

# Practical Booklet 8

Iconic equilibria

## Cambridge International AS & A Level Chemistry 9701

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## Introduction

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Practical work is an essential part of science. Scientists use evidence gained from prior observations and experiments to build models and theories. Their predictions are tested with practical work to check that they are consistent with the behaviour of the real world. Learners who are well trained and experienced in practical skills will be more confident in their own abilities. The skills developed through practical work provide a good foundation for those wishing to pursue science further, as well as for those entering employment or a non-science career.

The science syllabuses address practical skills that contribute to the overall understanding of scientific methodology. Learners should be able to:

- plan experiments and investigations
- collect, record and present observations, measurements and estimates
- analyse and interpret data to reach conclusions
- evaluate methods and quality of data, and suggest improvements.

The practical skills established at AS Level are extended further in the full A Level. Learners will need to have practised basic skills from the AS Level experiments before using these skills to tackle the more demanding A Level exercises. Although A Level practical skills are assessed by a timetabled written paper, the best preparation for this paper is through extensive hands-on experience in the laboratory.

The example experiments suggested here can form the basis of a well-structured scheme of practical work for the teaching of AS and A Level science. The experiments have been carefully selected to reinforce theory and to develop learners' practical skills. The syllabus, scheme of work and past papers also provide a useful guide to the type of practical skills that learners might be expected to develop further. About 20% of teaching time should be allocated to practical work (not including the time spent observing teacher demonstrations), so this set of experiments provides only the starting point for a much more extensive scheme of practical work.

## Guidance for teachers

### Aim

To investigate the changes in pH during titrations using strong and weak acids and alkalis and to understand the nature of buffer solutions.

### Outcomes

Syllabus section 7.2(c), (e) and (f) as well as experimental skills 2, 3, and 4

Further work: syllabus section 7.2(h) and (k) as well as experimental skill 1

### Skills included in the practical

A Level skills	How learners develop the skills
Planning	plan how to extend an investigation to answer a new question identify the steps necessary to carry out the procedure show an understanding of how and why the procedure suggested will be effective
Analysis	use graphs to draw attention to the key points in quantitative data describe and summarise the key points of a set of observations understanding the nature of buffer solutions
Evaluation	identify where repeated readings are appropriate suggest modifications to the experimental procedure that will improve the accuracy of finding the equivalence point
Conclusions	calculation of pH of solutions (including buffers) determine the equivalence point for each set of observations suggest an appropriate indicator/indicators for acid – alkali combinations

This practical provides an opportunity to build on essential skills introduced at AS Level.

AS Level skills	How learners develop the skills
MMO quality	set up and use the apparatus to an appropriate level of precision obtain results that are close to those of an experienced chemist
PDO recording	record the pH values and total volumes of alkali added
PDO display	show the level of precision of their readings
PDO layout	plot appropriate variables accurately on appropriate, clearly labelled x- and y-axes
ACE conclusions	carry out calculations involving moles of solutions

