

Quantitative Inorganic Analysis

University of Hargeusa

College of Education
Senior Students
Chemistry

Author

Ismail Abdi Ali

Contents

Chapter 1: introduction to Inorganic Quantitative Analysis	1
Analytical chemistry	1
Qualitative Analysis	1
Quantitative Analysis	1
Wet chemistry	1
Dry chemistry test	1
Analysis	2
Determination	2
Characterizing a Material	2
Standardization	2
Primary standard	2
A standard solution	3
Making a standard solution	3
How to make molar solution	5
Making Molar Solutions from Concentrated Aqueous Acids	7
Making Molar aqueous solution of H ₂ SO ₄ (1M)	8
Calculating the Molarity of 96% by Mass of Sulphuric acid	8
Preparing stock solutions of sulphuric acid by easy method	9
Typical Concentrations of Concentrated Acids and Bases	10
Chapter Two: Stoichiometry	11
Law of Conservation of Mass	11
Types of Reactions	11
1) Combination (synthesis)	12
2) Decomposition reaction	12
3) Single Displacement Reaction	12
4) Double Displacement	12
5) Acid-Base	13
6) Combustion	13

Practical activities of chemical reaction.....	13
Preparing the stock solution	22
Making diluted solutions from a more concentrated stock solution	22
Chapter 3: Titrimetric Analysis.....	24
Classification of Reactions in Titrimetric Analysis	25
1. Neutralization reactions, or acidimetry and alkalimetry	25
2. Complex formation reactions	25
3. Precipitation reactions:.....	26
4. Oxidation-reduction reactions:.....	26
Chapter 4: Complex ions and coordination compounds	28
Coordination compounds	28
The coordination sphere.....	28
ligands	28
Donor atoms	28
coordinate covalent bond.....	28
Formation of Complex ions.....	28
Mono dentate Ligands	29
Polydentate ligand	30
Chelate	30
Coordination number.....	33
Naming Coordination Compounds	33
Chapter 5 : Gravimetric analysis	40
The steps of gravimetric analysis.....	40
What is precipitation gravimetry?	40
Volatilization gravimetry.....	41
Summary	43

Chapter 1: introduction to Inorganic Quantitative Analysis

Analytical chemistry

Analytical chemistry is often described as the area of chemistry responsible for characterizing the composition of matter, both qualitatively (what is present) and quantitatively (how much is present) Chemical Analysis is subdivided into Qualitative Analysis and Quantitative Analysis.

Qualitative Analysis

Qualitative Analysis treats of the methods for determining the nature of the constituents of a substance, it is an analysis carried out to determine only the identity of a pure analyte, the identity of an analyte in a matrix, or the identity of several or all components of a mixture.

Quantitative Analysis

Quantitative Analysis is any method used for determining the amount of a chemical in a sample. The amount is always expressed as a number with appropriate units. An acid-base titration is an example of quantitative analysis.

In order to recognize a substance, we change it, usually with the help of another substance of known nature, into a new compound which possesses distinctive properties. This transformation we call a *chemical reaction*; and the substance by means of which the reaction is brought about, the *reagent*. A reagent is **a compound or mixture added to a system to start or test a chemical reaction**. A reagent can be used to determine the presence or absence of a specific chemical substance.

We distinguish between reactions in the *wet way* and reactions in the *dry way*.

Wet chemistry

Wet chemistry, also called wet chemical analysis, is a form of analytical chemistry that uses classical methods, such as **Gravimetry and Titration** to analyze elements and compounds in liquid samples. **Wet chemistry** is a form of **analytical chemistry** that uses classical methods such as observation to analyze materials. It is called wet chemistry because most of the analyzing is done in the liquid phase. Wet chemistry is also called bench chemistry since many tests are performed at lab benches.

Dry chemistry test

Dry chemistry analysis is the term used in contrast to **wet chemistry techniques**. A **dry chemistry analyzer** is a process in which a liquid test sample is added directly to a dry reagent strip manufactured specifically for each project.

The dry chemistry analyzer uses the moisture of the sample being tested as a solvent to cause a specific chemical reaction that leads to a chemical analysis.

This type of analysis is a class of analytical methods based on enzymatic methods. Dry chemistry is also known as dry reagent chemistry or solid phase chemistry.

Analysis

The analysis is a process that provides chemical or physical information about the constituents in the sample or the sample itself. The components of a sample that are to be identified or determined are often referred to as **analytes** and the word for the material in which the analyte is found is called the **matrix** of the analyte.

Determination

Determination is an analysis of a sample to find the identity, concentration, or properties of the analyte.

Characterizing a Material

A combination of the qualitative and quantitative analysis of a material or matrix is sometimes called **characterizing** the material. A total analysis such as this might involve a complete reporting of the properties of a material as well as the identity and quantity of component substances.

Standardization

Standardization is an analytical technique we use to find an unknown concentration using **a primary or a secondary standard solution**. The most commonly used technique for standardization of a solution is titration. For a standardization process, a standard solution is required as a reference. Standard solutions come in two types as primary standard solutions and secondary standard solutions. For accurate standardizations, we use primary standard solutions.

Primary standard

A primary standard is a compound which is stable, of high purity, highly soluble in water and of a high molar mass to allow for accurate weighing.

Some examples of primary standards for titration of solutions, based on their high purity, are provided

- Potassium bromate (KBrO_3) for standardization of sodium thiosulfate solutions
- Potassium hydrogen phthalate (usually called KHP) for standardization of aqueous base
- Sodium carbonate for standardization of aqueous acids: hydrochloric, sulfuric acid and nitric acid solutions

A standard solution

A standard solution is a solution of accurately known concentration prepared from a primary standard that is weighed accurately and made up to a fixed volume. To prepare a standard solution, a known mass of solute is dissolved and the solution is diluted to a precise volume. It is prepared using a standard substance, such as a primary standard. Standard solutions are used to determine the concentrations of other substances, such as solutions in titration

Standard solution concentration is usually expressed in terms of molarity (M) or moles per liter (mol/L). Not all substances are suitable solutes for standard solutions. The reagent must be stable, pure, and preferably of high molecular weight.

Making a standard solution

Aim

The purpose of this experiment is to prepare a standard solution of potassium hydrogen phthalate.

Introduction

Potassium hydrogen phthalate is a primary standard because it meets certain requirements.

- It must be available in a highly pure state.
- It must be stable in air.
- It must be easily soluble in water.
- It should have a high molar mass.

You will need to weigh accurately a sample of potassium hydrogen phthalate and use it to make a solution of concentration close to 0.10 mol dm^3 .

Requirements

- safety spectacles
- weighing boat or filter paper
- spatula
- potassium hydrogen phthalate (Irritant)
- balance capable of weighing to within 0.01 g

- beaker, 250 cm³
- wash bottle of distilled water
- stirring rod
- volumetric flask, 250 cm³
- filter funnel
- dropping pipette

Procedure

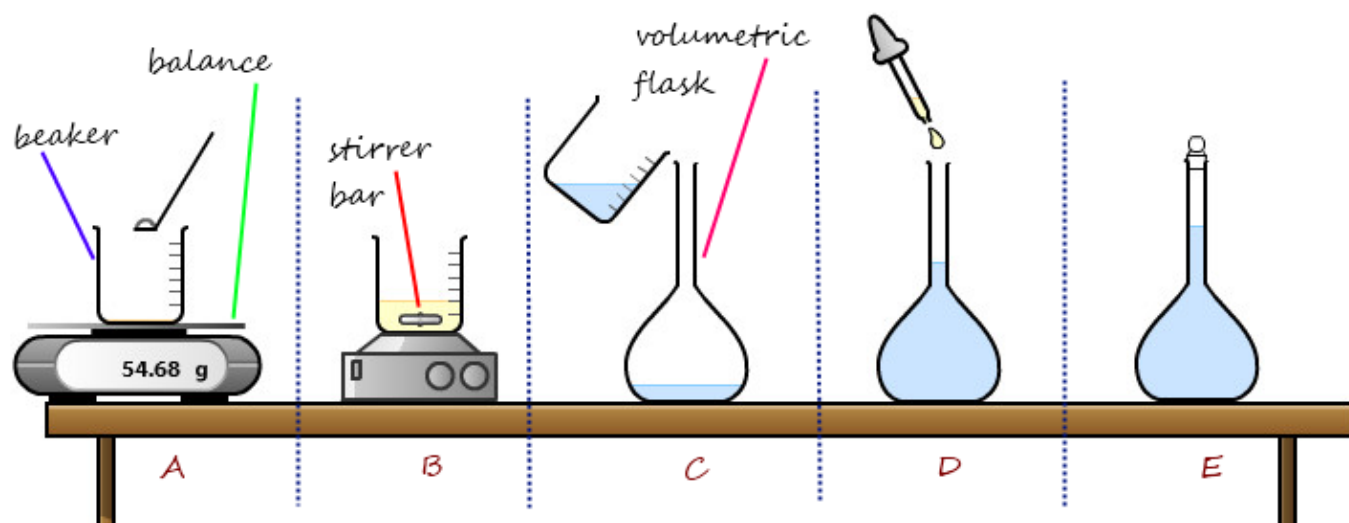
1. Transfer 5.1 g of potassium hydrogen phthalate into a weighing boat and weigh it to the nearest 0.01 g.

