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| **DEPARTMENT OF CHEMISTRY**  **FOURAH BAY COLLEGE – UNIVERSITY OF SIERRA LEONE** **CHEM 114**EXPERIMENTAL CHEMISTRY I **(to be taken alongside CHEM 111)** | |
| **CREDIT HOURS** | **1.5** |
| **REQUIREMENTS** | C6 in WASSCE Chemistry or equivalentTo be taken alongside CHEM 111 |
| **REQUIRED FOR** | **CHEM 124** |

## INTRODUCTION

Chemistry is the investigation of the composition, properties and transformations of substances and various elementary forms of matter. As part of the study of Chemistry, it is frequently necessary to study the composition, properties and transformations of substances experimentally; this requires a knowledge and understanding of various practical techniques and the development of the skills required to implement these techniques. CHEM114 is an introduction to some of the practical techniques used in Chemistry. A pass (C-) in 114 is required for all students wishing to specialise in Chemistry or Biology.

The course consists of a series of 10 practical experiments based on different chemical principles and introducing different practical techniques. Working in small groups, students are required to perform a series of practical tasks during the course of each experiment. Students are expected to keep a clear written record of all practical work carried out and all experiment results obtained.

## EQUIPMENT REQUIRED

Students must purchase the manuals required for CHEM 114 and CHEM 124 at the start of the academic year at a cost of Le 40,000; this cost includes a small caution fee used to cover the annual cost of replacing any broken glassware.

An official FBC Chemistry labcoat must be worn to every practical session. These can be purchased from the head technician at a cost of Le 70,000. Students will not be admitted into any practical session unless they are wearing an FBC Chemistry labcoat.

Safety glasses must also be worn to every practical session. These are widely available in town but can also be purchased from the head technician at a cost of Le 15,000. Students will be deducted marks if they are not wearing eye protection.

Students should also bring a pen, writing paper and a calculator to every practical session. Graph paper is also required for some reports.

**DRESS AND BEHAVIOUR EXPECTATIONS**

Students are not permitted to wear open footwear and long hair must be tied back. Casual, durable clothes and trainers are recommended.

Food and drink must not be consumed in any laboratory.

The chemicals and apparatus used in Chemistry can be harmful, especially if not handled correctly. Students are expected to act in a safe and responsible manner at all times. Ask the nearest staff member if you not sure how to handle apparatus or chemical safely.

If students break equipment with a value exceeding the annual contribution, the student is expected to replace the equipment at his/her own expense.

**ASSESSMENT**

**Continuous Assessment – 50%**

Students must submit a full report for each practical to the head technician by 12.00 pm on the Monday following the practical session. The report will only be accepted if the student has been marked present at both the beginning and the end of the practical and if their results have been signed by their allocated lecturer.

Most reports will be awarded a mark out of 10. Two reports per semester will be awarded a mark out of 20 and will be considered equivalent to two reports. The best eight reports achieved by each student over the semester will be added up to give a total out of 80. This mark will be scaled down to give a final continuous assessment mark out of 50.

**Practical Examination – 50%**

Students will sit a single practical examination at the end of the semester. This will consist of two practical exercises and some written questions. There are 70 marks available for the practical exam, which will be scaled down to give a final examination mark out of 50.

The mark from the continuous assessment and the mark from the practical examination are combined to give a total mark out of 100, which will determine the students’ overall grade for the module.

**PROCEDURE FOR PRACTICAL SESSIONS**

1. Students should arrive promptly for each session and sign in as they enter the laboratory; students may sign in from 08:30
2. At 09:00 the lecturers will introduce the main chemical concepts underlying the practical, demonstrate how to use any new apparatus and provide suggestions on how to best carry out the practical
3. At 09:30 the lecturers will ask the students to report to their work stations and check their equipment; each group will be briefed, issued with any additional equipment they need and be instructed to start the practical
4. Once all data has been collected, the students should ensure that their rough work is signed by the lecturer allocated to their work station and then clean and tidy their work station
5. Students should sign out with the head technician once their work station is clean and tidy; they should do this by 12:30
6. At 12:30 the lecturers will review the theory and provide guidance on completing the report
7. The session should finish promptly at 13:00

## REPORTS

* Each report should include a clear presentation of all observations, calculation and presentation of results and conclusions with all working shown clearly, and answers to any additional questions
* Rough work should be signed by a lecturer at the end of the session and should be submitted along with the report
* Marks will be awarded as follows:

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| Arrives by 9.00 am | 1 mark |
| Arrives before practical work begins | 1 mark |
| Arrives by 10.00 am | 1 mark |
| Arrives by 10.00 am and wears eye protection throughout practical session | 1 mark |
| Arrives by 10.00 am, wears appropriate clothing and behave appropriately throughout practical session | 1 mark |
| Report | 5 marks |
| TOTAL | 10 marks |

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| **4** | **Qualitative Analysis I – Identification of Cations** |
| **5** | **Qualitative Analysis II – Identification of Anions** |
| **6** | **Volumetric Analysis I: Acid-Base Reactions** |
| **7** | **Volumetric Analysis II: Precipitation Reactions** |
| **8** | **Volumetric Analysis III: Redox Reactions** |
| **9** | **Chemical Equilibrium** |
| **10** | **Colorimetric Determination of pH** |

**Week 1 – Physical Separation Techniques**

**Chemical Principles**

Many ionic compounds, such as NaCl and KCl, dissolve in water. Some, such as BaSO4, do not.

Ionic compounds almost never dissolve in organic solvents such as ethanol.

Organic compounds such as C6H5COOH usually dissolve in organic solvents such as ethanol.

Organic compounds sometimes dissolve in water.

Many solid/solid or solid/liquid mixtures can therefore be separated by using the fact that they have different solubilities in different solvents.

If a mixture of solids is added to a suitable solvent, the soluble solid will dissolve in the solvent to make a solution. The insoluble solid will not.

The insoluble solid can be separated from the solution by **filtration**. The solid remaining in the filter paper after filtration is called the **residue**. The solution which passes through the filter paper is called the **filtrate**.

After filtration, the residue should be washed (whilst still in the filter paper) with cold solvent to remove any remaining solution. The residue should then be dried in an oven to ensure that any remaining solvent evaporates. If the solvent is water, a **dessicator** can also be used. Complete drying can be achieved by heating in a crucible using ashless filter paper. The drying process is complete when the mass of the solid and its contents stops changing.

After filtration, the solute in the filtrate can be separated from the solvent by **evaporation**. The solution should be heated in an evaporating dish over a flame until the solvent has evaporated. In some cases, the solid may decompose on heating so you may need to stop heating just before the solvent has evaporated, leaving the remaining solvent to evaporate naturally.

It is often necessary in Chemistry to measure the mass of a substance. Two measurements are always required for the measurement of mass: the mass of the empty container and the mass of the container with its contents. The mass of the contents is the difference between the two. This technique is known as **weighing**. Weighing measurements should be recorded in a table as follows:

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| --- | --- |
| Mass of empty container /g |  |
| Mass of container plus contents /g |  |
| Mass of contents / g |  |

**Experiment 1 - Separating a mixture of sand and salt and hence finding the percentage by mass of each in the mixture**

**Equipment needed:** boiling tube, funnel, filter paper, stirring rod, 10 cm3 measuring cylinder, evaporating dish, access to 2dp mass balance, access to oven, access to hot flame

**Method:**

1. Weigh a piece of filter paper.
2. Weigh out approximately 1 g of the mixture onto the filter paper; pour the mixture into a boiling tube and add 10 cm3 of water, stirring well for a few minutes.
3. Weigh an empty evaporating dish and then filter the mixture into it.
4. Wash the residue with a little water, allowing the water to run into the evaporating dish.
5. Remove the filter paper, open it up and leave it to dry in the oven for 10 minutes.
6. Weigh the filter paper and residue and then put it back in the oven for another 10 minutes.
7. If the mass has decreased since the first measurement, put it back in the oven for 10 minutes and repeat until there is no further change in mass.
8. Place the evaporating dish onto a tripod above a flame and evaporate off the water.
9. Allow the mixture to cool in a dessicator and weigh the evaporating dish and contents.
10. Leave the evaporating dish in an oven or on a hot plate for 30 minutes and then weigh it again. If the mass has decreased since the first measurement, put it back in the oven for 10 minutes and repeat until there is no further change in mass.

**Experiment 2 - Separating a mixture of barium sulphate and sodium chloride and hence finding the percentage by mass of each in the mixture**

**Equipment needed:** boiling tube, funnel, ashless filter paper, stirring rod, 10 cm3 measuring cylinder, evaporating dish, crucible, access to 2dp mass balance, access to hot flame

**Method:**

1. Weigh a piece of filter paper.
2. Weigh out approximately 1 g of the mixture onto the filter paper; pour the mixture into a boiling tube and add 10 cm3 of water, stirring well for a few minutes.
3. Weigh an empty evaporating dish and then filter the mixture into it.
4. Wash the residue with a little water, allowing the water to run into the evaporating dish.
5. Remove the filter paper, open it up and leave it to dry in the oven for 10 minutes.
6. Weigh the filter paper and residue and then put it back in the oven for another 10 minutes.
7. If the mass has decreased since the first measurement, put it back in the oven for 10 minutes and repeat until there is no further change in mass.
8. Place the evaporating dish onto a tripod above a flame and evaporate off the water.
9. Allow the mixture to cool in a dessicator and weigh the evaporating dish and contents.
10. Leave the evaporating dish in an oven or on a hot plate for 30 minutes and then weigh it again. If the mass has decreased since the first measurement, put it back in the oven for 10 minutes and repeat until there is no further change in mass.

