# Laboratory work № 3. Standardisation of Potassium Permanganate Solution. Quantitative determination of hydrogen peroxide.

A useful property of potassium permanganate solution is its intense purple colour, which to serve as an indicator for most titrations. The surface of the permanganate solution rather than the bottom of the meniscus can be used to measure titrant volumes.

We can‘t prepare a primary standard solution from the crystalline $KMnO_4$ which is sold in the pharmacy because even the mark of -cp‖ (chemically pure) type doesn‘t satisfy the requirements necessary for the initial substances. It usually contains traces of $MnO_2$ in the form of admixtures which can catalyze the reaction of water oxidation by permanganate:

$$4MnO_4^- + 2H_2O = 4MnO_2 + 3O_2 + 4OH^-,$$

which is usually accompanied by the change in concentration of $KMnO_4$ solution. Besides, during the dissolving of even the purest $KMnO_4$ in water a part of it is used for the reaction with admixtures of organic substances which are always present in distilled water. Moreover, light can also influence the stability of $KMnO_4$ solution.

According to the principles indicated above, we should prepare $KMnO_4$ solution with the concentration approximately equal to the required one (usually 0.02 or 0.05 mol/L) and leave it for some days till the complete precipitation of $MnO_2$. After waiting 1 week, filter the solution by glass fibers into a clean amber glass bottle. The final solution is kept in dark.

As primary standards to determine the concentration of $KMnO_4$ solution we use anhydrous sodium oxalate $Na_2C_2O_4$ or oxalic acid dihydrate $H_2C_2O_4 \times 2H_2O$. These substances after all the preparations satisfy the requirements needed for primary standards and can be used for the preparation of standard solutions.

**Equipment and glassware:**

- 3 Erlenmeyer flasks, volume 100 mL;
- Buret on 25,00 mL fastened on a ring stand;
- Pipet on 10,00 mL;
- volumetric flask V=100,0 ml
- Glass or plastic funnel of small diameter;
- Bottle with DW.

**Reagents:**

- 0.02500 M solution of $H_2C_2O_4$;
- Solution of $KMnO_4$ with approximately concentration;
- 20 % solution of $H_2SO_4$

**Part I. Standardisation of Potassium Permanganate Solution.**

The reaction between permanganate ion and oxalic acid is complex and proceeds slowly even at elevated temperature unless manganese(II) is present as a catalyst. Thus, when the first few millilitres of the standard permanganate are added to a hot solution of oxalic acid, several seconds are required before the colour of the permanganate ion disappears. Solution of sodium oxalate are titrated at 60°C to 90°C. After the added permanganate is completely consumed (as indicated by the disappearance of colour), the solution is heated to about 60°C and titrated to a pink colour that persists for about 30 seconds.

**Procedure:**

1. Before the beginning of work to wash carefully all glassware as usual.
2. To wash buret by potassium permanganate solution.

3. To wash pipet by $H_2C_2O_4$ solution.
4. Using funnel to fill a buret by potassium permanganate solution and obtain absence of air bubbles in the tag of buret.
5. To take off funnel and show out the level of liquid in a buret to the zero mark.
6. To take aliquot of 0.02500M $H_2C_2O_4$ solution (10.00 ml) by a pipet in washed by distilled water Erlenmeyer flask.
8. Add 10 ml of 2 M $H_2SO_4$ solution
7. Heat solution to 80°C to 90°C one flask (*should not be boiled!*).
8. Titrate with potassium permanganate solution. The pink colour imported by one addition should be permitted to disappear below any father titrant is introduced.
9. Read the burette mark.
10. Repeat titration also two times. Calculate the approximate value of used potassium permanganate solution.
11. Calculate the exact concentration of the potassium permanganate solution accordance to equivalents law.

$$2KMnO_4 + 5H_2C_2O_4 + 3H_2SO_4 \rightarrow 2MnSO_4 + K_2SO_4 + 10CO_2 + 8H_2O$$

oxidizer 1 reducer 2 reducer 1 oxidizer 2

$$2/ \quad MnO_4^- + 8H^+ + 5e^- \longrightarrow Mn^{+2} + 4H_2O$$

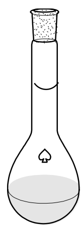
$$5/ \quad C_2O_4^{-2} \longrightarrow 2CO_2 + 2e^-$$

$$2MnO_4^- + 16H^+ + 5C_2O_4^{-2} \longrightarrow 2Mn^{+2} + 8H_2O + 10CO_2$$

$$c_{KMnO_4} = \frac{2 \cdot c_{H_2C_2O_4} \cdot V_{H_2C_2O_4}}{5 \cdot V_{KMnO_4}}$$

**Part II. Quantitative determination of hydrogen peroxide.**

**1.1.Preparation of working solution**



1. Transfer the received sample into volumetric flask.
2. Add 40-50 ml of distilled water to volumetric flask and mix.
3. Bring the level of liquid in a flask to the mark;
4. 1. Load a burette with potassium permanganate solution.
5. To conical flask pour in 10.00 ml aliquot of sample.
6. Add 10 ml of 2 M $H_2SO_4$ solution.
7. Titrate with potassium permanganate solution. The pink colour imported by one addition should be permitted to disappear below any father titrant is introduced.
8. Read the burette mark.
9. Repeat titration also two times. Calculate the approximate value of used potassium permanganate solution.
10. Repeat titration also two times. Calculate the median volumes of used potassium permanganate solution.

11. Calculate content of hydrogen peroxide in sample:

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

$$\begin{array}{ll|l} MnO_4^- + 8H^+ + 5e \rightleftharpoons & Mn^{2+} + 4H_2O & 2 \\ H_2O_2 - 2e \rightleftharpoons & O_2 + 2H^+ & 5 \end{array}$$

| № of titration | Volume of $KMnO_4$ used for titration $H_2C_2O_4Cl$, mL | Volume of $KMnO_4$ used for titration $H_2O_2$, mL |
|---|---|---|
| 1. | | |
| 2. | | |
| 3. | | |
| average | | |

$$\rho^* = \frac{5 \cdot c_{KMnO_4} \cdot V_{KMnO_4} \cdot 10^{-3} \cdot M(H_2O_2) \cdot V_к}{2 \cdot V_п \cdot V_{пробы} \cdot 10^{-3}},$$

**The results you present to teacher.**