Experiment 6
Synthesis of an Iron(III)-EDTA Complex

Student Handout

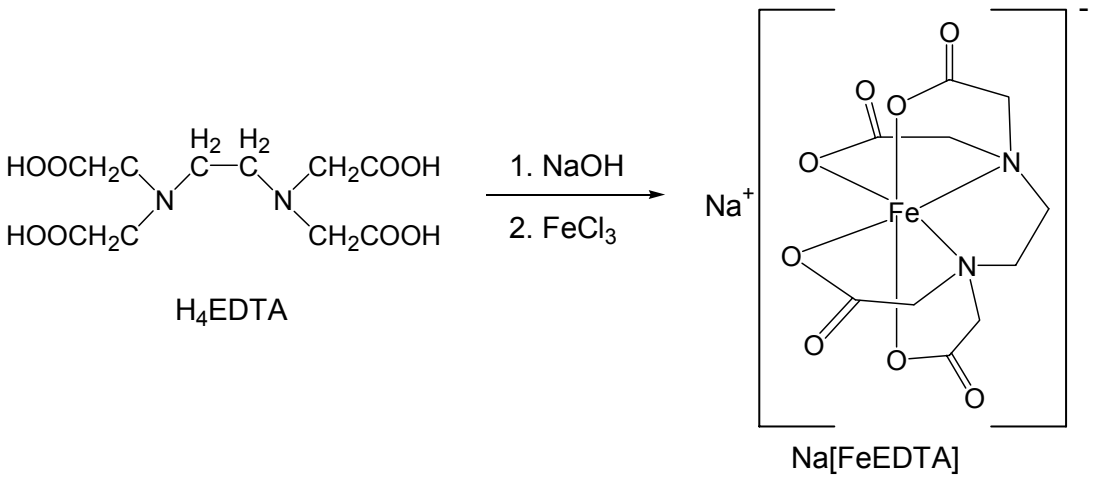
Purpose

To prepare the iron(III) ethylenediaminetetraacetalato complex, $Na[Fe(EDTA)]\cdot 3H_2O$.

Background

Ethylenediaminetetraacetic acid (H_4EDTA) is a very useful hexadentate ligand. It can react with various metal ions through its four oxygen and two nitrogen atoms to form chelates. It has been widely used in chelation therapy, a treatment which removes excessive metals such as lead and iron from our body. This compound can also be used as a food additive to prevent spoilage by removing the essential metal ions for bacterial growth.

In this investigation, you will synthesise an iron(III)-EDTA complex and perform simple chemical tests to compare the chemical properties of the complex with the free iron(III) ions.



H_4EDTA

Na[FeEDTA]

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Safety

Avoid direct contact of chemicals with skin. Dispose of chemical wastes, broken glassware and excess materials according to your teacher's instruction.

Ethanol is inflammable. Keep it away from ignition source.

Pay special attention when handling Bunsen burners.

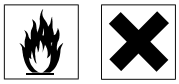
EYE PROTECTION MUST BE WORN

Materials and Apparatus Available

For synthetic work:

Ethylenediaminetetraacetic acid, disodium dihydrate ($Na_2H_2EDTA \cdot 2H_2O$) 

Iron(III) chloride hexahydrate 

Ethanol 

Sodium hydroxide (NaOH) 

Beaker

Deionised water
Bunsen burner
Filter vac
Suction flask

Büchner funnel
Filter paper
Spatula

For chemical tests:

0.1 M Iron(III) chloride ($FeCl_3$) solution 

0.1 M Sodium hydroxide (NaOH) solution 

0.1 M Sodium fluoride (NaF) solution
0.1 M Potassium thiocyanate (KSCN) solution
Dropper

Test tube rack
Test tube

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Experimental Procedure

Photos of the experiment are available at http://www.chem.cuhk.edu.hk/ssc.htm.

Make sure you record all the observations and data.

Part A: Synthesis of $Na[Fe(EDTA)]\cdot 3H_2O$

1. Dissolve 0.4 g (0.01 mol) of NaOH in 10 cm^3 of water, and then add 3.8 g (0.01 mol) of $Na_2H_2EDTA\cdot 2H_2O$.
2. Gently heat the solution until the solid dissolves to give a clear solution.
3. Dissolve 2.5 g (0.009 mol) of iron(III) chloride hexahydrate in 5 cm^3 of water, which is then added to the EDTA solution with swirling.
4. Gently boil off the water until most of the yellow powder precipitates out.
5. Cool down the solution and collect the precipitate by suction filtration.
6. Wash the product thoroughly with ice water until it is free of iron(III) ions.
7. Wash the product with ethanol twice and dry it with filter paper.
8. Weigh your product, calculate its theoretical and percentage yields.

Part B: Chemical tests

1. With heating, prepare 10 cm^3 of 0.1 M sodium iron(III) ethylenediaminetetraacetate solution.
2. Respectively dispense 10 drops of 0.1 M iron(III) chloride solution and 0.1 M sodium iron(III) ethylenediaminetetraacetate solution into test tubes.
3. Add 10 drops of 0.1 M sodium hydroxide solution to each of the test tubes and record the observations in a table.
4. Similarly, test both iron(III) chloride and sodium iron(III) ethylenediaminetetraacetate solutions with 0.1 M fluoride and thiocyanate solutions.

0.1 M solution tested	0.1 M Reagent	Observations
Fe^{3+}	NaOH	
$Fe(EDTA)^-$	NaOH	
Fe^{3+}	KSCN	
$Fe(EDTA)^-$	KSCN	
Fe^{3+}	NaF	
$Fe(EDTA)^-$	NaF	

5. Determine which ligand, the monodentate ligands or the hexadentate ligand $EDTA^{4-}$, binds more strongly to the iron(III) ions.

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Questions for Further Thought

1. Why should an excess base be avoided in Step 1 of Part A of the Procedure?
2. The stability constant for $Fe(EDTA)^{-}$ is 5×10^{25} dm^3 mol^{-1}. Calculate the concentration of Fe^{3+} ions when equal volumes of a 0.2 M $EDTA^{4-}$ solution and a 0.2 M Fe^{3+} solution are mixed together.
3. Which of the following ligands can form chelates: OH^{-}, CN^{-}, $H_2NCH_2CH_2NH_2$, $H_2NCH_2CH_2CH_3$, $(COOH)_2$ and CH_3COOH? Put an asterisk at the upper right corner of the coordinating atom(s) in the formula.
4. 30.00 cm^3 of a 0.2000 M EDTA solution is added to 20.00 cm^3 of an Fe^{3+} solution. The excess EDTA is then back-titrated with a 0.1000 M Pb^{2+} solution. 15.68 cm^3 of the Pb^{2+} solution is consumed. What is the concentration of the Fe^{3+} solution?

Reference

E. M. Cranton, *A Textbook on EDTA Chelation Therapy,* Human Sciences Press, New York, 1989, pp. 55 - 59.

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