$$n = MV \quad n = 0.1 \text{ mol/L} \times 0.25 \text{ L} = 0.025 \text{ mol. Mass} = 0.025 \times 204 = 5.1 \text{ g}$$

2. Put about 100 cm³ of water into a 250 cm³ beaker. Carefully transfer the bulk of the potassium hydrogen phthalate from the weighing boat into the beaker.
3. Reweigh the boat with any remaining potassium hydrogen phthalate to the nearest 0.01g.
4. Stir to dissolve the solid, adding more water if necessary.
5. Transfer the solution to the 250 cm³ volumetric flask through the filter funnel. Rinse the beaker well, making sure all liquid goes into the volumetric flask.
6. Add distilled water until the level is within about 1 cm of the mark on the neck of the flask. Insert the stopper and shake to mix the contents.
7. Using the dropping pipette, add enough water to bring the bottom of the meniscus to the mark. Insert the stopper and shake thoroughly ten times to ensure complete mixing. Simply inverting the flask once or twice does not mix the contents properly and is a very common fault.
8. Label the flask with the contents, its molarity and the date. This can now be used to calculate the unknown concentration of a base.

Calculating Molarity: Formula: $\text{C}_8\text{H}_5\text{KO}_4$ $5.1 \text{ g} / 204 \text{ g} \cdot \text{mol}^{-1} = 0.025 \text{ mol}$ Vol. = 0.25L

Molarity = n/v = 0.025 mol/0.25L = 0.1M



Molar mass of potassium hydrogen phthalate,	g mol^{-1}
Mass of bottle and contents before transfer, (m_1)	g
Mass of bottle and contents after transfer, (m_2)	g
Mass of potassium hydrogen phthalate, $m = (m_2 - m_1)$	g
Moles of potassium hydrogen phthalate, $n = m/M$	mol
Volume of solution, V	dm^3
Concentration of potassium hydrogen phthalate, $c = n/V$	mol dm^{-3}

Questions: What effect would each of the errors described below have on the concentration of potassium hydrogen phthalate?

- Some of the solid potassium hydrogen phthalate was spilled in making the transfer.
- Not enough water was added to bring the volume to the mark.

How to make molar solution

Molar solution is one mole of substance per one litre of solution

1mol of solute + water = one litre of molar solution

Concentration = number of moles / volume of solution in dm^3

Aim : to make molar solution of sodium hydroxide

Requirements

- safety spectacles
- watch glass
- spatula
- sodium hydroxide
- balance capable of weighing to within 0.01 g
- beaker, 250 cm³
- wash bottle of distilled water
- stirring rod
- volumetric flask, 250 cm³
- filter funnel
- dropping pipette

You can make a concentration of a molar solution by using any volume. Different volumes contain different masses of solute. This is how to find amount of mass in 250 ml of 1M of NaOH.

$n = MV$ $n =$ number of moles $M =$ molarity $V =$ volume of solution

$$n = M \times V \quad n = 1\text{mol/L} \times 0.25\text{L} = 0.25 \text{ mol}$$

$$\text{Mass} = 0.25 \times 40\text{g} = 10\text{g}$$

Procedure

1. Transfer 10 g of sodium hydroxide into a watch glass and weigh it to the nearest 0.01g.
2. Put about 100 cm³ of water into a 250 cm³ beaker. Carefully transfer the mass of the sodium hydroxide from the watch glass boat into the beaker.
3. Reweigh the boat with any remaining sodium hydroxide to the nearest 0.01g.
4. Stir to dissolve the solid, adding more water if necessary.

5. Transfer the solution to the 250 cm³ volumetric flask through the filter funnel. Rinse the beaker well, making sure all liquid goes into the volumetric flask.
6. Add distilled water until the level is within about 1 cm of the mark on the neck of the flask. Insert the stopper and shake to mix the contents.
7. Using the dropping pipette, add enough water to bring the bottom of the meniscus to the mark. Insert the stopper and shake thoroughly ten times to ensure complete mixing. Simply inverting the flask once or twice does not mix the contents properly and is a very common fault.
8. Label the flask with the contents, its molarity and the date. This can now be standardized by using potassium hydrogen phthalate.

Note: to avoid spoiling sodium hydroxide since you need only 25 ml or 50 ml etc use this table or calculate it by using above equation.

Sodium hydroxide /g	Add water until mark of	Concentration
40 g	1000 ml	1 M (= mol/ dm ³)
20 g	500 ml	1 M (= mol/ dm ³)
10 g	250 ml	1 M (= mol/ dm ³)
4 g	100 ml	1 M (= mol/ dm ³)
2 g	50 ml	1 M (= mol/ dm ³)
1 g	25 ml	1 M (= mol/ dm ³)

Making Molar Solutions from Concentrated Aqueous Acids

Making a standard molar solution from aqueous acids is a bit more involved than making a standard molar solution from a solid chemical. This is because nearly all liquid acids, no matter how

concentrated they are, are already diluted to some extent with water (for instance, pure HCl is a gas, not a liquid, and it is rarely solid in its pure form). Let's use sulfuric acid as our first example.

Making Molar aqueous solution of H₂SO₄ (1M)

The first step is to read the label on the bottle of the H₂SO₄ reagent. The label will tell you its molarity. Although there are a variety of concentrations of acids, concentrated H₂SO₄ often comes from the factory at a 18.0 Molar concentration (Table 2). This means that there are 18 moles of moles of H₂SO₄ in each liter of solution (note: do *not* rely on Table 1; always check the label on the bottle). You need to make a much more diluted solution, so you will add one mole of the concentrated reagent to a fresh batch of water. Your task is to calculate how many milliliters of reagent contain one mole of the acid.

We know from reading the label on the bottle ("18.0 Molar") that one liter of reagent contains 18.0 moles of H₂SO₄. This means that 1 ml of reagent contains 0.018 moles of

H₂SO₄. Therefore,

$$\frac{1 \text{ ml}}{0.018 \text{ moles}} = \frac{x \text{ ml}}{1 \text{ mole}}$$

Solving for x, we find that we need 55.6 ml of H₂SO₄ reagent.

Therefore, we *slowly* add 55.6 ml of the H₂SO₄ reagent to about 500 ml of distilled (deionized) water, and then we top it off with more water to exactly the "1-liter" mark on the flask.

You have successfully made a 1 Molar H₂SO₄ solution. This procedure works similarly with aqueous bases.

Caution: *Never* add water into a large volume of concentrated acid! You risk creating an explosion! The rule is: "Acid into water"

Always add a smaller volume of acid into a larger volume of water.

Calculating the Molarity of 96% by Mass of Sulphuric acid

The container of the sulphuric acid is usually labeled with its specific gravity (1.84 g/ml) and its mass % concentration (96% by mass)

Concentrated laboratory acid is 96 % H₂SO₄ by mass and has a density of 1.84 g ml⁻¹. What is its molarity?

