



IB Chemistry Investigations

Volume 2 (Higher Level)

COPY MASTERS

(For use with the IB Diploma programme)

(Fourth edition)

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Series editor: David Greig

Table of Contents

Standard Level

Investigation 1A – DETERMINING THE AMOUNT OF WATER OF CRYSTALLIZATION IN A HYDRATED SALT	1
Investigation 1B – DETERMINING THE COMPOSITION OF ‘COPPER(II) CARBONATE’ BY HEATING	5
Investigation 1C – HOW CONCENTRATED IS ‘CONCENTRATED’ NITRIC ACID?	7
Investigation 1D – DETERMINING THE MOLAR MASS OF A SOLUBLE ACID	10
Investigation 1E – IDENTIFYING FUELS FROM THEIR MOLAR MASSES	13
Investigation 2A – OBSERVING EMISSION SPECTRA	20
Investigation 3A – SOME REACTIONS OF THE HALOGENS	22
Investigation 4A – FORCES BETWEEN PARTICLES AND PHYSICAL PROPERTIES	26
Investigation 5A – MEASURING ENTHALPY CHANGES	31
Investigation 5B – INDIRECTLY DETERMINING AN ENTHALPY CHANGE APPLYING HESS’ LAW	36
Investigation 6A – TECHNIQUES FOR MEASURING REACTION RATES	40
Investigation 6B – FACTORS AFFECTING RATES OF CHEMICAL REACTIONS	49
Investigation 6C – A QUANTITATIVE INVESTIGATION OF REACTION RATES	56
Investigation 7A – AN INTRODUCTION TO EQUILIBRIUM	60
Investigation 7B – FACTORS AFFECTING VAPOUR PRESSURE	62
Investigation 7C – SHIFTING THE POSITION OF AN EQUILIBRIUM	65
Investigation 8A – DETERMINING THE SOLUBILITY OF ETHANEDIOIC (OXALIC) ACID	70
Investigation 8B – THE PERCENTAGE OF NITROGEN IN A FERTILISER	75
Investigation 8C – STRONG AND WEAK ACIDS AND BASES	78
Investigation 9A – COMMON OXIDISING AND REDUCING AGENTS	85
Investigation 9B – BIOCHEMICAL OXYGEN DEMAND (BOD) BY REDOX TITRATION	91
Investigation 9C – AN INTRODUCTION TO ELECTROCHEMISTRY	95
Investigation 10A – INVESTIGATING ORGANIC STRUCTURES USING MODELS	99
Investigation 10B – SOME REACTIONS OF HYDROCARBONS	106
Investigation 10C – REACTIONS OF ALCOHOLS AND HALOGENOALKANES	110
Investigation 11A – ANALYSIS OF ORGANIC SPECTRA	115
Investigation 22A (Option A) – INVESTIGATING CATALYSTS	132
Investigation 23A (Option B) – THE CHROMATOGRAPHIC SEPARATION OF AMINO ACIDS	136
Investigation 24A (Option C) – THE ENTHALPY OF COMBUSTION OF FUELS	138
Investigation 25A (Option D) – THE SYNTHESIS OF ASPIRIN	141

