

<b>HIRANUMA APPLICATION DATA</b>	Automatic Titrator	Data No.	O8	Feb. 03, 2022
<b>Factor standardization</b>	<b>Standardization of potassium permanganate titrant</b>			

## 1. Abstract

Potassium permanganate is a strong oxidizing agent, thus the potassium permanganate standard solution is used as a titrant for reducing agents such as iron(II) iron, and as a titrant for hydrogen peroxide and chemical oxygen demand (COD).

Factors are indicated on the commercially available standard solution for volumetric analysis. The factor determination is required when the standard solutions are prepared in the laboratory. Also it is effective to check the repeatability by the factor measurement using a standard material to check the performance of titrator system. *Japanese Industrial Standard JIS K 8001* and the *Japanese Pharmacopoeia* describe that sodium oxalate, which is a standard material for a volumetric analysis, should be used for the factor determination of potassium permanganate standard solution.

The measurement procedure is as follows. The sodium oxalate is taken into an Erlenmeyer flask, and add water and sulfuric acid to dissolve in acidic solution. And then put the flask on a hot stirrer to warm the solution to temperature about 35 °C. Dispense potassium permanganate standard solution up to about 2 mL before the end point, and react it until the solution color becomes transparent. After heating to the solution temperature of 55-60 °C, slowly titrate the remaining sodium oxalate with a potassium permanganate standard solution. As the reaction shown in formula (1), 2 mol of potassium permanganate is quantitatively reacted with 5 mol of sodium oxalate, and an inflection point appears on the titration curve at the end point.



- 1) Japanese Pharmacopoeia Eighteenth Edition
- 2) Japanese Industrial Standard JIS K8001 General rules for test methods of reagents

## 2. Configuration of instruments and reagents

### (1) Configuration of instruments

Main unit	: Automatic Titrator	COM Series
Electrode	: Platinum-reference combination electrode (long type)	PR-733BZ
Buret	: In order to use the Erlenmeyer flask as titration vessel, it is necessary to modify the buret tip and stirrer parts. (Refer to figure 1.)	
Stirrer	: Hot stirrer (commercial item) Laboratory stand (commercial item), Attach the shaft for the electrode holder (Refer to figure 2.) * Please contact us for specific installation examples.	

## (2) Reagents

- Titrant : 0.02 mol/L (0.1 N) potassium permanganate standard solution
- Standard material : Sodium oxalate (Standard material for volumetric analysis)  
(Certified value of purity for the standard material in this report: 99.96 %)
- Additive reagent : Diluted sulfuric acid solution (Approx. 65 %)  
Prepared by slowly adding 100 mL of sulfuric acid to 100 mL of DI water.

## 3. Measurement procedure

- (1) Take 0.15 g of sodium oxalate into a 200 mL Erlenmeyer flask and accurately weigh it.
- (2) Add 100 mL of DI water into the flask to dissolve the sample, and add 10 mL of diluted sulfuric acid solution to acidify the solution.
- (3) Put the flask on the hot stirrer to warm the solution to temperature about 35 °C with stirring.
- (4) Immerse the electrode and start measurement. Firstly, 20 mL of 0.02 mol/L potassium permanganate standard solution is dispensed with buret.
- (5) The flask is heated to 55-60 °C while the titrator counts the time set to "T. Timer" for 600 seconds.
- (6) Titration is performed with 0.02 mol/L potassium permanganate standard solution, and the inflection point on the titration curve is detected as the end point.

## 4. Measurement conditions and results

### Examples of titration conditions

Factor standardization with sodium oxalate standard material

Cndt No.	1	ConstantNo.	1	Mode No.	6
Method	Auto	Size	0 g	Pre Int	0 sec
Buret No.	1	Blank	0 mL	Del K	2
Amp No.	1	Molarity	0.02 mol/L	Del Sens	0 mV
D. Unit	mV	Factor	0.9996 *1	Int Time	3 sec
S-Timer	5 sec	K	335 *2	Int Sens	3 mV
C.P. mL	20 mL	L	0	BrT Speed	2
T Timer	600 sec	Unit	Fact2	Pulse	40
D.P. mL	0.2 mL	Formula			
End Sens	200		$S * F * 1000 / (K * M * (D - B))$		
Over mL	0.5 mL	Digits	4		
Max.Vol.	40 mL				