Solution

1. Find the mass of 1L of solution from the density
2. Find the mass of H₂SO₄ in 1L of the solution using the percent by mass
3. Convert the mass of H₂SO₄ to moles of H₂SO₄

- The mass of 1L (1000 ml) of the solution is:

$$1000 \text{ ml} \times 1.184 \text{ g/ml} = 1184 \text{ g}$$

- Mass of H₂SO₄ in 1L of the solution

$$1184 \text{ g} \times 96\% = 1136.64 \text{ g}$$

- Converting the mass of H₂SO₄ to moles of H₂SO₄

$$\text{Moles of H}_2\text{SO}_4 = \frac{1136.64 \text{ g}}{98 \text{ g/mol}} = 11.6 \text{ mol}$$

$$\text{Molarity} = \frac{\text{amount of solute (mol)}}{\text{Volume of solution (L)}}$$

$$\text{Molarity} = \frac{11.6 \text{ mol}}{1 \text{ L}} = 11.6 \text{ mol/L}$$

$$\text{Molarity of sulphuric acid} = 11.6 \text{ M}$$

Preparing stock solutions of sulphuric acid by easy method

Aim: calculating number of milliliters of 96% m/m of sulphuric acid that contains:

- 1) 1 mole or 98g of sulphuric acid
- 2) 0.5 mol or 49g of sulphuric acid

3) 0.4 mol or 39.2g of sulphuric acid

4) 0.2mol or 19.6g sulphuric acid

Given

1) Molar mass of sulphuric acid = 98g/mol

2) Specific gravity = 1.84g/ml

3) % assay = 96% by mass

$$D = \frac{\text{Mass}}{\text{Vol}} \quad V = \frac{M}{D} = \frac{98\text{g}}{1.84 \text{ g/ml}} = 53.26 \text{ ml}$$

number of milliliters of 96% m/m of sulphuric acid that contains: 1 mole
or 98g of sulphuric acid :

$$\frac{53.26 \times 100}{96} = 55.5\text{ml}$$

Therefore, we *slowly* add 55.5 ml of the H₂SO₄ reagent to distilled (deionized) water, and then, top it off with more water to exactly the "1-liter" mark on the flask.

2) Calculating number of milliliters of 96% m/m of sulphuric acid that contains 0.5 mol or 49g of sulphuric acid:

$$0.5 \text{ mol H}_2\text{SO}_4 = 0.5 \times 55.5\text{ml} = 27.75 \text{ ml}$$

0.5 mol H₂SO is in 27.75 ml

Typical Concentrations of Concentrated Acids and Bases
(as written on the labels of their containers)

Acid/base	WT %	specific gravity	molarity	
Hydrochloric acid	35	1.18g/ml	11.3	
Sulphuric acid	96	1.84 g/ml	18	

Nitric acid	70	1.4 g/ml	15.6	
Phosphoric acid	85	1.69 g/ml	14.7	
Acetic acid	99.7	1.05 g/ml	17.4	
Aqueous ammonia	28	0.89g/ml	14.7	

Chapter Two: Stoichiometry

Stoichiometry (/stɔɪki'ɒmɪtri/) is a section of chemistry that involves using relationships between reactants and products in a chemical reaction to determine desired quantitative data. In Greek, *stoikhein* means element and *metron* means measure, In order to use stoichiometry to run calculations about chemical reactions, it is important to first understand the relationships that exist between products and reactants and why they exist, for example understanding how to balance reactions and determining the molar mass

Law of Conservation of Mass

The Law of Conservation of Mass states that matter is neither created nor destroyed in a chemical reaction, every chemical reaction has the same elements in its reactants and products, though the elements they are paired up with often change in a reaction. In this reaction, magnesium (mg), and oxygen, are the elements present in both reactants, so based on the law of conservation of mass; they are also present on the product side of the equations.

Types of Reactions

There are 6 basic types of reactions.

In every chemical reaction, it's the atoms and molecules of the reactants that are getting rearranged to give us new products. And this rearrangement could happen in many ways, like for

example, here, the atoms and molecules are combining to give one single product. Or you can have a big molecule that breaks down into smaller molecules. Or you can have an exchange of atoms between the reactants to give us new products. So depending on the ways of rearrangement, we can classify chemical reactions into various types

1) Combination (synthesis)

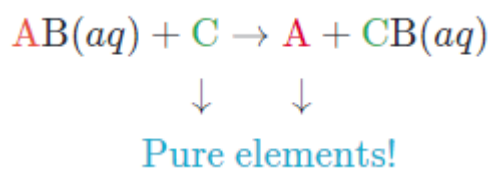
Reactions in which two or more reactants combine to give one product are called combination reactions for example the formation of HCl from the reaction of a chemical and H₂ and Cl₂

2) Decomposition reaction

In decomposition reaction, one reactant breaks down into two or more products for example copper (ii) carbonate decomposes when heated into copper oxide and carbon dioxide.

3) Single Displacement Reaction

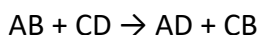
A **single replacement reaction**, sometimes called a **single displacement reaction**, is a reaction in which one element is substituted for another element in a compound. The starting materials are always pure elements, such as a pure zinc metal or hydrogen gas, plus an aqueous compound. When a replacement reaction occurs, a new aqueous compound and a different pure element will be generated as products. The general pattern of a single replacement reaction is shown below



4) Double Displacement

A double displacement reaction is **a type of reaction in which two reactants exchange ions to form two new compounds**. Double displacement reactions typically result in the formation of a product that is a precipitate

Double displacement reactions take the form:



- A double displacement reaction is a type of chemical reaction in which the reactant ions exchange places to form new products.
- Usually, a double displacement reaction results in precipitate formation.
- The chemical bonds between the reactants may be either covalent or ionic.

- A double displacement reaction is also called a double replacement reaction, salt metathesis reaction, or double decomposition.

5) Acid-Base

Acid- base reaction is a chemical reaction in which acid and a base react quantitatively with each other. Neutral substances are formed such as water and salts.

6) Combustion

Combustion is the formation of CO_2 and H_2O from the reaction of a chemical and oxygen O_2

Practical activities of chemical reaction

Experiment: single displacement reaction

Reaction of acids with metals

Objective

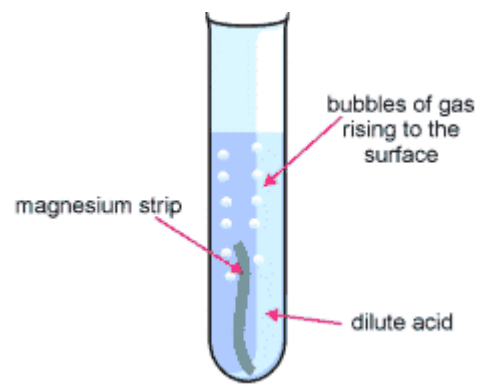
- 1) To show single displacement reaction, magnesium reacts with sulphuric acid
- 2) Making magnesium sulphate from dilute sulphuric acid and magnesium ribbon

Apparatus

1. a beaker/test tube
2. Measuring cylinder – 25 ml
3. Filter paper
4. Filter funnel
5. Tripod
6. Gauze or pipe clay triangle
7. Bunsen burner
8. Magnesium ribbon
9. Dilute sulphuric acid
10. Evaporating dish

Procedure

1. Put 15 cm^3 of 0.5 M sulfuric acid into the 100 cm^3 beaker or test tube.
2. Add magnesium metal to the acid and carefully observe the reaction.
3. Remove the excess magnesium metal by filtering.
4. **Evaporate some of the water by** heating carefully on the tripod.



