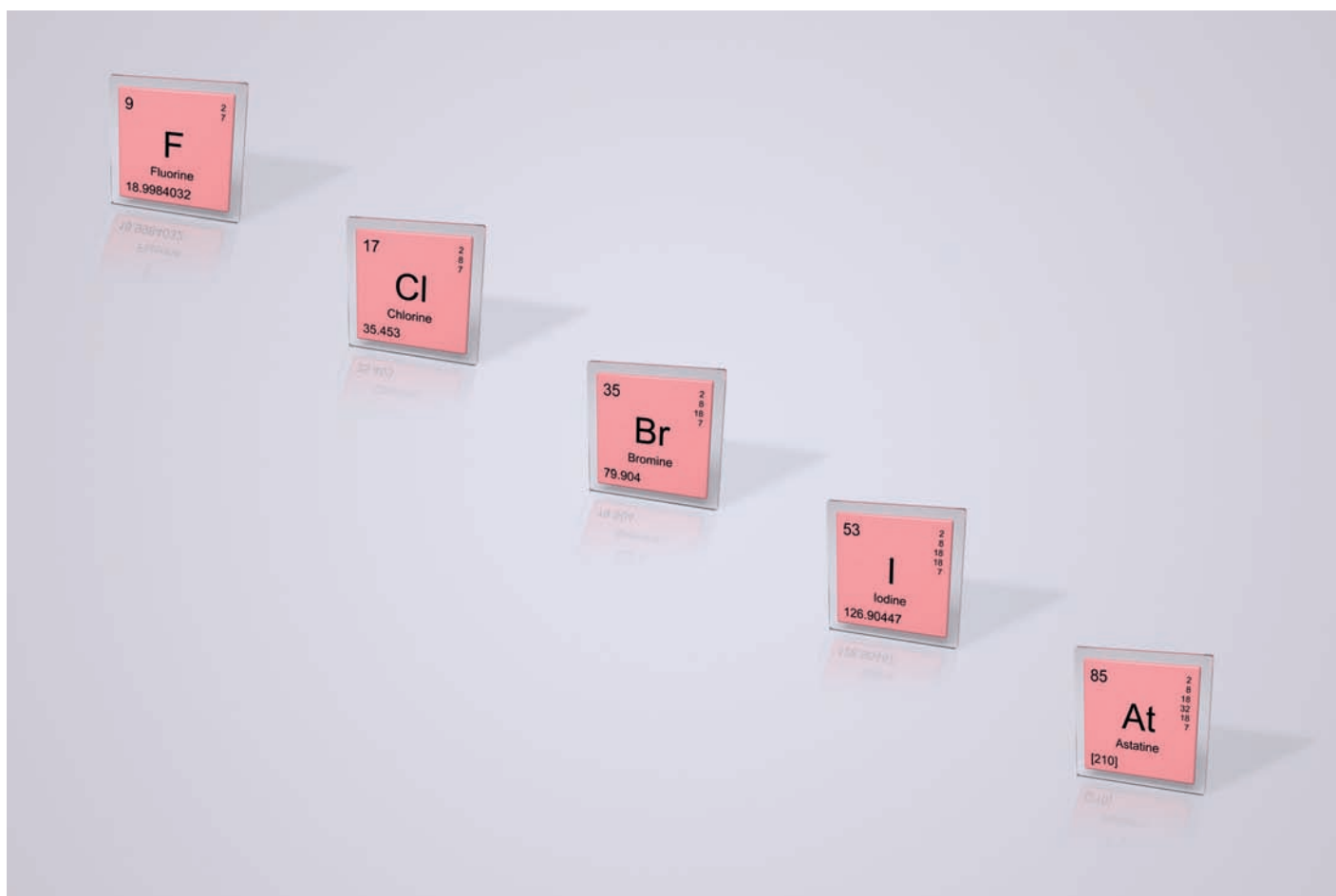


Halogens

Teacher Notes



Halogens is funded as part of the Reach and Teach educational programme supported by the Wolfson Foundation



THE
WOLFSON
FOUNDATION

Halogens

Why focus on G&T and higher achievers?

Within the education system every child has the right to develop their learning so as to maximise their potential.

These exercises are designed to give students enthuse and enrich activities that although related to the curriculum are in fact taking the learning experience to the next level whilst also showing chemistry in a familiar context. This has been found to be a successful model for not only improving learning but also for raising levels of motivation. Higher achieving students can find the restraints of the standard curriculum to be demotivating leading to underachievement.

The different activities are designed to improve a number of skills including practical work/dexterity, thinking/analysis skills, literacy, research activities, use of models and teamwork. Students should also gain confidence through the activities and improve the ability to express themselves.

Some of the activities would appear to be complex for KS3, however at this stage in their learning high achieving students are open to new concepts and are ready to explore issues without pre-conceptions. They are keen to link ideas and develop concepts and understanding. It can prove to be an uplifting experience.

Introduction

The halogens, group 7, are an important group of elements both chemically and socially. They are unusual in that they are coloured and diatomic. They also have an order of reactivity that is the opposite of the metals in groups 1 & 2. In fact they are all very reactive, fluorine being the most reactive non-metal.

Socially they have been used for providing clean drinking water, antiseptics, bleach, insecticides, polymers and much more. They are involved in many aspects of our daily life and are in some cases essential to our diet. It is therefore surprising that in their elemental form they can prove extremely toxic.

This programme is designed to develop students understanding of the halogens and the halides. It investigates both the source and uses of the materials as well as the chemistry involved. Working through the activities will also develop thinking and research skills.

Topic	Type of activity	Summary	Timing (mins)	KS3	KS4	KS5	Page
Silver halides	Practical	The identification of halogens by the formation of silver halides. Includes the uses of silver halides.	30	√	√	√	11
Reactions of aqueous solutions of halogens	Practical	Displacement reactions of the halides. Produces an order of reactivity Also looks at the colour of halogens in aqueous and non	30	√	√	√	13

		polar solvents.					
Halogens reaction with iron	Demonstration	Illustrates the reaction between metals and halogens. Reinforces the order of reactivity.	20	√	√	√	18
Acidic and bleaching properties	Practical	Investigating the products of aqueous halogens.	15	√	√	√	22
Which bleach is the most cost effective	Practical Investigation	An indirect titration that assesses the bleaching power of different commercial bleaches.	50+		√	√	23
Extracting iodine from seaweed	Practical	An experiment illustrating how halogens can be extracted from seaweed which has concentrated the substance from sea water.	45		√	√	26
Extension question	Summative assessment	Thought provoking questions to assess knowledge and understanding.	20		√	√	29

The first activity introduces halogens and halides by investigating their identification using silver nitrate solution and observing the insoluble silver halides of different colours. It also links to the ionic equations involved and a use of the process, namely photography. The difference between the halogens is further explored in the second activity where an order of reactivity is established by observing a range of displacement reactions. Solubility in aqueous and non-polar organic solvents, and the colours of the halogens are also observed. The demonstration of the reaction of the halogens with metals to form metal halides reinforces, in a dramatic way, the order of reactivity. It also emphasises the colours.

Activity 4 looks at two basic properties of the aqueous solutions of halogens, the acidic and bleaching properties. Although relatively simple this is a thought provoking experiment particularly when considering the charge on such products, for example the chlorine in chloric(I) acid. The salts of the chloric(I) acid, commonly referred to as hypochlorites, are studied in this applied activity where they investigate the cost effectiveness of commercial bleach samples by indirect titration.

The sources of the halogens are explored in the next activity where iodine is extracted from sea weed. Sea water contains many of the halides which can be concentrated in various marine plants. This experiment uses laminaria which is a good source of iodine.

The final exercise is a challenging summative assessment based around the chemistry of iodine and encompassing much of the chemistry encountered in this range of activities. It is designed for key stage 5 but could be attempted by year 11 students.

Aims and objectives

The aims and objectives of these activities are:

- Developing questioning skills through problem solving.
- Exploring the use of models to expand understanding
- Develop practical skills and dexterity.
- Promote independent learning.

Chemistry topics:

- Properties of the halogens.
- Properties of halides.
- Identification of halogens.
- Extraction from natural sources.
- Acid and bleaching properties.
- Displacement reactions.
- Indirect titration.

These exercises can be used with key stages 3, 4, and 5 as indicated on the *Possible Routes*.