Higher Level

Investigation 13A – SOME CHEMICAL REACTIONS OF TRANSITION METALS	145
Investigation 15A – ENTHALPIES OF SOLUTION	155
Investigation 15B – THE ENTHALPY CHANGE OF A DISPLACEMENT REACTION	159
Investigation 15C – DETERMINING ENTROPY CHANGES	163
Investigation 16A – DETERMINING THE RATE EXPRESSION FOR A REACTION	169
Investigation 16B – DETERMINING THE ACTIVATION ENERGY FOR A REACTION	173
Investigation 17A – MEASURING AN EQUILIBRIUM CONSTANT BY LIGHT ABSORPTION	176
Investigation 17B – INVESTIGATION OF THE ESTER FORMATION EQUILIBRIUM	179
Investigation 18A – SOME MEASUREMENTS ON WEAK ACIDS AND BASES	182
Investigation 18B – pH CHANGES DURING ACID-BASE TITRATIONS	185
Investigation 18C – DETERMINING THE pK_a OF AN INDICATOR	190
Investigation 19A – INVESTIGATING ELECTRODE POTENTIALS	194
Investigation 19B – INVESTIGATING THE ELECTROLYSIS OF AQUEOUS SODIUM CHLORIDE	199
Investigation 19C – SOME FACTORS AFFECTING ELECTROLYSIS	204
Investigation 20A – FACTORS THE HYDROLYSIS OF HALOGENOALKANES	210
Investigation 20B – A TWO STAGE ORGANIC SYNTHESIS	214
Investigation 22B (Option A) – MEASURING K_{sp} FOR A SPARINGLY SOLUBLE SALT	219
Investigation 23B (Option B) – ENZYME ACTIVITY AND THE MICHAELIS CONSTANT	224
Investigation 24B (Option C) – SOME FACTORS AFFECTING VOLTAIC CELLS	228
Investigation 25B (Option D) – RAOULT'S LAW AND FRACTIONAL DISTILLATION	233

This practical is designed to investigate the chemical reactions of selected first row transition metals, looking particularly at their oxidation states and the complex ions that they form.

METHOD & DATA COLLECTION

For each part of the experiment, record what you observe and attempt to explain these results in terms of appropriate theory, writing balanced equations (preferably ionic ones) or half equations in the case of redox reactions, for the reactions that occur.

1. Vanadium

- a) Fill a test tube to a depth of 4 cm with an acidified solution containing vanadate(V) ions. Add a piece of granulated zinc and observe the colour of the solution throughout the course of the rest of the Investigation.

Observations

Interpretation

Equation(s)

2. Chromium

- a) Fill a test tube to a depth of 2 cm with a solution containing chromium(III) ions. Add a few drops of aqueous sodium hydroxide and then an excess of sodium hydroxide. **Save the product for part b.**

Observations

Interpretation

Equation(s)

- b) Transfer the product from a) to a boiling tube, add an equal volume of aqueous hydrogen peroxide and warm gently.

Observations

Interpretation

Equation(s)

SAMPLE PAGES

- c) Fill a test tube to a depth of 2 cm with a solution containing chromate(VI) ions. Add dilute sulfuric acid a few drops at a time until a colour change occurs. Now add aqueous sodium hydroxide until there is a further colour change.

Observations

Interpretation

Equation(s)

3. Manganese

- a) Fill a test tube to a depth of 2 cm with a solution containing manganese(II) ions. Add a few drops of aqueous sodium hydroxide and then an excess of sodium hydroxide. Shake the test tube for a minute or two, observing the precipitate carefully.

Observations

Interpretation

Equation(s)

SAMPLE PAGES

- b) Fill a test tube to a depth of 2 cm with aqueous hydrogen peroxide and add a little manganese(IV) oxide. Test the gas evolved with a glowing splint. Note any changes to both the solution and the solid.

Observations

Interpretation

Equation(s)

- c) Put a 2 cm depth of aqueous potassium manganate(VII) into three test tubes. To one add an equal volume of dilute sulfuric acid. To the second, add a pellet of solid sodium hydroxide and leave the third unchanged. Now, add two drops of cyclohexene to all three and shake to mix the layers (Note – the double bond in alkenes is easily oxidised).

Warning: Do not handle solid sodium hydroxide with your fingers!



Observations

Interpretation

Equation(s)

4. Iron

- a) Fill a test tube to a depth of 2 cm with a solution containing iron(II) ions. Add a few drops of aqueous sodium hydroxide and then an excess of sodium hydroxide. Leave the test tube to stand for about ten minutes and then observe closely what happens near the surface.