1. What did you observe in this reaction?
2. How do you know when the reaction is over?

Experiment: Acid-Base

Acid+ Insoluble base

Objective

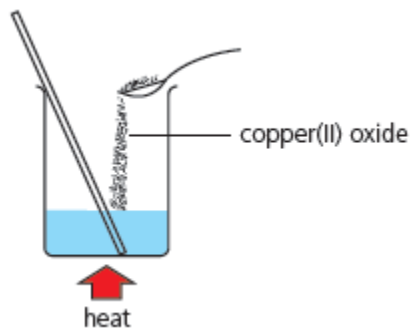
- 1) To show acid –base reaction, copper (II) oxide reacts with sulphuric acid
- 2) Making copper (II) sulphate from copper (II) oxide and sulphuric acid

Apparatus and chemicals

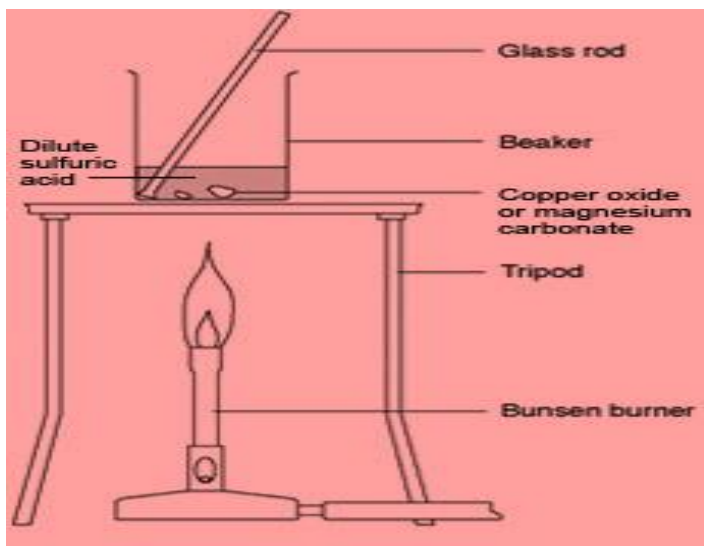
1. beaker 250 ml (borosilicate glass)
2. stirring rod
3. spatula
4. evaporating dish
5. funnel
6. filter paper
7. black copper (II) sulphate powder
8. sulphuric acid
9. bunsen burner/spirit lamp

Procedure

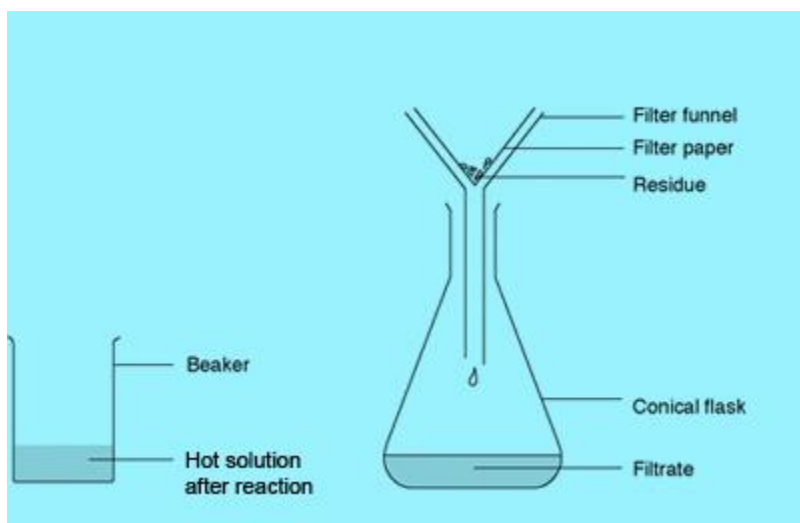
1. A spatula of copper (II) oxide is added to dilute sulphuric acid .



2. gently warm the solution .



3. more copper oxide is added until no more dissolve and settle at the bottom.
4. the excess solid is filtered from the solution



5. the solution is heated to evaporate some of the water. Blue copper(II) sulphate crystals start to form.

Experiment: Double Displacement

Precipitation of lead iodide

Objective

Precipitation of lead iodide from aqueous solutions of potassium iodide and lead nitrate

Hazards

Both lead nitrate and potassium iodide are very poisonous. Lead nitrate is also a strong oxidizing agent. Handle them with care.

Apparatus

1. Two small beakers
2. Test tube
3. Stirring rod
4. Spatula
5. Distilled water
6. Potassium iodide
7. Lead nitrate

Procedure

Make a solution of lead nitrate

1. Measure 30 ml of distilled water in a small beaker. Add a spatula of lead nitrate and stir the solution until all crystals dissolve

Make a solution of potassium iodide.

2. In the same way measure 20 ml of distilled water in another small beaker. Add a spatula of potassium iodide and stir the solution.
3. Mix the solutions together in a test tube.
4. Make a filter using the conical flask, funnel and filter paper.
5. Filter the mixture.



precipitation of lead iodide

Observation

This is a very **quick demonstration** that shows the reaction of two **aqueous solutions** can **react** together. White **lead nitrate** and white potassium iodide react to make yellow lead iodide precipitate.

Conclusion

When you simply mix two chemicals together, their particles will combine, forming two brand new compounds. For example, when you add lead nitrate to potassium iodide, you will witness a double-replacement reaction in which the lead combines with the iodide and becomes lead iodide, while the potassium combines with nitrate and becomes potassium nitrate

: Experiment: Decomposition

Thermal Decomposition of carbonates

Objective

To investigate the decomposition of copper (II) carbonate

[Bubbling CO_2 through limewater ($\text{Ca}(\text{OH})_2(\text{aq})$) is a test for carbon dioxide. In the presence of CO_2 the limewater turns milky due to the formation of $\text{CaCO}_3(\text{s})$.]

Apparatus

1. Two Test tubes

2. Bungs
3. Retort stand
4. Copper carbonate
5. Spatula
6. Tongs
7. Delivery tub

Procedure

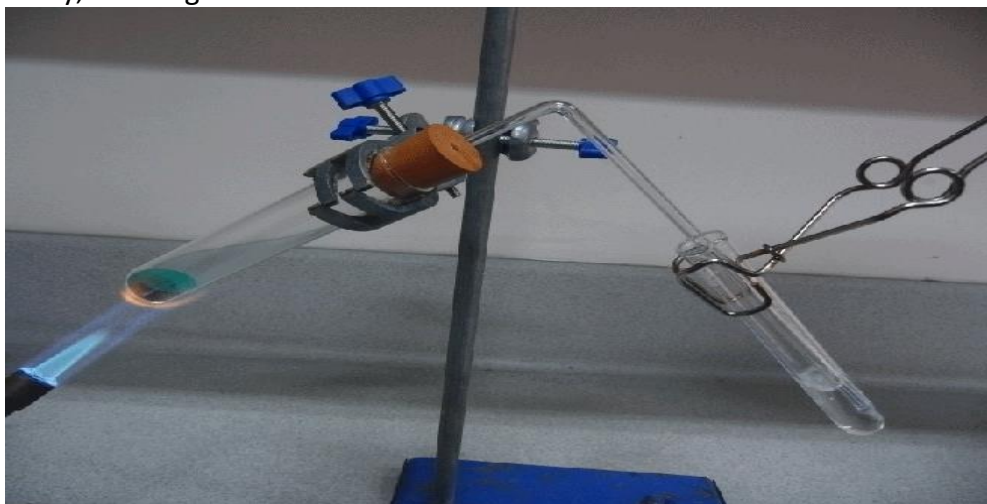
1. Set up the Apparatus as shown
2. Put a spatula of copper carbonate in one test tube and seal the test tube with the rubber
3. Carefully stick the delivery tube through the rubber stopper.
4. Pour lime water into the other test tube.
5. Place the other end of the delivery tube into the test tube containing the lime water.
6. Heat the copper carbonate over the Bunsen flame and observe what happens to colours of copper carbonate powder and the lime water

Observation

1. you can see in the picture below: It shows the thermal decomposition of copper (II) carbonate:

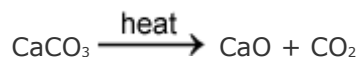


2. On the left of the picture the green copper (II) carbonate starting material can be seen turning black due to the formation of copper (II) oxide.
3. On the right the colorless gas produced is being bubbled through limewater which turns milky, showing it to be carbon dioxide



Conclusion

Metal carbonates such as copper carbonate break down when heated strongly. This is called thermal decomposition. Other metal carbonates decompose in the same way. Here are the equations for the thermal decomposition of calcium carbonate (lime stone):



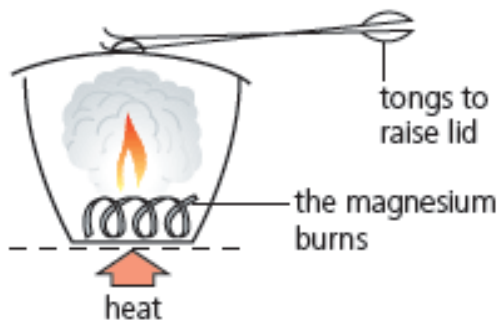
Experiment Empirical Formula

Objective

Determining the empirical formula of the reaction of magnesium with oxygen

Apparatus

1. Crucible with lid
2. Bunsen burner
3. Tripod with Gauze
4. Crucible tongs
5. Triangle pipe clay
6. Magnesium ribbon



Procedure

1. Weigh a crucible and lid, empty. Then add a coil of magnesium ribbon and weigh it again, to find the mass of the magnesium.
2. Heat the crucible. Raise the lid carefully at intervals to let oxygen in. The magnesium burns brightly.
3. When burning is complete, let the crucible cool (still with its lid on). Then weigh it again. The increase in mass is due to oxygen.

