Unit 3: Finding out about substances

In this unit students learn about:

- how to take and prepare samples for analysis
- the use of standard procedures to ensure that the results of analysis can be replicated
- how to conduct qualitative analysis and a volumetric (quantitative) analysis
- how a chromatographic analysis and colorimetric analysis are conducted
- how to calculate a molar enthalpy change from experimentation and that energy changes take place during chemical reactions.

Students' portfolio evidence should consist of:

- a report on four analytical exercises including a qualitative analysis of an unknown inorganic chemical compound and an acid/base volumetric (quantitative) analysis
- the measurement of the molar enthalpy change of combustion of one reaction.

The activities

The activities give students experience of and an opportunity to demonstrate their ability to:

- obtain and prepare samples for analysis
- carry out qualitative chemical analysis and quantitative chemical analysis using titrations
- use chromatographic techniques and colorimetric techniques in analysis
- determine energy changes that take place when substances react.

The grid below summarises the activities. They provide more opportunities than students will probably have the time for and so suitable ones will need to be selected.

Note: The activity summaries refer to those aspects that link directly to the specifications. All are set in context and have some other parts to them.

A Large scale problems

Students:

- determine the temporary and permanent hardness of water by **titration** with standard soap solution
- devise and use an **acid-base titration** to determine the concentration of acid in a descaler, using information from a *Technique* sheet
- devise a test to compare the effectiveness of different descalers.

B Eggs need protection

Students:

- prepare samples of eggs for analysis following a standard procedure
- determine the calcium carbonate content of the shell using an **acid-base back titration**
- use **paper chromatography** to identify amino acids that make up the protein found in egg white (albumen)
- use **thin layer chromatography** to identify some of the lipids in egg yolk.

C Analysing soft drinks

Students::

- practice a technique by following standard procedures to determine the purity of citric acid using an **acid-base titration** (both with an indicator and using a pH measurements)
- modify one of these standard procedures to determine the acid content of a dilutable blackcurrant juice drink
- use **colour matching** and **colorimetry** to determine how much the juice has been diluted in a drink.

D Analysing food colourings

Students:

- compare the effectiveness of **paper and thin layer chromatography** for identifying food dyes present in a commercial food colouring; they are given Technique sheets
- follow a standard procedure to separate the food dyes by column chromatography, in readiness for determining their concentrations
- follow a standard procedure to determine the concentrations using **colorimetry.**

E Etchings: art and science

Students:

- use **qualitative chemical tests** to identify the ions present in an etchant
- use **qualitative chemical tests** to investigate the effectiveness of different etchants on different metals
- use **colour matching, colorimetry** and **redox titrations** to decide whether an etchant is 'spent' and needs replacing.

F The mine water problem

Students:

- follow a standard procedures to detect pollutant cations in a water sample using **qualitative chemical tests**
- follow standard procedures to determine the copper(II) ion concentration is a water sample by (a) **colour matching**, (b) **colorimetry**
- use a standard procedure to determine the concentration of zinc ions in a sample containing both zinc and copper(II) ions by **titration.**

G Analysing analgesics

Students:

- use a standard procedure to identify the active ingredients in an analgesic by **thin layer chromatography**
- follow standard procedures to determine (a) ibuprofen using an **acid-base titration**, (b) aspirin using an **acid-base back titration**, (c) paracetamol using a **redox titration.**

H Practice calculations

Students:

- practice changing masses of substance into moles and vice versa
- practice calculating reacting quantities using chemical equations
- practice titration calculations.

I Runaway exotherms

Students:

- measure **molar enthalpy of combustion**
- analyse patterns in molar enthalpy change data
- monitor temperature change
- find out about making use of exothermic reactions.