**Experiment 3 - Separating a mixture of benzoic acid and sodium chloride and hence find the percentage by mass of each in the mixture**

**Equipment needed:** boiling tube, funnel, ashless filter paper, stirring rod, 10 cm3 measuring cylinder, evaporating dish, crucible, access to 2dp mass balance, access to sand bath

**Method:**

1. Weigh a piece of filter paper.
2. Weigh out approximately 1 g of the mixture onto the filter paper; pour the mixture into a boiling tube and add 10 cm3 of ethanol, stirring well for a few minutes.
3. Weigh an empty evaporating dish and then filter the mixture into it.
4. Wash the residue with a little ethanol, allowing the water to run into the evaporating dish.
5. Remove the filter paper, open it up and leave it to dry in the oven for 10 minutes.
6. Weigh the filter paper and residue and then put it back in the oven for another 10 minutes.
7. If the mass has decreased since the first measurement, put it back in the oven for 10 minutes and repeat until there is no further change in mass.
8. Place the evaporating dish onto a sand bath and evaporate off the ethanol.
9. Allow the mixture to cool in a dessicator and weigh the evaporating dish and contents.
10. Leave the evaporating dish in an oven for 10 minutes and then weigh it again. If the mass has decreased since the first measurement, put it back in the oven for 10 minutes and repeat until there is no further change in mass.

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| **Report**   1. Record all of your results clearly 2. Calculate the percentage by mass of each substance in the mixture for each experiment 3. How did your total mass of separated substances compare with the original mass of the mixture? What might have caused a lower total than your original? What might have caused a higher total than your original? 4. Give two reasons why it is important not to use much more than 10 cm3 of water to dissolve the salt in Experiments 1 and 2, and not more than 10 cm3 of ethanol in Experiment 3 |

**Week 2 – Demonstration of Various Physical Methods and Processes**

**Chemical Principles**

1. **Crystallisation and Fractional Crystallisation**

Most substances are more soluble in solvents at higher temperatures.

A **saturated** solution is a solution in which no more of the solute will dissolve. A saturated solution can be easily prepared by adding a solute gradually to a solvent until no more of the solid dissolves.

If a saturated solution is heated, the excess solute should dissolve in the solvent as the solute becomes more soluble. If this solution is allowed to cool, the solute will become less soluble again and it will appear as a solid. This process is known as **crystallisation**.

If a saturated solution containing a mixture of components is heated until all excess solute dissolves, and then allowed to cool, the two components will often crystallise at different temperatures, and so one solute will crystallise before the other. This is known as **fractional crystallisation** and can be used to separate two components which are both soluble in a solvent.

1. **Sublimation**

Some solids turn directly into a gas on gentle heating. This is known as **sublimation**. A mixture of two solid components can be separated if one of the components sublimes and the other does not. Ammonium chloride and iodine both sublime.

If the mixture is heated in a dish, the solid which sublimes will turn into a gas, and the one which does not will remain in the dish. If an inverted funnel is placed over the dish, the gas will often condense again on the funnel to give a layer of solid.

1. **Gravimetric Analysis**

Many ionic compounds in the solid state contain a fixed ratio of water molecules in the crystal structure. This water is known as **water of crystallisation** and any solid which contains water within its crystal structure is said to be **hydrated.**

When hydrated solids are heated, they lose their water of crystallisation, which usually escapes as a gas. The mass of the solid decreases as a result. By measuring the change in mass of a solid before and after dehydration, it is possible to deduce the formula of the hydrated solid:

S.xH2O(s) 🡪 S(s) + xH2O(g)

- Let initial mass of solid (S.xH2O) = a; let final mass of solid (S) = b so mass of water = a – b

- Moles of S = b/mr(S), moles of water = (a-b)/18

- So x = =

The analysis of a substance by measuring changes in mass is known as **gravimetric analysis**.

**Experiment 1 – Observing Crystallisation**

**Equipment needed:** test tube, test tube rack, stirring rod, tongs, watch glass, access to KNO3 with spatula, access to a water bath

1. Pour 5 cm3 of water into a test tube
2. Add one spatula measure of potassium nitrate (KNO3) to the test tube and swirl until it fully dissolves
3. Keep adding KNO3 to the test tube, one spatula at a time, and swirling, until no further KNO3 will dissolve (you have now made a saturated solution of KNO3)
4. Heat the saturated solution in a water bath until the remaining KNO3 dissolves
5. Pour the hot solution onto a watch glass and record your observations

**Experiment 2 – Separating a mixture of copper sulphate and potassium chlorate (V) by Fractional Crystallisation**

**Equipment needed:** boiling tube, boiling tube rack, stirring rod, tongs, watch glass, access to KClO3/CuSO4 mixture with spatula, access to a water bath, two pieces of filter paper

1. Pour 10 cm3 of water into a boiling tube
2. Add the KClO3/CuSO4 mixture to the boiling tube, one spatula measure at a time and swirling in between each addition, until no further solid with dissolve (ie the solution is saturated)
3. Heat the saturated solution in a water bath until the remaining solid dissolves
4. Pour the hot solution onto a watch glass and record your observations
5. Use a spatula to remove the crystals as they form; place the KClO3 crystals on one piece of filter paper and the CuSO4 crystals on another

**Experiment 3 – Observing Sublimation**

**Equipment needed:** test tube, test tube rack, small spatula, access to a Bunsen burner

1. Place approximately 0.5 g of I2 into a clean dry test tube
2. Heat the iodine gently over a Bunsen burner until it starts to sublime
3. Record your observations

**Experiment 4 – Using Sublimation to separate a mixture of sodium chloride and ammonium chloride**

**Equipment needed:** evaporating dish, glass funnel, spatula, access to sand bath

1. Place approximately 0.1 g of the NaCl/NH4Cl onto an evaporating dish
2. Please an inverted glass funnel over the dish and place onto a sand bath
3. Record all your observations
4. Remove the funnel and try to collect some of the NH4Cl

**Experiment 5 – Determining the water of crystallisation of hydrated copper sulphate by gravimetric analysis**

**Equipment needed:** crucible, access to mass balance, access to clay pipe triangle and tripod, access to Bunsen Burner, access to hydrated copper sulphate with spatula

1. Weigh an empty crucible
2. Weigh out approximately 1 g of hydrated copper sulphate crystals into a crucible
3. Weigh the crucible with the hydrated copper sulphate in it
4. Place the crucible in a clay pipe triangle on a tripod over a Bunsen burner and heat for 15 minutes using a small blue flame
5. Using tongs, remove the crucible from the clay pipe triangle and allow it to cool – **be careful not to drop the crucible – it is hot!**
6. Weigh the crucible again
7. Repeat step 4, but this time heat for only 5 minutes
8. Remove the crucible, allow it to cool and weigh it again
9. If the mass has decreased again, repeat steps 8 and 9 until there is no further change in mass

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| Report  1. Write down your observations from Experiment 1 and explain what is happening. 2. Write down your observations from Experiment 2 and answer the following questions: 3. How effectively were you able to separate the two solids? 4. What was the difference between the solids which allowed them to be separated in this way? 5. Why would the techniques used in Week 1 not be effective for this separation? 6. Write down your observations from Experiment 3 and explain what is happening 7. Write down your observations from Experiment 4 and evaluate the effectiveness of this technique as a way of separating the two solids 8. Write down your observations from Experiment 5 and explain what is happening 9. Calculate the value of x in CuSO4.xH2O |

**Week 3 – Solubility and Solubility Curves**

### Chemical Principles

The **solubility** of a substance is the quantity of that substances (in grams or moles) which can dissolve in a stated amount of solvent or solution (usually 100 ml or 1 dm3) at a given temperature.

A **solubility curve** is a graph of solubility against temperature and hence shows the relationship between solubility and temperature.

The solubility of most salts increases with increasing temperature, although there are a number of exceptions. In a small number of cases, the solubility of a substance may increase with increasing temperature up to a certain critical temperature, and then decrease again if the temperature is increased further; this happens if the most stable form of the solid changes - for example, sodium sulphate is most stable in hydrated form (Na2SO4.10H2O) at temperatures below 32.4 oC but in anhydrous form at temperatures above 32.4 oC; its solubility in water increases with increasing temperature up to a maximum at 32.4 oC but decreases with increasing temperature above 32.4 oC.

The temperature of maximum (or minimum) solubility is called the transition point. It occurs at the same temperature for all solvents.

The solubility of a substance at different temperatures can be prepared by adding a known mass of solute to a fixed quantity of the solvent, heating the mixture until it dissolves, then allowing the mixture to cool and observe the temperature at which crystals form; the solution can then be diluted and the procedure repeated at the new, lower concentration.

