

Modelling the Solvay process

MATERIALS

10 g solid NaCl
 10 g solid ammonium chloride (NH_4Cl)
 10 mL concentrated ammonia (NH_3)
 small cylinder of carbon dioxide gas, or gas generator using 1 M hydrochloric acid (HCl) and pellets of calcium carbonate
 20 mL limewater
 small beakers
 Hirsch funnel and access to vacuum filtration (optional)
 Pyrex test-tube
 small conical flask
 red litmus paper

Introduction

Perform the reactions in the carbonator, converter and ammonia recovery. **Caution:** All steps of the investigation must be performed in a fume cupboard. Carry out a risk assessment for all chemicals used in this activity.

On completion of this investigation, you will be able to:

- identify risk factors involved in the laboratory modelling of the Solvay process
- identify the chemical process involved in each separate investigation
- compare the effectiveness of laboratory preparation with that of the industrial process
- identify difficulties with laboratory modelling of the Solvay process.



Syllabus

Perform a first-hand investigation to assess risk factors and then carry out, where possible, each of the chemical steps involved in the Solvay process, identifying any difficulties associated with the laboratory modelling of the process.

Procedure

- 1 Dissolve 3.5–4.5 g NaCl in 10 mL distilled or deionised water in a small beaker. This is your brine solution.
- 2 Your teacher will dissolve 5–6 g ammonium chloride (NH_4Cl) in 10 mL concentrated ammonia (NH_3) in a small beaker. (This represents an ammonium saturator.)
- 3 Transfer the brine solution into a beaker containing the saturated ammonia solution and stir. If the solution is clear, add a small amount of ammonium chloride until some is left undissolved.
- 4 Decant the clear solution into a small, clean, dry conical flask. Bubble carbon dioxide gas through the solution vigorously from a gas cylinder or a gas generator. (Carbon dioxide gas can be steadily generated by dripping 1 M HCl solution onto calcium carbonate chips in the gas generator.) A white precipitate will start forming after 5–7 minutes.
- 5 Filter the precipitate through a Hirsch funnel using vacuum filtration. If this is not available, decant the liquid into another beaker and scoop up the precipitate. (Filter paper will not work).
- 6 Place some of the dry precipitate into a clean test-tube. Add a few drops of water to dissolve and test it with 0.1 M barium nitrate solution for the presence of carbonate ions. A faint white precipitate will confirm this.

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- 7 Place the rest of the solid precipitate into a Pyrex test-tube. Heat gently at first, then strongly, until only a small amount of precipitate remains.
- 8 To regenerate ammonia from the filtrate, mix equal volumes of filtrate and saturated calcium hydroxide ($\text{Ca}(\text{OH})_2$) or limewater in a conical flask and heat. Hold moist red litmus paper in the mouth of the flask. The ammonia given out will change the colour of the paper from red to blue.

Discussion

- 1 List the chemical processes involved in the industrial production of sodium carbonate.
- 2 Write chemical equations for each step in the Solvay process.
- 3 Evaluate the effectiveness of laboratory modelling of each step of the Solvay process.

FOLLOW-UP

Demonstrate, using chemical equations, that limestone and salt are the raw materials and sodium carbonate and calcium chloride are the net products.

Risk assessment

Ammonia: Toxic by all routes of exposure. Concentrated ammonia is to be handled in the fume cupboard by the teacher only.

Disposal of waste

All liquids can be washed down the sink in the fume cupboard.

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Hints

Investigation 8.2: A qualitative analysis of an equilibrium reaction

- The following equilibrium is investigated: $\text{Cu}^{2+} + 3\text{I}^- \rightleftharpoons \text{CuI}(\text{s}) + \text{I}_2(\text{aq})$
- In Procedures A and B, the same colour of equilibrium mixture (dirty grayish brown) is observed as well as the presence of all other species in the equilibrium. The brown colour of the original equilibrium interferes slightly with the blue colour of the copper complex with the ammonia solution, but it is still possible to detect the presence of a blue colour.

