جامعة الانبار

كلية : الصيدلة

قسم : الكيمياء الصيدلانية

اسم المادة باللغة العربية: الكيمياء التحليلية

اسم المدة باللغة الإنكليزية: **Analytical Chemistry**

المرحلة: الأولى

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عنوان المحاضرة باللغة العربية: الكيمياء التحليليلة العملي التجربة السابعة

عنوان المحاضرة باللغة الإنكليزية: **Practical Analytical Chemistry (7)**

Exp. 7

Preparation and Standardization of 0.1 N $KMnO_4$

Potassium permanganate, a powerful oxidant, is perhaps the most widely used of all standard oxidizing agents. The color of a permanganate solution is so intense that an indicator is not ordinarily required. The reagent is readily available at modest cost. On the other hand, the tendency of permanganate to oxidize chloride ion is a disadvantage because hydrochloric acid is such a useful solvent.

One of the most obvious properties of potassium permanganate is its intense purple color, which commonly serves as the indicator for titrations.

The permanganate end point is not permanent and gradually fades. Decolorization results from the reaction of the excess permanganate with the relatively large concentration of manganese (II) ion that is present at the end point.

$$2MnO_4^- + 3Mn^{2+} + 2H_2O \qquad\qquad 5MnO_2(s) + 4H^+$$

Solid potassium permanganate, which is ordinarily used for the preparation of permanganate solutions, is inevitably contaminated with manganese dioxide to some extent; as a result, preparation of standard permanganate solutions directly by weight is not possible.

A permanganate solution possessing reasonable stability can be obtained provided a number of precautions are observed. Perhaps the most important variable affecting stability is the catalytic influence of manganese dioxide. This contaminant occurs in the starting material and is also produced when permanganate oxidizes organic matter in the water used to prepare the solution. Removal of manganese dioxide by filtration markedly enhances the stability of standard permanganate solutions. Sufficient time must be allowed for complete oxidation of contaminants in the water before filtration; the solution may be boiled to hasten the oxidation. Paper cannot be used for the filtration since it reacts with the permanganate to form the undesirable dioxide.

Standardized solutions should be stored in the dark. If any solid is detected in the solution, filtration and restandardization are necessary. In any event, restandardization every one to two weeks is a good precautionary measure.

In acidic solution, permanganate oxidizes oxalic acid to carbon dioxide and water.

The reaction of permanganate (oxidizing agent) with oxalic acid (reducing agent) is oxidation-reduction reaction, involves electron transfer.

The permanganate solution is added from the buret to the solution of oxalic acid in the conical flask until the color is changed to a pink in the end point.

$$2(MnO_4^- + 8H^+ + 5e^- \qquad\qquad Mn^{2+} + 4H_2O) \quad +7 \qquad +2$$

$$5(C_2O_4^{2-} \qquad\qquad\qquad 2CO_2 + 2e^-) \quad +3 \qquad +4$$

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

The complete chemical equation is:

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

Procedure

Preparation of 0.1 N $KMnO_4$

Dissolve 3.2 g of $KMnO_4$ in about 1 liter of distilled water. Heat to boiling, and keep hot for about 1 hr. Cover and let stand overnight. Filter the solution through a fine-porosity sintered glass crucible or through a Gooch crucible with an asbestos mat. Store the solution in a clean, glass-stoppered bottle, and keep in the dark when not in use.

Standardization against oxalic acid

Full the buret with $KMnO_4$ solution. Transfer by a pipette 10 ml from the 0.1N oxalic acid ($H_2C_2O_4$) solution to a conical flask, and add 10 ml 4N H_2SO_4 solution. Heat the mixture to 60-90°C for few minutes, and titrate by adding the permanganate solution, stirring vigorously with a thermometer. The reagent should be introduced slowly so that the pink color is discharged before further additions are made. If the solution temperature drops below 60°C heat. The end point is the first persistent pink color.

If a brown precipitate is formed during titration, it is attributed to one of the followings:

1- The temperature degree of the solution below 60°C.
2- The addition of permanganate was fast.
3- The amount of H_2SO_4 is not sufficient.
4- The vigorous heating lead to dissociation of oxalic acid.

$$\text{no. of the } KMnO_4 \text{ milliequivalents} = \text{no. of the } H_2C_2O_4 \text{ milliequivalents}$$

$$N_1 \times V_1 = N_2 \times V_2$$

$$N_1 \times (\text{from buret}) = 0.1 \times 10$$

The strength of permanganate solution = N_1 × equivalent weight (molecular wt./5)
= $N_1 \times 158/5$