## Method

- **Learners must wear eye protection for these investigations.**
- The pH changes during the reaction of sodium hydroxide with hydrochloric acid are investigated. It is important that both solutions have the same concentration. Before learners carry out their work, the relative concentrations should be checked by the teacher (or technician) using a conventional titration with thymolphthalein as indicator: the end point should be within the range 24.8 – 25.2 cm<sup>3</sup>.
- It is easier to identify the buffer action that takes place if the ethanoic acid (the weak electrolyte) is pipetted into the beaker and the sodium hydroxide (strong alkali) is added from the burette 5.0 cm<sup>3</sup> at a time.
- Buffer action takes place when about 12.5cm<sup>3</sup> of NaOH are added, that is half of the ethanoic acid has been neutralised. This is because the concentrations of ethanoic acid (unreacted) and sodium ethanoate (produced) are equal at this point in the titration. If time allows, the experiment should be repeated with smaller volumes of sodium hydroxide added at around 12.5 cm<sup>3</sup>, so that the buffering effect is more clearly observed.
- In the same way, it is desirable that learners should observe in more detail the nature of pH changes near the end-point. In order to do this, the experiment should be repeated with a smaller volume of sodium hydroxide added at around 25.0cm<sup>3</sup>.
- Learners should notice the sudden change in pH that takes place at the end-point. This sudden change allows the use of conventional indicators to show the end-point of a titration, because the indicator goes right through its colour change range while only a couple of drops of titrant are added at the end point.
- It is possible for learners to use narrow range universal indicator papers in place of a pH probe, if the latter is not available in sufficient numbers. In this case it is easier to use a conical flask for the reaction and swirl the solution after each addition of alkali. Then dip a glass rod (stirrer) into the mixture and touch the tip of the rod onto the appropriate indicator paper.
- Each learner/pair of learners should carry out the experiment with ethanoic acid and sodium hydroxide. If time allows, different learners/pairs of learners should carry out the experiment using one other combination of strong/weak acid/alkali (NaOH + HCl, HCl + aq NH<sub>3</sub>, NH<sub>3</sub> + CH<sub>3</sub>COOH). Class members can then pool their results before plotting graphs.
- Using these graphs, learners can discuss which indicators, if any, would be suitable to determine the end-points of these titrations.

## Further work

- Learners investigate the characteristics of buffer solutions. The buffering action breaks down if too much acid or alkali is added to the buffer solution, because one of the chemicals needed in the buffering mixture gets used up.

## Results

- Learners should tabulate results clearly with unambiguous headings and correctly displayed units.
- All volumes and pH readings should be shown to a consistent number of decimal places.
- Plotted results should cover at least half of the available grid and be drawn with a sharp pencil. The plotting should be accurate (within  $\frac{1}{2}$  a small square and in the correct half of the small square, any points on a line having the centre of the mark on the line).

## Interpretation and evaluation

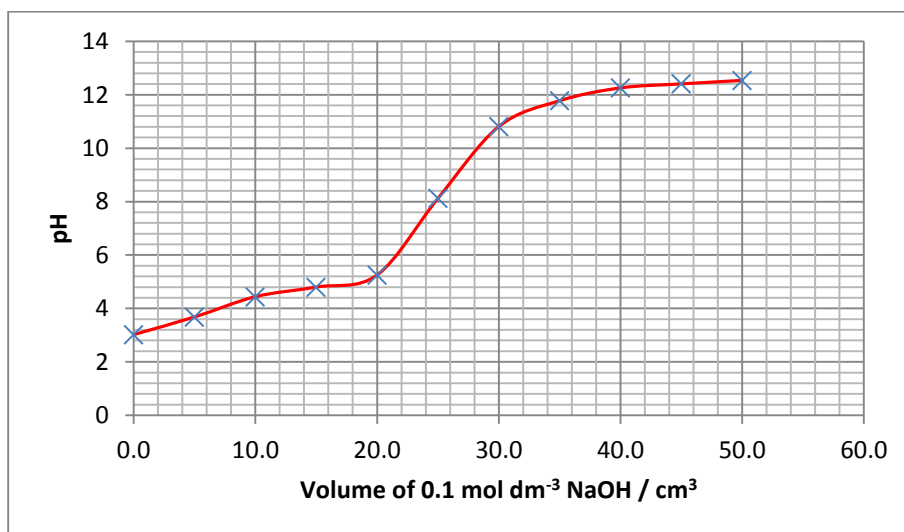
- The definition of pH can be introduced or revised.
- Discussion can take place to determine where the equivalence point lies on each graph.
- Possible reasons for different end-point pH values for the different combinations can be brainstormed. (Incomplete dissociation of the weak acid or alkali should be revised.)
- Possible ways of improving the end-point determination can be discussed. (Add NaOH in much smaller volumes as soon as the pH begins to change more rapidly than previously. This will give more points around the end-point so a more accurate line can be drawn.)
- The suitability of the indicators for each system can be discussed. (The indicator(s) selected should have colour changes in the almost vertical portion of the graph so that a colour change would be apparent on adding one drop ( $0.05 \text{ cm}^3$ ) of solution from the burette.)

