

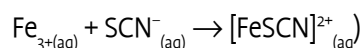
Determination of iron by thiocyanate colorimetry

Use this method if you do not have access to a colorimeter

Introduction

Iron is one of the many minerals required by the human body. It is used in the manufacture of the oxygen-carrying proteins haemoglobin and myoglobin. A deficiency of iron in the body can leave a person feeling tired and listless, and can lead to a disorder called anemia. Many of the foods we eat contain small quantities of iron.

In this analysis the iron present in an iron tablet (dietary supplement) or a sample of food is extracted to form a solution containing Fe^{3+} (ferric) ions. To make the presence of these ions in solution visible, thiocyanate ions (SCN^-) are added. These react with the Fe^{3+} ions to form a blood-red coloured complex:



By comparing the intensity of the colour of this solution with the colours of a series of standard solutions, with known Fe^{3+} concentrations, the concentration of iron in the tablet or food sample may be determined. This technique is called colorimetry.

Equipment and Materials Required

- ferric ammonium sulfate $\text{FeNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ standard solutions: 2, 4, 6, 8 and $10 \times 10^{-5} \text{ mol L}^{-1}$ (see below for preparation)
- iron tablet (dietary supplement) or sample of iron-containing food (eg: silverbeet, spinach, raisins, peanuts, walnuts, etc.)
- 1 mol L^{-1} ammonium thiocyanate solution (see below for preparation)
- 1 mol L^{-1} sulfuric acid
- 0.15 mol L^{-1} potassium permanganate solution (see below for preparation)
- 100 mL beaker
- 100, 200, 250 and 500 mL volumetric flasks
- 5 mL pipette
- 10 mL measuring cylinder
- 100 mL conical flask
- at least 6 boiling tubes (or large test tubes)

- distilled water

Method:

1. **Preparation of Fe^{3+} standard solutions:** NB: It may take several days to dissolve the Fe^{3+} salt used here, so carry out this preparation well in advance of the rest of the experiment. Weigh out about 3.0 g of ferric ammonium sulfate ($\text{FeNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$). Use a mortar and pestle to grind the salt to a fine powder. Accurately weigh 2.41 g of the powder into a 100 mL beaker and add 20 mL of concentrated sulfuric acid (Caution: concentrated acids are highly corrosive – wear safety glasses and rubber gloves and take care when handling). Leave powder to soak in acid overnight. The next day, carefully pour the acid/powder slurry into a 500 mL volumetric flask, rinsing the beaker into the flask a few times with water, then make up to the mark with distilled water. Let this solution stand for several days until the ferric ammonium sulfate powder has fully dissolved. If possible, insert a magnetic stirrer bar and stir the solution to speed up this dissolving process.

Use a pipette to transfer 20 mL of ferric ion solution to a 200 mL volumetric flask and make up to the mark with distilled water. This gives a solution with $[\text{Fe}^{3+}] = 0.001 \text{ mol L}^{-1}$. To prepare a $2 \times 10^{-5} \text{ mol L}^{-1}$ standard solution pipette 10 mL of the 0.001 mol L^{-1} solution into a 500 mL volumetric flask, add 10 mL of 1 mol L^{-1} sulphuric acid, and then make up to the mark with distilled water. Repeat this procedure in separate 500 mL volumetric flasks, pipetting in 20, 30, 40 and 50 mL of 0.001 mol L^{-1} Fe^{3+} solution in turn, to obtain 4, 6, 8 and $10 \times 10^{-5} \text{ mol L}^{-1}$ solutions respectively.

(NB: if you do not have five 500 mL volumetric flasks you can use one flask to prepare each standard in turn. After preparing each standard, pour the solution into a labelled glass vessel which has a lid (eg: a glass bottle). Then rinse your 500 mL volumetric flask thoroughly with distilled water before using it to prepare your next standard solution.)

2. **Preparation of 1 mol L^{-1} ammonium thiocyanate solution:** Weigh 38 g of solid ammonium thiocyanate into a 500 mL volumetric flask and make up to the mark with distilled water.

3. **Preparation of 0.15 mol L^{-1} potassium**

permanganate solution (only required for analysis of iron tablet): Weigh 2.4 g of solid potassium permanganate into a 100 mL volumetric flask and make up to the mark with distilled water.

