the analysis environment, external factors and sample nature for optimal conditions before the measurement.