

Sheet No.

GT-310-PE-047E

Energy & Petroleum Products

Standardization of 0.1 mol/L Perchloric Acid 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

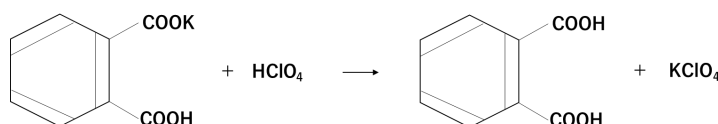
Outline

This application sheet describes the standardization of 0.1 mol/L perchloric acid solution in acetic acid using an alternative solvent, as described in ASTM D2896. The appendix of the standard 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

Potassium hydrogen phthalate is 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. The inflection point is detected as the end point. The factor is calculated from the amount of potassium hydrogen phthalate 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) (To record the temperature when temperature correction is required if the titrant temperature differs between standardization and titration)

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Reagents

- [Titrant] ■ 0.1 mol/L Perchloric acid solution in acetic acid
- [Reagents] ■ Potassium hydrogen phthalate (certified reference material): Dried at 120 °C for 2 hours, then cooled
- Acetic acid (special grade)
- Xylene (special grade)
- Acetone (special grade)
- Sodium perchlorate electrolyte: saturated solution of sodium perchlorate (NaClO_4) in glacial acetic acid

Analytical Procedure**[Blank titration]**

- 40 mL of acetic acid was added to a 150 mL tall beaker, and 80 mL of xylene and 10 mL of acetone was added to it.
- It was titrated with a solution of perchloric acid.

[Sample titration]

- 0.1 to 0.2 g of potassium hydrogen phthalate was weighed in a 150 mL tall beaker up to digits of 0.1 mg. 40 mL of warm acetic acid* was added to dissolve it, and 80 mL of xylene and 10 mL of acetone was also added.
- After cooling to room temperature, It was titrated with a solution of perchloric acid.

* acetic acid at approximately 50 °C was used.

[Calculation]

$$\text{Factor} = 1,000 \times W / FW / (A1 - B) / Q$$

- 1,000: Unit conversion factor for 1/ mL to 1/ L
- W: Mass of potassium hydrogen phthalate sample (g)
- FW: Formula weight of potassium hydrogen phthalate sample (= 204.23)
- A1: Volume of 0.1 mol/L sodium acetate solution in acetic acid used for sample titration (mL)
- B: Volume of 0.1 mol/L sodium acetate solution in acetic acid used for blank titration (= 0 mL)
- Q: Concentration of 0.1 mol/L sodium acetate 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 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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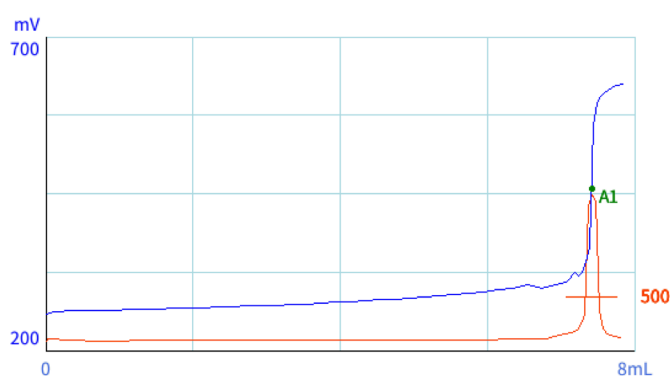
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Measurement Results

Sample	Sample amount (g)	Titration volume (mL)	Factor	Average	RSD (%)
	0.1502	7.4031	0.993		
	0.1474	7.2816	0.991	0.992	0.1
	0.1552	7.6605	0.992		



Sample name: Potassium hydrogen phthalate
 End point: 7.4031 mL 459.0 mV
 Start of measurement: 0.000 mL 260.0 mV
 End of measurement: 7.830 mL 625.3 mV
 Measurement time: 18 min 03 s
 Start temperature: 25.4°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
Initial wait time:	60 s	60 s
Drop control:	Individual	Individual
Max. drop volume:	50 μ L	200 μ L
Min. drop volume:	10 μ L	50 μ L
Stability criteria:	Individual	Individual
Delta potential:	1 mV	1 mV
Delta time:	12 s	12 s
E1:	Inflection/Set-Potential* ¹	Inflection/Set-Potential* ¹
E1 potential:	460 mV	460 mV
E1 potential width:	250 mV	250 mV
E1 differential evaluation value:	500 mV/mL	500 mV/mL
E1 evaluation points:	3	5
Max. derivative threshold:	10 mL	20 mL
End derivative:	50 mV/mL	50 mV/mL

*1: If no inflection point is detected, the E1 potential is used to detect the endpoint. If the potential for the end point (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.