**Water Analysis**

Teacher / Technician Guide





**Researching Chemistry**

**Higher**

Photo: Wikipedia, GDFL

Higher Physics Topical Investigation Skin Cancer—Prevention and Cure

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**Investigation Brief**

Suntan creams stop harmful UV radiation reaching the skin. Manufacturers’ products are rated with a Sun Protection Factor (SPF). Suntan creams can have SPF values from 6 to over 50.

UV radiation monitors normally measure irradiance in output intensity per unit area. Thus, a typical low intensity UV lamp may emit approximately 10 mWcm-2.

The aim of this investigation is to determine the effect of various suntan creams on the transmission of UV radiation. In particular, the relationship between SPF and absorption should be found.

**Investigation Notes**

UV lamps can be harmful. Make sure that you read the safety leaflet which is supplied with the UV lamp. Some cheap UV monitors do not measure the irradiance of UV radiation. Rather, they give an indication of UV index. It is possible to undertake this investigation with such a monitor, but results will be less reliable and accurate. UV radiation does not pass through many transparent materials (including glass). However, UV transparent acrylics are readily available.

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**Overview of the assignment and activities.**

The investigation will cover the following key areas:-

Nature’s Chemistry - Mandatory Knowledge

Ester, fats and oils

• Fats and oils, esters condensation and hydrolysis reactions

Soaps, detergents and emulsions

• Hydrolysis of esters. Structure of soap ions including covalent tail, (hydrophobic), and an ionic head (hydrophilic), cleansing action of soaps

• Production, action and use of detergents

Chemistry in Society

Chemical Analysis

• Volumetric titrations, volumetric analysis for quantitative reactions. Standard solutions, acid base and redox titrations

Researching Unit

• Filtration

• Distillation

• Use of a balance

• Titration

The assignment has two stages:

• Research stage

• Communication stage

The research stage involves gathering information/data from the internet, books, newspapers, journals, experiment/practical activity or any other appropriate source. Candidates must select, use and record their referenced sources.

Group work approaches are acceptable as part of the research stage when gathering information/data or undertaking an experiment/practical activity, but assessors must ensure that candidates are able individually to meet the evidence requirements of this assessment.

In the course of their assignment, candidates are required to:

* choose a relevant topic in chemistry (the assessor must review the appropriateness of the topic chosen)
* state appropriate aim (s)
* research the topic by selecting relevant data/information
* carry out risk assessment of procedure
* process and present relevant data/information
* analyse data/information
* state conclusions
* evaluate their investigation
* explain the underlying chemistry of the topic researched
* present the findings of the research in a report

The evidence for this assignment will consist of the report. Of the total of 20 marks available for the assignment, the marking instructions provide 16 marks for skills and 4 marks for knowledge and understanding. The table below shows how these marks are allocated to each of the criteria against which the evidence will be assessed.

|  |  |
| --- | --- |
| Criteria | Mark allocation |
| Aim(s) | 1 |
| Applying knowledge and understanding of chemistry | 4 |
| Selecting information | 2 |
| Risk assessment | 1 |
| Processing and presenting data/information | 4 |
| Analysing data/information | 2 |
| Conclusion(s) | 1 |
| Evaluation | 3 |
| Presentation | 2 |

Detailed marking instructions for the Higher Assignment can be found using the following link:

<http://www.sqa.org.uk/files_ccc/GAInfoHigherChemistry.pdf>

**Introduction**

The support pack contains a list of suggested investigative practical work related to the theme of Water. Teachers should feel free to edit these investigations to suit the needs of their own students. For example, some teachers may wish to remove some of the experimental guidance to make the planning process more challenging for able students. Additionally, the list of investigations is by no means exhaustive; teachers are free to develop their own alternative investigation topics to enhance the experience for students.

The Water Analysis support pack for the Researching Chemistry unit contains a selection of four suggested investigations.

|  |  |
| --- | --- |
| Investigation | Title |
| A | How hard is the water? How does the concentration of Calcium and Magnesium ions affect hardness of water? |
| B | How much iron is in water? |
| C | What is the concentration of phosphates in water? |
| D | Making and testing Soap and Detergent |

Many schools are likely to organise the learners into groups for investigative work issuing a different brief with each group. It is vital that teachers ensure that pupil’s research and write up of the investigation is their own work (with the exception of raw data/ results).

**Why is this topical?**

Access to clean water is of huge importance worldwide. While it is commonly thought to be a problem affecting poorer nations, pollution from industry and agriculture cause problems in richer countries as well. In the summer of 2014, pollution in Lake Erie caused an algal bloom resulting in the citizens of Toledo and surrounding regions to have to boil their water before drinking it.

In order to determine the safety of a water supply, it is necessary to analyse the water to determine what substances are in it and what remedial measures might be needed to make it potable (drinkable).

**Investigation A:**

*“How hard is the water? How does the concentration of Calcium and Magnesium ions affect hardness of water?”*

**Introduction**

Water makes up around 66.7% of the surface of the Earth. It is essential for all forms of life on Earth. In Scotland, drinking water is available on tap but depending on the area and the supply of water there are many dissolved ions.

The pupils’ task is to compare the hardness of different water samples by finding out the concentration of calcium and magnesium ions present in the sample. The technique of titration is used to determine the concentration of ions present. If bottled water is used as one of the samples the accuracy of the technique can be measured by comparing the values given on the label of mineral water.

**Background research**

The first stage of carrying out research in chemistry is to review what is already known about the topic of interest. Chemists use books, scientific papers, journals and the internet to carry out background research.

Pupils’ first task in the Researching Chemistry unit is to *independently* carry out background research into one of the investigations listed, which should be agreed with the teacher. In school, it is likely that background research will be carried out on the internet.

Once the pupil has completed their background research, they must then complete the unit assessment tasks and store their research evidence in a safe place.

**Assessment tasks**

1. **Record at least two sources of information relevant to the investigation**. Sufficient detail should be given to allow someone else to find the sources easily. For a website, the URL shown here is perfectly adequate <http://www.biodieselfillingstations.co.uk/>.
2. **Write a brief summary** of the information of relevance contained in each of the sources you have identified.

**Focus questions**

A1 What ions are present in water and how does this vary in different areas?

A2 How does tap water compare to bottled mineral water in term of the ions present?

A3 What ions are responsible for making water hard and how can the hardness of water be tested for.

A4 Give details of chemical reactions and formulae equations involved.

A5 How does the hardness of water affect the foaming capacity of soaps?

A6 Many countries add fluoride ions to drinking water. Why are fluoride ions added to water and why do some countries not allow fluoride to be added to drinking water.

A7 Why are chloride ions added to water?

**Advice on using the internet for background research**

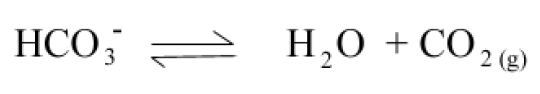
The web allows access a huge amount of information – don’t get side-tracked! Promising sites should be bookmarked so that they can be easily accessed at a later date. Tables, graphs and pictures can be copied into a folder. It is worthwhile spending a few moments considering which keywords may be the best to enter into the search engine. Ensure sites are credible. For more advice on effective web-based research see the Education Scotland resource on <http://www.educationscotland.gov.uk>

**Background to Water Hardness**

**Types of Hardness**

There are two types of water hardness, temporary and permanent.

Temporary Hardness is due to the bicarbonate ion, HCO3-, being present in the water. This type of hardness can be removed by boiling the water to expel the CO2, as indicated by the following equation:



Bicarbonate hardness is classified as *temporary hardness*.

Permanent hardness is due to the presence of the ions Ca2+, Mg2+, Fe3+ and SO42-. This type of hardness cannot be eliminated by boiling. The water with this type of hardness is said to be *permanently hard*.

The determination of water hardness is a useful test that provides a measure of quality of water for households and industrial uses. Originally, water hardness was defined as the measure of the capacity of the water to precipitate soap. Hard water is not a health hazard. People regularly take calcium supplements.

Drinking hard water contributes a small amount of calcium and magnesium toward the total human dietary needs of calcium and magnesium. In some parts of the UK, consuming extremely hard water could be a major contributor of calcium and magnesium to the diet.

Hard water does cause soap scum, clog pipes and clog boilers. Soap scum is formed when the calcium ion binds with the soap. This causes an insoluble compound that precipitates to form the scum you see. Soap actually softens hard water by removing the Ca2+ ions from the water.

When hard water is heated, CaCO3 precipitates out, which then clogs pipes and industrial boilers. This leads to malfunction or damage and is expensive to remove.

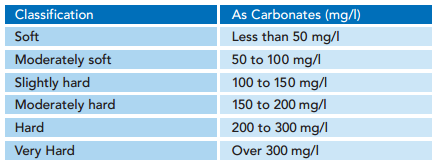
The ions involved in water hardness, i.e. Ca2+ (aq) and Mg2+ (aq), can be determined by two titrations with a chelating agent, ethylenediaminetetraacetic acid (EDTA), usually in the form of disodium salt. The titration reaction is:

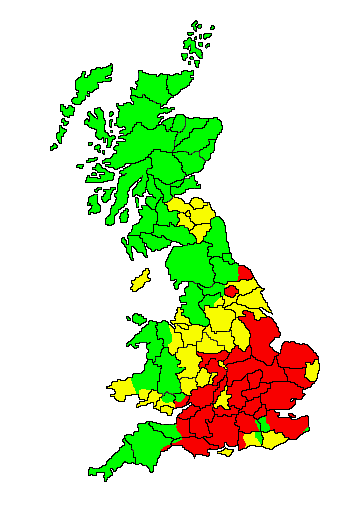
**Ca2++ EDTA4------> CaEDTA2-**

Total hardness (calcium and magnesium) is determined by titrating at pH 10 (using an ammonia buffer) with Eriochrome black T as the indicator.