Practical Chemistry for schools and colleges is an interactive CD rom for chemistry students and teachers to support practical work in post-16 courses. It was sent free of charge to schools and colleges and would be useful in several of the activities. The CD rom contains detailed video footage of techniques including using burettes and pipettes, carrying out titrations and thin layer chromatography. It's published by the Royal Society of Chemistry in association with the Chemistry Video Consortium, ICI and Sigma-Aldrich. See:
http://www.chemsoc.org/networks/learnnet/pract-cd.htm

The CD rom might be a useful to introduce techniques or to recall demonstrations of techniques just prior to using them.

A

AQA
AS GCE APPLIED SCIENCE
UNIT 3: FINDING OUT ABOUT SUBSTANCES

LARGE SCALE PROBLEMS

Specification links

This investigation relates to the following sections of Unit 3: Finding out about substances

Content

- How to obtain and prepare sample for analysis
- Volumetric analysis (acid-base titration)

Assessment evidence

- an acid/base volumetric (quantitative) analysis

science

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THE INVESTIGATION

LARGE SCALE PROBLEMS

Scale can be a problem if you live in a hard water area. Boiling hard water produces limescale. You've probably seen the effect. Kettles, coffee makers and shower heaters - in fact anything that's used to heat water - fur up. Scale formation is a serious problem in industries where water is heated or heat exchangers are used.

It's been estimated that, in an average hard water area, it takes only two years for 6 mm of limescale to form in pipes or on heat exchangers. This cuts energy efficiency by 40%. Each year billions of pounds are wasted in higher energy bills, lost production and replacing equipment. Lime causes large scale problems in both senses of the phrase.

Contents

The science at work

What is hard water?

There are various tell-tale signs that water is hard. If you wash in it using soap, a scum forms. If you boil it in a kettle, the kettle gets furred up (perhaps not very noticeably until you have used the kettle many times). The 'hardness' is due to dissolved calcium and magnesium salts.

The main salt is calcium hydrogencarbonate, with smaller amounts of calcium sulfate. Magnesium hydrogencarbonate and magnesium sulfate are also present. They cause the same problems. However, the magnesium ion concentration in solution is much lower than the calcium ion concentration.

Here is the chemistry …

Soap is a sodium or potassium salt of a fatty acid (a large naturally occurring carboxylic acid) such as stearic acid. The insoluble calcium salt forms, for example, when sodium stearate reacts with calcium hydrogencarbonate:

$$\underset{\text{sodium stearate}}{2RCOONa\ (aq)} + Ca(HCO_3)_2\ (aq) \rightarrow \underset{\text{calcium stearate ('scum')}}{(RCOO)_2Ca\ (s)} + 2NaHCO_3\ (aq)$$

Limescale and fur are calcium carbonate. It forms when hard water is boiled:

$$Ca(HCO_3)_2\ (aq) \rightarrow CaCO_3\ (s) + CO_2\ (g) + H_2O\ (l)$$

Hardness: permanent and temporary

There are two types of hardness: temporary and permanent. Temporary hardness causes furring, limescale and scum. It's due to calcium and magnesium hydrogencarbonates. Permanent hardness doesn't cause furring or limescale, but it does form a scum when washing with soap. It's due to calcium and magnesium sulfates.

How do descalers work?

The active ingredient in descalers is an acid. This dissolves the limescale. For example,

$$CaCO_3\ (s) + 2HCl\ (aq) \rightarrow CaCl_2\ (aq) + CO_2\ (g) + H_2O\ (l)$$

Various acids are used, including citric acid, hydrochloric acid, methanoic acid, phosphoric acid, sulfamic acid and hydroxyethanoic acid. Some examples are given in *Datasheet: Active ingredients in commercial descalers.*

Your brief

The investigation comes in two parts. In the first part you'll investigate the causes of hardness. You'll collect some samples of tap water and estimate its hardness by titration using soap solution as the indicator. You'll also investigate the effect of boiling water on its hardness.

In the second part you'll look at some commercial descalers. These contain various acids. Your task is to determine the concentrations of the acids and to compare the effectiveness of the descalers. Work in a group, with each person analysing one descaler. Together, discuss how to compare the effectiveness of the descalers and devise a method to use. Use this method to compare descalers and to see how the results relate to the acidity measurements you carried out.

