

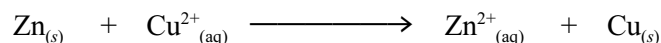
Lab: Quantitative Redox Titration Involving Permanganate Ion

Objective

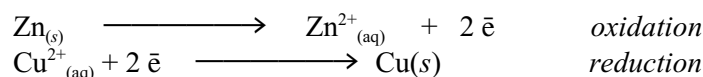
In this experiment, one of the main types of chemical reactions, oxidation/reduction (or redox) reactions, will be used in the titration analysis of an iron compound, and in the determination of the percentage of hydrogen peroxide in ordinary hydrogen peroxide.

Introduction

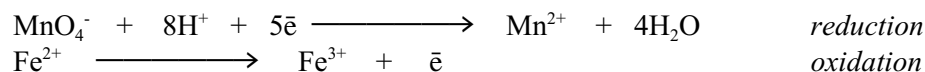
Oxidation/reduction processes form one of the major classes of chemical reactions. In redox reactions, electrons are transferred from one species to another. For example, in the following simple reaction:



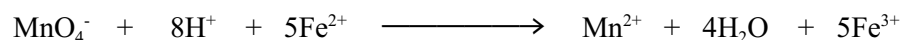
electrons are transferred from elemental metallic zinc to aqueous copper(II) ions. This is most easily seen if the overall reaction is written instead as two half-reactions, one for the oxidation and one for the reduction:



In this experiment you will use potassium permanganate, KMnO_4 , as the titrant in the analysis of an unknown sample containing **iron**. Although this method can easily be applied to the analysis of realistic iron *ores* (i.e. – the native state in which an element is found in nature), for simplicity your unknown sample will contain only iron in the +2 oxidation state. In acidic solution, potassium permanganate rapidly and quantitatively *oxidizes* iron(II) to iron(III), while itself being *reduced* to manganese(II). The half reactions for the process are:



When these half-reactions are combined to give the overall balanced chemical reaction equation, a factor of *five* has to be used with the iron half-reaction *so* that the number of electrons lost in the overall oxidation will equal the number of electrons gained in the reduction:



Potassium permanganate has been one of the most commonly used oxidizing agents because it is extremely powerful, as well as inexpensive and readily available; and serves as a chemical indicator.

Potassium permanganate is especially useful among titrants since it requires no indicator to signal the endpoint of a titrant. Potassium permanganate solutions — even at fairly dilute concentrations — are intensely colored purple. The product of the permanganate reduction half-reaction, manganese(II), in dilute solution shows almost *no* color. Therefore, during a titration using KMnO_4 , when one drop *excess* of potassium permanganate has been added to the sample, the sample will take on a pale red/pink color (since there are no more sample molecules left to convert the purple MnO_4^- ions to the colorless Mn^{2+} ions).

It does have some drawbacks, however. Because KMnO_4 is such a strong oxidizing agent, it reacts with practically *anything* that can be oxidized: this tends to make solutions of KMnO_4 difficult to store without decomposition or a change in concentration. Because of this limitation, it is common to prepare, standardize, and then use KMnO_4 solutions for an analysis all on the same day. It is *not* possible to prepare directly KMnO_4 standard solutions determinately by mass: solid potassium permanganate cannot be obtained in a completely pure state due to the high reactivity mentioned above. Rather, potassium permanganate solutions are prepared to be an approximate concentration, and are then standardized against a known primary standard sample of the same substance which is to be analyzed in the unknown sample.

Safety

1. Potassium permanganate is a strong oxidizing agent and can be damaging to skin, eyes and clothing. Wash after handling.
2. Potassium permanganate will stain skin and clothing if spilled.
3. Sulphuric acid solutions are damaging to the skin, eyes and clothing — especially if allowed to concentrate through evaporation of water. If sulphuric acid solution is spilled on the skin, wash immediately with copious amounts of water.
4. Iron salts may be irritating to the skin. Wash after handling.
5. Both sulphuric acid and potassium permanganate can react violently with hydrogen peroxide.