Equipment needed: boiling tube, burette, stirring rod, thermometer, access to water bath, access to mass balance, access to KClO3 with spatula, access to Na2B4O7.10H2O with spatula

**Experiment 1 – Determine the solubility curve for potassium chlorate (V)**

1. Weigh out approximately 1.5 g of KClO3 into a boiling tube. Record the exact mass added.
2. Add 5.0 cm3 of water from a burette into the boiling tube.
3. Heat the mixture, stirring well, until the solid dissolves.
4. Insert a thermometer into the boiling tube; allow the mixture to cool and note the temperature at which crystals first appear or at which the solution becomes turbid. Record this temperature.
5. Add a further 3.0 cm3 of water from the burette into the boiling tube and repeat steps 3 and 4.
6. Repeat step 5 until a total of 20 cm3 have been added (ie carry out step 5 five times)

**Experiment 2 – Determine the transition point of borax**

1. Weigh out approximately 3 g of Na2B4O7.10H2O into a boiling tube. Record the exact mass added.
2. Add 5.0 cm3 of water from a burette into the boiling tube.
3. Heat the mixture, stirring well, until the solid dissolves.
4. Insert a thermometer into the boiling tube; allow the mixture to cool and note the temperature at which crystals first appear or at which the solution becomes turbid. Record this temperature.
5. Add a further 3.0 cm3 of water from the burette into the boiling tube and repeat steps 3 and 4.
6. Repeat step 5 until a total of 20 cm3 have been added (ie carry out step 5 five times)

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| **Report**   1. Calculate the molar mass of KClO3 and hence the moles used; hence calculate the molarities of the solutions after each addition of water and complete the table below:  |  |  |  |  | | --- | --- | --- | --- | | Moles of KClO3 | Total volume of water /cm3 | Molarity of solution | Lowest temperature at which this molarity can exist /oC | |  | 5.0 |  |  | |  | 8.0 |  |  | |  | 11.0 |  |  | |  | 14.0 |  |  | |  | 17.0 |  |  | |  | 20.0 |  |  |  1. Plot a graph of the solubility of KClO3 against temperature 2. Calculate the molar mass of Na2B4O7.10H2O and hence the moles used; hence calculate the molarities of the solutions after each addition of water and complete the table below:  |  |  |  |  | | --- | --- | --- | --- | | Moles of Na2B4O7.10H2O | Total volume of water /cm3 | Molarity of solution | Lowest temperature at which this molarity can exist /oC | |  | 5.0 |  |  | |  | 8.0 |  |  | |  | 11.0 |  |  | |  | 14.0 |  |  | |  | 17.0 |  |  | |  | 20.0 |  |  |  1. Plot a graph of the solubility of Na2B4O7.10H2O against temperature and use your graph to determine the transition point of borax |

**Week 4 – Qualitative Analysis of Cations in Solution**

Qualitative Analysis is the identification of an unknown substance.

Cations in solution can be identified from:

* the colour of the solution
* whether or not they give a precipitate with certain reagents, the colour of that precipitate and the solubility of the precipitate in other reagents
* whether or not they form complex ions with certain reagents, observable by the solution turning a characteristic colour

In this experiment you will be given 20 solutions, labelled A – T. Each solution contains a different one of the following cations:

|  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| NH4+ | Na+ | K+ | Mg2+ | Ca2+ | Sr2+ | Ba2+ | Al3+ | Sn2+ | Pb2+ |
| Cr3+ | Mn2+ | Fe2+ | Fe3+ | Co2+ | Ni2+ | Cu2+ | Zn2+ | Ag+ | Cd2+ |

You are required to carry out a series of observations and simple chemical tests in order to identify which cation is in which solution.

The available tests are:

1. **hydrochloric acid followed by dilute ammonia**

* dilute HCl will precipitate Ag+ and Pb2+ as chlorides
* AgCl dissolves in dilute NH3 to give a complex ion but PbCl2 does not

**Test: add 1 cm3 of dilute HCl to 1 cm3 of the unknown solution; if a precipitate if formed add dilute NH3 dropwise until the test tube is half-full**

1. **hydrogen sulphide**

* H2S in acidic conditions will precipitate Ag+, Pb2+, Cu2+, Sn2+ and Cd2+ as sulphides
* the colour of the precipitate can help identify the cation
* Fe3+ can oxidise H2S to a yellow precipitate of sulphur

**Test: bubble H2S into 1 cm3 of the unknown solution for 10 seconds; if no precipitate is formed after 10 s, bubble H2S into the solution for a further 20 s**

1. **sodium hydroxide solution**

* NaOH will precipitate all cations except NH4+, Na+, K+, Ba2+ and Sr2+ as hydroxides, although Ca(OH)2 does not form instantly
* the colour of the precipitate can help identify the cation
* Pb(OH)2, Zn(OH)2, Cr(OH)3, Al(OH)3 and Sn(OH)2 are amphoteric and dissolve in excess sodium hydroxide to give hydroxoanions

**Test: add a few drops of dilute NaOH to 1 cm3 of the unknown solution; continue to add NaOH, a few drops at a time, until the test tube is half-full**

1. **ammonia solution**

* NH3 is alkaline and will precipitate all cations except NH4+, Na+, K+, Ba2+, Sr2+ and Ca2+ as hydroxides
* the colour of the precipitate can help identify the cation
* AgOH, Cu(OH)2, Cd(OH)2, Cr(OH)3, Ni(OH)2, Mn(OH)2, Zn(OH)2 and Co(OH)2 dissolve in excess dilute NH3 to give complex ions
* the colour of the complex can help identify the cation

**Test: add a few drops of dilute NH3 to 1 cm3 of the unknown solution; continue to add NH3, a few drops at a time, until the test tube is half-full**

1. **dilute sulphuric acid**

* H2SO4 will precipitate Pb2+, Ag+, Ba2+ and Sr2+ ions as sulphates; Ca2+ also forms a precipitate slowly

**Test: add 1 cm3 of dilute H2SO4 to 1 cm3 of the unknown solution**

You are NOT required to carry out all five of these tests on every solution; you are only required to carry out enough tests to identify all 20 cations; should these five tests be insufficient to identify a particular cation, there are some other tests available to you if you ask a technician:

1. potassium dichromate and ethanoic acid

precipitates Ag+, Pb2+ and Ba2+ as chromates; the colour of the precipitate can help identify the cation

1. potassium hexacyanoferrate (II) solution

precipitates Cu2+, Fe2+, Fe3+, Zn2+ and Ca2+ as cyanoferrates; the colour of the precipitate can help identify the cation

1. uranyl magnesium acetate solution

precipitates Na+ as a magnesium uranyl acetate

## The expected observations for each chemical test are shown below:

|  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
|  | Cation | Colour in solution | (a)  HCl followed by NH3 | (b)  Acidic H2S | (c)  NaOH until in excess | (d)  NH3 until in excess | (e)  H2SO4 | Extra Test |
| 1 | Ag+ | Colourless | White ppt soluble in NH3 | Black ppt | Brown ppt | brown ppt soluble in excess | White ppt | K2CrO4/CH3COOH – red ppt |
| 2 | Pb2+ | Colourless | White ppt insoluble in NH3 | Black ppt | White ppt soluble in excess | White ppt insoluble in excess | White ppt | K2CrO4/CH3COOH – yellow ppt |
| 3 | Cu2+ | Pale blue | No reaction | Black ppt | Pale blue ppt | Pale blue ppt, soluble in excess to give deep blue solution | No reaction | K4Fe(CN)6 – red/brown ppt |
| 4 | Sn2+ | Colourless | No reaction | Brown ppt | White ppt soluble in excess | White ppt insoluble in excess | No reaction |  |
| 5 | Cd2+ | Colourless | No reaction | Yellow ppt | White ppt | White ppt soluble in excess | No reaction |  |
| 6 | Fe2+ | Pale green | No reaction | No reaction | Dirty green ppt, turning orange on standing | Dirty green ppt, turning orange on standing | No reaction | K4Fe(CN)6 – pale blue ppt |
| 7 | Fe3+ | Yellow/orange | No reaction | Yellow ppt | Red-brown ppt | Red-brown ppt | No reaction | K4Fe(CN)6 – pale blue ppt |
| 8 | Cr3+ | Dark green | No reaction | No reaction | Green ppt, soluble in excess | Green ppt, soluble in excess | No reaction |  |
| 9 | Ni2+ | Green | No reaction | No reaction | Green ppt | Green ppt soluble in excess | No reaction |  |
| 10 | Mn2+ | Pale pink /colourless | No reaction | No reaction | White ppt, darkens on standing, | White ppt soluble in excess | No reaction |  |

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| --- | --- | --- | --- | --- | --- | --- | --- | --- |
|  | Cation | Colour in solution | (a)  HCl followed by NH3 | (b)  Acidic H2S | (c)  NaOH until in excess | (d)  NH3 until in excess | (e)  H2SO4 | Extra Test |
| 11 | Zn2+ | Colourless | No reaction | No reaction | White ppt soluble in excess | White ppt soluble in excess | No reaction | K4Fe(CN)6 – white ppt |
| 12 | Al3+ | Colourless | No reaction | No reaction | White ppt  Soluble in excess | White ppt | No reaction |  |
| 13 | Co2+ | Pink | No reaction | No reaction | Blue ppt, turning pink on standing | Blue ppt, soluble in excess (yellow solution) | No reaction |  |
| 14 | Ca2+ | Colourless | No reaction | No reaction | Faint white ppt in excess | No reaction | White ppt may form slowly | K4Fe(CN)6 – white ppt |
| 15 | Sr2+ | Colourless | No reaction | No reaction | No reaction | No reaction | White ppt |  |
| 16 | Ba2+ | Colourless | No reaction | No reaction | No reaction | No reaction | White ppt | K2CrO4/CH3COOH – yellow ppt |
| 17 | NH4+ | Colourless | No reaction | No reaction | Pungent gas evolved on warming | No reaction | No reaction |  |
| 18 | Na+ | Colourless | No reaction | No reaction | No reaction | No reaction | No reaction | UMA – yellow ppt |
| 19 | K+ | Colourless | No reaction | No reaction | No reaction | No reaction | No reaction |  |
| 20 | Mg2+ | Colourless | No reaction | No reaction | White ppt | White ppt | No reaction |  |

|  |
| --- |
| **Report**   1. Identify the cation present in solutions A – T and explain how you were able to identify each cation  * Indicate which tests you carried out on each solution * Indicate your observations in full for each test * Indicate what you have concluded from each test  1. For the regular tests (a) – (e) which gave a result, write an equation for the reaction taking place. 2. Explain any problems you encountered with identifying the cation present in each solution. |

**Week 5 – Qualitative Analysis of Anions in Solution**

Qualitative Analysis is the identification of an unknown substance.

Anions in solution can be identified from:

* whether or not they give a precipitate with certain reagents, the colour of that precipitate and the solubility of the precipitate in other reagents
* whether or not they evolve gases with certain reagents, and if so, which gas is evolved

In this experiment you will be given 10 solids, labelled A – J. Each solid contains a different one of the following ions as well as a K+ or Na+ ion:

|  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| CO32- | HCO3- | SO42- | SO32- | S2- | NO3- | NO2- | Cl- | Br- | I- |

You are required to carry out a series of observations and simple chemical tests in order to identify which anion is in which solid.