Investigation 8.3: Properties of sulfuric acid

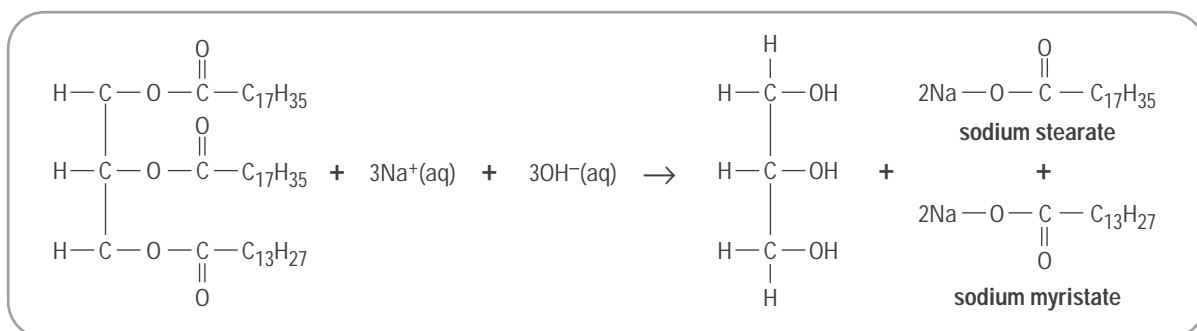
- Observe fumes that develop as sulfuric acid reacts with potassium iodide, forming a dark grey to black solid: $\text{HSO}_4^- + 3\text{H}^+ + 2\text{I}^- \rightarrow \text{SO}_2(\text{g}) + \text{I}_2(\text{aq}) + 2\text{H}_2\text{O}$
- Filter paper changes from orange to blue-green in the presence of sulfur dioxide gas.
- The reaction of sugar with concentrated sulfuric acid is spectacular.
- When dehydrating sugar, a black pillar will form with a lot of heat given off as well as fumes: $\text{C}_{12}\text{H}_{22}\text{O}_{11}(\text{s}) \rightarrow 12\text{C}(\text{s}) + 11\text{H}_2\text{O} + \text{heat}$
- The large amount of heat is produced as a result of water forming and diluting the concentrated sulfuric acid.
- Dehydrating blue copper sulfate crystals with concentrated sulfuric acid produces white copper sulfate (CuSO_4).

Investigation 8.4: Electrolysis of sodium chloride

- If the electrolysis is performed in a beaker, add a few drops of phenolphthalein indicator solution to identify OH^- ions forming at the negative electrode (cathode). Hold a piece of dry litmus paper over the anode just above the liquid (a bleaching effect will identify chlorine gas being produced).
- If the electrolysis is carried out using a voltmeter, collect some gas above the cathode in a dry test-tube and test with a lighted taper—a 'pop' will indicate the production of hydrogen gas. Collect the gas from the other electrode into a test-tube containing moist litmus paper and stopper the test-tube. The colour of the litmus paper will fade in the presence of chlorine gas.
- At the cathode: $2\text{H}_2\text{O} + 2\text{e}^- \rightarrow \text{H}_2(\text{g}) + 2\text{OH}^-$
At the anode: $2\text{Cl}^- \rightarrow \text{Cl}_2(\text{g}) + 2\text{e}^-$

Investigation 8.5: The saponification reaction

- The saponification reaction is the hydrolysis reaction of oils or fats with a strong alkali solution. Fats and oils are esters of long-chain fatty acids and glycerol. When oil is hydrolysed it produces glycerol and the sodium salt of fatty acid (soap):



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- The emulsifying property of soap is apparent in the test-tube containing soap and oil. The test-tube without soap has oil clearly visible after shaking, while the test-tube with soap is cloudy but with no visible oil present.
- The industrial production of soap uses cheaper fats such as tallows. It is fully mechanised and automated. However, the procedure is similar to the laboratory production. It starts with the reflux of fats with NaOH and the product is precipitated in ice-cold brine solution. To obtain fine, pure soap, the product is milled into fine soap particles and washed continuously with cold brine in order to rinse out residual NaOH.
- Cold brine is used for washing, because soap is soluble in pure water.

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Investigation 8.6: Properties of an emulsion

RESULTS		TABLE 1 SUMMARY OF RESULTS		
Sample	Type of emulsion	PROPERTY		
		Feel/Texture	Colour of soluble dye	Electrical conductivity
test-tube A (sodium oleate)	O/W	creamy, cold	blue	fair
test-tube B (calcium oleate)	W/O	greasy	red	poor
milk	O/W	creamy, cold	blue	poor
cleansing cream	O/W	creamy, cold	blue	fair
moisturising cream	W/O	greasy	red	poor
mayonnaise	O/W	creamy, cold	blue	good

Students should be able to draw a model of each emulsion and the action of surfactants in each case, as shown in Figures 1 and 2.

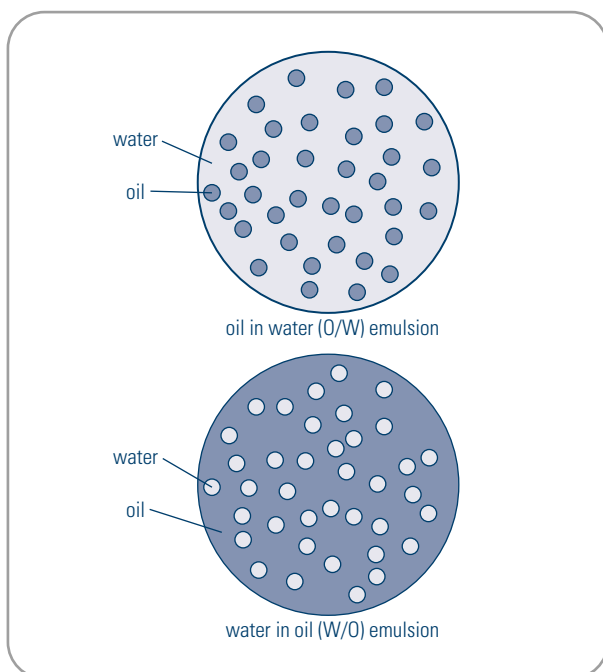


FIGURE 1
A representation of O/W and W/O emulsions.

