**SS3 FIRST TERM E-LESSON NOTE**

**Volumetric Analysis (Titration)**

**Heat of Neutralization & Redox Titrations**

**Test for Common Gases**

**Qualitative Analysis I**

**Qualitative Analysis II**

**Qualitative Analysis III**

**Qualitative Analysis IV - Anions**

**Test for Fat and Oil; Protein; Starch**

**Petroleum I**

**Petroleum I**

Topic: Volumetric Analysis

Introduction

Volumetric analysis is an analytical method or procedure for working out the titre or concentration of an analyte in a solution. This is done by measuring the volume of a standard solution of an appropriate reagent whose precise concentration is already known.

Preparing A Standard Solution

A standard solution is a solution in which its concentration is known.

The steps taken in preparing a standard solution are:

Determine the volume and concentration that you want to prepare.

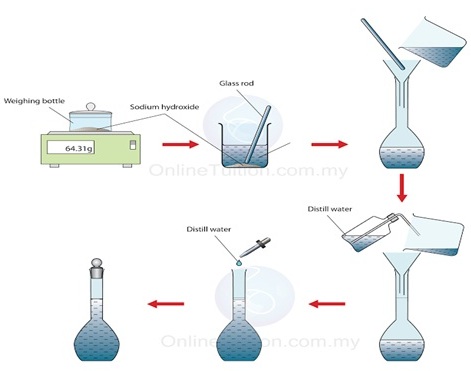
Calculate the mass of solute needed to give the required volume and concentration.

Weigh the solute

Dissolve the solute completely dissolved in distilled water and then transfer it to a volumetric flask partially filled with distilled water.

Add distilled water to the calibration mark of the volumetric flask.

Invert the flask and shake it to make sure thorough mixing.



Acid Base Titration

Some materials used during acid – base titration and precautions in using some of them

weighing balance

chemical balance

pipette

burette

retort stand

filter paper

funnel

white tile

standard volumetric flask

conical flask

Pipette

rinse the pipette with the solution it will be measured with e.g. base

avoid air bubbles in the pipette

make sure the mark to be read is at same level with your eye

do not blow the last drop on the burette

Burette

rinse the burette with acid or allow it to drain after rinsing

make sure the burette jar is filled

avoid air bubbles in the burette

make sure that burette is not leaking4remove the funnel befor taking your reading

avoid inconsistent burette reading

Conical flask

do not rinse with any of the solutions used in the titration but with distilled water

wash down with distilled water any drop of the solution that stick by the sides of the conical flask

Concentration of a Solution

The concentration of a solution tells you how much solute is dissolved in 1 unit volume of solution.

The volume of a solution is measured in dm³ (litres)  1 dm³ = 1000 cm³.

The amount of solute can be measured in grams or moles.

2 units of concentration used in chemistry are g dm-3 and mol dm-3

Concentration in g dm-3

Concentration is the number of moles of solute per liter of solution.

A concentration of 10 g dm-3 means there is 10 g of solute dissolved in1 dm3 of solution.

Concentration=Mass of solute (g) / Volume of solution (dm3)

Example 1:  
Calculate the concentration of the solution if 28g of NaOH is dissolve in 250cm3 of water.  
Answer:  
Mass of solute = 28g  
Volume of solvent = 250cm³ = 0.25dm³

Concentration = Mass / Volume

=28g / 0.25dm3

=112g/dm3

Concentration in mol dm-3 (Molarity)

Molarity is probably the most commonly used unit of concentration. It is the number of moles of solute per liter of solution.

A concentration of 2 mol dm-3 means there are 2 moles of solute dissolved in 1 dm3 of solution.

Molarity=Mole of solute(mol) / Volume of solution(dm3)

Example 2:  
What is the molarity of a solution made when water is added to 0.2 mol of CaCl2 to make 100 cm³ of solution? [RAM: Ca = 40; Cl = 35.5]

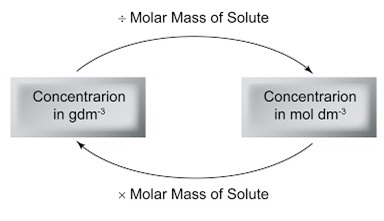
Answer:  
Number of mole of solute = 0.2 mol  
Volume of solvent = 100 cm³ = 0.1 dm³

Molarity = Number of Mole / Volume

= 0.2mol / 0.1dm3

=2mol/dm3

Conversion of Concentration Unit



The chart above shows how to convert the units of concentration from g dm-3 to mol dm-3 and vice versa.

The molar mass of the solute is equal to the relative molecular mass of the solute.

Example 3:  
The concentration of a Potassium chloride solution is 14.9 g dm-3. What is the molarity ( mol dm-3) of the solution? [Relative Atomic Mass: Cl = 35.5; K = 39]

Answer:  
Relative Formula Mass of Potassium Chloride (KCl)  
= 39 + 35.5 = 74.5

Molar Mass of Potassium Chloride = 74.5 g/mol

Molarity of Potassium Chloride

Molarity = ConcentrationMolar Mass=14.9gdm−374.5gmol−1=0.2mol/dm3

Molarity and Number of Moles

Number of mole of solute in a solution can be calculated by using the following formula

n=MV / 1000

where  
n = number of mole of solute  
M = molarity of the solution  
V = volume of the solution in cm3

