

### **Experiment 3**

#### **Determination of the Percentage of Oxalate in Iron(II) Oxalate by Redox**

##### **Titration**

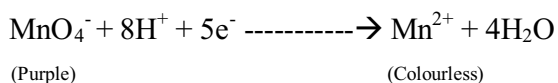
28-9-2005

##### **Objective**

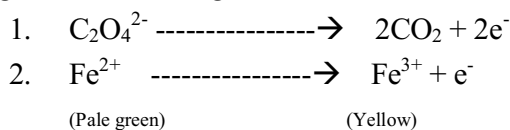
To determine the percentage by mass of oxalate in a sample of iron(II) oxalate

##### **Introduction**

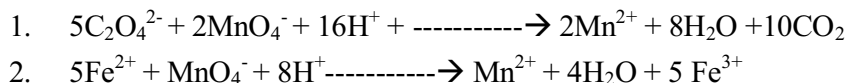
Acidified potassium permanganate is a strong oxidizing agent. It oxidizes substances and reduces itself.



Both iron (II) and oxalate ions can be oxidized. When they come in contact with an oxidizing agent, the following reactions occur:



So, when potassium permanganate is added to acidified iron (II) oxalate, two redox reactions occur.



Notice the color changes of the solution. At the beginning, the color of solution inside the conical flask should be pale green due to the iron (II) ions present. Then after a certain amount of potassium permanganate is added, it should change to yellow because iron (III) ions are formed. Finally, the color of the solution should change from yellow to purple after 1 excess drop of permanganate is added. This is because there would be no more reducing agents to react with the permanganate ions, and the purple color of the permanganate ions would be seen in the conical flask.

##### **Procedure**

1. Approximately 2 grams of iron (II) oxalate was weighed out with a weighing bottle.
2. The oxalate powder was poured into a beaker. Dilute sulphuric acid was added.
3. The beaker was heated and the solution inside was stirred until the solution turns very pale yellow..
4. The solution in the beaker was poured into a volumetric flask and made up to 250 cm<sup>3</sup> with distilled water.
5. Potassium permanganate was poured into a burette.
6. 25 cm<sup>3</sup> of the oxalate solution was transferred to a conical flask. About 10 cm<sup>3</sup> of sulphuric acid was added into the conical flask.

7. The conical flask was heated until the solution inside reaches 60°C.
8. A few drops of potassium permanganate were added to the conical flask. The flask was shaken until a yellow colour appeared.
9. Potassium permanganate was further added until the solution turned pink. The reading was taken.
10. Steps 5 to 9 was repeated 3 more times.

### **Data and Calculation**

Weight of bottle with  $\text{FeC}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$  = 10.093g

Weight of bottle after  $\text{FeC}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$  was poured into beaker = 8.311g

Mass of  $\text{FeC}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$  used = 1.782g

|                                              | 1                    | 2                    | 3                    | 4                    |
|----------------------------------------------|----------------------|----------------------|----------------------|----------------------|
| Final reading                                | 29.9 cm <sup>3</sup> | 29.0 cm <sup>3</sup> | 29.9 cm <sup>3</sup> | 29.4 cm <sup>3</sup> |
| Initial reading                              | 2.6 cm <sup>3</sup>  | 1.7 cm <sup>3</sup>  | 2.5 cm <sup>3</sup>  | 2.0 cm <sup>3</sup>  |
| Volume of $\text{KMnO}_4$ used<br>(rejected) | 27.3 cm <sup>3</sup> | 27.3 cm <sup>3</sup> | 27.4 cm <sup>3</sup> | 27.4 cm <sup>3</sup> |

### **Volume of $\text{KMnO}_4$ used in titration**

Average volume of  $\text{KMnO}_4$  used =  $(27.3 + 27.4 + 27.4)/3 = 27.367 \text{ cm}^3$

No. of moles of  $\text{KMnO}_4$  used =  $27.367/1000 \times 0.02 = 5.473 \times 10^{-4}$  moles

For every 3 moles of  $\text{MnO}_4^-$  used, 5 moles of  $\text{FeC}_2\text{O}_4$  is reacted.

So, no. of moles of  $\text{FeC}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$  in every 25 cm<sup>3</sup> =  $5.473 \times 10^{-4} / 3 \times 5 = 9.122 \times 10^{-4}$  moles

There are also  $9.122 \times 10^{-4}$  moles of oxalate in every 25 cm<sup>3</sup>. Then there would be  $9.122 \times 10^{-3}$  moles of oxalate in 250 cm<sup>3</sup> of the solution.