Calcium alone can be determined using the same EDTA solution but this time using sodium bydroxide (to make the solution more alkaline) and Murexide as the indicator.

The table below shows the levels of dissolved solids in waters of differing hardness.



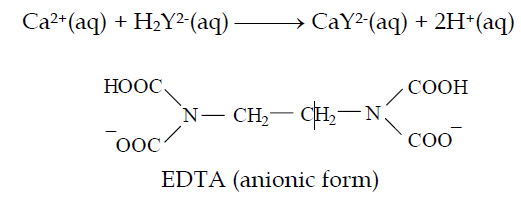
This map shows the hardness of the water supply in different pars of Great Britain.

**Red = Hard**

**Yellow = Medium**

**Green = Soft**

The ions involved in water hardness, i.e. Ca2+(aq) and Mg2+(aq), can be determined by titration with a chelating agent, ethylenediaminetetraacetic acid (EDTA), usually in the form of disodium salt. The titration reaction is:



Eriochrome Black T is commonly used as indicator for the above titration. At pH 10, Ca2+(aq) ion first complexes with the indicator as CaIn+(aq) which is wine red. As the stronger ligand EDTA is added, the CaIn+(aq) complex is replaced by the CaY2-(aq) complex which is blue. The end point of titration is indicated by a sharp colour change from wine red to blue.

Titration using Eriochrome Black T as indicator determines total hardness due to Ca2+(aq) and Mg2+(aq) ions. Hardness due to Ca2+(aq) ion is determined by a separate titration at a higher pH, by adding NaOH solution to precipitate Mg(OH)2(s), using murexide (or hydroxynaphthol blue) as indicator.

**References**

<http://www.sserc.org.uk/index.php/chemistry/higher-chemistry-revised/1136-principles-to-production-h/3468-water-testing>

<http://www.scottishwater.co.uk/you-and-your-home/water-quality/water-quality-factsheets/12-hardness-in-water>

<http://www.bbc.co.uk/schools/gcsebitesize/science/triple_ocr_gateway/chemistry_out_there/hardness_of_water/revision/1/>

<http://www.rsc.org/learn-chemistry/resource/res00000426/testing-water-hardness?cmpid=CMP00000496#!cmpid=CMP00000454>

**Practical procedure taken from**

<http://www.sserc.org.uk/index.php/chemistry/higher-chemistry-revised/1136-principles-to-production-h/3468-water-testing>

<http://www.york.ac.uk/chemistry/schools/chemrev/projects/>

**Planning the investigation**

The next stage in the investigation is to plan and carry out an appropriate experimental procedure that will allow the pupil to find out quantitatively the mass of calcium and magnesium ions in water.

Whilst planning experimental work the following may be considered:

* Which water sample(s) will be investigated?
* how to find out the actual calcium and magnesium ion content in the water?
* which chemicals will be required?
* what apparatus will be required?
* the hazards that might be involved and how will risks be minimised.

**Equipment and Materials Required**

|  |  |
| --- | --- |
| 0.001 M EDTA solution | 0.5% Eriochrome Black T solution  (This has a very short shelf life – prepare fresh each day). |
| 1M NaOH | 0.5% Murexide |
| pH 10 ammonia buffer | Burette |
| Clamp and stand |  |

**Method**

*Experiment 1 – Total Hardness Determination*

1. Fill a 50 cm3 burette with 0.001 mol l‑1 EDTA solution, making sure the tip is full and free of air bubbles..
2. Add 50.00 cm3 of an unknown hard water solution into a 100 cm3 beaker.
3. Add 10 cm3 of Ammonia buffer to the beaker.
4. Add 0.5 cm3 of Eriochrome Black T indicator.
5. Titrate with the 0.001 mol l-1 EDTA until the colour changes from wine red to pure blue. Read burette to +/- 0.10 cm3.
6. Repeat the titration until the final volumes agree to +/- 0.20 cm3.

*Experiment 2 – Calcium Determination*

1. Fill a 50 cm3 burette with 0.001 mol l-1 EDTA solution, making sure the tip is full and free of air bubbles.
2. Add 50.00 cm3 of an unknown hard water solution into a 100 cm3 beaker.
3. Add 4 cm3 of 1.0 mol l-1 Sodium Hydroxide.
4. Add 0.5 cm3 of Murexide indicator.
5. Titrate with the 0.001 mol l-1 EDTA until the colour changes from salmon pink to orchid purple. Read burette to +/- 0.10 cm3.
6. Repeat the titration until the final volumes agree to +/- 0.20 cm3.

**Calculations**

From the equation, 1 mole of calcium complexes with 1 mole of EDTA, so it is easy to work out the concentrations.

Molarity of EDTA solution is 0.001 mol l-1

1cm3 of 0.001 mol l-1 EDTA = 0.1 mg of CaCO3

Calcium– take the figure from experiment 2

Magnesium– subtract the result from experiment 2 (calcium) from the result of experiment 1 (total hardness)

**Reporting results**

The final stage of any scientific investigation involves reporting the results. Scientists use a wide range of communication methods to report their results, including scientific papers, laboratory reports, blogs, videos, scientific posters, podcasts, PowerPoints, web pages, etc.

**Assessment task**

Once the pupil has agreed the format of their scientific communication with the teacher, the pupil should produce a report on their investigation containing the following key features:

* a clear statement of the aim of the investigation
* Background/ Underlying Chemistry including research from at least two different sources.
* a brief explanation of how volumetric titration technique can be used to determine the calcium and magnesium ion concentration in water.
* your experimental observations and results, including the calibration graph
* a comparison of the actual and the calculated ion concentration in bottled mineral water.
* reasons why your experimental results are different from the actual results (if they are different)
* an evaluation description of any ways in which the results could be improved
* a valid conclusion, based on the evidence in the report, which relates to the aim.

**Investigation B:**

“How much iron is in water?”

**Introduction**

Water makes up around 66.7% of the surface of the Earth. It is essential for all forms of life on Earth. In Scotland, drinking water is available on tap but depending on the area and the supply of water there are many dissolved ions.

The pupils’ task is to find out the mass of iron in tap water from different areas or from fresh sources of water in the environment, such as rivers or springs. The technique of colorimetry can be used to measure the concentration of iron ions in an unknown sample of water using a calibration graph and comparing the concentration found to the value stated on the label of mineral water.

**Background research**

The first stage of carrying out research in chemistry is to review what is already known about the topic of interest. Chemists use books, scientific papers, journals and the internet to carry out background research.

The pupils’ first task in the Researching Chemistry unit is to *independently* carry out background research into one of the investigations listed, which will be agreed with the teacher. In school, it is likely that background research will be carried out on the internet.

Once the pupil has completed their background research, the pupil must then complete the unit assessment tasks and store their research evidence in a safe place.

**Assessment tasks**

1. **Record at least two sources of information relevant to your focus question**. Sufficient detail should be given to allow someone else to find the sources easily. For a website, the URL shown here is perfectly adequate <http://www.biodieselfillingstations.co.uk/>.
2. **Write a brief summary** of the information of relevance contained in each of the sources identified.

**Focus questions**

B1 Why does water contain iron? Where does the iron come from?

B2 What is iron needed for in our diets?

B3 What iron ions are the most common form?

B4 What compounds does iron form in water and in the human body?

B5 What are the effects of high concentrations of iron?

B6 Give details of the reactions involved and formulae equations.

**Advice on using the internet for background research**

The web allows access a huge amount of information – don’t get side-tracked! Promising sites should be bookmarked so that they can be easily accessed at a later date. Tables, graphs and pictures can be copied into a folder. It is worthwhile spending a few moments considering which keywords may be the best to enter into the search engine. Ensure sites are credible. For more advice on effective web-based research see the Education Scotland resource on <http://www.educationscotland.gov.uk>

**Background to Iron in Water**

Iron is the second most abundant metal in the earth's crust, of which it accounts for about 5%. Elemental iron is rarely found in nature, as the iron ions Fe2+ and Fe3+ readily combine with other elements to form oxides, hydroxides, carbonates, and sulphides. Iron is most commonly found in nature in the form of its oxides.

***Iron uptake in humans***

***Food***

Iron occurs as a natural constituent in plants and animals. Liver, kidney, fish, and green vegetables contain 20–150 mg/kg, whereas red meats and egg yolks contain 10–20 mg/kg. Rice and many fruits and vegetables have low iron contents (1–10 mg/kg).

***Water***

The median iron concentration in rivers has been reported to be 0.7 mg/litre. In anaerobic groundwater where iron is in the form of iron(II), concentrations will usually be 0.5–10 mg/litre, but concentrations up to 50 mg/litre can sometimes be found.

Concentrations of iron in drinking-water are normally less than 0.3 mg/litre but may be higher in countries where various iron salts are used as coagulating agents in water-treatment plants and where cast iron, steel, and galvanized iron pipes are used for water distribution.

