Colorimetric analysis of aspirin

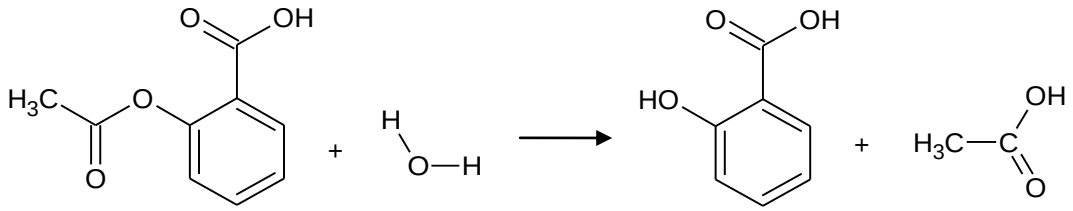
Student worksheet

Health and safety note:

Wear appropriate eye protection (splash-proof goggles). 1 mol dm^{-3} sodium hydroxide solution is corrosive. Iron(III) chloride solutions are an irritant at concentrations greater than 0.2 mol dm^{-3}.

Principle

Aspirin hydrolyses to produce 2-hydroxybenzoic acid and ethanoic acid (shown below).



2-hydroxybenzoic acid produces a violet-blue complex when mixed with iron(III) ions. The amount of 2-hydroxybenzoic acid in a solution made by hydrolysing aspirin can be determined by adding iron(III) ions and measuring the intensity of the violet-blue solution. From this the amount of aspirin can be calculated.

Equipment and materials

- Colorimeter and suitable filter (green/yellow). A solution of the complex displays maximum absorption at about 530 nm
- Cuvette
- 100 cm^3 conical flask
- Bunsen burner, tripod, gauze and heat-proof mat (or an electric hotplate)
- Aspirin − Harmful
- 500 cm^3 volumetric flask (x2)
- 50 cm^3 burette
- 100 cm^3 volumetric flask (x5)
- 1 cm^3 pipette + pipette filler
- 1 mol dm^{-3} sodium hydroxide solution − Corrosive
- 0.02 mol dm^{-3} iron(III) chloride solution

Note: the volumetric flasks need to be washed thoroughly before being used to make a new solution, as does the pipette which should also be rinsed 2-3 times with the new solution before measuring out the required volume.

Method: Preparing standard solutions and obtaining a calibration graph

1. Weigh accurately about 0.4 g of aspirin into a 100 cm^3 conical flask, add 10 cm^3 of 1.0 mol dm^{-3} sodium hydroxide solution and warm the mixture gently at 50 °C for ten minutes.
2. Cool the solution and transfer quantitatively to a 500 cm^3 volumetric flask. Make up to the mark with deionised water. This is the stock solution. It contains the hydrolysis product (sodium 2-hydroxybenzoate) from a 0.80 g dm^{-3} solution of aspirin.

3. Use a burette to measure 10 cm^3 of stock solution into a 100 cm^3 volumetric flask. Make it up to volume with 0.02 mol dm^{-3} iron(III) chloride solution. This is standard solution A. In a similar way make up standard solutions B to G using 8 cm^3, 6 cm^3, 4 cm^3, 2 cm^3, 1 cm^3 and 0.5 cm^3 of stock solution.

Standard solution	Volume of stock solution /cm^3	Concentration equivalent of aspirin /g dm^{-3}
A	10	0.080
B	8	0.064
C	6	0.048
D	4	0.032
E	2	0.016
F	1	0.008
G	0.5	0.004

4. Measure the absorbance of each standard solution using the colorimeter, fitted with a suitable filter.
5. Plot a graph of absorbance against concentration equivalent of aspirin. This is the calibration graph.

Method: Analysing aspirin tablets

1. Make a note of the mass of aspirin the manufacturer states is in each tablet. This is given on the packaging.
2. Weigh accurately one tablet and put it into a 100 cm^3 conical flask. Carefully crush the tablet with a stirring rod and add 10 cm^3 of 1 mol dm^{-3} sodium hydroxide solution and warm at 50 °C gently for ten minutes.
3. Cool the solution and transfer quantitatively to a 500 cm^3 volumetric flask, if necessary filtering to remove any insoluble material. Make up to the mark with deionised water.
4. Pipette 1 cm^3 of into a 10 cm^3 volumetric flask. Make it up to volume with 0.02 mol dm^{-3} iron(III) chloride solution. Measure the absorbance of the unknown using the colorimeter, fitted with a suitable filter.
5. Use the calibration graph to determine the concentration of aspirin in the solution and use this to work out the mass of aspirin in the tablet.