Preparation of iron tablet for analysis:

1. Place iron tablet in a 100 mL beaker and use a measuring cylinder to add 20 mL of 1 mol L⁻¹ sulfuric acid. Allow the tablet's coating to break down and its contents to dissolve. You may help this process by using a stirring rod to carefully crush the tablet and stir the solution. (NB: iron tablets sometimes contain filler materials that may not fully dissolve in acid)
2. Once the iron tablet is dissolved, add 0.15 mol L⁻¹ potassium permanganate solution dropwise, swirling the beaker after each addition. Iron tablets usually contain ferrous sulfate, with iron present as Fe²⁺ ions. Since Fe²⁺ does not form a coloured complex with thiocyanate, permanganate ions are added to oxidise all the Fe²⁺ to form Fe³⁺ ions. For the first few drops of permanganate, the purple colour will disappear immediately upon addition to the iron solution; however, as further drops are added the colour will begin to linger for a little longer. Stop adding potassium permanganate drops when the purple colour persists for several seconds after addition – usually no more than about 2 mL of 0.15 mol L⁻¹ permanganate solution will be required.
3. Transfer the iron solution to a 250 mL volumetric flask, rinsing the beaker with distilled water a few times and transferring the washings to the volumetric flask. Make up to the mark with distilled water, stopper the flask and mix well.
4. Use a pipette to transfer 5 mL of iron solution to a 100 mL volumetric flask and make up to the mark with distilled water. This diluted solution will be used for colorimetric analysis.

Preparation of food sample for analysis:

1. Accurately weigh a few grams (typically 2 – 5 g is required, depending on iron content of sample) of your food sample into a crucible.
2. Heat the crucible over a bunsen burner (see Figure 1) until the sample is reduced completely to ash, or (preferably) combust the sample directly in the bunsen flame (as shown in Figure 2), reducing it to ash. NB: be very careful with the bunsen flame while heating/combusting your sample. Also beware that the crucible will become very hot during this process, so handle it only with crucible tongs – or preferably not at all – until

it has cooled.

3. When the sample and crucible have cooled, use a stirring rod to crush the ash to a fine powder (see Figure 3). Use a measuring cylinder to add 10 mL of 1 mol L⁻¹ hydrochloric acid and stir for 5 minutes, making sure that all the ash is soaked.
4. Add 5 mL of distilled water and filter the solution into a 100 mL conical flask to remove the ash. This filtered solution will be used for colorimetric analysis.

Colorimetric analysis:

NB: this analysis method applies to samples prepared using either of the two methods above (iron tablets or food samples).

1. Accurately measure 10 mL of your sample solution into a clean, dry boiling tube/test tube. NB: this is most accurately done using a 10 mL pipette; however, it is possible to do this accurately enough (and with less hassle) using a clean 10 mL measuring cylinder if you measure carefully.
2. Next, measure 10 mL of each Fe³⁺ standard solution into separate boiling tubes (one standard per tube) in order of increasing concentration, beginning with the 2 × 10⁻⁵ mol L⁻¹ standard. It is a good idea to first rinse your pipette or measuring cylinder with a few mLs of the 2 × 10⁻⁵ mol L⁻¹ standard). NB: Make sure you label each boiling tube appropriately with the name of the sample or standard it contain. A test tube rack is very useful for holding and transporting your tubes (see Figure 4). Alternatively you can use a large beaker to hold them.
3. Using a 10 mL measuring cylinder, measure 10 mL of 1 mol L⁻¹ ammonium thiocyanate solution into each of six small clean vessels – six boiling tubes is ideal. You should now have one measured portion of thiocyanate solution for each of your iron solutions.
4. As quickly as possible, pour 10 mL of thiocyanate solution (the portions measured out above) into each of your iron solutions.
5. Mix the solutions by swirling. A stable red colour will appear over the next few minutes.
6. Allow the red colour to develop for 15 minutes. Then estimate the concentration of Fe³⁺ ions in your iron sample by identifying which of your Fe³⁺ standards matches its colour the closest. Figure 4 illustrates the range of colour intensities that you can expect from your set of Fe³⁺ standards. Tip: If you are using boiling/test tubes all of identical sizes, the best way to compare colours is by looking at your solutions from above – looking down into the tubes (see Figure 5).

7. If the colour of your unknown iron solution is stronger than the colour of your highest concentration Fe^{3+} standard you will need to modify the above procedure. In the case of an iron tablet, you should repeat the analysis with a more dilute solution of the dissolved iron tablet. In the case of a food sample, you should repeat the analysis using a smaller mass of your food.