The investigation

Temporary and permanent hardness

The hardness of water can be estimated by measuring the volume of standard soap solution required to give a lather or froth. You can use this method to compare unboiled water (total hardness) and boiled water (permanent hardness). The difference is a measure of the water's temporary hardness.

Use *Standard Procedure: Comparing the hardness of water* to compare the types of hardness in different water supplies.

Questions

1 Why do you use soap solution and not a detergent solution as the 'indictor'? [Hint: find out why detergents were developed.]

2 How are soaps and detergents made?

3 Find out how the hardness might be determined with greater accuracy.

4 Some medical evidence suggests 'some degree of water hardness' is advantageous in drinking water. Find out what these advantages might be (check data from more than one source).
(http://news.bbc.co.uk/1/hi/health/3396141.stm)

Optional

5 If hardness in water is removed before use, the need for a descaler is unnecessary. Hardness can be removed by use of a 'water softener' containing ion exchange resin. Describe how such an ion exchange resin works to remove water hardness. Include any chemical equations.

Commercial descalers

Carry out a survey to find a range of commercial descalers. In each case identify the acid or acids that are present. Write the formulae of the acids and equations for their reactions with calcium carbonate. You might look in a supermarket and try a web search.

Questions

1. What is the difference between a strong acid and a weak acid?
2. Classify the acids you have identified in descalers as strong or weak.
3. For weak acids, write equations to show the ionic equilibrium in aqueous solution.
4. What is the difference between descalers recommended for domestic use and those recommended for industrial use?

Acid content of commercial descalers

You will be given a sample of a commercial descaler. Devise and use a procedure to determine the concentration of acid in it. You will find useful information in *Technique: Acid-base titrations*.

Think about:

- which alkali to titrate the acid against;
- which indicator to use;
- carrying out some investigations to estimate the approximate concentration of acid;
- what assumptions you need to make to calculate the concentration of acid;
- how to calculate the acid concentration.

Questions

1. How did you choose the pH indicator?
2. How might the acid concentration be determined without the use of a pH indicator?
3. Summarise the analyses of other descalers investigated by members of your group.

Comparing the effectiveness of descalers

Devise a test to compare the effectiveness of the commercial descaler you are investigating. The method does not need to be of high accuracy, ± 5% will be fine. However, it must be a rapid and meaningful comparative test. Use the test to compare decalers investigated by other people in your group.

Questions

1. List the descalers in order of effectiveness according to you test.
2. Is there a relationship between the acidity of the descaler and its effectiveness?

Your findings

Write a report of your investigation into water hardness and descalers. It's important to:

- accurately record all your analytical data (observations and measurements);
- carry out all calculations properly, giving sources of error and calculating the percentage error;
- evaluate the significance of your results and the confidence you have in them.

Do not write out the standard procedures for comparing hardness, but do reference it in your portfolio. However, if you modified it you must record what you did differently and explain why.

If you work in a group, make it clear what you did and acknowledge results you obtained from other members of the group.

Don't forget to ...

- set out your report clearly and logically - headings and sub-headings will help;
- begin by explaining the vocational relevance of the analyses;
- include a risk assessment;
- record numerical data to the correct number of significant figures;
- show all the steps in each calculation;
- estimate errors and say something about your level of confidence in the data;
- collect and record relevant data from other members of your group.

Useful resources

www.kamco.co.uk
www.chemexnorthwales.co.uk
www.plumbworld.co.uk
www.simplegreen.co.uk
www.premierproducts.co.uk
http://www.metrohm.co.uk/bt_titration.asp

Standard Procedure: Comparing the hardness of water

1 Scope

This procedure may be used to estimate the total, temporary and permanent hardness of water. It may be used to determine the hardness of water from any source, for example, tap water, rain, river or lake. More accurate values may be obtained using edta titrations.

