BOILING LIQUIDS AT REDUCED PRESSURE

Water boils at significantly less than 100°C when the pressure above it is reduced.

- **EQUIPMENT** thermometer (-10°C to +110°C)
 - 1 L round bottomed flask (thick walled)
 - 2-holed rubber stopper to fit round bottomed flask
 - 3-way stop cock
 - boiling chips
 - tripod
 - bunsen burner
 - retort stand
 - clamp and boss head
 - water pump, with trap and vacuum tubing
- **REAGENTS** tap water (500 mL)
- **PREPARATION** Insert the thermometer through one hole of the 2-holed rubber stopper and insert one arm of the 3-way stop cock through the other hole.
 - Half fill the flask with tap water and add several boiling chips.
 - Firmly place the stopper in the mouth of the flask and adjust the thermometer so that the bulb is below the surface of the water.
 - Place the flask on the tripod, open the stop cock and heat the flask with the bunsen burner until the temperature is 50–60°C.
 - Remove the flask from the tripod and clamp it to the stand.

PROCEDURE • Attach the vacuum tubing from the trap to one of the open arms of the stop cock.

- Connect the trap to the pump, turn the pump to maximum water flow and adjust the stop cock so that air is drawn from the flask.
- Focus the camera on the thermometer and read the temperature that the water boils at.
- **RESULTS** When the pressure inside the flask is lowered the water will boil at less than 100°C. As the water boils the temperature decreases and the boiling will stop.

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PHASE CHANGE UNDER PRESSURE

The passing of a weighted wire through a block of ice with the block of ice remaining intact shows how, under the influence of pressure, localised melting can occur without a change in temperature.

- **EQUIPMENT** tripod, 40 cm tall
 - 2 rulers, 30 cm long, wood or plastic
 - Nichrome wire, 29 BNS, half a metre
 - 2 kg weight
 - ice-cream container, plastic, 17 x 17 x 9 cm deep

REAGENTS • water (200 mL)

• dry ice, solid CO₂ (a big piece)

PREPARATION • Make a hemi-cylindrical block of ice by placing 100 to 200 mL of water into the ice-cream container.

• Put the container into a freezer resting on one *edge*.

PROCEDURE • Remove the ice by twisting the container.

- Pass the wire-loop up through the centre of the tripod and slip it over the block of ice.
- Rest the ice across the rulers, rounded surface uppermost.
- Allow the 2 kg weight to hang down freely so that it is fully supported by the ice block.
- Repeat the above procedure using a suitably shaped block of solid carbon dioxide.
- **RESULTS** During the following six to eight minutes the weighted wire will melt a path through the ice until it has completely passed through. Weight and wire will crash to the bench below. However, the path forced through the ice will have immediately refrozen behind the wire: the ice block is once again intact. This results from the melting point of water decreasing with increasing pressure.

The weighted wire will not pass through the "dry ice" as the melting point of carbon dioxide increases with increasing pressure.

CAUTION

Solid CO_2 sublimes at -78°C and can cause frostbite.

LE CHATELIER'S PRINCIPLE: EFFECT OF TEMPERATURE ON EQUILIBRIUM

Three glass bulbs containing similar concentrations of N_2O_4 and NO_2 are subjected to temperature changes. The intensity of the brown colour alters accordingly.

EQUIPMENT

3 sealed glass bulbs containing N_2O_4/NO_2



- 4 x 1 L beakers (large enough to allow the glass bulbs to rest comfortably on the rim)
- crystallising dish, large enough to allow glass bulb to fit inside
- hot-plate
- light box
- crushed ice
- salt

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- hot water
- **PREPARATION** In one of the beakers, bring 500 mL of water to the boil and maintain its temperature on the hot-plate.
 - Place crushed ice and some salt into the crystallising dish.
 - Set up the light box.
 - Rest each glass bulb on the rim of an empty beaker and place it beside the light box.
- **PROCEDURE** Place the beakers with the three bulbs on the light box and note the uniformity of the colour of their contents.
 - Place one of the bulbs over the beaker with boiling water, set another into the crushed ice/salt mixture and maintain the third at room temperature.
 - After some time, compare the contents of the three bulbs once more.
- **RESULTS** The colour of the heated gas will darken and that of the cooled gas will become pale.

LE CHATELIER'S PRINCIPLE: EFFECT OF CONCENTRATION

Shifts in equilibrium caused by changes in concentration can be observed by means of a reaction which has a pronounced colour change.