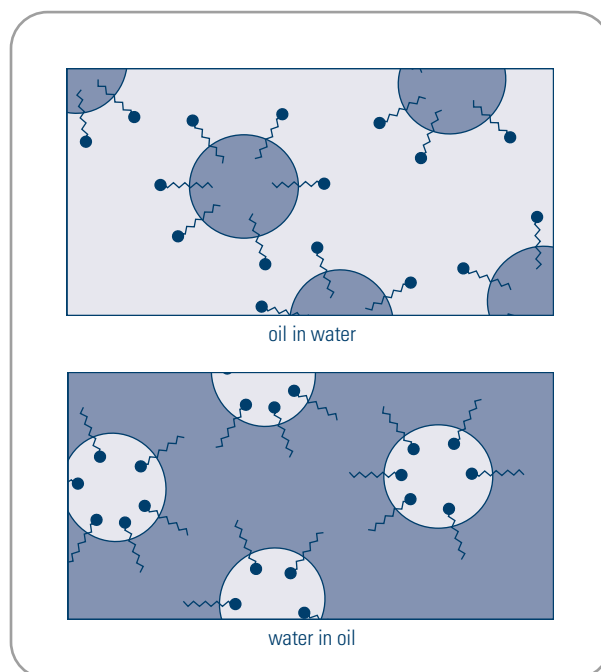


FIGURE 2
The behaviour of surfactant molecules in emulsions.

Investigation 8.7: Modelling the Solvay process

Difficulties associated with the laboratory modelling:

- **Reaction in lime kiln:** $\text{CaCO}_3(\text{s}) \rightarrow \text{CaO} + \text{CO}_2$
Students could try to decompose calcium carbonate by heating and bubbling the gas through limewater and observing how fast the bubbles are produced. However, most schools do not have strong enough heat from their Bunsen burners to decompose calcium carbonate. With such a low pressure, they will not be able to saturate the brine/ammonia solution to produce NaHCO_3 . In industry, such a process produces gas under a steady pressure above 150 kPa.
- The brine solution was not purified. The most common contaminants are CaCl_2 , MgCl_2 and traces of heavy metals. These must be precipitated from the brine as carbonates are filtered off before entering the reaction vessel. (This step was not done in the lab.)
- **Reaction in slaker:** $\text{CaO}(\text{s}) + \text{H}_2\text{O} \rightarrow \text{Ca}(\text{OH})_2(\text{aq}) + \text{E}$
This could be easily demonstrated by using very dry CaO , which has been in the oven at 150°C for a few hours, and cooled in a desiccator. Addition of water releases a lot of heat. Use a thermometer of $0\text{--}350^\circ\text{C}$ to measure the temperature of the mixture.
- **Reaction in saturator:** $\text{NaCl}(\text{aq}) + \text{NH}_3(\text{g}) \leftrightarrow \text{Na}^+(\text{aq}) + \text{Cl}^-(\text{aq}) + \text{NH}_3(\text{aq})$
We cannot produce saturated ammonia in the laboratory, as we are not allowed to generate NH_3 gas. Instead, we are saturating the solution with NH_4^+ ions. This is not the same as in the industrial closed reaction vessel, which contains ammonia gas under pressure above the saturated liquid. When brine is added to the reaction vessel, equilibrium is established between all ions and the ammonia vapour.
- **Reaction in carbonator:**
 $\text{NaCl}(\text{aq}) + \text{CO}_2 + \text{NH}_3(\text{g}) + \text{H}_2\text{O} \rightarrow \text{NaHCO}_3(\text{s}) + \text{NH}_4\text{Cl}(\text{aq})$
By bubbling CO_2 through the liquid in the conical flask, we do not have equilibrium such as in the industrial process, and our precipitate is a mixture—some NaHCO_3 but mostly ammonium chloride (NH_4Cl) and NaCl . This is demonstrated by heating the solid, which represents the converter.
- **Reaction in converter:** $2\text{NaHCO}_3(\text{s}) \rightarrow \text{Na}_2\text{CO}_3(\text{s}) + \text{CO}_2(\text{g})$
In the laboratory our solid on heating produces the sublimation of ammonium chloride and some NaCl , with traces of Na_2CO_3 . (The test for the presence of carbonates in the dissolved residue is not convincing.) In industry, sodium hydrogen carbonate is a pure solid that converts to sodium carbonate on heating.
- **Ammonia recovery:**
 $2\text{NH}_4\text{Cl}(\text{aq}) + \text{Ca}(\text{OH})_2(\text{aq}) \rightarrow \text{CaCl}_2(\text{aq}) + 2\text{NH}_3(\text{g}) + 2\text{H}_2\text{O}$
Regeneration of ammonia can only be done qualitatively in the laboratory. In the industrial process this is done in a closed system.

