Quick quantitative chemistry – the microscale way

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Learn how to do quantitative chemistry experiments involving reaction rates using microscale techniques that are relatively easy and quick to set up, without expensive equipment.

Quantitative chemistry allows students to generate experimental data and link it with their knowledge of particles, chemical equations, and numerical equations. This can be difficult for students to master and requires practice and familiarization with the equations and formulae involved.

Microscale chemistry methods use less reagents, reduce costs, decrease the time taken for preparation, and minimize the production of hazardous substances.[1] These microscale practical activities are relatively simple and quick to do, and thus, can help students focus on the chemistry and reduce the load on working memory. Despite the small masses and volumes involved, the data generated from microscale experiments shows equivalent or better results than traditional equipment, although a comparison of techniques is a useful exercise in error analysis.

Rates of reaction lessons can bring together concepts about how reactions are proceeding and how the particles are interacting. This can lead into collision theory, which identifies the factors required for a successful collision (sufficient energy and correct orientation of the particles) and factors that affect the rate of reaction, such as concentration, temperature, surface area, and the presence of catalysts.[2]

These activities can be done with students aged 16–18 and can take about 50–100 minutes each, depending on the number of repeats performed.

Activity 1: Investigating the effect of concentration on rate of reaction

The reaction between sodium thiosulfate and hydrochloric acid produces a fine precipitate of solid sulfur. This allows the relative rate of reaction to be calculated by measuring the time needed for the reaction mixture to become opaque.

$$Na_2S_2O_3(aq) + 2HCl(aq) \rightarrow S(s) + SO_2(g) + 2NaCl(aq) + H_2O(l)$$
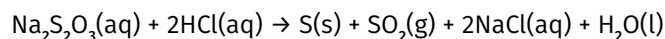
This microscale method has advantages over the large-scale reaction. Smaller amounts of chemicals are used, thereby reducing cost and preparation time. Due to the reduced volumes used, the total volume of toxic sulfur dioxide produced is significantly reduced, lowering the exposure to students and teachers. A stop bath is included, which stops the reaction by neutralizing hydrochloric acid and acidic sulfur dioxide, preventing further release of sulfur dioxide.

The time taken to do the experiment is reduced and repeats can be done if desired.

Materials
- Distilled water
- 1 M hydrochloric acid (caution: irritant)
- 0.1 M sodium thiosulfate
- Syringes (1 ml and 5 ml)
- Small glass vial (about 1 cm in diameter)
- Pen and paper
- Timer
- 0.5 M sodium carbonate
- Phenolphthalein solution in ethanol (0.1% w/v) (caution: highly flammable)
- Eye protection
- Large beaker (250 ml) for stop bath

Procedure
1. Make the reaction stop bath by adding 10 cm³ sodium carbonate solution to a 250 ml beaker and adding a few drops of phenolphthalein solution.
2. Draw a cross on a piece of paper.
3. Add 0.5 cm³ of hydrochloric acid to the vial using a 1 ml syringe. Place the vial over the cross.
4. Start the timer and add 4.5 cm³ of sodium thiosulfate solution from a 5 ml syringe to the reaction and measure the time until the cross can no longer be seen through the vial from above (figure 1). Record this time.
5. Pour the reaction mixture into the stop bath, clean the vial, and repeat using reaction mixtures 2–8 shown below.

Reaction number	1	2	3	4	5	6	7	8
1 M hydrochloric acid (cm³)	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Water (cm³)	0	0.5	1.0	1.5	2	2.5	3.0	3.5
0.1 M sodium thiosulfate solution (cm³)	4.5	4.0	3.5	3.0	2.5	2.0	1.5	1.0

Figure 1: As the reaction of sodium thiosulfate and hydrochloric acid proceeds, the sulfur precipitate formed obscures a cross drawn under the vial.
Image courtesy of Adrian Allan

6. From the results, plot a graph of relative rate of reaction (1/t) against sodium thiosulfate volume (cm³) for each vial (figure 2).

Results
A sample table of results obtained from this experiment is given below.