***Estimated total exposure and relative contribution of drinking-water***

Reported daily intakes of iron in food - the major source of exposure - range from 10 to 14 mg.

Drinking-water containing 0.3 mg/litre would contribute about 0.6 mg to the daily intake.

Intake of iron from air is about 25 μg/day in urban areas.

***Effects on humans***

Iron is an essential element in human nutrition. Estimates of the minimum daily requirement for iron depend on age, sex, physiological status, and iron bioavailability and range from about 10 to 50 mg/day.

The average lethal dose of iron is 200–250 mg/kg of body weight, but death has occurred following the ingestion of doses as low as 40 mg/kg of body weight. Autopsies have shown haemorrhagic necrosis and sloughing of areas of mucosa in the stomach with extension into the submucosa. Chronic iron overload results primarily from a genetic disorder (haemochromatosis) characterized by increased iron absorption and from diseases that require frequent transfusions. Adults have often taken iron supplements for extended periods without deleterious effects, and an intake of 0.4–1 mg/kg of body weight per day is unlikely to cause adverse effects in healthy persons .

This experiment allows for the analysis of iron content in water or food samples. The iron is present in a solution containing Fe3+ (ferric) ions. To make the presence of these ions in solution visible, thiocyanate ions (SCN−) are added. These react with the Fe3+ ions to form a blood-red coloured complex:

Fe3+ (aq) + SCN−(aq) → [FeSCN]2+(aq)

By comparing the intensity of the colour of this solution with the colours of a series of standard solutions, with known Fe3+ concentrations, the concentration of iron in the tablet or food sample may be determined. This technique is called colorimetry.

<http://www.sserc.org.uk/index.php/chemistry/higher-chemistry-revised/1136-principles-to-production-h/3468-water-testing>

<http://www.youtube.com/watch?v=e6jDGzVuQxg>

**Planning the investigation**

The next stage in the investigation is to plan and carry out an appropriate experimental procedure that will allow the pupil to find out quantitatively the concentration of iron ions in the water samples.

Note that most bottled water does not contain iron so it is advisable to discourage pupils from using samples of bottled water.

Whilst planning the experimental work the following may be considered:

* Which water sample(s) will be investigated?
* What is the source of the water supply?
* How will the iron concentration be determined?
* If the iron concentration is too low it cannot be determined so how can the concentration be increased?
* Which chemicals will be required?
* What apparatus will be required?
* The hazards that might be involved and how you will minimise risk.

**Equipment and Materials Required**

|  |  |
| --- | --- |
| ferric ammonium sulphate FeNH4(SO4)2•12H2O standard solutions: | 2, 4, 6, 8 and 10 × 10−5 mol l-1  (see below for preparation) |
| 1 mol l-1 ammonium thiocyanate solution (see below for preparation) | 1 mol l-1 sulphuric acid |
| 1 mol l-1  hydrochloric acid | 100 cm3 beaker |
| 200 and 500 cm3 volumetric flasks | 5 cm3 pipette |
| 100 cm3 conical flask | Test tubes (5 for the references plus however many samples you are doing) |
| Colorimeter & cuvettes | distilled water |

**Method**

***Preparation***

*Preparation of 1 mol l-1 ammonium thiocyanate solution*

1. Weigh 38 g of solid ammonium thiocyanate into a 500 cm3 volumetric flask and make up to the mark with distilled water.

*Preparation of Fe3+ standard solutions*

NB: It may take several days to dissolve the Fe3+ salt used here, so carry out this preparation well in advance of the rest of the experiment.

1. Weigh out about 3.0 g of ferric ammonium sulphate (FeNH4(SO4)2•12H2O). Use a mortar and pestle to grind the salt to a fine powder. Accurately weigh 2.41 g of the powder into a 100 cm3 beaker and add 20 cm3 of concentrated sulphuric acid [corrosive]. Leave powder to soak in acid overnight.
2. The next day, carefully pour the acid/powder slurry into a 500 cm3 volumetric flask, rinsing the beaker into the flask a few times with water, then make up to the mark with distilled water. Let this solution stand for several days until the ferric ammonium sulphate powder has fully dissolved. If possible, insert a magnetic stirrer bar and stir the solution to speed up this dissolving process.
3. Use a pipette to transfer 20 cm3 of ferric ion solution to a 200 cm3 volumetric flask and make up to the mark with distilled water. This gives a solution with [Fe3+] = 0.001 mol l-1.
4. To prepare a 2 × 10−5 mol l−1 standard solution pipette 10 cm3 of the 0.001 mol l−1 solution into a 500 cm3 volumetric flask, add 10 cm3 of 1 mol l-1 sulphuric acid, and then make up to the mark with distilled water.
5. Repeat this procedure in separate 500 cm3 volumetric flasks\*, pipetting in 20, 30, 40 and 50

cm3 of 0.001 mol l-1 Fe3+ solution in turn, to obtain 4, 6, 8 and 10 × 10−5 mol l-1 solutions respectively.

(\* if you do not have five 500 cm3 volumetric flasks you can use one flask to prepare each standard in turn. After preparing each standard, pour the solution into a labelled glass vessel which has a lid (eg: a glass bottle). Then rinse your 500 cm3 volumetric flask thoroughly with distilled water before using it to prepare your next standard solution.)

***The experiment***

1. Accurately measure 2 cm3 of your sample solution into a clean, dry test tube\*.
2. Next, measure 2 cm3 of each Fe 3+ standard solution into separate test tubes (one standard per tube) in order of increasing concentration, beginning with the 2 × 10−5 M standard. It is a good idea to first rinse your pipette or measuring cylinder with a few cm3 of the 2 × 10−5 M standard.

NB: Make sure you label each boiling tube appropriately with the name of the sample or standard it contains.

1. Add 2 cm3 of 1 M1 ammonium thiocyanate solution to each iron solution in sequence, with 2 minutes between each addition\*\*. These additions must be carefully timed so that all samples react for the same period of time.
2. Mix the solutions by swirling. A stable red colour will appear over the next few minutes.
3. As near as possible to 15 minutes after adding thiocyanate\*\*\*, pour your samples into a cuvette and measure the absorbance at a wavelength of 490 nm for each coloured solution using your colorimeter.

These measurements will be made in sequence − one sample every two minutes − reflecting the timing of the thiocyanate additions above.

The measured absorbance of light is a direct measure of the intensity of the solution’s colour.

*\* You can, if you wish, simply add the solutions and mix in a cuvette.*

*\*\* If you have a colorimeter to hand, you should be able to do it faster than this. 1 sample per minute is easily achievable and with practice, one every 30s is quite possible.*

*\*\*\* As long as you are reasonably close to the time, all should be well. When left for another 5 minutes, the darkest of the reference samples only dropped from a reading of 0.44 to 0.42. An extra 10 minutes caused a further drop to 0.40.*

**Calculations**

1. Using only the absorbance results obtained for your Fe3+ standard solutions (not your unknown iron sample), prepare a graph with [Fe3+ ] (in 10-5 M) as the horizontal axis and absorbance (at 490 nm) as the vertical axis.
2. Draw a line of best fit for your data points that goes through the origin (because absorbance must be zero when Fe3+ concentration is zero).
3. You can use this graph to determine the iron content of your water samples.

The concentration of substances in drinking water is usually given as parts per million (ppm). 1ppm = 1 mg per l (for low concentrations at least).

To work out the concentration in ppm:

1. Multiply the figure in mol l-1l by the Atomic mass of Iron - 55.845
2. This gives the number of g l-1
3. Divide by 1000
4. This gives the value in mg l-1 = ppm

The concentrations in ppm Fe3+ of your standard solutions are:

|  |  |
| --- | --- |
| x10-5 mol l-1 | [Iron] ppm |
| 10 | 5.68 |
| 8 | 4.55 |
| 6 | 3.41 |
| 4 | 2.27 |
| 2 | 1.14 |

**Reporting results**

The final stage of any scientific investigation involves reporting the results. Scientists use a wide range of communication methods to report their results, including scientific papers, laboratory reports, blogs, videos, scientific posters, podcasts, PowerPoints, web pages, etc.

**Assessment task**

Once the pupil has agreed the format of the scientific communication with the teacher, the pupil should produce a report on their investigation containing the following key features:

* a clear statement of the aim of the investigation
* Background/ Underlying Chemistry including research from at least two different sources.
* a brief explanation of the techniques involved, e.g. colorimetry, and how the technique is used to determine the mass of iron in water samples.
* your experimental observations and results, including the calibration graph
* a comparison of the actual and the calculated ion concentration of known samples.
* reasons why the experimental results are different from the actual results (if they are different)
* an evaluation description of any ways in which the results could be improved
* a valid conclusion, based on the evidence in the report, which relates to the aim.
* A further investigation could be carried out to find the effect of iron concentration on the hardness of water.

**Investigation C:**

*“What is the concentration of phosphates in water?”*

**Introduction**

Water makes up around 66.7% of the surface of the Earth. It is essential for all forms of life on Earth. In Scotland, drinking water is available on tap but depending on the area and the supply of water there are many dissolved ions. Water is used for washing and has a foaming capacity depending on the hardness or softness of the water. Phosphate ions are often present in water samples.

**Background research**

The first stage of carrying out research in chemistry is to review what is already known about the topic of interest. Chemists use books, scientific papers, journals and the internet to carry out background research.

The pupils’ first task in the Researching Chemistry unit is to *independently* carry out background research into one of the investigations listed, which will be agreed with the teacher. In school, it is likely that background research will be carried out on the internet.