Observations

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Interpretation

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Equation(s)

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- b) Fill a test tube to a depth of 2 cm with a solution containing iron(III) ions. Add a few drops of aqueous sodium hydroxide and then an excess of sodium hydroxide.

Observations

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Interpretation

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Equation(s)

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SAMPLE PAGES

- c) Put solutions containing iron(II) ions and iron(III) ions into two separate test tubes and then add a few drops of aqueous potassium thiocyanate to both.

Observations

Interpretation

Equation(s)

5. Cobalt

- a) Fill a test tube to a depth of 2 cm with a solution containing cobalt(II) ions. Add concentrated hydrochloric acid, a drop at a time, until there is no further colour change. Add water, a little at a time, until the colour change is **just** reversed. Now warm the solution gently.

Warning: Concentrated acids are very corrosive, take precautions!

**Observations**

Interpretation

Equation(s)

6. Nickel

- a) Fill a test tube to a depth of 2 cm with a solution containing nickel(II) ions. Add concentrated hydrochloric acid, a drop at a time, until there is no further colour change.

Observations

Interpretation

Equation(s)

- b) Fill a test tube to a depth of 2 cm with a solution containing nickel(II) ions. Add a few drops of aqueous ammonia and then an excess of the reagent.

Observations

Interpretation

Equation(s)

SAMPLE PAGES

7. Copper

- a) Fill a test tube to a depth of 2 cm with a solution containing copper(II) ions. Add a few drops of aqueous sodium hydroxide and then an excess of sodium hydroxide.

Observations

Interpretation

Equation(s)

- b) Fill a test tube to a depth of 2 cm with a solution containing copper(II) ions. Add a few drops of aqueous ammonia and then an excess of the reagent.

Observations

Interpretation

Equation(s)

SAMPLE PAGES

- c) Fill a test tube to a depth of 2 cm with a solution containing copper(II) ions. Add concentrated hydrochloric acid, a drop at a time, until there is no further colour change.

Observations

Interpretation

Equation(s)

- d) One quarter fill a boiling tube with an aqueous solution of copper(II) ions and dissolve two spatulas of glucose in this solution. Heat the mixture to boiling and then, maintaining this temperature, add aqueous sodium hydroxide, a drop at a time, until there is no further change.

Observations

Interpretation

Equation(s)

SAMPLE PAGES

- e) Put a 2 cm depth of a solution containing copper(II) ions into a test tube. Add an equal volume of aqueous potassium iodide. Add aqueous sodium thiosulfate, a drop at a time, until there is no further colour change.

Observations

Interpretation

Equation(s)

SAMPLE PAGES

IB Chemistry Investigations

Volume 2 (Higher Level)