The lack of suitable indicator for a weak acid / weak alkali combination can be discussed (lack of significant near-vertical section). (Each weak acid / weak alkali system will have a different pH value at the end-point which will depend on the  $K_a$  /  $pK_a$  and  $K_b$  /  $pK_b$  values.

## Typical results

$0.1 \text{ mol dm}^{-3}$  sodium hydroxide titrated with  $25.0 \text{ cm}^3$   $0.1 \text{ mol dm}^{-3}$  ethanoic acid

$pK_a = 4.62$ ,  $K_a = 2.4 \times 10^{-5} \text{ mol dm}^{-3}$



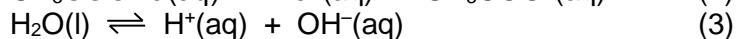
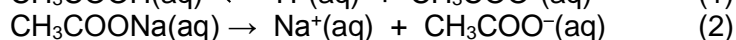
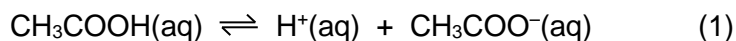
**Further work**

- Before starting further work the nature of buffer solutions can be introduced or revised. The graph of sodium hydroxide versus ethanoic acid can be used to illustrate the partial neutralisation of the weak acid by the strong alkali. Learners should note that the pH changes only slightly until the buffering capacity of the system is exceeded.
- Learners could use their graphs to determine the relative volumes of sodium hydroxide and ethanoic acid which correspond to a pH of 5.0. They can scale these volumes to work out how to prepare 25 cm<sup>3</sup> of buffer solution of pH = 5.

Learners should suggest how the appropriate volume of ethanoic acid could be measured.

The reasons for the pH value differing from 5.0 for the solution made can be discussed. (Smaller additions of NaOH beyond 20.0 cm<sup>3</sup> to obtain more points in the region where neutralisation occurs would give a more accurate volume of NaOH to use to prepare the buffer solution.)

- The equations involved in buffer systems can be used and the effects of adding small volumes of strong acid or alkali checked against experimental results.



The common ion effect of the ethanoate ion from (2) on the equilibrium shown in (1) can be discussed.

- Increasing the concentrations of the solutions making the buffer would give it greater buffering capacity.

## Information for technicians








Each learner will require:

- (a) Eye protection
- (b) 2 x 100 cm<sup>3</sup> beaker
- (c) 1 x 50 cm<sup>3</sup> burette
- (d) 1 x burette stand and clamp
- (e) 1 x filter funnel (for filling burette)
- (f) 1 x 25 cm<sup>3</sup> pipette
- (g) 1 x pipette filler
- (h) 1 x glass rod
- (i) 2 x teat / dropping pipettes
- (j) 1 x pH probe / pH meter
- [MH]** (k) 25 cm<sup>3</sup> pH7 buffer solution (ammonium ethanoate)
- [MH]** (l) 150 cm<sup>3</sup> 0.100 mol dm<sup>-3</sup> sodium hydroxide
- [MH]** (m) 120 cm<sup>3</sup> 0.100 mol dm<sup>-3</sup> ethanoic acid
- [N] [MH]** (n) 120 cm<sup>3</sup> 0.100 mol dm<sup>-3</sup> ammonia
- [MH]** (o) 120 cm<sup>3</sup> 0.100 mol dm<sup>-3</sup> hydrochloric acid
- [MH]** (p) 20 cm<sup>3</sup> 0.100 mol dm<sup>-3</sup> potassium hydroxide
- (q) paper towel

### Additional Instructions

If pH probes / pH meters are not available then narrow range universal indicator papers may be used with the chart of colour and pH value for each. If universal indicator papers are used then learners should use conical flasks instead of one of the 100 cm<sup>3</sup> beakers for the reaction mixtures.

