CIE Chemistry A-Level

Practicals for Papers 3 and 5

Rate of Reaction

Disappearing cross: Change in rate of the reaction of sodium thiosulphate with hydrochloric acid as temperature is changed:

$Na_2S_2O_3 + 2HCl \rightarrow 2NaCl + SO_2 + S + H_2O$

Method	Accuracy	Explanation
1. Add about 10 cm of 1 mol dm^{-3} hydrochloric acid to the 'acid' tube. Place this tube into a plastic container (without the cross under it).	• Hold the glass tubes and **vertically** in the plastic container.	
2. Use a measuring cylinder to add 10.0 cm of 0.05 mol dm^{-3} sodium thiosulfate solution to the second tube. Place this tube into the plastic container with the **cross under it** and carefully place a **thermometer in this tube**.		
3. Record the **start** temperature and then add 1 cm of the acid to the thiosulfate solution and start timing.		
4. Look down through the tube from above and record the time for the cross to **disappear from view**.		
5. Record the **final** temperature of the reaction mixture, and then pour the cloudy contents of the tube into the sodium carbonate solution.	• The temperature at which each experiment is carried out must be known as accurately as possible. This is done by measuring the initial and the final temperature to find a **mean** temperature.	This acts as the 'stop bath'.
6. Now add water from a very hot water tap (or kettle) to the plastic container. The water should be no hotter than 55 °C. Add cold water if necessary.		
7. Measure another 10.0 cm of 0.05 mol dm^{-3} **sodium thiosulfate** solution into a clean tube. Insert this tube into the correct hole in the plastic container (i.e. the one with the cross under it).		

8. Leave the tube to warm up for about 3 minutes.		
9. Repeat steps (3) to (6) in order to obtain results for at least **5 different temperatures** in total.		

Safety:

- To minimise the escape of **toxic** sulfur dioxide during the experiment a lid is advised. Two holes should be made in the lid using a hot wide cork borer. These holes should securely hold the glass tubes and vertically in the plastic container. Could also perform the experiment in a **fume cupboard**.
- Wear **eye protection**, a lab coat and **gloves** as HCl is an irritant.
- Ensure that the investigation is carried out in a **well-ventilated room** and that appropriate measures are taken to dispose of waste solutions.

Stop baths:

- Containers of sodium carbonate solution and phenolphthalein (**stop baths**) should be available to students so that the acid and sulfur dioxide can be **neutralised** at any point during the experiment.
- Once the colour of the solution in the stop bath changes, the sodium carbonate has been used up and the stop bath will need to be replenished.
- The stop bath should be placed in a fume cupboard, if available.

Analysing the data:

- In these experiments at different temperatures, the **concentrations** of all the reactants are the **same**.
- The **time taken** to produce the **same amount of sulfur** at **different temperatures** is an indication of **rate of the reaction**.
- A graph of the amount of sulfur produced against time can be plotted.
- The initial rate of reaction = (amount of sulfur)/time so the initial rate of reaction is **proportional** to 1/time.
- This is an approximation for rate of reaction as it does **not** include concentration. This can be used because it is assumed that the amount of sulphur produced is **fixed** and constant.

Initial rate method: 'Iodine Clock' experiment

- Hydrogen peroxide reacts with iodide ions to form iodine and the thiosulfate ion **immediately** reacts with iodine:
  $H_2O_2\,(aq) + 2H^+\,(aq) + 2I^-(aq) \rightarrow I_2\,(aq) + 2H_2O\,(l)$
  $2S_2O_3^{2-}\,(aq) + I_2\,(aq) \rightarrow 2I^-\,(aq) + S_4O_6^{2-}\,(aq)$
- **$S_2O_3^{2-}$** ions are used to **remove iodine** as it forms.

Initial rate method: 'Iodine Clock' experiment …

Method	Accuracy	Explanation
1. Fill the 50 cm^3 burette with potassium iodide solution.	• **Rinse** a 50 cm^3 burette with potassium iodide before	
2. Transfer 10.0 cm^3 of hydrogen peroxide solution from a burette to a 100 cm^3 beaker	• Beaker should be clean and dry	
3. Use a 50 cm^3 measuring cylinder to add 25 cm^3 of sulfuric acid to a 250 cm^3 beaker.	• Beaker should be **clean and dry**	
4. Use a 25 cm^3 measuring cylinder to add 20 cm^3 of distilled (deionised) water into the 250 cm^3 beaker.		
5. Use a plastic dropping pipette to add about 1 cm^3 of starch solution to this beaker.		
6. Use the burette to add 5.0 cm^3 of potassium iodide solution to the mixture in the 250 cm^3 beaker.		
7. Finally, add 5.0 cm^3 of sodium thiosulfate solution from a burette to the mixture in the 250 cm^3 beaker.		
8. **Stir** the mixture in the 250 cm^3 beaker. Pour the hydrogen peroxide solution from the 100 cm^3 beaker into the 250 cm^3 beaker and **immediately start the timer**.	• Stir the mixture	
9. **Stop** the timer when the mixture in the 250 cm^3 beaker turns blue-black. Record the time.		
10. Rinse the 250 cm^3 beaker with distilled (deionised) water and dry it with a paper towel.		
11. **Repeat** steps in four further experiments changing the concentration of potassium iodide.		This will allow the order of reaction to be determined.
12. Plot a **graph of initial rate (y) versus concentration (x)** to determine the **order**.		

Improvements:

● Use a **colorimeter** to **minimise human error** in timing.

Continuous monitoring method:

Method	Accuracy	Explanation
1. Add 50 cm^3 of 0.8 mol dm^{-3} hydrochloric acid to a conical flask.		
2. Set up the gas syringe or alternative **gas collection equipment**.		
3. Add a 6 cm strip of magnesium ribbon to the conical flask, place the **bung** firmly into the top of the flask and **start the timer**.	Swirl the flask every few seconds.	
4. **Record the volume of hydrogen** gas collected every 15 seconds for 2.5 minutes.		
5. **Alter the concentration of HCl** and repeat steps (1) to (4).		

Experiment considerations:

- A typical gas syringe only measures 100 cm^3 of gas so you don't want a reaction to produce more than this volume. **Quantities of reactants** need to be calculated carefully.
- Measuring **initial rate is preferential** as the concentrations are known at the start of the reaction.
- In reactions where there are several reactants, if the concentration of **one of the reactant** is kept in a large **excess** then that reactant will appear not to affect rate and will be essentially zero order. This is because its concentration stays virtually constant and does not affect rate.

Analysis:

- **Plot a graph** of volume of hydrogen produced on the y-axis against time in seconds for each hydrochloric acid concentration. Draw a line of best fit.
- Draw a tangent to each line of best fit at time, t = 0 s.
- Calculate the **gradient of each tangent** in order to deduce the initial rate of each reaction at each concentration.
- Compare the rate values obtained.

Other ways of following the reaction:

- Colorimeter: If any of the reagents or products are coloured (normally iodine), the reaction can be followed by **measuring time vs absorbance**. The absorbance is proportional to the concentration of iodine (a calibration graph is required to calculate the exact concentration of iodine).

- **Quenching**: Aliquots of a reaction mixture can be sampled at different times (without disturbing the reaction). The aliquots are quenched to stop the reaction by either: cooling, diluting, or neutralising an acid/base catalyst. The aliquots can then be titrated against to workout concentrations of reagent present.
- Measuring **mass** lost
- Measuring **pH**