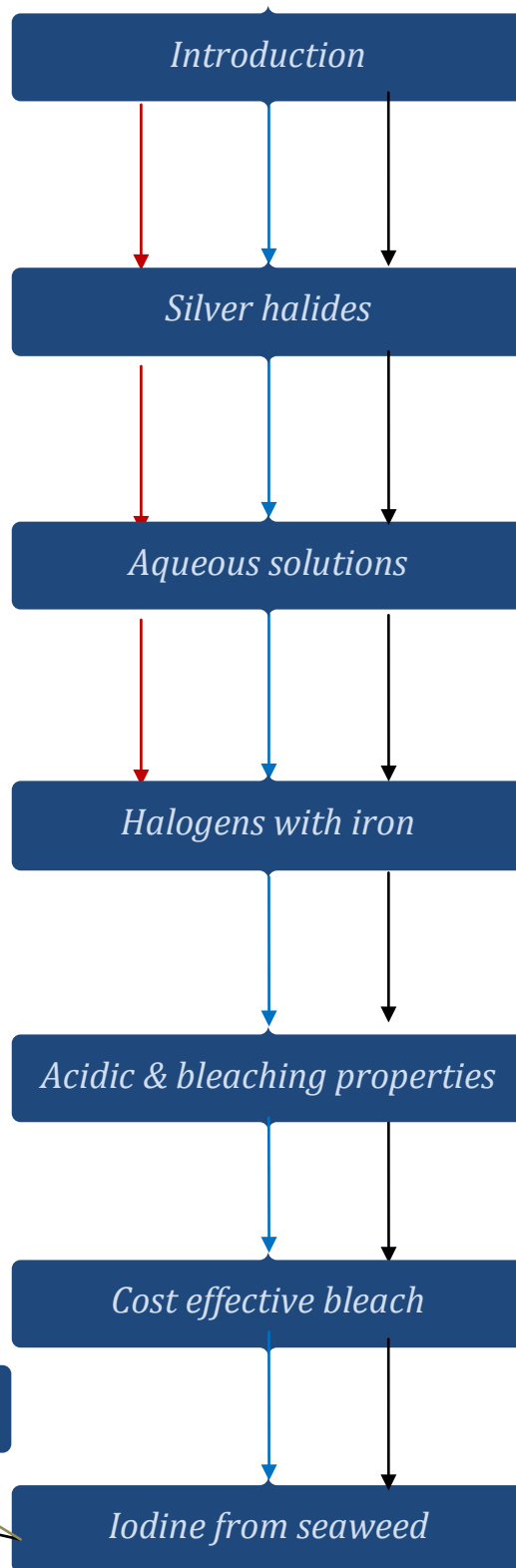
These activities have proved very successful with high achieving key stage 3 students who have followed the prescribed pathway and have been stimulated into further independent learning.

At key stage 4 this activity enhances understanding of key industrial processes.

As well as developing key concepts and understanding, these exercises provide a reinforcement and revision tool for a number of topics from the A level syllabus.

At all levels there is promotion of questioning skills, independent learning and research skills.

Possible routes



The introduction leads into a demonstration to illustrate the identification of halogens and their use in photography.

Moving forward exploring the variable reactivity of the halogens – displacement reactions. Introduce solubility in water and organic solvents and the related colour.

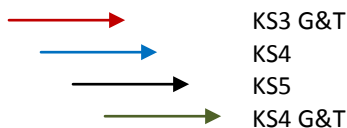
This activity reinforces the idea of a reactivity series by the comparison of the reactions of different halogens with iron. It also revisits the colour of halogens

A simple experiment that looks at the products formed from reacting chlorine with water. Introduces the variable oxidation states of chlorine.

A quantitative indirect titration exercise that explores the cost effectiveness of commercial bleach.

This activity explores the sources of halogens by undertaking the extraction of iodine from seaweed. Solubility is also explored.

Key



This extension questions is a demanding look at iodine. It is synoptic for this whole package.

Extension question

Iodine from seaweed

Halogens

The halogens form Group VII of the periodic table. They and their compounds have both a good and bad effect upon the world we live in. They are reactive substances that form diatomic molecules that are coloured and span the full range of solids to gases at room temperature. Although non-metals they still have an order of reactivity just like the metals. They readily react with metals to form salts.

The name halogen, initially first used in the nineteenth century, is derived from the Greek word meaning a 'salt producer' or 'salt former' as they are all found in the sea as salts.

Name	Symbol	Molecule	State (at room temp)	Colour
fluorine	F	F ₂	gas	pale yellow
chlorine	Cl	Cl ₂	gas	yellow green
bromine	Br	Br ₂	liquid	orange brown
iodine	I	I ₂	solid	grey vapour violet
astatine	At	Short lived radioactive isotope		

The general reactivity is due to the fact that they have seven electrons in their outer electron shell, an unpaired electron in the *p* orbital. The size of the atom also plays a part in the reactivity.

Symbol	Atomic number	Electron configuration
F	9	1s ² 2s ² 2p ⁵
Cl	17	1s ² 2s ² 2p ⁶ 3s ² 3p ⁵
Br	35	1s ² 2s ² 2p ⁶ 3s ² 3p ⁶ 4s ² 4p ⁵
I	53	1s ² 2s ² 2p ⁶ 3s ² 3p ⁶ 4s ² 4p ⁶ 5s ² 5p ⁵

Uses

The halogens have many uses within everyday life. Most of the uses have a very positive impact but there are one or two high profile developments that show a less positive side. Chlorine was used as a chemical weapon in the early twentieth century as well as a component of nerve gases. The halogens generally have been used to produce CFCs (chloro-fluoro-carbons) used in the past as refrigerants and propellants. These had a significant effect in removing ozone protection from UV radiation in the atmosphere.

However, without the simple use of chlorine in drinking water to kill bacteria many more people would have died from diseases such as cholera. They have also been used to produce pesticides, sterilising agents, polymers and solvents to name but a few.

Halogen	Uses
fluorine	Fluoride in toothpaste Manufacture of PTFE, the non-stick coating on pans Etching glass using hydrofluoric acid CFC refrigerants and propellants
chlorine	Biocide in water Bleach Manufacture of: PVC Pesticides/weedkillers Disinfectants Solvents
bromine	Manufacture of: Medicines Pesticides Photographic films Compounds added to petrol
iodine	Antiseptics Food supplements

All the isotopes of astatine are radioactive, and so this element will not be considered further here.

Do not forget that one of the common essential compounds of our diet is the result of a metal reacting with a halogen namely sodium chloride – salt.

Appearance

Fluorine is a toxic pale yellow gas, chlorine is a toxic pale green gas, bromine is a toxic and corrosive brown volatile liquid, and iodine is a shiny black solid which easily sublimates to form a violet vapour on heating.

General Reactivity

The elements of Group 7, the Halogens, are a very similar set of non-metals. They all exist as diatomic molecules, X_2 , and oxidise metals to form halides. The halogen oxides are acidic, and the hydrides are covalent. Fluorine is the most electronegative element of all. Generally, electronegativity and oxidising ability decrease on descending the Group. The result of this decreasing electronegativity is increased covalent character in the compounds, so that AlF_3 is ionic whereas $AlCl_3$ is covalent.

Fluorine shows some anomalies because of the small size of its atom and ion. This allows several F atoms to pack around a different central atom, as in AlF_6^{3-} compared with $AlCl_4^-$. The F-F bond is also unexpectedly weak because the small size of the F atom brings the lone pairs closer together than in other halogens, and repulsion weakens the bond.

Occurrence and Extraction

The halogens are too reactive to occur free in nature. Fluorine is mined as fluorspar, calcium fluoride and cryolite. It is extracted by electrolysis as no oxidant will oxidise fluorides to fluorine. Chlorine is also found in minerals such as rock-salt, and huge quantities of chloride ions occur in seawater, inland lakes and subterranean brine wells. It is obtained by the electrolysis of molten sodium chloride or brine. Bromine is also found as the bromide ion in seawater, and in larger quantities in brine wells, from which it is extracted. Iodine is mined as sodium iodate(V), $NaIO_3$, which is present in Chile saltpetre. It is obtained by reaction with sodium hydrogensulfite.

Physical Properties

At room temperature all the halogens exist as diatomic molecules. The melting points, boiling points, atomic radii and ionic radii all increase on descending the Group. The shapes of the covalent molecules and ions are readily explained by VSEPR (valence shell electron pair repulsion) theory and these compounds are often used to illustrate the theory. Fluorine is never surrounded by more than 8 electrons in its outer shell, whereas the other halogens may be surrounded by up to 14.