### Hazard symbols

		
GHS02 ( <i>flammable F</i> )	GHS03 ( <i>oxidising O</i> )	GHS05 ( <i>corrosive C</i> )
		
GHS06 ( <i>acutely toxic T</i> )	GHS07 ( <i>moderate hazard MH</i> )	GHS08 ( <i>health hazard HH</i> )
		
	GHS09 ( <i>hazardous to the aquatic environment N</i> )	



# Worksheet

## Aim

To investigate the changes in pH during titrations using strong and weak acids and alkalis and to understand the nature of buffer solutions.

## Method

### Safety:

- Wear eye protection.
- 0.1 mol dm<sup>-3</sup> sodium hydroxide [H]
- 0.1 mol dm<sup>-3</sup> potassium hydroxide [H]

### Hazard symbols

H = harmful or irritating substance

1. Pour sufficient pH7 buffer solution into a 100 cm<sup>3</sup> beaker (or smaller) so that the bulb of the pH probe is covered by solution. Calibrate the pH meter so it reads 7.0 when the probe is placed in the buffer solution.
2. Using a pipette filler, wash the 25 cm<sup>3</sup> pipette with a little 0.1 mol dm<sup>-3</sup> ethanoic acid and discard the washings. Transfer 25.0 cm<sup>3</sup> of your acid solution into a 100 cm<sup>3</sup> beaker. (Touch the bottom of the pipette against the wall of the beaker or onto the surface of the solution to deliver the correct volume.)
3. Wash the burette with a little 0.1 mol dm<sup>-3</sup> sodium hydroxide and discard the washings. Then fill the burette to the 0.00 cm<sup>3</sup> mark, making sure that the region under the tap is full.
4. Remove the pH probe from the buffer solution, rinse it with a little distilled water and shake it gently to remove excess water. Place the probe in the solution of acid. Take and record a pH reading. This is your reading with 0.0 cm<sup>3</sup> alkali added.
5. Add 5.0 cm<sup>3</sup> of 0.1 mol dm<sup>-3</sup> sodium hydroxide to the acid in the beaker. Stir the mixture thoroughly. Measure and record the new pH reading. Record the exact volume of alkali added and the pH value.
6. Add a second 5.0 cm<sup>3</sup> portion of your alkali to the mixture in the beaker. Stir the mixture thoroughly and then take a new pH reading. Record the **total** volume of alkali added and the pH value. It does not matter if the volume added is not exactly 10.0 cm<sup>3</sup> as long as you record the accurate volume of alkali you have run out of the burette so far.
7. Continue adding 5.0 cm<sup>3</sup> portions of alkali to the beaker, stirring, and taking new readings of the total volume and pH until you have added a total volume of 50.0 cm<sup>3</sup> of alkali. (Take care not to spill the reaction mixture as you stir when larger total volumes of alkali are added.) Record all data in a table.
8. Repeat steps 1 - 5 of the procedure. Measure the pH when the following **total** volumes of sodium hydroxide have been added: 10.0, 12.0, 12.2, 12.4, 12.6, 12.8, 13.0, 15.0 cm<sup>3</sup>

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## Worksheet, *continued*

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9. Following on from step 9, measure the pH when the following **total** volumes of sodium hydroxide have been added: 20.0, 22.0, 24.0, 24.4, 24.6, 24.8, 25.0, 25.2, 25.4, 25.6, 26.0, 28.0, 30.0 cm<sup>3</sup>.
10. Repeat the procedure using different combinations of strong/weak acid/alkali.
  - (a) 0.1 mol dm<sup>-3</sup> sodium hydroxide (burette) with 25.0 cm<sup>3</sup> of 0.1 mol dm<sup>-3</sup> hydrochloric acid (pipette).
  - (b) 0.1 mol dm<sup>-3</sup> ammonia (burette) with 25.0 cm<sup>3</sup> of 0.1 mol dm<sup>-3</sup> hydrochloric acid (pipette).
  - (c) 0.1 mol dm<sup>-3</sup> ammonia (burette) with 25.0 cm<sup>3</sup> of 0.1 mol dm<sup>-3</sup> ethanoic acid (pipette).
11. For each combination of acid and alkali, plot a graph of pH against volume of alkali added.