Example 4  
How many moles of zinc sulphate is present in 200cm3 of 0.1 mol dm-3 zinc sulphate solution?  
Answer:  
Molarity, M = 0.1 mol dm-3  
Voloume, V = 200cm3

n=MV / 1000

n= (0.1)(200) / 1000 = 0.02mol

Example 5  
A solution of barium hydroxide have molarity 0.1 mol dm-3. What is the concentration of the solution in g dm-3? [Relative Atomic Mass: Ba = 137; O = 16; H = 1]  
Answer:  
Relative Formula Mass of barium hydrokxide, Ba(OH)2  
= 137 + 2(16+1) = 171

Molar Mass of Potassium Chloride = 171 g/mol

Concentration =  Molarity × Molar Mass

=0.1mol/dm3 × 171gmol−1 = 17.1gmol−1

Solubility of substance

The term solubility refers to the maximum amount of material that will dissolve in a given amount of solvent at a given temperature to produce a stable solution.

Calculations on Solubility

Examples 1: If 12.2 g of lead(II) trioxonitrate(V) were dissolved in 21 g of distilled water at 20oC, calculate the solubility of the solute in mol dm-3.

Solution:

Molar mass of Pb(NO3)2 = 331 g

12.2 g of Pb(NO3)2 = 12.2/331 = 0.037 mole

21 g of water at 20oC dissolved 0.037 mole of Pb(NO3)2

1000 cm3 of water at 20 oC dissolved 1000 x 0.037 / 21

                                                = 1.76 moles of Pb(NO3)2

At 20oC, the solubility of Pb(NO3)2 in water is 1.76 mol dm-3

Using formula Method

Solubility = mass/molar mass x 1000/vol

            = 12.2/331 x 1000/21

            = 1.7751 mol dm-3

            = 1.76 mol dm-3

Example 2: The solubility of potassium trioxonitrate (V) is exactly 1800 g per 1000 g water at 83oC and 700 g per 1000 g water at 40oC. Calculate the mass of potassium trioxonitrate (V) that will crystallize out of solution if 155 g of the saturated solution at 83 oC is cooled to 40oC.

Solution:

                                                                       Solvents          +          Solution           = Solution

Masses involved at 83oC                     1000 g                         1800 g             = 2800 g

Masses involved at 40oC                     1000 g                         700 g               = 1700 g

Solute deposited on cooling from 83oC to 40oC = 2800 – 1700 = 1100 g

On cooling from 83oC to 40oC,

2800 g of saturated solution deposit 1100 g of solute

155 g of saturated solution will deposit = 1100 x 155 / 2800

                                                            = 60.89 g of solute

A titration calculation

The method for titration calculations is the one you have used for mole calculations already:

Write a balanced equation for the reaction.

Find the number of moles of the known substance.

Use the balanced equation to find the number of moles of the unknown substance.

Work out the mass, concentration or volume of the unknown.

The formula:

MAVA/MBVB = NA/NB

MA = Molarity of acid in mol/dm3

VA = Volume of the acid in cm3

MB = Molarity of base in mol/dm3

VB = Volume of the base in cm3

NA = Number of moles of acid

NB = Number of moles of base

Example 1: Calculate:

(a) the mass of anhydrous Na2CO3 present in 300cm3 of 0.1M

(b) the number of Na2CO3 particles present in the solution (Na = 23, C = 12, O = 16)

Solution:

(a) Molarity of the Na2CO3 solution = 0.1 M

Molar mass of Na2CO3 = 23 x 2 + 12 + 16 x 3 = 106g/mol

Concentration (g/dm3) = Molarity x molar mass

0.1  x 106

= 10.6g/dm3

This means 1000cm3 of 0.1M solution contain 10.6g of Na2CO3

300cm3 of 0.1 M solution will contain: 300 x 10.6 / 1000

                                                            = 3.18g of Na2CO3

(b) Number of Na2CO3 particles = molarity x 6.02 x 1023

                                                            0.1 x 6.02 x 1023

                                                            = 0.602 x 1023

This means 1000cm3 of 0.1 M solution contain 0.602 x 1023 Na2CO3 particles

300cm3 of 0.1M solution contain

Example 2: 20.30cm3 of hydrochloric acid solution was titrated against 25cm3 of 0.1M sodium hydroxide solution. Calculate:

(i)   the concentration of the acid in mol/dm3

(ii)  the concentration of the acid in g/dm3

Solution:

Equation of reaction

HCl (aq) + NaOH (aq) ——–> NaCl (aq) + H2O (l)

   1         :         1

(i) MA =?, MB = 0.1 M, VA = 20.30cm3, VB = 25cm3, NA = 1, NB = 1

MAVA/MBVB = NA/NB

MA = MBVB NA / VA NB

MA = 0.1 x 25 x 1 / 20.30 x 1

MA = 0.123 mol/dm3

(ii)  Molarmass of Acid = 36.5 g/mol, Concentration of Acid in mol/dm3 = 0.123 mol/dm3

        Concentration of Acid in  g/dm3 = Concentration of Acid in mol/dm3 x Molarmass

                                                                           =  0.123 x 36.5

                                                                           =  4.49 g/dm3

 Assessment

Calculate the concentration of the solution if 25g of NaOH is dissolve in 500cm3 of water.

32.30cm3 of hydrochloric acid solution was titrated against 25cm3 of 0.2M sodium hydroxide solution. Calculate: a. the concentration of the acid in mol/dm3  b. the concentration of the acid in g/dm3

If 21.2 g of Calcium Trioxocarbonate were dissolved in 15 g of distilled water at 30oC, calculate the solubility of the solute in mol dm-3.