Once the pupil has completed their background research, the pupil must then complete the unit assessment tasks and store their research evidence in a safe place.

**Assessment tasks**

1. **Record at least two sources of information relevant to the Investigation** Sufficient detail should be given to allow someone else to find the sources easily. For a website, the URL shown here is perfectly adequate <http://www.biodieselfillingstations.co.uk/>.
2. **Write a brief summary** of the information of relevance contained in each of the sources you have identified.

**Focus questions**

C1 How do phosphates enter the water supply?

C2 What are the effects of phosphates on the water treatments plants?

C3 What is the limit in ppm of phosphates in the discharge from sewage treatment plants, according to the Environment Agency for Scotland.

C4 How can phosphate levels be controlled?

**Advice on using the internet for background research**

The web allows access a huge amount of information – don’t get side-tracked! Promising sites should be bookmarked so that they can be accessed at a later date. Tables, graphs and pictures can be copied into a folder. It is worthwhile spending a few moments considering which keywords may be the best to enter into the search engine. Ensure sites are credible. For more advice on effective web-based research see the Education Scotland resource on <http://www.educationscotland.gov.uk>

**Phosphate Background**

Phosphates may occur dissolved in water in two forms.

The first form—orthophosphate—are produced by natural processes such as decay and are found in sewage. This very useful form of phosphorus is the one used by plants and animals for growth.

The second form of phosphate— polyphosphate—are used for treating boiler waters and are found in many household detergents and soaps. In water, they change into the ortho form.

Organic phosphates are important in nature. Their occurrence may result from the breakdown of organic pesticides which contain phosphates. They may exist in solution, as particles, loose fragments or in the bodies of aquatic organisms.

Rainfall can cause varying amounts of phosphates to wash from farm soils into nearby waterways. Phosphate will stimulate the growth of phytoplankton and aquatic plants which provide food for fish. This may cause an increase in the fish population and improve the overall water quality. However, if an excess of phosphate enters the waterway, algae and aquatic plants will grow wildly, choke up the waterway and use up large amounts of oxygen. This condition is known as eutrophication or over-fertilization of receiving waters. This rapid growth of aquatic vegetation eventually dies and as it decays it uses up oxygen. This process in turn causes the death of aquatic life because of the lowering of dissolved oxygen levels.

Phosphates are not toxic to people or animals unless they are present in very high levels. Digestive problems could occur from extremely high levels of phosphate. Phosphate itself does not have notable adverse health effects. However, phosphate levels greater than 1.0 may interfere with coagulation in water treatment plants. As a result, organic particles that harbour microorganisms may not be completely removed before distribution.

The Environment Agency for England and Wales has suggested that discharge from sewage treatment plants should have no higher than 1-2 ppm phosphate.

<http://www.sserc.org.uk/index.php/chemistry/higher-chemistry-revised/1136-principles-to-production-h/3468-water-testing>

<http://www.water-research.net/index.php/phosphates>

<http://pubs.rsc.org/en/Content/ArticleLanding/1993/AN/AN9931800553#!divAbstract>

**Equipment and Materials Required**

|  |  |
| --- | --- |
| 0.00844 mol l-1 potassium antimonyl tartrate | Dissolve 1.3715 g K(SbO)C4H4O6.0.5H2O in 400 cm3 distilled water in a 500 cm3 volumetric flask and dilute to volume.) |
| 0.0162 mol l-1 | Dissolve 20 g (NH4)6Mo7O24.4H2O in 500 cm3 distilled water. |
| 0.1 mol l-1 Ascorbic acid | Dissolve 1.76 g ascorbic acid in 100 cm3 distilled water |
| Cuvettes |  |
| Colorimeter |  |

**Method**

*determination of phosphate concentration*

1. Make up your combined reagent: Mix the above reagents in the following proportions for 100 cm3 of the combined reagent: (Mix after the addition of each reagent)
   1. 50 cm3 2.5 mol l-1 H2SO4
   2. 5 cm3 potassium antimonyl tartrate solution
   3. 15 cm3 ammonium Molybdate solution
   4. 30 cm3 ascorbic acid solution.

Let all reagents reach room temperature fefore they are mixed and mix them in the order given. If turbidity forms in the combined reagent, shake and let stand for a frew minutes until turbidity disappears before proceeding. The reagent is stable for 4 hours.

1. Make a series of dilutions of your phosphate solution as follows: 0.5 cm3 of solution with 9.5 cm3 distilled water; 1 cm3 with 9 cm3 of distilled water and so on.
2. Place 5 cm3 of each dilution of the reference solution of your samples in a series of test tubes.
3. Add 1 cm3 of the combined reagent to each and mix thoroughly.
4. Leave the solutions for between 10 and 15 minutes.
5. Transfer some of the coloured solutions to cuvettes and read the absorbance at 590nm.
6. Plot the absorbance of your diluted reference solutions on a graph. This is your reference graph.
7. Read off the results for your water samples from the standard graph you obtained in 7.

**Reporting results**

The final stage of any scientific investigation involves reporting the results. Scientists use a wide range of communication methods to report their results, including scientific papers, laboratory reports, blogs, videos, scientific posters, podcasts, PowerPoints, web pages, etc.

**Assessment task**

Once the pupil has agreed the format of their scientific communication with the teacher, the pupil should produce a report on their investigation containing the following key features:

* a clear statement of the aim of the investigation
* Background/ Underlying Chemistry including research from at least two different sources.
* a brief explanation of the techniques involved e.g. colorimetry, and how this technique is used determine the phosphate concentrations in water.
* experimental observations and results, including the calibration graph
* a comparison of the actual and the calculated ion concentration in bottled mineral water.
* reasons why the experimental results are different from the actual results (if they are different)
* an evaluation description of any ways in which the results could be improved
* a valid conclusion, based on the evidence in the report, which relates to the aim.

# Investigation D

*“Making and testing Soap and Detergent“*

**Introduction**

Solid soaps are a widely used commercial product and are the sodium salts of fatty acids. Soaps can be made using naturally occurring fats by hydrolysing the large ester molecules using sodium hydroxide to produce the sodium salt of the fatty acid and glycerol. If the glycerol is not removed then the soap will also contain moisturising properties. Soaps are surfactants and reduce the surface tension of water.

Soaps need to be safe to use so chemical tests need to be done to ensure the soap will not cause damage to the user.

**Background research**

The first stage of carrying out research in chemistry is to review what is already known about the topic of interest. Chemists use books, scientific papers, journals and the internet to carry out background research.

The pupils’ first task in the Researching Chemistry unit is to *independently* carry out background research into one of the investigations listed, which will be agreed with the teacher. In school, it is likely that background research will be carried out on the internet.

Once the pupil has completed their background research, the pupil must then complete the unit assessment tasks and store their research evidence in a safe place.

**Assessment tasks**

1. **Record at least two sources of information relevant to your focus question**. Sufficient detail should be given to allow someone else to find the sources easily. For a website, the URL shown here is perfectly adequate <http://www.biodieselfillingstations.co.uk/>.
2. **Write a brief summary** of the information of relevance contained in each of the sources you have identified.

**Focus questions**

D1 What chemicals are the active ingredients in commercial soaps?

D2 What are the traditional methods and two processes used to make soap?

D3 How do soaps remove grease and oil? What is the action of soaps?

D4 What types of fats or oils can be used in making natural soap?

D5 How are synthetic soaps made?

D6 How are soaps scented?

D7 When making soap, How is it possible to test when the soap is neutralised?

D8 Give details of chemical equations and formulae for making soap.

D9 Why is it necessary to test soap?

D10 What tests will be carried out on the soap and how does this compare to commercial soaps.

D11 What affects the foaming capacity of soap ?

D12 How can the foaming capacity of soap be determined?

D13 What is the expected pH of the soap?

D14 How can water dispersal be tested?

**Advice on using the internet for background research**

The web allows access a huge amount of information – don’t get side-tracked! Promising sites should be bookmarked so that they can be accessed easily at a later date. Tables, graphs and pictures can be copied into a folder. It is worthwhile spending a few moments considering which keywords may be the best to enter into the search engine. Ensure sites are credible. For more advice on effective web-based research see the Education Scotland resource on <http://www.educationscotland.gov.uk>

**References**

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<http://www.rsc.org/learn-chemistry/resource/res00000985/soap-from-bacon?cmpid=CMP00001530>

<http://www.creative-science.org.uk/hollywood5.html>

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<http://www.creative-science.org.uk/hollywood5.html>

<http://www.elmhurst.edu/~chm/vchembook/554soap.html>

<http://www.nuffieldfoundation.org/practical-chemistry/making-soaps-and-detergents>

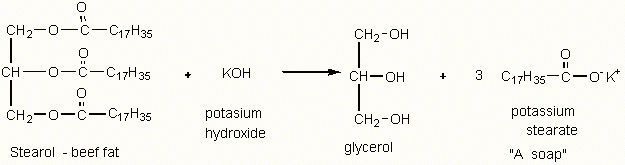
http://www.chymist.com/Soap%20and%20detergent.pdf

<http://www.sserc.org.uk>

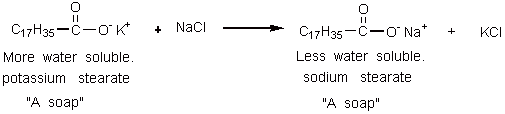
**Background to Soap Making**

**Soaps, Detergents and Cleaning**

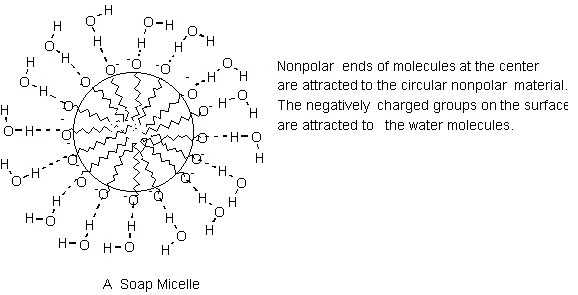
Fats are isolated from plants and animals. The properties such as solubility relate to their chemical structures. Fats are heated with a strong base to convert them into soaps. The fat you use to make soap, reacts with potassium hydroxide to produce a potassium soap, the potassium salt of the fatty acid. One typical animal fat, stearol, reacts with KOH to form potassium stearate, a soap. Most naturally occuring fats produce a mixture of different salts of fatty acids when they are converted to soap.



