A microscale acid–base titration

This investigation is part of the **Nuffield practical collection**, developed by the Nuffield Foundation and the Royal Society of Chemistry. Delve into a wide range of chemical concepts and processes with this collection of over 200 step-by-step practicals: rsc.li/43bjGql

Learning objectives

1. Safely carry out a microscale titration of sodium hydroxide and hydrochloric acid.
2. Use practical results to calculate an unknown concentration.

Success criteria

Learners will carry out the microscale titration, aiming to achieve concordant results. They will use their results to carry out either a structured or unstructured calculation to find the unknown concentration of sodium hydroxide solution.

Introduction

In this experiment, learners use a microscale titration apparatus – prepared from pipettes, a syringe and some rubber or plastic tubing – to carry out a titration, filling the microscale burette with hydrochloric acid and placing sodium hydroxide solution in a beaker. They then calculate the exact concentration of the sodium hydroxide solution.

For this microscale technique, learners need to be capable of careful manipulation to carry the experiment out successfully. They also need to be familiar with the concept of the mole and capable of performing the calculations from the results of the experiment.

On such a small scale, safety issues are minimal and the time taken to carry out a titration is reduced as the volumes being reacted are so small. It is possible for a class to carry out the practical work and calculations in a one-hour session.

Scaffolding

A scaffolded (✪) sheet is available, which guides learners through the calculation stepwise. The unscaffolded (✪✪) version offers less guidance on the steps. If appropriate, you can alter the scaffolded version to remove some steps, for example prompts to convert volumes, or remove the provided equations.

The scaffolded sheet provides learners with a results table and units. The unscaffolded sheet requires learners to produce their own. You can alter this to provide a table without units, for example.

For a simpler introduction to microscale titration which focuses on observing the end-point of the reaction, try this Microscale titration experiment with integrated instructions: rsc.li/rsc.li/4iclogx

Technician notes

Equipment

Apparatus

- Graduated glass pipette, 2 cm^3
- Pipette, 1 cm^3, and pipette filler to fit (or a 1 cm^3 plastic syringe)
- Plastic syringe, 10 cm^3
- Fine-tip poly(ethene) dropping pipette
- Small lengths of rubber, plastic or silicone tubing
- Beakers, 10 cm^3, x 2
- Clamp stand with two bosses and clamps
- Safety glasses

Chemicals

- Dilute hydrochloric acid, 0.10 M, about 10 cm^3
- Sodium hydroxide solution, approx. 0.1 M (IRRITANT), about 10 cm^3
- Phenolphthalein indicator solution (HIGHLY FLAMMABLE), a few drops

Preparing the microscale titration apparatus

The microscale titration apparatus, or microscale burette, replaces the normal burette. To make the microscale titration apparatus, cut off the tip end of a fine-tip poly(ethene) dropping pipette and push the tip carefully onto the end of a 2 cm^3 graduated glass pipette. Clamp a plastic syringe, 10 cm^3 capacity, above the adapted pipette as shown in the picture, and connect the two with rubber, plastic or silicone tubing. Because the diameters of the syringe nozzle and of the top of the pipette may be quite different, two pieces of tubing, one to fit each end, will probably be needed; these can then be joined by an adaptor. A suitable adaptor can be made by cutting off the lower end of a 1 cm^3 plastic syringe, such that the syringe body diameter fits the wider tubing, and the syringe tip fits the narrower tubing (see the diagram and photograph: rsc.li/4brRDbJ).

Learners can build their own microscale titration apparatus from supplied components, but this is likely to take the learners more time than the titration itself! For that reason, it is preferable to prepare a class set of these in advance (or ready-made microscale titration kits can be purchased online).

*A suitable poly(ethene) dropping pipette would be fine-tip standard, non-sterile, 3.3 cm^3 capacity.

