

Preparation of oxalate complexes of iron biology essay



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To prepare two oxalate complexes of iron namely, Potassium Trioxalatoferrate Trihydrate and Iron Oxalate and to analyse the products for iron and oxalate respectively.

One of the properties known to be characterised by transition metals such as iron is complex ion formation since they are able to form stable complexes. In this experiment, two complex of iron are being formed with the oxalate ion being the common ligand in both. Potassium Trioxalatoferrate (III) Trihydrate and Iron (II) Oxalate are the two complexes being formed and are represented by the following chemical structures:

Figure 1: Chemical structures of Potassium Trioxalatoferrate (III) Trihydrate and Iron (II) Oxalate respectively.

The oxalate ion, apart from acting as a Lewis base can be referred to as a bidentate ligand since an oxalate ion can donate two pairs of electrons (one from each oxygen) to the iron (III) or Iron (II) cation acting as a Lewis acid from two oxygen atoms as can be seen in figure 1 above.

Iron can form a variety of complexes with most of them having an octahedral geometry. In this experiment, the Iron (II) oxalate formed is characterised by an Fe^{2+} as the central metal cation. This is then oxidised to Fe^{3+} in order to synthesise the Potassium Trioxalatoferrate (III) Trihydrate complex characterised by an Fe^{3+} as the central metal cation.

Certain complexes such as the Potassium Trioxalatoferrate (III) Trihydrate complex are unstable to light and therefore they are said to be photosensitive. For this reason, it is a must to store such a complexes under

dark conditions in order to prevent the reduction of the Fe^{3+} ion back to the Fe^{2+} ion.

The amount of oxalate within a complex can be determined using titrimetric analysis. Potassium permanganate is titrated with the oxalate ion and the amount of oxalate can be determined through this redox titration. No indicator is necessary in such a titration due to the fact that the endpoint is characterised by a faint pink colour resulting from the fact that at the endpoint, excess unreacted permanganate ions are present in the solution since all the oxalate ions would have been consumed.

The amount of iron in a complex on the other hand can be analysed following the addition of zinc to the complex solution followed by heating. Once this is carried out, the resulting solutions can be treated with potassium permanganate in a redox titration as described previously above and hence, the amount of iron in a complex can be determined.

In this experiment, heating is involved in the redox titrations due to the fact that since the reaction is rather slow at room temperature, in order for one to observe a quick colour change at the endpoint, the solution needs to be heated to around 60°C .

Method

Chemicals used

Ferrous ammonium sulphate

Hydrogen peroxide

Sulfuric acid

Ethanol

Oxalic acid

Zinc

Ferrous oxalate

Potassium permanganate

Potassium oxalate

Apparatus used

Buchner funnel

Heating mantle

Burette

Weighing boat

Thermometer

Filter paper

Magnetic stirrer

Glass wool

Analytical balance

Measuring cylinder

Procedure

Part a – Preparation of Iron (II) oxalate

15g of ferrous ammonium sulphate were dissolved in 50mL warm water which had been acidified with 1mL 2M sulfuric acid.

To this, a solution of 10% 75mL oxalic acid was added with rapid stirring. The mixture was gently heated until its boiling point was reached and the yellow precipitate of ferrous oxalate formed was allowed to settle.

The precipitate was removed by filtration on a Buchner funnel and washed thoroughly with hot water followed by acetone.

The product was allowed to dry on a funnel under suction and was then weighed.

Part B – Preparation of Potassium Trioxalatoferrate (III) Trihydrate.

3. 25g of ferrous oxalate was suspended in a warm solution of (5g in 15mL water) potassium oxalate.

To this, 15mL 20 vol. Hydrogen peroxide was added from a burette whilst the solution was stirred continuously and the temperature was maintained at 40°C.

The solution contained a precipitate of ferric hydroxide and this was removed by heating the solution to its boiling point and adding 10mL 10% oxalic acid.

Further small amounts of oxalic acid was added drop wise until the precipitate just dissolved.

The hot solution was filtered and 15mL ethanol was added to the filtrate in order to re dissolve any crystals that formed by gentle heating.

The solution was placed in a dark cupboard to crystallize since the product formed was photosensitive.

The crystals were collected by filtration on a Buchner funnel and later washed with an equivolume mixture of ethanol and water followed by acetone. The crystals were then dried and weighed.