The potassium soap formed from your fat is converted to a sodium soap by replacing the potassium ions with sodium ions. A large excess of sodium chloride supplies the sodium ion. You may also notice that the potassium soap is softer than the sodium soap. In addition there is a difference in the way the sodium and potassium soaps behave in water.

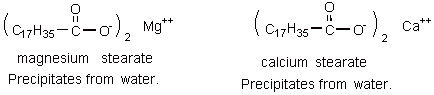


Both potassium and sodium soaps dissolve in water and are effective as cleaning agents. Each has a polar end to the molecule identified by the negative charge and an end that is primarily carbon and hydrogen. The polar end attracts polar water molecules. The other end, hydrocarbon end, attracts oils and other water insoluble materials like fat or grease. Water is a polar solvent and dissolves polar and ionic molecules. Gasoline is nonpolar and dissolves nonpolar materials such as fat or oil. A way to remember this behavior is the simple axiom; "Like dissolves like."

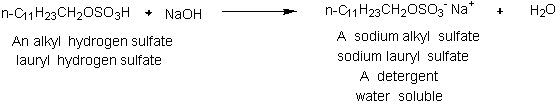


The nonpolar ends of the molecule associate with the fat, grime or dirt which is also nonpolar, The polar or ionic end of the molecule attracts the water molecules. A spherical structure with the polar portions of the molecule on the surface and the nonpolar parts of the molecule in the center is attracted to the water and carries the non-water-soluble material away with it. This spherical shaped unit of soap and grime is a **micelle**.

Magnesium and calcium salts of the same fatty acids that make up potassium and sodium soaps are not water soluble. When sodium or potassium soaps are put into water containing calcium and magnesium ions, the cloudyness, scum or curds consist of less soluble calcium and magnesium soaps. To achieve the same washing or cleaning action, more soap must be added.



There are other materials that also have cleaning capacity like soaps. The molecules of detergents also have polar and  nonpolar ends. They clean like soaps except that their calcium and magnesium salts are generally more soluble in water than their soap counterparts.  In recent years many different detergents have been introduced for use in cleaning. The conversion of one alkyl sulfate into a detergent is shown below.



**Planning the investigation**

The next stage in the investigation is to plan and carry out an appropriate experimental procedure that will allow the pupil to make a soap product. The pupil will then carry out a series of tests on the soap to ensure its safety for use. The tests can then be compared to the same test results from commercial soaps.

Whilst planning experimental work consider the following:

* What fat will be used and how will this be obtained?
* How will the sodium salt of the fatty acid be obtained?
* What are the desirable properties of the soap (moisturising, deep cleaning)
* What scent will the soap have and how will this scent be obtained.
* What tests will be carried out on the soap?
* Which chemicals will be required?
* What apparatus will be required?
* The hazards that might be involved and how you will minimise risk.

**Equipment and Materials Required**

|  |  |
| --- | --- |
| Test-tubes, 4 (with corks) | Castor oil, about 5 cm3 |
| Boiling tubes, 3 | Ethanol (IDA, Industrial Denatured Alcohol) (HIGHLY FLAMMABLE, HARMFUL), 5 cm3 |
| Test-tube rack | Sodium hydroxide solution, 5 mol l-1 (CORROSIVE), 10 cm3 |
| Dropping pipette | Sodium chloride, 10 g |
| Measuring cylinders (10 cm3), 2 | Concentrated sulfuric acid (CORROSIVE), 2 cm3 |
| Beakers (100 cm3), 2 | Purified water (distilled or deionised) |
| Beaker (250 cm3) | Glass rod |
| Spatula | Bunsen burner |
| Tripod stand | Heat resistant mat |
| Boiling water from kettle | Ice bath |
| Filter flask, funnel, filter paper and pump |  |

**Method**

**Castor oil** is used as a source of vegetable oils which, on reaction with warm concentrated alkali, form **soaps**. The vegetable oils in castor oil also contain hydroxy-groups (–OH) which will react readily with concentrated sulfuric acid, forming a long chain molecule with an ionic sulphonate group on the end. Such molecules behave as **detergents**.

*Experiment 1 – Making Soap*

1. Place about 2 cm3 of castor oil in a 100 cm3 beaker using a dropping pipette, followed by 5 cm3 of ethanol. Stir with a glass rod to mix.
2. Add 10 cm3 of sodium hydroxide solution..
3. Prepare a water bath containing near-boiling water from an electric kettle so that you can safely lower the small beaker into it without spillage. A 250 cm3 beaker may be used as the water bath. Do not use too much water, as the small beaker needs to be supported without risk of the water coming over the top.
4. Stir the mixture in the beaker with a glass rod for 5 minutes. If the water bath cools too much, you may need to renew with fresh boiling water.
5. Meanwhile in a boiling tube make a saturated solution of sodium chloride by shaking solid sodium chloride with 10 cm3 of water until no more will dissolve. Allow to settle.
6. After 5 minutes, add the saturated sodium chloride solution to the small beaker and stir.
7. Add desired scent at this point and mix thoroughly.
8. Cool the mixture by changing to a cold water bath (or an ice bath if available).
9. Soft, white lumps of the soap will gradually form in the mixture. Leave for a few minutes to improve the yield. During this time the soap may rise to the surface and form a soft crust on cooling.
10. Using a pump, with a fresh filter paper damped down in the funnel, filter off the soap, breaking up the crust with a glass rod if necessary
11. Allow the soap to drain on a paper towel – do not touch it with your fingers, as it may still contain sodium hydroxide.
12. Use a spatula to transfer a little of the soap to a test-tube, and add a few cm3 of purified water. Shake well! What happens? You have made a soap!

*Experiment 2 – Making Detergent*

1. Add 4 cm3 of concentrated sulfuric acid to a boiling tube (your teacher may do this for you).
2. Using the dropping pipette, add 2 cm3 of castor oil very carefully to the boiling tube, swirling gently to mix. Does the test-tube become hot?
3. Add 10 cm3 of cold water to a boiling tube (about 3 - 4 cm depth), then carefully pour the reaction mixture from the first tube into the water. The liquid may be very slow-flowing (viscous) and contain concentrated acid, so be careful and take your time over this.
4. Stir to remove the excess of acid into the water and then decant (pour off) the water down the sink, leaving a pinkish-grey sludge. Wash the product again with two more portions of water.
5. Use a spatula to transfer a small quantity of the product to a clean test-tube. Add a few cm3 of water, and shake well. What happens? You have made a detergent!

Castor oil - Castor oil is the best oil for making soap in the school laboratory, but reasonable results can be obtained from olive oil and rape-seed oil. For making detergent by sulfonation, the hydroxy-group on the carbon chain is essential, and castor oil is necessary.

 At least two electric kettles, strategically situated around the laboratory, will be needed to allow students to fill their water-baths (250 cm3 beakers or larger, or a small metal waterbath). These should be pre-heated to boiling at the start of the lesson so that a rapid re-heat is possible when students have to fill their water baths. It may be safer for students to take the electric kettles to their working places than to carry beakers of boiling water around the laboratory.

An ice-bath will help to speed up the precipitation of the soap produced. Again, two or more placed around the laboratory will probably be necessary.

The solid soap needs to be filtered off using a pump. Four water-pumps set up around the laboratory, with filter flasks (100 cm3) and either small Buchner or Hirsch funnels, will allow the class to filter their products efficiently. Filter papers should be available to size for each funnel. If water pumps are not available, other types of suction pump can be used.

**Testing Soap**

Cosmetic products must go through exhaustive testing prior to launch to ensure that they are effective and safe to use. Manufacturers employ extremely exacting on-going testing to ensure that every bottle of soap produced meets the product specification exactly.

Having produced a soap or detergent, it is now time to test the product against its competitors. It is advised to compare 3 commercial soaps: a cheap one, a mid-price one and an expensive one.

**Preparation**:

A soap solution can be made by dissolving soap flakes (or shavings from bar soap) in ethanol – use IDA (Industrial Denatured Alcohol) (HIGHTLY FLAMMABLE, HARMFUL) . Do not dilute with water.

