

Ministry Of Higher Education and scientific Research

Al-Mustansiriya University

Collage of Science

Department of Chemistry

Practice Volumetric Chemical Analysis

First year / 2012



Edited by

Ruba Fahmi Abbas

Supervised by

Lecturer Dr.

Mohammed Abdul-Hadi Khalil

Lecturer Dr.

Abbas Shebeeb Hasan

3//Reduction - Oxidation titration (Re-Dox):-

Is a type of titration based on a redox reaction between the analyte and titrant. Redox titration may involve the use of a redox indicator. A titration characterized by the transfer of electrons from one substance to another (from the reductant to the oxidant) with the end point determined calorimetrically or potentiometrically or by titration.

A redox indicator (also called an oxidation-reduction indicator) is an indicator that undergoes a definite color change at a specific electrode potential.

The requirement for fast and reversible color change means that the oxidation-reduction equilibrium for redox indicator system needs to be established very fast. Therefore only a few classes of organic redox systems can be used for indicator purposes. There are two common types of redox indicators:

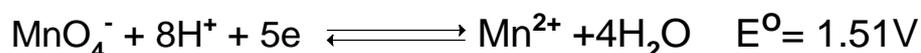
- metal-organic complexes (Ex. phenanthroline)
- true organic redox systems (Ex. Methylene blue)

Sometimes colored inorganic oxidants or reductants (Ex. Potassium manganate, Potassium dichromate) are also incorrectly called redox indicators.

Almost all redox indicators with true organic redox systems involve a proton as a participant in their electrochemical reaction.

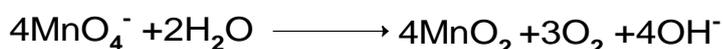
Experiment No.(6):- Determination of iron (Fe⁺²)

Theory:-Permanganate ion is strong oxidation reagent, half – reaction for the permanganate is:



Permanganate solution decomposes slowly and thus require occasional restandardization.

Aqueous solution of permanganate is not stable because the ion tends to oxidize water:



The reaction between iron and permanganate performed in acidic medium:



Procedure:-

A-Prepare 0.1 N oxalic acid in 50 ml volumetric flask.

$$0.1 = \frac{\text{Wt.}}{\text{Eq.Wt.}} * \frac{1000}{50}$$

B-Prepare (50 ml) 0.1 N KMNO₄:-

$$0.1 = \frac{\text{Wt.}}{\text{Eq.Wt.}} * \frac{1000}{50}$$

Weigh 0.6 g KMnO₄. Dissolve with D.W in volumetric flask capacity 50 ml. Heat the solution for one hour, filtrate the solution with glass wool.

C- Standarazation of KMNO₄ solution:-

1. Transfer 5 ml from oxalic acid , with pipette, to conical flask capacity 300 ml
2. Add 5 ml dilute sulphuric acid.
3. Heat the mixture on the water bath for 5 min.
4. Titration against KMNO₄ until the faint pink color is appear.
5. Repeat step 3 three time.
6. Calculate the normality of permanganate

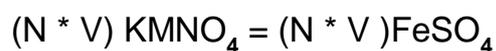
$$(N * V) \text{KMNO}_4 = (N * V) \text{H}_2\text{C}_2\text{O}_4$$

$$N * V = 0.1 * 5$$

D-Determination of FeSO₄ solution:-

1. Transfer 10 ml from FeSO₄.

2. add 5 ml dilute sulphuric acid,
3. Titration against KMnO_4 until the faint pink color is appear
4. Repeat this step three times.
5. Calculate the normality of FeSO_4 :-



$$N * V = ? * 10$$

Discussion:-

1. Why is potassium permanganate not used as a primary standard?
2. Not use indicator why?
3. Added sulphuric acid why?
4. Heat and filtrate the mixture why?
5. Why is the color of solution change from colorless to pink?
6. What equivalent weight of KMnO_4 in acidic, basic and neutral medium?