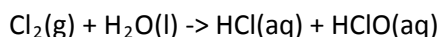
Chemical Properties

Group 7, or the halogens, are the most non-metallic elements, with fluorine being the most non-metallic element of all - so much so that it reacts with almost anything and is a difficult element to work with. Its compounds on the other hand are some of the most stable and difficult to decompose.

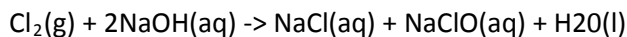
The most characteristic chemical feature of the halogens is their ability to oxidise. Fluorine has the strongest oxidising ability, so other elements which combine with fluorine have their highest possible oxidation number. Fluorine is such a strong oxidising agent that it must be prepared by electrolysis. Chlorine is the next strongest oxidising agent, but it can be prepared by chemical oxidation. Most elements react directly with chlorine, bromine and iodine, with decreasing reactivity going down the Group, but often the reaction must be activated by heat or UV light. The oxidation of thiosulfate ions, $S_2O_3^{2-}$, by the halogens is quantitative. This means that oxidising agents can be estimated accurately; the oxidising agent is reacted with excess I^- ions, and the liberated I_2 titrated with standard thiosulfate solution. The end point

is detected with starch as indicator, which forms a dark blue complex with iodine. Chlorine, bromine and iodine disproportionate in the presence of water and alkalis.

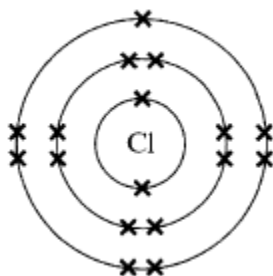
Consider chlorine, it is a green poisonous gas which dissolves in water to form an acid solution,



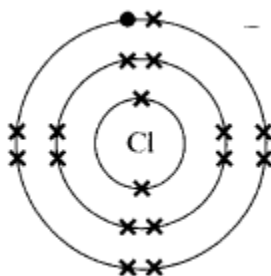
and it readily reacts with alkalis to form salts. With sodium hydroxide,



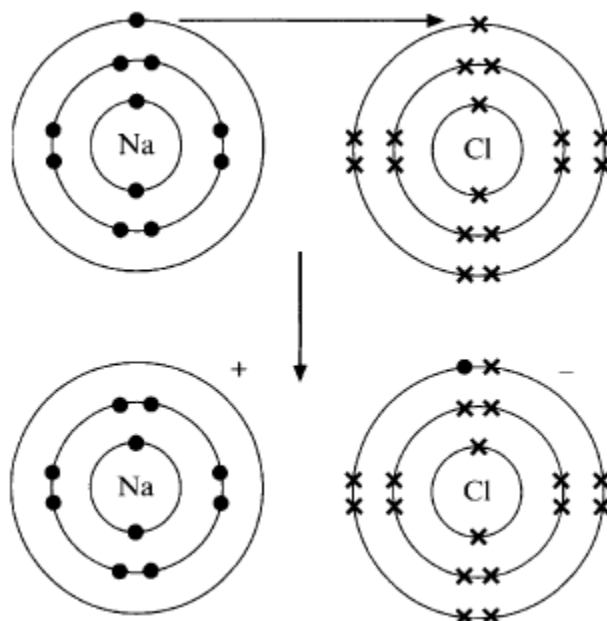
All the halogens have seven electrons in their outermost energy level. Let us take chlorine as our example. Its ground state configuration is $1s^2 2s^2 2p^6 3s^2 3p^5$. We can see this in below.



If a chlorine atom can manage to get hold of an extra electron from somewhere, its configuration will be $1s^2 2s^2 2p^6 3s^2 3p^6$, which is the same as the configuration of argon, a noble gas. So what's the difference? An argon atom has 18 electrons and 18 protons, so it is neutral; the chlorine atom did have 17 electrons, but it has gained one so it also has 18 electrons, but it still has 17 protons. The chlorine now has one more negative charge than positive charges, so it is no longer neutral; it has an overall negative charge of -1. Atoms are neutral; this new particle is negatively charged, so instead of calling it a chlorine atom, we call it a chloride ion as below.



Now if a sodium atom and a chlorine atom were to collide with each other. The sodium can get rid of one electron by giving it to the chlorine atom: both now have got the desired 'noble gas' configuration. This type of diagram is usually called a 'dot and cross' diagram because the electrons from the different atoms are shown as dots and crosses (although, of course, there is no real difference between the electrons of different atoms).



Just look at what is produced: one positive sodium ion and one negative chloride ion. In any real sample of sodium and chlorine, of course, there would be billions of ions, but for every one sodium ion there would be one chloride ion. They would attract each other because they have opposite charges to form a compound, sodium chloride. Its formula is NaCl because: the ratio of sodium ions to chloride ions is 1:1.

Oxides and Oxoacids

There are no fluorine oxides as F is more electronegative than O. Chlorine, bromine and iodine each form several oxides which are thermally unstable, such as chlorine dioxide ClO_2 . The only fluorine oxoacid, HOF, is unstable at room temperature, but there are many oxoacids of the other halogens. The best known salts of these are; hypochlorite, chlorate(I) ClO^- , chlorite, chlorate(III) ClO_2^- , hypochlorate, chlorate(V) ClO_3^- , perchlorate, chlorate(VII) ClO_4^- . These are all powerful oxidising agents.

Halides

The halogens can combine with each other to form interhalogens and polyhalide ions.

Polyhalide ions have the general formula $[\text{Y-X-Y}]^-$. It is not possible for F to represent X in a polyhalide ion as it cannot expand its octet.

Hydrides

Hydrogen halides have the general formula HX. HF is a colourless liquid which boils at 19.5°C , and all the other hydrogen halides are colourless gases. HF is a liquid due to the extensive hydrogen bonding which occurs between molecules. All the hydrogen halides dissolve easily to give acidic solutions, the most widely used being hydrochloric acid, HCl. All except HF are typical acids; they liberate carbon dioxide from carbonates and form salts with basic oxides. HF is a weak acid because the H-F bond is very strong, and because hydrogen-bonding occurs between F^- and HF in solution.

Organic Compounds

The halogens form organic compounds which are best known for their industrial and environmental impact, such as PVC, DDT and TCP.

Oxidation States and Electron Affinities

Fluorine in all its compounds has an assigned oxidation number of -1, as it is the most electronegative of all the elements. The other halogens show a wide range of oxidation numbers, and the redox chemistry of these halogens is important. The oxidation numbers most commonly shown are odd; there are few compounds with even oxidation numbers and they are often thermally unstable. Chlorine is the third most electronegative element after F and O. The halide ions are readily formed by accepting one electron, as this completes an octet of valence electrons. The electron affinity decreases on descending the Group.

Reactivity

Non-metals also vary in reactivity. Unlike the metals, the order of reactivity for these is the same as the order of the elements in the group:

Fluorine

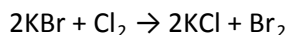
Chlorine

Bromine

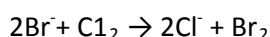
Iodine

Astatine

Thus, for example, if you pass chlorine gas through potassium bromide solution, the solution will go reddish-brown because the chlorine displaces the bromine from the potassium bromide:

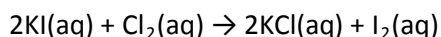


or



The higher element in the reactivity series displaces the lower one from a solution of its salt.

If you pass chlorine gas into potassium iodide solution, the solution goes brown because the chlorine displaces iodine from the potassium iodide. Iodine in KI has an oxidation number of -1 and this rises to 0 in the elemental iodine, so iodine has been oxidised. The opposite occurs with chlorine.



Industrial information

The halogens are probably the most important group of the Periodic Table used in industry. Fluorine is widely used as an oxidising agent. HF is used to etch glass. Chlorine is used for chlorinating drinking water, and in many organochlorine compounds. Some of these, such as the insecticide DDT, are effective but environmentally damaging, and much controversy surrounds their use. Chlorine dioxide is used to bleach

wood pulp for paper making, as it gives a good whiteness without degrading the paper. Hypochlorites are used in domestic bleaches. Potassium chlorate(V) is used as an oxidant in fireworks and matches.

Activity 1: Silver halides

Teacher Notes

Insoluble silver halides form as precipitates when solutions of silver salts are added to solutions containing halide ions.

The silver chloride, bromide and iodide can be distinguished by their colours and their solubility in ammonia solution, providing tests for these halide ions in solution.

The use of silver halides in film photography is illustrated by the effect of light on the precipitates.

Lesson organisation

These reactions can be demonstrated or investigated as a class practical.

Reactions can be confined to the silver halides as part of an investigation of Group 7 chemistry. Using ammonia to distinguish between the silver halides is more appropriate at an advanced level although it is becoming more apparent in GCSE specifications.