2 Definitions

Standard soap solution: a known amount of soap dissolved in a given volume of aqueous alcohol

Scum: a precipitate formed by reaction between soap and aqueous calcium and magnesium ions

3 Principle

Water often contains small amounts of calcium hydrogencarbonate, calcium sulfate, magnesium hydrogencarbonate and magnesium sulfate. These compounds make water 'hard'. More precisely, it is the calcium ions and magnesium ions that cause hardness.

Soap is a sodium or potassium salt of a fatty acid (a large naturally occurring carboxylic acid) such as stearic acid or palmitic acid. The sodium and potassium salts are soluble in water. The calcium and magnesium salts are not.

When, for example, sodium stearate is added to a solution containing calcium ions the following reaction occurs:

$2RCOO^-$ (aq) + Ca^{2+} (aq) → $(RCOO)_2Ca$ (s)

To estimate hardness, an aqueous solution of soap is reacted quantitatively with calcium and magnesium ions in water. A scum forms (the insoluble calcium and magnesium salts). When all the calcium and magnesium ions have been precipitated, a slight excess of soap produces a permanent lather.

Types of hardness:

- Temporary - can be removed by boiling (on boiling it produces limescale)
- Permanent - cannot be removed by boiling
- Total - sum of temporary and permanent hardness

Hardness is expressed as 'milligrams of calcium carbonate per litre of water' (mg $CaCO_3$ per dm^3). Remember: 1 litre = 1 dm^3.

4 Equipment and materials

- water sample for analysis
- 250 cm^3 stoppered conical flask
- 50 cm^3 burette
- 50 cm^3 measuring cylinder
- 250 cm^3 beaker
- tripod and gauze
- Bunsen burner
- standard soap solution (contains ethanol, therefore FLAMMABLE)

5 Procedure

1. Consult a risk assessment for this procedure and consider whether it needs to be adapted to suit the particular conditions under which you are working. Implement the control measures identified, modifying them as necessary, but first ask your teacher to check your risk assessment.
2. Fill a 50 cm^3 burette with standard soap solution.
3. Using a measuring cylinder, put 50 cm^3 of water sample into a 250 cm^3 conical flask.
4. From the burette, add 1.0 cm^3 of soap solution. Stopper the flask and shake vigorously for about 10 seconds. If a lather forms and lasts for at least two minutes, the water is declared soft (i.e. it contains no 'hardness') and the analysis is finished. If it's hard, a scum will form.
5. Add further 1.0 cm^3 portions of soap solution, stoppering and shaking the flask vigorously for about 10 seconds each time, until a permanent lather forms. A permanent lather is one that lasts for at least 2 minutes. Record titre V_1.
6. Using a measuring cylinder, put another 50 cm^3 of water sample into a 250 cm^3 beaker. Heat to boiling and continue boiling for 10 minutes. A white precipitate may form. CARE: when using a Bunsen burner, be sure the flame is not close to the soap solution.
7. Allow the water sample to cool. Pour back into the measuring cylinder and make up to 100cm^3 with distilled water.
8. Transfer this water sample to a 250 cm^3 conical flask.
9. Titrate the water sample by repeating stages 4 and 5. Record titre V_2.

6 Calculations

1 cm^3 standard soap solution is equivalent to 1 mg $CaCO_3$

- Total hardness = $20V_1$ mg $CaCO_3$ per dm^3
- Permanent hardness = $20V_2$ mg $CaCO_3$ per dm^3
- Temporary hardness = $20(V_1 - V_2)$ mg $CaCO_3$ per dm^3

7 Test Report

Your report should include:

- table of your burette readings and titres;
- values for total, permanent and temporary hardness expressed as mg $CaCO_3$ per dm^3.

Technique: Acid-base titrations

The science

An acid reacts with a base to give a salt and water only:

acid + base → salt + water

This is a neutralisation reaction. Remember: an alkali is a soluble base.