The available tests are:

1. **hydrochloric acid**

* H+ will give an acid-base reaction with CO32- and HCO3- to produce CO2, with SO32- to produce SO2, with S2- to produce H2S, with NO2- to produce NO2; in all cases effervescence will be observed
* The gas evolved can be tested as follows:

|  |  |  |
| --- | --- | --- |
| CO2 | bubble through limewater | Turns limewater cloudy |
| SO2 | dichromate paper  bubble through limewater | Turns green  Turns limewater cloudy |
| NO2 | brown gas |  |
| H2S | smelly gas (rotten eggs)  dichromate paper  lead ethanoate paper | Turns green  Turns black |

**Test: add 1 cm3 of dilute HCl to a small spatula measure of the unknown solid; if a gas is evolved check the colour and smell; then add dichromate or lead ethanoate paper as appropriate; if both tests negative bubble through limewater**

1. **barium chloride solution**

* Ba2+ will give precipitates with HCO3-, CO32-, SO32- which dissolve in HCl with effervescence
* Ba2+ will give a precipitate with SO42- which is insoluble in HCl

**Test: dissolve a small spatula measure of the unknown solid in water; add 1 cm3 of BaCl2; if a precipitate is formed add HCl gradually until in excess**

1. **Silver nitrate solution**

* Ag+ will give precipitates with CO32-, SO32-, S2-, Cl-, Br- and I- and with excess NO2- and SO42-
* Ag2CO3 and Ag2SO3 dissolve in HNO3
* All precipitates except AgBr and AgI dissolve in dilute NH3

**Test: dissolve a small spatula measure of the unknown solid in water; add 1 cm3 of AgNO3; if a precipitate is formed, split the precipitate into two portions; to the first add HNO3 and to the second add NH3**

1. **Chlorine water**

* Chlorine will oxidise I- and Br-
* the colours of the halogens formed can be used to identify the ion
* organic solvents give more clarity to the colour

**Test: dissolve a small spatula measure of the unknown solid in water; add 1 cm3 of chlorine water; if the solution turns yellow, orange or brown add 1 cm3 of cyclohexane and shake well**

You are NOT required to carry out all three of these tests on every anion; you are only required to carry out enough tests to identify all 10 anions; should these three tests be insufficient to identify a particular anion, there are some other tests available to you:

1. magnesium sulphate solution

* Mg2+ will give a precipitate with CO32- but not HCO3-

Test: **Dissolve a small spatula measure of the unknown solid in water; add 1 cm3 of MgSO4**

1. Potassium manganate (VII) solution

* MnO4- is reduced and decolorised by SO32-, NO2- and Cl- it is also reduced by S2-, Br- and I- but the resulting solutions are also coloured

**Test: Dissolve a small spatula measure of the unknown solid in water; add 1 cm3 of acidified KMnO4**

## The expected observations for each chemical test are shown below:

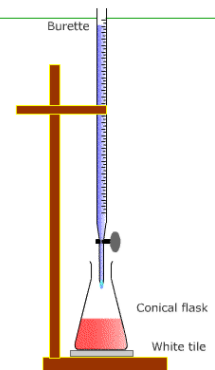
|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
|  | Cation | Colour in solution | (a)  (i) HCl to solid  (ii) test on gas | (b)  (i) BaCl2 to solution  (ii) add HCl | (c)  (i) AgNO3 to solution  (ii) add HNO3 or NH3 | (d)  (i) add Cl2  (ii) add C6H12 | Extra Test |
| 1 | CO32- | Colourless | Effervescence (CO2) | white ppt soluble in HCl | White ppt  Soluble in HNO3 and NH3 | No reaction | White ppt with MgSO4 |
| 2 | HCO3- | Colourless | Effervescence (CO2) | white ppt soluble in HCl | White ppt  Soluble in HNO3 and NH3 | No reaction | No reaction with MgSO4 |
| 3 | SO42- | Colourless | no effervescence | white ppt | Faint white ppt in excess | No reaction |  |
| 4 | SO32- | Colourless | Effervescence (SO2) | white ppt soluble in HCl | White ppt  Soluble in HNO3 and NH3 | No reaction | Decolorises KMnO4 |
| 5 | NO3- | Colourless | no effervescence | no reaction | No reaction | No reaction |  |
| 6 | NO2- | Colourless | effervescence  (NO2) | no reaction | Faint white ppt in excess | No reaction | Decolorises KMnO4 |
| 7 | S2- | Colourless | effervescence  (H2S) | no reaction | Black ppt  soluble in NH3 | Yellow ppt | Decolorises KMnO4; yellow ppt |
| 8 | Cl- | Colourless | no effervescence | no reaction | White curdy ppt  soluble in NH3 | No reaction | Decolorises KMnO4 |
| 9 | Br- | Colourless | no effervescence | no reaction | Cream ppt | Yellow/orange solution; turns cyclohexane orange | Yellow/orange solution with KMnO4 |
| 10 | I- | Colourless | no effervescence | no reaction | Yellow ppt | Yellow/brown solution; turns cyclohexane purple | Yellow/brown solution with KMnO4 |

|  |
| --- |
| **Report**   1. Identify the anion present in solutions A – J and explain how you were able to identify each anion  * Indicate which tests you carried out on each solution * Indicate your observations in full for each test * Indicate what you have concluded from each test  1. For the regular tests (a) – (d) which gave a result, write an equation for the reaction taking place. 2. Explain any problems you encountered with identifying the anion present in each solution. |

Weeks 6, 7 and 8 – Volumetric Analysis

**Principles of Volumetric Analysis**

* **Volumetric analysis** is the quantitative investigation of a solution using one or more measurements of volume



* The most common type of volumetric analysis is **titration**; during a titration, the volume of one solution required to react completely with another is measured; in most cases one solution is added gradually from a **burette** into another solution in a **conical flask** until the **equivalence point** is reached; the equivalence point of a titration is the point at which the volume of solution added from the burette is just enough to react completely with the solution in the conical flask.
* The phrase “solution A is titrated against solution B” means that a known volume of solution A should be placed in a conical flask and solution B placed in a burette; solution B should be added to solution A until the equivalence point is reached.
* Titrations can be used in all three of the main types of inorganic reaction:

1. **Acid-base** reactions
2. **Precipitation** reactions
3. **Redox** reactions

The equivalence point needs to be clearly visible; in most cases, a suitable indicator is added to the conical flask, although some redox reactions are auto-indicating

* The main purpose of titrations is to determine the **concentration** of a solution; this is known as **standardisation**. A solution whose concentration is known accurately is known as a standard solution; concentration can be expressed as **molarity**, **mass concentration** or **normality**

Molarity: moles of solute per dm3 of solution (moldm-3)

Mass concentration: mass of solute per dm3 of solution (gdm-3)

Normality: equivalents of solute per dm3 of solution (Eqdm-3)

Once one solution has been standardised, it can be used to standardise the solution it is reacting with.

* Titrations can also be used to determine the molar mass or percentage purity of a solid, if it is soluble in water; a standard solution of the substance should be prepared first, using a volumetric flask
* The main apparatus used in titrations are pipettes, burettes and conical flasks; volumetric flasks are also used if a standard solution needs to be prepared from a solid or from a concentrated solution

* A **pipette** is an apparatus used to deliver a known volume of solution accurately into another container; most pipettes have a single graduation mark and can therefore only be used to deliver a fixed volume of solution (usually 5 cm3, 10 cm3 or 25 cm3)

Using a pipette

1. The pipette should first be rinsed with the liquid to be pipetted
2. The pipette should then be filled using a pipette filler to around 2 cm above the graduation mark
3. The filler should then be removed quickly and replaced with a finger
4. The pressure of the finger should then be relaxed until the base of the meniscus lies on the graduation line; the pipette should be held vertically with the line at eye level to confirm this
5. The pipette should then be moved so its tip is above the container (usually a conical flask) into which liquid is to be transferred, and then be allowed to empty into the container under gravity
6. The tip of the pipette should then be placed into the solution for one second; no further attempt should be made to remove any liquid remaining in the pipette

When used correctly the measurement error in a pipette should only be 0.05 cm3.