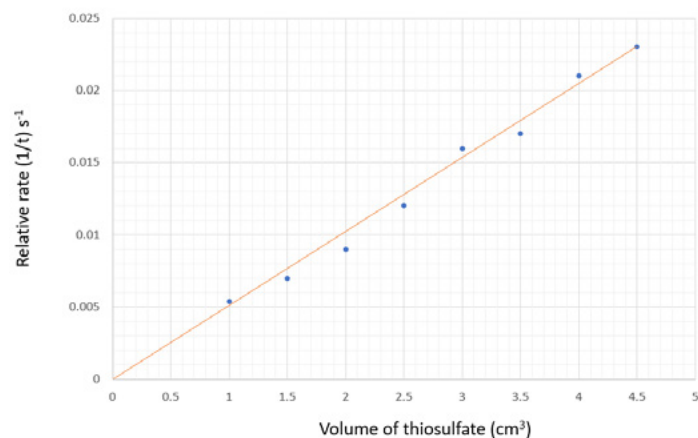| Volume of sodium thiosulfate (cm³)	Time taken for cross to disappear (s)	Relative rate of reaction, 1/t (s⁻¹)
1.0	183	0.0054
1.5	143	0.007
2.0	111	0.009
2.5	82	0.015
3.0	63	0.016
3.5	58	0.017
4.0	47	0.021
4.5	43	0.023

Figure 2: Graph showing the relationship between volume of sodium thiosulfate and relative rate of reaction
Image courtesy of Adrian Allan

Discussion

The results in figure 2 demonstrate that the relationship between the volume of thiosulfate solution and the relative rate of reaction is directly proportional. Since the total volume is always the same, the volume of thiosulfate solution can be taken as a measure of thiosulfate-ion concentration. An extension activity for older students could involve calculating the concentrations of thiosulfate ions and plotting concentration against relative reaction rate.

Collison theory can be discussed with students. Increasing the concentration results in more collisions between particles, since there are more particles occupying the same volume of space.

The data points for the slowest reactions have a higher chance of error and anomalous results because the time to obscure the cross is more gradual. This is a good discussion point and allows students the opportunity to repeat reactions and use averages, if desired.

This experiment can be done with larger volumes or with fewer reactions, depending on time and resources. An Arrhenius plot of the data collected can also be drawn, and Arrhenius equations can be used to determine the activation energy for the reaction, which is approximately 50 kJ/mol.[3] If microwell plates are available, the reaction can be done using dropper bottles instead of syringes.[4]

Optional extension

This activity can be extended to investigate the effect of temperature on the relative rate of reaction. By immersing the vial in hot or cold water, the reaction mixture can be heated or cooled quickly. The temperature of the reaction should be below 60C° to minimize exposure to sulfur dioxide.[5]

Activity 2: Investigating the effect of temperature on rate of reaction

The aim of this experiment is to find the effect of varying temperature on the relative rate of reaction between ethanedioic (oxalic) acid and an acidified solution of potassium permanganate:

$$5(COOH)_2(aq) + 6H^+ + 2MnO_4^{2-}(aq) \rightarrow 2Mn^{2+}(aq) + 10CO_2(g) + 8H_2O(l)$$
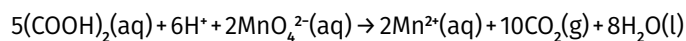
Initially, the reaction mixture is purple in colour due to the presence of permanganate ions, but it will turn colourless as soon as they are used up. This colour change allows us to follow the course of the reaction. Using dropper bottles allows the use of smaller volumes without sophisticated measuring apparatus and gives students more time to focus on the content rather than measuring accurately.

Safety notes

Some of the chemicals are harmful; wear eye protection. If any chemical splashes on skin, it should be washed off immediately with water.

