Sheet No.
GT-310-PE-044E Energy & Petroleum Products

Standardization of 0.1 mol/L Sodium Acetate Solution in Acetic Acid (ASTM D2896)

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

Outline

In the ASTM D2896, 0.1 mol/L sodium acetate solution in acetic acid is used as the titrant for back titration. The standardization method of the solution is divided into Procedure A (120 mL) and Procedure B (60 mL) depending on the amount of titration solvent used. Testing can be performed as manual titration using a buret or automatic recording titration using an automatic buret.
For this application sheet, standardization was performed using automatic titration according to Procedure B, and measurement 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 at a speed of 1.0 mL/min maximum, while recording the potential difference between a glass electrode and the reference electrode. The inflection point is detected as the end point. The factor is calculated from the amount of 0.1 mol/L perchloric acid solution and the volume of titrant used up to the end point.

$$HClO_4 + CH_3COONa \longrightarrow NaClO_4 + CH_3COOH$$

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]
- 0.1 mol/L Sodium acetate solution in acetic acid
- 0.1 mol/L Perchloric acid solution in acetic acid

[Reagents]
- Titration solvent: 2 parts chlorobenzene (special grade) mixed with 1 part acetic acid (special grade)
- Sodium perchlorate electrolyte: saturated solution of sodium perchlorate ($NaClO_4$) in glacial acetic acid

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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. 60 mL of titration solvent was added to a 150 mL tall beaker.
2. 4.00 mL of 0.1 mol/L perchloric acid solution in acetic acid was added and titrated with 0.1 mol/L sodium acetate solution in acetic acid using the GT-310. *

*: In this application sheet, two GT-310BRT units were used for the sample titration of step 2. When using just one GT-310BRT unit, replace the cassette after adding 4mL of 0.1 mol/L perchloric acid solution with a cassette of 0.1 mol/L sodium acetate solution.

[Calculation]

Factor = (4 − B) × fp1 / A1

With temperature correction*1

Factor = [4 × {1 + 0.001 × (X2 − t)} − B × {1 + 0.001 × (X2 − X3)}] × fp1 / A1

4: Volume of 0.1 mol/L perchloric acid solution (= 4 mL)
B: Volume of 0.1 mol/L perchloric acid solution used for blank titration (= 0 mL)
fp1: Factor of 0.1 mol/L perchloric acid solution (= 0.993)*2
A1: Volume of titrant used for sample titration (mL)
X2: 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)
X3: 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. GT310-PE-043E).

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.
- Before use, it was confirmed that the buret with temperature sensor had an accuracy of ± 0.02 mL.
- The electrode should be rinsed and immersed in purified water for at least 5 minutes before each titration.

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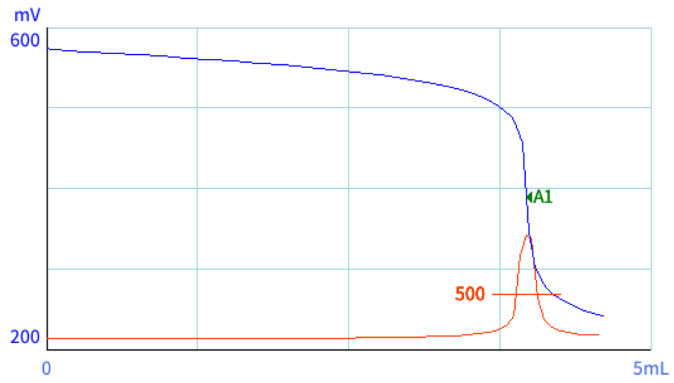
Measurement Results

Sample	Sample amount (mL)	Titration volume (mL)	Factor	Average	RSD (%)
0.1 mol/L perchloric acid solution in acetic acid	4	3.9646	1.002	**1.002**	**0.1**
		3.9654	1.002		
		3.9564	1.004		



Sample name:	0.1 mol/L perchloric acid solution in acetic acid	
End point:	3.9646 mL	389.2 mV
Start of measurement:	0.000 mL	573.7 mV
End of measurement:	4.597 mL	242.6 mV
Measurement time:	12 min 30 s	
Start temperature:	25.3°C	

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■ Default values were used for parameters not listed below.

	Blank titration	Sample titration
Stirrer speed:	2.5	2.5
Titration mode:	TAN/TBN standard method: OIL-A	TAN/TBN standard method: OIL-A
Detector:	mV1	mV1
Preset 1:	None	Preset-V*1
P1 buret number:		1
P1 titrant:		0.1 M HClO4/AcOH
P1 injection volume:		4 mL
Titrant:	0.1 M HClO4/AcOH	0.1 M AcONa/AcOH
Buret number:	1	2
Initial wait time:	60 s	120 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 [mV]	1 mV
Delta time:	12 s	12 s
E1:	Inflection/Set-Potential*2	Inflection/Set-Potential*2
E1 potential:	450 mV	450 mV
E1 potential width:	250 mV	250 mV
E1 derivative threshold:	500 mV/mL	500 mV/mL
E1 evaluation points:	3	5
Max. titration volume:	10 mL	20 mL
End derivative:	50 mV/mL	50 mV/mL

*1: Set because step 2 was automated using two GT-310BRT units.

*2: If no inflection point is detected, the E1 potential is used to detect the endpoint. If the potential for end point 1 (A1) is the same as the E1 potential, the titration amount should be 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.