TEACHING NOTES

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Syllabus Correlation Table

No.	Title	Topic	Syllabus reference
1A	Determining the Water of Crystallisation	1.2	Obtaining and using experimental data for deriving empirical formulas from reactions involving mass changes.
1B	Determining the Composition of "Copper Carbonate"	1.2	
1C	How Concentrated is "Concentrated" Nitric Acid?	1.3	Use of the experimental method of titration to calculate the concentration of a solution by reference to a standard solution
1D	Determining the Molar Mass of a Soluble Acid	1.3	
1E	Identifying fuels from their molar masses	1.3	Obtaining and using experimental values to calculate the molar mass of a gas from the ideal gas equation.
2A	Observing Emission Spectra	2.2	Emission spectra could be observed using discharge tubes of different gases and a spectroscope. Flame tests could be used to study spectra.
3A	Some Reactions of the Halogens (Group 7)	3.2	Experiment with chemical trends directly in the laboratory or through the use of teacher demonstrations.
4A	Forces Between Particles and Physical Properties	4.1	Students could investigate compounds based on their bond type and properties.
5A	Measuring Enthalpy Changes	5.1	A calorimetry experiment for an enthalpy of reaction should be covered and the results evaluated. Experiments could include calculating enthalpy changes from given experimental data (energy content of food, enthalpy of melting of ice or the enthalpy change of simple reactions in aqueous solution).
5B	Using Hess' Law to Determine Enthalpy Changes	5.2	A calorimetry experiment for an enthalpy of reaction should be covered and the results evaluated. Experiments could include Hess's Law labs.
6A	Techniques for Measuring Reaction Rates	6.1	Investigation of rates of reaction experimentally and evaluation of the results. Experiments could include investigating rates by changing concentration of a reactant or temperature.
6B	Factors Affecting Rates of Chemical Reactions	6.1	Investigation of rates of reaction experimentally and evaluation of the results. Experiments could include investigating rates by changing concentration of a reactant or temperature.
6C	A Quantitative Investigation of Reaction Rates	6.1	Investigation of rates of reaction experimentally and evaluation of the results. Experiments could include investigating rates by changing concentration of a reactant or temperature.
7A	An Introduction to Equilibrium	7.1	The characteristics of chemical and physical systems in a state of equilibrium. Physical and chemical systems should be covered.
7B	An Introduction to Vapour Pressure	7.1	The characteristics of chemical and physical systems in a state of equilibrium. Physical and chemical systems should be covered.
7C	Predicting the Effect of Changes on an Equilibrium	7.1	Le Châtelier's principle can be investigated qualitatively by looking at pressure, concentration and temperature changes on different equilibrium systems.
8A	The Solubility of Ethanedioic (Oxalic) Acid	8.2	Candidates should have experience of acid-base titrations with different Indicators. The evidence for these properties could be based on a student's experimental experiences.
8B	Percentage of Nitrogen Content in a Fertilizer	8.2	Candidates should have experience of acid-base titrations with different Indicators. The evidence for these properties could be based on a student's experimental experiences.

No.	Title	Topic	Syllabus reference
8C	A Comparison of Strong and Weak Acids and Bases	8.4	Students should be familiar with the use of a pH meter and universal indicator. Students should have experimental experience of working qualitatively with both strong and weak acids and bases. Examples to include: H_2SO_4 (aq), HCl (aq), HNO_3 (aq), NaOH (aq), NH_3 (aq).
9A	Common Oxidizing and Reducing Agents	9.1	Experiments could include demonstrating the activity series, redox titrations and using the Winkler Method to measure BOD.
9B	Redox Titrations	9.1	Experiments could include demonstrating the activity series, redox titrations and using the Winkler Method to measure BOD.
9C	Investigating some Electrochemical Cells	9.2	Performance of laboratory experiments involving a typical voltaic cell using two metal/metal-ion half-cells.
10A	Investigating Organic Structures using Models	10.1	Construction of 3-D models (real or virtual) of organic molecules.
10B	Reactions of Hydrocarbons	10.2	Experiments could include distinguishing between alkanes and alkenes.
10C	Reactions of Organic Compounds containing Oxygen	10.2	Experiments could include reflux and distillation.
11A	Analysis of Organic Spectra	11.3	Deduction of information about the structural features of a compound from percentage composition data, MS, ^1H NMR or IR.
OPTION TOPICS			
22A	Investigating the behaviour of catalysts	A.3	Experiments could include investigating the decomposition of potassium sodium tartrate with cobalt chloride and the decomposition of hydrogen peroxide with manganese (IV) oxide.
23A	The chromatographic separation of amino acids	B.2	Experiments could involve identification of amino acid mixtures by paper chromatography.
24A	The enthalpy of combustion of fuels	C.1	The energy density of different fuels could be investigated experimentally.
25A	The synthesis of aspirin	D.2	Experiments could include the synthesis of aspirin.

