Sheet No.
GT-310-ME-014E Pharmaceuticals and Cosmetics

Analysis of Hydrogen Peroxide in Oxydol (Japanese Pharmacopoeia)

Related standard: The Japanese Pharmacopoeia 18th edition

Outline

Oxydol is defined in the Japanese Pharmacopoeia as a 2.5 to 3.5 w/v% hydrogen peroxide solution containing an appropriate stabilizer. For this application sheet, the concentration of hydrogen peroxide in oxydol was measured using potentiometric titration based on the assay specified in the Japanese Pharmacopoeia. The measurement results were obtained, with a relative standard deviation (RSD) of less than 1 %.

Principle

Redox titration is performed using potassium permanganate. Under acidic conditions, potassium permanganate acts as a pentavalent oxidizing agent, resulting in the reaction shown below. 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 concentration of hydrogen peroxide is calculated from the volume of the oxydol sample and the amount of potassium permanganate added up to the end point.

$$2\ KMnO_4 + 5\ H_2O_2 + 3\ H_2SO_4 \rightarrow 2\ MnSO_4 + K_2SO_4 + 5\ O_2 + 8\ H_2O$$

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)

[Reagent]
- Dilute sulfuric acid: Carefully add 5.7 mL of sulfuric acid (special grade) to 10 mL of water, cool using running water or an ice bath, then add water to make up 100 mL (10 %).

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Analytical Procedure

1. 1 mL sample was transferred to a 50 mL beaker using a volumetric pipette.
2. 10 mL of water and 10 mL diluted sulfuric acid were added.
3. The solution was titrated with a solution of 0.02 mol/L potassium permanganate.

* Although a 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 flask.

[Calculation]

Concentration (w/v%) = A1 × X1 × f / W / 10

A1: Volume of 0.02 mol/L potassium permanganate solution required for titration (mL)
X1: Mass of hydrogen peroxide equivalent to 1 mL of 0.02 mol/L potassium permanganate solution (= 1.701 mg/mL)
f: Factor of 0.02 mol/L potassium permanganate solution (= 1.0015) *1
W: Amount of sample (= 1 mL)
1/10: Unit conversion factor for mg to g (1/1000) multiplied by unit conversion factor for percentage (100)

*1: Standardization was performed based on the Japanese Pharmacopoeia. (Refer to the application sheet GT-310-ME-015E "Standardization of Potassium Permanganate Solution" and GT-310-ME-016E "Standardization of Potassium Permanganate Solution (Japanese Pharmacopoeia (1))"

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.
- The electrode liquid junction must be fully immersed during measurement. While the liquid junction may not be fully immersed at the start of titration due to the small amount of solvent specified in the standard, measurement is possible provided it is immersed after the preset dripping.

Measurement Results

Sample	Sample amount (mL)	Titration volume (mL)	Concentration (w/v%)	Average (w/v%)	RSD (%)
Oxydol	1	17.5797	2.995	**2.995**	**0.1**
		17.5618	2.992		
		17.5958	2.998		

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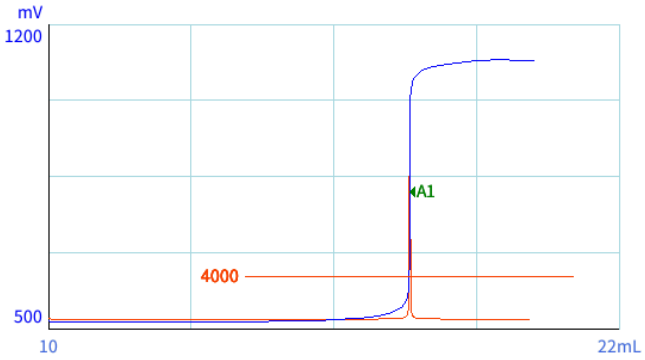

Sample name:	Oxydol	
End point:	17.5797 mL	816.6 mV
Start of measurement:	10.000 mL	520.2 mV
End of measurement:	20.185 mL	1,115.4 mV
Measurement time:	7 min 10 s	

■ Default values were used for parameters not listed below.

Stirrer speed:	3.5
Titration mode:	General titration
Detector:	mV
Preset 1:	Volume*1
P1 injection volume:	10 mL
Initial fill rate:	50 %*2
Drop volume control:	Individual [Normal*3]
Max. drop volume:	200 µL
Min. drop volume:	10 µL
Stability criteria:	Individual [Fast*3]
Delta potential:	5 mV
Delta time:	3 s
E1:	Inflection
E1 potential:	800 mV
E1 potential width:	500 mV
E1 derivative threshold:	4,000 mV/mL
E1 evaluation points:	25
Max. titration volume:	28 mL

*1: Used to ensure that the electrode liquid junction is immersed and reduce the time required

*2: Used so that the initial aspiration is performed after discharging 50 % (10 mL) to avoid buret aspiration around the end point

*3: Parameters other than the settings 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.