

Sheet No.

GT-310-PE-048E

Energy & Petroleum Products

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

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

Outline

In ASTM D2896, 0.1 mol/L sodium acetate solution in acetic acid is used as the titrant for back titration in determining base number. This application sheet describes standardization using an alternative solvent. The appendix of ASTM D2896 describes the use of xylene as an alternative solvent to chlorobenzene. It also notes that approximately 10 % of acetone can be added to the alternative solvent to reduce 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, standardization was performed using automatic titration, 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 titrant (0.1 mol/L sodium acetate solution in acetic acid), and 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]	■ 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 xylene (special grade) mixed with 1 part acetic acid (special grade) ■ Sodium perchlorate electrolyte: saturated solution of sodium perchlorate (NaClO_4) in glacial acetic acid ■ Acetone (special grade)

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Analytical Procedure

[Blank titration]

1. 120 mL of titration solvent was added to a 250 mL tall beaker, and 10 mL of acetone was added to it.
2. It was titrated with a solution of perchloric acid in acetic acid.

[Sample titration]

1. 120 mL of titration solvent was added to a 250 mL tall beaker, and 10 mL of acetone was added to it.
2. 8.00 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 automation of sample titration step 2.

When using just one GT-310BRT unit, replace the cassette after adding 8 mL of 0.1 mol/L perchloric acid solution in acetic acid with a cassette of 0.1 mol/L sodium acetate solution in acetic acid.

[Calculation]

$$\text{Factor} = (8 - B) \times \text{fp1} / A1$$

With temperature correction*1

$$\text{Factor} = [8 \times \{1 + 0.001 \times (X2 - t)\} - B \times \{1 + 0.001 \times (X2 - X3)\}] \times \text{fp1} / A1$$

8: Volume of 0.1 mol/L perchloric acid solution (= 8 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 (= 0.992)*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 here, 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 and outer solutions at regular intervals (at least once a week).
- 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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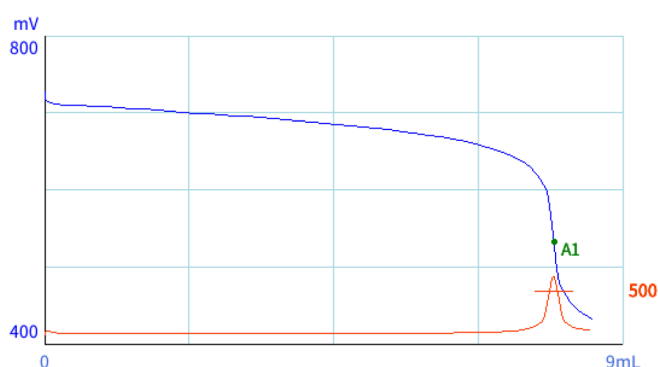
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Measurement Results

Sample	Sample amount (mL)	Titration volume (mL)	Factor	Average	RSD (%)
0.1 mol/L perchloric acid solution in acetic acid	8	7.9129	1.003	1.002	0.2
		7.9055	1.004		
		7.9312	1.001		



Sample name: 0.1 mol/L perchloric acid solution in acetic acid

End point: 7.9129 mL 533.6 mV

Start of measurement: 0.000 mL 718.3 mV

End of measurement: 8.506 mL 434.2 mV

Measurement time: 16 min 46 s

Start temperature: 25.9°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 HClO ₄ /AcOH
P1 injection volume:		8 mL
Titrant:	0.1 M HClO ₄ /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	1 mV
Delta time:	12 s	12 s
E1:	Inflection/Set-Potential ^{*2}	Inflection/Set-Potential ^{*2}
E1 potential:	480 mV	480 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 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.

* 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.