Cambridge Assessment International Education

Practical Booklet 3

Reacting masses and volumes, a titration exercise

Cambridge International AS & A Level Chemistry 9701

Version 2.0

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Introduction

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 determine the stoichiometric equation for the reaction between amidosulfonic (sulfamic) acid, NH_2SO_3H, and sodium hydroxide, NaOH, by a titration method.

Outcomes

Syllabus section 1.5 (a), (b)(iii) and (c) as well as experimental skills 2 and 3

Further work: syllabus section 6.1

Skills included in the practical

AS Level skills		How learners develop the skills
Manipulation, measurement and observation	*Successful collection of data and observations*	set up and use the apparatus to the level of precision indicated
	Quality of measurements or observations	obtain results that are close to those of an experienced chemist
	Decisions relating to measurements or observations	decide on the end point colour of the indicator decide how many titres are needed
Presentation of data and observations	*Recording data and observations*	record the burette readings with appropriate headings and units
	Display of calculation and reasoning	show the level of precision of their burette readings show working in the calculation and use significant figures appropriate to the precision of measurements
	Data layout	results clearly tabulated
Analysis, conclusions and evaluation	*Interpretation of data or observations and identifying sources of error*	calculate numbers of moles from titration data calculate maximum percentage errors for burettes and pipettes
	Drawing conclusions	write a correct equation for the reaction decide which piece of apparatus gives the greatest maximum percentage error

Method

Safety

- Learners must wear safety glasses for this investigation.

- Learners should be shown how to make up a standard solution by weighing a solid and using a volumetric flask. It is essential that they have opportunity to practise this technique until they carry it out accurately. Standard solutions for use in a titration are sometimes made up by diluting a more concentrated solution, using a pipette and volumetric flask.

- Learners should be shown how to use pipettes (with fillers) and burettes with precision and accuracy. They should know how to run out a pipette in the proper manner. They should also understand and be able to carry out the different approaches needed when a burette is used for a rough titration and when it is used in an accurate titration. These techniques should also be practised. Two accurate titres within 0.1 cm^3 should always be obtained in any experiment.

- The accuracy of the titration technique is due to the precision of the apparatus and to the use of dilute solutions. This means that the one drop of reactant (approximately 0.05 cm^3) needed to obtain an end point contains a very small fraction of a mole so is very sensitive.

- Leaners should appreciate that the amount of indicator used should be reasonably small, but sufficient to allow the colour change to be clearly identifiable. Many end-point colour changes are subtle, so it is important the learners have used a range of indicators in their titration work.

- The advantage of using solids such as amidosulfonic acid or potassium hydrogen phthalate is that they are primary standards which are chemically stable and can be used to find the concentration of the other reagent accurately. However, more common solids are satisfactory for elementary work: anhydrous sodium carbonate can be used as the alkali when an acid is being investigated.

- This method can be used for **further work** when studying redox reactions. Learners should carry out titrations with potassium manganate(VII) to investigate reducing agents, such as iron(II) sulfate or hydrogen peroxide. Titrations in which iodine is produced, and then titrated with sodium thiosulfate, can be used to investigate oxidising agents.

- Types of investigation possible include:
  (a) determination of the stoichiometric equation for a reaction;
  (b) investigation of the change in oxidation number of one of the reactants;
  (c) determination the concentration of one of the reactants;
  (d) determination of the percentage purity of a reactant.

Results

Learners should tabulate the initial and final burette readings and titres (volume used) for the rough and as many accurate titres as deemed necessary with unambiguous headings and units shown as / cm^3 or (cm^3) (as specified in the syllabus). Burette readings for the accurate titrations should always be recorded to the nearest 0.05 cm^3 and titres are considered to be concordant/consistent if they are within 0.10 cm^3.

Interpretation and evaluation

- You can discuss which titres to use in calculating the volume of alkali to be used in the calculations and the number of decimal places to use in the answer. (Selected titres should have a spread of ≤ 0.20 cm^3; answers should be arithmetically correct to 2 dp.)

- The equations $n = m/A_r$ and $n = cV$ can be introduced or revised.

- The appropriate number of significant figures can be discussed. For the number of moles, answers to 3 or 4 sf are appropriate given the precision of the measuring instruments and that the concentration of the alkali is shown to 3 sf.