* A **burette** is an apparatus used to deliver a variable quantity of solution accurately into another container; the burette most widely used in laboratory chemistry can deliver volumes of up to 50 cm3

Preparing a burette for first use

1. The burette, including the tip, should first be rinsed with the liquid it is being used to deliver
2. The burette should then be fixed into a suitable stand and clamped vertically; the height of the tip above the ground should be just enough to pass an upright conical flask underneath it
3. The burette should then be partially filled using a funnel; liquid should be allowed to flow out until the tip of the burette is completely full of liquid; it may be necessary to flick the tip with a fingernail to dislodge any air bubbles

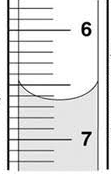
* A **titration** is then carried out as follows:

Carrying out a titration

1. The burette should then be refilled to somewhere in between 1 cm3 and 5 cm3 and the funnel removed
2. The level of the base of the meniscus should then be read to 2 decimal places (the second decimal place should be 0 or 5 – an estimation of whether the meniscus level lies on a mark (0) or in between two marks (5)); this volume is called the initial volume and it should be clearly recorded
3. The liquid should be allowed to flow out of the burette into the conical flask until the equivalence point of the titration has been reached; at the first attempt, a suitable flow rate is approximately 1 cm3 per second; the conical flask should be swirled continuously
4. The new level of the base of the meniscus should then be read to 2 decimal places; this volume is called the final volume and it should be clearly recorded
5. The difference between the initial and final volumes is called the titre volume
6. The entire process should then be repeated, using a new sample of solution in the conical flask; on this occasion the liquid can be allowed to flow fast until the volume delivered is around 3 cm3 less than the first titre volume, and then the flow rate should be reduced to one drop per second, swirling the conical flask continuously, until the equivalence point is reached again and the final volume clearly recorded to 2 decimal places
7. The process should be repeated indefinitely until two titre volumes within 0.1 cm3 of each other have been recorded

## Reading a burette to 2 dp

Look closely at the level of the base of the meniscus; if it is sitting on a graduation mark, use 0 as your second decimal place; if it is sitting in between graduation marks, use 5 as your second decimal place. The correct reading of the burette opposite is 6.65 cm3



* The results should be recorded in a table as follows:

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Titration No. | Rough | 1 | 2 | 3 | 4 | 5 |
| Final volume/cm3 |  |  |  |  |  |  |
| Initial volume/cm3 |  |  |  |  |  |  |
| Titre volume/cm3 |  |  |  |  |  |  |
| Concordant |  |  |  |  |  |  |
| Average titre/cm3 |  | | | | | |

* The two values within 0.1 cm3 of each other should be ticked as concordant; in some cases a titre volume may be concordant with two previous titre volumes; in this case the two titres within 0.05 cm3 of each other should be used; if all three are 0.1 cm3 apart then all three should be used; the rough titration should not be used as one of the concordant values
* The average of the concordant titre volumes should be calculated and recorded to one decimal place
* An averaged titre volume should be accurate to 0.1 cm3 if all procedures have been followed correctly
* A **volumetric flask** is in apparatus designed to contain a specific amount of solution, usually 250 cm3; it is used to prepare standard solutions from solids (by dissolving) or from concentrated solutions (by dilution)

Preparing a standard solution

1. Weigh out accurately an agreed mass of the solid on a piece of paper and transfer it to a clean beaker; weigh the paper again after the transfer to ensure the mass transferred is accurately known
2. Dissolve the solid in around 50 cm3 of water
3. Transfer the solution into a volumetric flask using a funnel; rinse out the beaker twice into the volumetric flask to ensure that all washings are transferred
4. Shake the mixture well, and then add water, continuing to shake, until the base of the meniscus of the water lies on the graduation mark; continue to shake well

When used correctly the measurement error in a volumetric flask should only be 0.2 cm3.

**Week 6 - Acid-Base Titrations**

* Most acids and bases are colourless; an indicator is therefore required to identify the equivalence point; during acid-base titrations, a large and sudden change in pH occurs at the equivalence point and this point can therefore be identified by using an indicator which changes colour over the same pH range; the pH change which occurs at the equivalence point depends on the acid-base properties of the salt formed, which in turn depends on the strength of the acid and base used in the titration. A general guide for 1 moldm-3 solutions is as follows (note that these are guidelines only; the exact pH change depends on the Ka and Kb values of the acid and base and on the molarity of the solutions):

|  |  |  |
| --- | --- | --- |
| Acid | Base | Approximate pH change at equivalence point |
| Strong | strong | 3 – 11 |
| Weak | strong | 7 – 11 |
| Strong | weak | 3 – 7 |
| Weak | weak | no sharp change |

* The indicators most frequently used in titrations are methyl orange and phenolphthalein; each indicator has a characteristic **end-point** (equal to its pKIn value) which is the pH at which the indicator changes colour, most indicators change colour over a pH range approximately equal to pKIn ± 1

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Indicator | end-point (pKIn) | pH range of colour change | Colour in acid | Colour in alkali | suitability |
| Methyl orange | 3.7 | 3.1 – 4.4 | Pink | Yellow | Strong acids only |
| Phenolphthalein | 9.3 | 8.3 – 10.0 | Colourless | Pink | Strong bases only |

* Either indicator can be used for titrations between strong acids and strong bases; if the acid is weak, phenolphthalein should be used; if the base is weak, methyl orange should be used; there is no suitable indicator for weak acid-weak base titrations
* Because indicators themselves have acid-base properties, they will influence the titre volume and therefore as little of the indicator should be added as possible. Three drops of indicator is always sufficient for a 25 cm3 sample of solution in a conical flask

**Apparatus needed per group:**

50 cm3 burette with clamp, stand and boss or suitable alternative

25 cm3 pipette and pipette filler

conical flask

white tile

two 100 cm3 beakers

access to a standard borax solution (B) (ask a technician for its mass concentration)

access to methyl orange indicator and a suitable dropping pipette

access to phenolphthalein indicator and a suitable dropping pipette

access to the hydrochloric acid solution (A)

access to the sodium hydroxide solution (E)

access to the unknown organic acid solution (D) (ask the technician for its mass concentration)

access to limewater (C) and a suitable measuring cylinder

access to the solution containing sodium carbonate and sodium hydrogencarbonate (F)

access to barium chloride solution and a suitable measuring cylinder

**Note:** You may carry out the experiments in any order, but Experiment 5 must be carried out either first or last

**Note:** If you do not have time to carry out all six experiments, ensure that you complete Experiments 4 and 5 individually; you may team up with your partner group to ensure that you complete all of experiments 1, 2 and 3 between the two groups; you may not share results outside the two groups.

# **Experiment 1 – Standardisation of a solution of hydrochloric acid using borax**

Borax (Na2B4O7·10H2O) is a weak base whose concentration remains stable over time. It is therefore useful in the standardisation of acids. Borax dissolves in water to give the tetraborate ion B4O72-, which is diprotic and reacts with strong acids in solution as follows: B4O72- + 2H+ + 5H2O → 4 H3BO3

Hydrochloric acid (HCl) is a strong monoprotic acid

**Procedure:**

1. Prepare the burette for first use with Solution A (if you have not already done so)
2. Pipette 25.0 cm3 of Solution B into a conical flask
3. Add three drops of methyl orange indicator
4. Titrate Solution B against Solution A
5. Repeat the titration until two concordant titre values have been obtained
6. Record your results in a table

# **Experiment 2 – Standardisation of a solution of sodium hydroxide using HCl**

Sodium hydroxide (NaOH) is a strong monoprotic base. It reacts with strong acids in solution as follows: OH- + H+ → H2O

**Procedure:**

1. Prepare the burette for first use with Solution A (if you have not already done so)
2. Pipette 25.0 cm3 of Solution E into a conical flask
3. Add three drops of methyl orange indicator
4. Titrate Solution E against Solution A
5. Repeat the titration until two concordant titre values have been obtained
6. Record your results in a table

# **Experiment 3 – Determination of the solubility of calcium hydroxide using HCl**

Calcium hydroxide (Ca(OH)2) is a sparingly soluble base, which dissolves in water as follows:

(Ca(OH)2) Ca2+ + 2OH-

The solubility of a substance is the concentration of its saturated solution. The hydroxide ions in the solution react with strong acids in solution as follows: OH- + H+ → H2O

A saturated solution of Ca(OH)2 is commonly known as limewater.

**Procedure:**

1. Prepare the burette for first use with Solution A (if you have not already done so)
2. Using a measuring cylinder, transfer 25 cm3 of Solution C into a conical flask
3. Add three drops of methyl orange indicator
4. Titrate Solution C against Solution A
5. Repeat the titration until two concordant titre values have been obtained
6. Record your results in a table

**Experiment 4** **- Determination of a mixture of sodium carbonate and sodium hydrogencarbonate using HCl**

Sodium carbonate (Na2CO3) dissolves in water to give carbonate ions (CO32-). CO32- is a weak diprotic base; when a strong acid is gradually added to it, it initially forms the hydrogencarbonate ion (HCO3-) as follows:

CO32- + H+ 🡪 HCO3-

The equivalence point of this titration is associated with a pH change from approximately 10 – 8 and can therefore be detected using phenolphthalein.

Sodium hydrogencarbonate (NaHCO3) dissolves in water to give HCO3- ions; HCO3- is itself a weak base, and when a strong acid is added to it, it reacts as follows:

HCO3- + H+ 🡪 CO2 + H2O

The equivalence point of this titration is associated with a pH change from approximately 5 – 3 and can therefore be detected using methyl orange.

When a mixture of Na2CO3 and NaHCO3 is titrated against a strong acid, the carbonate ions will react with the acid first and the first equivalence point will be detected when this reaction is complete. The HCO3- formed during this titration and the HCO3- originally present with both then react with the acid, and the second equivalence point will be detected when this reaction is complete.