Materials
- Distilled water in a dropper bottle
- 1 M sulfuric acid in a dropper bottle (caution: skin and eye irritant – wear eye protection)
- 0.02 M potassium permanganate solution in dropper bottle (caution: oxidizing, harmful)
- 0.2 M oxalic acid in dropper bottle (caution: harmful)
- Small glass vial (about 1 cm in diameter, with a capacity of at least 2 ml)
- Beaker of hot water recently boiled in a kettle
- Timer
- Small thermometer
- White tile

Procedure
1. Add 40 drops of distilled water, 5 drops of sulfuric acid, and 2 drops of potassium permanganate to a glass vial.
2. Insert a small thermometer into the vial and place the vial in a beaker of hot water until the mixture reaches 40C°. Note the temperature.
3. Place the vial on a white tile.
4. Add a drop of oxalic acid to the vial and start the timer at the same time.

5. Gently stir the mixture with a thermometer.
6. When the reaction mixture just turns colourless, stop the timer, and record the time in seconds (figure 3).
7. Repeat the experiment another three times, but heat the initial reaction mixture with water, sulfuric acid, and potassium permanganate first to 50C°, then 60C°, and finally to 70C°. More temperatures can be done if time allows.
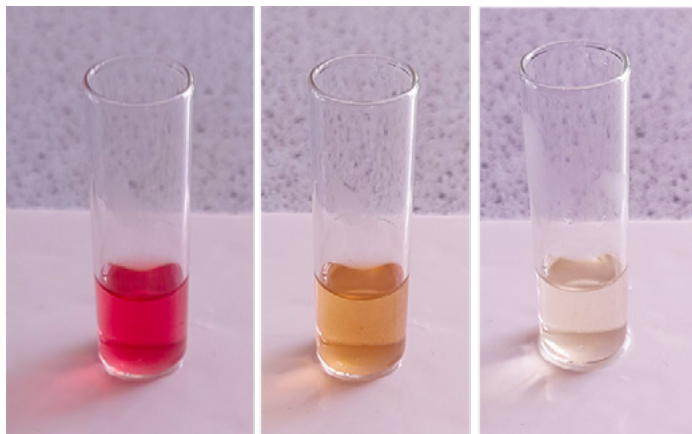

Figure 3: As the reaction of oxalic acid with acidified potassium permanganate solution progresses, the solution becomes colourless.
Image courtesy of Adrian Allan

Results

A sample table of results is shown below and the corresponding graph can be drawn (figure 4).

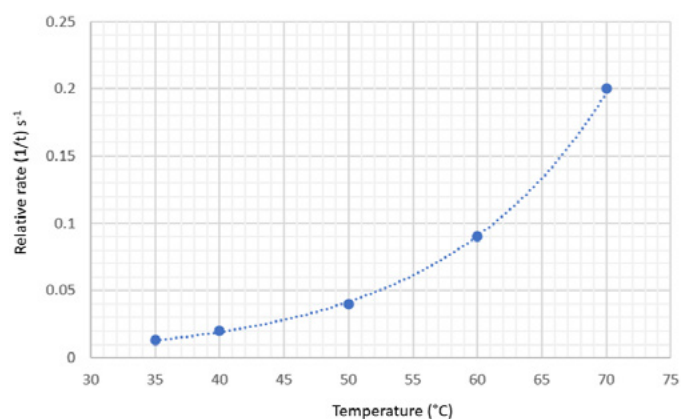| Temperature of reaction mixture at start of reaction (°C)	Time for reaction to go colourless (s)	Relative rate, $1/t$ (s^{-1})
34	78	0.0128
40	51	0.02
50	25	0.04
60	11	0.09
70	5	0.2

Figure 4: Graph showing the relationship between temperature of oxalic acid and acidified permanganate and rate of reaction
Image courtesy of Adrian Allan

Discussion

The results in figure 4 demonstrate that the relative rate of reaction increases with rising temperature. However, since the graph of rate against temperature is a curve, the rate is not directly proportional to temperature. In fact, it can be seen from the graph that the rate of reaction doubles if there is a temperature rise of about 10C°.