Extension

- $NaOH + NH_2SO_3H \rightarrow NH_2SO_3Na + H_2O$
  Using the full equation as a starting point, discussion can take place about what constitutes a neutralisation reaction and which are ‘spectator’ ions.
  $H^+(aq) + OH^-(aq) \rightarrow H_2O(l)$

- The choice of indicator and their colour changes at the end point can be discussed. Thymolphthalein is suitable if a strong alkali, such as NaOH, is used in the titration.

Bromophenol blue or methyl orange are suitable if a strong acid, such as HC*l*, is used in the titration

- Errors in measurements made with pipettes, burettes and balances can be discussed such as the effect of the number of readings for one measurement, what a maximum and minimum error would be in each case. (The max % error of the balance will depend on the precision of the balance used.)

Specimen results

Mass of amidosulfonic acid /g = 2.42
Mean titre /cm^3 = 24.83

Calculation

Moles of NH_2SO_3H in each titration = (2.42/97.1) ÷ 10 = 2.49×10^{-3} mol
Moles of NaOH = 2.48×10^{-3} mol
Reacting ratio of moles is 1:1
Max % error of pipette = 0.24%

Max % error of burette = 0.40%

Further work

- Other acid-base titrations can be carried out, using different indicators.

- Redox titrations, using potassium manganate(VII) or sodium thiosulfate, can be used to:
  (a) determine the stoichiometric equation for a reaction;
  (b) investigate the change in oxidation number of one of the reactants;
  (c) determine the concentration of one of the reactants.

Information for technicians

Each learner will require:

(a) Eye protection

(b) 1 × 50 cm^3 burette

(c) 1 × burette stand and clamp

(d) 1 × filter funnel (for filling burette)

(e) 1 × 25 cm^3 pipette

(f) 1 × pipette filler

(g) 1 × 100 cm^3 beaker

(h) 1 × 250 cm^3 volumetric (graduated) flask

(i) 1 × filter funnel (for transferring solution)

(j) 2 × 150 cm^3 or 250 cm^3 conical flask

(k) 1 × white tile

(l) 1 × glass rod

(m) 1 × spatula

(n) paper towel

(o) Access to a balance reading to **at least** 1 dp.

[HH] (p) approximately 2.5 g amidosulfonic acid (supplied in a stoppered container)

[MH] (q) 140 cm^3 0.100 mol dm^{-3} sodium hydroxide

(r) 300 cm^3 distilled water

Hazard symbols

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

Worksheet

Aim

To determine the stoichiometric equation for the reaction between amidosulfonic (sulfamic) acid, NH_2SO_3H, and sodium hydroxide, NaOH, by a titration method.

Method

Safety

- Wear eye protection.
- Amidosulfonic acid (solid) is a health hazard.
- Sodium hydroxide 0.100 mol dm^{-3} is a moderate hazard.

(1) Weigh a small beaker. Record the balance reading.

(2) Add between 2.40 g and 2.45 g of amidosulfonic acid and re-weigh. Record the new reading.

(3) Add approximately 40 cm^3 of distilled water to the beaker and stir to dissolve most of the acid.

(4) Transfer the **solution** to a 250 cm^3 volumetric flask. Do not transfer any remaining solid.

(5) Add approximately 25 cm^3 of distilled water to the beaker and stir to dissolve any remaining acid. Transfer this solution to the volumetric flask and repeat until all the acid has dissolved.

(6) Add distilled water to the beaker to wash out any remaining acid solution and transfer the washings to the volumetric flask.

(7) Make the solution up to the mark. Stopper the flask and shake it to mix the solution thoroughly.

Titration

(8) Clamp the burette carefully so that it is held vertically. Wash the burette with a little aqueous sodium hydroxide and discard the washings. Then fill the burette (through the funnel inserted at the top). Make sure that the region under the tap is full and the alkali level is on the scale. Remove the funnel.

(9) Take a reading at eye level of the position of the bottom of the meniscus on the scale. Burettes have scale markings every 0.1 cm^3 so are read to the nearest 0.05 cm^3. Record this initial burette reading in a suitable table of results.

(10) Use a pipette filler to introduce a small volume of solution of the acid into the 25 cm^3 pipette, wash the pipette and discard the washings. Using a pipette filler, fill the pipette until the bottom of the meniscus is on the marker line when the pipette is held vertically at eye level. Transfer the 25.0 cm^3 of your acid solution into a conical flask. Touch the bottom of the pipette against the wall of the flask or onto the surface of the solution to deliver the correct volume.