**Procedure:**

1. Prepare the burette for first use with Solution A (if you have not already done so)
2. Pipette 25.0 cm3 of Solution F into a conical flask
3. Add three drops of phenolphthalein indicator
4. Titrate Solution F against Solution A until the equivalence point is reached
5. Add three drops of methyl orange indicator
6. Continue to titrate Solution F against Solution A until the equivalence point is reached
7. Record your results in a table

# **Experiment 5 – Identification of an organic acid using NaOH**

Most organic acids are weak monoprotic acids of the form HA. They react with solutions of strong bases as follows: HA + OH- 🡪 A- + H2O

**Procedure:**

1. Prepare the burette for first use with Solution D
2. Pipette 25.0 cm3 of Solution E into a conical flask
3. Add three drops of phenolphthalein indicator
4. Titrate Solution E against Solution D
5. Repeat the titration until two concordant titre values have been obtained
6. Record your results in a table

|  |
| --- |
| **Report**  Your report must include the following:   1. All of your original titration results tables with your report, signed by the lecturer allocated to your group (20 marks) 2. A record of the mass concentrations of the borax and organic acid solutions (1 mark) 3. Calculations of the following, showing clear working and chemical equations when necessary:  * The molarity of solution A (HCl) (from Experiment 1) * The molarity of solution E (NaOH) (from Experiment 2) * The molarity of solution C (limewater) (from Experiment 3) * The molar mass of the organic acid in solution D (from Experiment 5) * The molarity of Na2CO3 in solution F (from Experiment 4.1) * The molarity of NaHCO3 in solution F (from Experiment 4.1 and Experiment 4.2 independently)   (24 marks)   1. A comparison of your two results for the molarity of NaHCO3 in solution F (2 marks)   TOTAL 47 marks for report + 3 marks for attendance and contribution = 50 marks |

Use the following questions to help you with your report:

1. Use the mass concentration of B to calculate the molarity of Solution B
2. Hence calculate the number of moles of borax in 25 cm3 of solution B
3. Hence deduce the number of moles of HCl needed to react with this quantity of borax
4. Use your titre volume from Experiment 1 and your answer to Q3 to calculate the **molarity of solution A**
5. Use your titre volume from Experiment 2 and your answer to Q4 to calculate the number of moles of HCl needed for the titration in Experiment 2
6. Hence deduce the number of moles of NaOH present in 25 cm3 of solution E
7. Hence calculate the **molarity of solution E**
8. Use your titre volume from Experiment 3 and your answer to Q4 to calculate the number of moles of HCl needed for the titration in Experiment 3
9. Hence deduce the number of moles of Ca(OH)2 present in 100 cm3 of solution C
10. Hence calculate the **molarity of solution C**
11. Use your answer to Q6 to deduce the number of moles of the organic acid required to react with 25 cm3 of solution E
12. Use your titre volume from Experiment 5 and your answer to Q11 to deduce the molarity of solution D
13. Use the mass concentration of D and your answer to Q11 to calculate the **molar mass of the organic acid** **in solution D**
14. Use your first titre volume in Experiment 4.1 and your answer to Q4 to calculate the number of moles of HCl needed to react with the CO32- in solution F
15. Hence deduce the number of moles of CO32- in 25 cm3 of solution F
16. Hence calculate the **molarity of the Na2CO3 in solution F**
17. Use your second titre volume in Experiment 4 and your answer to Q4 to deduce the number of moles of HCl needed to react with the HCO3- in 25 cm3 of solution F after the first equivalence point has been reached
18. Hence deduce the number of moles of HCO3- in 25 cm3 of solution F after the first equivalence point has been reached
19. Use your answers to Q15 and Q18 to deduce the number of moles of HCO3- originally present in 25 cm3 of solution F
20. Hence calculate the **molarity of the NaHCO3 in solution F from Experiment 4**

**Week 7 - Precipitation Titrations**

* Precipitation titrations are very useful for the determination of chloride ions in solution; chloride ions react with silver ions to give the very insoluble precipitate of AgCl: Ag+(aq) + Cl-(aq) 🡪 AgCl(s); in most cases the solution containing chloride ions is titrated against a solution of silver nitrate (AgNO3) until all of the Cl- has precipitated as AgCl
* The equivalence point of this reaction can be observed in two ways:

1. The Mohr method: a small quantity of potassium chromate (K2CrO4) is added to the conical flask as an indicator; the CrO42- ions in the solution form a red precipitate with Ag+ ions (Ag2CrO4); but because AgCl is less soluble than Ag2CrO4, AgCl precipitates first and the red precipitate of Ag2CrO4 is only observed when there are no Cl- ions remaining in solution
2. The Fajans method: dichlorofluorescin is an organic compound which changes its colour to violet when it binds to Ag+ ions, but since Ag+ will precipitate with Cl- until there are no Cl- ions remaining, the violet colour is only observed after the equivalence point; the violet colour is easier to see if starch is also added

Both methods only work in neutral solution

**Apparatus needed per group:**

50 cm3 burette with clamp, stand and boss or suitable alternative

25 cm3 pipette and pipette filler

250 cm3 volumetric flask

conical flask

white tile

two 100 cm3 beakers

funnel

access to solid potassium chloride (to prepare S)

access to a 2 dp mass balance

access to a solution containing a mixture of KCl and HCl (Q)

access to a solution of AgNO3 (P)

access to a solution of NaOH (E) (ask the technician for its molarity)

access to a solution containing a mixture of KCl and NaCl (R) (ask the technician for its mass concentration)

access to CaCO3 powder

access to potassium chromate solution and a suitable dropping pipette

access to dichlorofluoroescein solution and a suitable dropping pipette

access to starch solution and a suitable dropping pipette

access to phenolphthalein indicator and a suitable dropping pipette

**Note:** You may carry out Experiments 1 and 2 in either order. You must carry out Experiment 3 last.

**Experiment 1 – Preparation of a standard solution of potassium chloride and its use to standardise a solution of AgNO3**

Potassium chloride (KCl) is a soluble chloride which can be easily prepared as a standard solution of known molarity.

**Procedure:**

1. Prepare the burette for first use with Solution P (if you have not used Solution P in the burette before)
2. Weigh out accurately 0.90 – 1.0 g of KCl and use it to prepare a standard solution of KCl (Solution S
3. Pipette 25.0 cm3 of Solution S into a conical flask
4. Add 1 cm3 of K2CrO4 indicator
5. Titrate Solution S against Solution P
6. Repeat the titration until two concordant titre values have been obtained
7. Record your results in a table

**Experiment 2 – Determination of a solution containing sodium chloride and KCl**

Sodium chloride (NaCl) is a soluble chloride.

**Procedure:**

1. Prepare the burette for first use with Solution P (if you have not used Solution P in the burette before)
2. Pipette 25.0 cm3 of Solution R into a conical flask
3. Add three drops of dichlorofluorescein and 1 cm3 of starch solution
4. Titrate Solution R against Solution P
5. Repeat the titration until two concordant titre values have been obtained
6. Record your results in a table

**Experiment 3 – Determination of a solution containing HCl and KCl**

Calcium carbonate (CaCO3) is an insoluble base which can be used to neutralise strong acids such as HCl:

CaCO3 + 2H+ 🡪 Ca2+ + CO2 + H2O

**Procedure:**

**Part 1**

1. Pipette 25.0 cm3 of Solution Q into a conical flask
2. Add a slight excess of CaCO3; the solution should remain slightly milky after swirling
3. Add either K2CrO4 or dichlorofluorescein/starch as per Experiment 1 or 2 (your choice)
4. Titrate Solution Q against Solution P
5. Repeat the titration until two concordant titre values have been obtained
6. Record your results in a table

**Part 2**

1. Prepare the burette for first use with Solution Q
2. Pipette 25.0 cm3 of Solution E into a conical flask
3. Add three drops of phenolphthalein indicator
4. Titrate Solution E against Solution Q
5. Repeat the titration until two concordant titre values have been obtained
6. Record your results in a table

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| **Report**  Your report must include the following:   1. All of your original titration results tables with your report, signed by the lecturer allocated to your group (12 marks) 2. A record of the mass of KCl used in Experiment 1 (2 marks) 3. Calculations of the following, showing clear working and chemical equations when necessary:  * The molarity of Solution S (KCl) (from Experiment 1) * The molarity of Solution P (AgNO3) (from Experiment 1) * The molarity of KCl and NaCl in Solution R (from Experiment 2) * The molarity of HCl and KCl in solution Q (from Experiment 3)  1. marks)   4) An explanation for why it is necessary to add CaCO3 in Experiment 3 (2 marks)   1. An evaluation of the different methods of indicating silver chloride precipitation titrations (2 marks) 2. An estimate of the total percentage apparatus error in your calculation of the molarity of Solution P from Experiment 1, taking into consideration the mass measurement, the pipette, the burette and the volumetric flask (5 marks)   TOTAL 43 marks for report + 2 marks for attendance and contribution = 45 marks |

Use the following questions to help you with your report:

1. Use your mass of KCl to calculate the **molarity of your Solution S**
2. Hence calculate the number of moles of KCl in 25 cm3 of Solution S
3. Hence deduce the number of moles of AgNO3 needed to react with this quantity of KCl
4. Use your titre volume from Experiment 1 and your answer to Q3 to calculate the **molarity of Solution P**
5. Use your titre volume from Experiment 2 and your answer to Q4 to calculate the number of moles of AgNO3 needed to react with Solution R
6. Hence deduce the number of moles of Cl- present in 25 cm3 of Solution R
7. Hence calculate the molarity of Cl- in Solution R
8. Let the total molarity of Cl- be n, the total mass concentration be m and the molarity of NaCl be x:
9. write an expression for the mass concentration of NaCl in terms of x
10. write an expression for the molarity of KCl in terms of n and x
11. hence write an expression for the mass concentration of KCl in terms of n and x
12. hence write an expression for m in terms of n and x
13. Use the mass concentration of Solution R, your answer to Q7 and your expression in Q8d to calculate the **molarity of NaCl in Solution R**
14. Use your answers to Q7 and Q8e to calculate the **molarity of KCl in Solution R**
15. Use your first titre volume in Experiment 3 and your answer to Q4 to calculate the number of moles of AgNO3 needed to react with Solution Q
16. Hence deduce the total number of moles of Cl- in 25 cm3 of solution Q
17. Hence calculate the total molarity of Cl- in Solution Q
18. Use the molarity of solution E to calculate the number of moles of NaOH in 25 cm3 of Solution E
19. Hence deduce the number of moles of HCl needed to react with 25 cm3 of Solution E
20. Use your second titre volume in Experiment 3 and your answer to Q13 to calculate the **molarity of HCl in Solution Q**
21. Use your answers to Q11 and Q14 to calculate the **molarity of KCl in Solution Q**

**Week 8: Redox Titrations**

* Reducing agents in solution can be determined by titrating them against a standard solution of potassium manganate (VII) (KMnO4); the manganate acts as an oxidising agent as follows:

