

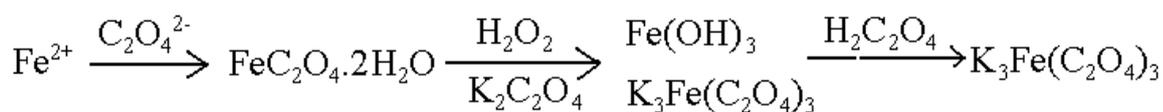
Preparation of an Iron Complex

Introduction

In this experiment you will prepare a complex compound of iron; potassium trioxalatoferrate(III), $K_3Fe(C_2O_4)_3$. In this compound each oxalate ligand has two bonds to the iron(III) ion, and hence this compound is an example of a chelate. You will aim at obtaining the maximum yield of product of the highest purity permitted by this method. Your final product should be in the form of bright green crystals, free from dust specks, filter paper fibres, etc.

Background

It is of interest to compare the properties of chelated iron(III) ion with that of the normal iron(III) ion. The compound will be prepared by first preparing iron(II) oxalate, $FeC_2O_4 \cdot 2H_2O$, and then oxidizing this with hydrogen peroxide, in the presence of potassium oxalate, to convert it to the ferric-oxalate ion $Fe(C_2O_4)_3^{3-}$



Thus all the iron in the iron(II) ammonium sulphate is converted to ferrioxalate, one third of the ferrioxalate being formed from the iron(III) hydroxide. Both H_2O_2 and $Fe(OH)_3$ are unstable to heat. Potassium trioxalato ferrate(III) is photosensitive, i.e. it decomposes when exposed to light, reforming iron(II) oxalate.

Chemicals: 2 M sodium hydroxide, ethanol, ethanedioate (oxalic) acid, 1.8 M hydrogen peroxide, iron(II) ammonium sulphate, potassium ethanedioate (oxalate)

Technician's note

1. sodium ethanedioate (oxalate) cannot be employed in this experiment.
2. Iron(II) ammonium sulphate should be fresh and green in appearance.
3. Molarity of hydrogen peroxide should be roughly determined by titration.

Procedure

1. Dissolve 7.5 g of iron(II) ammonium sulphate, $FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$ in 25 cm³ of warm water. Add to this warm solution, with stirring, 32.5 cm³ of an oxalic acid solution made by dissolving 10 g of the acid in 100 cm³ of water.
2. Cautiously heat the mixture to boiling, and then allow the yellow granular precipitate to settle.
3. Decant the supernatant liquid and wash the precipitate by stirring it with 50 cm³ of hot water. Allow the precipitate to settle and again decant the clear liquid. Repeat with a further 50 cm³ of hot water.

4. Suspend the washed precipitate in a warm solution of 5 g of potassium oxalate in 15 cm³ of water. Add slowly from a burette 12.5 cm³ of 1.8 M H₂O₂, stirring continuously and keeping the temperature at about 40 °C.

N.B.

If hydrogen peroxide is added too quickly, the heat liberated would decompose the peroxide and the iron(II) ion will not be completely oxidized to iron(III) compound. Your yield of product in the last step reflects how good your performance in this experiment.

5. Heat the mixture to boiling.
6. Add the boiling mixture, in one portion, 10 cm³ of a solution of oxalic acid containing 10 g of the acid in 100 cm³ of water. If the mixture has not turned to a clear green, add oxalic acid solution, drop by drop, until it becomes clear (up to 2.5 cm³ may be required.)
7. The solution should now be green due to the presence of the ferrioxalate ion. Filter the hot solution if necessary and add 15 cm³ of ethanol.
8. If any crystal appears, redissolve them by warming the solution. Set aside in the dark to crystallize. After crystallization, decant off the supernatant liquid, wash by decantation with a little water, and finally with ethanol.
9. Spread the crystals on a fresh filter paper and allow them to dry in the air. Weigh the dry crystal and record the result.

Questions for discussion

1. Iron(II) ammonium sulphate is used as the starting material rather than the use of iron(II) sulphate. Explain.
2. What is the name of the product in step (1) - (2)?
3. Explain the use of hot water rather than cold water in step 3. What is the purpose of carrying out step 3?
4. (a) Why is potassium oxalate solution used rather than water in step 4? (b) Why should the temperature of the mixture be kept at about 40 °C?
5. (a) In step 5, what do you observe? (b) Write an equation for the reaction in step 5.
6. Write an equation for the reaction in step 6. Why should excess oxalic acid be avoided in step 6?
7. What is the function of the use of ethanol in step 7? Why is it effective?
8. Why is it necessary to exclude light in step 8?
9. Why are the crystals finally washed with ethanol?

End