Procedure

a Pour about 3 cm³ of each of the halide solutions into separate test-tubes.

b Add a few drops of silver nitrate solution to the test-tube containing potassium chloride solution. A white precipitate of silver chloride forms.

c Add a few drops of silver nitrate solution to potassium bromide solution. A cream or off-white coloured precipitate of silver bromide forms.

d Add a few drops of silver nitrate solution to potassium iodide solution. A yellow precipitate of silver iodide forms.

e Pour half the contents of the three test-tubes into another three labelled test-tubes.

f Place one set of three test-tubes in a cupboard and the other set in bright light, such as on a window sill, and leave for 5–10 mins. In bright light, the silver chloride darkens quickly, the silver bromide more slowly, and the silver iodide is not affected at all. Compare with the solutions kept in the dark.

g–j Use ammonia solution to distinguish between the silver halides.

g Prepare fresh samples of the silver halide precipitates as above, a–d.

h Slowly add an equal volume of *dilute* ammonia solution to the test-tube containing silver chloride using a teat pipette. Shake well after each addition to mix the contents. The precipitate dissolves, giving a colourless solution.

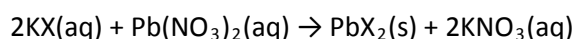
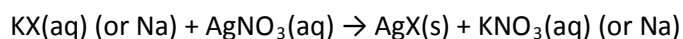
i Add an equal volume of *dilute* ammonia solution to the test-tube containing silver bromide. Shake to mix. The precipitate does not dissolve. Now add concentrated ammonia solution to almost fill the test-tube, stopper the tube and invert to mix. Most of the precipitate dissolves.

j Add an excess of *concentrated* ammonia solution to the test-tube containing silver iodide, stopper and invert to mix. The precipitate does not dissolve.

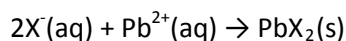
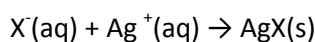
Teaching notes

Any spillages of silver or lead nitrate on the skin should be washed off with plenty of water. Silver nitrate causes black stains on the skin and clothing which wear off slowly.

These precipitation reactions can be represented by the following equations, where X = Cl, Br or I:



or the general ionic equations:



The silver chloride experiment can be modified to produce a photographic paper on which an image can be recorded. Here a simple extension is to filter off the freshly prepared silver chloride precipitate (covering the funnel to exclude light), and then opening the filter paper out onto a white tile and placing it in bright light. The silver chloride darkens quickly. Partly covering the precipitate on the paper will emphasise the effect of light.

The decomposition of silver chloride is an example of a photochemical reaction. Silver metal and chlorine atoms are produced.

The optional experiments involving ammonia to distinguish between the silver halides should be tried beforehand. This is because the result depends on the relative amounts of the precipitate and ammonia. Silver chloride and iodide are not usually a problem - the silver iodide loses some of its yellow colour. But the extent to which the silver bromide dissolves depends on the actual concentration of ammonia in the test-tube.

Technician notes

Apparatus and chemicals

Eye protection

Each working group requires:

Test-tube rack (see note 1)

Test-tubes, 6

Boiling tubes, 3

Test-tube holder (to fit a boiling tube)

Teat pipettes, 2

Beaker (250 cm³ or 500 cm³)

Bunsen burner

For optional part:

Test-tubes, 3

Teat pipettes, 2

Corks or rubber bungs to fit test-tubes, 3

About half a test-tube of each of the following solutions:

Potassium (or sodium) chloride solution about 0.1 mol dm³ (**Low Hazard**)

Potassium (or sodium) bromide solution about 0.1 mol dm³ (**Low Hazard**)

Potassium (or sodium) iodide solution about 0.1 mol dm³ (**Low Hazard**)

Silver nitrate solution, about 0.05 M (**Low Hazard but stains fingers and clothing** at this concentration, Danger to the environment*) about 1 cm³

For optional part, in a fume cupboard:

Dilute ammonia solution ~0.1 M (**Low Hazard**)

Concentrated ammonia solution ~8 M (**Corrosive, Danger to the environment**),

Teat pipettes

Corks or rubber bungs to fit test-tubes, 3

Technical notes

Potassium (or sodium) chloride solution (**Low Hazard**) Refer to CLEAPSS Hazcard 47B and Recipe Book sheet 68 or 82

Potassium (or sodium) bromide (**Low Hazard**) Refer to CLEAPSS Hazcard 47B

Potassium (or sodium) iodide solution (**Low Hazard**) Refer to CLEAPSS Hazcard 47B and Recipe Book sheet 72

Silver nitrate solution (**Low Hazard but stains fingers and clothing** at concentration used, **Danger to the environment**) Refer to CLEAPSS Hazcard 87 and Recipe Book sheet 77

Dilute ammonia solution (**Low Hazard**) Refer to CLEAPSS Hazcard 6 and Recipe Book sheet 6, and L195 Safer chemicals, safer reactions

Concentrated ammonia solution (**Corrosive, Danger to the environment**) Refer to CLEAPSS Hazcard 6 and Recipe Book sheet 6

Place sufficient test-tube racks to accommodate 3 test-tubes per group in a brightly lit part of the laboratory – such as on a window sill, and sufficient to accommodate 3 test-tubes per group in a dark part of the laboratory - such as in an empty cupboard.

Activity 2: Reactions of aqueous solutions of the halogens

Teacher notes

This activity compares the colours of three halogens in aqueous solution and in a non-polar solvent. These halogens also react to a small extent with water, forming acidic solutions with bleaching properties.

Halogens undergo redox reactions with metal halides in solution, displacing less reactive halogens from their compounds. These displacement reactions are used to establish an order of reactivity down Group 7 of the Periodic Table.

Lesson organisation

This series of simple experiments illustrates some of the chemical properties of the halogens following an introduction to the physical properties of the Group 7 elements. It can be done as a demonstration or as a class experiment.

Investigating the solubility of the halogens in a non-polar solvent can be left out, or only shown as a demonstration.

If it is done as a class experiment you should allow 30 minutes.

Procedure

HEALTH & SAFETY: Wear eye protection

The halogens in water and a hydrocarbon solvent

a Pour about 2 cm³ of each of the aqueous halogen solutions into separate test-tubes. Add equal volumes of hydrocarbon solvent to each tube, stopper the tube and, holding your thumb over the bung, shake the mixture by inverting the test-tube a few times.

b Allow the two layers to settle. Observe and record the colour of each layer. It may be necessary to shake the test-tubes again to transfer more of the halogen from the water to the hydrocarbon layer.

c What does this tell you about the solubility of the halogens in the different solvents. What is the difference between the solvents.

Acidic and bleaching properties of halogen solutions

a Place a piece of Universal Indicator paper on a white tile. Transfer a drop of chlorine water onto the paper using a glass rod. Observe and record the colour(s) of the paper. Explain what you see – think about the species present.

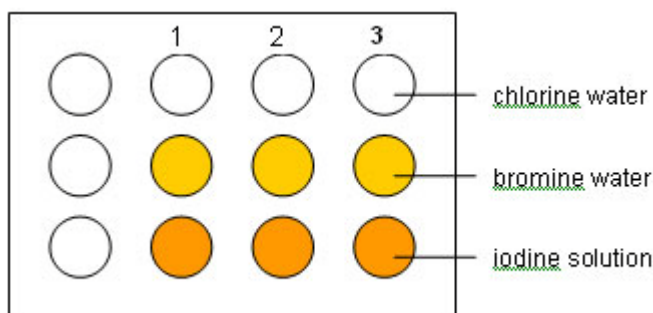
b Wipe the glass rod and the tile clean with a paper towel or tissue. Place a fresh piece of indicator paper on the tile and transfer a drop of bromine water onto it using the glass rod. Observe the colour of the paper.

c Repeat **b**, using the iodine solution.

d Is there a common theme or are there differences between the halogens.

Displacement reactions

a Using a plastic pipette put two drops of chlorine solution in each of three dimples in the spotting tile, as shown below. In the same way and using a clean plastic pipette for each solution, add bromine water, and iodine solution to the spotting tile.



b Add two drops of potassium chloride solution to each of the three dimples in column 1 of the tile. Observe and record any colour changes that take place.

c Add two drops of potassium bromide solution to each of the three dimples in column 2 of the tile. Observe and record any colour changes that take place.

d Add two drops of potassium iodide solution to each of the three dimples in column 3 of the tile. Observe and record any colour changes that take place.

e For reactions in which bromine or iodine are suspected to have formed, the reaction could be repeated with 2 cm³ of each solution in a test tube, and hexane could then be added to confirm the presence of bromine or iodine.

f Decide upon a series of reactivity for Group 7.

