Sheet No.
GT-310-FO-013E Food & Beverage

Standardization of Sodium Hydroxide Solution (Official Analysis Method of the National Tax Agency)

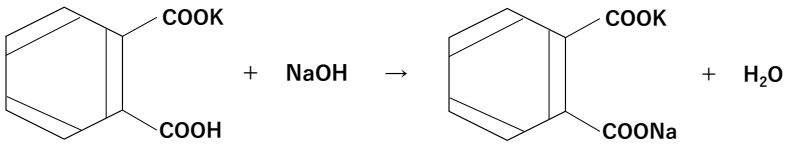
Reference standard: Official Analysis Method of the National Tax Agency, 3-5-1 Reagents

Outline

The Official Analysis Method of the National Tax Agency describes test methods for the goods, subject to indirect tax and related items (e.g., alcoholic beverages). Standardization was performed on 0.1 mol/L sodium hydroxide solution used to test total acid (free acid) and amino acids based on 3-5-1 Reagents. The Official Analysis Method of the National Tax Agency describes a procedure using phenolphthalein as an indicator, with the end point defined as the point at which the solution turns light red.
Potentiometric titration was conducted using a glass electrode, and results were obtained with a relative standard deviation of less than 1 %.

Principle

Potassium hydrogen phthalate is neutralized using sodium hydroxide solution.
Titration is performed while recording pH with a glass electrode. The end point is detected in at a rapid change in pH occurs. The factor of the sodium hydroxide solution is calculated from the mass of the potassium hydrogen phthalate and the volume of sodium hydroxide solution titrated to the end point.



Apparatus

Automatic titrator: GT-310
Electrodes: GLASS ELECTRODE, L=105 (GTPH1B),
REFERENCE ELECTRODE, L=105 (D-J) (GTRE10B) (Outer solution: 1 mol/L potassium nitrate solution, Inner solution: 1 mol/L potassium chloride solution)

Reagents

[Titrant] ■ Sodium hydroxide solution 0.1 mol/L (for volumetric analysis)
[Reagent] ■ Potassium hydrogen phthalate (certified reference material)

Sheet No.
GT-310-FO-013E

Standardization of Sodium Hydroxide Solution (Official Analysis Method of the National Tax Agency)

Analytical Procedure

1. Approximately 0.5 g of potassium hydrogen phthalate, dried under the conditions specified in the certification, was precisely weighed into a 100 mL beaker, and then approximately 50 mL of purified water was added.
2. Titrated with 0.1 mol/L sodium hydroxide solution.

[Calculation]

Factor (f) = W / X1 / A1 × X2 × 10

W: Mass of potassium hydrogen phthalate sample (g)
X1: Mass of potassium hydrogen phthalate equivalent to 1 mL of 0.1 mol/L sodium hydroxide solution (= 20.42 mg/mL)
A1: Titration volume (mL)
X2: Purity of potassium hydrogen phthalate (= 100.00 %)
10: Factor to convert units, g・% to mg

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.
- Perform pH calibration before measurement.

Measurement Results

Sample	Sample amount (g)	Titration volume (mL)	Factor	Average (%)	RSD (%)
Potassium hydrogen phthalate	0.5055	24.8677	0.9955	**0.9947**	**0.1**
	0.5048	24.8527	0.9947		
	0.5083	25.0423	0.9940		

Sheet No.
GT-310-FO-013E

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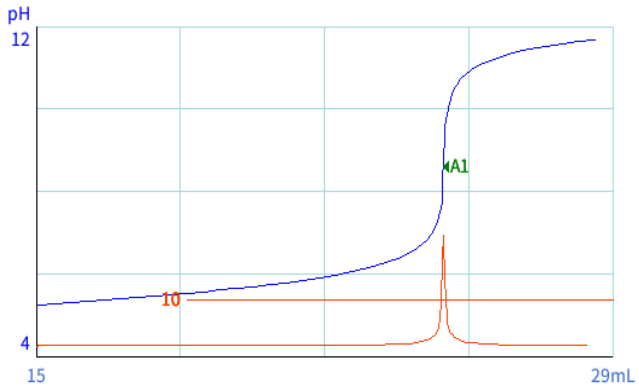

Sample name:	Potassium hydrogen phthalate	
End point:	24.8527 mL	pH 8.6198
Start of measurement:	15.0000 mL	pH 5.2948
End of measurement:	28.788 mL	pH 11.6914
Measurement time:	5 min 29 s	

■ Default values were used for parameters not listed below.

Detector:	pH
Titration mode:	General titration
Preset 1:	Volume*1
P1 injection volume:	15 mL
Initial fill rate:	75 %*2
Drop volume control:	Individual [Normal*3]
Max. drop volume:	400 µL
Min. drop volume:	20 µL
Stability criteria:	Individual [Fast*3]
Delta potential:	pH 0.1
Delta time:	3 s
E1:	Inflection
E1 reference potential:	pH 7
E1 potential width:	pH 5
E1 derivative threshold:	pH 10 /mL
E1 evaluation points:	20
Max. titration volume:	35 mL

*1: Used to reduce the time required.
*2: Used so that the initial aspiration is performed after discharging 75 % (15 mL) to avoid buret aspiration at the endpoint.
*3: Parameters other than the “Individual” may be used.

* 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.