Observation

To work out the empirical formula, you need to know the masses of elements that combine. *The only way to do this is by experiment.*

The results showed that 0.3g of magnesium combined with 0.2g of oxygen. Draw up a table again:

Elements that combine	magnesium	oxygen
Masses that combine	2.4 g	1.6 g
Relative atomic masses (A_r)	24	16
Moles of atoms that combine	2.4/24 0.1	1.6/16 0.1
Ratio in which atoms combine	1:1	
Empirical formula	MgO	

So the empirical formula for the oxide is **MgO**.

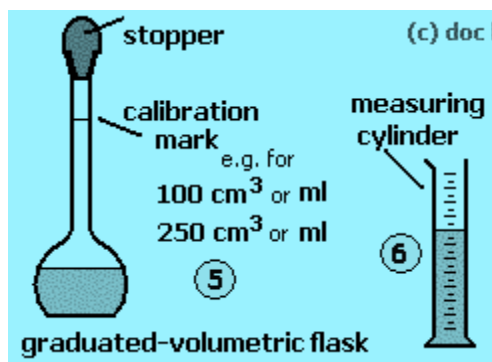
Conclusion

Explain what you have observed

Experiment: Volumetric Calculations

Objective

To learn how to make diluted solutions from a more concentrated stock solution. You will learn how to use the dilutions equation, and prepare several different dilutions from a concentrated NaOH stock solution.



Apparatus

1. volumetric flasks-250ml
2. glass stirring rod
3. Digital scale
4. Sodium hydroxide
5. Distilled water
6. spatula
7. measuring cylinder -25 ml
8. beaker -100 ml
9. measuring cylinder -40 ml
10. weighing paper (paper used for weighing NaOH)

Procedure

Preparing the stock solution (0.4M)

1. Put the weighing paper on the scale and touch the TARE button of the scale
2. weigh 4 g of NaOH on the digital scale (Do not put NaOH on the scale but on paper)
3. Half fill the small beaker about 50 ml of distilled water.
4. Add the weighed solid sodium hydroxide to the water and stir with glass stirring rod.
5. Transfer the solution to the volumetric flask.
6. Add more distilled water up to the mark 250 ml.
7. Now you have 0.4M NaOH stock solution (C_1)

Making diluted solutions from a more concentrated stock solution

1. Measure 20 ml of 0.4M NaOH from the stock solution. (V_1)
2. add more distilled water to sodium hydroxide solution in the measuring cylinder to the mark 40 ml (V_2)
3. Calculate the desired volume (V_2) by using the equation below.

Diluting Concentrated Solutions Equation:

$$C_1V_1 = C_2V_2$$

Where C_1 = concentration of starting (stock) solution

V_1 = volume to use of the stock solution to make diluted solution

C_2 = desired concentration of diluted solution

V_2 = desired volume of the diluted sample

Observation

Dilution means to reduce the concentration of a solution. For example, in this experiment 20 ml of 0.4 M of sodium hydroxide is diluted to 40 ml of 0.2 M.

A solution can be diluted by adding more solvent to the stock solution (the starting solution before dilution) in the same vessel.

A solution can be diluted by using a pipette to transfer some of the stock solution to the volumetric flask and then adding solvent up to the mark.

Conclusion

For each of the following molar solutions, show the calculation (equation with all units) for the preparation of each solution. Then, describe how you would make the solution in an appropriate container.

- a. 1 L of 0.5 M KNO_3 solution.
- b. 500 ml of 0.4M of NaOH
- c. 250 ml of 0.8 NaOH

Chapter 3: Titrimetric Analysis

The term 'titrimetric analysis' refers to quantitative chemical analysis carried out by determining the volume of a solution of accurately known concentration which is required to react quantitatively with a measured volume of a solution of the substance to be determined. The solution of accurately known strength is called the **standard solution**. The weight of the substance to be determined is calculated from the volume of the standard solution used and the chemical equation and relative molecular masses of the reacting compounds.

The term 'volumetric analysis' was formerly used for this form of quantitative determination but it has now been replaced by **titrimetric analysis**. It is considered that the latter expresses the process of titration rather better, and the former is likely to be confused with measurements of volumes, such as those involving gases. In titrimetric analysis the reagent of known concentration is called the **titrant** and the substance being titrated is termed the **titrand (the analyte)**. The alternative name has not been extended to apparatus used in the various operations; so, the terms volumetric glassware and volumetric flasks are still common, but it is better to employ the expressions graduated glassware and graduated flasks.

The standard solution is usually added from a long-graduated tube called a burette. The process of adding the standard solution until the reaction is just complete is termed a **titration, and** the substance to be determined is **titrated**. The point at which this occurs is called the **equivalence point** or the (Stoichiometric) **end point**. The completion of the titration is detected by some physical change, produced by the standard solution itself (e.g. the faint pink colour formed by potassium permanganate) or, more usually, by the addition of an auxiliary reagent, known as an indicator. After the reaction between the substance and the standard solution is practically complete, the indicator should give a clear visual change (either a colour change or the formation of turbidity) in the liquid being titrated. The point at which this occurs is called the **end point of the titration**.

For use in titrimetric analysis a reaction must fulfil the following conditions.

1. There must be a simple reaction which can be expressed by a chemical equation; the substance to be determined should react completely with the reagent in Stoichiometric or equivalent proportions.

2. The reaction should be relatively fast. (Most ionic reactions satisfy this condition.) In some cases, the addition of a catalyst may be necessary to increase the speed of a reaction.
3. There must be an alteration in some physical or chemical property of the solution at the equivalence point.
4. An indicator should be available which, by a change in physical properties (colour or formation of a precipitate), should sharply define the end point of the reaction.

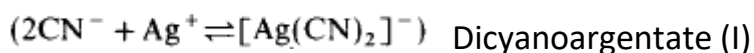
Classification of Reactions in Titrimetric Analysis

The reactions employed in titrimetric analysis fall into four main classes. The first three of these involve no change in oxidation state as they are dependent upon the combination of ions. But the fourth class, oxidation-reduction reactions, involves a change of oxidation state or, expressed another way, a transfer of electrons.

1. Neutralization reactions, or acidimetry and alkalimetry : These include the titration of free bases, or those formed from salts of weak acids by hydrolysis, with a standard acid (**acidimetry**), and the titration of free acids, or those formed by the hydrolysis of salts of weak bases, with a standard base (**alkalimetry**). The reactions involve the combination of hydrogen and hydroxide ions to form water.

Also, under this heading must be included titrations in non-aqueous solvents, most of which involve organic compounds.

2. **Complex formation reactions**: These depend upon the combination of ions, other than hydrogen or hydroxide ions, to form a soluble, slightly dissociated ion or compound, as in the titration of a solution of a cyanide with silver nitrate



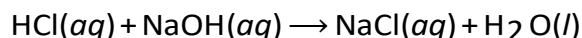
Ethylene diamines tetra acetic acid, largely as the disodium salt of EDTA, is a very important reagent for complex formation titrations and has become one of the most important reagents used in titrimetric analysis. Equivalence point detection by the use of metal-ion indicators has greatly enhanced its value in titrimetry.