The concentration of a Potassium chloride solution is 28.9 g dm-3. What is the molarity ( mol dm-3) of the solution? [Relative Atomic Mass: Cl = 35.5; K = 39]

WEEK 2

Heat of Neutralization & Redox Titrations

CONTENT

Determination of Heat of Neutralization

Meaning of Redox titrations

Redox titrations of Acidified KMnO4(aq) and Fe2+(aq);

Redox titrations of C2O42-(aq) and NaOH(aq)

Determination of Heat of Neutralization

The heat of neutralization can be determined in the laboratory using a thermally insulated container, with the cork having a thermometer and stirrer.

Method: Transfer 100cm3 of 0.5M hydrochloric acid solution into a beaker. Record the temperature of this solution. Transfer 100cm3 of 0.5M sodium hydroxide into another beaker. Record its temperature also. Allow the two solutions to attain the same temperature. Then transfer the 100 cm3 of sodium hydroxide solution into the hydrochloric acid quickly. Fit the cork immediately. Stir the solution very well. While stirring the solution, take the thermometer reading at time intervals until a constant temperature is obtained. Note the highest temperature obtained.

Results

Initial temperature of the acid and base = T1 oC

Final temperature of the mixed solutions = T2 oC

Change in temperature = T2 – T1 = ΔT

Mass of the mixture solution after neutralization, m = 200g

Calculations

The heat change during the reaction of 100 cm3 of 0.5M HCl = mC ΔT.

The number of moles in 100cm3 of HCl = 100×0.51000=0.005moles100×0.51000=0.005moles

Therefore the enthalpy change during neutralization is heat evolve divide by the number of moles of acid in 100 cm3.

= mCΔTn

Oxidation-Reduction Titration

Oxidation-Reduction Titration or Redox titration is a volumetric analysis involving an oxidizing agent and reducing agent. In this titration, an oxidizing agent (also known as the oxidant) is titrated against a reducing agent (otherwise known as the reductant).

The unknown concentration of the analyte is then determined using calculations. At the end of it, the oxidizing agent becomes reduced and the reducing agent becomes oxidized. Examples of redox titration are potassium tetraoxomanganate (VII) – iron(II) and  thiosulphate–iodine titrations.

Redox Titrations of Acidified KMnO4 Solution and a Fe2+ Salt

(1). When 0.011 moldm–3 potassium tetraoxomanganate (VII), KMnO4(aq) solution acidified with teraoxosulphate (VI) acid was titrated against a solution containing 14.0 g of an iron (II) salt in 500cm3 of solution, 21.30 cm3 of acidified KMnO4(aq) oxidized 25.00 cm3 of the iron (II) salt solution. The equation for the reaction is:

MnO4–(aq) + 5Fe2+(aq) + 8H+(aq) → Mn2+(aq) + 5Fe2+(aq) + 4H2O(l)

(a) From the information provided, calculate the:

(i) moles of MnO4– used in the titration.

(ii) moles of Fe2+ titrated

(iii) concentration of Fe2+ in mol dm–3

(iv) concentration of Fe2+ in gdm–3

(v) percentage of iron in the salt

(b) Give reason(s) why

(i) it is necessary to acidify KMnO4(aq) in the titration

(ii) hydrochloric acid cannot be used to acidify KMnO4 solution in the titration

Results and Calculations

Molar concentration of oxidant, Cox = 0.0110 moldm-3

Volume of oxidant, Vox used = 21.30 cm3

(a)(i). Number of moles of the oxidant = molar concentration x volume in dm3

= 0.0110moldm−3×21.30cm31000cm3=0.00234moles0.0110moldm−3×21.30cm31000cm3=0.00234moles

(ii). From the balanced equation for the reaction:

1 mole of MnO4–(aq) will oxidize 5 moles of Fe2+(aq)

Therefore, the number of moles of B titrated = 0.00234 х 5 = 0.0117 moles

(iii). The concentration of B in moldm-3;

25 cm3 of B contains 0.0117 moles

1000 cm3 will contain = 100025×=0.0117=0.468moldm−3100025×=0.0117=0.468moldm−3

Alternatively:

Cox×VoxCred×Vred=noxnred0.110×21.30Cred×25=15=Cox×VoxCred×Vred=noxnred0.110×21.30Cred×25=15=

Cred = 0.110×21.30×525×1=0.4686moldm−30.110×21.30×525×1=0.4686moldm−3

(iv). concentration of the Fe2+ in gdm–3 = concentration of Fe2+ in moldm–3 х molar mass

0.468 moldm–3 х 56 gmol–1 = 26.21 gdm–3

(v). percentage of iron in the salt is:

500 cm3 contains 14 g of the salt

∴ 1000 cm3 = 1000×145001000×14500

= 28 g of the salt

Therefore, percentage of iron in the salt

= 26.21×10028.0=93.61%26.21×10028.0=93.61%

b(i). It is necessary to acidify the oxidant so as to prevent unwanted reactions which may introduce errors to the titration.

(ii). Dilute HCl(aq) cannot be used because in the presences of the strong oxidizing agent, tetraoxomanganate (VII) ion, the chloride ions in hydrochloric acid are oxidized to chlorine gas.