Teaching notes

A results table similar to the one below could be used for the recording of results. It has been completed with expected observations.

	Colour after shaking with hydrocarbon solvent	Effect on indicator paper	Reaction with potassium chloride solution	Reaction with potassium bromide solution	Reaction with potassium iodide solution
Chlorine water	Aqueous layer: pale yellow-green to colourless Hydrocarbon layer: colourless to pale yellow-green	Turns red, then rapidly bleaches white	No reaction	Yellow-orange colour of bromine appears	Brown colour of iodine appears
Bromine water	Aqueous layer: yellow-orange to colourless Hydrocarbon layer: colourless to yellow-orange	Turns red, then slowly bleaches white	No reaction	No reaction	Colour darkens from yellow-orange to brown

	Colour after shaking with hydrocarbon solvent	Effect on indicator paper	Reaction with potassium chloride solution	Reaction with potassium bromide solution	Reaction with potassium iodide solution
Iodine solution	Aqueous layer: brown to colourless Hydrocarbon layer: colourless to purple	Paper stained brown	No reaction	No reaction	No reaction

The halogens are more soluble in the hydrocarbon and move to this top layer, when shaken with a hydrocarbon solvent. For chlorine and bromine the colour does not change. You might need a white background to see the colour of the chlorine solution. However, for iodine there is a colour change, from brown in water to purple in the hydrocarbon layer.

Where no displacement reaction takes place between a halogen solution and a halide solution, it may be that some lightening in the colour of the solution is observed and this can be explained by the effect of dilution.

Take care to limit students' exposure to chlorine and bromine water fumes. Some students with respiratory problems can show an allergic reaction to chlorine, the onset of which may be delayed.

Iodine is the least soluble of the halogens in water. It is more soluble in potassium iodide solution, so the 'iodine solution' here is actually iodine in potassium iodide solution.

Reactions of aqueous solutions of the halogens

Technician notes

Apparatus and chemicals

Eye protection

One group of students requires:

Test-tube rack, to hold 10 test-tubes

Test-tubes, 10

Cork or rubber bungs to fit, 4

Plastic dropping pipettes, 6

White spotting tile

White tile

Glass rod

Paper towel or tissue

Universal Indicator paper (about 2 cm strips), 3

About 10 cm³ of each of the following halogen solutions in stoppered test-tubes (see notes 1 and 2):

Chlorine water, 0.1% (w/v) (The gas is **Toxic, Irritant, Dangerous for the environment** but the solution is **Low Hazard**)

Bromine water, 0.1% (w/v) (**Harmful**)

Iodine solution, 0.5% (w/v) (**Dangerous for the environment** at this concentration)

Half a test-tube of each of the following solutions (see note 3):

Potassium (or sodium) chloride solution, about 0.1 mol dm⁻³ (**Low hazard**)

Potassium (or sodium) bromide solution, about 0.1 mol dm⁻³ (**Low hazard**)

Potassium (or sodium) iodide solution, about 0.1 mol dm⁻³ (**Low hazard**)

Optional:

Cyclohexane (**Highly flammable, Harmful, Danger for the Environment**) or other suitable non-polar solvent, about 10 cm³ (see note 1)

Technical notes

Chlorine water (Chlorine gas escapes, which is **Toxic, Irritant, Dangerous for the environment** but the solution is **Low Hazard** at the concentration used) Refer to CLEAPSS Hazcard 22B, Recipe Book sheet 25

Bromine water (**Harmful**) Refer to CLEAPSS Hazcard 15B, Recipe Book sheet 17

Iodine solution (**Dangerous to the environment** at concentration used) Refer to CLEAPSS Hazcard 54, Recipe Book sheet 50

Potassium chloride solution (**Low hazard**) Refer to CLEAPSS Hazcard 47B, Recipe Book sheet 68

Sodium chloride solution (**Low hazard**) Refer to CLEAPSS Hazcard 47B, Recipe Book sheet 82

Potassium iodide solution (**Low hazard**) Refer to CLEAPSS Hazcard 47B, Recipe Book sheet 72

Cyclohexane (**Highly flammable, Harmful, Danger for the Environment**) Refer to CLEAPSS Hazcard 45B

1 Each group of students should be supplied with stoppered test-tubes containing about 10 cm³ of each of the aqueous solutions of the halogens and one of cyclohexane (optional).

2 The halogen solutions can be diluted further to minimise the amount of chlorine or bromine fumes given off but should not be so dilute that their distinctive colours are not clearly visible in the test-tubes (a white background may be needed for chlorine water).

3 The concentration of the potassium (or sodium) iodide should be adjusted so that it gives a light brown solution on adding the chlorine water. If these reagents are too concentrated, a black precipitate of iodine often results instead of a brown solution.

4 At the end of the experiments all mixtures and solutions should be returned to a suitable waste container in a fume cupboard for safe disposal.

Activity 3: Halogen reactions with iron

Teacher notes

In this demonstration experiment, iron wool is heated in the presence of chlorine gas and the vapours of bromine and iodine. Exothermic redox reactions occur, causing the iron wool to glow. Iron(III) halides (FeX_3) are formed as coloured solids:

The vigour of the reactions corresponds to the order chlorine > bromine > iodine, showing the trend of decreasing reactivity of the elements down Group 7.

To view a video clip of these demonstration experiments, click [here](http://media.rsc.org/videoclips/clips/ReactionHalFeWool.mpg) (<http://media.rsc.org/videoclips/clips/ReactionHalFeWool.mpg>).

Lesson organisation

These experiments must be carried out in a fume cupboard as both the reactants and products are hazardous. Teachers attempting this demonstration for the first time are strongly advised to carry out a trial run before doing it in front of a class.

Time allowed should be at least 20 min, depending on the amount of discussion and testing of the products between each experiment.

In addition to using this demonstration to show the relative reactivity of the halogens, the reaction of chlorine or bromine with iron could be used on its own to show the reaction between a reactive non-metallic element and a metal.

Procedure

HEALTH & SAFETY: Work in a fume cupboard throughout each stage of this demonstration. Wear suitable safety goggles. Have at least 500 cm³ of 1M sodium thiosulfate readily available to treat any spillages of liquid bromine. Double-check that the acid you are using to generate chlorine is concentrated **hydrochloric acid**. Several accidents have occurred when teachers have inadvertently used concentrated sulfuric acid.

Chlorine

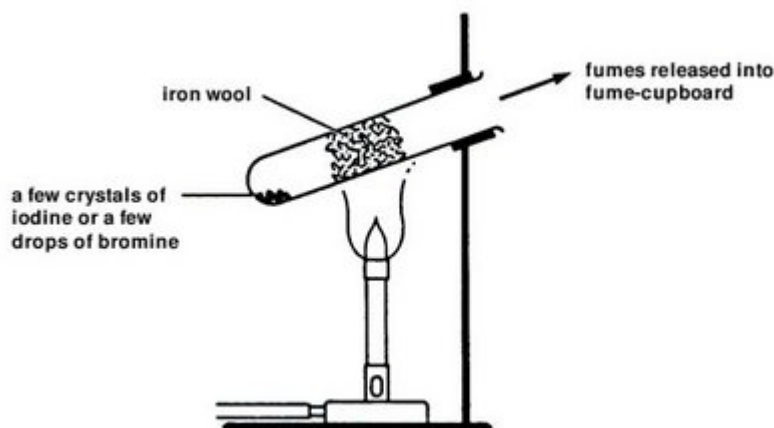
- Place a 1 g tuft of cleaned iron wool in the reduction tube so that it is well spread out. Leave at least a 1 cm gap between the stopper and the iron wool.
- Connect the reduction tube to the chlorine generator with a short length of rubber tubing. Clamp it in position over a Bunsen burner.
- Pass a slow stream of chlorine over the iron wool from the chlorine gas generator. Do this by allowing the hydrochloric acid to drip *slowly* on to the potassium manganate(VII). After a few seconds, it should be possible to see the greenish colour of the chlorine gas filling the reduction tube, as all the air is expelled.
- The iron wool may ignite without any heating. If not, gently heat at the end nearest to the chlorine generator until the wool does ignite (no further heating should be required).
- A vigorous reaction will occur and the glow will spread along the wool in the tube, producing clouds of brown iron(III) chloride. Some of this may emerge as a smoke from the end of the reduction tube.
- Continue passing chlorine over the iron wool until no further reaction occurs. Stop the chlorine supply and allow the tube to cool.