3. **Precipitation reactions:** These depend upon the combination of ions to form a simple precipitate as in the titration of silver ion with a solution of a chloride. No change in oxidation state occurs.
4. **Oxidation-reduction reactions:** Under this heading are included all reactions involving change of oxidation number or transfer of electrons among the reacting substances. The standard solutions are either oxidizing or reducing agents. The principal oxidizing agents are potassium permanganate, potassium dichromate, cerium (IV) sulphate, iodine, potassium iodate, and potassium bromate. Frequently used reducing agents are iron (II) and tin (II) compounds, sodium thiosulphate, arsenic (III) oxide, mercury(I) nitrate, vanadium (II) chloride or sulphate, chromium (II) chloride or sulphate, and titanium (III) chloride or sulphate

Example

Titrimetric Analysis

The end point in a titration of a 50.00-mL sample of aqueous HCl was reached by addition of 35.23 mL of 0.250 M NaOH titrant. The titration reaction is:

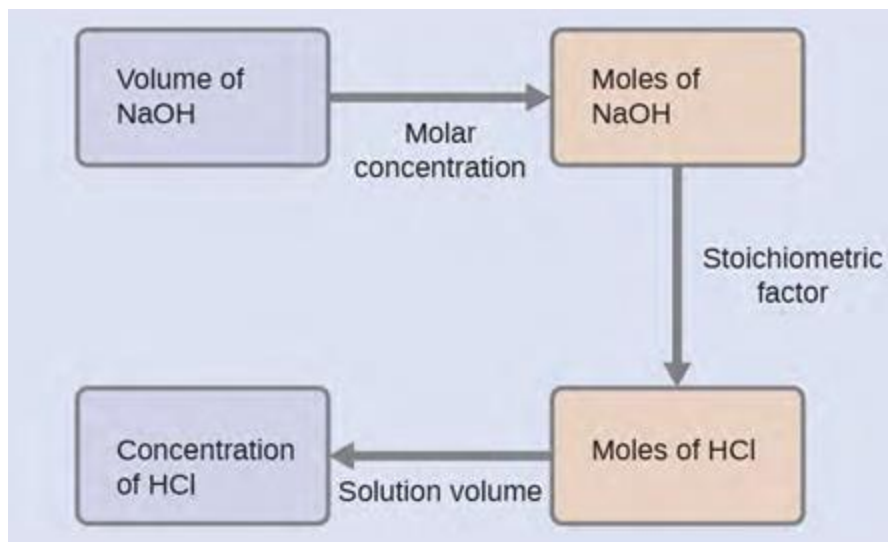


What is the molarity of HCl?

Solution

As for all reaction stoichiometry calculations, the key issue is the relation between the molar amounts of the chemical species of interest as depicted in the balanced chemical equation. since the amounts of reactants are provided and requested are expressed as solution concentrations.

For this exercise, the calculation will follow the following outlined steps



The molar amount of HCl is calculated to be:

$$35.23 \text{ mL NaOH} \times \frac{1 \text{ L}}{1000 \text{ mL}} \times \frac{0.250 \text{ mol NaOH}}{1 \text{ L}} \times \frac{1 \text{ mol HCl}}{1 \text{ mol NaOH}} = 8.81 \times 10^{-3} \text{ mol HCl}$$

Using the provided volume of HCl solution and the definition of molarity, the HCl concentration is:

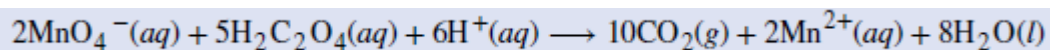
$$M = \frac{\text{mol HCl}}{\text{L solution}}$$

$$M = \frac{8.81 \times 10^{-3} \text{ mol HCl}}{50.00 \text{ mL} \times \frac{1 \text{ L}}{1000 \text{ mL}}}$$

$$M = 0.176 \text{ M}$$

Check Your Learning

A 20.00-mL sample of aqueous oxalic acid, $\text{H}_2\text{C}_2\text{O}_4$, was titrated with a 0.09113-M solution of potassium permanganate.



A volume of 23.24 mL was required to reach the end point. What is the oxalic acid molarity?
Answer: 0.2648 M

Chapter 4: Complex ions and coordination compounds

Coordination compounds

coordination complex **Coordination compounds** are neutral substances (i.e. uncharged) in which **at least** one ion is present as a complex.

A **coordination complex** is a substance in which a metal atom or ion accepts electrons from (and thus associates with) a group of neutral molecules or anions called **ligands**. A complex can be an anion, a cation ion, or a neutral molecule.

The coordination sphere

- The coordination sphere is the collection of components of a coordination compound that includes the central atom and the ligands surrounding this central atom given along with the net electrical charge of the compound.
- The electrical charge of a coordination compound is mentioned in coordination spheres.
- Example: $[\text{Fe}(\text{CN})_6]^{4-}$ hexacyanoferrate (II)

ligands

a ligand is an anion or neutral molecule that binds to a central metal atom to form a coordination complex.

Donor atoms

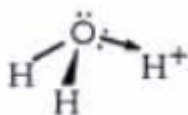
Within a ligand, **the atom that is directly bonded to the metal atom/ion** is called the donor atom.

coordinate covalent bond

A coordinate covalent bond is a covalent bond in which one atom (i.e., the donor atom) supplies both electrons. This type of bonding is different from a normal covalent bond in which each atom supplies one electron.

Formation of Complex ions

In aqueous solution, H^+ ions are attached to polar water molecules by coordinate bonds forming H_3O^+ ions. In the same way, other cations also exist in aqueous solution as hydrated ions with formulae of the type, $[\text{M}(\text{H}_2\text{O})_n]$ ion.



Co-ordinate bonding in an H_3O^+

H^+ ions are very small and it is assumed that each one associates with only one water molecules. The larger size of other cations does, however, allow them to associate with two, four or even six water molecules. For example, Ag^+ ions exist in an aqueous solutions as $[\text{Ag}(\text{H}_2\text{O})_2]^+$ diaquasilver(I), Cu^{2+} ions exist as $[\text{Cu}(\text{H}_2\text{O})_4]^{2+}$ tetraaquacopper(II) and Fe^{3+} ions exist as $[\text{Fe}(\text{H}_2\text{O})_6]^{3+}$ hexaaquairon(III)

Other polar molecules, besides water, can also co-ordinate with metal cations. Thus in an aqueous ammonia Cu^{2+} exists mainly as $[\text{Cu}(\text{NH}_3)_4]^{2+}$ tetraminecopper(II) and Ag^+ exists as $[\text{Ag}(\text{NH}_3)_2]^+$ diamminesilver(I). Anion, such as Cl^- , OH^- , and $\text{S}_2\text{O}_3^{2-}$, can also associate with cations in the same way as polar molecules like H_2O and NH_3 . Thus Ag^+ exists as $[\text{Ag}(\text{S}_2\text{O}_3)_2]^{3-}$ in sodium thiosulphate solution and Cu^{2+} ions form $[\text{CuCl}_4]^{2-}$ in concentrated hydrochloric acid.

Ions such as $[\text{Ag}(\text{S}_2\text{O}_3)_2]^{3-}$ and $[\text{Ag}(\text{NH}_3)_2]^+$, in which a metal ion is associated with anions or neutral molecules, are known as **complex ions**. The anions and molecules co-ordinated to the central cation are called **ligands**. Each ligand contains at least one atom bearing a lone pair of electrons which can be donated to the central cation forming a co-ordinate (dative) bond.

Most ligands form only one co-ordinate bond with a cation. These ligands, which include H_2O , NH_3 and Cl^- , are said to be **unidentate** because they have only 'one tooth' with which to attach themselves to the central cation in a complex. (The word 'dens' in Latin means tooth.) In some cases, ligands form two or more co-ordinate bonds to a central metal ion and these ligands are said to be **polydentate**, meaning 'many teeth' /

Mono dentate Ligands

Monodentate ligands are also called "one-toothed" because they bite the metal atom only in one place.