Key to Required Practicals

No.	Topic	Requirement
1	Topic 1.2	Determination of the molecular formula of a compound from its empirical formula and molar mass.
2	Topic 1.3	Obtaining and using experimental values to calculate the molar mass of a gas from the ideal gas equation.
3	Topic 1.3	Use of the experimental method of titration to calculate the concentration of a solution by reference to a standard solution
4	Topic 5.1	A calorimetry experiment for an enthalpy of reaction should be covered and the results evaluated.
5	Topic 6.1	Investigation of rates of reaction experimentally and evaluation of the results.
6	Topic 9.2	Performance of laboratory experiments involving a typical voltaic cell using two metal/metal-ion half-cells.
7	Topic 10.1	Construction of 3-D models (real or virtual) of organic molecules.
8	Topic 15.1 / Topic 19.1	Perform lab experiments which could include single replacement reactions in aqueous solutions.

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No.	Title	Topic	Syllabus reference
13A	Some Chemical Reactions of Transition Metals	13.1 & 13.2	The oxidation states of vanadium and manganese, for example, could be investigated experimentally.
15A	Enthalpy of Solution	15.1	Enthalpy of solution, hydration enthalpy and lattice enthalpy are related in an energy cycle.
15B	Determining the enthalpy change of a reaction	15.1 / 19.1	Perform lab experiments which could include single replacement reactions in aqueous solutions.
15C	Determining Entropy Changes	15.2	Application of $\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ$.
16A	Determining the Rate Expression for a Reaction	16.1	Deduction of the rate expression for an equation from experimental data and solving problems involving the rate expression.
16B	Measuring the Activation Energy for a Reaction	16.2	Determining and evaluating values of activation energy and frequency factors from data.
17A	Measuring an Equilibrium Constant by Light Absorption	17.1	The equilibrium constant for an esterification reaction and other reactions could be experimentally investigated.
17B	Investigating an Ester Formation Equilibrium	17.1	The equilibrium constant for an esterification reaction and other reactions could be experimentally investigated.
18A	Some Measurements on Weak Acids and Bases	18.2	The properties of strong and weak acids could be investigated experimentally. Experiments could include determination of the pK_a of a weak acid.
18B	pH Changes during Acid-Base Titrations	18.3	Experiments could include investigation of pH curves.
18C	The Acid Dissociation Constant of an Indicator	18.3	Experiments could include investigation of the pK_a of an indicator.
19A	Understanding Electrode Potentials	19.1	Calculation of cell potentials using standard electrode potentials.
19B	The electrolysis of aqueous sodium chloride	19.1	Explanation of the products formed during the electrolysis of aqueous solutions.
19C	A Quantitative Investigation into Electrolysis	19.1	Determination of the relative amounts of products formed during electrolytic processes.
20A	Some Factors Affecting Halogenoalkane Reactions	20.1	A range of experiments exploring various types of reactions and functional group interconversions could be done.
20B	The nitration of methyl benzoate	20.1	A range of experiments of organic synthetic reactions could be done. Core techniques of organic chemistry could include reflux, distillation, filtration, purification (including chromatographic techniques), separations and extractions.
22B	Solubility product of a sparingly soluble compound	A.10	Experiments could include investigations of K_{sp} .
23B	Enzyme activity and the Michaelis constant	B.7	Experiments could include measuring enzyme activity with changing conditions of temperature, pH and heavy metal ion concentration.
24B	Factors affecting cell potential	C.6	The factors that affect the voltage of a cell and the lead–acid battery could be investigated experimentally.
25B	Investigating optical activity	20.3 / D.7	Distinction between optical isomers using a polarimeter. Explanation of the use of a polarimeter to identify enantiomers.

Syllabus relevance: Topic 13.1 & 13.2

The oxidation states of vanadium and manganese, for example, could be investigated experimentally.

Apparatus (per group)

- 6 × test tubes
- 2 × boiling tubes
- 100 cm³ beaker
- Wooden splints

Chemicals required

About 10 cm³ per student of aqueous solutions of the following: The concentration is not vital, from 1 mol dm⁻³ to 0.1 mol dm⁻³ will do unless stated otherwise.