Part C – The analysis of the products for Iron and Oxalate

For Iron (II) oxalate:

0. 3g of oxalate were dissolved in 25mL 2M sulfuric acid and the solution was heated to 60°C and titrated with 0. 2M standard potassium permanganate solution until the first permanganate pink colour was observed.

2g of zinc dust was added and the solution was boiled for 25 minutes. The solution was filtered through glass wool and the residual zinc was washed with 2M sulfuric acid.

The washings were added to the filtrate and the solution was titrated with standard potassium permanganate.

The percentages of iron, oxalate and water of recrystallisation in the product were determined and hence, the empirical formula could be derived.

For Potassium trioxalatoferrate (III) trihydrate:

0.2 g of Potassium trioxalatoferrate (III) trihydrate were dissolved in 25 mL 2M sulfuric acid and titrated with 0.02M permanganate.

The solution was treated with zinc dust and re-titrated with permanganate as described in the analysis of Iron (II) oxalate above.

The percentages of iron and oxalate in the complex were determined and this was compared to the theoretical value.

Precautions:

It was made sure that in the preparation of Potassium Trioxalatoferrate (III) Trihydrate, ethanol was added to the filtrate in order to re-dissolve any crystals that formed by gentle heating.

It was made sure that in the preparation of Potassium Trioxalatoferrate (III) Trihydrate, the solution was placed in a dark cupboard to crystallize since the product formed was photosensitive.

It was made sure that for the preparation of Potassium Trioxalatoferrate (III) Trihydrate, the temperature was maintained at 40°C to prevent hydrogen peroxide decomposition.

Observations:

Ferrous (II) oxalate had a yellow precipitate and at the end a yellow powder was obtained.

The endpoint of the redox titrimetric titration was marked by a faint pink colouration.

Ferric hydroxide had a brown precipitate which turned into a green solution upon excess oxalic acid was added.

Potassium Trioxalatoferrate (III) Trihydrate formed was in the form of green crystals.

3. Results and Calculations

Results:

Part A:

Ferrous ammonium sulphate weighed – 15. 042g

10% oxalic acid measured – 75mL

Mass of ferrous (II) oxalate obtained – 5. 586g

Part B:

Ferrous (II) oxalate used – 3. 269g

Potassium oxalate used – 5. 008g

Mass of Potassium Trioxalatoferrate (III) Trihydrate obtained – 2. 205g

Part C:

Ferrous (II) oxalate used – 0. 320g

Potassium Trioxalatoferrate (III) Trihydrate used – 0. 200g

Zinc used – 2g

Volume of permanganate required in the redox titration between iron (II) oxalate and permanganate – 49. 5mL

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Volume of permanganate required in the redox titration between iron (II) oxalate and permanganate in the presence of zinc – 15. 50mL

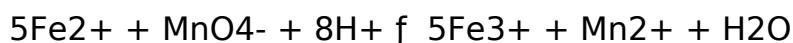
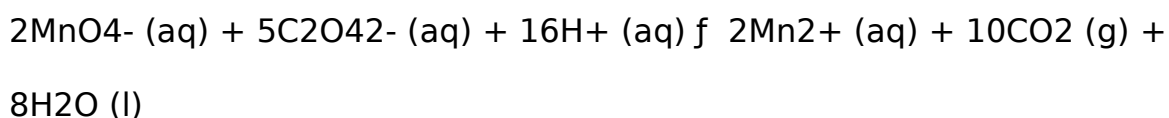
Volume of permanganate required in the redox titration between Potassium Trioxalatoferrate (III) Trihydrate and permanganate – 24. 50mL

Volume of permanganate required in the redox titration between Potassium Trioxalatoferrate (III) Trihydrate and permanganate in the presence of zinc – 4. 00mL

Calculations:

Analysis of products for Iron Oxalate for Iron(II) oxalate

The equations taking place in the reaction are:



Moles permanganate reacting with oxalate and iron = Concentration of permanganate x Volume of permanganate required:

$$\text{Moles permanganate} = 0.02 \times (49.50 / 1000)$$

$$\text{Moles permanganate} = 0.00099 \text{ moles}$$

Moles permanganate reacting with iron (II) = Concentration of permanganate x Volume of permanganate required:

$$\text{Moles permanganate} = 0.02 \times (15.5 / 1000)$$

Moles permanganate = 0.00031 moles

Therefore, moles of permanganate reacting with the oxalate ions = Total number of moles - Number of moles of permanganate reacting with iron.

