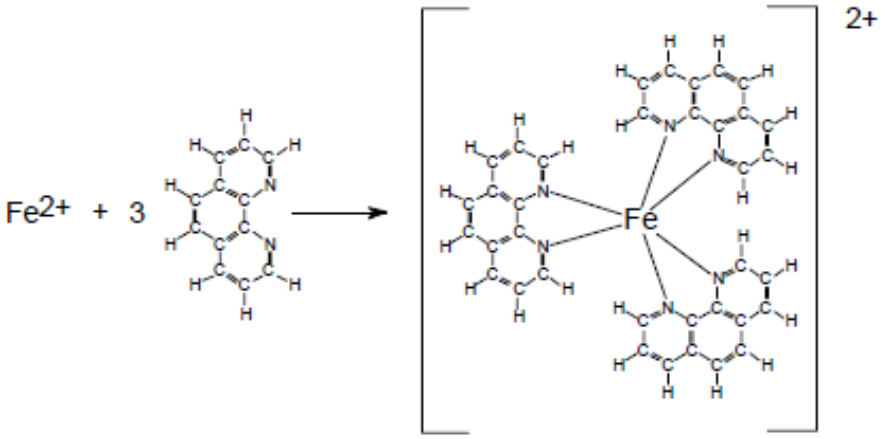
# Iron by 1,10-phenanthroline assay

## Student worksheet

### Principle

Iron(II) ions form a deep red solution with 1,10-phenanthroline. Generally, it is suitable for solutions that are 5 mg $dm^{-3}$, or less, $Fe^{2+}(aq)$. The colour is due to the complex that forms:




You can use this reaction for the quantitative analysis of low concentrations of $Fe^{2+}(aq)$ in solution. You can find the concentration of the solution of $Fe^{2+}$ using a colorimeter. You can also use simple colour matching although the results will be less precise. This method is suitable for solutions that contain 5 mg $dm^{-3}$, or less, $Fe^{2+}(aq)$.

If iron is present in the +3 oxidation state, it must be reduced to the +2 oxidation state before complex formation. This can be done by reaction with hydroxylamine hydrochloride:

$$2Fe^{3+}(aq) + 2NH_2OH(aq) + 2OH^-(aq) \rightarrow 2Fe^{2+}(aq) + N_2(g) + 4H_2O(l)$$

### Equipment and materials

- burettes x 3
- 1 $cm^3$ pipette
- 2 $cm^3$ pipette
- 100 $cm^3$ volumetric flask x 7 (or re-use one flask)
- colorimeter and suitable filter (green) – a solution of the complex displays maximum absorption at 510 nm
- iron(II) ammonium sulfate solution containing 0.100 g $dm^{-3}$ $Fe^{2+}$ (100 ppm), (15 $cm^3$)
- hydroxylamine hydrochloride solution (if used), 1.5 mol $dm^{-3}$ (7 $cm^3$)
- 1,10-phenanthroline solution, 5 x $10^{-3}$ mol $dm^{-3}$ (105 $cm^3$)
- sodium ethanoate solution, 1 mol $dm^{-3}$ (14 $cm^3$)
- solution of unknown $Fe^{2+}$ concentration,(15 $cm^3$)

### Method

**Care:** Wear eye protection. Hydroxylamine hydrochloride solution is harmful.

1. Fill two burettes, one with the standard iron(II) solution and one with deionised water.
2. Label six 50 or 100 $cm^3$ beakers A to F and use the burettes to add the solutions shown in the table:

| Volumetric flask | A | B | C | D | E | F |
|---|---|---|---|---|---|---|
| Volume of standard iron(II) solution / $cm^3$ | 5.0 | 4.0 | 3.0 | 2.0 | 1.0 | 0.0 |

3. To each volumetric flask, use a 1 or 2 $cm^3$ pipette to add 1.0 $cm^3$ of 1.5 mol $dm^{-3}$ hydroxylamine hydrochloride solution (if used) and 2.0 $cm^3$ of 1 mol $dm^{-3}$ sodium ethanoate solution. From a burette add 15.0 $cm^3$ of 5 x $10^{-3}$ mol $dm^{-3}$ 1,10-phenanthroline solution. Make up the volume to 100 $cm^3$ using distilled or deionised water.

| Beaker | A | B | C | D | E | F |
|---|---|---|---|---|---|---|
| ppm iron | 5 | 4 | 3 | 2 | 1 | 0 |

4. Mix each solution well and leave to stand for 10 minutes.
5. Measure the absorbance of each solution. If the colour of the solutions is too intense for the colorimeter to measure, dilute the solution by adding more water to the beakers from the burette and measure the absorbance of the diluted solutions.
6. Plot a graph of absorbance (*y* axis) against $Fe^{2+}(aq)$ concentration (in ppm iron) (*x* axis) for beakers A-F.
7. Using a burette, add 10 $cm^3$ of the iron(II) solution of unknown $Fe^{2+}$ concentration to a 100 $cm^3$ volumetric flask. Use a 1 or 2 $cm^3$ pipette to add 1.0 $cm^3$ of 1.5 mol $dm^{-3}$ hydroxylamine hydrochloride solution (if used) and 2.0 $cm^3$ of 1 mol $dm^{-3}$ sodium ethanoate solution. From a burette add 15.0 $cm^3$ of 5 x $10^{-3}$ mol $dm^{-3}$ 1,10-phenanthroline solution. Make up the volume to 100 $cm^3$ using distilled or deionised water.
8. Measure the absorbance of the solution made up using of the iron(II) solution whose concentration is unknown.
9. Use the graph to find the concentration of $Fe^{2+}(aq)$ as ppm iron in the unknown. Remember that the iron(II) solution has been diluted ten-fold in the volumetric flask.