- EQUIPMENT
- 4 conical flasks: 1 x 3 L, 2 x 1 L
- 2 x 100 mL measuring cylinders
- light box •

REAGENTS

- iron(III) chloride, FeCl₂ (1.2 M, 100 mL)
- ammonium thiocyanate, NH, SCN (1 M, 100 mL)
- distilled water

PREPARATION . Dissolve iron(III) chloride (19.46 g) in water (100 mL) in a measuring

- cylinder.
- Dissolve ammonium thiocyanate (7.61 g) in water (100 mL) in a • measuring cylinder.
- Place the iron(III) solution (1.5 mL) and the thiocyanate solution (1.5 mL) into the 3 L flask. Make up to 2 L with distilled water.
- Using the 1 L flasks divide this solution into two. •
- Set up the light box and place the flasks and cylinders on it.
- PROCEDURE Add 50 mL of iron solution to one of the flasks and 50 mL of • thiocyanate solution to the other.

RESULTS Addition of iron(III) will cause the system to turn dark red.

 $[Fe(OH_2)_{e}]^{3+}(aq) + SCN^{-}(aq) \zeta [Fe(OH_2)_{e}(SCN)]^{2+}(aq) + H_2O(I)$

Addition of thiocyanate will also intensify the colour.

You could also use solid sodium fluoride to show the reverse reaction where you end up in a slight yellowish solution in the flask.

CARBONATE REACTIONS

Dry ice (solid carbon dioxide) is added to a flask of calcium hydroxide. A precipitate of calcium carbonate forms, redissolves, and re–forms when the flask is heated.

- EQUIPMENT 3 measuring cylinders: 2 x 250 mL, 1 x 500 mL
 - 1 x 500 mL flask
 - hot-plate

REAGENTS • 300 mL clear limewater (calcium hydroxide solution, Ca(OH)₂).

- Prepare by adding a few teaspoonfuls of calcium hydroxide or calcium oxide to 1 L water.
- Prepare the demonstration one day before to allow any solid present to settle.
- dry ice, solid CO₂.

PROCEDURE • Place 300 mL of clear limewater in a 500 mL flask.

- Drop in a chunk of dry ice.
- Observe the formation of a precipitate and the redissolving to form a clear solution again.
- Heat over a low flame or on a hot plate.

Solid CO, sublimes at -78°C and can cause frostbite.

CAUTION

RESULTS

1) Carbon dioxide reacts with water to form CO_3^{2-} ions through a series of equilibria.

 $CO_2(g) + H_2O(I) \oplus H_2CO_3(aq)$ $H_2CO_3(aq) \oplus H^+(aq) + HCO_3^-(aq)$

 $HCO_{3}^{-}(aq) \Phi H^{+}(aq) + CO_{3}^{2-}(aq)$

2) If calcium ions are in excess, the carbonate ions react with the calcium ions to form insoluble calcium carbonate.

 $Ca^{2+}(aq) + CO_3^{2-}(aq) \Phi CaCO_3(s)$

3) The additional dissolving of carbon dioxide and formation of acid means that acid is now in excess causing the calcium carbonate to dissolve.

$$CaCO_{3}(s) + CO_{2}(aq) + H_{2}O(l) \Phi Ca^{2+}(aq) + 2HCO_{3}^{-}(aq)$$

4) Upon heating, carbon dioxide is driven from the solution, acidity is decreased, and calcium carbonate precipitates again.

DISTRIBUTION OF IODINE: COMPETING EQUILIBRIA

The distribution of iodine between an organic and an aqueous layer is independent of the direction in which equilibrium is approached.

EQUIPMENT • 2 large tes

- 2 large test tubes with rubber stoppers
- test tube rack
- 2 x 50 mL measuring cylinders
- spatula
- light box

REAGENTS

- potassium iodide, KI (1 M, 50 mL)
 - 50 mL organic solvent (toluene)
 - iodine (0.02 g)

PREPARATION • Dissolve potassium iodide (8.3 g) in water (50 mL).