- g) When cool, disconnect the reduction tube and rinse a little of the product into a clean beaker with some distilled water. Pour some of this solution into a clean test-tube and test with a few drops of silver nitrate solution. A white precipitate of silver chloride will form, confirming the presence of chloride ions.

Bromine See Standard procedure: [liquid bromine](http://www.practicalchemistry.org/print/standard-techniques/handling-liquid-bromine-and-preparing-bromine-water,59,AR.html)

(<http://www.practicalchemistry.org/print/standard-techniques/handling-liquid-bromine-and-preparing-bromine-water,59,AR.html>).

- a) Wear suitable protective gloves and take care to avoid spillage when handling liquid bromine. It is Corrosive and Very toxic. Transfer about 0.5 cm^3 liquid bromine into one of the boiling tubes, using the teat pipette. Care is needed to avoid spillage - the density and volatility of the bromine cause it to drip very easily from the pipette. Keep the bromine container and the mouth of the test-tube close together. Replace the lid of the bromine container immediately.
- b) Using tongs or tweezers, place a 1 g tuft of cleaned iron wool into the boiling-tube so that it is well spread out and almost fills the boiling-tube. Leave a 2 cm gap between the iron wool and the surface of the liquid bromine.
- c) Clamp the test-tube near the top and at an angle of about 45° – see diagram.



- d) Heat the test-tube, gently at first, with a yellow-tipped blue flame (air hole on Bunsen burner slightly closed). Do this by moving the flame slowly between the bottom half of the iron wool and the bromine. As the bromine vapour starts to rise up into the iron wool, heat the wool more strongly.
- e) Remove the heat when the wool starts to glow due to the heat of the reaction. Note the extra heating required to get this reaction started compared to the reaction involving chlorine. The iron will become coated with yellow-brown iron(III) bromide, and a brown 'smoke' may escape from the mouth of the test-tube.
- f) When the reaction appears to be over, use tongs or tweezers to remove some of the remaining iron wool from the test-tube.
- g) Rinse the iron wool in a few cm^3 of deionised/distilled water in a small beaker. Pour out some of the resulting solution into a clean test-tube and test with a few drops of silver nitrate solution. Formation of a cream precipitate of silver bromide confirms that bromide ions are present.

Iodine

- a) Transfer about 0.5 g of solid iodine (**Harmful**) into one of the boiling tubes. Place a 1 g tuft of cleaned iron wool in the test-tube and clamp it as before.

- b) Working in a fume cupboard, heat the test-tube with a yellow-tipped blue flame (air hole on Bunsen burner slightly closed). Heat gently at first by moving the flame slowly between the bottom half of the iron wool and the iodine.
- c) As the purple iodine vapour starts to rise up into the iron wool, heat the wool more strongly. Remove the heat when the reaction causes a dull glow – see Additional notes below. Some red-brown iron(III) iodide should form.
- d) When the reaction appears to be over, remove some of the remaining iron wool from the test-tube with tweezers and rinse it in a few cm³ of deionised/distilled water in a small beaker. Pour some of the resulting solution into a clean test-tube and test it with a few drops of silver nitrate solution. Formation of a yellow precipitate of silver iodide confirms that iodide ions are present.

Additional teaching notes, hints and tips

The order in which the experiments are done is a matter of choice, but it is probably best to leave the most reactive halogen (chlorine) to last, to end with a vigorous reaction - and confirm a class prediction?

The reaction with iodine is much less vigorous than that with bromine and it may be difficult to see a glow at all. A couple of trial experiments beforehand may be necessary to get the right balance between heating the iodine and getting the iron hot enough for a reaction to start. If the iron is heated too vigorously, it may start to glow from reaction with the oxygen in any air that may still be present in the test-tube.

The general equation for the reactions involved is:



Halogen reactions with iron

Technician notes

Apparatus and chemicals

Eye protection for teacher *and* students, protective gloves for teacher

Access to a fume cupboard

Apparatus to set up a chlorine generator

Boiling-tubes, 2

Reduction tube (see note 1)

Beakers (100 cm³), 3

Tweezers

Teat pipette (preferably glass, with a narrow tip)

Test-tubes, 3 small, and a test-tube rack

Bunsen burner and heat resistant mat

Bosses, clamps and stands

The approximate quantities of chemicals below are sufficient for one demonstration.

Iron wool, 3 tufts about 1 g mass each (see note 2)
Liquid bromine (**Corrosive, Very toxic, Danger to the environment**), 0.5 cm³ (see note 3)
Sodium thiosulfate solution (**Low hazard**), 500 cm³ at 1 mol dm⁻³ (see note 3)
Iodine solid (**Harmful, Danger to the environment**), 0.5 g.
Hexane (**Highly flammable, Harmful**), 100 cm³ (see note 2)
Silver nitrate solution, 10 cm³ at approximately 0.1 mol dm⁻³. Care - stains skin and clothes.
Chlorine generator (**Toxic, Danger to the environment**) (see note 4)
Potassium manganate(VII) (**Oxidising agent, Harmful, Danger to the environment**), 10g (see note 4)
Concentrated hydrochloric acid (**Corrosive**), 100 cm³ (see note 4)
Deionised or distilled water

Technical notes

Liquid bromine (**Corrosive, Very toxic, Danger to the environment**). Refer to CLEAPSS Hazcard 15A. See Standard procedure: [Liquid bromine](#).

Sodium thiosulfate solution (**Low hazard**).

Iodine solid (**Harmful, Danger to the environment**). Refer to CLEAPSS Hazcard 54.

Hexane (**Highly flammable, Harmful**). Refer to CLEAPSS Hazcard 45A

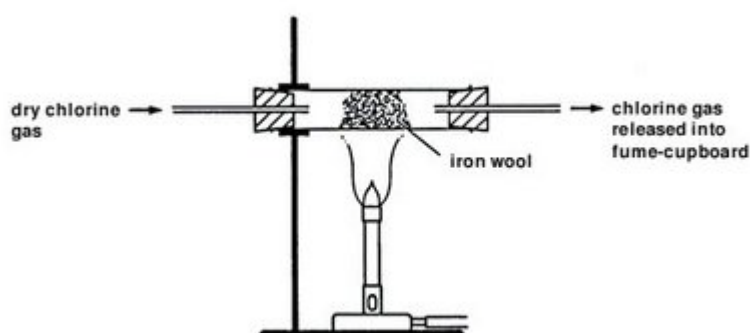
Silver nitrate solution. Refer to CLEAPSS Hazcard 87.

Chlorine generator (**Toxic, Danger to the environment**). Refer to CLEAPSS Hazcard 22A. See CLEAPSS Recipe Book sheet 24 and standard procedure: [Generating, collecting, and testing gases](http://www.practicalchemistry.org/print/standard-techniques/generating-collecting-and-testing-gases,52,AR.html) (<http://www.practicalchemistry.org/print/standard-techniques/generating-collecting-and-testing-gases,52,AR.html>).

Potassium manganate(VII) (**Oxidising agent, Harmful, Danger to the environment**). Refer to CLEAPSS Hazcard 81

Concentrated hydrochloric acid (**Corrosive**). Refer to CLEAPSS Hazcard 47A

1 The reduction tube should be fitted with a one-holed rubber stopper fitted with short length of glass tubing. Alternatively, an 8 to 10 cm length of wide-bore glass tubing with a stopper at each end fitted with a short length of glass tubing could be used. See diagram below.



2 The finest grade iron wool is best since it provides the maximum surface area. Iron wool is often sold with a thin layer of grease on its surface to stop it rusting. Working in a fume cupboard, the layer of grease can be removed by dipping the iron wool in hexane (or alternative) a few times. The solvent must be allowed to completely evaporate.

3 Wear suitable protective gloves when handling bromine. Have at least 500 cm³ of 1M sodium thiosulfate readily available to treat any spillages.

4 Double-check that the acid you are using to generate chlorine is concentrated **hydrochloric acid**. Several accidents have occurred when teachers have inadvertently used concentrated sulfuric acid.

Activity 4: Acidic and bleaching properties of halogen solutions

These halogens also react to a small extent with water, forming acidic solutions with bleaching properties.

Procedure

Wear eye protection

- Place a piece of Universal Indicator paper on a white tile. Transfer a drop of chlorine water onto the paper using a glass rod. Observe and record the colour of the paper.
- Wipe the glass rod and the tile clean with a paper towel or tissue. Place a fresh piece of indicator paper on the tile and transfer a drop of bromine water onto it using the glass rod. Observe the colour of the paper.
- Repeat b, using the iodine solution.