These reactions could be summed up to give a net reaction that will show what raw materials are needed. All other by-products are reused in the process. Multiply the reaction in the carbonator by a factor of 2 to balance and cancel out the same species. The net reaction is:



This reaction shows that limestone and salt are the raw materials and sodium carbonate and calcium chloride are the net products.

FOLLOW - UP

1 zinc atoms in $2.2\text{ g} = 2 \times 10^{21}$
 iodine atoms in $0.84\text{ g} = 4 \times 10^{21}$

2 TeO_2
 3 X_2S_5 (arsenic compound)

Sample risk assessment record**INVESTIGATION 9: Masses of combining elements**

Assessment team: _____ Date: _____ Year/group: **11**

Ref: *Heinemann Chemistry Practical Manual*

Chemicals used	Conc. (mol/L)	Amount	User code	DG class/haz	Procedure as per Appendix D?	What are the hazards?
iodine	solid	1.0 g	11-12	8/5.1 DHS	needs SSRA	Toxic by all routes of exposure
ethanol	pure	30 mL	7-12	3 Haz	yes	Highly flammable
Product:						
zinc iodide	solid	1 g	11-12	Haz	yes	Slightly toxic if ingested, corrosive

**Disposal of waste**

Collect specimen tubes with leftover iodine (if any) and alcohol. Zinc pieces cannot be reused, as they will very quickly react with oxygen, forming oxide. Leftover zinc pieces can be disposed of in the garbage bin. Zinc iodide can be collected or disposed of in the garbage.

How are risks controlled?

- wear safety glasses
- use gloves
- wash hands after use
- teacher to evaporate alcohol to dry product

Assessment of risk:

Risk for this investigation is **not significant**, provided appropriate control measures are in place as indicated above.

Approved by: _____ Date: _____

Investigation 10**Physical properties of water****Rationale**

At each station, students investigate either one physical property of water, or the unique effects caused by the polar nature of water molecules and hydrogen bonding. At Station 2, you should allocate a different task (one of five) to each group. Draw the Results table on the board, so that all groups can enter their results.

**Syllabus**

Perform an investigation and gather data to solve problems and analyse information involving calculation of the density of water as a liquid and a solid using $\text{density} = \text{mass}/\text{volume}$. Analyse information by using models to account for the differing densities of ice and liquid water. Plan and perform an investigation to identify and describe the effect of antifreeze and salt on the freezing point and/or boiling point of water. Choose equipment and perform first-hand investigations to demonstrate the following properties of water: surface tension, adhesion, cohesion, viscosity.

Background knowledge

Students should have already covered SI units for density, and terms such as capillarity, adhesion, cohesion and surface tension. These terms will be further reinforced during this investigation. Students should also have learned about the polarity of water and the reason for its polarity.

Hints

- Station 2: In order to estimate the freezing point of ethylene glycol solution and salt solution, place all three beakers with their thermometers in a deep freezer, and inspect every 20 minutes. Water will freeze in about 20 minutes, while the other two solutions will not freeze at all during the class time. Measure the temperature of the freezer and report these estimated freezing points as 'below freezer temperature'.
- Station 3: Instead of floating a wire ring with a string across it, you may set up a floating razor blade. This is more effective than a wire ring. However, due to safety concerns, razor blades are not mentioned.
- Station 4: Set up the simple equipment as in Figure 2. Tape different-sized capillary tubes to the ruler, making sure that one end of the largest capillary tube is dipped into oil before mounting it. For coloured water, use water-soluble food colouring or a crystal of potassium permanganate.
- Station 5: A test for the viscosity of liquids is best demonstrated by the set-up shown in Figure 3. Four glass tubes of length 40–50 cm and diameter 10–15 mm are sealed at the bottom with plasticine and hot wax. The appropriate liquid is poured to 5 mm from the top. The top is sealed with plasticine and wax, and the tubes mounted with strong clear adhesive tapes on stiff cardboard or a plywood board.

