Sheet No.
GT-310-PE-032E Energy & Petroleum Products

Standardization of 0.1 mol/L Perchloric Acid Solution in Acetic Acid (JIS K 2501)

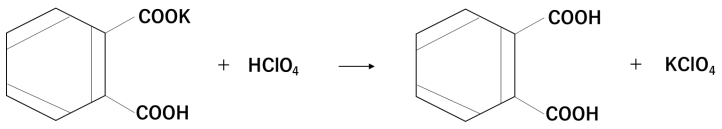
Related standard: JIS K 2501:2003 Petroleum products and lubricants - Determination of neutralization number 9. Potentiometric titration (Base number, perchloric acid titration)

Outline

In “Determination of neutralization number - 9. Potentiometric titration (Base number, perchloric acid titration)” of JIS K 2501, 0.1 mol/L perchloric acid solution in acetic acid is used as the titrant. The standard states that standardization should be performed at least once a week. The standardization method is divided into Procedure A (120 mL) and Procedure B (60 mL) depending on the amount of titration solvent used.
For this application sheet, standardization was performed using automatic titration according to Procedure B, and results were obtained with a relative standard deviation (RSD) of less than 1 %.

Principle

Potassium hydrogen phthalate is neutralized using perchloric acid. Titration is performed while recording the potential difference between a glass electrode and the reference electrode.
The titration is performed by 0.1 mol/L perchloric acid solution in acetic acid at a speed of 1.0 mL/min maximum, and the inflection point is detected as the end point.
The factor is calculated from the amount of potassium hydrogen phthalate and the volume of 0.1 mol/L perchloric acid solution in acetic acid used up to the end point.



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)

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Reagents

[Titrant]
- Perchloric acid solution in acetic acid 0.1 mol/L (for non-aqueous titration)

[Reagents]
- Potassium hydrogen phthalate (certified reference material): Dried at 120 °C for 2 hours, then cooled
- Acetic acid (special grade)
- Chlorobenzene (special grade)
- Sodium perchlorate electrolyte: saturated solution of sodium perchlorate monohydrate (special grade) in acetic acid. Ensure that excess crystals remain.

Analytical Procedure

[Blank titration]
1. 20 mL of acetic acid was added to a 150 mL tall beaker, and 40 mL of chlorobenzene was added to it.
2. It was titrated with a titrant of perchloric acid in acetic acid.

[Sample titration]
1. 0.05 to 0.1 g of potassium hydrogen phthalate was weighed in 150 mL tall beaker with accuracy of 0.1 mg. 20 mL of warm acetic acid* was added to dissolve it, and 40 mL of chlorobenzene was also added.
2. After cooling to room temperature, it was titrated with a solution of perchloric acid in acetic acid.

* Acetic acid at 50 °C was used based on JIS K 0050 defines warm water as being 40 to 60 °C.

[Calculation]

Factor = 1,000 × W / FW / (A1 − B) / Q

1,000:	Unit conversion factor for mL to L
W:	Mass of potassium hydrogen phthalate (g)
FW:	Molar mass of potassium hydrogen phthalate (= 204.23 g/mol)
A1:	Volume of 0.1 mol/L perchloric acid solution in acetic acid required for sample titration (mL)
B:	Volume of 0.1 mol/L perchloric acid solution in acetic acid required for blank titration (= 0 mL)
Q:	Concentration of 0.1 mol/L perchloric acid solution in acetic acid (= 0.1 mol/L)

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 and outer solutions at regular intervals (at least once a week).
- Before use, it was confirmed that the buret with temperature sensor had a accuracy of 20 mL ± 0.02 mL.

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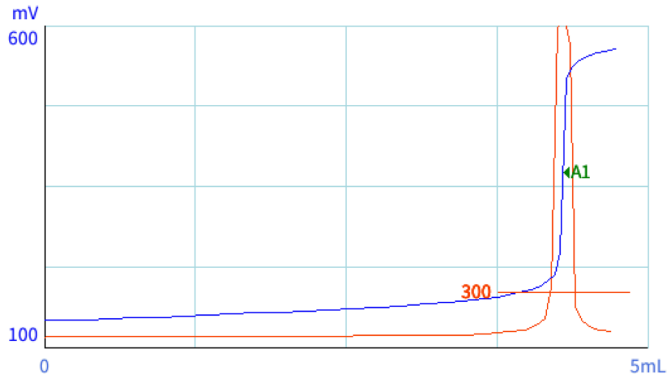
Measurement Results

Sample	Sample amount (g)	Titration volume (mL)	Factor	Average	RSD (%)
Potassium hydrogen phthalate	0.0875	4.2933	0.998	**0.997**	**0.1**
	0.0864	4.2477	0.996		
	0.0922	4.5286	0.997		



Sample name:	Potassium hydrogen phthalate	
End point:	4.2933 mL	375.5 mV
Start of measurement:	0.000 mL	143.6 mV
End of measurement:	4.724 mL	564.0 mV
Measurement time:	13 min 11 s	
Start temperature:	22.6 °C	

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■ Default values were used for parameters not listed below.

	Blank titration*	Sample titration*
Stirrer speed:	2.5	2.5
Detector:	mV1	mV1
Titration mode:	TAN/TBN standard method: OIL-A	TAN/TBN standard method: OIL-A
Initial wait time:	60 s	60 s
Drop control:	Individual	Individual
Max. drop volume:	50 µL	200 µL
Min. drop volume:	10 µL	50 µL
Stability criteria:	Individual	Individual
Delta potential:	1 mV	1 mV
Delta time:	12 s	12 s
E1:	Inflection/Set-Potential	Inflection/Set-Potential
E1 potential:	500 mV	500 mV
E1 derivative threshold:	250 mV	250 mV
E1 differential evaluation value:	300 mV/mL	300 mV/mL
E1 evaluation points:	3	5
Max. titration volume:	10 mL	20 mL
End derivative:	50 mV/mL	50 mV/mL

* If no inflection point is detected, the E1 potential is used to detect the end point. If the potential for the titration amount (A1) is the same as the E1 potential, the titration amount is treated as 0.

*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.