0.00099 - 0.00031

= 0.00068 moles

From the stoichiometry of the equation it is observed that 2 moles of permanganate react with 5 moles of oxalate, thus:

Moles of oxalate = $5/2$ (0.00068) = 0.0017 moles

Grams of oxalate = number of moles x mass of oxalate

Grams of oxalate = 0.0017 x 88

Grams of oxalate = 0.150 grams

Therefore % oxalate in the product:

$(0.150 / 0.320) \times 100 = 46.9 \%$

From the stoichiometry of the equation it is observed that 1 mole of permanganate react with 5 moles of Iron, thus:

Moles of oxalate = 5 (0.00031) = 0.00155 moles

Grams of Iron (II) = number of moles x mass of oxalate

Grams of Iron (II) = 0.00155 x 56

Grams of Iron (II) = 0.087 grams

Therefore % Iron in the product:

$$(0.087 / 0.320) \times 100 = 27.19\%$$

The mass of water = Total mass of complex - (Mass of oxalate + iron (ii))

$$\text{Mass of water} = 0.320 - (0.150 + 0.087) = 0.083\text{g}$$

Therefore moles = grams / RMM

$$\text{Moles water} = 0.083 / 18$$

$$\text{Moles water} = 0.0046 \text{ moles}$$

Therefore % water in product:

$$(0.083 / 0.320) \times 100 = 25.9\%$$

To calculate the empirical formula:

Iron Oxalate Water

$$0.00155 : 0.0017 : 0.0046$$

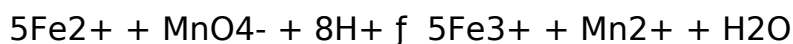
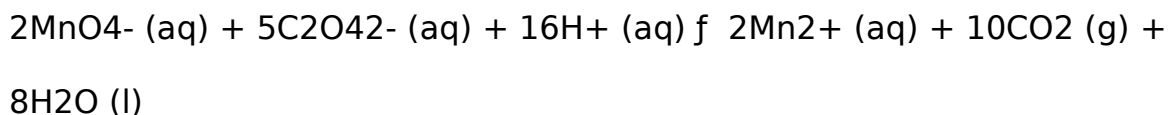
$$0.00155 : 0.00155 : 0.00155$$

$$1 : 1 : 3$$

Thus empirical formula is $\text{FeC}_2\text{O}_4 \cdot 3\text{H}_2\text{O}$

Analysis of products for Iron Oxalate for Potassium trioxalatoferrate (III) trihydrate.

The equation taking place in the reaction are:



Moles permanganate reacting with oxalate = Concentration of permanganate x Volume of permanganate required:

$$\text{Moles permanganate} = 0.02 \times (24.5 / 1000)$$

$$\text{Moles permanganate} = 0.00049 \text{ moles}$$

From the stoichiometry of the equation it is observed that 2 moles of permanganate react with 5 moles of oxalate, thus:

$$\text{Moles of oxalate} = 5/2 (0.00049) = 0.00123 \text{ moles}$$

$$\text{Grams of oxalate} = \text{number of moles} \times \text{mass of oxalate}$$

$$\text{Grams of oxalate} = 0.00123 \times 88$$

$$\text{Grams of oxalate} = 0.108 \text{ grams}$$

Therefore % oxalate in the product:

$$(0.108 / 0.200) \times 100 = 54 \%$$

Moles permanganate reacting with iron (III) = Concentration of permanganate x Volume of permanganate required:

$$\text{Moles permanganate} = 0.02 \times (4.00 / 1000)$$

$$\text{Moles permanganate} = 8 \times 10^{-5} \text{ moles}$$

From the stoichiometry of the equation it is observed that 1 mole of permanganate react with 5 moles of Iron, thus:

$$\text{Moles of oxalate} = 5 (8 \times 10^{-5}) = 0.0004 \text{ moles}$$

Grams of Iron = number of moles x mass of oxalate

$$\text{Grams of Iron} = 0.0004 \times 56$$

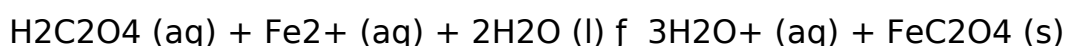
$$\text{Grams of Iron} = 0.0224 \text{ grams}$$

