# Core practical 13b: Use a clock reaction to determine a rate equation

## Objectives

- To use a clock reaction to find the order of reaction with respect to iodide ions
- To use a clock reaction to find the order of reaction with respect to peroxodisulfate ions

## Safety

- Wear a lab coat and goggles.
- Tie long hair back.

## Specification links

- Practical techniques 1, 4, 11, 12
- CPAC 1a, 2a, 2b, 3a, 4a, 4b

## Procedure

1. Measure 10.0 $cm^3$ of potassium iodide solution into a small beaker standing on a white tile.
2. Add 5.0 $cm^3$ of sodium thiosulfate solution to the potassium iodide solution.
3. Add 10 drops of starch solution to the mixture in the small beaker. Starch acts as the indicator and must be used in each experiment.
4. Measure out 10.0 $cm^3$ of the sodium peroxodisulfate solution. Pour this into the mixture prepared in steps 1 and 2. Start the stop clock.
5. Stop the clock when a blue colour appears in the beaker and note the time taken.
6. Make a copy of Table 2 from the 'Analysis of results' section. Use this to record your results.
7. Repeat steps 1–5 using the volumes of sodium peroxodisulfate and potassium iodide solutions shown in Table 1. The total volume including the sodium thiosulfate solution must add up to 25.0 $cm^3$, which can be achieved by adding the correct volume of distilled/deionised water.

**Table 1**

| Mixture | Vol. $S_2O_8^{2-}$/$cm^3$ | Vol. $I^-$/$cm^3$ | Vol. $S_2O_3^{2-}$/$cm^3$ | Vol. $H_2O$/$cm^3$ |
|---|---|---|---|---|
| (a) | 10.0 | 10.0 | 5.0 | 0.0 |
| (b) | 10.0 | 8.0 | 5.0 | 2.0 |
| (c) | 10.0 | 6.0 | 5.0 | 4.0 |
| (d) | 10.0 | 4.0 | 5.0 | 6.0 |
| (e) | 10.0 | 2.0 | 5.0 | 8.0 |
| (f) | 8.0 | 10.0 | 5.0 | 2.0 |
| (g) | 6.0 | 10.0 | 5.0 | 4.0 |
| (h) | 4.0 | 10.0 | 5.0 | 6.0 |
| (i) | 2.0 | 10.0 | 5.0 | 8.0 |

## Notes on procedure

- This experiment is very sensitive to changes in temperature and catalysis. Ensure that all glass equipment is clean and allow solutions to reach room temperature before beginning the practical (particularly the starch solution).
- It is recommended that each group of students completes all nine experiments. They do not take long to carry out when students have got themselves organised. If you are short of time you could split the group so that some students change only the concentration.
- If you have sufficient burettes, students could use them instead of measuring cylinders to measure out solutions.

## Answers to questions

1. One procedural error is misjudging the appearance of the blue colour in the solution.

   Another arises from the addition of the starch, which increases the total volume of the mixture slightly.

   Measurement uncertainties can occur in measuring volumes of solutions.

   For a 2 $cm^3$ volume, the uncertainty is: ± 0.1 $cm^3$

   % uncertainty = $\frac{0.2}{2} \times 100 = 10\%$

   For a 10 $cm^3$ volume, the uncertainty is ± 0.1 $cm^3$

   % uncertainty = $\frac{0.2}{10} \times 100 = 2\%$

2. The procedural errors are difficult to overcome; one change is to have two students timing simultaneously and using the average value.

   Measurement uncertainties can be minimised by using a graduated pipette or a burette.

3. Rate = $k[S_2O_8^{2-}][I^-]$

4. Step 1 is the rate-determining step. The rate of the reaction is second order and involves one peroxodisulfate ion and one iodide ion.