Weight of  $\text{C}_2\text{O}_4^{2-}$  in the sample =  $9.122 \times 10^{-3} \times (12 \times 2 + 16 \times 4) = 0.803\text{g}$

Percentage by mass of  $\text{C}_2\text{O}_4^{2-}$  in the sample (found by experiment)  
 $= 0.803 / 1.782 \times 100\% = 45.04\%$

Molar mass of  $\text{FeC}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$  as written on its bottle = 179.9 g/mole

Percentage by mass of  $\text{C}_2\text{O}_4^{2-}$  in the sample (theoretically)  
 $= (24 + 16 \times 4) / 179.9 \times 100\% = 48.92\%$

### **Conclusion**

Percentage by mass of  $\text{C}_2\text{O}_4^{2-}$  in the sample, as found by experiment, is 45.04%.  
 Percentage by mass of  $\text{C}_2\text{O}_4^{2-}$  in theory is 48.92%.

### **Discussion**

1. There was one procedure that I mentioned in the procedures section. It was that the iron (II) oxalate needed to be heated to 60°C before it can be titrated.

Why is this step necessary? It was because the redox reaction between oxalate ions and permanganate ions were too slow. Why was the redox reaction between the two ions slow? This is due to 2 reasons. First, both ions are anions. They both carry the same negative charge. So, when they come close together, they would repel each other. The second reason is because of the structure of the 2 ions. The reactions between the two ions involve a lot of bond breaking and bond formation. So, it would take a long time for reaction.

Then to speed up the reaction of the oxalate and permanganate ions, the solution needs to be heated before it is titrated. But doing so needs a lot of time. How can we shorten the time required for heating the solution up to  $60^{\circ}\text{C}$ ? There are 2 ways. First, we can heat the conical flask with a stronger flame. We can adjust the flame intensity by enlarging the air hole. Second, we can heat 1 conical flask and titrate another one which has been heated at the same time. These methods can be used but I only used the first one.

Why? If I use the second method, it requires a lot of skill. If the conical flask being heated boils while I was titrating the other one, it would be very dangerous. So, I only used a stronger flame.

### **Answers to Study Question**

1. This is because the permanganate ions itself can act as an indicator. Before it is reacted, the permanganate ions are purple in colour. When it is dropped into the conical flask and reduced, it turns to manganese ions and changes to colorless. So, the solution in the conical flask retains its color before titration. But after all the reducing agents in the conical flask has reacted, adding 1 more drop of permanganate ion will change the whole color of the solution pink.
2. If brown precipitates were formed during titration, I need to add more sulphuric acid and reheat the solution. Adding sulphuric acid can dissolve the manganese (IV) oxide. The temperature is lowered in this step. So, the solution needs to be reheated again.
3. The redox reaction takes a relatively long time to complete. So, to allow the redox reaction to go to completion before permanganate ions are further added, it has to be shaken well and for a period of time. This would allow the colour to change before any permanganate ions are added.
4. As mentioned above, the heating of the solution is to speed up the reaction. If heating is insufficient, it would take a long time for the purple color of the permanganate which is just added to change colour. So, a persistent colour

change of the solution would not indicate the end point.

5. To make sure that exactly  $25\text{cm}^3$  of oxalate solution is obtained, pipetting of it must be done with great care. Otherwise, if more or less of the solution is obtained, the titrations would become inaccurate.
6. It takes time to let the oxalate ions, iron (II) ions and permanganate ions to react with each other. So, the permanganate ions may not be decolourized directly. Also, at the start, the permanganate ions experience more repulsion from the oxalate ions. So, it may need more time for the permanganate ions to react with the oxalate ions.
7. Add an excess of potassium iodide and sulphuric acid into  $25\text{cm}^3$  of potassium iodate. Pour this solution into a conical flask. Then, pour sodium thiosulphate into a burette. Titrate the solution in the conical flask with the thiosulphate. Just as the brown colour of iodine fades, add a drop of starch indicator. Find out the volume of thiosulphate required to decolourize the blue-black color of the starch solution. Then calculate the number of moles of thiosulphate ions used in the titration, and the molarity would be found.