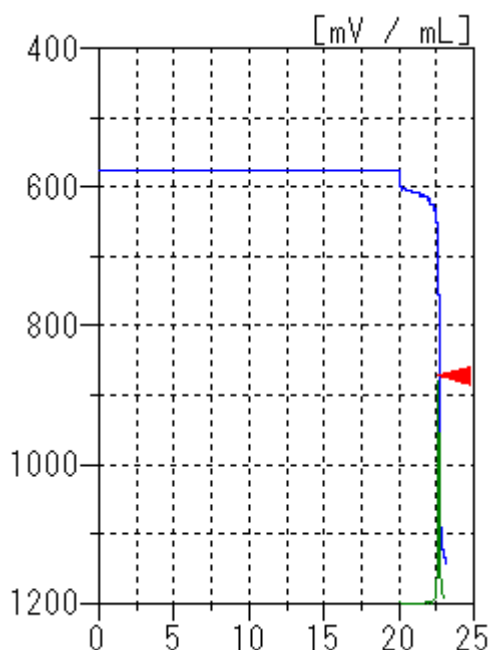
\*1: Purity of sodium oxalate / 100

\*2: It is the mass (g) of sodium oxalate that reacts with 1 mol of potassium permanganate by the reaction formula (1). The value is 2.5 times the molecular weight (134) of sodium oxalate.

## Measurement results

Measurement results of factor standardization

Measurement No.	Sample size (g)	Titrant volume (mL)	Factor	Statistical results
1	0.1568	23.554	0.9932	Avg. 0.993
2	0.1510	22.620	0.9959	SD 0.003
3	0.1554	23.424	0.9898	RSD 0.31 %



Examples of titration curves

## 5. Note

(1) About a sampling of the standard material

Sodium oxalate is used for the standardization of potassium permanganate standard solution in redox titration. The standard material for volumetric analysis comes with a certificate value of the purity and uncertainty. If the certification and traceability are required for the management of test result, such as quality records, the standard material for volumetric analysis is used. It is necessary to prepare the standard material with pretreatment such as drying as described in its instruction before use.

The weighing amount of sodium oxalate is described as about 0.3 g in the Japanese Pharmacopoeia and 0.20 to 0.24 g in JIS K8001. The titrant volume expected from the reaction ratio shown in formula (2) below are 45 mL and 30 to 35 mL, respectively. In this report, the weighing amount was reduced to 0.15 g in order to reduce the titrant volume to about 20 mL.

$$1 \text{ mL of } 0.02 \text{ mol/L potassium permanganate} = 6.700 \text{ mg of sodium oxalate} \quad \dots(2)$$

(2) About selection of electrode and modification of buret tip and stirrer to be compatible with Erlenmeyer flask and hot stirrer.

In this report, since a 200 mL Erlenmeyer flask is used for the titration vessel, the electrode is required a long and combination type platinum-reference combination electrode PR-733BZ, and the buret tip is replaced to a tube type (Refer to Figure 1). To heat the flask during titration, prepare a hot stirrer and laboratory stand. A shaft for electrode holder is attached on the laboratory stand to move up and down the electrode (Refer to Figure 2).

Please contact us for specific installation examples. When using a 200 mL conical beaker, the half-cell type electrodes such as PT-301 and RE-201Z are applicable.