## Sample data

| Mixture | Conc. $S_2O_8^{2-}$ /mol dm$^{-3}$ | Conc. $I^-$/mol dm$^{-3}$ | Time/s | $\frac{1}{t}$ ($\propto$ rate)/s$^{-1}$ |
|---|---|---|---|---|
| (a) | 0.08 | 0.08 | 10 | 10.0 |
| (b) | 0.08 | 0.064 | 13 | 7.6 |
| (c) | 0.08 | 0.048 | 17 | 5.9 |
| (d) | 0.08 | 0.032 | 25 | 4.0 |
| (e) | 0.08 | 0.016 | 48 | 2.1 |
| (f) | 0.064 | 0.08 | 13 | 7.6 |
| (g) | 0.048 | 0.08 | 17 | 5.9 |
| (h) | 0.032 | 0.08 | 24 | 4.2 |
| (i) | 0.016 | 0.08 | 47 | 2.1 |

- Both graphs of rate against concentration should produce straight lines with positive gradients that pass through the origin.
- The rate with respect to peroxodisulfate ions is first order.
- The rate with respect to iodide ions is first order.

# Core practical 13b: Use a clock reaction to determine a rate equation

## Objectives

- To use a clock reaction to find the order of reaction with respect to iodide ions
- To use a clock reaction to find the order of reaction with respect to peroxodisulfate ions

## Safety

- Wear a lab coat goggles.
- Tie long hair back.

## All the maths you need

- Translate information between graphical, numerical and algebraic forms.
- Plot two variables from experimental or other data.

## Equipment

- 100 $cm^3$ of 0.2 mol $dm^{-3}$ sodium peroxodisulfate solution
- 100 $cm^3$ of 0.2 mol $dm^{-3}$ potassium iodide solution
- 50 $cm^3$ of 0.05 mol $dm^{-3}$ sodium thiosulfate solution
- 20 $cm^3$ of 1% starch solution
- distilled/deionised water
- white tile
- four 10 $cm^3$ measuring cylinders
- dropping pipettes
- four 100 $cm^3$ beakers
- four 250 $cm^3$ beakers
- stop clock

## Procedure

1. Measure 10.0 $cm^3$ of potassium iodide solution into a small beaker standing on a white tile.
2. Add 5.0 $cm^3$ of sodium thiosulfate solution to the potassium iodide solution.
3. Add 10 drops of starch solution to the mixture in the small beaker. Starch acts as the indicator and must be used in each experiment.
4. Measure out 10.0 $cm^3$ of the sodium peroxodisulfate solution. Pour this into the mixture prepared in steps 1 and 2. Start the stop clock.
5. Stop the clock when a blue colour appears in the beaker and note the time taken.
6. Make a copy of Table 2 from the 'Analysis of results' section. Use this to record your results.
7. Repeat steps 1–5 using the volumes of sodium peroxodisulfate and potassium iodide solutions shown in Table 1. The total volume, including the sodium thiosulfate solution, must add up to 25.0 $cm^3$, which can be achieved by adding the correct volume of distilled/deionised water.

**Table 1**

| Mixture | Vol. $S_2O_8^{2-}$/$cm^3$ | Vol. $I^-$/$cm^3$ | Vol. $S_2O_3^{2-}$/$cm^3$ | Vol. $H_2O$/$cm^3$ |
|---|---|---|---|---|
| (a) | 10.0 | 10.0 | 5.0 | 0.0 |
| (b) | 10.0 | 8.0 | 5.0 | 2.0 |
| (c) | 10.0 | 6.0 | 5.0 | 4.0 |
| (d) | 10.0 | 4.0 | 5.0 | 6.0 |
| (e) | 10.0 | 2.0 | 5.0 | 8.0 |
| (f) | 8.0 | 10.0 | 5.0 | 2.0 |
| (g) | 6.0 | 10.0 | 5.0 | 4.0 |
| (h) | 4.0 | 10.0 | 5.0 | 6.0 |
| (i) | 2.0 | 10.0 | 5.0 | 8.0 |