- Acidified ammonium vanadate(V) (1.2 g of solid dissolved in 100 cm³ dil H₂SO₄)
- Chromium(III) sulfate
- Potassium chromate (VI)
- Hydrogen peroxide
- Manganese(II) sulfate
- Potassium manganate(VII) [0.01 mol dm⁻³]
- Iron(II) ammonium sulfate
- Iron(III) chloride
- Potassium thiocyanate
- Potassium iodide
- Cobalt(II) chloride
- Nickel(II) sulfate
- Copper(II) sulfate
- Sodium thiosulfate [0.2 mol dm⁻³]

Supplies of the following normal laboratory reagents (~50 cm³ per student)

- Aqueous sodium hydroxide [2 mol dm⁻³]
- Aqueous ammonia [2 mol dm⁻³]
- Dilute sulfuric acid [1 mol dm⁻³]
- Concentrated hydrochloric acid

The following solids:

- Granulated zinc (~2 g per group)
- Solid manganese(IV) oxide (~2 g per group)
- Solid glucose (~2 g per group)

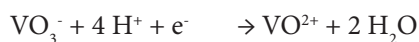
Notes

Probable timing—90 minutes laboratory time

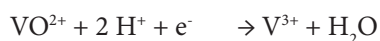
1. Vanadium

- a) **Observations** – The yellow solution starts to effervesce when the piece of zinc is added. It initially goes through green to a blue solution. The solution then gradually turns green again and eventually changes to a lilac colour.

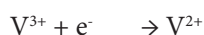
Interpretation – The first green colour is a mixture of the original yellow, vanadium(V) and the blue of the vanadium(IV) state that is being produced. The solution then turns green owing to the formation of the vanadium(III) ion and then changes to the lilac colour of vanadium(II).

Equation(s)

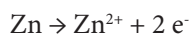
Yellow → Blue



Blue → Green

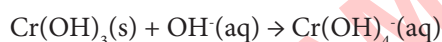
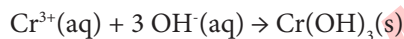


Green → Lilac

**2. Chromium**

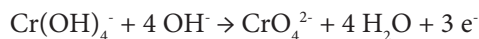
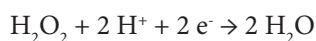
- a) **Observations** – The chromium(III) ions react with the hydroxide ions to form a murky green precipitate, which redissolves in excess of the alkali to form a clear bright green solution.

Interpretation – The initial precipitate is chromium(III) hydroxide which is amphoteric and redissolves to form a solution of the chromate(III) ion.

Equation(s)

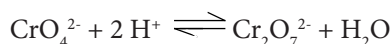
- b) **Observations** – The bright green solution becomes gradually more yellow in colour until eventually the solution becomes a bright yellow. There is gentle bubbling of the mixture, especially when heated.

Interpretation – The hydrogen peroxide, which is being reduced to water, oxidises the chromate(III) ion to the chromate(VI) ion,

Equation(s)

- c) **Observations** – The yellow solution turns an orange colour as the acid is added to it and then this colour change is reversed by addition of the alkali.

Interpretation – The yellow dichromate ion is in equilibrium with the orange dichromate ion. The hydrogen ions from the acid drive the position of equilibrium to the right, whereas the hydroxide ions from the alkali drive it to the left.

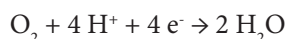
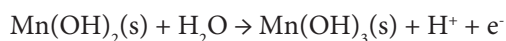
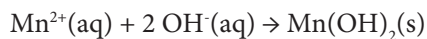
Equation(s)

Yellow → Orange

3. Manganese

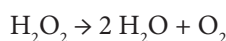
- a) **Observations** – When the alkali is added to the almost colourless solution, a pale buff coloured precipitate forms. On shaking, the colour slowly darkens to a brown.

Interpretation – The hydroxide ions react with the manganese(II) ions to precipitate out manganese(II) hydroxide. This is then slowly oxidised by oxygen in the air to form the darker coloured manganese(III) hydroxide.