Station 1: DENSITY OF WATER AND ICE

RESULTS		
TABLE 1 STATION 1—SAMPLE RESULTS FOR CALCULATING DENSITY OF WATER		
Sample number	Volume of water (mL)	Mass of water (g)
1	5.5	5.51
2	6.7	6.68
3	7.4	7.39
4	8.2	8.21
5	9.3	9.2

TABLE 2	
Test	Observations
ice in water	8–10% of ice cube surface is protruding
ice in acetone	ice sinks to the bottom

Calculations

- 1 Graph of the data—see Figure 1.
- 2 Value of slope (from the graph) = $5.5/5.5 = 1.0$, hence density of water = 1.0 g/mL
- 3 Relative density of ice: since only 90–92% of the ice cube is submerged in water, the relative density of ice is 90–92% that of water, or 0.90 to 0.92.

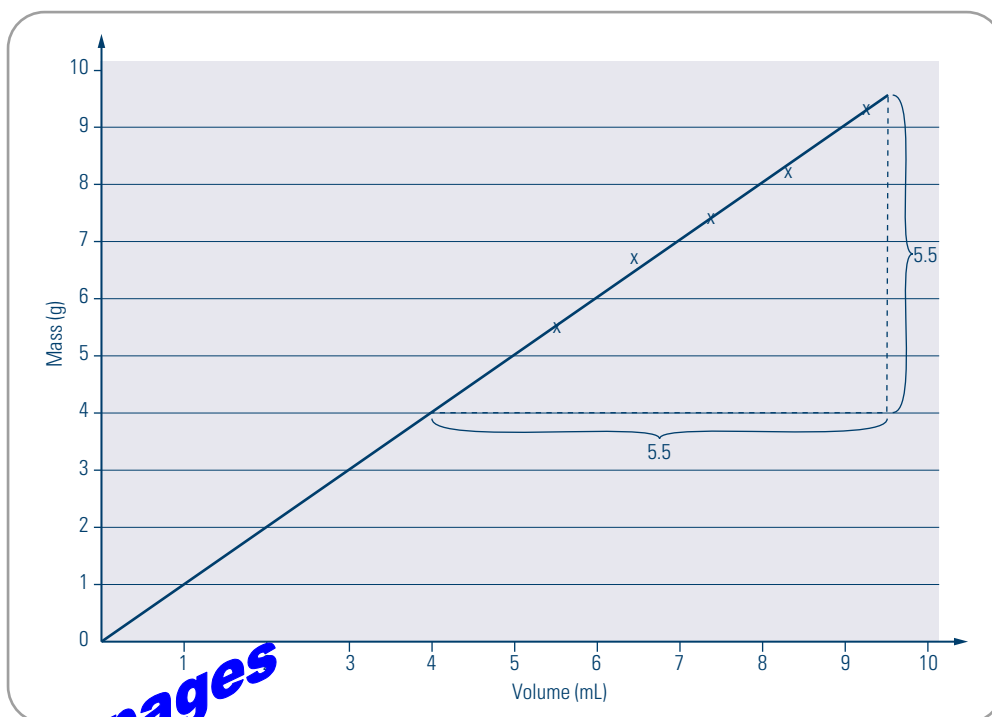


FIGURE 1
Determination of the density of water

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Station 2: FREEZING AND BOILING POINTS OF WATER

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RESULTS			RESULTS	
TABLE 3			TABLE 4	
Liquid	Estimated time (mins) taken to freeze	Estimated freezing temperature	Liquid	Boiling point (°C)
water	20–30 minutes	0°C	distilled water	100
20% ethylene glycol	did not freeze during class time	< -10°C	20% ethylene glycol	102
20% salt solution (NaCl)	did not freeze during class time	< -10°C	30% ethylene glycol	103
			50% ethylene glycol	104

Station 3: SURFACE TENSION OF WATER

RESULTS	
TABLE 5	
Test	Observations
water on glass	drop spreads almost flat
water on PVC	drop stays round
water on greased paper	drop fully rounded and moves freely
object floating on water	wire loop sits high on the surface, no submersion
object floating with detergent added	after 2 drops of detergent added, wire loop submerges and sinks
effect of charged ebonite rod on water	stream of water moves towards the rod
effect of charged Perspex rod on water	stream of water moves towards the rod

Station 4: CAPILLARITY, ADHESION AND COHESION

Two slides sandwiched together (Figure 1 in student practical manual) are redrawn by students with the meniscus decreasing from the end where glass touches to the end where the matchstick is inserted.

Students also redraw the set-up of the equipment (Figure 2

in student practical manual) and draw the meniscus in each tube. The first tube, which is oiled, does not have a meniscus above the level of water in the beaker. The same sized tube without oil has a meniscus about 5 mm above the level of the water. The narrower the tube, the higher the meniscus.