(2). A is a solution containing 16.20 g dm-3 of impure ethanedioic acid.

B is 0.100 mol dm-3 sodium hydroxide solution.

On titrating A against B, using phenolphthalein as indicator, 31.50cm3 of A was found to react completely with 25.00 cm3 portions of B.

From information provided above, calculate the:

i. concentration of A in mol dm-3;  
ii. the percentage purity of the ethanedioic acid;

The equation for the reaction involved is:

H2C2O4(aq)+2NaOH(aq) → Na2C2O4(aq) + 2H2O(l)

[H = 1; C = 12; O = 16]

Solution

CA =?  
VA = 31.50 cm3  
nA = 2  
CB = 0.100 moldm-3  
VB = 25.00 cm3  
nB = 1

(i) CAVACBVB=nAnBCA=CBVBnAVAnBCA=0.1×25×231.50×1=0.159moldm−3CAVACBVB=nAnBCA=CBVBnAVAnBCA=0.1×25×231.50×1=0.159moldm−3

The concentration of H2C2O4(aqis 0.159 moldm-3

(ii). The percentage purity of the ethanedioic acid

 Concentration in gdm-3 = Concentration in moldm-3 x molar mass

= 0.159 moldm-3 x 90 gmol-1

= 14.31g

Percentage purity = purityimpure=1001=14.3116.20=100188.3%

EVALUATION

1. A solution of sodium trioxocarbonate (IV) contains 10.6g in 250cm3 of solution. Calculate the concentration of the solution. [Na2CO3 = 106.0]
2. 25.0 mol dm-3
3. 10.6 mol dm-3
4. 1.0 mol dm-3
5. 0.4 mol dm-3

2. Which of the following apparatuses can be used to measure accurately a specific volume of a liquid?

1. Pipette
2. Conical flask
3. Measuring cylinder
4. Beaker

3. Phenolphthalein indicator is more suitable for titration involving

1. Strong base and weak acid
2. Strong base and strong acid
3. Strong acid and weak base
4. Strong acid and strong base

**WEEK 3**

Test for Common Gases

CONTENT

Test for Oxygen gas, O2;

Test for Hydrogen gas, H2;

Test for Ammonia gas, NH3;

Test for Carbon (IV) oxide gas, CO2;

Test for Hydrogen chloride gas, HCl;

Test for Sulphur (IV) oxide gas, SO2.

Test/Identification of Gases

Gases are identified using their properties. Properties such as colour, odour, combustibility, action on litmus paper, and so on are the common properties used to identify gases. The table below summarizes the common gases and key properties used to identify them.

Common Gases and the Important Properties used in Identifying them

|  |  |  |  |
| --- | --- | --- | --- |
| Gas | Physical Properties | Test | Result |
| Hydrogen, H2(g) | Colourless, odourless gas; neutral to litmus paper | Ignite | Burns with ‘pop’ sound |
| Oxygen, O2(g) | Colourless, odourless gas; neutral to litmus paper | Insert a glowing splint | The splint rekindles |
| Carbon (IV) oxide, CO2(g) | Colourless, odourless gas; acidic | Bubble the gas through lime water | Limewater turns milky |
| Chlorine, Cl2(g) | Greenish-yellow;choking smell | (i) Insert moist blue litmus paper  (ii) Insert a moist starch-iodide paper | Litmus paper turns red then becomes bleached to white  Moist starch-iodide paper turns blue |
| Ammonia, NH3(g) | Colourless;Pungent smell; alkaline | (i) Insert moist red litmus paper.(ii) Contact with fumes of concentrated hydrochloric acid. | (i) Litmus paper turns blue. (ii) Gives white dense fume of NH4Cl with HCl. |
| Hydrogen Chloride, HCl(g) | Colourless, odourless;gives acidic solution in water | (i) Insert damp blue litmus. (ii) Apply a drop of silver nitrate on the end of a glass rod and hold it over gas | (i) Litmus paper turns red (ii) A white precipitate |
| Hydrogen sulphide, H2S(g) | Colorless gas;rotten egg smell | Test with damp lead (II) ethanoate (lead acetate) paper | Lead(II) ethanoate paper turns black |
| Sulphur (IV) oxide, SO2(g) | Colourless, odourless; acidic | Test with freshly made potassium dichromate (VI) paper | Paper turns from orange to green |
| Nitrogen (IV) oxide, NO2(g) | Reddish brown; pungent smell;Acidic | (i). Insert moist blue litmus paper (ii). Pass into silver trioxonitrate (V) solution | (i). Litmus paper turns red (ii). Cream precipitate is formed. |

WEEK 4

Qualitative Analysis I

CONTENT

Reagent for the test for cations

Identification of NH4+; Ca2+; and Pb2+ ions

Chemical Reagents used to Test for Cations in Solution

Identification of NH4+; Ca2+; and Pb2+ ions

Chemical Reagents used to Test for Cations in Solution

Dilute aqueous sodium hydroxide, NaOH(aq) and dilute aqueous ammonia solution, NH3 (aq) are used for the test for cations of inorganic salts in solution. Generally, the reagents react with the various cations to form hydroxides which may or may not dissolve in water. Base on the action of a cation with these reagents, the cation is identified. However, some of the actions of the cations with these mentioned reagents may not be completely reliable. If a test is very reliable for the identification of a substance, such a test is considered a confirmatory test for such a substance.

