

Sheet no.

GT-310-PE-043E Energy & Petroleum Products

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

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

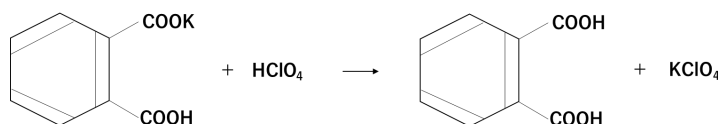
Outline

0.1 mol/L perchloric acid solution in acetic acid is used as the titrant in ASTM D2896. The standardization method is divided into Procedure A (120 mL) and Procedure B (60 mL) depending on the amount of titration solvent. 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

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. Then, 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 for temperature correction 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)
- Chlorobenzene (special grade)
- Sodium perchlorate electrolyte: saturated solution of sodium perchlorate (NaClO₄) in glacial acetic acid

Analytical Procedure

[Blank titration]

- 20 mL of acetic acid was added to a 150 mL tall beaker, and 40 mL of chlorobenzene was added to it.
- It was titrated with a solution of perchloric acid in acetic acid.

[Sample titration]

- 0.05 to 0.1 g of potassium hydrogen phthalate was weighed up to digits of 0.1 mg in 150 mL tall beaker. 20 mL of warm acetic acid* was added to dissolve it. Then, 40 mL of chlorobenzene was added.
- It was cooled to room temperature and titrated using 0.1 mol/L perchloric acid solution in acetic 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: Molar mass of potassium hydrogen phthalate sample (= 204.23 g/mol)
- 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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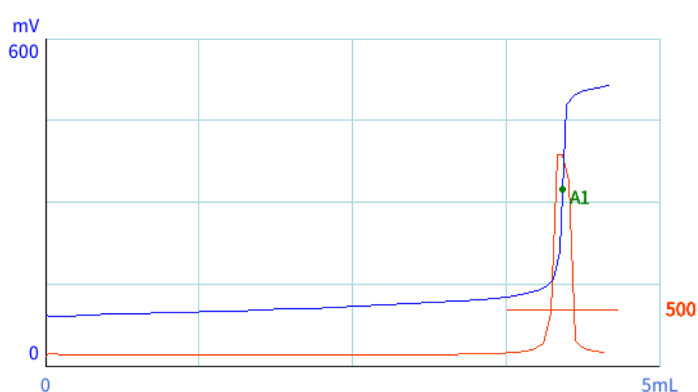
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Measurement Results

| Sample | Sample amount (g) | Titration volume (mL) | Factor | Average | RSD (%) |
|------------------------------|-------------------|-----------------------|--------|--------------|------------|
| Potassium hydrogen phthalate | 0.0852 | 4.2029 | 0.993 | 0.993 | 0.1 |
| | 0.0855 | 4.2218 | 0.992 | | |
| | 0.0903 | 4.4480 | 0.994 | | |



Sample name: Potassium hydrogen phthalate

End point: 4.2029 mL 324.0 mV

Start of measurement: 0.000 mL 95.5 mV

End of measurement: 4.576 mL 514.0 mV

Measurement time: 9 min 42 s

Start temperature: 22.8°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 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: 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.