Calculations:

1. Assume that the concentration of Fe^{3+} in your unknown iron solution is approximately equal to that of the Fe^{3+} standard whose colour was the closest match.
2. Use this concentration to calculate the mass of iron (in mg) in your original tablet or food sample (NB: the molecular weight of iron is 55.8 g mol^{-1}). Remember to take into account any dilutions that you performed while preparing your sample solution.

Additional Notes:

1. If you are using a colorimeter instrument, it is not critical to have identical boiling/test tubes for each iron solution – any small vessels (eg: small beakers or glass vials) will do. However, if you are analysing the colour intensities by eye, as above, it is important to have identical vessels in order to make an accurate comparison – a set of identical boiling/test tubes is ideal.
2. You may notice that even the standard giving the closest colour match to your sample is still quite different – meaning that your estimated Fe^{3+} concentration is not very accurate. In order to obtain a more accurate result you may wish to try the following procedure:
 - Identify which two Fe^{3+} standards are the closest colour matches to your unknown solution: one must be slightly darker in colour and the other slightly lighter than your unknown. The concentration of your unknown solution must lie somewhere between the concentrations of these two standards. For this example, let us pretend that the two closest standards are 4×10^{-5} and $6 \times 10^{-5} \text{ mol L}^{-1}$.
 - Take 5 clean, dry boiling tubes. To the first tube we add 10 mL of the $4 \times 10^{-5} \text{ mol L}^{-1}$ standard and 10 mL of the $6 \times 10^{-5} \text{ mol L}^{-1}$ standard. We now have a new standard that is $5 \times 10^{-5} \text{ mol L}^{-1}$.
 - Next, we measure 5 mL of this $5 \times 10^{-5} \text{ mol L}^{-1}$ standard into a new tube and also add 5 mL of the $4 \times 10^{-5} \text{ mol L}^{-1}$ standard. We now have another new standard that is $4.5 \times 10^{-5} \text{ mol L}^{-1}$.
 - If we measure 5 mL of the $5 \times 10^{-5} \text{ mol L}^{-1}$ standard



Figure 1. Set up used for heating a silverbeet sample in a crucible.



Figure 2. Set up used for combusting a silverbeet sample directly in the bunsen burner flame.

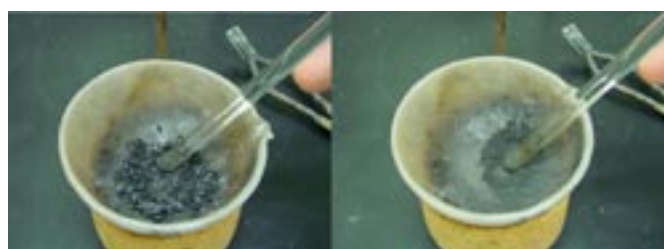


Figure 3. Left photo: ash remaining after combustion of a silverbeet sample. Right photo: silverbeet ash which has been ground to a fine powder using a stirring rod.

into another new tube and add 5 mL of $6 \times 10^{-5} \text{ mol L}^{-1}$, we will make another new standard that is $5.5 \times 10^{-5} \text{ mol L}^{-1}$.

- We finish by measuring 10 mL of the $4 \times 10^{-5} \text{ mol L}^{-1}$ standard into one boiling tube, and 10 mL of the $6 \times 10^{-5} \text{ mol L}^{-1}$ standard into a different tube.
- We now have a set of five boiling tubes, each containing exactly 10 mL of a different Fe^{3+} standard solution, with concentrations of 4, 4.5, 5, 5.5 and $6 \times 10^{-5} \text{ mol L}^{-1}$. If we now repeat the colorimetric analysis of our unknown iron solution using these new standards instead of the original ones, we should obtain a much more accurate value for Fe^{3+} concentration.

Contact Us

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Figure 4. Blood-red coloured solutions produced by the ferric thiocyanate complex ion ($\text{Fe}[\text{SCN}]_2^+$). These solutions were prepared using the following range of Fe^{3+} standards: 2, 4, 6, 8, 10, $12 \times 10^{-5} \text{ mol L}^{-1}$.



Figure 5. The same boiling tubes as in Figure 4 (prepared using Fe^{3+} standard solutions with concentrations of $2 - 12 \times 10^{-5} \text{ mol L}^{-1}$) viewed from above, looking directly down the length of the tube. This view provides the most accurate “eyeball” comparison of the solutions’ colour intensities.