The result of qualitative analysis for ions is reported in a table under the headings test, observation, and inference. The action performed is stated under test, the observations made are recorded under observation and the conclusion or conclusions drawn are reported under inference.

Identification of NH4+; Ca2+; and Pb2+ ions

The test, observation and inference for the identification of NH4+; Ca2+ and Pb2+ ions are presented below.

Test for Cations in Solution using NaOH(aq)

|  |  |  |
| --- | --- | --- |
| Test | Observation | Inference |
| Sample solution  + NaOH(aq) + heat | A gas with characteristic pungent smell, which turns moist/wet red litmus paper blue, is given off. | Gas is NH3(g) from NH4+.  Gas is alkaline. |
| Sample solution + NaOH(aq)  –  in drops  – in excess | White precipitate is formed.  Precipitate is insoluble in excess NaOH(aq) | Ca2+ or Pb2+ present  Ca2+ present |
| Sample solution + NaOH(aq)  –  in drops  – in excess | White gelatinous is precipitate formed.   Precipitate is soluble in excess NaOH(aq) | Zn2+ or Al3+ present |
| Sample solution + NaOH(aq)  –  in drops  – in excess | Powdery/chalky white precipitate formed.      Precipitate is soluble in excess NaOH(aq) | Ca2+ or Pb2+ present  Pb2+ present |

Test for Cations in Solution using NH3(aq)

|  |  |  |
| --- | --- | --- |
| Test | Observation | Inference |
| Sample solution+NH3(aq)- in drops- in excess | No visible reaction | Ca2+ or NH4+ present |
| Sample solution+NH3(aq) – in drops  – in excess | White gelatinous precipitate is formed.   Precipitate is soluble in excess NH3(aq)  Precipitate is insoluble in excess NH3(aq) | Al3+ or Zn2+ present  Zn2+ present   Al3+ present |
| Sample solution + NH3(aq) – in drops  – in excess | White powdery or chalky precipitate is formed.   Precipitate is insoluble in excess NH3(aq). | Pb2+ present |

Confirmatory Test for NH4+; Ca2+ and Pb2+ ions

|  |  |  |
| --- | --- | --- |
| Test | Observation | Inference |
| Sample solution + NaOH(aq)      + warming | No visible reaction.     Colourless gas with pungent smell is evolved. The gas turns red litmus paper blue and forms white dense fume with HCl(aq) | The gas is NH3(g) from NH4+ |
| Sample solution + few drops of saturated Na2CO3(aq) or NaHCO3(aq)in drops then in excess | White precipitate is formed. Precipitate soluble in excess Na2CO3(aq) or NaHCO3(aq) | Ca2+ confirmed |
| Sample solution + dil. Or conc. HCl(aq) + heat and allow to cool  Sample solution + KI(aq) or K2Cr2O4(aq) | White precipitate is formed. Precipitate dissolved on heating and reappear on coolingYellow precipitate is formed | Pb2+ present     Pb2+ confirmed |
| Sample solution + (NH4)2S(aq)Sample solution + Potassium hexacyanoferrate (II) solution (K4(CN)6)(aq) | Dirty white precipitate is formedWhite precipitate  is formed | Zn2+ confirmed     Zn2+ confirmed |
| Sample solution + NH4OH(aq) followed by NH4Cl(aq) | White precipitate is formed | Al3+ confirmed |

WEEK 5

Qualitative Analysis II

CONTENT

Identification of cations: Cu2+; Fe2+ and Fe3+

Test for Cations in Solution using NaOH (aq)

|  |  |  |
| --- | --- | --- |
| Test | Observation | Inference |
| Sample solution + NaOH(aq) – in drops   – in excess   + Heat | Pale/light blue is precipitate formed.  Precipitate is insoluble in excess NaOH(aq)  Precipitate turned black. | Cu2+ present    CuO is formed |
| Sample solution + NaOH(aq)  in drops  – in excess | Dirty green gelatinous precipitate is formed.  Precipitate is insoluble in excess NaOH(aq) | Fe2+ present |
| Sample solution + NaOH(aq)– in drops  – in excess | Reddish brown gelatinous precipitate formed.  Precipitate is insoluble in excess NaOH(aq) | Fe3+ present |

Test for Cations in Solution using NH3(aq)

|  |  |  |
| --- | --- | --- |
| Test | Observation | Inference |
| Sample solution + NH3(aq) -in drops  -in excess | Pale/light blue precipitate is formed.   Precipitate soluble in excess NH3(aq) to form dark blue solution. | Cu2+ present |
| Sample solution + NH3(aq)- in drops  – in excess | Dirty green gelatinous precipitate is formed.  Precipitate is insoluble in excess NH3(aq). | Fe2+ present |
| Sample solution + NH3(aq) – in drops  – in excess | Reddish brown gelatinous precipitate is formed.   Precipitate is insoluble in excess NH3(aq) | Fe3+ present |

Confirmatory Test for Cu2+; Fe2+ and Fe3+

|  |  |  |
| --- | --- | --- |
| Test | Observation | Inference |
| Sample solution + NH3(aq) in excess   Sample solution + Potassium hexacyanoferrate (II) solution [K4Fe(CN)6)aq) | Deep blue solution is formed  Brown precipitate is formed | Cu2+ confirmed  Cu2+ confirmed |
| Sample solution + Potassium hexacyanoferrate (III) solution,  (K3(CN)6)(aq) | Light blue precipitate formed is | Fe2+ confirmed |
| Sample solution + Potassium hexacyanoferrate (III) (K3(CN)6)(aq) | Deep blue precipitate is formed | Fe3+ confirmed |
| Sample solution + Potassium thiocyanate solution (KSCN)(aq) | Blood red solution is formed | Fe3+ confirmed |