As with all chemical reactions, the acid and base react in a fixed ratio. For example:

- NaOH (aq) + HCl (aq) → NaCl (aq) + H2O (l)
  tells us sodium hydroxide and hydrochloric acid react in a 1:1 mole ratio
- $2NaOH$ (aq) + H_2SO_4 (aq) → Na_2SO_4 (aq) + $2H_2O$ (l)
  tells us sodium hydroxide and sulfuric acid react in a 2:1 mole ratio

The reaction can be monitored using:

- an acid-base indicator (one that has different colours in acid and base)
- a pH electrode and meter (there is a rapid change in pH at neutralisation)

Technique in outline

Suppose you want to find the concentration of a base in solution.

You would take a known volume of the solution and react it with an acid of known concentration (we call this a standard solution). From the volume of acid required to neutralise the base you can calculate the concentration.

Here is an example:

You have a solution of sodium hydroxide solution of unknown concentration. By experiment you find that 25 cm^3 of the solution requires 20 cm^3 of 0.100 mol dm^{-3} hydrochloric acid to react completely.

- 20 cm^3 of 0.100 mol dm^{-3} hydrochloric acid contains $\frac{20 \times 0.100}{1000}$ moles of hydrochloric acid
- From the equation NaOH (aq) + HCl (aq) → NaCl (aq) + H_2O (l)
  1 mole of hydrochloric acid reacts with 1 mole of sodium hydroxide
- Therefore there were $\frac{20 \times 0.100}{1000}$ moles of sodium hydroxide in 25 cm^3 of solution
- Therefore there are $\frac{20 \times 0.100 \times 1000}{1000 \times 25}$ moles of sodium hydroxide in 1000 cm^3 of solution
- Therefore the concentration of the sodium hydroxide solution = 0.080 mol dm^{-3}
  Remember: 1000 cm^3 = 1 dm^3

A typical procedure

Equipment and materials

- balance to weigh to ± 0.001 g
- 50 cm^3 burette
- 25 cm^3 pipette and safety filler
- 250 cm^3 volumetric flask
- 250 cm^3 conical flask
- funnel
- a suitable standard alkaline solution
- a suitable pH indicator, for example:
  - methyl orange for a strong acid/weak base titration
  - bromothymol blue or litmus for a strong acid/strong base titration
  - phenolphthalein for a weak acid/strong base titration [HIGHLY FLAMMABLE, HARMFUL]

Safety

Wear protective clothing and eye protection (preferably goggles giving chemical splash protection). Always use a pipette filler; never pipette by mouth.

A risk assessment must be carried out. Check it with your teacher before starting work.

Method

1 Prepare solution by weighing out a quantity of the sample and dissolving in water. Quantitatively transfer solution to a 250 cm^3 volumetric flask, make up to the mark with distilled water and mix thoroughly.

2 Pipette 25 cm^3 into a 250 cm^3 conical flask. Add three to five drops of a suitable indicator.

3 Titrate against a suitable standard solution in a burette. If the sample is acidic, use a standard alkali solution. If sample is a base, use a standard acid.

4 The 'end point' is when the indicator changes colour. The first titration is usually a 'rough' titre, which means that it may be a little more than needed for an accurate titration.

5 Repeat stages 3 to 4 sufficient times to achieve a set of reproducible (± 0.05 cm^3) results.

6 Take an average titre of the reproducible results.

Related modern techniques

Acid-base titrations are used in many analytical laboratories. However, carrying titrations out manually can be time-consuming even for an experienced analyst. Laboratories with large numbers of samples to analyse often use an automated robotic titrator.

A standard solution is added at a constant, measured rate to the to a sample solution. The pH of the reaction mixture is monitored constantly. The resulting electronic data are fed to a computer which produces a graph of pH against volume of standard solution added. An appropriate programme computes the data and provides the result of the analysis.

