Laboratory work № 4. Standartisation of Sodium Thiosulfate.

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:

- Dry salt of $K_2Cr_2O_7$;
- Solutions of sodium thiosulfate with approximately concentration – 0.05 M;
- Starch solution;*
- 20% solution of H_2SO_4,
- 10% solution of KI.

***Preparation of Starch Solution**

Material Required: Starch (arrowroot): 1.0 g.

Procedure : Weigh 1.0 g starch in a glass in a glass pestle-mortar and triturate thoroughly with 10 ml of cold DW. Boil separately 200 ml of DW in a beaker and add the starch paste to it with vigorous stirring. The resulting mixture is boiled gently for a further period of 30 minutes till a transluscent and thin liquid having an uniform consistency is obtained.

Note: 1) The prepared solution of starch undergoes rapid deterioration, hence it is always desired to use freshly prepared solution every day,

2) It is now more or less believed that the iodine is held as an 'absorption complex' within the helical chain of the macromolecule β-amylose *i.e.*, a component of most starches. However, another component, α-amylose, is undesirable because it produces a red-colouration with iodine which is not readily reversible, and 3) 'Soluble Starch' comprises principally of β-amylose, with the α-fraction having been removed. Always, it is a practice to prepare indicator-solutions from this product exclusively.

Part I. Standardisation of Sodium Thiosulfate Against Potassium Dichromate.

Procedure:

1.1. Preparation of Potassium Dichromate Standard Solution

1. Calculate a mass of primary standard sample for preparation 100,0 ml of 0.008300 M solution:

$m = C \cdot V \cdot M = 0{,}00835 \cdot 0{,}1 \cdot 294{,}185 \approx 0{,}25$ г $K_2Cr_2O_7$

M.m. $K_2Cr_2O_7$= 294,185 g;

2. Weight potassium dichromate sample on hand balance with 0,1 g accuracy.
3. Weight empty volumetric flask on analytical balance. Transfer the dichromate into volumetric flask and weight. (To carry taken mass of potassium dichromate in a volumetric flask through a dry filtering funnel). Weight volumetric flask with potassium dichromate on analytical balance. **m=_____g**
4. Add 40-50 ml of distilled water to volumetric flask, dissolve dichromate (mix solution carefully).
5. Bring the level of liquid in a flask to the mark;

1.2. Standardisation of Sodium Thiosulfate Solution

1. Before the beginning of work to wash carefully all glassware as usual.

2. To wash buret by thiosulphate solution.
3. To wash pipet by working solution.
4. Using funnel to fill a buret by thiosulphate 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. Pipette 10-ml aliquot of prepared standard potassium dichromate solution into 100 ml Erlenmeyer flask.
7. Add 10 ml of 20% H_2SO_4 solution and 5 ml of 10% KI solution into 100 ml Erlenmeyer flask.
8. Cover the flask with a stopper and put into dark place at 10 min (to let the reaction between $K_2Cr_2O_7$ and KI go up to the completion and avoid the degradation of I_2 induced by the light).
9. Titrate the sample solution with thiosulfate until the solution becomes ***pale yellow***. 10. Introduce 0.5 ml of starch indicator, and titrate with constant stirring to the disappearance of the blue colour.
11. Read the burette mark.
12. To repeat titration of a new portion of working solution.

№ of titration	Volume of $Na_2S_2O_3$ used for titration, mL	average volume, ml
1.		_____
2.		
3.		

13. Calculate precision concentration of the sodium thiosulphate standard solution accordance to equivalents law.

$$\overset{x}{K_2Cr_2O_7} + 6KI + 7H_2SO_4 \rightarrow \overset{3x}{3I_2} + Cr_2(SO_4)_3 + 4K_2SO_4 + 7H_2O$$

$$\overset{3x}{I_2} + \overset{6x}{2Na_2S_2O_3} \rightarrow 2NaI + Na_2S_4O_6$$

$$n\,(Na_2S_2O_3) = 6n\,(K_2Cr_2O_7)$$

$$C(Na_2S_2O_3) = \frac{6 \cdot m(K_2Cr_2O_7) \cdot 10{,}00}{M(K_2Cr_2O_7) \cdot 0{,}1000 \cdot \overline{V}(Na_2S_2O_3)}$$

The results you present to teacher.