MnO4- + 8H+ + 5e- 🡪 Mn2+ + 4H2O

The half-equation requires the presence of acid; sulphuric acid (H2SO4) is used for this purpose as it is a strong acid and the SO42- ion does not interfere with the redox reaction (unlike Cl- or NO3-); the H2SO4 is added to the reducing agent conical flask before starting the titration

The MnO4- ion is an intense purple colour; as it is added to the reducing agent, the purple colour disappears, as the Mn2+ is (almost) colourless; at the equivalence point, when the reducing agent in the conical flask has been used up, the excess MnO4- turns the solution pink; the titration is therefore auto-indicating and no additional indicator is required

* Oxidising agents in solution can be determined by reacting them with an excess of potassium iodide solution (KI); the I- ions reduce the oxidising agent and are oxidised to iodine (I2): 2I- 🡪 I2 + 2e-

The I2 produced can be titrated against a standard solution of sodium thiosulphate (Na2S2O3) according to the following reaction: I2 + 2S2O32- 🡪 S4O62- + 2I-

The brown colour of the I2 becomes gradually paler as its concentration in the conical flask decreases, eventually turning pale yellow and then colourless at the equivalence point; if a small amount of starch solution is added when the solution is yellow, the remaining iodine forms a deep blue colour which remains intense until the equivalence point is reached, when it disappears; the starch therefore acts as the indicator for this titration

**Apparatus needed per group:**

50 cm3 burette with clamp, stand and boss or suitable alternative

25 cm3 pipette and pipette filler

250 cm3 volumetric flask

conical flask

white tile

two 100 cm3 beakers

funnel

access to solid ferrous ammonium sulphate (to prepare Solution O)

access to a 2 dp mass balance

access to potassium manganate (VII) solution (K)

access to potassium dichromate (VI) solution (L)

access to sodium thiosulphate solution (M)

access to copper sulphate solution (N)

access to sulphuric acid and a suitable measuring cylinder

access to potassium iodide solution and a suitable measuring cylinder

access to starch solution and a suitable dropping pipette

**Note:** You should carry out Experiment 1 before Experiment 2; you can carry out Experiments 3 and 4 in either order but either complete Experiments 1 and 2 before starting Experiments 3 and 4, or complete Experiments 3 and 4 before starting Experiment 1

**Experiment 1 – Preparation of a standard solution of ammonium iron (II) sulphate and its use to standardise a solution of potassium manganate (VII)**

Ammonium iron (II) sulphate ((NH4)2Fe(SO4)2.6H2O) contains Fe2+ ions, which are reducing agents and can react with MnO4- ions in acidic conditions: Fe2+ 🡪 Fe3+ + e-

Because the Fe3+ ions are orange in colour, they can interfere with the visibility of the equivalence point.

**Procedure:**

1. Prepare the burette for first use with Solution K
2. Weigh out accurately 4.9 – 5.0 g of (NH4)2Fe(SO4)2.6H2O and use it to prepare a standard solution of (NH4)2Fe(SO4)2.6H2O (Solution O) using H2SO4 instead of water
3. Pipette 25.0 cm3 of Solution O into a conical flask
4. Add approximately 25 cm3 of H2SO4
5. Titrate Solution O against Solution K
6. Repeat the titration until two concordant titre values have been obtained
7. Record your results in a table

**Experiment 2 – Determination of a solution of potassium dichromate (VI) using Fe2+ and back-titration against KMnO4**

Potassium dichromate (K2Cr2O7) contains the dichromate ion Cr2O72-, which is an oxidising agent in acidic conditions: Cr2O72- + 14H+ + 6e- 🡪 2Cr3+ + 7H2O; one way of determining it is to react it with an excess of Fe2+ ions, which will react with the dichromate as follows: Cr2O72- + 14H+ + 6Fe2+ 🡪 2Cr3+ + 7H2O + 6Fe3+.

The amount of Fe2+ remaining after this reaction can be determined by titration against KMnO4.

**Procedure:**

1. Pipette 25.0 cm3 of Solution L into a conical flask
2. Pipette 35.0 cm3 of Solution O into the same conical flask (by pipetting 25.0 cm3 and then 10.0 cm3)
3. Add approximately 35 cm3 of H2SO4, swirling well to allow the reaction to complete
4. Add approximately 5 cm3 of H3PO4
5. Titrate the mixture of Solution O and Solution R against Solution K
6. Repeat the titration until two concordant titre values have been obtained
7. Record your results in a table

**Experiment 3 – Determination of a solution of Na2S2O3 using K2Cr2O7 and KI**

**Procedure:**

1. Prepare the burette for first use with Solution M (if you have not already done so)
2. Pipette 25.0 cm3 of Solution L into a conical flask
3. Add approximately 25 cm3 of H2SO4
4. Add approximately 10 cm3 of KI, swirling well to allow the reaction to complete
5. Titrate Solution L against Solution M until the brown colour has faded slightly
6. Add 1 cm3 of starch solution and continue titrating until the blue colour disappears
7. Repeat the titration until two concordant titre values have been obtained
8. Record your results in a table

**Experiment 4 – Determination of a solution of copper sulphate using KI and Na2S2O3**

Copper sulphate (CuSO4) contains Cu2+ ions. Cu2+ is an oxidising agent and can react with I- ions as follows:

2Cu2+ + 4I- 🡪 2CuI + I2

**Procedure:**

1. Prepare the burette for first use with Solution M (if you have not already done so)
2. Pipette 25.0 cm3 of Solution N into a conical flask
3. Add approximately 10 cm3 of KI, swirling well to allow the reaction to complete
4. Add approximately 20 cm3 of distilled water and swirl again
5. Titrate Solution N against Solution M until the brown colour has turned yellow
6. Add 1 cm3 of starch solution and continue titrating until the blue colour disappears
7. Repeat the titration until two concordant titre values have been obtained
8. Record your results in a table

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| **Report**  Your report must include the following:   1. All of your original titration results tables with your report, signed by the lecturer allocated to your group (12 marks) 2. A record of the measurement of the mass of (NH4)2Fe(SO4)2.6H2O used to prepare Solution O (2 marks) 3. Calculations of the following, showing clear working and chemical equations when necessary:  * The molarity of Solution O ((NH4)2Fe(SO4)2.6H2O) (from Experiment 1) * An equation for the reaction between Fe2+ in Solution O and MnO4- in Solution K * The molarity of Solution K (KMnO4) (from Experiment 1) * The molarity of Solution R (K2Cr2O7) (from Experiment 2) * An equation for the reaction between Cr2O72- in Solution R and I- * The molarity of Solution M (Na2S2O3) (from Experiment 3) * The molarity of solution N (CuSO4) (from Experiment 4)   (20 marks)   1. An explanation for why the starch indicator used in Experiments 3 and 4 should be added when the solution is yellow, rather than at the start of the titration (2 marks) 2. An estimate of the total percentage apparatus error in your calculation of the molarity of Solution P from Experiment 1, taking into consideration the mass measurement, the pipette, the burette and the volumetric flask (5 marks)   TOTAL 41 marks for report + 2 marks for attendance and contribution = 43 marks |

Use the following questions to help you with your report:

1. Use your mass of (NH4)2Fe(SO4)2.6H2O to calculate the **molarity of your Solution O**
2. Hence calculate the number of moles of Fe2+ in 25 cm3 of Solution O
3. **Write an equation for the reaction between Fe2+ and MnO4-**
4. Use your answers to Q2 and Q3 to deduce the number of moles of MnO4- required to react with the Fe2+ in the conical flask in Experiment 1
5. Use your titre volume from Experiment 1 and your answer to Q4 to calculate the **molarity of Solution K**
6. Use your answer to Q1 to calculate the number of moles of Fe2+ added to the conical flask in Experiment 2
7. Use your titre volume from Experiment 2 and your answer to Q5 to calculate the number of MnO4- needed to react with the excess Fe2+ in Experiment 2
8. Use your answers to Q3 and Q7 to calculate the moles of Fe2+ present in the conical flask in Experiment 2 before the titration in Experiment 2
9. Use your answers to Q6 and Q8 to calculate the number of moles of Fe2+ which reacted with the Cr2O72- in Experiment 2
10. Use your answer to Q9 and the equation for the reaction between Fe2+ and Cr2O72- to deduce the number of moles of Cr2O72- added to the conical flask in Experiment 2
11. Hence calculate the **molarity of Solution R**
12. Use your answer to Q11 to calculate the number of moles of Cr2O72- added to the conical flask in Experiment 3 (this should be the same as your answer to Q10)
13. **Write an equation for the reaction between Cr2O72- and I-**
14. Use your answers to Q12 and Q13 to deduce the number of moles of I2 produced in the reaction in Experiment 3
15. Hence deduce the number of moles of S2O32- needed to react with this amount of I2
16. Use your titre volume from Experiment 3 and your answer to Q15 to calculate the **molarity of Solution M**
17. Use your answer to Q16 and your titre volume from Experiment 4 to deduce the number of moles of S2O32- used in the titration
18. Hence deduce the number of moles of I2 present in the conical flask
19. Hence deduce the number of moles of Cu2+ added to the conical flask
20. Hence calculate the **molarity of Solution N**

**Week 9 – Chemical Equilibrium**

**Chemical Principles**

Reactions which can proceed in both directions depending on the relative amounts of reactants and products are said to be **reversible**; such reactions eventually reach a state of **dynamic equilibrium**, which means that the rate of the forward reaction and the rate of the reverse reaction are the same; as a result the amounts of reactants and products remains constant.

The relative amounts of reactants and products in the system when dynamic equilibrium has been reached is known as the **position of equilibrium**; it can be quantified using an equilibrium constant.

If the conditions are changed after a system has reached dynamic equilibrium, the position of equilibrium will also change; it is possible to make qualitative predictions about how the position of equilibrium will move using **Le Chatelier’s principle**: “If a change is imposed on a system in dynamic equilibrium, the equilibrium will move in such a way as to oppose the effect of that change”.