Station 5: VISCOSITY OF LIQUIDS

RESULTS		
TABLE 6		
Liquid	Structural formula	Time in seconds
water	$\begin{array}{c} \text{H} \quad \text{H} \\ \diagdown \quad / \\ \text{O} \end{array}$	2
ethylene glycol	HO-CH ₂ -CH ₂ -OH	3
acetone	H ₃ C-CO-CH ₃	1
glycerol	$\begin{array}{c} \text{H}_2\text{C}-\text{OH} \\ \\ \text{HC}-\text{OH} \\ \\ \text{H}_2\text{C}-\text{OH} \end{array}$	10

Discussion

- No, since each quantity has its own definite mass and volume.
 - Yes, since water expands when it freezes and heats up.
 - Yes, dissolved salts can alter the density of water. Salt water is more dense than fresh water.
- Since less than 10% of the ice is protruding above the water surface, it must be 10% less dense. Since water density is 1.0 g/mL, ice must be around 0.9 g/mL.
- Ice floats on water because its lattice structure is not

close-packed—there are empty spaces between molecules. (Use molecular models to arrange six water molecules like a benzene ring, orienting each oxygen to attract hydrogen.)

- Since 0.92 g ice has a volume of 1 mL, 1000 g (1 L) water would have volume of:

$$\frac{1000 \text{ g} \times 1.0 \text{ mL}}{0.92 \text{ g}} = 1087 \text{ mL}$$
- Ethylene glycol has much higher boiling point (198°C) and much lower freezing point (-15.6°C) than water. Ethylene glycol is added to a car radiator to raise the

boiling point of the water above 100°C, thus preventing the radiator boiling in hot weather. Ethylene glycol also lowers the freezing point of water, preventing the radiator from freezing when the temperature drops below -5°C.

- b Salt lowers the freezing temperature of water on the road to below -5°C so that dangerous ice does not form.
- 6 a Ethylene glycol has large molecules with two OH groups on each side. When these molecules come between water molecules, they form stronger hydrogen bonds between molecules of water and ethylene glycol than between water molecules. More energy is needed to overcome these intermolecular forces, and so the boiling point of the water increases.
- b Sodium chloride dissolves in water and breaks up into positive sodium ions and negative chloride ions. Water molecules surround each ion with the positive end of the water molecules surrounding negative chloride ions, and the negative ends of the water molecules surrounding positive sodium ions. In order for water to freeze, molecules of water must come together. However, in this case, sodium and chloride ions hold them apart. Ethylene glycol, with its strong hydrogen bonds, also keeps water molecules apart. A lot of energy must be removed before this attraction weakens, and hence the freezing point is lowered.
- 7 The reason for the differences in boiling points of these three compounds lies in the strength of the intermolecular forces between molecules. The electron-attracting ability of oxygen is 3.5 (on a relative scale called electronegativity), while hydrogen is 2.1, nitrogen 3 and sulfur 2.5. As a result, a covalent bond between hydrogen and oxygen is strongly polar (electronegativity difference is 1.4). The two covalent O-H bonds in a water molecule are at an angle of 109°. The water molecule is therefore polar. One polar molecule is attracted to another by intermolecular forces, which in the case of water molecules are called hydrogen bonding.
- Ammonia is a triangular pyramidal molecule and is not as strongly polar (electronegativity difference is only 0.9). The intermolecular attraction between the nitrogen of one molecule and the hydrogen of another, also called hydrogen bonding, is weaker than in water.
- Hydrogen sulfide has very weakly polar covalent bonds (electronegativity difference is 0.4), while the angle of the H-S bonds is larger than in water. The molecule is only weakly polar and has weak intermolecular forces.
- 8 a Surface tension can be considered a 'skin' on the surface of water. It is a measure of the resistance of a liquid to increasing its surface area.
- b Strong hydrogen bonds cause molecules of water to 'stick' together, creating strong cohesive forces.
- c Water has the stronger surface tension. Molecules of detergent reduce the strong cohesive forces between water molecules.
- d If a water molecule is in contact with a polar substance such as glass, which contains oxygen atoms, it will strongly adhere to it (adhesive forces), thus wetting the surface. However, grease and PVC have non-polar surfaces so cohesive forces between the water molecules are stronger than the weak adhesive forces, resulting in spherical drops.
- 9 The spherical shape of a water drop is due to a combination of surface tension and cohesive forces. A molecule at the surface of a liquid experiences cohesive (intermolecular) forces from the water molecules beside and below it. There are no molecules above it, so the overall force is towards the centre of the sphere.
- 10 Intermolecular forces, or hydrogen bonding, are responsible for fusing two drops together when they touch.
- 11 a Adhesion is the attraction of one material to another via intermolecular forces.
- b Cohesion is the attraction between molecules in a substance—that is, intermolecular forces acting between the same type of molecules.
- 12 Adhesion and cohesion forces are responsible for the capillary action of water. As the capillary tube gets narrower, the surface area of glass in contact with water increases; thus adhesive forces are much stronger than cohesive and gravitational forces. As a result, water rises.
- 13 Glycerol, ethylene glycol, water, acetone. Viscosity is a measure of the strength of cohesive intermolecular forces. Glycerol, with three hydrogen bonds per molecule, has the highest viscosity; glycol has two hydrogen bonds per molecule, while water has only one. Acetone is a polar molecule but without hydrogen bonding. There are only weak interactions between its molecules, so it has the lowest viscosity.
- 14 Water molecules are polar and rotate freely in the liquid state. When a positively charged rod is placed close to a stream of water, the negatively charged ends of water molecules (oxygen) line up towards the positive rod. When a negatively charged rod is used, the reverse occurs: the hydrogen ends of water molecules line up towards the rod.