Equation(s)

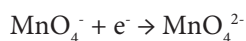
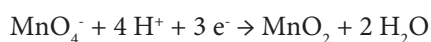
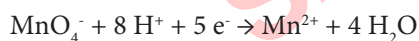
- b) **Observations** – When the black solid is added to the colourless solution there is rapid effervescence, a colourless gas is evolved and the mixture becomes significantly warmer.

Interpretation – The manganese(IV) oxide catalyses the decomposition of hydrogen peroxide into water and oxygen.

Equation(s)

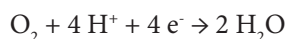
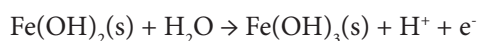
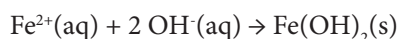
- c) **Observations** – The initial colour of the manganate(VII) solution is purple in all three cases. In the acidic solution the cyclohexene forms a colourless layer and slowly the aqueous layer is decolourised to leave a clear, colourless solution. In the case of the neutral solution the reaction with the cyclohexene causes the aqueous layer to become brown and cloudy, whilst with the alkaline solution it changes from purple to a deep dark green colour.

Interpretation – In each case the manganate(VII) ion is oxidising the cyclohexene to cyclohexan-1,2-diol. The reduction product varies according to the concentration of hydrogen ions. In acidic solution manganate(VII) ion is reduced to the almost colourless manganese(II) ion; in neutral solution insoluble, brown manganese(IV) oxide is produced, and in alkaline conditions the product is the dark green manganate(VI) ion.

Equation(s)**4. Iron**

- a) **Observations** – The very pale green solution reacts with the alkali to form a murky green gelatinous precipitate. Whilst there is no immediate sign of further reaction over the next hour the precipitate gradually darkens and near the surface of the solution becomes reddish-brown in colour.

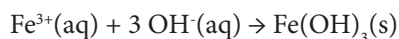
Interpretation – The hydroxide ions react with the iron(II) ions to precipitate out iron(II) hydroxide. This oxidation by oxygen in the air to form the reddish brown iron(III) hydroxide, is much slower than in the case of manganese.

Equation(s)

- b) **Observations** – The yellow-brown solution reacts with the alkali to form a gelatinous red-brown precipitate, but no further change occurs on adding excess of the reagent.

Interpretation – The hydroxide ions react with the iron(III) ions to precipitate out iron(III) hydroxide. Unlike the chromium and aluminium equivalents this is not amphoteric in the normal sense and unless very high concentrations of hydroxide ions are present it will not redissolve.

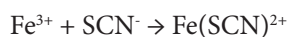
Equation(s)



- c) **Observations** – With the iron(II) solution there is a slight darkening of the almost colourless solutions to a pale straw colour. With the iron(III) the yellow-brown solution turns an intense blood-red colour when the colourless thiocyanate solution is added.

Interpretation – Iron(III) ions react with the thiocyanate ion to produce a very highly coloured complex ion. With iron(II) ions there is no reaction, but solutions of iron(II) salts almost always give a mild positive reaction owing to the presence of iron(III) ions in them as a result of oxidation by atmospheric oxygen.

Equation(s)

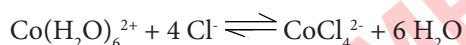


5. Cobalt

- a) **Observations** – The pale pink solution gradually changes colour, through shades of purple, to a bright blue colour as the concentrated acid is added. This colour change is reversed on dilution, but restores as the mixture is heated.

Interpretation – The chloride ions from the acid gradually displace the water ligands from around the pink hexaaquacobalt(II) ion to form the blue tetrachlorocobalt(II) ion. On reducing the chloride ion concentration the equilibrium is displaced back to the left, but heating causes it to shift to the right once more, indicating that the forward reaction is endothermic.