Datasheet: Active ingredients in commercial descalers

Chemex products

Chemex Sanclean

http://www.chemexnorthwales.co.uk/products/datasheet/sanclean.pdf

Concentrated Toilet Cleaner and Descaler SANCLEAN is a perfumed clinging biocidal toilet cleaner. It's thixotropic nature ensures that it clings to the enamel allowing a four way action that disinfects, cleans, deodorises and descales.

Contains hydrochloric acid 10-30%

Chemex Leisureclean

http://www.chemexnorthwales.co.uk/products/datasheet/leisureclean.pdf

Pool and Shower Room Cleaner LEISURECLEAN is a sanitising pool and shower cleaner. It has a unique four-way action that disinfects, cleans, deodorises and descales at the same time.

Contains hydrochloric acid 10-30%

Chemex Descale

http://www.chemexnorthwales.co.uk/products/datasheet/descale.pdf

Lime scale remover Non-tainting acid descaler for kettles, dishwashers, boilers etc. Carefully designed formulation with inhibitors achieves maximum descaling with minimum metal attack.

Dilute up to 3:1 with water

Contains hydrochloric acid 10-30%

Chemex Brickmate

http://www.chemexnorthwales.co.uk/products/datasheet/brickmate.pdf

Brick and Mortar Cleaning BRICKMATE removes mortar, scale, plaster, lime, mineral deposits, rust and corrosion from brick and concrete floors. Also from plant and equipment, glass and painted surfaces.

Dilute up to 8:1 with water

Contains hydrochloric acid 10-30%

Fernox products

Fernox DS-3

http://www.plumbworld.co.uk/info-42

Fernox DS-3 is used for removing limescale from any type of water heater to restore its efficiency. It is safe to use with steel, stainless steel, cast iron, copper, brass, PVC, ABS rubber polythene and most plastic pipework.

Contains sulfamic acid

Kamco products

SCALEBREAKER FX: descaling liquid

Descaling liquid for pipework, radiators, heating & cooling systems and equipment with either rust or limescale deposits. Dissolves iron oxides at ambient temperatures. Non fuming. Safe to use with steel, stainless steel, cast iron, copper, brass and most plastics / rubber.

Contains citric acid and phosphoric acid

SCALEBREAKER HD: high strength descaling liquid

SCALEBREAKER HD is a powerful & economic liquid for use in cleaning heavily scaled equipment, where speed and high solvency are critical. Rapidly dissolves limescale and rust. Contains wetting agents to penetrate heavy deposits. Safe to use with steel, cast iron, copper, brass and most plastics.

Contains hydrochloric acid >32% (with colour changing indicator)

SCALEBREAKER SR: descaling crystals for limescale deposits

SCALEBREAKER SR is safe to handle & store, but a strong acid when dissolved in water. Non fuming. Safe to use with steel, stainless steel, cast iron, copper, brass, aluminium, PVC, polythene and most plastics / rubbers. Dissolves up to 50% of its own weight of scale.

Contains sulfamic acid ca 98%

SCALEBREAKER CG: food-grade descaling crystals

Biodegradable crystals for solution in water. Very safe to handle and store. Removes limescale deposits from equipment used for food preparation and catering. Non fuming and non toxic. Safe to use with steel, stainless steel, cast iron, copper, brass, aluminium and most plastics.

Contains citric acid >99.8%

Datasheets for Kamco products: http://www.kamco.co.uk/MSDS.htm

Simple Green products

Lime Scale Remover

http://www.simplegreen.co.uk/produkte_03.php.htm

Concentrated Lime Scale Remover

Biodegradable - non-abrasive - non-flammable

Contains no bleach, ammonia or phosphorus. Removes lime scale and soap scum, deodorizes with fresh, pleasant scent.

Specially formulated to effectively dissolve and remove lime scale, lime scale deposits and soap scum. Regular use delays build-up of lime scale deposits. Deodorizes with pleasant wintergreen scent.