WEEK 6

Qualitative Analysis III

CONTENT

Reagents for test for anions

Identification of anions: SO42-; SO32-; CO32-; S2-; Cl–; and I–

Chemical Reagents to Test for Anions in Solution

Identification of Anions

Chemical Reagents to Test for Anions in Solution

One of the following pairs of chemical reagents which are most appropriate is used to test for the presence of an anion of inorganic salts.

i. Dilute barium chloride solution, BaCl2(aq), and dilute hydrochloric acid solution, HCl(aq)

ii. Dilute barium trioxonitrate (V) solution, Ba(NO3)2(aq), and dilute trioxonitrate (V) acid, HNO3(aq);

iii. Dilute aqueous silver trioxonitrate (V) solution, AgNO3(aq), and dilute trioxonitrate (V) acid, HNO3(aq)

iv. Freshly prepared aqueous Iron (II) tetraoxosulphate (IV) FeSO4(aq), with concentrated tetraoxosulphate (VI) acid, H2SO4, is used to test for trioxonitrate (V) nitrate ion, NO–3

v. And a few others.

Note that often the pair in (i) and (ii) give same result.

Identification of Anions

Test for SO42-, SO32-, CO32-and S2- using dilute BaCl2(aq) and HCl(aq) or Ba(NO3)2(aq) and HNO3(aq) as test reagents

|  |  |  |
| --- | --- | --- |
| Test | Observation | Inference |
| Sample solution + BaCl2(aq)   + HCl(aq) in excess | White precipitate formed.  Precipitate insoluble in excess HCl(aq) | SO42-, SO32-, CO32- or S2- present  SO42- present |
| Sample solution   + BaCl2(aq)    + HCl(aq) in excess | White precipitate formed.   A colourless gas with a chocking smell is given off. Gas turns potassium dichromate (IV) paper from orange to green. | SO42-, SO32-, CO32- or S2- present  SO32- is present.  Gas is SO2 gas |
| Sample solution  + BaCl2(aq)    + HCl(aq) in excess | White precipitate formed.   A colourless gas with a chocking smell is given off. Gas turns potassium dichromate (IV) paper from orange to green. | SO42-, SO32-, CO32- or S2- present  SO32- is present.Gas is SO2 gas |
| Sample solution   + BaCl2(aq)    + HCl(aq) in excess | White precipitate formed.    Effervescence occurs, a colourless, odourless gas which turns moist blue litmus red and lime water milky is given off. | SO42-, SO32-, CO32- or S2- present  Acidic gas is present.  Gas is CO2 from CO32- or HCO3– |
| Sample solution   + BaCl2(aq)     + HCl(aq) in excess | White precipitate formed.      A colourless gas with rotten egg smell is given off. Gas turns lead (II) ethanoate paper black. | SO42-, SO32-, CO32- or S2- present  S2- present.Gas is H2S |
| Sample solution   +HCl(aq)  +BaCl2(aq) | No visible reaction.  White precipitate formed. Precipitate is insoluble. | SO42- present |

Test for SO32-, CO32-, S2- , Cl– and I– using AgNO3(aq) and HNO3(aq)

|  |  |  |
| --- | --- | --- |
| Test | Observation | Inference |
| Sample solution +AgNO3(aq)  + dil. HNO3(aq)   in excess | White precipitate    Precipitate insoluble in excess HNO3(aq) | SO32-, CO32-, Cl– or S2- present   SO32-; S2- or Cl– present |
| Sample solution +AgNO3(aq)    + dil. HNO3(aq)   in excess | White precipitate    Effervescence occurs, a colourless, odourless gas which turns moist/damp/wet blue litmus red and lime water milky is given off. | SO32-, CO32-, Cl– or S2- present  Gas is CO2 from CO32- or HCO3–.  Gas is acidic. |
| Sample solution +AgNO3(aq)   + dil. HNO3(aq)   in excess | White precipitate   White precipitate formed. Precipitate soluble in excess NH3(aq) | SO32-, CO32-, Cl– or S2- present  Cl– present |
| Sample solution +AgNO3(aq)    + dil. HNO3(aq)   in excess | White precipitate     Yellow precipitate formed. Precipitate insoluble in concentrated NH3(aq) | SO32-, CO32-, Cl– or S2- present  I– present |

WEEK 7

Qualitative Analysis IV – Anions

CONTENT

Additional test for identification of SO42-;

SO32-; CO32-; NO3–; S2-; Cl–; and I–

Test for CO32-, SO32- and S2- using Dilute Acids

Test for CO32-, SO32- and S2- using Dilute Acids

Test for CO32-, SO32- and S2- using dilute acids

|  |  |  |
| --- | --- | --- |
| Test | Observation | Inference |
| Dry sample or residue or solution + dil. HCl(aq) or H2SO4(aq) | Sample dissolved. Effervescence occurs, a colourless, odourless gas, which turns moist blue litmus red and lime water milky is liberated. | Acidic gas present  Gas is CO2 from CO32- or HCO3– |
| Dry sample or residue or solution + dil. HCl(aq) or H2SO4(aq) | Sample dissolved. Effervescence occurs. Colourless gas with a sharp irritating smell is given off. The gas turns potassium dichromate (IV) paper from orange to green. | SO2(g) (from SO32-) is liberated |
| Dry sample or residue or solution + dil. HCl(aq) or H2SO4(aq) + KMnO4(aq) or K2Cr2O7(aq) | Purple colour of KMnO4(aq) turns colourless or orange colour of K2Cr2O7(aq) turns green | Reducing agent present |
| Dry sample or residue or solution + dil. HCl(aq) or H2SO4(aq) | Sample dissolved. Effervescence occurs. Colourless gas with characteristic rotten egg smell is given off. | H2S(g) from S2- present |