Equation(s)

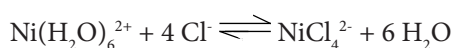


6. Nickel

- a) **Observations** – The green coloured solution gradually changes colour to a brighter yellow-green shade.

Interpretation – The chloride ions from the acid gradually displace the water ligands from around the green hexaaquanickel(II) ion to form the yellow-green tetrachloronickel(II) ion.

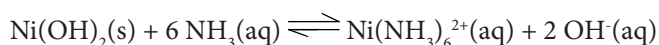
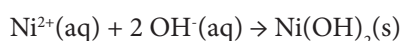
Equation(s)



- b) **Observations** – Initially the green solution becomes cloudy owing to the formation of a pale green precipitate. As excess ammonia is added the solution clears to a clear, pale blue colour.

Interpretation – Initially the hydroxide ions present in the aqueous ammonia react with the nickel(II) ions to form a precipitate of nickel(II) hydroxide, though if the concentrations of the two solutions are low this stage may not be apparent. On adding excess ammonia the precipitate redissolves to form the pale blue coloured hexaamminenickel(II) complex ion.

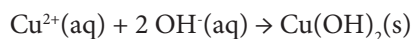
Equation(s)



7. Copper

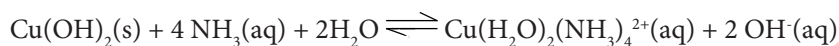
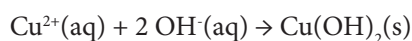
- a) **Observations** – The blue coloured solution reacts with the alkali to form a pale blue precipitate, but there is no further reaction with excess alkali.

Interpretation – The blue hexaaquacopper(II) ions react with the hydroxide ions in the alkali to precipitate copper(II) hydroxide, which is not amphoteric and hence does not redissolve in excess.

Equation(s)

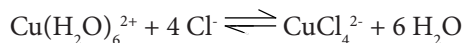
- b) **Observations**–Initially the blue solution becomes cloudy and a pale blue precipitate forms. As excess ammonia is added the precipitate redissolves to form a clear, intense royal blue coloured solution.

Interpretation–Initially the hydroxide ions present in the aqueous ammonia react with the copper(II) ions to form a precipitate of copper(II) hydroxide. On adding excess ammonia the precipitate redissolves to form the strongly coloured, royal blue tetraamminecopper(II) complex ion.

Equation(s)

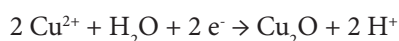
- c) **Observations** – The blue colour solution gradually changes colour, through shades of green, to a yellow colour when a large excess of the concentrated acid is added.

Interpretation–The chloride ions from the acid gradually displace the water ligands from around the blue hexaaquacopper(II) ion to form the yellow tetrachlorocopper(II) ion, the intermediate colours being caused by a mixture of these colours and/or partial ligand replacement.

Equation(s)

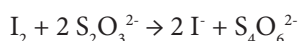
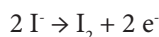
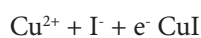
- d) **Observations** – The white glucose dissolves readily in the blue solution without any significant change in its colour, even on heating. On adding the alkali the mixture becomes cloudy and the colour changes through green and yellow to a red-brown precipitate.

Interpretation – In alkaline conditions the glucose reduces copper(II) ions to the insoluble, red-brown copper(I) oxide.

Equation(s)

- e) **Observations** – The blue solution reacts with the colourless iodide solution to give a cloudy brown mixture. On adding the thiosulfate the brown colouration disappears to leave an off-white precipitate in a colourless solution.

Interpretation – The blue copper(II) ions are reduced to copper(I) ions which react with iodide ions to form an off-white precipitate of insoluble copper(I) iodide. The oxidation of the iodide ions produces a brown solution of iodine. When aqueous thiosulfate ions are added these react with the iodine, converting it back to colourless iodide ions so the colour of the copper(I) iodide is clearer.

Equation(s)

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