Chemical Analysis by Redox Titration

Introduction:

Titration is a common method for determining the amount or concentration of an unknown substance. The method is easy to use if the quantitative relationship between two reacting substances is known. The method is particularly well-suited to acid-base and oxidation-reduction reactions. In this experiment, you will conduct redox titrations using a standardized permanganate solution. You will be trying to find the % hydrogen peroxide in a commercially sold solution.

Permanganate ion is a powerful oxidizing agent, especially in acidic solution, which can be used to analyze (by titration) solutions containing many different species. In these titration reactions, the intensely colored MnO_4^- ion is reduced to form the colorless Mn^{2+} ion.

Species Titrated / Species Formed in Titration

H_2O_2 O_2

An advantage of using the permanganate ion in the titration of colorless unknown solutions is that it is "self-indicating". As long as the reducing agent remains present in the sample, the color of MnO_4^- quickly disappears as it is reduced to Mn^{2+}. However, at the endpoint, all the reducing agent has been used up so the next drop of MnO_4 solution is sufficient to cause an easily detected color change, from colorless to a faint, permanent peach/pink. So we know that at the endpoint, the oxidizing agent (MnO_4^-) and reducing agent (H_2O_2) have reacted exactly in proportion to their stoichiometry in the balanced redox equation. If we know how much of the oxidizing agent we added, then we can figure out exactly how much reducing agent was present in the unknown.

Titration Notes:

1. Always rinse buret with water (from a beaker, not the faucet) first. Second, rinse with a small amount of the titrant and drain it through the tip.
2. Fill the buret with the titrant using a funnel.
3. Fill the buret tip by momentarily opening the pinchclamp or stopcock.
4. Now you are ready to read the initial volume (bottom of the meniscus). Remember that burets are graduated in a downward direction. The first estimated digit will probably be the hundredths place.
5. Do not waste time trying to fill the buret to zero for each titration.
6. Do not start above the 0 mL mark or titrate past the 50 mL mark.
7. Always use white paper underneath your sample flask so that you will notice slight color changes.
8. Learn to swirl the flask without removing it from underneath the buret.
9. Use a drop, drop, drop pace until you see the color change becoming more than local (where the titrant meets the sample). Now proceed dropwise.
10. Second and third trial titrations should always be fast assuming the sample will be about the same because you now know approximately how much titrant is needed. If the first titration required 25 mL than you can add 22 mL all at once and then proceed cautiously.
11. Remember that the amount of water used to dilute the sample is not crucial because it does not affect "how many" of the sample molecules are present in the sample flask. Diluting with water allows you to see the color change easier.
12. Always rinse sample flasks before using.
13. Always label multiple burets and sample flasks.
14. Did you add your indicator?

Materials

50 mL buret, ringstand, buret clamp
250 mL Erlenmeyer flask
0.100 $KMnO_4$ Solution (acidic)
3.0 M H_2SO_4
White paper
Commercial hydrogen peroxide solution
Distilled water bottle

Goggles Must Be Worn at All Times in the Lab!!!

PART A: Titration of an Unknown Hydrogen Peroxide Solution

Procedure:

1. Obtain approx. 10.xxx g (record its mass accurately) of the commercial hydrogen peroxide solution. Transfer the solution to a clean 250 mL Erlenmeyer flask. Rinse the peroxide container twice with a small amount of water and add the rinsings to the flask. Dilute the sample in the flask to about 75 mL with water, then add about 20 mL of 3 M H_2SO_4.
2. Obtain a 50 mL buret and rinse it with water. Do a final rinse with a small amount (5 mL) of the standard MnO_4^- solution. Fill the buret with the standard solution. Fill the buret tip by momentarily opening the stopcock. Record the initial reading.
3. Place a sheet of white paper under the sample flask. Now slowly begin titrating the H_2O_2 solution while it is continuously being stirred by gently swirling the flask. Continue titrating until you see the color of MnO_4^- begin to persist locally in the solution, at which point, you should slow down to dropwise additions. Continue until one added drop of MnO_4^- solution produces a faint peach/pink color that lasts at least 30 seconds. This is the first excess MnO_4^- which is not being reduced by the H_2O_2. Record the final buret reading.
4. Complete a second trial. For excellent work, the calculated percents need to be within 1% of each other.

Data:

* mass of H_2O_2 sample for each trial
* concentration of standard solution
* initial buret reading for each trial
* final buret reading for each trial
* volume of MnO_4^- used in each trial
* observations of reaction

Calculations:

1. Write the balanced net ionic equation for the reaction (show the working equation as well). Identify the oxidizing and reducing agents.
2. Calculate the milligrams of H_2O_2 in the sample for each trial.
3. Calculate the $\%H_2O_2$ by mass in the commercial sample for each trial.
4. Calculate the average $\%H_2O_2$ in the commercial hydrogen peroxide solution.