- Place two very small crystals of iodine (about the size of a pinhead) of similar size, one in each test tube.
- To one test tube add potassium iodide solution (25 mL); to the other organic solvent (25 mL).
- Stopper both tubes and shake until the iodine dissolves completely. (This may take some time.)
- **PROCEDURE** Display both test tubes on the light box and draw attention to the contrast in colour.
 - Then add organic solvent (25 mL) to the test tube containing potassium iodide solution, and potassium iodide solution (25 mL) to the test tube containing organic solvent.
 - Stopper both tubes and shake.
 - Place both tubes on the light box and compare the colour intensities in both layers.
- **RESULTS** The colour intensities of both layers in each test tube should be identical. In the aqueous layer, the iodine forms the tri-iodide ion,

$$I_2(s) + I(aq) \Phi I_3(aq)$$

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I_2(\text{org}) \Phi I_2(\text{aq})
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The iodine is slightly soluble in water and more soluble in the organic solvent. The overall equilibrium position will be the result of the two competing equilibria above.

METATHESIS REACTION BETWEEN TWO SOLIDS

Two dry white solids are mixed together in a beaker without heating. A yellow solid forms.

- EQUIPMENT 1 L beaker
 - stirring rod

REAGENTS

- lead nitrate, Pb(NO₃)₂ (50 g)
- potassium iodide, KI (50 g)

PROCEDURE

- Mix 50 g of each of the two compounds in a beaker with a glass stirring rod.
- No heat is required.

RESULTS

• When the two compounds are mixed, lead iodide is formed.

 $Pb(NO_3)_2(s) + 2KI(s) \zeta PbI_2(s) + 2KNO_3(s)$

white white yellow white

DISPOSAL Add 10% sodium sulfide solution slowly to the contents until precipitation is complete. Filter, and pour the filtrate down the sink. The black precipitate of PbS should be disposed of in a solid waste container.

DEMONSTRATION 5.8

"NATURAL" pH INDICATORS

EQUIPMENT	• 3 x 300 mL beakers for each pigment to be tested
REAGENTS	 hydrochloric acid, HCl (1 M) sodium hydroxide, NaOH (1 M) sodium hydrogencarbonate, NaHCO₃ (solid) as a pH 7 buffer ethanol (600 mL) red cabbage red roses or any bright coloured flower(by request)
PROCEDURE	 Extract the pigment from the flowers by soaking them in ethanol. Boil red cabbage in 600 mL of water to extract the purple colour. Pour about 200 mL of each solution into each of three beakers. Add HCl to one, NaOH to the other and pH 7 buffer to the third.
RESULTS	Each of the pigments has a distinct alkaline and acid colour and can therefore be used as a pH indicator.

Relative rates of reaction of strong and weak acids

This demonstration illustrates the relative rates of reaction of two different acids of the same concentration with magnesium metal .

- **EQUIPMENT** 2 x 250 mL conical flasks
 - 2 balloons (one purple and one red)
 - lightbox

REAGENTS

- hydrochloric acid, HCl (1 M, 150 mL)
- acetic acid, CH₃COOH (1 M, 150 mL)
- magnesium metal, Mg (2 x 2 g)

PREPARATION • Place 2 g of the magnesium metal in each balloon.

- Pour the hydrochloric acid (150 mL) into one flask and the acetic acid (150 mL) into the other flask.
- Attach the neck of each balloon to the mouth of the respective conical flask (the purple balloon for the weak acid and the red balloon for the strong acid), making sure that none of the magnesium in the balloons spills into the flasks.
- Place the two flasks on the lightbox.
- **PROCEDURE** Simultaneously dump the magnesium from the balloons into the flasks.
- **RESULTS** In each case the magnesium metal and the acid react to form hydrogen gas. However the red balloon is filled up faster then the purple one by the evolving hydrogen gas.

The equation for the reaction of magnesium with acid is:

Mg(s)+ 2H⁺(aq) $\zeta \Box H_2(g)$ + Mg²⁺(aq)

Thus the rate of evolution of $H_2(g)$ depends on the concentration of $H^+(aq)$ in the acid solution. The rate of reaction with magnesium metal is much faster for the strong acid than the weak acid, since the strong acid has a greater concentration of $H^+(aq)$.

ACIDIFICATION OF SODIUM HYDROXIDE WITH "DRY ICE"

The change in pH that occurs when CO₂ dissolves in water can be demonstrated with universal indicator by the addition of dry ice to sodium hydroxide solution.
 EQUIPMENT

 2 L beaker (tall thin type)
 tongs
 large universal indicator/pH chart

 REAGENTS

 distilled or deionised water (1000 mL)
 sodium hydroxide (1 M)
 universal indicator

• dry ice, solid CO_2 (enough to fill a 500 mL beaker)

PREPARATION • Place 1 L of distilled water into the beaker and add universal indicator.
 Add 1 M sodium hydroxide solution until the pH reaches 12 (purple).