Apparatus

Buret and clamp with stand, Erlenmeyer flasks, beakers, suction pump, pipets, filter funnel

Reagents

0.0200 mol dm⁻³ KMnO_{4(aq)}, 3.00 mol dm⁻³ H₂SO_{4(aq)}, unknown concentration of FeSO_{4(aq)}, sample of H₂O₂

Procedure

Part A: To determine the amount of iron in an unknown iron (II) solution

1. Set up a titration apparatus. Rinse the buret with a small amount of KMnO₄ solution. Completely fill the buret with freshly prepared 0.0200 mol dm⁻³ KMnO_{4(aq)}. (Note: since potassium permanganate solutions are so intensely coloured, it is generally impossible to see the curved meniscus that the solution forms in the buret. In this case, it is acceptable to make your liquid level readings at the point where the top surface of the permanganate solution comes in contact with the wall of the buret. A piece of paper held behind the buret may be of help also in the reading of the buret.) Record the initial buret reading to the nearest 0.02 cm³ in Table I.
2. Obtain a sample of an unknown concentration of FeSO_{4(aq)} and place it in a 100 cm³ beaker.
3. Pipet 25 cm³ of the FeSO_{4(aq)} using a suction pump. Add enough distilled water to each flask to make up about 50 cm³.
4. Add exactly 10.0 cm³ of 3.00 mol dm⁻³ H₂SO_{4(aq)}
5. Place the flask containing the unknown iron (II) sample under the tip of the buret, and begin adding potassium permanganate to the sample a few cm³ at a time, swirling the flask after each addition of permanganate.
6. Continue adding the permanganate a few cm³ at a time, with swirling, until the red streaks begin to become more persistent.
7. At this point, begin adding the permanganate one drop at a time, swirling the flask constantly to mix. The end-point is the appearance of a permanent pink colour.
8. After the end-point has been reached, record the final buret reading, (to the nearest 0.02 cm³). Calculate the volume of permanganate solution used to the nearest 0.02 cm³.

Part B: To determine the Percentage of H₂O₂ in a Hydrogen Peroxide sample.

1. Retain the apparatus from Part A.
2. Determine the mass of two clean dry 250 cm³ flasks, and record in Table II.
3. Carefully transfer between 1.00 cm³ and 2.00 cm³ of H₂O₂ into each of the two flasks using a 10 cm³ pipet and a suction pump. (It is preferable to use a different volume in each of the two flasks).
4. Determine the mass of each flask and record in Table II.
5. Add ~ 35 cm³ of distilled water and 10 cm³ of 3.00 mol dm⁻³ H₂SO_{4(aq)} to each flask.
6. Titrate each flask with 0.0200 mol dm⁻³ KMnO_{4(aq)} to a slightly pink end point.
7. Record all buret readings in Table II.

Data Table I: Determining the Concentration of a Solution of Fe⁺²

	Trial 1	Trial 2	Trial 3
Unknown #, [KMnO _{4(aq)}] = 0.0200 mol dm ⁻³			
Initial volume of KMnO _{4(aq)}			
Final volume of KMnO _{4(aq)}			
Volume of KMnO _{4(aq)} required			

Data Table II: Determining the Concentration of a H₂O₂ solution

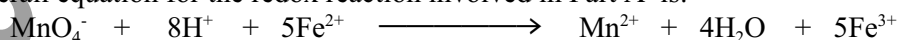
[KMnO _{4(aq)}] = 0.0200 mol dm ⁻³	Trial 1	Trial 2
mass of empty flask (g)		
mass of flask + H ₂ O ₂ (g)		
Initial volume of KMnO _{4(aq)} (cm ³)		
Final volume of KMnO _{4(aq)} (cm ³)		
Volume of KMnO _{4(aq)} required (cm ³)		

Data Analysis

Balance the overall redox reaction for each reactions. Once the reactions are balanced, you can use the stoichiometric mole ratio to complete your calculations.

Part A

The overall equation for the redox reaction involved in Part A is:



From Data Table I, determine the average volume of 0.200 mol dm⁻³ KMnO_{4(aq)} used to titrate the sample of Fe⁺².

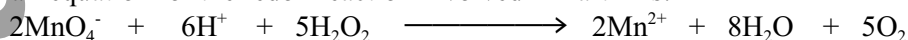
Calculate the number of moles of potassium permanganate required for each Fe⁺² sample.

Calculate the number of moles of Fe⁺² present in the sample, from the balanced equation.

Knowing that a 25.0 cm³ sample was used for the titration, determine the concentration of Fe⁺² in the sample.

Part 2

The overall equation for the redox reaction involved in Part B is:



Using Trial 1, determine the volume of KMnO_{4(aq)} used, and hence the number of moles of MnO₄⁻¹.

Use the balanced equation, to determine the number of mole of H₂O₂ used.

Knowing the molar mass of H₂O₂, determine the mass of H₂O₂ in the sample.

Hence, determine the percentage of H₂O₂ in the solution, and compare with the percentage given on the bottle.

Use similar calculation for Trial 2, and thus, determine the average value for the percentage of H₂O₂ in the sample.

Conclusion

Provide a concluding statement for the objective of this lab.