These results can be used to discuss with students the concept of activation energy, which is the minimum energy required for particles to have a successful collision. Diagrams showing the energy distribution of particles can be used to show how a small increase in temperature can give a significant increase in reaction rate, as more molecules will have an energy greater than the activation energy (figure 5).
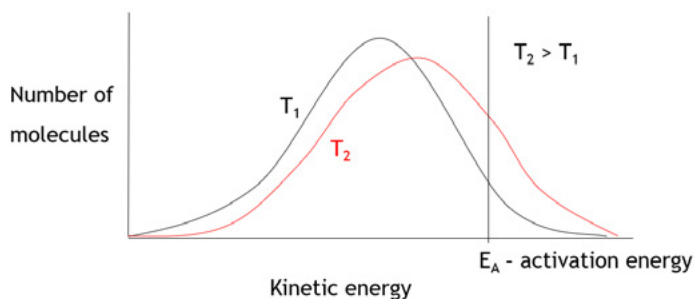

Figure 5: Distribution of energy among particles at different temperatures. As the temperature increases from T_1 to T_2, more particles have an energy greater than the activation energy ($_{EA}$). This increases the chance of successful collisions, resulting in an increase in reaction rate.
Image courtesy of Adrian Allan

Acknowledgements

References

[1] Worley B (2021) Little wonder: microscale chemistry in the classroom. *Science in School* **53**.

[2] The Royal Society of Chemistry resource to teach reaction dynamics and mechanisms: https://edu.rsc.org/cpd/teaching-rates-of-reaction-post-16/4013857.article

[3] Worley B, Paterson D (2021) *Understanding Chemistry through Microscale Practical Work* pp 58–59. Association for Science Education. ISBN: 978-0863574788

[4] The Science on Stage webinar on microscale chemistry: https://youtu.be/LM97yXJlotQ?si=e_IGnqLuTiJdPV84

[5] A video of a small-scale thiosulfate acid reaction with explanations: https://science.cleapss.org.uk/resource/thiosulfate-acid-reaction-rate-and-temperature.vid

Resources

- Follow a video make-it guide for constructing the microscale conductivity meter.
- Demonstrations of the experiments in this article can be found in this Science on Stage webinar on microscale chemistry.
- Watch a demonstration of the microscale electrolysis of copper chloride.
- Read an introduction to microscale chemistry in the classroom: Worley B (2021) Little wonder: microscale chemistry in the classroom. *Science in School* **53**.
- Teach about pH chemistry using microscale methods: Worley B, Allan A (2021) Little wonder: pH experiments the microscale way. *Science in School* **54**.
- Teach the chemistry of precipitation using microscale-chemistry methods: Worley B, Allan A (2022) Pleasing precipitation performances – the microscale way. *Science in School* **57**.
- Try a classroom activity to extract essential oils from fragrant plants: Allan A, Worley B, Owen M (2018) Perfumes with a pop: aroma chemistry with essential oils. *Science in School* **44**: 40–46.
- Learn how the hydrogen economy might work in practice: Mitov M, Hubenova Y (2014) A classroom hydrogen economy. *Science in School* **30**: 31–35.
- Learn how to make indicators from butterfly tea: Prolongo M, Pinto G (2021) Tea-time chemistry. *Science in School* **52**.
- Try some engaging experiments with bioluminescence that integrate biology, chemistry, and engineering: Wegner C, Hammann M, Zehne C (2021) Bioluminescence: combining biology, chemistry, and bionics. *Science in School* **53**.

AUTHOR BIOGRAPHY

Dr Adrian Allan is a teacher of chemistry at Dornoch Academy, UK. He was selected to represent the UK at the Science on Stage conferences in 2017 and 2019. He has presented Science on Stage webinars and workshops around Europe on microscale chemistry and using magic to teach science.

Bob Worley, FRSC, is the (semiretired) chemistry advisor for CLEAPSS in the UK. He taught chemistry for 20 years, and in 1991, he joined CLEAPSS, which provides safety and advisory support for classroom experiments. In carrying out these duties, he gained an interest in miniaturizing experiments to improve safety and convenience. He was awarded the 2021 Excellence in Secondary and Further Education Prize for significant and sustained contributions to the development and promotion of safe practical resources for teachers worldwide.

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