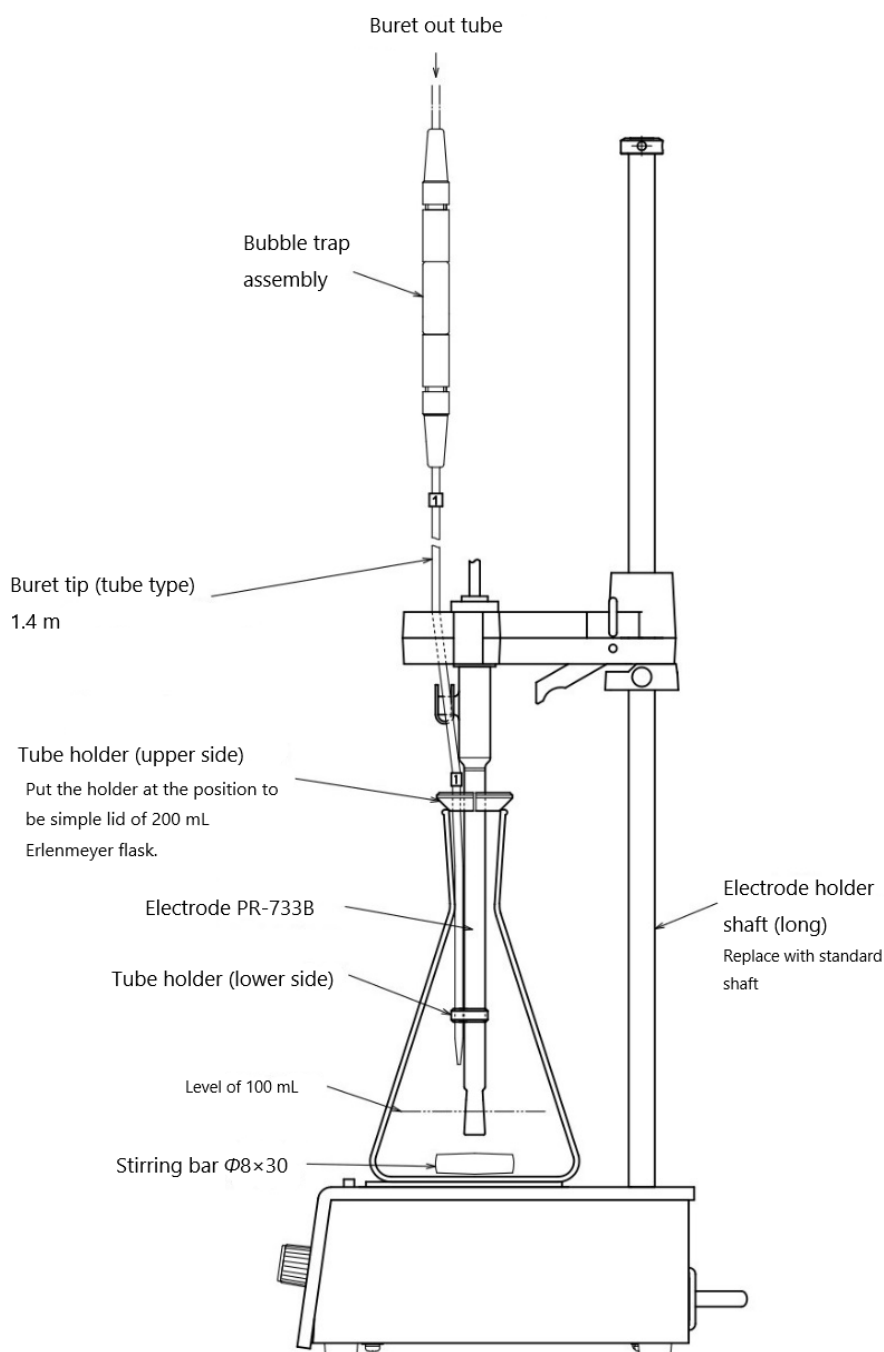


Figure 1 An modification example of buret tip and stirrer parts compatible with Erlenmeyer flasks