**Equipment and Materials Required**

|  |  |
| --- | --- |
| 1 test tube rack | 4 Test tubes |
| 4 petri dishes | 1 10ml measuring cylinder |
| 1 250ml measuring cylinder | 4 large test tubes (with stoppers) |
| 2 1ml Pasteur pipettes | Universal indicator |
| Cooking oil | India ink |
| Distilled water |  |

**Test A - Determination of pH**:

1. Put one drop of the soap in a test-tube.

2. Fill the tube 3/4 full with distilled water.

3. Add two drops of Universal Indicator solution.

4. Find the pH value of the soap solution.

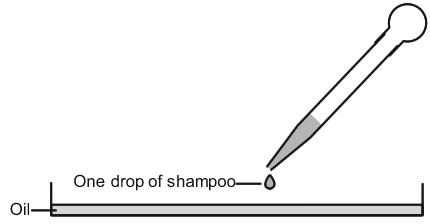
5. Record your results in the table.

**Health and Safety**

* Avoid any skin contact with the Universal indicator.
* Immediately wash any Universal indicator of fexposed skin.

*Information: Most soap are neutral or slightly acidic. Acidic solutions cause the cuticle (outer layer) of the hair to shrink and lay flatter on the shaft of the hair. Basic solutions cause the cuticle to swell and open up. Acidic solutions make the hair seem smoother. Basic solutions make hair seem frizzier.*

**Test B - Oil Test**:

1. Pour cooking oil into a petri dish to make a thin film over the base.

2. Place the dish on the dark paper.

3. Use a dropping pipette to drip one drop of soap onto the oil film (see diagram).

4. Wait until the drop has stopped spreading out, then measure the diameter of the faint iridescent halo formed around the soap drop. This will probably be between 3 and 6 cm in diameter. Write this down in the 'oil test' column of the table.

Test C and Test D are done together make sure you read Test D before you start.

**Test C - Shake Test - Determination of Foam Formation**:

1. Put approx 50 ml of the 1% soap solution into a 250ml graduated cylinder and record the volume.

2. Cover the cylinder with your hand and shake 10 times.

3. Record the total volume of the contents after shaking

4. Calculate the volume of the foam only and record your answer

5. Record the size of the bubbles (as small, medium, or large)

*Information: A good soap should have a foam volume of 100 ml or more in the shake test. Bubbles should be small. The smaller the bubbles the longer the foam will persist*.

**Test D - Foam Quality and Retention**:

1. Immediately after the Shake Test (Test C), Begin timing.

2. Record the volume of foam at 1-minute intervals for 5 minutes in the table.

*Information: In a good soap, foam retention should remain stable for at least 5 minutes.*

**Test E - Dirt Dispersion**:

1. Put two drops of soap in a large test tube

2. Add 10 ml of distilled water

3. Add 1 drop of India Ink

4. Stopper the test tube and shake it ten times.

5. Estimate the amount of ink in the foam (as None, Light, Moderate, or Heavy) and record

*Information: Soaps that cause the ink to concentrate in the foam are considered poor quality. The dirt should stay in the water portion. Dirt that stays in the foam will be difficult to rinse away. It will redeposit on the hair*.

**Reporting results**

The final stage of any scientific investigation involves reporting the results. Scientists use a wide range of communication methods to report their results, including scientific papers, laboratory reports, blogs, videos, scientific posters, podcasts, PowerPoints, web pages, etc.

**Assessment task**

Once the pupil has agreed the format of their scientific communication with the teacher, the pupil should produce a report on their investigation containing the following key features:

* a clear statement of the aim of your investigation
* Background/ Underlying Chemistry including research from at least two different sources.
* Chemical equations and formulae related to the reactions involved.
* a brief explanation of the chemistry techniques involved.
* your experimental observations and results.
* a brief explanation of how the soap was made
* How was the soap tested for safely
* The expected mass of soap and the percentage yield calculation.
* reasons why the experimental results are different from the actual results (if they are different)
* an evaluation description of any ways in which the results could be improved
* a valid conclusion, based on the evidence in the report, which relates to the aim.

**Further Investigations**

Further investigations could include:

Testing the foaming capacity of soap with hard and soft water.

How do the ions present affect the foaming capacity.

**Technician’s Guide**

**Investigation A**

Students determine the concentration of calcium and magnesium in water samples by means of two titrations. The sample solution is titrated against EDTA at 2 different pH values. One measures total hardness and the other calcium only. Subtracting this from the total hardness gives the concentration of magnesium.

**Equipment and Materials Required**

|  |  |
| --- | --- |
| 0.001 M EDTA solution | 0.5% Eriochrome Black T solution  (This has a very short shelf life – prepare fresh each day). |
| 1M NaOH | 0.5% Murexide |
| pH 10 ammonia buffer | Burette |
| Clamp and stand |  |

**Preparing the Reagents**

*EDTA*

A 0.001 M solution contains 0.372g per litre of EDTA 9Disodium salt) per litre.

As it can be difficult to measure this small amount accurately, it might be better to prepare the solution by serial dilution from a 0.1m solution (37.2g per litre)

*Ammonia Buffer (pH 10)*

1. Dissolve 17.5g of ammonium chloride (NH4Cl) in 142ml of concentrated ammonia (0.880).
2. Dilute to 250 cm3 with distilled water.

*Murexide indicator*

1. Grind 0.20 g of murexide to a fine powder with 20 g of potassium nitrate

Rather than making a solution of this, it is easier to just add a spatula tip of the solid to the water sample and swirl to dissolve.

*1M Sodium hydroxide*

1. Wearing gloves and goggles (BS EN166 3) add 4g of sodium hydroxide to about 70cm3 of distilled water and stir until dissolved.
2. Dilute to 100 cm3.

*Eriochrome Black T preparation*

1. Put on gloves and protective eyewear and weigh out approximately 0.5 g of solid Eriochrome Black T, (EBT) on a balance and transfer it to a small beaker or flask. Add about 50 cm3 of 95 percent ethyl alcohol and swirl the mixture until the EBT has fully dissolved.
2. Weigh out 4.5 g of hydroxylamine hydrochloride on a balance and transfer it to the beaker or flask containing the EBT. Swirl until the hydroxylamine hydrochloride has fully dissolved.
3. Transfer the solution containing the EBT and hydroxylamine hydrochloride to a 100 cm3 graduated cylinder. Add enough 95 percent ethyl alcohol to bring the total volume to exactly 100 cm3.
4. Transfer the EBT solution from the 100-mL graduated cylinder to a dropper bottle and label the bottle "0.5% Eriochrome Black T in Ethanol."

Tips & Warnings

* EBT indicator solutions typically exhibit very short shelf lives. Always prepare a fresh EBT solution when performing complexometric titrations.
* Hydroxylamine hydrochloride is highly toxic and corrosive to skin and mucous membranes. Avoid direct skin contact. Wear rubber gloves and protective eyewear at all times when handling this compound.
* Ethyl alcohol is flammable. Avoid working near open flames or other possible sources of ignition.

**Method**

*Experiment 1 – Total Hardness Determination*

1. Fill a 50 cm3 burette with 0.001 mol l‑1 EDTA solution, making sure the tip is full and free of air bubbles..
2. Add 50.00 cm3 of an unknown hard water solution into a 100 cm3 beaker.
3. Add 10 cm3 of Ammonia buffer to the beaker.
4. Add 0.5 cm3 of Eriochrome Black T indicator.
5. Titrate with the 0.001 mol l-1 EDTA until the colour changes from wine red to pure blue. Read burette to +/- 0.10 cm3.
6. Repeat the titration until the final volumes agree to +/- 0.20 cm3.

*Experiment 2 – Calcium Determination*

1. Fill a 50 cm3 burette with 0.001 mol l-1 EDTA solution, making sure the tip is full and free of air bubbles.
2. Add 50.00 cm3 of an unknown hard water solution into a 100 cm3 beaker.
3. Add 4 cm3 of 1.0 mol l-1 Sodium Hydroxide.
4. Add 0.5 cm3 of Murexide indicator.
5. Titrate with the 0.001 mol l-1 EDTA until the colour changes from salmon pink to orchid purple. Read burette to +/- 0.10 cm3.
6. Repeat the titration until the final volumes agree to +/- 0.20 cm3.

**Investigation B:**

**Equipment and Materials Required**

|  |  |
| --- | --- |
| ferric ammonium sulphate FeNH4(SO4)2•12H2O standard solutions: | 2, 4, 6, 8 and 10 × 10−5 M (see below for preparation) |
| 1 Mammonium thiocyanate solution | 1 mol l-1 sulphuric acid |
| distilled water | 100 cm3 beaker |
| 200 and 500 cm3 volumetric flasks | 5 cm3 pipette |
| 100 cm3 conical flask | Test tubes (5 for the references plus however many samples you are doing) |
| Colorimeter & cuvettes |  |

**Preparation of solutions**

*Preparation of 1 mol l-1 ammonium thiocyanate solution*

1. Weigh 38 g of solid ammonium thiocyanate into a 500 cm3 volumetric flask and make up to the mark with distilled water.

*Preparation of Fe3+ standard solutions*

NB: It may take several days to dissolve the Fe3+ salt used here, so carry out this preparation well in advance of the rest of the experiment.