Contains hydroxyethanoic acid 5-10%

Datasheets for Simple Green products: http://www.simplegreen.co.uk/msds.php.htm

Bird Brand products

Bird Brand Multi Purpose Decaler

http://www.birdbrand.co.uk/msds/Kettle%20descaler.doc

For the removal of lime scale, kettles, showerheads, coffee makers, steam irons.

Contains methanoic acid 10-90%

Premiere Products

http://www.premiereproducts.co.uk/

Freshaloo Daily toilet maintainer, disinfectant and deodoriser

http://www.premiereproducts.co.uk/pdfs/freshaloo.pdf

For cleaning toilets and urinals

Viscous formulation for maximum cleaning power

pH 1.5

Contains 6.4% sulfamic acid (though datasheet says 5% but less than 15%)

T.D'Scale.

A daily toilet maintainer, descaler, disinfectant and deodorant for hard water areas
http://www.premiereproducts.co.uk/pdfs/tdscale.pdf
For cleaning toilets and urinals
Viscous formulation for maximum descaling and cleaning power
pH 0.5-1.0
Contains 8% hydrochloric acid (though datasheet says 5% but less than 15%)

T.D.30.

Powerful, concentrated, quick-acting acid toilet cleaner, descaler, bactericide and deodorant
http://www.premiereproducts.co.uk/pdfs/td30.pdf
Extremely effective in removing acid lime stains in toilets
For the periodic cleaning of toilets and urinals
Contains a quaternary bactericide to kill all common germs
Viscous formulation to ensure maximum adhesion to vertical surfaces
Contains a corrosion inhibitor
pH 1.5
Chemicals (Hazard Information and Packaging) classified "Irritant"
Contains 18% orthophosphoric acid (though datasheet says 15% but less than 30%)

T.D.10.

An aggressive, concentrated, quick-acting acid toilet cleaner, descaler, bactericide and deodorant
http://www.premiereproducts.co.uk/pdfs/td10.pdf
For the periodic cleaning of toilets and urinals
Contains a quaternary bactericide to kill all common germs
Contains a powerful detergent to assist penetration and soil removal
Contains a corrosion inhibitor
Directional pouring cap
A deep pink colour with pleasant almond deodorant
pH 0.2
CHIP classified "Irritant"
Contains 18% hydrochloric acid

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Outline

This assignment is about the hardness of water and the use of descalers. Students use:

- a titration using soap solution
- acid-base titration

They also plan and use a rate of reaction experiment to compare the effectiveness of descalers and relate this to their acidity.

It is suggested that students work individually, but the group's results should be shared. Students carry out practical work individually, but could work in small groups to share ideas, plan practical work, share and evaluate results. However, each student should write their own interpretation.

Hardness

A range of water samples can be analysed. These may come from students' homes and/or be 'artificial' samples prepared in advance (see *Technical Information*). Give each student a sample (about 500 cm^3) to analyse.

This is not an 'accurate' titration (the end-point is determined only to the nearest 1 cm^3). However, it provides good practice of some essential skills used in volumetric analysis.

Students might be asked to bring samples of tap water from their homes. If so, remind them to:

- use a plastic bottle with screw top;
- let the water run for a while before collecting the sample (remembering to wash the bottle 2-3 times before filling with water);
- collect about 500 cm^3.

Descaler acidity

Each student pair could investigate a different commercial descaler.

There are many descalers available, but they can be rather expensive. 'Artificial' commercial descalers can be made by making a solution of a given acid to a known concentration. *Datasheet: Active ingredients in commercial descalers* gives the composition of some descalers.

In determining of the acidity of a descaler it may be necessary to do a rough titration with 1 mol dm^{-3} sodium hydroxide to determine a dilution factor before titration with standard 0.100 mol dm^{-3} sodium hydroxide. In this case, the student needs to do a quantitative dilution.

Descaler effectiveness

Limescale is essentially calcium carbonate, though very small quantities of magnesium carbonate may also be present. The effectiveness of descalers can be compared by their reaction with calcium carbonate. It is, of course, essential that students make comparisons under controlled conditions. The particle size of calcium carbonate is crucial. It may be interesting for some students to use powdered calcium carbonate and others to use marble chips. Importantly, of course, the marble chips must all be about the same size.