## Analysis of results

**Table 2**

| Mixture | Conc. $S_2O_8^{2-}$ /mol dm$^{-3}$ | Conc. $I^-$/mol dm$^{-3}$ | Time/s | $\frac{1}{t}$ (∝ rate)/s$^{-1}$ |
|---|---|---|---|---|
| (a) | | | | |
| (b) | | | | |
| (c) | | | | |
| (d) | | | | |
| (e) | | | | |
| (f) | | | | |
| (g) | | | | |
| (h) | | | | |
| (i) | | | | |

1. Calculate the concentration of iodide ions in each of the 25.0 cm$^3$ solutions for experiments (a)–(e). Write these values in Table 2.
2. Use the times recorded in each experiment to work out the rates for these experiments. Write these values in Table 2.
3. Plot a graph of rate against concentration. Deduce the order of the reaction with respect to iodide ions.
4. Using the data from the first experiment (a) and the data from (f)–(i), work out the concentration of the peroxodisulfate ion in 25.0 cm$^3$ of solution. Write these values in Table 2.
5. Work out the rate for each of these concentrations. Write these values in Table 2.
6. Plot a graph of rate against concentration. Deduce the order of the reaction with respect to peroxodisulfate ions.

## Learning tips

- The initial rate is the instantaneous rate at the start of a reaction when the time, *t* is zero ($t$ = 0). The initial rate can be found by measuring the gradient of a tangent drawn at $t$ = 0 on a concentration−time graph.
- A clock reaction is a more convenient way of obtaining the initial rate of a reaction by taking a single measurement. The time, *t,* is measured from the start of an experiment for a visual change to be observed. This often involves a colour change or the formation of a precipitate.
- If there is no significant change in rate during this time, it can be assumed that the average rate of reaction will be the same as the initial rate. The initial rate is then proportional to $\frac{1}{t}$.

## Questions

1. Identify the main sources of uncertainty in the procedure used and the measurements recorded in this experiment. Calculate the percentage uncertainty for any measurements taken.
2. Suggest ways of minimising these uncertainties.
3. What is the overall rate equation for this reaction? The equation for the reaction is:
   $S_2O_8^{2-} + 2I^- \rightarrow 2SO_4^{2-} + I_2$
4. A suggested mechanism for the reaction is:
   step 1 $I^- + S_2O_8^{2-} \rightarrow (S_2O_8I)^{3-}$
   step 2 $(S_2O_8I)^{3-} + I^- \rightarrow 2SO_4^{2-} + I_2$

   Which of these steps is the rate-determining step? Use the rate equation to justify your answer.

# Core practical 13b: Use a clock reaction to determine a rate equation

## Objectives

- To use a clock reaction to find the order of reaction with respect to iodide ions
- To use a clock reaction to find the order of reaction with respect to peroxodisulfate ions

## Safety

- Wear goggles.
- Consult CLEAPSS Hazcards® 47B, 95B and 95C. Perform a risk assessment using up-to-date information before this practical is carried out.
- This experiment is very sensitive to changes in temperature and catalysis. Ensure all glass equipment is clean and allow solutions (particularly the starch solution) to reach room temperature before beginning.

| Equipment per student/group | Notes on equipment |
|---|---|
| 100 $cm^3$ of 0.2 mol $dm^{-3}$ sodium peroxodisulfate solution | Low hazard at this concentration.<br>Oxidising and harmful at higher concentrations. |
| 100 $cm^3$ of 0.2 mol $dm^{-3}$ potassium iodide solution | Low hazard |
| 50 $cm^3$ of 0.05 mol $dm^{-3}$ sodium thiosulfate solution (If you find the reaction too slow with a thiosulfate concentration of 0.05 mol $dm^{-3}$, then try reducing the concentration to 0.02 or 0.01 mol $dm^{-3}$.) | Low hazard |
| 20 $cm^3$ of 1% starch solution | Make from fresh, in boiling water, on the day of the practical. |
| distilled/deionised water | |
| four 10 $cm^3$ measuring cylinders | |
| dropping pipettes | |
| four 100 $cm^3$ beakers | |
| four 250 $cm^3$ beakers | |
| stop clock | |
| white tile | |

## Notes