Le Chatelier’s principle can be applied as follows:

* if extra reactant is added, the position of equilibrium will move to the right to remove that reactant
* if some reactant is removed, the position of equilibrium to move to the left to replace that reactant
* if extra product is added, the position of equilibrium will move to the left to remove that product
* if some product is removed, the position of equilibrium will move to the right to replace that product
* if the temperature is increased, the position of equilibrium will move in the endothermic direction to lower the temperature
* if the temperature is decreased, the position of equilibrium will move in the exothermic direction to increase the temperature

**Experiment 1 – investigating the reaction between Fe3+ and SCN- ions**

Fe3+ ions react with SCN- ions in aqueous solution as follows:

[Fe(H2O)6]3+(aq) + SCN-(aq)[Fe(H2O)5(SCN)]2+(aq) + H2O(l)

[Fe(H2O)5(SCN)]2+ is a deep red colour; the position of this equilibrium can be studied by observing the intensity of the red colour in the solution.

**Equipment needed:** eight test tubes, one test tube rack, one 100 cm3 beaker, access to water bath, access to 0.1 moldm-3 FeCl3, 0.1 moldm-3 NH4SCN and 0.1 moldm-3 NH4Cl, each with their own dropping pipette

**Procedure:**

1. Add 2 cm3 of iron (III) chloride (FeCl3) and 2 cm3 of ammonium thiocyanate (NH4SCN) to 100 cm3 of distilled water in a beaker. Ensure that the solutions are well mixed.
2. Pour the resulting solution into four different test tubes, so that they are all approximately half-full; label the test tubes A, B, C and D.
3. Add 2 cm3 of iron (III) chloride to test tube A.
4. Add 2 cm3 of ammonium thiocyanate to test tube B.
5. Add 2 cm3 of ammonium chloride solution to test tube C.
6. Add 2 cm3 of water to test tube D.
7. Pour half of each solution into four more test tubes labelled E, F, G and H.
8. Warm test tubes E, F, G and H in a water bath for a few minutes.
9. Observe how the intensity of the colour changes following each action.

**Experiment 2 – investigating the hydrolysis of bismuth (III) chloride**

Bismuth chloride is hydrolysed according to the following equation:

BiCl3(aq) + H2O(l)BiOCl(s) + 2HCl(aq)

BiOCl is insoluble and appears as turbidity; the position of this equilibrium can be studied by observing the degree of turbidity in the mixture.

**Equipment needed:** three test tubes, one test tube rack, one 10 cm3 measuring cylinder, one burette, access to a solution of BiCl3, access to a water bath, tongs

**Procedure:**

1. Use a 10 cm3 measuring cylinder to pour 2 cm3 of BiCl3 into a test tube.
2. Prepare a burette for first use with distilled water; record the initial reading on the burette.
3. Add water dropwise from the burette to the test tube containing the BiCl3 until the solution becomes turbid; note the volume of water which must be added before this happens.
4. Use a 10 cm3 measuring cylinder to pour 2 cm3 of BiCl3 into another test tube.
5. Heat the mixture in a water bath until its temperature is approximately 50 oC.
6. Add water dropwise from the burette to the test tube containing the BiCl3 until the solution becomes turbid; note the volume of water which must be added before this happens.
7. Use a 10 cm3 measuring cylinder to pour 2 cm3 of BiCl3 into another test tube.
8. Heat the mixture in a water bath until its temperature is approximately 80 oC.
9. Add water dropwise from the burette to the test tube containing the BiCl3 until the solution becomes turbid; note the volume of water which must be added before this happens.

**Experiment 3 – chromate ions and dichromate ions**

Chromium can exist in the +6 oxidation state either as the yellow chromate ion, CrO42- or as the orange dichromate ion, Cr2O72-, depending on the pH:

CrO42-(aq) + 2H+(aq)Cr2O72-(aq) + H2O(l)

The position of equilibrium in this reaction can be studied by observing whether the solution appears as yellow or orange.

**Equipment needed:** two test tubes, one test tube rack, access to 0.04 moldm-3 K2CrO4, 0.1 moldm-3 HCl and 0.1 moldm-3 NaOH, each with its own dropping pipette, access to a water bath, tongs

**Procedure:**

1. Pour around 4 cm3 of K2CrO4 into a test tube.
2. Add dilute HCl to the test tube dropwise until the solution just becomes orange.
3. Transfer half of the solution into another test tube.
4. Add one or two drops of NaOH to the second test tube – just enough to turn the solution yellow again.
5. Place both test tubes in a water bath for five minutes and observe any change in colour.

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| **Report**     1. Record all your observations from Experiment 1 in a table; explain your observations in each test tube and deduce whether the reaction in Experiment 1 is exothermic or endothermic. 2. Record all your observations from Experiment 2 in a table; explain your observations and deduce whether the reaction in Experiment 2 is exothermic or endothermic. 3. Record all your observations from Experiment 3 in a table; explain your observations and deduce whether the reaction in Experiment 3 is exothermic or endothermic. |

**Week 10 – Colorimetric Determination of pH**

**Chemical Principles**

The use of colour to analyse a substance is known as colorimetry. Colorimetry is useful in the determination of pH.

The **pH** of a solution is the negative logarithm of the hydrogen ion concentration: -log[H+].

One way to determine the pH of a solution is using an **acid-base indicator**; these are substances which change colour depending on the pH. Indicators change gradually over a pH range of around 2 units and the colour of the indicator can give an accurate measurement of the pH, provided that the pH is inside the pH range over which the indicator changes colour. Different indicators change colour over different pH ranges and so the approximate pH must be known before a suitable indicator can be selected:

* Phenolphthalein changes colour between pH 8.3 and pH 10.0
* Methyl orange changes colour between pH 3.2 and pH 4.4
* Methyl red changes colour between pH 4.4 and pH 6.3

**Universal Indicator** is a mixture of indicators, each of which changes colour at a different pH in such a way that universal indicator gives a slightly different colour over the full range of pH values from 3 to 11. It is thus a good way of determining the approximate pH of a solution whose pH is unkwown.

In order to determine the pH of a solution using indicators, it is necessary to compare the colour of the indicator in the unknown sample to the colour of the indicator in a solution of fixed, known pH known as a buffer solution. A **buffer** **solution** is a solution with a stable known pH which is not significantly affected by contamination.

Most buffer solutions are made by partially neutralising a weak acid. For example, if 0.05 moles of NaOH are added to 0.1 moles of CH3COOH, the NaOH will partially neutralise the CH3COOH, resulting in 0.05 moles of CH3COOH (a weak acid) and 0.05 moles of CH3COONa (a weak base) remaining in the solution. This mixture can neutralise both acidic and alkaline contamination and can therefore maintain a stable pH.

**Experiment 1 – Determining the approximate pH of a solution**

You are provided with eight buffer solutions with pH values of 3, 4, 5, 6, 7, 8, 9 and 10. Each solution has had two drops of universal indicator added to it.

1. Using a measuring cylinder, pour 5 cm3 each of solutions A, B, C, D and E into five separate test tubes.
2. Add two drops of universal indicator to each test tube and shake well.
3. Compare the colour of each solution to the colours of the buffer solution samples and hence deduce the pH of each of solutions A, B, C, D and E to the nearest 1 units.

**Experiment 2 – Determining the precise pH of a solution**

You are provided with Solution E, which is known to have a pH value of approximately 5. The pH of this solution can be accurately determined by preparing a number of solutions with the following pH values: 4.6, 4.8, 5.0, 5.2 and 5.4.

In this case, a single indicator which shows a significant colour change over this range is more useful than universal indicator; the pKIn of methyl red is 5.1 and it changes colour from red to yellow over a pH range of 4.4 – 6.3. It is therefore a good indicator to use for solutions covering this pH range.

The accurate pH of Solution E can therefore be determined by adding two drops of methyl red indicator to 5 cm3 of Solution E and comparing its colour to the five buffer solutions of similar pH, each of which has also had two drops of methyl red added to it.

The buffer solutions can be prepared as follows:

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| pH | Volume of 0.5 moldm-3 CH3COOH /cm3 | Volume of 0.5 moldm-3 NaOH /cm3 |
| 4.6 | 35.8 | 10.0 |
| 4.8 | 20.0 | 10.0 |
| 5.0 | 16.3 | 10.0 |
| 5.2 | 14.0 | 10.0 |
| 5.4 | 12.5 | 10.0 |

1. Set up two burettes, one containing 0.5 moldm-3 CH3COOH and the other containing 0.5 moldm-3 NaOH.
2. To five separate beakers, add the required quantities of ethanoic acid and sodium hydroxide to prepare buffer solutions with pH values of 4.6, 4.8, 5.0, 5.2 and 5.4. Shake the solutions well.
3. Using a measuring cylinder, pour 5 cm3 of each buffer solution into five different test tubes.
4. Add 2 drops of methyl red indicator to each test tube and shake well.
5. Place the five test tubes in a test tube rack in ascending order of pH.
6. Using a measuring cylinder, pour 5 cm3 of solution E into a test tube.
7. Add 2 drops of methyl red indicator to the test tube and shake well.
8. Compare the colour of Solution E to the colours of the buffer solution samples and hence deduce the pH of each of Solution E to the nearest 1 dp.

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| Report  1. State the pH of Solutions A, B, C and D and explain how you deduced them. 2. Given that the pKa of CH3COOH is 4.8, show that the mixing of 14.0 cm3 of 0.5 moldm-3 CH3COOH and 14.0 cm3 of 0.5 moldm-3 NaOH creates a buffer solution with pH 5.2 3. State the pH of Solution E and explain how you deduced it. 4. Explain how you could determine the pH accurately of a solution with a pH of around 6. Explain which acid you would use to prepare the buffer solution and which indicator you would use. |