- **PROCEDURE** Add solid CO₂.
 - Observe colour changes.

RESULTS Over the next few minutes, the pH will drop steadily to below pH 4.

 $CO_2(s) \Phi CO_2(g) \Phi CO_2(aq) \Phi H_2CO_3 \Phi H^+ + HCO_3^-$

 $H^{+}(aq) + OH^{-} \zeta \Box H_{2}O$

BUFFERED AND NON-BUFFERED SOLUTIONS

The difference between buffered and non-buffered solutions is shown by taking one of each solution, at the same pH, adding universal indicator to both then adding acid or base and noting the colour change or lack thereof.

- **EQUIPMENT** 1 x 2 L beaker
 - 6 x 1 L beakers
 - 2 x 50 mL measuring cylinders
 - 4 glass stirring rods
 - light box
- **REAGENTS** potassium dihydrogenphosphate, KH₂PO₄ (150 g)
 - sodium hydrogenphosphate (anhydrous), Na₂HPO₄ (150 g)
 - universal indicator
 - sodium hydroxide, NaOH (0.1 M, 100 mL)
 - hydrochloric acid, HCl (0.1 M, 100 mL)
 - deionised water
 - large universal indicator/pH chart

PREPARATION For the buffer prepare the following solutions:

- Using one of the 1 L beakers, dissolve 150 g of potassium dihydrogenphosphate in water and make up to 800 mL.
- In another 1 L beaker dissolve 150 g of sodium hydrogenphosphate (anhydrous) and make up to 800 mL.
- Add universal indicator to both solutions.
- In the 2 L beaker, combine 700 mL of the (basic) sodium hydrogenphosphate solution with 600 mL of the (acidic) potassium dihydrogenphosphate solution.
- Divide this buffer solution into two equal parts and place these in two of the 1 L beakers.
- Place similar volumes of plain water into two further 1 L beakers.
- Add universal indicator to both and, if necessary, adjust to pH 7 with small amounts of acid or hydroxide solution.
- Label all beakers clearly and line these up on the lightbox.
- Supply acid and hydroxide solutions in flasks and place one (labelled) measuring cylinder with each .
- Add 25 to 50 mL of acid solution to one of the non-buffered solutions and the same amount to one of the buffered solutions.
 - Add between 25 and 50 mL of alkaline solution to the other nonbuffered solution and to the second buffered solution.
- **RESULTS** Dramatic colour changes occur in the non-buffered liquids. The buffered systems remain green:

 $H_2PO_4^{-}(aq) + OH^{-}(aq) \oplus HPO_4^{2-}(aq) + H_2O(I)$

 $HPO_4^{2-}(aq) + H^+(aq) \Phi H_2PO_4^{-}(aq)$

ACID-BASE INDICATORS: A CARBON-DIOXIDE-ACTIVATED CHEMICAL REACTION

The addition of carbon dioxide to a solution changes the colour of the indicator, showing that the solution has become acidic.

EQUIPMENT • 2 x 500 mL conical flasks

ethanol, CH₃CH₂OH (95%, 250 mL)



- sodium hydroxide, NaOH (1 M, 3 mL)
- thymolphthalein indicator
- phenol red indicator

PREPARATION • Place ethanol (about 250 mL) in a 500 mL conical flask.

- Add 5-6 drops of thymolphthalein indicator and just enough dilute sodium hydroxide to produce a very pale blue colour (approx' 3 mL).
 Stopper the flask until it is used.
- Stopper the flask until it is used.
 Prepare the second solution as directed above, except use 1-2 drops
- of phenol red in 250 mL of water.Add half a drop of sodium hydroxide to produce a red solution.
- Be sure to protect the solution from CO_2 in the air prior to the demonstration.

• The two flasks containing the coloured solutions are passed around the class, the red solution up one side of the room and the blue solution up the other side of the room.

- Each student is invited to remove the stopper, speak into the flask, and politely request the colour to change.
- **RESULTS** After approximately 30 students have talked to the solution, the red solution will suddenly turn clear with a yellow tinge!

 $H_2O(I) + CO_2(g) \oplus H_2CO_3(aq) \oplus H^+(aq) + HCO_3^-(aq)$

The blue solution will take **much** longer to change colour, but it will eventually go clear. Swirling speeds up the reaction.