Results

- Using the results of the experiment with chlorine water explain your observations.
- Compare the results for all the solutions and explain the differences.

Apparatus and chemicals

Eye protection

One demonstration or one group of students requires:

Plastic dropping pipettes, 6

White tile

Glass rod

Paper towel or tissue

Universal Indicator paper, 3

About 10 cm³ of each of the following halogen solutions in stoppered test-tubes (see notes 1 and 2):

Chlorine water, 0.1% (w/v) (The gas is **Toxic, Irritant, Dangerous for the environment** but the solution is **Low Hazard**)

Iodine solution, 0.5% (w/v) (**Dangerous for the environment** at this concentration)

Technical notes

Chlorine water (Chlorine gas escapes, which is **Toxic, Irritant, Dangerous for the environment** but the solution is **Low Hazard** at the concentration used) Refer to CLEAPSSR Hazcard 22B, Recipe Book sheet 25

Iodine solution (**Dangerous to the environment** at concentration used) Refer to CLEAPSSR Hazcard 54, Recipe Book sheet 50

1. Each group of students should be supplied with stoppered test-tubes containing about 10 cm³ of each of the aqueous solutions of the halogens.
2. At the end of the experiments all mixtures and solutions should be returned to a suitable waste container in a fume cupboard for safe disposal.
3. It is essential for the acidity test that the chlorine water is just that. Some samples of 'chlorine water' that can be purchased from suppliers are actually chlorine in sodium hydroxide solution: this can give unexpected results in the test for pH.

Activity 5: Which bleach is the most cost effective?

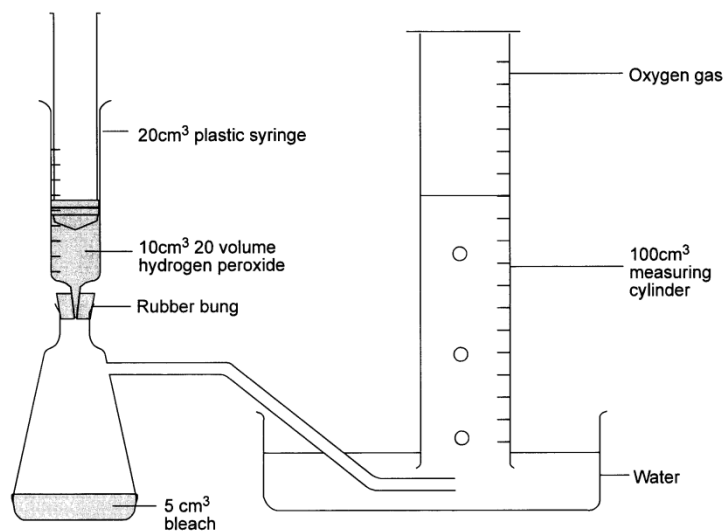
Different domestic bleaches contain different amounts of chlorine. This is not necessarily reflected in the price. This practical will help to determine which is the most cost effective product. Excess hydrogen peroxide is added to household bleach and the volume of oxygen produced is measured. The concentration of sodium chlorate(I) in the bleach can be calculated. The students are asked to be the scientist working for a consumer magazine. This month they are investigating chlorine-containing bleaches. They are asked to write an article for the magazine giving the results, explaining the methodology of the tests and ultimately which is the most cost effective bleach. This exercises the students' literacy skills, a key aspect of the GCSE curriculum.

The procedure

Use the pipette to measure 5 cm³ of household bleach into the flask and replace the bung. Fill the syringe with 10 cm³ of 20 volume hydrogen peroxide and fit its nozzle into the rubber bung. Fill the measuring cylinder with water. Squirt the peroxide into the bleach. The solutions will react and oxygen will be given off and collected in the measuring cylinder. Shake to ensure complete mixing and take a reading of the volume of the gas collected when gas has stopped being given off.

Repeat for each bleach.

Calculate the percentage of sodium chlorate(I) in the bleach.

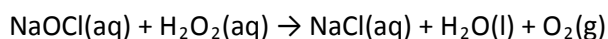


Generating oxygen

Write a report for the 'Consumer Watch-dog' magazine, giving the results, explaining the methodology of the tests and ultimately which is the most cost effective bleach.

Teacher notes

The active ingredient of household bleach is sodium chlorate(I) (sodium hypochlorite). This reacts with hydrogen peroxide:



So one mole of oxygen is equivalent to one mole (74.5 g) of sodium hypochlorite and the percentage of sodium hypochlorite in the original bleach solution can be calculated as follows:

If the total volume of gas in the measuring cylinder is $V \text{ cm}^3$, then the volume of oxygen produced will be $V - 10 \text{ cm}^3$ because the hydrogen peroxide will displace 10 cm^3 of air. At room temperature this is approximately $(V-10)/24\ 000$ mole. So 5 cm^3 of bleach solution contained $(V-10)/24\ 000$ mole of sodium hypochlorite which is $[(V-10)/24\ 000] \times 74.5 \text{ g}$. So in 100 cm^3 of solution there would be $20 \times [(V-10)/24\ 000] \times 74.5$, i.e. $0.062(V-10) \%$ sodium hypochlorite.

So normal household bleach (concentration of less than 5 %) should give a maximum of 90 cm^3 of gas in total. The density of bleach is about 1.2 g cm^{-3} so the w/v percentage could be converted into w/w if required.

For classes that might have difficulty with the calculations, the measuring cylinder could be directly calibrated in % sodium hypochlorite before the demonstration eg 90 cm³ could be marked 5 % etc. See the calculations above.

If a list of the prices of different brands of bleach was compiled, a value for money figure could be worked out such as 'grams of hypochlorite per penny'. A spreadsheet would be ideal for this type of calculation.

Extension

Some brands of thickened bleach seem to give lower results than expected. It is best to add water to these after pipetting 5 cm³ of bleach into the flask. Students may need help to appreciate that this dilution does not affect the amount of bleach present. Results could be compared with those obtained by adding excess potassium iodide to the bleach and titrating the liberated iodine with standard sodium thiosulphate.

Which bleach is the most cost effective?

Technician notes

Different domestic bleaches contain different amounts of chlorine. This is not necessarily reflected in the cost.

Description

Excess hydrogen peroxide is added to household bleach and the volume of oxygen produced is measured. The concentration of sodium chlorate(I) in the bleach can be calculated.

Apparatus

Each group will require

Eye protection.

One 250 cm³ Buchner flask with a one-holed rubber bung to fit.

One 10 cm³ plastic syringe.

About 30 cm of rubber tubing to fit the side arm of the Buchner flask.

One 100 cm³ measuring cylinder.

Trough.

One 5 cm³ pipette and filler.

Chemicals

The quantities given are for one experiment.

5 cm³ of each of three different household bleaches (some may be **irritants**).

The cost and volume of each bottle will need to be supplied.

10 cm³ of 20 volume hydrogen peroxide solution (**Irritant** at this concentration) See CLEAPSS Hazcard 50 and Recipe Book sheer 45.

Activity 6: Extracting iodine from seaweed

Teacher Notes

Ribbon seaweeds (sp. laminaria) contain iodine compounds which they obtain by extracting iodide ions from seawater. This iodine can be extracted from the seaweed by heating it in air to an ash, in which the iodine is present as iodide. The iodide is dissolved out of the residue by boiling water, the solution filtered and the iodide oxidised to iodine using hydrogen peroxide. The iodine colours the solution brown at this stage. Isolating the iodine from this solution can be done by solvent extraction, followed by evaporation of the solvent.

Procedure

HEALTH & SAFETY: Carry out the first stage in a fume cupboard. Wear eye protection.

1. Pile the seaweed up on the tin lid supported on a tripod and begin heating with a strong Bunsen flame. It may have to be added a portion at a time during the heating, given the quantity to be reduced to ash. When all has been turned to ash, about a dessertspoonful of residue will remain.
2. Boil the ash with about 20 cm³ of purified water in a beaker, and filter while hot. Collect the clear filtrate in a second beaker and allow to cool.
3. Turn off the Bunsen burner.
4. Add about 2 cm³ of dilute sulfuric acid to the solution, followed by hydrogen peroxide solution. A deep brown colour of iodine is formed as hydrogen peroxide oxidises the iodide ions present to iodine.
5. Transfer the mixture to a separating funnel and add 10 - 20 cm³ of cyclohexane. Stopper the separating funnel, secure it with your thumb, and shake vigorously for about 30 s. With the separating funnel inverted, release any pressure that has built by opening the tap briefly.
6. Clamp the funnel and allow the layers to separate. The cyclohexane will form a layer on top of the aqueous layer, and be coloured purple by the iodine now dissolved in it.
7. Run off the lower aqueous layer and discard down the sink with running water.
8. Run the purple cyclohexane layer into an evaporating basin, and set aside to evaporate in the fume cupboard. **DO NOT HEAT!**
9. Iodine crystals will form slowly, probably in time to be shown to the class in the next lesson.