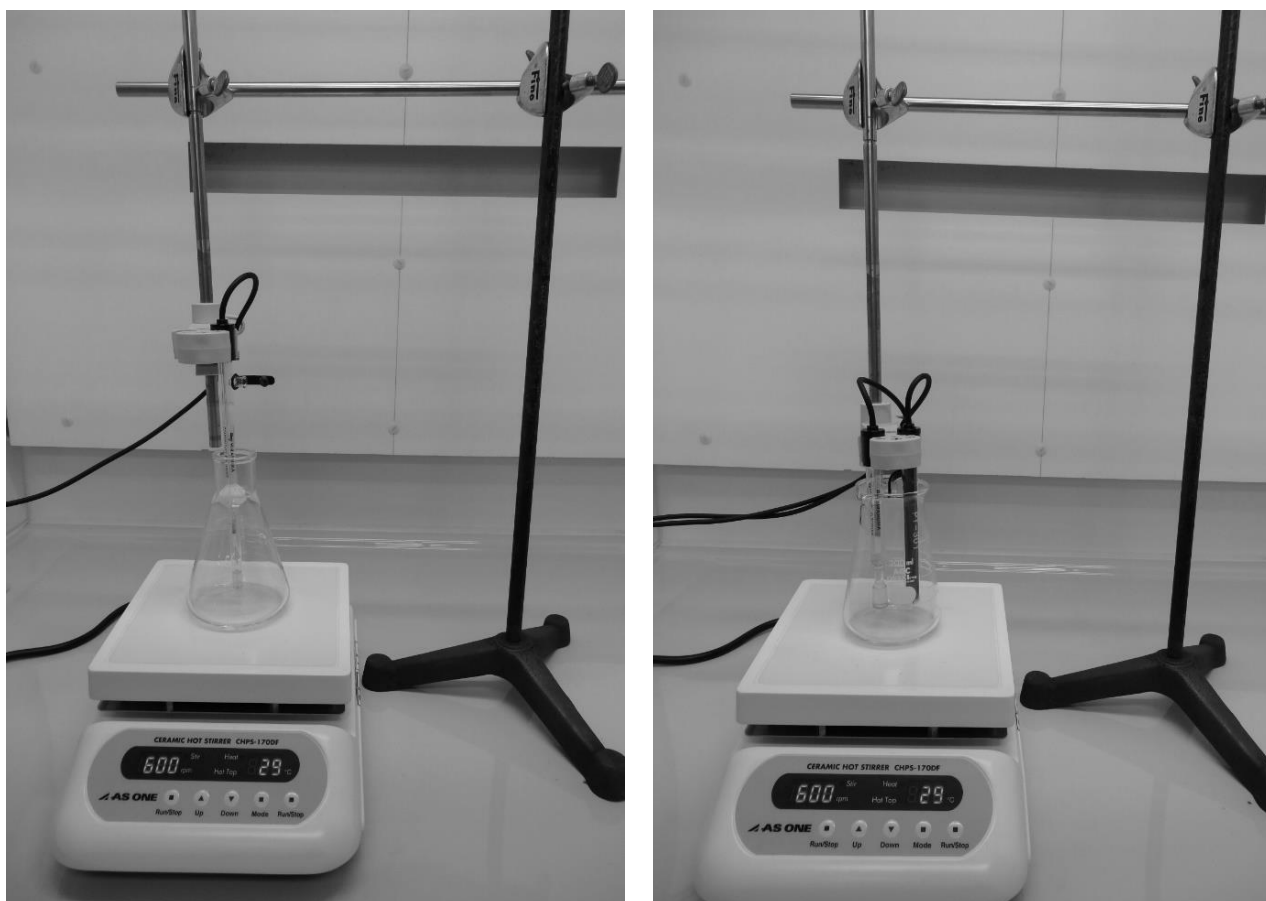


Figure 2 An installation example of electrodes compatible with hot stirrer and titration vessel.

(Left) Titration vessel: 200 mL Erlenmeyer flask

Electrode: Platinum-reference combination electrode PR-733BZ(Long type)

(Right) Titration vessel: 200 mL conical beaker

Electrode: Platinum electrode PT-301, Reference electrode RE-201Z

(3) Performance check method with potassium permanganate titrant that does not require hot stirrer.

If the factor determination of potassium permanganate standard solution is not required, and it is necessary to check the performance of titrator system (device, electrode, and titrant), the measurement procedure can be simplified from this report. By using the standard solution of ammonium iron (II) sulfate as the standard sample, heating with a hot stirrer is not required, so a beaker can be used as the titration vessel. As a result, the long and combination type electrodes are not required, but the half-cell electrode type such as PT-301 and RE-201Z can also be applied.

However, since the standard solution of ammonium iron (II) sulfate is inferior in stability to sodium oxalate, it should not be used for the factor determination of potassium permanganate, but only for the performance check of the titrator system based on repeatability. Please refer to Application Data No. O9 in details.

Keywords : Factor standardization, Redox titration, Potassium permanganate, Sodium oxalate