FOLLOW - UP

- 1 In their roots and trunk, trees have small capillaries called xylem tubes, through which water enters with the help of osmotic pressure and capillary action.
- 2 Bricks have many small capillaries in their structure. In brick houses, the foundation must be insulated against moisture penetration because capillary action may cause permanent dampness.
- 3 a Many minerals in the lithosphere have water molecules chemically bound into their structure.
b Pure water is found in vapour or as tiny droplets in clouds in the atmosphere.
c The oceans and polar ice caps (part of the hydrosphere) are solutions of water with various dissolved minerals.
- 4 Density of water is 1000 kg/m³, density of ice is 920 kg/m³.
- 5 The density of the jewellery is the most distinguishable physical property. Immerse the jewellery in a container of water and collect the overflowing water. Measure this volume. It will be equal to the volume of the jewellery. Weigh the jewellery and calculate its density. (Archimedes discovered that any object immersed in water displaces a volume of water equivalent to its own volume.)
- 6 a 22 500 kg/m³ b 225 g c 0.022 m³ or 22 L

INVESTIGATION 10: Physical properties of water

 Assessment team: _____ Date: _____ Year/group: **11**

 Ref: *Heinemann Chemistry Practical Manual*

Chemicals used	Conc. (mol/L)	Amount	User code	DG class/haz	Procedure as per Appendix D?	What are the hazards?
ethylene glycol	pure	250 mL	7-12	DHS CW	yes	Toxic by all routes of exposure
acetone	pure	250 mL	7-12	3 CW	yes	Highly flammable, irritant vapour, skin irritant



Disposal of waste

Reuse pure liquids from station 5. All other solutions can be washed down the sink.

How are risks controlled?

- wear safety glasses
- use gloves
- fume cupboard
- wash hands after use

Assessment of risk:

Risk for this investigation is **not significant**, provided appropriate control measures are in place as indicated above.

Approved by: _____ Date: _____

Investigation 11

Solubility of a range of substances

Rationale

Students investigate the solubilities of various compounds and attempt to recognise that polar substances dissolve in water (polar solvent), while non-polar substances do not dissolve in a polar solvent. Students also look at the interaction of each class of compound with water on a molecular level.



Syllabus

Perform a first-hand investigation to test the solubilities in water of a range of substances that include ionic, soluble molecular, insoluble molecular, covalent networks and large molecules.

Background knowledge

Students have learned about major classes of compounds and their structures: ionic, polar covalent, molecular, macromolecular and covalent network solids. They should also know how to interpret the colour of universal indicator.

For the first time, students will be preparing the pre-lab safety form by referring to MSDS sheets provided by the teacher.

Hints

- Test dilute hydrochloric acid, without further dilution, for conductivity and pH. You may demonstrate conductivity and pH of ammonia solution, as well.
- Blue copper sulfate solution may mask the green colour of universal indicator, so try to find out its pH using a pH meter.

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Investigation 14

Qualitative analysis of heavy metals in industrial waste water

Rationale

Students get involved in local environmental issues and learn about the importance of monitoring their local environment. They will investigate, individually or in groups, a selected industrial site to analyse discharged water. Students will plan, design and carry out a qualitative analysis for the presence of heavy metals, using drinking water as a control.

Students will carry out a risk assessment record of their own investigation, which will clearly identify and assess control measures.

Syllabus

Gather and process information from first-hand investigations or secondary sources and use available evidence to discuss the importance of monitoring concentrations of heavy metals in industrial wastes.

Background knowledge

Students should know about the type of industrial processes that may produce heavy metals as waste products. They should have some idea of how industries manage their industrial waste. Students will apply the knowledge gathered in previous investigations to identify heavy metal cations in polluted water.

Hints

- Give students a blank sample risk assessment record to complete for their own practical investigation. Refer them to Appendix D in the CSIS package, volume 2, or appropriate MSDS sheets.
- Collecting discharged water before it reaches the local creek or sea may be difficult. When there is discharge from several manufacturers in the area, identification of a particular company may prove difficult. For these reasons, you could prepare 'polluted water' in the laboratory from a 'phantom' site.
- If a local creek is suspect, you must accompany students when water is collected. Anyone collecting water samples must wear gloves.
- Students will not be able to identify each heavy metal ion, but the idea is to confirm their presence or absence and identify the need for monitoring concentrations of heavy metals.
- Even if students obtain all negative results, the value of the exercise is not diminished as they will be able to research more reliable and accurate methods of monitoring waste water.
- Students write up a scientific report of their investigation. The Aim is to test a water sample for the presence of heavy metals, and compare the results with drinking water. Students write their own procedure in point form. They may choose to test with sodium sulfide only, or use other reagents such as carbonates and hydroxides. In conclusion, students make a qualitative statement and discuss the reliability of their results in terms of the solubility of the salts of heavy metals, and the use of control samples (see Discussion below).