Lesson organisation

Under suitable circumstances it could be considered for a class experiment, using a smaller quantity of seaweed and up to the point of seeing the iodine colour in aqueous solution. There are safety considerations involving the use of the flammable solvent as a class experiment for larger groups. Burning seaweed produces some very smelly fumes, it is thus recommended that this procedure is performed in a fume hood.

However, with suitable small groups, extraction of the aqueous iodine into a non-polar solvent to show its characteristic purple colour could be carried out on a test-tube scale, using only a few cm³ of the solvent. Solid iodine would be not be isolated.

All solvent residues must be placed in a container in a fume cupboard for subsequent disposal.

The practical is rather lengthy, possibly taking 30 - 45 minutes to the point where the extracted iodine solution can be left to evaporate. Teachers may wish to consider commencing the heating of the seaweed prior to the start of the lesson, which may perhaps enable 15 minutes of lesson time to be saved.

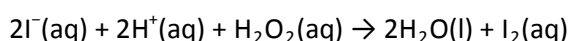
Teaching notes

The seaweed contains a wide range of elements drawn from sea-water by the living algae, giving concentrations in the organism that may be considerably higher than in sea-water itself. In the past, this natural concentration of useful elements, for which there was then no other economic source, gave rise to a large scale 'cottage industry' network around the west coasts of Britain and Ireland, employing thousands in gathering and burning the kelp on the shore. The story of these past times can be used to enliven the waiting times at the different stages of the demonstration – see Weblinks below.

The two principal products were soda ash (sodium carbonate) for the alkali industry in the 18th and early 19th centuries, and iodine in the later nineteenth century. Kelp was also used, and still is, as an excellent fertiliser. Soda ash production from kelp ceased when the chemical industry itself found ways of producing sodium carbonate from sodium chloride, and iodine production from kelp ceased when the extraction of iodine from Chile saltpetre became dominant. In both events, thousands were put out of work, leading to large numbers emigrating from Scotland and Ireland to America and elsewhere. Nowadays, a small-scale industry continues, providing the raw material for expensive kelp-derived health supplements containing several 'essential minerals' including iodine. A typical kelp capsule may contain 0.1% iodine. The main kelp product at present, though, is for alginates used in food and drink, cosmetics and dozens of other everyday products.

Burning the kelp converts the iodine compounds in the kelp into iodide anions. Bromide and chloride ions are also present. The cations in the ash include sodium and potassium, so the leached solution in effect contains sodium and potassium halides.

Adding hydrogen peroxide in acid solution preferentially oxidises the iodide anions to iodine:



Although iodine is sparingly soluble in water, it is much more soluble in the presence of more iodide ions, and the brown colour is due to this effect, though some of the iodine may be present as a dark brown precipitate.

Web Links

There is a wealth of information available on the internet as background to this experiment. Just a small sample of these sites is given below

For biological background on kelp:

<http://www.pznw.co.uk/marine/kelp.html>

For background information on the chemistry of iodine, and its historical discovery:

<http://en.wikipedia.org/wiki/Iodine>

For historical details of kelp-burning communities, try:

<http://www.orkneyjar.com/tradition/kelpburning.htm>

An internet search for 'kelp' produces hundreds of health food supplement suppliers, whose websites give information about the mineral contents of kelp capsules and their claimed benefits. These may be useful in promoting discussion about scientific evidence and how it is used and misused in such contexts.

Extracting iodine from seaweed

Technician notes

Apparatus and chemicals

Each practical will require:

Eye protection

Access to fume cupboard

Beakers (250 cm³), 2

Beaker tongs

Filter funnel

Filter paper

Evaporating basin (75 cm³ or similar)

Separating funnel (100 cm³)

Bunsen burner and tripod

Heat resistant mat

Large tin lid (size to fit across the top of the tripod)

Hydrogen peroxide solution, '20 volume' (about 1.6 mol dm⁻³) (**Irritant** at this concentration), 10 cm³

Cyclohexane (**Highly flammable, Harmful, Dangerous for environment**), about 20 cm³ (see note 2)

Distilled or deionised water

Sulfuric acid, 1 mol dm⁻³ (**Irritant** at this concentration), 5 cm³

Kelp or ribbon seaweed (*Laminaria*), about a dozen 50 cm lengths (see note 1)

Technical notes

Hydrogen peroxide (**Irritant** at concentration used) Refer to CLEAPSS Hazcard 50 and Recipe book sheet 45

Cyclohexane (**Highly flammable, Harmful, Dangerous for environment**) Refer to CLEAPSS Hazcard 45B

Sulfuric acid (**Irritant** at concentration used) Refer to CLEAPSS Hazcard 98A and Recipe Book sheet 98.

1 *Laminaria* seaweed: this is the flat, brown, ribbon-like seaweed commonly called kelp, and any good source will suffice. Freshly collected material is likely to be the best, but some biological suppliers may be able to provide. Allow to dry, and keep dry, until ready to use.

2 Note that in the original version of this experiment, the solvent used was tetrachloromethane, but with restrictions on the use of chlorinated hydrocarbons this compound and related solvents should not be used in this experiment even if stocks are still held.

3 All solvent residues must be placed in a container in a fume cupboard for subsequent disposal.

Activity 7: Extension question

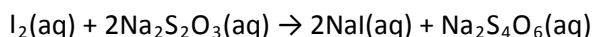
This question is about Iodine Number

This old method for determining the degree of unsaturation in a fat or oil is still used by most students following a food science course. You are asked to process the following experimental results.

Iodine monochloride is used to determine the degree of unsaturation in oils. The iodine monochloride adds rapidly to the carbon-carbon double bonds present. In an experiment, 0.127g of an unsaturated oil was treated with 25.00 cm³ of 0.100 mol dm⁻³ iodine monochloride solution. The mixture was kept in the dark until the reaction was complete. The unreacted iodine monochloride was then treated with an excess of aqueous potassium iodide, forming iodine.

The liberated iodine was found to react with 40.00 cm³ of 0.100 mol dm⁻³ sodium thiosulfate.

- (i) Suggest why it is necessary to keep the mixture of oil and iodine monochloride in the dark. (1)
- (ii) Write an equation for the reaction between iodine monochloride and potassium iodide. (1)
- (iii) Calculate the number of moles of sodium thiosulfate which were used in the titration. (1)
- (iv) Calculate the number of moles of iodine liberated, given that iodine reacts with sodium thiosulfate according to the equation:



Hence calculate the number of moles of unreacted iodine monochloride. (1)

- (v) Calculate the number of moles of iodine monochloride that reacted with the unsaturated oil. (1)

Direct addition of iodine to an unsaturated oil is slower than the addition of iodine monochloride.

However, unsaturation is quoted as the iodine number. The iodine number is the number of grammes of iodine that in theory can be added to 100 g of oil.

- (vi) Calculate the iodine number of this oil, given that one mole of iodine monochloride is equivalent to one mole of iodine molecules. (1)

Answer

This question is about Iodine Number

(i) By keeping the mixture of oil and iodine monochloride in the dark, free radical substitution of alkyl groups is prevented. (1)

(ii) $\text{ICl}(\text{aq}) + \text{KI}(\text{aq}) \rightarrow \text{KCl}(\text{aq}) + \text{I}_2(\text{aq})$ (1 mark – must give full equation and not ionic equation – do not penalise for incorrect state symbols in this question)

(iii) 0.00400 moles (1)

(iv) 0.00200 moles of unreacted iodine monochloride (1)

(v) 0.000500 moles (1)

(vi) 100 (1)

(–1 mark for incorrect sig figs in iii), iv) and v) **Total 6**

Glossary

diatomic	A molecule consisting of two identical atoms.
displacement	Where a more reactive element displaces a less reactive one from a solution of its salts.
electronegativity	The ability of an atom to attract the bonding electron pair in a covalent bond.
oxidation	Loss of electrons.
precipitate	A solid formed by the reaction of two substances in solution.
reduction	Gain of electrons.

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