Sheet No.
GT-310-ME-015E Pharmaceuticals and Cosmetics

Standardization of Potassium Permanganate Solution (Japanese Pharmacopoeia (2))

Reference standard: The Japanese Pharmacopoeia 18th edition

Outline

The section titled “General Test / 9.21 Standard Solution for Volumetric Analysis” in the Japanese Pharmacopoeia provides information on the preparation and standardization of standard solutions for volumetric analysis. The standardization of 0.02 mol/L potassium permanganate solution involves titrating sodium oxalate with potassium permanganate solution until a light red color persists for 30 seconds. For this application sheet, the potentiometric titration method that assumes the inflection point as the end point was performed. For the method using the point at which the color persists for 30 seconds as the end point, refer to Application Sheet GT310-ME-016E “Standardization of Potassium Permanganate Solution (Japanese Pharmacopoeia (1))”. The measurement results showed a relative standard deviation (RSD) of less than 1 %.

Principle

Redox titration of sodium oxalate is performed using potassium permanganate. Under acidic conditions, potassium permanganate acts as a pentavalent oxidizing agent, resulting in the reaction shown below. Since this reaction rate is slow, the solution is heated to accelerate the reaction. Since manganese (II) ions produced by the redox reaction act as a catalyst, potassium permanganate is reacted in advance to generate manganese (II) ions. Afterward, the solution is reheated and titration is performed. Titration is performed while recording the potential difference between a platinum electrode and the reference electrode. The end point is detected at a rapid change in potential difference occurs. The factor of the 0.02 mol/L potassium permanganate solution is calculated from the mass of sodium oxalate and the volume of potassium permanganate solution used to reach the end point.

$$2KMnO_4 + 5Na_2C_2O_4 + 8H_2SO_4 \rightarrow 2MnSO_4 + 5Na_2SO_4 + K_2SO_4 + 10CO_2 + 8H_2O$$

Apparatus

Automatic titrator: GT-310
Electrodes: PLATINUM ELECTRODE, L=105 (GTPT1B),
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]
- Potassium permanganate solution 0.02 mol/L (as specified in Japanese Pharmacopoeia)

[Reagents]
- Sodium oxalate (certified reference material)
- Diluted sulfuric acid (1:20): Sulfuric acid (special grade) was diluted 20 times by volume with pure water.

Analytical Procedure

Standardization of Potassium Permanganate Solution (Japanese Pharmacopoeia (2))

1. Sodium oxalate was dried between 150 °C to 200 °C for 1 to 1.5 hours (in this application sheet, 175 °C for 1.5 hours), then cooled in a desiccator (with silica gel).
2. Approximately 0.3 g was accurately weighed into a 500 mL beaker* and dissolved in 30 mL of purified water.
3. 250 mL of diluted sulfuric acid (1:20) was then added, and the solution temperature was adjusted to between 30 °C and 35 °C. While this was gently stirred, 40 mL of solution was quickly added (the GT-310BRT dispensing function was used here). The mixture was left to stand until the red color disappeared.
4. After heating to between 55 °C and 60 °C, titration was performed using the 0.02 mol/L potassium permanganate solution.

* Although a 500 mL conical flask is used in the standard, a beaker was used instead. This was due to the small solvent volume, which would not allow full immersion of the electrode in a solution.

[Calculation]

Factor (f) = W / X1 / (A1 + 40) × X2 × 10

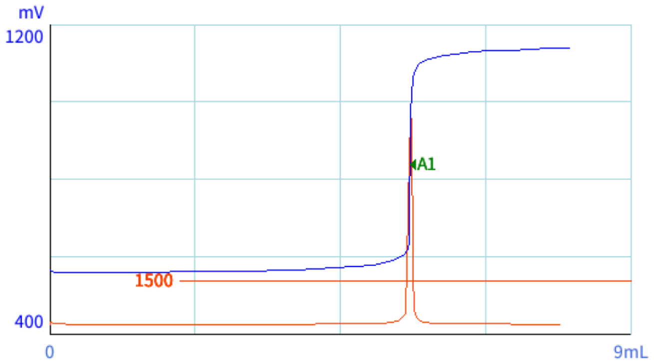
W: Mass of sodium oxalate (g)
X1: Mass of sodium oxalate equivalent to 1 mL of 0.02 mol/L potassium permanganate solution (= 6.7 mg/mL)
A1: Volume of 0.02 mol/L potassium permanganate solution required for titration (mL)
40: Volume of 0.02 mol/L potassium permanganate solution added in Step 3 (= 40 mL)
X2: Purity of sodium oxalate (= 99.96 %)
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.

Measurement Results

Sample	Sample amount (g)	Titration volume (mL)	Factor	Average	RSD (%)
Sodium oxalate	0.3058	5.5789	1.001	**1.000**	**0.1**
	0.3065	5.7179	1.000		
	0.3068	5.8386	0.999		



Sample name:	Sodium oxalate	
End point:	5.5789 mL	840.5 mV
Start of measurement:	0.000 mL	566.3 mV
End of measurement:	8.035 mL	1,139.7 mV
Measurement time:	8 min 38 s	

■ Default values were used for parameters not listed below.

Stirrer speed:	3.5
Titration mode:	General titration
Detector:	mV
Initial wait time:	20 s
Drop volume control:	Individual [Normal^{*1}]
Max. drop volume:	300 μL
Min. drop volume:	20 μL
Stability criteria:	Individual [Normal^{*1}]
Fixed wait time:	2 s
Delta potential:	1 mV
Delta time:	2 s
E1:	Inflection
E1 potential:	500 mV
E1 potential width:	1,000 mV
E1 derivative threshold:	1500 mV/mL
E1 evaluation points:	14
Max. titration volume:	15 mL
Over titration volume:	1 mL

*1: 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.