Therefore % Iron in the product:

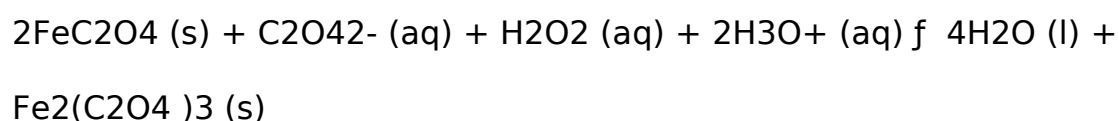
$$(0.0224 / 0.200) \times 100 = 11.20\%$$

Discussion:

In the first part of the experiment, ferrous ammonium sulphate, also known as Mohr's Salt was treated with warm water and sulphuric acid in order to prevent the formation of rust coloured iron hydroxides and oxides. This was followed by oxalic acid. The oxalate ions replace some or all of the sulphate ligands surrounding the Fe²⁺ ion and as a result, a yellow precipitate of ferrous oxalate forms. The reaction taking place is as follows:

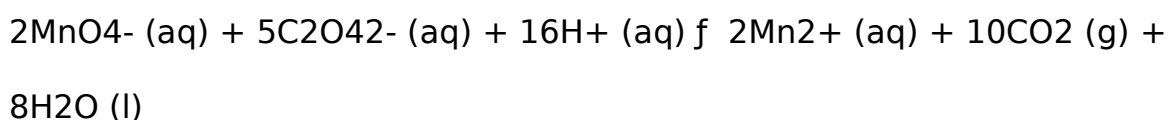


In order to oxidise the Fe^{2+} ion into an Fe^{3+} ion in ferrous oxalate, hydrogen peroxide, acting as an oxidising agent is added to a solution of ferrous oxalate and potassium oxalate. Temperature control is very crucial in this step due to the fact at high temperatures, hydrogen peroxide can decompose and thus would not be able to oxidise the iron (II) to iron (III) required to prepare the Potassium trioxalatoferrate (III) trihydrate complex. It is important to make sure that all the iron (ii) has been oxidised to iron (iii) due to the fact that since each complex consists of a different number of oxalate ligands, if a mixture of the two complex ions is present, the empirical formula determination would become difficult. The reaction taking place is as follows:

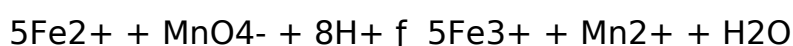


When the $\text{Fe}_2(\text{C}_2\text{O}_4)_3$ precipitate was dissolved, $[\text{Fe}(\text{C}_2\text{O}_4)_3]^{3-}$ forms. This reacts with the potassium ions in solution introduced via the potassium oxalate and forms potassium trioxalatoferrate (III) which is photosensitive and thus must be stored in the dark.

In the analysis of the oxalate ion, no indicator is required in the redox titration between permanganate and the oxalate ions due to the fact that at the end point, since potassium permanganate is an oxidising agent, it oxidises the oxalate ions in solution into carbon dioxide and as a result, permanganate is itself reduced to Mn^{2+} therefore a faint pink colour is observed at the endpoint. The reactions taking place are as follows:



In order to analyse the iron content in the complexes formed, zinc is added followed by heating the solution. Once this was complete, the solution was treated with permanganate in a redox titration similar to the one described previously above. The reaction taking place is as follows:



Conclusion:

This experiment has shown that iron being a transition metal can exist as various oxidation states. These oxidation states can then form a variety of complexes with various ligands. The complexes that are formed can then be analysed using a redox titration in order to determine the percentages of iron and oxalate in the complex.

In this experiment, the empirical formula of Iron (II) oxalate was found to be $\text{FeC}_2\text{O}_4 \cdot 3\text{H}_2\text{O}$ and consisted of 46.9% oxalate, 27.19% Iron (II) and 25.9% water whereas the Trioxalatoferrate (III) Trihydrate consisted of 54% oxalate and 11.20% iron (III)