The Brown Ring Test (NO3–)

|  |  |  |
| --- | --- | --- |
| Test | Observation | Inference |
| Sample solution + freshly prepared FeSO4(aq) + Conc. H2SO4 solution in drops down the side of the test tube while holding the test tube in a slanting position. | Brown ring formed at the liquid junction | NO3– confirmed |

Dioxonitrate (III) ion, NO2– test

|  |  |  |
| --- | --- | --- |
| Test | Observation | Inference |
| Sample solution + freshly prepared FeSO4(aq) + CH3COOH solution in drops | Reaction mixture turns brown | NO2– present |

Additional Confirmatory Test for Some Anions

Hydrogen Trioxocarbonate (IV) ion, HCO3–

|  |  |  |
| --- | --- | --- |
| Test | Observation | Inference |
| Sample solution + BaCl2(aq) or MgSO4(aq) solution  + heating until boiling | No visible reaction   White precipitate formed | HCO3– is confirmed |
| Sample solution + few drops of phenolphthalein  + heat | No visible reaction. Solution remains colourless.  Solution turns pink. | HCO3– is confirmed |

Trioxosulphate (IV) ions, SO32-

|  |  |  |
| --- | --- | --- |
| Test | Observation | Inference |
| Sample solution + H2SO4(aq) + few drops of KMnO4(aq) or few drops of K2Cr2O7(aq) | Purple KMnO4(aq) is decolourisedOrK2Cr2O7(aq) changes from orange to green. | SO2(g) from SO32-  which reduces dichromate (VI) to chromate (III). |
| Sample solution + FeCl2(aq) in drops + warm | Yellow solution changes to reddish brown colour. | Oxidizing agent is present. Fe2+ has been oxidized to Fe3+ |

Iodide ion, I–

|  |  |  |
| --- | --- | --- |
| Test | Observation | Inference |
| Sample + dil. HNO3(aq) + AgNO3(aq) in excess | Yellow precipitate formed. Precipitate insoluble in concentrated NH3(aq) | I– confirmed. |

Sulphide ion, S2-

|  |  |  |
| --- | --- | --- |
| Test | Observation | Inference |
| Sample solution + Pb(CH3COO)2(aq) or Pb(NO3)2(aq) + warming | Black precipitate of lead (II) sulphide formed. | S2- confirmed. |

WEEK 8

Test for Fat and Oil; Protein; Starch | Week 8

CONTENT

Test for sugars

Test for fat and oil

Test for protein

Test for starch

Test for Sugar

Reducing Sugar: E.g. Glucose, Fructose, Lactose and Maltose

|  |  |  |
| --- | --- | --- |
| Test | Observation | Inference |
| Few drops of the sample + Fehling’s solution + warm | Solution gives yellow precipitate, which turns brick red on warming. | Reducing sugar is present |
| Few drops of the sample + Benedict’s solution + warm | Solution gives orange red or yellow precipitate. | Reducing sugar is present |

Non-non-reducing sugars do not react with Fehling’s nor Benedict’s solution. Therefore, they do not give any precipitate with these reagents.

Tests for Fat and Oil

|  |  |  |
| --- | --- | --- |
| Test | Observation | Inference |
| 2cm3 of sample + water + (3 – 5 drops of) Sudan (III) stain | Red stain is formed | Sample is melted fat or oil |
| Few drops of sample on a piece of white paper or filter paper | Translucent spot (grease spot) is observed on the paper | Sample is melted fat or oil |

Tests for Protein

The Biuret Test

|  |  |  |
| --- | --- | --- |
| Test | Observation | Inference |
| Sample + NaOH(aq) solution + CuSO4(aq) solution in drop, followed by thorough shaking + warm | Violet or purple precipitate formed | Sample is protein |

The Millions test

|  |  |  |
| --- | --- | --- |
| Test | Observation | Inference |
| Sample solution + millions reagent+ heat | White precipitate is formed. Precipitate turns deep red or brick red on heating. | Protein is present |

The Xanthoproteic Test

|  |  |  |
| --- | --- | --- |
| Test | Observation | Inference |
| Sample + conc. HNO3(aq) in drops + heat + few drops of NaOH(aq) or NH4OH(aq) | Yellow precipitate, which turns orange on addition of NaOH(aq) or NH4OH(aq) | Sample is protein |

Tests for Starch

|  |  |  |  |
| --- | --- | --- | --- |
|  | Test | Observation | Inference |
| I. | 2 cm3 of the sample + Iodine solution | Solution turns blue-black | Sample is starch |
| II. | Solution from (a) above + gentle heating followed by cooling | The blue-black colouration disappears on heating and reappears on cooling | Sample is starch |

WEEK 9

Petroleum I

CONTENT

Origin and composition of petroleum (crude oil)

Nigerian and world crude oil reserve

Exploration and drilling of crude oil

Origin and Composition of Petroleum

The word petroleum means rock oil. Petroleum consists of Natural gas and Crude oil. Natural gas contains methane gas and other hydrocarbon gas mixtures. Natural gas may be liquefied under pressure to produce Liquefied Natural gas, LNG. LNG is a colourless, odourless, non-toxic gas which is predominantly used as domestic gas for cooking and heating. It may also be used as transportation fuel in special engines. The odour of the domestic gas is a result of the addition of tert-buthynol (also known as mercaptan). The odour of mercaptan is necessary to make it possible to detect gas leaks.