SALTS OF ACIDS AND BASES

The pH of solutions of various salts are determined.

- EQUIPMENT pH meter
 - large universal indicator/pH chart
 - liaht box
 - 10 x 500 mL beakers
 - 10 stirring rods
 - pH electrode

REAGENTS

- universal indicator
 - freshly deionised water (or freshly boiled out water) **

- sodium hydroxide, NaOH (1 M, 30 mL)
- sodium carbonate, Na₂CO₃ (10 g)
- sodium hydrogencarbonate, NaHCO₂ (10 g)
- sodium chloride, NaCl (10 g)
- ammonium chloride, NH₂Cl (10 g)
- ammonium acetate, CH₃COONH₄ (10 g)
- aluminium sulfate, $AI_2(SO_4)_3$ (10 g)
- potassium hydrogensulfate, KHSO₄ (10 g)
- hydrochloric acid, HCI (1 M, 30 mL)

Place the water (270 mL) in the beakers and add universal indicator. **PREPARATION** •

- Put the beakers on the light box, with the stirring rods.
- Place the solids and solutions in sample tubes, in order of decreasing • pH.
- PROCEDURE Add the substances to the beakers, one by one, with stirring.
 - Observe the colour changes and measure the pH.
- RESULTS The salts are arranged in order of increasing pH so that their colours with universal indicator form a rainbow.

SOLUBILITY AND COMPLEX ION EQUILIBRIA OF SILVER: THE "ONE-POT DEMONSTRATION"

The sequential addition of clear, colourless solutions alternately precipitates and redissolves.

- **EQUIPMENT** 1 L conical flask
 - 6 x 10 mL & 2 x 25 mL measuring cylinders (stoppered)
 - 1 x 50 mL & 1 x 100 mL measuring cylinders (stoppered)
 - safety glasses
 - light box

REAGENTS

Prepare the solutions as set out below. Note that the iodide and thiosulfate solutions must be made up freshly. The remainder may be made up in advance provided that the silver nitrate solution is kept in a dark bottle.



1.	sodium hydroxide	1 M	2 mL
2.	sodium hydrogenphosphate	1 M	5 mL
3.	nitric acid	5 M	45 mL
4.	sodium chloride	1 M	3 mL
5.	ammonia	concentrated	50 mL
6.	potassium bromide	1 M	3 mL
7.*	sodium thiosulfate	1 M	30 mL
8.	sodium sulfide	1 M	5 mL

* to be made fresh.

- **PREPARATION** Place the solutions into the appropriate measuring cylinders which must be clearly numbered as indicated.
- **PROCEDURE** Line up the measuring cylinders in their numeric sequence.
 - Place the flask of silver nitrate solution (start with 400ml of 0.1M) on the light box.
 - Add the solutions to the flask one by one swirling gently after each addition. It is essential that the numeric sequence is strictly adhered to!

High Risk Demonstration:

- Refer to HIRAC
- Set up in Red Tray

Chapter 5: Equilibrium

RESULTS	The following	change should	occur after	each addition:
	ine renewing	onango onoaia	oooar artor	ouon addition.

	OBSERVATION	K_{sp} or K_{stab}
Ag⁺ + NO ₃ -	colourless solution	
χ □NaOH (1)	χ	
AgOH	Brown precipitate	10-7.70
χ Na ₂ HPO ₄ (2)	χ	
Ag ₃ PO ₄	Yellow precipitate	10 -16
χ □HNO₃ (3)	χ	
3Ag+ + NO ₃ - + HPO ₄ -	Precipitate dissolves	
χ NaCl (4)	χ	
AgCl	White precipitate	10-9.74
χ NH ₃ (aq) (5)	χ	
[Ag(NH ₃) ₂]+ + Cl-	Precipitate dissolves	10+7
χ KBr (6)	χ	
AgBr	Pale yellow precipitate	10 -12.3
$\chi \Box Na_2S_2O_3$ (7)	χ	
3Na⁺ + [Ag(S₂O₃)₂]³-	Precipitate dissolves	10 ⁺¹³
χ	χ	
Ag ₂ S	Black precipitate	10-50

CAUTION

A comparatively large quantity of cyanide is produced. Do not leave it unattended at any stage.

Once the demonstration is complete, add sodium hypochlorite solution and allow to stand for 24 hours, then discard.