

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

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

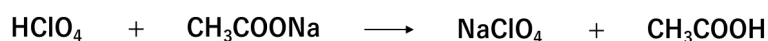
Outline

In “Determination of neutralization number - 9. Potentiometric titration (Base number, perchloric acid titration)” of JIS K 2501, 0.1 mol/L sodium acetate solution in acetic acid is used as the titrant for back titration. The standard states that standardization should be performed at least once a week. The standardization is performed by neutralization titration with 0.1 mol/L perchloric acid solution in acetic acid. 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

In sample titration, perchloric acid is neutralized using sodium acetate. Blank titration is used to correct for the amount of perchloric acid consumed by the titration solvent. 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 sodium acetate solution in acetic acid at a speed of 1.0 mL/min maximum. The inflection point is detected as the end point. The factor is calculated from the amount of 0.1 mol/L perchloric acid solution in acetic acid and the volume of titrant 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)

Reagents

[Titrants]	<ul style="list-style-type: none">■ Sodium acetate solution in acetic acid 0.1 mol/L (for non-aqueous titration)■ Perchloric acid solution in acetic acid 0.1 mol/L (for non-aqueous titration)
[Reagents]	<ul style="list-style-type: none">■ Titration solvent: 1 part acetic acid (special grade) mixed with 2 parts chlorobenzene (special grade)■ Sodium perchlorate electrolyte: saturated solution of sodium perchlorate monohydrate (special grade) in acetic acid (special grade)

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Analytical Procedure

[Blank titration]

1. 60 mL of titration solvent was added to a 150 mL tall beaker.
2. It was titrated with a solution of perchloric acid in acetic acid. *

[Sample titration]

1. 4.00 mL of 0.1 mol/L perchloric acid solution in acetic acid was added to a 150 mL tall beaker using a GT-310BRT unit, and dissolved in 60 mL of titration solvent.
2. It was titrated with a solution of perchloric acid in acetic acid. *

* Here, two GT-310BRT units were used. If using one GT-310BRT unit, replace the reagent cassette.

[Calculation]

$$\text{Factor} = (4 - B) \times X1 / A1$$

With temperature correction^{*1}

$$\text{Factor} = [4 \times \{1 + 0.001 \times (X2 - X3)\} - B \times \{1 + 0.001 \times (X2 - X4)\}] \times X1 / A1$$

- 4: Volume of 0.1 mol/L perchloric acid solution in acetic acid sample (mL)
B: Volume of 0.1 mol/L perchloric acid solution in acetic acid used for blank titration (= 0 mL)
X1: Factor of 0.1 mol/L perchloric acid solution in acetic acid (= 0.994)^{*2}
A1: Volume of 0.1 mol/L sodium acetate solution in acetic acid used for sample titration (mL)
X2: Temperature of 0.1 mol/L perchloric acid solution in acetic acid at standardization (°C)
X3: Temperature of 0.1 mol/L perchloric acid solution in acetic acid after titrating 4 mL (°C)
X4: Temperature of 0.1 mol/L perchloric acid solution in acetic acid 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 here, as the temperature difference was within 5 °C.

*2: Standardization was performed in accordance with JIS K2501 (see application sheet no. GT-310-PE-033E).

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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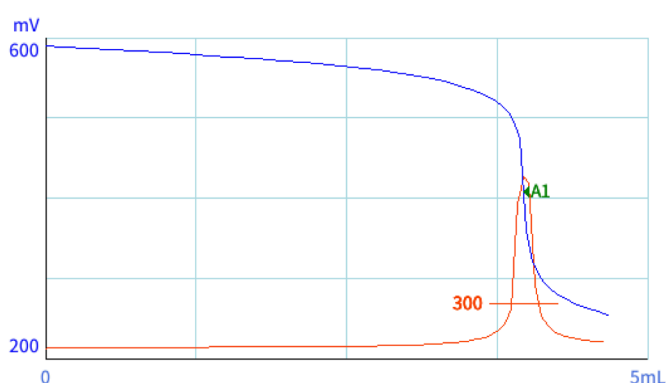
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Measurement Results

Sample	Sample amount (mL)	Titration volume (mL)	Factor	Average	RSD (%)
0.1 mol/L perchloric acid solution in acetic acid	4	3.9629	1.0033	1.0031	0.0
		3.9631	1.0033		
		3.9646	1.0029		



Sample name: 0.1 mol/L perchloric acid solution in acetic acid
 End point: 3.9629 mL 408.3 mV
 Start of measurement: 0.000 mL 589.7 mV
 End of measurement: 4.662 mL 256.0 mV
 Measurement time: 15 min 07 s
 Start temperature: 22.3 °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
Titrant:	0.1 mol/L HClO ₄ /AcOH	0.1 mol/L AcONa/AcOH
Buret number:	1	2
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 potential width:	250 mV	250 mV
E1 derivative threshold:	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 endpoint. 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.