1. Weigh out about 3.0 g of ferric ammonium sulphate (FeNH4(SO4)2•12H2O). Use a mortar and pestle to grind the salt to a fine powder. Accurately weigh 2.41 g of the powder into a 100 cm3 beaker and add 20 cm3 of concentrated sulphuric acid [corrosive]. Leave powder to soak in acid overnight.
2. The next day, carefully pour the acid/powder slurry into a 500 cm3 volumetric flask, rinsing the beaker into the flask a few times with water, then make up to the mark with distilled water. Let this solution stand for several days until the ferric ammonium sulphate powder has fully dissolved. If possible, insert a magnetic stirrer bar and stir the solution to speed up this dissolving process.
3. Use a pipette to transfer 20 cm3 of ferric ion solution to a 200 cm3 volumetric flask and make up to the mark with distilled water. This gives a solution with [Fe3+] = 0.001 M.
4. To prepare a 2 × 10−5 M standard solution pipette 10 cm3 of the 0.001 M solution into a 500 cm3 volumetric flask, add 10 cm3 of 1 M sulphuric acid, and then make up to the mark with distilled water.
5. Repeat this procedure in separate 500 cm3 volumetric flasks\*, pipetting in 20, 30, 40 and 50 cm3 of 0.001 M Fe3+ solution in turn, to obtain 4, 6, 8 and 10 × 10−5 M solutions respectively.

(\* if you do not have five 500 cm3 volumetric flasks you can use one flask to prepare each standard in turn. After preparing each standard, pour the solution into a labelled glass vessel which has a lid (eg: a glass bottle). Then rinse your 500 cm3 volumetric flask thoroughly with distilled water before using it to prepare your next standard solution.)

The recipe for the iron standard solution will give 5 litres of 0.001M stock solution.

Following the instructions will give 500 cm3 of each of the reference standards

**Quantities required**

For a group with three samples to examine carrying out the test three times on each sample,

10 cm3 of each standard iron solution

100 cm3 of ammonium thiocyanate

**Colorimetric analysis**

1. Accurately measure 2 cm3 of your sample solution into a clean, dry test tube\*.
2. Next, measure 2 cm3 of each Fe 3+ standard solution into separate test tubes (one standard per tube) in order of increasing concentration, beginning with the 2 × 10−5 M standard. It is a good idea to first rinse your pipette or measuring cylinder with a few cm3 of the 2 × 10−5 M standard.
3. NB: Make sure you label each boiling tube appropriately with the name of the sample or standard it contains.
4. Add 2 cm3 of 1 M ammonium thiocyanate solution to each iron solution in sequence, with 2 minutes between each addition\*\*. These additions must be carefully timed so that all samples react for the same period of time.
5. Mix the solutions by swirling. A stable red colour will appear over the next few minutes.
6. As near as possible to 15 minutes after adding thiocyanate\*\*\*, pour your samples into a cuvette and measure the absorbance at a wavelength of 490 nm for each coloured solution using your colorimeter. These measurements will be made in sequence − one sample every two minutes − reflecting the timing of the thiocyanate additions above. The measured absorbance of light is a direct measure of the intensity of the solution’s colour.

*\* You can, if you wish, simply add the solutions and mix in a cuvette.*

*\*\* If you have a colorimeter to hand, you should be able to do it faster than this. 1 sample per minute is easily achievable and with practice, one every 30s is quite possible.*

*\*\*\* As long as you are reasonably close to the time, all should be well. When left for another 5 minutes, the darkest of the reference samples only dropped from a reading of 0.44 to 0.42. An extra 10 minutes caused a further drop to 0.40.*

**Investigation C:**

**Equipment and Materials Required**

|  |  |
| --- | --- |
| 0.00844 mol l-1 potassium antimonyl tartrate | Dissolve 1.3715 g K(SbO)C4H4O6.0.5H2O in 400 cm3 distilled water in a 500 cm3 volumetric flask and dilute to volume.) |
| 0.0162 mol l-1 | Dissolve 20 g (NH4)6Mo7O24.4H2O in 500 cm3 distilled water. |
| 0.1 mol l-1 Ascorbic acid | Dissolve 1.76 g ascorbic acid in 100 cm3 distilled water |
| Cuvettes |  |
| Colorimeter |  |

**Preparation**

Make up your combined reagent: Mix the above reagents in the following proportions for 100 cm3 of the combined reagent: (Mix after the addition of each reagent)

1. 50 cm3 2.5 M H2SO4
2. 5 cm3 potassium antimonyl tartrate solution
3. 15 cm3 ammonium Molybdate solution
4. 30 cm3 ascorbic acid solution.

Let all reagents reach room temperature before they are mixed and mix them in the order given. If turbidity forms in the combined reagent, shake and let stand for a frew minutes until turbidity disappears before proceeding. The reagent is stable for 4 hours.

**Quantities required**

20cm3 of the combined reagent will allow a group to produce the reference graph and test 10 sample.

**Method**

*Experiment 1 – determination of phosphate concentration*

1. Make a series of dilutions of your phosphate solution as follows: 0.5 cm3 of solution with 9.5 cm3 distilled water; 1 cm3 with 9 cm3 of distilled water and so on.
2. Place 5 cm3 of each dilution of the reference solution of your samples in a series of test tubes.
3. Add 1 cm3 of the combined reagent to each and mix thoroughly.
4. Leave the solutions for between 10 and 15 minutes.
5. Transfer some of the coloured solutions to cuvettes and read the absorbance at 590nm.
6. Plot the absorbance of your diluted reference solutions on a graph. This is your reference graph.
7. Read off the results for your water samples from the standard graph you obtained in

**Equipment and Materials Required**

|  |  |
| --- | --- |
| Test-tubes, 4 (with corks) | Castor oil, about 5 cm3 |
| Boiling tubes, 3 | Ethanol (IDA, Industrial Denatured Alcohol) (HIGHLY FLAMMABLE, HARMFUL), 5 cm3 |
| Test-tube rack | Sodium hydroxide solution, 5 M (CORROSIVE), 10 cm3 |
| Dropping pipette | Sodium chloride, 10 g |
| Measuring cylinders (10 cm3), 2 | Concentrated sulfuric acid (CORROSIVE), 2 cm3 |
| Beakers (100 cm3), 2 | Purified water (distilled or deionised) |
| Beaker (250 cm3) | Glass rod |
| Spatula | Bunsen burner |
| Tripod stand | Heat resistant mat |
| Boiling water from kettle | Ice bath |
| Filter flask, funnel, filter paper and pump |  |

**Method**

*Experiment 1 – Making Soap*

1. Place about 2 cm3 of castor oil in a 100 cm3 beaker using a dropping pipette, followed by 5 cm3 of ethanol. Stir with a glass rod to mix.
2. Add 10 cm3 of sodium hydroxide solution..
3. Prepare a water bath containing near-boiling water from an electric kettle so that you can safely lower the small beaker into it without spillage. A 250 cm3 beaker may be used as the water bath. Do not use too much water, as the small beaker needs to be supported without risk of the water coming over the top.
4. Stir the mixture in the beaker with a glass rod for 5 minutes. If the water bath cools too much, you may need to renew with fresh boiling water.
5. Meanwhile in a boiling tube make a saturated solution of sodium chloride by shaking solid sodium chloride with 10 cm3 of water until no more will dissolve. Allow to settle.
6. After 5 minutes, add the saturated sodium chloride solution to the small beaker and stir.
7. Add desired scent at this point and mix thoroughly.
8. Cool the mixture by changing to a cold water bath (or an ice bath if available).
9. Soft, white lumps of the soap will gradually form in the mixture. Leave for a few minutes to improve the yield. During this time the soap may rise to the surface and form a soft crust on cooling.
10. Using a pump, with a fresh filter paper damped down in the funnel, filter off the soap, breaking up the crust with a glass rod if necessary
11. Allow the soap to drain on a paper towel – do not touch it with your fingers, as it may still contain sodium hydroxide.
12. Use a spatula to transfer a little of the soap to a test-tube, and add a few cm3 of purified water. Shake well! What happens? You have made a soap!

**Quantities needed**

Each group will need

2cm3 of castor (or other) oil,

10 cm3 ethanol,

10 cm3 5M sodium hydroxide and

10cm3 of saturated sodium chloride solution (or 4g of solid sodium chloride to dissolve into 10cm3 of water)

*Experiment 2 – Making Detergent*

1. Add 4 cm3 of concentrated sulfuric acid to a boiling tube (your teacher may do this for you).
2. Using the dropping pipette, add 2 cm3 of castor oil very carefully to the boiling tube, swirling gently to mix. Does the test-tube become hot?
3. Add 10 cm3 of cold water to a boiling tube (about 3 - 4 cm depth), then carefully pour the reaction mixture from the first tube into the water. The liquid may be very slow-flowing (viscous) and contain concentrated acid, so be careful and take your time over this.
4. Stir to remove the excess of acid into the water and then decant (pour off) the water down the sink, leaving a pinkish-grey sludge. Wash the product again with two more portions of water.
5. Use a spatula to transfer a small quantity of the product to a clean test-tube. Add a few cm3 of water, and shake well. What happens? You have made a detergent!

**Quantities needed**

Each group will need

2 cm3 of castor (or other) oil,

4 cm3 sulphuric acid (concentrated)

Castor oil is the best oil for making soap in the school laboratory, but reasonable results can be obtained from olive oil and rape-seed oil. For making detergent by sulphonation, the hydroxy-group on the carbon chain is essential, and so castor oil is necessary.

 At least two electric kettles, strategically situated around the laboratory, will be needed to allow students to fill their water-baths (250 cm3 beakers or larger, or a small metal waterbath). These should be pre-heated to boiling at the start of the lesson so that a rapid re-heat is possible when students have to fill their water baths. It may be safer for students to take the electric kettles to their working places than to carry beakers of boiling water around the laboratory.