### Results

Record **all** your observations.

Record and tabulate the results of your experiments.

Tabulate your results clearly with unambiguous headings and correctly displayed units.

All volumes and pH readings should be shown to a consistent number of decimal places.

Plotted results should cover at least half of the available grid and be drawn with a sharp pencil. The plotting should be accurate (within ½ a small square and in the correct half of the small square, any points on a line having the centre of the mark on the line.)

### Interpretation and evaluation

1. The graph of your experiment with sodium hydroxide and ethanoic acid should be approximately S-shaped. All the ethanoic acid has reacted at the mid-point of the steepest section of the graph.  
From your graph, what volume of aqueous sodium hydroxide is needed to neutralise 25.0 cm<sup>3</sup> of aqueous ethanoic acid? (Are the two solutions of exactly the same concentration?)
2. What is the pH of the mixture at this point?  
Suggest why your answer is not pH = 7.
3. From your graph, find the pH of the mixture when **half** the ethanoic acid has reacted. At this point the concentration of acid equals the concentration of base.  
Use this information and the equation below to calculate the acid dissociation constant for ethanoic acid. (You may convert the equation into a log expression.)

$$K_a = [\text{H}^+(\text{aq})][\text{CH}_3\text{COO}^-(\text{aq})] / [\text{CH}_3\text{COOH}(\text{aq})]$$

## Worksheet, *continued*

4. The syllabus lists four commonly used indicators. The names and ranges of pH over which their colours change is given below:

Indicator	pH range
methyl orange	2.9 to 4.6
bromophenol blue	3.0 to 4.5
thymol blue	8.0 to 9.6
thymolphthalein	9.3 to 10.5

Which indicator(s) would be suitable for determining the end-point for a titration of sodium hydroxide with ethanoic acid if no pH meter was available?

5. Find the equivalence points and pH at the end-point for the other 3 pairs of acid-alkali. Suggest, and explain, which of the above indicators would be suitable for determining the end-point for these titrations.  
Why would it be difficult to carry out a similar titration involving ammonia and a weak acid?

### Further work

- Decide how to use your results with the aqueous sodium hydroxide and aqueous ethanoic acid to make a buffer solution with pH 5.0.

Make 25 cm<sup>3</sup> of the pH 5.0 buffer using the same solutions as used in the original experiment. (You may wish to use additional apparatus.)

Test the pH of your buffer solution.

Is it exactly 5.0? If not, what errors may have occurred?

- Use 5 cm<sup>3</sup> portions of your buffer solution for each of the following experiments.
  - To the first portion, add five drops of 0.1 mol dm<sup>-3</sup> potassium hydroxide. Record the new pH.  
Add five more drops of KOH and record the new pH.  
Keep adding five drops of KOH each time and record the pH of the solution.  
Keep adding KOH until there is a significant change in the pH.  
Why is there a limit to the volume of potassium hydroxide you are able to add with only a small change in pH?
  - To the second portion, add five drops of 0.1 mol dm<sup>-3</sup> hydrochloric acid. Record the new pH.  
Add five more drops of the dilute hydrochloric acid to the mixture and record the new pH.. Keep adding five drops of hydrochloric acid each time and note what happens to the pH value after each addition.  
How would you make a buffer solution capable of resisting pH change when larger volumes of hydrochloric acid are added?

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