(11) Place the conical flask on the white tile under the burette.

(12) Add sufficient drops of bromophenol blue indicator to be able to see the yellow colour.
(If you are using methyl orange indicator the colour will be red at this stage.)

(13) Carry out a 'rough' titration. Determine the approximate volume of alkali needed to neutralise the acid in the conical flask. Swirl the flask between additions of alkaline solution from the burette. Add about 5 cm^3 of alkali at a time until the indicator colour starts to change colour as more alkali is added. Then add about 1 cm^3 of alkali at a time until the indicator turns blue (yellow if methyl orange is used).

Worksheet, *continued*

(14) Read the new level of the alkali in the burette. (Remember, the meniscus should be at eye level.) Record this final burette reading in your result table.

(15) Discard the contents of the conical flask, wash it with water and discard the washings.

(16) Pipette 25.0 cm^3 of acid solution into the conical flask and add indicator.

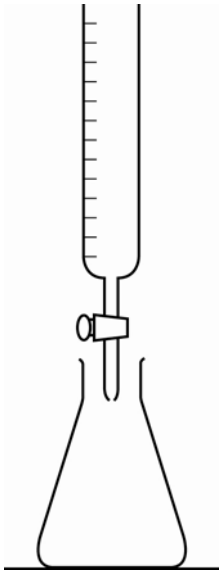
(17) If needed, top up the burette with the aqueous sodium hydroxide, take a reading of the level and record it. This is the initial burette reading

(18) Carry out an 'accurate' titration. Add aqueous sodium hydroxide from the burette to the aqueous amidosulfonic acid until the indicator just stays blue (or just stays yellow with methyl orange) when the solution is swirled. You should add the alkali a drop at a time, when close to the end point, until the colour of the indicator changes.

(19) Take the new reading of the alkali level and record it. This is the final burette reading.

(20) Repeat steps 15 – 19 until you have 2 concordant 'accurate' titres, that is, titres no more than 0.10 cm^3 apart.

Diagram of apparatus



Results

Record **all** your observations.

Tabulate the initial and final burette readings and titres (volume used) for the rough titration and as many accurate titres as deemed necessary with unambiguous headings and units shown as / cm^3 or (cm^3) (as specified in the syllabus).
Burette readings for the accurate titrations should always be recorded to the nearest 0.05 cm^3 and titres are considered to be concordant/consistent if they are within 0.10 cm^3.

Worksheet, *continued*

Interpretation and evaluation

Calculation

Use the Periodic Table for any data required.

(1) Calculate the number of moles of amidosulfonic acid, NH_2SO_3H, weighed out.
(2) Use your answer to (1) to calculate the number of moles of amidosulfonic acid present in the 25.0 cm^3 pipetted into your conical flask.
(3) Calculate the mean volume of sodium hydroxide that will be used in your calculations.
(4) Use your answer to (3) to calculate the number of moles of sodium hydroxide, 0.100 $mol\,dm^{-3}$ NaOH, required to neutralise the acid in the conical flask.
(5) Use your answers to (2) and (4) to calculate the mole ratio of alkali : acid.
(6) Write a balanced equation for the reaction between sodium hydroxide and amidosulfonic acid.

Extension

(7) Write an ionic equation, including state symbols, for the reaction.

Points to consider

(1) A pipette is marked as being accurate to ± 0.06 cm^3.
What is the maximum percentage error in the volume of amidosulfonic acid you pipetted into the conical flask?
(2) A single burette reading is accurate to ± 0.05 cm^3. What would be the maximum percentage error if your titre was 24.85 cm^3?
(3) The end point in a titration is when the indicator changes to the desired colour on adding one drop of reagent. One drop has a volume of approximately 0.05 cm^3. Calculate the number of moles of 0.100 $mol\,dm^{-3}$ of sodium hydroxide contained in this volume.
(4) The error in the balance reading is ± half the smallest division. For a 2 dp balance the error would be ± 0.005 g. Calculate the maximum percentage error for the mass of amidosulfonic acid you weighed out.
(5) Which piece of apparatus, the burette, pipette or the balance, caused the greatest percentage error in your experiment?

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