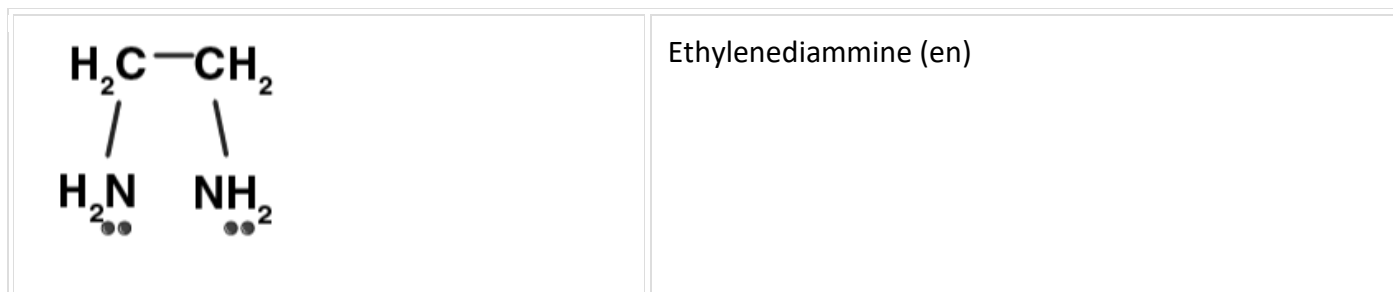
Examples

Water, ammonia, hydroxide and bromide Br^-

Polydentate ligand

Polydentate ligand: a ligand that is attached to a central metal ion by bonds from two or more donor atoms. Ethylenediamine and oxalate ions are **Polydentate** or **Bidentate** because they contain two donor atoms

Ethylenediamine donates two lone pairs of electrons to the central metal atom. it is known as bidentate ligands. They are often referred to as a “chelating ligands”. The complex which contains chelating ligands is called “Chelates



Polydentate ligand is a ligand that is attached to a central metal ion by bonds from two or more donor atoms.

The ligand such as edta (ethylenediaminetetracetate) in $[Ag(edta)]^{3-}$ can form as many as six coordinate bonds with the central ion. (4 from oxygens and two from nitrogen atoms). The complex ions formed between polydentate ligands and cations are known as chelates or chelated complex. $[Ag(edta)]^{3-}$ is an example

Chelate

a compound containing a ligand bonded to a central metal atom at two or more points

The Greek word ‘chelos’ means ‘claw’ and the polydentate ligands form a claw-like grip on the central metal ion



What is the coordination number of the complex hexacyanoferrate (II) $[\text{Fe}(\text{CN})_6]^{4-}$ and hexacyanoferrate (III) $[\text{Fe}(\text{CN})_6]^{3-}$

Either the complex ion should be $[\text{Fe}(\text{CN})_6]^{4-}$ or $[\text{Fe}(\text{CN})_6]^{3-}$. Whatever it may be coordination number will be 6 because CN is an unidentate ligand and central metal ion Fe^{2+} or Fe^{3+} is attached to 6 CN^- ions

What coordination number is $\text{Co}(\text{NH}_3)_5(\text{NO}_2)\text{Cl}_2$ in the complex?

Coordination number of Co in this complex compound is 6 because cobalt (III) ion makes 6 coordinate bonds with five ammonia molecules and a nitrite ion which act as ligands.

What coordination number is $[\text{Pt}(\text{en})_2]\text{CO}_3$?

(bis(ethylenediamine)platinum(II) carbonate)

The coordination sphere is $[\text{Pt}(\text{en})_2]^{2+}$, since $(\text{CO}_3)^{2-}$ exists as an anion.

So here Pt is the central metal ion or the coordination center (which is generally a metal) is attached to 2 ions i.e. (en) i.e. ethylenediamine.

So, since **Pt** is surrounded by **2 (en)** in the coordination sphere,

And since **en** is a **bidentate** i.e. attaches itself from two sides due to presence of lone pairs.

we can say coordination no. is 4.

The intention of this practical is to study the formation of complexes and to determine the relative strength with which different ligands form complex ions.

REQUIREMENTS

Each student, or pair of students, will need:

Eye protection

6 test tubes

2 teat pipettes

Access to approximately 0.1 mol dm^{-3} solutions of the following:

Potassium thiocyanate (**harmful solid**).
Dissolve 9.7 g of KSCN in distilled water and make up to 1 dm^3 .

Sodium 2-hydroxybenzoate (salicylate).
Dissolve 16.0 g of $\text{HOC}_6\text{H}_4\text{COONa}$ in distilled water and make up to 1 dm^3 .

Procedure

CARE!

Eye protection must be worn for all these experiments.

A Put 10 drops of iron(III) chloride solution in a test tube.

- 1 The complex ion in this solution is $[\text{Fe}(\text{H}_2\text{O})_6]^{3+}$. What is its colour and what ligand does it contain?

B Add potassium thiocyanate solution, $\text{KSCN}(\text{aq})$, drop by drop until no further change occurs. (Keep half of the resulting solution for part E. Use the other half for part C.)

- 2 What is the colour of the solution now? What ligand is present in the complex ion?

C If the solution from B is too deeply coloured, dilute it until the true colour of the complex is evident. Now, add sodium 2-hydroxybenzoate (salicylate) solution drop by drop until there is no further change in colour.

3 What is the colour of the solution now? What ligand is now present in the complex ion?

Coordination number

- The number of atoms(**donners**) of the ligands that are directly bond to the central metal atom or ion by coordinate bonds is known as the coordination number of the metal atom or ion.
- It is actually the number of coordinate covalent which the ligands form with the central metal atom or ion

Complex	Metal	Coordination of metal number
$K_4[Fe(CN)_6]$	+ 2	6
$[Cu(NH_3)_4]SO_4$	+ 2	4
$Ni(CO)_4$	0	4

What is the coordination number of $K_4 [Fe (CN) _6]$?

Naming Coordination Compounds

The coordination compounds are named in the following way:

A. When naming coordination compounds, always name the cation before the anion. This rule holds regardless of whether the complex ion is the cation or the anion. (This is just like naming an ionic compound.)

B. In naming the complex ion:

1. Name the ligands first, in alphabetical order, and then name the central metal. Note: In the chemical formula the central metal is written before the ligands.

2. The names of some common ligands are listed in Table 1.

-Anionic ligands end in "-o." For anions that end in "-ide"(e.g. chloride, hydroxide), "-ate" (e.g. sulfate, nitrate), and "-ite" (e.g. nitrite), change the endings as follows:

-ide → -o; e.g., chloride → chloro and hydroxide → hydroxo

-ate → -ato; e.g., sulfate → sulfato and nitrate → nitrato

-ite → -ito; e.g., nitrite → nitrito

For neutral ligands, the common name of the molecule is used (e.g. $\text{H}_2\text{NCH}_2\text{CH}_2\text{NH}_2$ (ethylenediamine)). Important exceptions: water is called 'aqua', ammonia is called 'ammine', carbon monoxide is called 'carbonyl', and the N_2 and O_2 molecules are called 'dinitrogen' and 'dioxygen'.

Names of Some Common Ligand

Anionic Ligands	Names	Neutral Ligands	Names
Br^-	bromo	NH_3	ammine
F^-	fluoro	H_2O	aqua
O^{2-}	oxo	NO	Nitrosyl
OH^-	hydroxo	CO	Carbonyl
CN^-	cyano	O_2	dioxygen
$\text{C}_2\text{O}_4^{2-}$	oxalato	N_2	dinitrogen
Br^-	bromo	$\text{C}_5\text{H}_5\text{N}$	pyridine
CO_3^{2-}	carbonato	$\text{H}_2\text{NCH}_2\text{CH}_2\text{NH}_2$	ethylenediamine

CH ₃ COO ⁻	acetato		
----------------------------------	---------	--	--

3. The Greek prefixes di-, tri-, tetra-, etc. are used to designate the number of each type of ligand in the complex ion. If the ligand already contains a Greek prefix (e.g. ethylenediamine) or if it is a polydentate ligand (i.e. it can attach at more than one coordination site), the prefixes bis-, tris-, tetrakis-, and pentakis- are used instead. (See examples 3 and 4.) The numerical prefixes are listed in this Table.

Number	Prefix	Number	Prefix	Number	Prefix	Number
1	mono	5	penta (pentakis)	9	nona (ennea)	1
2	di (bis)	6	hexa (hexakis)	10	deca	2
3	tri (tris)	7	hepta	11	undeca	3
4	tetra (tetrakis)	8	octa	12	dodeca	4

4. After naming the ligands, name the central metal. If the complex ion is a cation, the metal is named same as the element. For example, Co in a complex cation is called cobalt and Pt is called platinum. (See examples 1-4.) If the complex ion is an anion, the name of the metal ends with the suffix -ate. (See examples 5 and 6.) For example, Co in a complex anion is called cobaltate and Pt is called platinate. For some metals, the Latin names are used in the complex anions (e.g. Fe is called ferrate and not ironate).