- Suggested answers to Procedure step 4 are as follows:
 - a Cadmium causes brittle bones, liver and kidney damage. Chromium(VI) salts are carcinogenic. Lead attacks the nervous system, and retards brain activities and the physical development of children. Mercury causes brain damage, and attacks the nervous system, kidney and liver.
 - b Cadmium is used in the manufacture of nickel-cadmium batteries. Chromium is used in the processing of leather. Lead is used in the production and recycling of lead-acid batteries. Mercury is used in the paper industry, and in chlorine and sodium hydroxide production by the electrolysis of brine.
 - c In the 1960s, a local paper manufacturer in Minamata Bay, Japan, was continuously discharging mercury in the bay, which was the main supply of fish for this region. Thousands of people eating contaminated fish became ill with so-called 'mad cat' disease. Mercury in this area also caused birth defects in the children of affected parents.

d

TABLE 1
SYDNEY WATER LIMITS FOR DISPOSAL
OF HEAVY METALS INTO THE SEWER

Metal	Limit (g per day)
cadmium	4
chromium	8
copper	4
iron	80
lead	8
mercury	0.05
nickel	8

- e The Atomic Absorption Spectrophotometer or AAS (made by Varian Instruments Ltd) is used by many analytical laboratories for monitoring heavy metals in water samples.

RESULTS

TABLE 2 SAMPLE RESULTS TABLE

Sample	S ²⁻	OH ⁻	CO ₃ ²⁻
site 1			
site 2			
site 3			
drinking water	clear	clear	clear
0.1 M lead nitrate	black ppt	white ppt	white ppt

Discussion

- 1 $\text{Pb}^{2+} + \text{S}^{2-} \rightarrow \text{PbS(s)}$
 $\text{Pb}^{2+} + 2\text{OH}^- \rightarrow \text{Pb(OH)}_2\text{(s)}$
 $\text{Pb}^{2+} + \text{CO}_3^{2-} \rightarrow \text{PbCO}_3\text{(s)}$
- 2 The control samples are drinking water and 0.1 M lead nitrate, or any other heavy metal solution. Controls are necessary to establish if the selected reagent works, and to find out if the amount of precipitate in the sample water is more than in the reference (drinking water).
- 3 The validity is checked against repeated tests of the samples and control. If there is slight precipitate in a sample and none in the control (drinking water), we can conclude that heavy metals are present.

- 4 AAS is recommended to identify and quantify heavy metals.
- 5 It is important to identify the source of the water in terms of the industrial processes being carried out there.
- 6 Whether the results are positive or negative, it is advisable to find out (by talking to technical experts) what procedures are employed by the manufacturer to recycle, re-use

- or dispose of heavy metals, in order to gain an insight into the effective disposal or recycling of heavy metals.
- 7 Each student in a group must identify precisely his or her responsibility and contribution to the overall results. For example, two students collect samples while two prepare reagents, then each carries out at least one sample test and control test.

Sample risk assessment record

INVESTIGATION 14: Qualitative analysis of heavy metals in industrial waste water

Assessment team: _____ Date: _____ Year/group: **11**

Ref: *Heinemann Chemistry Practical Manual*

Chemicals used	Conc. (mol/L)	Amount	User code	DG class/haz	Procedure as per Appendix D?	What are the hazards?
sodium hydroxide	0.5	1 mL	7-12	8. DHS	yes	Corrosive to skin and eyes
sodium carbonate	0.5	1 mL	7-12	DHS	yes	Slightly toxic if ingested
lead nitrate	0.1	1 mL	7-12	5.1/6.1	yes	Highly toxic by all routes of exposure
lead hydroxide	solid	< 0.1 g	11-12	6.1 DHS	yes	Highly toxic by all routes of exposure
lead carbonate	solid	< 0.1 g	11-12	6.1 DHS	yes	Highly toxic by all routes of exposure
lead sulfide	solid	< 0.1 g	11-12	6.1 DHS	yes	Highly toxic by all routes of exposure



Disposal of waste

Solutions can be washed down the sink. Re-use plastic wells.

How are risks controlled?

- wear safety glasses
- use gloves when collecting water sample
- wash hands after use

Assessment of risk:

Risk for this investigation is **not significant**, provided appropriate control measures are in place as indicated above.

Approved by: _____ Date: _____