If students are to compare their results it will be necessary that they agree on a 'standard method'. This might be done by asking students to work in pairs to think about how it might be done. The whole group could be brought together to share the ideas and agree the comparative test to be used by all of them. Students should be able to call upon their experiences at GCSE.

Possible methods for monitoring the rate of reaction:

- rate of loss of mass (as CO_2) given off;
- volume of CO_2 (using a gas syringe).

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Standard procedure: Comparing the hardness of water

The Procedure requires that 1 cm^3 soap solution ≡ 1 mg $CaCO_3$, necessitating a standard solution, e.g. Wanklyn (Griffin /Fisher Scientific code J/7500/17; Philip Harris B6A71043). This is a solution of potassium oleate in a water/ethanol mixture [FLAMMABLE], and is relatively expensive. If the water samples are hard, the volumes taken for analysis could be reduced, adjusting the calculation formulae in the Procedure accordingly.

50 cm^3 of water with a hardness of 200 mg $CaCO_3$ per dm^3 would require 10 cm^3 of the standard soap solution.

Students might be asked to bring samples of tap water from their homes. If so, remind them to:

- use a plastic bottle with screw top;
- let the water run for a while before collecting the sample (remembering to wash the bottle 2-3 times before filling with water);
- collect about 500 cm^3.

'Artificial' hard water samples can be made:

- Permanent hard water can be made by making a saturated solution of calcium sulfate in distilled water. The solubility of calcium sulfate is 0.24 g in 100 cm^3 of water. Therefore, a saturated solution has a permanent hardness of 176 mg $CaCO_3$ per dm^3.
- Temporary hard water can be made by bubbling carbon dioxide through limewater until it goes clear. Lime water contains about 0.16 g calcium hydroxide in 100 cm^3 of water. The resulting solution after bubbling CO_2 through has a temporary hardness of 216 mg $CaCO_3$ per dm^3.
- These solutions can be mixed in different proportions to give water samples containing differing amounts of permanent and temporary hardness.

The hardness of water depends on its source. Ranges sometimes quoted are:

Soft	0-75 mg $CaCO_3$ per dm^3
Moderately hard	75-150 mg $CaCO_3$ per dm^3
Hard	150-300 mg $CaCO_3$ per dm^3
Very hard	>300 mg $CaCO_3$ per dm^3

Equipment and materials

Standard procedure: Comparing the hardness of water

- water sample for analysis
- 250 cm^3 stoppered conical flask
- 50 cm^3 burette
- 50 cm^3 measuring cylinder
- 250 cm^3 beaker
- tripod and gauze
- Bunsen burner
- Wanklyn standard soap solution (FLAMMABLE)

Technique: Acid-base titrations

- balance to weigh to ± 0.001 g
- 50 cm^3 burette
- 25 cm^3 pipette
- 250 cm^3 volumetric flask
- 250 cm^3 conical flask

- funnel
- a suitable standard solution
- a suitable pH indicator, for example:

  methyl orange for a strong acid/weak base titration

  bromothymol blue or litmus for a strong acid/strong base titration

  phenolphthalein for a weak acid/strong base titration [HIGHLY FLAMMABLE, HARMFUL]

  'Artificial' descalers can be made using information from *Datasheet: Active ingredients in commercial descalers*. [CORROSIVE OR IRRITANT]

Comparing effectiveness of descalers

This will depend on the methods chosen by students. It is likely to include some or all of the following:

- 100 cm^3 beaker
- stopwatch
- balance to weigh ± 0.001 g
- 25 cm^3 measuring cylinder
- calcium carbonate (powdered or marble chips of as similar size to one another as possible))
- thermometer, 0 to 100 ºC
- gas syringe
- descalers [CORROSIVE OR IRRITANT]