Safety and hazards

- Read our standard health and safety guidance: rsc.li/3zyJLkx
- Wear safety glasses throughout.
- Dilute hydrochloric acid, HCl(aq) – see CLEAPSS Hazcard HC047a and CLEAPSS Recipe Book RB043 or refer to your local safety advisory body.
- Sodium hydroxide solution, $NaOH$(aq), approx. 0.1 M, (IRRITANT at concentration used) – see CLEAPSS Hazcard HC091a and CLEAPSS Recipe Book RB085 or refer to your local safety advisory body. Learners are to calculate the concentration of the sodium hydroxide solution so the bottle should not be labelled with the exact concentration.
- Phenolphthalein indicator solution (HIGHLY FLAMMABLE) – see CLEAPSS Hazcard HC032 and CLEAPSS Recipe Book RB000 or refer to your local safety advisory body.

Method

A full method is provided in the student worksheet.

Teaching notes

This microscale technique minimises apparatus and chemical requirements and takes less time to perform than titration on the usual scale. Although the solutions used do present minor hazards, the use of such small quantities reduces risks from those hazards to very low levels. Nevertheless, make sure that learners take all the usual precautions in handling these solutions. The main risk is from misuse of the syringe or pipettes, especially if containing hazardous solutions.

The technique also makes the point that quantitative chemical experimentation does not always have to be performed on the traditional 'bucket' scale at school level.

An example of the results can be found in the student PowerPoint.

Answers

Scaffolded sheet

1. The equation shows that the ratio of hydrochloric acid to sodium hydroxide is 1:1.
2. Learners should select concordant results only.
   Using model results:
   - $Average\ titre = \frac{1.01+1.01+1.02}{3} = 1.01\ cm^3 = 1.01 \times 10^{-3}\ dm^3$

- Moles of hydrochloric acid = $1.01 \times 10^{-3} \times 0.1 = 1.01 \times 10^{-4}$ mol

3. a) As it is a 1:1 ratio, the moles of sodium hydroxide is also 1.01×10^{-4} mol.
   b) Volume of sodium hydroxide = 1.0 cm^3 = 1.0×10^{-3} dm^3

$$Concentration\ of\ sodium\ hydroxide = \frac{1.01 \times 10^{-4}}{1.0 \times 10^{-3}} = 0.101\ mol\ dm^{-3}$$

4. Producing a pure dry salt via titration:
   - The student must repeat the experiment **without indicator** as this would contaminate the salt.
   - The student should use evaporation and crystallisation to remove the water from the solution, leaving the pure, dry salt.
     Allow any description of this method e.g. use of a water bath to heat the solution.
   - If the salt is pure, it should have a fixed melting point which matches the accepted value of the melting point in databases.

Unscaffolded sheet

1. $HCl_{(aq)} + NaOH_{(aq)} \rightarrow NaCl_{(aq)} + H_2O_{(l)}$
   $H^+{}_{(aq)} + OH^-{}_{(aq)} \rightarrow H_2O_{(l)}$
2. $Moles\ of\ hydrochloric\ acid = \frac{volume\ in\ cm^3}{1000} \times 0.1 = 1.01 \times 10^{-3} \times 0.1 = 1.01 \times 10^{-4}\ mol$
3. Moles of sodium hydroxide = 1.01×10^{-4} mol

$$Concentration\ of\ sodium\ hydroxide = \frac{1.01 \times 10^{-4}}{volume\ in\ dm^3} = \frac{1.01 \times 10^{-4}}{1.0 \times 10^{-3}} = 0.101\ mol\ dm^{-3}$$

4. Key steps for producing a pure dry salt:
   - Student completes the titration using indicator to find the endpoint accurately.
   - Repeat the experiment **without indicator** as this would contaminate the salt.
   - Add the same volume of sodium hydroxide and hydrochloric acid as was used in the first titration.
   - Use evaporation and crystallisation to separate the salt from the solution. To do this, heat the salt in an evaporating basin over a water bath until most of the water has evaporated/the solution is saturated/you have reached the crystallisation point.
   - Remove from the heat and allow to dry slowly.
   - Pat the crystals dry.

To test if the salt is pure, use **melting point analysis**. The salt should have a fixed melting point if pure, and a melting point range if impure. The melting point can be compared to a database.