Redox Titration

Purpose

Understand the definition of redox and the principle of equivalent titration

Principle

Oxidation generally refers to an increase in the oxidation state (or valence) of an atom or an ion, that is, an electron loss.In principle, a reduction in the oxidation state indicates an electron is obtained.The oxidation-reduction (redox) reaction is referred to as an electron transfer reaction. In a redox reaction, if one of the reactants is oxidized, the other reactant must be reduced.Many elements, especially transition metal elements, can exhibit multiple oxidation states. Take manganese as an example, there may be five oxidation states of 2^+, 3^+, 4^+, 6^+, and 7^+, of which 6^+ and 3^+ areveryunstable and existed only in aqueous solution. Permanganate ion, MnO_4^-, is a strong oxidant and it is easy to oxidize other reactants, i.e. it is easily reduced by itself.In this experiment, you will start with an acidic solution of permanganate ions, calibrate the concentration with a known amount of sodium oxalate, $Na_2C_2O_4$, and then use the potassium permanganate solution as a standard solution to analyze the oxalic acid of the unknown samples. The content of root, the reaction of potassium permanganate and sodium oxalate in sulfuric acid solution is

$$2MnO_4^-(aq) + 5C_2O_4^{2-}(aq) + 16H^+(aq) \rightarrow 2Mn^{2+}(aq) + 10CO_2(g) + 8H_2O(l)$$

In this reaction, Mn(VII) is reduced to Mn(II) and C(III) is oxidized to C(IV). In an oxidation-reduction reaction, not all atoms in a reactant undergo redox, but in fact only a few atoms participate in the reaction. These atoms are called "redox atoms". The equivalent weight of the reactants is the number of molecular weights divided by the number ofoxidation-reduction states of the contained redox atoms, and the mass of one equivalent of reactants simultaneously contains the mass of one equivalent of redox atoms.In dilute solution, MnO_4^- is pink, and Mn^{2+} is colorless, so MnO_4^- itself is a good indicator during titration. $C_2O_4^{2-}$is titrated with MnO_4^-. When MnO_4^-isdropped into the $C_2O_4^{2-}$ solution, all MnO_4^-isreduced to Mn^{2+} and the solution remains colorless until all the $C_2O_4^{2-}$ reactions arecompleted, at which point the next drop of MnO_4^-becomes excessive and the solution turnsPink indicating the end of the titration.

Procedure

(A) Calibration of the concentration of MnO_4^-solution

（1） In a 250 mL beaker, weigh approximately 0.10 g of dried $Na_2C_2O_4$,add 20mL of water to dissolve $Na_2C_2O_4$, and then slowly add 2.0mL of 18M H_2SO_4 to acidify. Stir the mixture and heat the solution to 80°C (not to exceed 90°C). Immediately titrate with MnO_4^-solution and carefully stir with glass rod (or stirrer). Record the desired volume of MnO_4^- solution at the end of the titration (light pink, at least 15 seconds). Always pay attention to the solution temperature, keep the solution temperature at 60 ~ 80°C, and reheat if necessary.

（2） Repeat the procedureonce

(B) Analysis ofoxalateinunknown sample

(1) Accurately weigh about 0.10 g of the previously dried unknown sample (note the unknown number)
(2) Repeat step (A) once
(3) Calculate the weight and percentage of$C_2O_4^{2-}$by mass of the unknown sample.

Safety

1. The remaining untitrated 0.1 N $KMnO_4$ solution (including the buret) should be poured into the recycle bottle.
2. Waste liquid should be poured into the waste container.
3. Remove the burette hydrant switch, soak the oxalic acid solution to wash the stain, and place it in order (thin gasket → rubber ring → knob). After checking by the assistant, put it back in the burette rack.

Data

(A) Calibration of the concentration of MnO_4^- solution

#	1	2
$Na_2C_2O_4$ mass（g）		
$KMnO_4$ volumebefore titrate（mL）		
$KMnO_4$ volume after titrate (mL)		
$KMnO_4$volume used（mL）		
$KMnO_4$concentration（N）		
$KMnO_4$average concentration（N）		

Formula:

Equivalent number of $Na_2C_2O_4$ = Equivalent number of $KMnO_4$

$$\frac{W_t(Na_2C_2O_4)}{134/2} = N_{KMnO_4} \times V_{KMnO_4}(L)$$

（B）Analysis unknown of oxalate

#	1	2
Unknown sample mass（g）		
$KMnO_4$ volume before titrate（mL）		
$KMnO_4$ volume after titrate (mL)		
$KMnO_4$ volume used（mL）		
$C_2O_4^{2-}$ percentage by mass（%）		
Average percent by mass（%）		