THE ROYAL SOCIETY OF CHEMISTRY

54. The analysis of aspirin tablets

Topic

Organic chemistry/chemical analysis.

Timing

20 min.

Description

In this experiment students measure the amount of free 2-hydroxybenzoic acid (salicylic acid) in 2-ethanoyloxybenzenecarboxylic acid (aspirin) tablets. 2-hydroxybenzoic acid (salicylic acid), being a substituted phenol, reacts with Fe^{3+} ions to produce a purple colour. The colour is matched against that produced by a set of standard solutions of 2-hydroxybenzoic acid (salicylic acid) in a well-plate.

Apparatus (per group)

- One 24-well plate
- One 100 cm^3 beaker
- Cotton wool
- One plastic pipette (standard form, *eg* Aldrich ref: Z13, 500-3)
- Two plastic pipettes (fine tip, *eg* Aldrich ref: Z13, 503-8).
- Sheet for microscale filtration technique.

Chemicals (per group)

- Various 2-ethanoyloxybenzenecarboxylic acid (aspirin) tablets

Solutions contained in plastic pipettes (fine-tip), see p. 2

- Iron(III) nitrate solution
- 2-Hydroxybenzoic acid (salicylic acid) (working) solution
- Deionised water.

1. Stock 2-hydroxybenzoic acid (salicylic acid) solution (0.1% w/v)

Dissolve 0.100 g of 2-hydroxybenzoic acid (salicylic acid) in *ca* 20 cm^3 of a 1:1 mixture of ethanol and deionised water in a 100 cm^3 beaker. Make up to 100 cm^3 in a volumetric flask.

2. Working 2-hydroxybenzoic acid (salicylic acid) solution (0.0025 g 2-hydroxybenzoic acid (salicylic acid) /25 cm^3)

Dilute 2.5 cm^3 of the stock solution to 25 cm^3 in a volumetric flask with a 1:1 ethanol/water mixture.

3. Iron(III) nitrate solution, 0.1 mol dm^{-3}.

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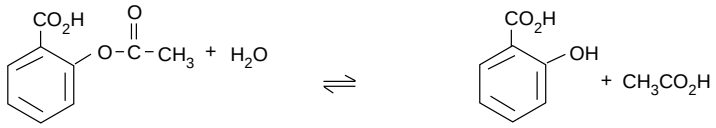
Observations

The set of standard solutions should give a range of intensities of a bluish colour. Students should be careful to add the correct number of drops as indicated. The experiment works best with old tablets containing some free 2-hydroxybenzoic acid (salicylic acid). New tablets with minimal free acid do not give any blue coloration but merely the colour of iron(III) in solution (yellow) so they do not fit into the range of standards.

The equation by which 2-hydroxybenzoic acid (salicylic acid) is formed is:



2-Ethanoyloxybenzenecarboxylic acid (Aspirin)

2-Hydroxybenzoic acid (Salicylic acid)

This experiment gives students an opportunity to consider the practical effect of equilibrium. Old 2-ethanoyloxybenzenecarboxylic acid (aspirin) tablets, which may have become damp with time, will contain more free 2-hydroxybenzoic acid (salicylic acid) because the presence of water causes the position of equilibrium to be shifted to the right in the above equation.

Reference

This experiment is based on a similar procedure given in the publication G. Rayner-Canham and A. Slater, *Microscale chemistry – laboratory manual*. Don Mills, Ontario: Addison-Wesley, 1994.

Safety

Students must wear eye protection.

It is the responsibility of the teacher to carry out a risk assessment.

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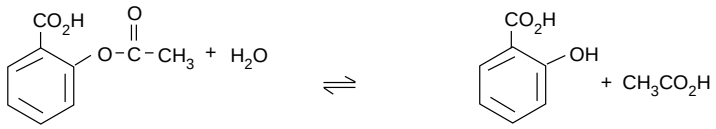
In this experiment you will be finding out how much 2-hydroxybenzoic acid (salicylic acid) is present in 2-ethanoyloxybenzenecarboxylic acid (aspirin) tablets.

2-Hydroxybenzoic acid (salicylic acid) is formed in the following reaction:



2-Ethanoyloxybenzenecarboxylic acid (Aspirin)

2-Hydroxybenzoic acid (Salicylic acid)

Instructions

Part A The preparation of standard solutions

In this part of the experiment you will be preparing a set of standard solutions with different colour intensities from the standard 2-hydroxybenzoic acid (salicylic acid) solution. You will be using these to match the intensity of the colour produced from the 2-ethanoyloxybenzenecarboxylic acid (aspirin) solution and so find out how much 2-hydroxybenzoic acid (salicyclic acid) there is in your 2-ethanoyloxybenzenecarboxylic acid (aspirin) tablet.

Taking your 24-well plate, add drops of solutions as indicated below:

Well no	A1	A2	A3	A4	A5	A6
No. of drops of: 2-ethanoyloxybenzene-carboxylic acid (salicyclic acid) soln.	5	15	25	35	45	50
Water	45	35	25	15	5	0
Iron(III) nitrate solution	5	5	5	5	5	5
Resulting mass (mg) of 2-hydroxybenzoic acid (salicyclic acid) per 25 cm^3 solution	0.25	0.75	1.25	1.75	2.25	2.5

Part B The analysis of 2-ethanoyloxybenzenecarboxylic acid (aspirin) tablets

1. Record the mass of a 2-ethanoyloxybenzenecarboxylic acid (aspirin) tablet and place it in a 100 cm^3 beaker.

2. Add 10 cm^3 of the 50% ethanol–water mixture (from a measuring cylinder) and swirl the mixture. The tablet will begin to disintegrate.

3. Using the microscale filtration method (p. 5), filter the mixture into a 25 cm^3 volumetric flask. Wash the beaker with a small quantity of the ethanol–water mixture and add to the flask. Make up to the mark, stopper and mix.

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4. Add 50 drops of this 2-ethanoyloxybenzenecarboxylic acid (aspirin) solution to well B3 followed by five drops of the iron(III) nitrate solution.

5. Match the colour to that of one of the standard solutions.

Calculations

Calculate the percentage of 2-hydroxybenzoic acid (salicylic acid) in the 2-ethanoyloxybenzenecarboxylic acid (aspirin) tablet as follows.

1. Identify the standard well that matches the colour intensity of the 2-ethanoyloxybenzenecarboxylic acid (aspirin) sample well.

2. The mass of 2-hydroxybenzoic acid (salicylic acid) (in 25 cm^3) in the solution from this standard well is therefore the same as the mass of 2-hydroxybenzoic acid (salicylic acid) in the 25 cm^3 of solution of your 2-ethanoyloxybenzenecarboxylic acid (aspirin) tablet solution.

3. Divide this mass (mg) by the mass of your 2-ethanoyloxybenzenecarboxylic acid (aspirin) tablet (mg) and multiply this value by 100 to give a percentage by mass.

Question

1. By considering the equation for the formation of 2-hydroxybenzoic acid (salicylic acid) from 2-ethanoyloxybenzenecarboxylic acid (aspirin), are there any differences in how much 2-hydroxybenzoic acid (salicylic acid) is present in both old and new bottles of 2-ethanoyloxybenzenecarboxylic acid (aspirin) tablets?