Crude oil is a dark brown viscous liquid which shows green fluorescence. It has an unpleasant odour. The major impurity in crude oil is sulphur. Sulphur as an impurity in petroleum causes environmental pollution and so it must be removed. Sulphur free crude oil is in high demand. Nigerian crude oil is relatively sulphur free.

Exploration and Drilling of Crude Oil

Rocks that are likely to have crude oil are identified through geophysical survey, remote sensing which makes use of aerial photographs to locate and map surface features, core drilling, and mapping the earth’s rock layers.

Mapping is done by exploding dynamite in deep holes in the earth’s crust and recording the reflections of the shock waves from the rock layers. From this data, a geologist will detect possible oil bearing rocks.

Crude oil is extracted by drilling wells into the ground. The drilling is carried out using a rotary rig supported by a derrick. When the well is drilled to the layers of the rocks that bear oil, the gas may come out under high pressure. The crude is then pumped out. The crude is then stored in hard steel tanks and transported for refining.

WEEK 10

Petroleum I

CONTENT

Location of Nigerian refineries

Fractional distillation and products

Cracking and reforming of petroleum

Location of Refineries in Nigeria

Crude oil is separated into different fractions by the separation process called Fractional Distillation in a fractionating column in petroleum refineries. In Nigeria, refineries are found in Port Harcourt, Warri and Kaduna. Dangote group is building a private refinery in Lagos, Nigeria.

Fractional Distillation and Products

During the refining of petroleum by fractional distillation, components of the petroleum separate into components called fractions according to their molar masses and boiling point. Each fraction contains several compounds which fall within a certain boiling point range. Heavier fractions separate to the bottom, and light fractions separate to the top. The lightest fractions, which are mainly gasses, vapourize and rise to the top of the distillation tower, and are collected, and so on.

After the fractional distillation separation, the products are further refined as necessary through various processes like cracking, reforming, or alkylation.

The table below summarized the various fractions of crude oil and its uses.

|  |  |  |  |
| --- | --- | --- | --- |
| Fraction | Number of Carbon | Boiling Point | Uses of fraction |
| Petroleum gases(methane, ethane, propane and butane) | C1 – C4 | <40oC | They are mainly used as fuel for homes and industries and for the production of petrochemicals. Methane, CH4, is used to produce H2, tetrachloromethane. |
| Gasoline (petrol) | C5 – C10 | – 180oC | Petrol is used as fuel in cars and generators. It is also used as a solvent for paint and grease. |
| Paraffin oil(Kerosene) | C11 – C15 | 180 – 250oC | It is used as fuel for lighting and heating. It is also used as aviation fuel. |
| Diesel(Gas oil and diesel) | C16 – C20 | 250 -300oC | It is used as fuel for heating and in diesel engines and as a raw material for cracking of petroleum. |
| Lubricating oil | C20 – C30 | – 350oC | Vaseline is used in hair care products. Paraffin is used in making candles, creams, etc. |
| Bitumen(Asphalt) | > C30 | >350oC | Bitumen is used in road surfacing, coating pipes, tar for roofing, etc. |

Cracking and Reforming of Petroleum

Cracking

Cracking is the breaking up of longer hydrocarbon molecules into smaller ones at high temperature and pressure and in the presence of the catalysts.

Types of Cracking

Thermal cracking: Thermal cracking involves heating heavy oil or large hydrocarbons e.g. diesel oil, kerosene at high pressure to give low molecular mass hydrocarbon.

C11H24Kerosense→thermalcrackingC8H18Petrol+C3H6Propane

Catalytic cracking: Catalytic cracking involves the use of catalyst such as SiO2-Al2O3, natural clay, etc. to break down large hydrocarbons into smaller molecular mass hydrocarbons.

C18H36→C6H14Hexane+C8H18Petrol+2C2H4Ethene

Cracking increases the yield of petrol.

Reforming

Reforming is the conversion of straight-chain hydrocarbons using catalyst into branch chain isomer, cycloalkanes, and benzenes. The purpose of reforming is to obtain a higher grade of petrol from a lower grade of petrol i.e. improve the quality of petrol.

Reforming Techniques

Reforming techniques involve: isomerization, cyclization and aromatization

Differences between Cracking and Reforming

|  |  |
| --- | --- |
| Cracking | Reforming |
| – Improves the quantity of petrol  – Breaks large factions of hydrocarbons into smaller ones  – Involves high temperature, high pressure and the use of catalyst | – Improves the quality of petrol  – Rearranges the fractions  – Does not involve high temperature and pressure. It only uses catalyst |

Importance of Cracking

It increases the yield of petrol

It produces petrochemicals such as ethene, propene etc. which are the major raw materials in chemical industry.

Importance of Reforming

To produce high quality petrol