An ice-bath will help to speed up the precipitation of the soap produced. Again, two or more placed around the laboratory will probably be necessary.

The solid soap needs to be filtered off using a pump. Four water-pumps set up around the laboratory, with filter flasks (100 cm3) and either small Buchner or Hirsch funnels, will allow the class to filter their products efficiently. Filter papers should be available to size for each funnel. If water pumps are not available, other types of suction pump can be used.

Testing soap/detergents

**Equipment and Materials Required**

|  |  |
| --- | --- |
| 1 test tube rack | 4 Test tubes |
| 4 petri dishes | 1 10 cm3 measuring cylinder |
| 1 250 cm3 measuring cylinder | 4 large test tubes (with stoppers) |
| 2 1 cm3 Pasteur pipettes | Universal indicator |
| Cooking oil | India ink |
| Distilled water | Ethanol (IDA) |

**Preparation**

Procedure: Prepare a 1% solution of your soap. Mix 2cm3 of soap with 200 ml of distilled water. Put the soap in the beaker first and add the water slowly. Swirl the beaker to mix the soap and water. Don’t shake it. You don’t want to create foam.

A soap solution can also be made by dissolving soap flakes (or shavings from bar soap) in ethanol – use IDA (Industrial Denatured Alcohol) (HIGHTLY FLAMMABLE, HARMFUL)

**Quantities needed**

In the case of universal indicator, oil and ink, only a few drops are required. Students can prepare their own solutions of soap.

**Method**

**Test A - Determination of pH**:

1. Put one drop of the soap in a test-tube.

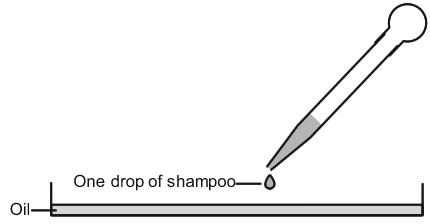
2. Fill the tube 3/4 full with distilled water.

3. Add two drops of Universal Indicator solution.

4. Find the pH value of the soap solution.

5. Record your results in the table.

**Test B - Oil Test**:

1. Pour cooking oil into a petri dish to make a thin film over the base.

2. Place the dish on the dark paper.

3. Use a dropping pipette to drip one drop of soap onto the oil film (see diagram).

4. Wait until the drop has stopped spreading out, then measure the diameter of the faint iridescent halo formed around the soap drop. This will probably be between 3 and 6 cm in diameter. Write this down in the 'oil test' column of the table.

Test C and Test D are done together make sure you read Test D before you start.

**Test C - Shake Test - Determination of Foam Formation**:

1. Put approx 50 ml of the 1% soap solution into a 250ml graduated cylinder and record the volume.

2. Cover the cylinder with your hand and shake 10 times.

3. Record the total volume of the contents after shaking

4. Calculate the volume of the foam only and record your answer

5. Record the size of the bubbles (as small, medium, or large)

**Test D - Foam Quality and Retention**:

1. Immediately after the Shake Test (Test C), Begin timing.

2. Record the volume of foam at 1-minute intervals for 5 minutes in the table.

**Test E - Dirt Dispersion**:

1. Put two drops of soap in a large test tube

2. Add 10 ml of distilled water

3. Add 1 drop of India Ink

4. Stopper the test tube and shake it ten times.

5. Estimate the amount of ink in the foam (as None, Light, Moderate, or Heavy) and record

Risk Assessment: (based on HSE 5 Step Risk Assessment

|  |  |
| --- | --- |
| Activity assessed | Water tests |
| *Date of assessment* | July 2014 |
| *Date of review (****Step 5****)* |  |
| *School* |  |
| *Department* |  |

| Step 1 | Step 2 | Step 3 | | Step 4 | | |
| --- | --- | --- | --- | --- | --- | --- |
| *List Significant hazards here:* | *Who might be harmed and how?* | *What are you already doing?* | *What further action is needed?* | *Action by whom?* | *Action by when?* | *Done* |
| **Calcium / Magnesium** |  |  |  |  |  |  |
| EDTA is a skin, eye and respiratory irritant | Technician by inhaling powder preparing solutions | Wear gloves and eye protection. Avoid raising dust. |  |  |  |  |
| Sodium hydroxide is corrosive  1M sodium hydroxide solution is corrosive | Technician preparing solutions  Technician, teacher or pupils by splashes | Wear gloves and goggles (BS EN166 3).  Wear goggles (BS EN166 3). |  |  |  |  |
| Ammonia .880 is corrosive and the fumes are toxic (Cat 3)  The ammonia buffer is corrosive and gives off toxic fumes (Cat 3) | Technician preparing buffer solution.  Technician, teacher or pupils by splashes or inhaling fumes | Wear gloves and goggles (BS EN166 3). Handle in a fume cupboard  Wear goggles (BS EN166 3). Work in a well-ventilated ares and keep lid off bottle for as short a time as possible. |  |  |  |  |
| Eriochrome black T is an eye irritant  Ethanol is flammable  Hydroxylamine hydrochloride is harmful by ingestions/skin contact, a skin/eye irritant, a skin sensitiser a category 2 carcinogen and can damage organs on repeated exposure.  Eriochrome Black T indicator solution is a skin sensitiser and a category 2 carcinogen. | Technician by inhalation preparing solution.  Technician preparing solution.  Technician preparing solution.  Technician, teacher or pupils by splashes | Wear eye protection. Avoid raising dust.  Keep away from sources of ignition. Wear gloves and eye protection.  Wear gloves and goggles (BS EN166 3).  Wear gloves and goggles (BS EN166 3). |  |  |  |  |
| Murexide indicator (ammonium purpurate) has no significant hazard  The reaction mixture is of no significant hazard. |  |  |  |  |  |  |
| **Iron** |  |  |  |  |  |  |
| Sulphuric acid is corrosive  1M sulphuric acid is irritant to skin and eyes | Technician preparing solutions, by splashing.  Technician, teacher, pupils preparing diluted reference solutions, by splashing. | Wear gloves and goggles (BS EN166 3)  Wear gloves and eye protection. |  |  |  |  |
| Iron III ammonium sulphate is a skin/eye irritant  The reference iron solutions are of no significant hazard | Technician preparing solutions, by splashing. | Wear gloves and eye protection. |  |  |  |  |
| Ammonium thiocyanate is harmful by ingestion, skin contact and inhalation.  1M Ammonium thiocyanate solution is of no significant hazard. | Technician preparing solutions | Avoid raising dust. Wear gloves and eye protection. |  |  |  |  |
| **Phosphate** |  |  |  |  |  |  |
| Potassium antimonyl tartrate is harmful by ingestion and skin contact.  0.00844M solution has no significant hazard. | Technician preparing solutions. | Wear gloves and eye protection |  |  |  |  |
| Sulphuric acid is corrosive  2,5M sulphuric acid is also corrosive | Technician preparing solutions, by splashing.  Technician, teacher, pupils preparing diluted reference solutions, by splashing. | Wear gloves and goggles (BS EN166 3)  Wear gloves and goggles (BS EN166 3) |  |  |  |  |
| Sodium phosphate is corrosive.  Potassium phosphate is a skin/eye irritant  Phosphate solutions are of no significant hazard | Technician preparing solutions.  Technician preparing solutions. | Avoid raising dust. Wear gloves and goggles (BS EN166 3)  Avoid raising dust. Wear gloves and eye protection |  |  |  |  |
| The combined reagent is a skin/eye irritant | Technician, teacher, pupils by splashing. | Wear gloves and eye protection. |  |  |  |  |
| Ammonium molybdate and the reaction mixture are of no significant hazard |  |  |  |  |  |  |
| **Soap / detergent** |  |  |  |  |  |  |
| Sodium hydroxide is corrosive  5M sodium hydroxide solution is also corrosive | Technician preparing solutions  Technician, teacher or pupils by splashes | Wear gloves and goggles (BS EN166 3).  Wear gloves and goggles (BS EN166 3). Take great care to keep away from eyes. |  |  |  |  |
| Ethanol is flammable | Technician preparing solution. | Keep away from sources of ignition. Wear gloves and eye protection. |  |  |  |  |
| Sodium chloride and castor oil have no significant hazards |  |  |  |  |  |  |
| Sulphuric acid is corrosive | Technician, teacher, pupils by splashing. | Wear gloves and goggles (BS EN166 3) |  |  |  |  |
|  |  |  |  |  |  |  |

|  |
| --- |
| **Description of activity:**  **Calcium / magnesium**  Water samples are titrated against EDTA solution. Using murexide and eriochrome black T indicators. The solution is made alkaline by pH 10 ammonia buffer for the total hardness or sodium hydroxide for the magnesium.  **iron**  Solutions of known iron concentration as well as unknowns are reacted with ammonium thiocyanate. This results in a red compound. The reference samples are read in a colorimeter and a reference curve is constructed which is used to determine the iron concentration in the water samples.  **Phosphate**  Samples of water and phosphates of known concentration are incubated with the reagent. The presence of phosphate produces a blue complex. This is determined by colorimeter and the results for water compared with the reference curve.  **Soap / detergent**  Students prepare and test samples of soap and detergents |

|  |
| --- |
| **Additional comments:**  All solutions can be washed to waste with copious quantities of cold running water. |