Name of Metals in Anionic Complexes

Name of Metal	Name in an Anionic Complex
Iron	Ferrate
Copper	Cuprate
Lead	Plumbate
Silver	Argentate
Gold	Aurate
Tin	Stannate

5. Following the name of the metal, the oxidation state of the metal in the complex is given as a Roman numeral in parentheses.

C. To name a neutral complex molecule, follow the rules of naming a complex cation. **Remember:** Name the (possibly complex) cation **BEFORE** the (possibly complex) anion. See examples 7 and 8.

For historic reasons, some coordination compounds are called by their common names. For example: $\text{Fe}(\text{CN})_6^{3-}$ and $\text{Fe}(\text{CN})_6^{4-}$ are named ferricyanide and ferrocyanide respectively, and $\text{Fe}(\text{CO})_5$ is called iron carbonyl.

Examples:

Give the systematic names for the following coordination compounds:

1. $[\text{Cr}(\text{NH}_3)_3(\text{H}_2\text{O})_3]\text{Cl}_3$

Answer: triamminetriaquachromium (III) chloride

Solution:

•The complex ion is found inside the parentheses. In this case, the complex ion is a cation.

•The ammine ligands are named first because alphabetically, "ammine" comes before "aqua."

•The compound is electrically neutral and thus has an overall charge of zero. Since there are three chlorides associated with one complex ion and each chloride has a -1 charge, the charge on the complex ion must be $+3$.

•From the charge on the complex ion and the charge on the ligands, we can calculate the oxidation number of the metal. In this example, all the ligands are neutral molecules. Therefore, the oxidation number of chromium must be the same as the charge of the complex ion, $+3$.

2. $[\text{Pt}(\text{NH}_3)_5\text{Cl}]\text{Br}_3$

Answer: pentaamminechloroplatinum(IV) bromide

Solution:

- The complex ion is a cation, and the counter anions are the 3 bromides.
- The charge of the complex ion must be +3 since it is associated with 3 bromides.
- The NH_3 molecules are neutral while the chloride carries a -1 charge.
- Therefore, the oxidation number of platinum must be +4.

3. $[\text{Pt}(\text{H}_2\text{NCH}_2\text{CH}_2\text{NH}_2)_2\text{Cl}_2]\text{Cl}_2$

Answer: dichlorobis(ethylenediamine)platinum(IV) chloride

Solution:

- Since Ethylenediamine is a bidentate ligand, the prefix bis- is used instead of the prefix di-

4. $[\text{Co}(\text{H}_2\text{NCH}_2\text{CH}_2\text{NH}_2)_3]_2(\text{SO}_4)_3$

Answer: tris(ethylenediamine)cobalt(III) sulfate

Solution:

- The sulfate has a charge of -2 and is the counter anion in this molecule.
- Since it takes 3 sulfates to bond with two complex cations, the charge on each complex cation must be +3.
- Since ethylenediamine is a neutral molecule, the oxidation number of cobalt in the complex ion must be +3.
- Again, remember that you never have to indicate the number of cations and anions in the name of an ionic compound.

5. $\text{K}_4[\text{Fe}(\text{CN})_6]$

Answer: potassium hexacyanoferrate (II)

Solution:

•Potassium is the cation and the complex ion is the anion.

•Since there are 4 K^+ associated with the complex ion (each K^+ having a +1 charge), the charge on the complex ion must be -4 .

•Since each ligand carries -1 charge, the oxidation number of Fe must be $+2$.

•The common name of this compound is potassium ferrocyanide.

6. $Na_2[NiCl_4]$

Answer: sodium tetrachloronickelate (II)

Solution:

•The complex ion is the anion so we have to add the suffix $-ate$ to the name of the metal.

7. $Pt(NH_3)_2Cl_4$

Answer: diamminetetrachloroplatinum (IV)

Solution:

•This is a neutral molecule because the charge on Pt^{+4} equals the negative charges on the four chloro ligands.

•If the compound is $[Pt(NH_3)_2Cl_2]Cl_2$, even though the number of ions and atoms in the molecule are identical to the example, it should be named: diamminedichloroplatinum(IV) chloride because the platinum in the latter compound is only four coordinated instead of six coordinated. (difference is coordination and ionicity).

8. $Fe(CO)_5$

Answer: pentacarbonyliron (0)

Solution:

•Since it is a neutral complex, it is named in the same way as a complex cation. The common name of this compound, iron carbonyl, is used more often.

9. $(\text{NH}_4)_2[\text{Ni}(\text{C}_2\text{O}_4)_2(\text{H}_2\text{O})_2]$

Answer: ammonium diaquabis (oxalato)nickelate(II)

Solution: The oxalate ion is a bidentate ligand.

10. $[\text{Ag}(\text{NH}_3)_2][\text{Ag}(\text{CN})_2]$

Answer: diamminesilver(I) dicyanoargentate(I)

You can have a compound where both the cation and the anion are complex ions. Notice how the name of the metal differs even though they are the same metal ions.

Can you give the molecular formulas of the following coordination compounds?

- 1. hexaammineiron(III) nitrate*
- 2. ammonium tetrachlorocuprate(II)*
- 3. sodium monochloropentacyanoferrate(III)*
- 4. potassium hexafluorocobaltate(III)*

Can you give the name of the following coordination compounds?

- 5. $[\text{CoBr}(\text{NH}_3)_5]\text{SO}_4$*
- 6. $[\text{Fe}(\text{NH}_3)_6][\text{Cr}(\text{CN})_6]$*
- 7. $[\text{Co}(\text{SO}_4)(\text{NH}_3)_5]^+$*
- 8. $[\text{Fe}(\text{OH})(\text{H}_2\text{O})_5]^{2+}$*

Answers:

- 1. $[\text{Fe}(\text{NH}_3)_6](\text{NO}_3)_3$*
- 2. $(\text{NH}_4)_2[\text{CuCl}_4]$*

3. $\text{Na}_3[\text{FeCl}(\text{CN})_5]$

4. $\text{K}_3[\text{CoF}_6]$

5. *pentaamminebromocobalt(III) sulfate*

6. *hexaammineiron(III) hexacyanochromate (III)*

7. *pentaamminesulfatocobalt(III) ion*

8. *pentaaquahydroxoiron(III) ion*

Chapter 5 : Gravimetric analysis

Gravimetric analysis is the process of isolating and weighing an element or a definite compound of the element as pure a form as possible.

The steps of gravimetric analysis

The steps commonly followed in gravimetric analysis are (1) preparation of a [solution](#) containing a known weight of the sample, (2) separation of the desired constituent, (3) weighing the isolated constituent, and (4) computation of the amount of the particular constituent in the sample from the observed weight of the isolated substance.

The separation of the element or of the compound containing it may be affected in a number of ways, the most important of which are: (a) precipitation methods; (b) volatilization or evolution methods; (c) electroanalytical methods; and (d) extraction and chromatographic methods.

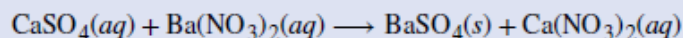
Only (a) and (b) will be discussed in this chapter. They are 2 common types of gravimetric analysis. Both involve changing the phase of the analyte to separate it from the rest of a mixture, resulting in a change in mass.

What is precipitation gravimetry?

Precipitation gravimetry is an analytical technique that uses a precipitation reaction to separate ions from a solution. The chemical that is added to cause the precipitation is called the *precipitant* or *precipitating agent*. The solid precipitate can be separated from the liquid components using filtration, and the mass of the solid can be used along with the balanced chemical equation to calculate the amount or concentration of ionic compounds in solution.

Gravimetric Analysis

A 0.4550-g solid mixture containing CaSO_4 is dissolved in water and treated with an excess of $\text{Ba}(\text{NO}_3)_2$, resulting in the precipitation of 0.6168 g of BaSO_4 .



What is the concentration (percent) of CaSO_4 in the mixture?

The mass of CaSO_4 that would yield the provided precipitate mass is

$$0.6168 \text{ g BaSO}_4 \times \frac{1 \text{ mol BaSO}_4}{233.43 \text{ g BaSO}_4} \times \frac{1 \text{ mol CaSO}_4}{1 \text{ mol BaSO}_4} \times \frac{136.14 \text{ g CaSO}_4}{1 \text{ mol CaSO}_4} = 0.3597 \text{ g CaSO}_4$$

The concentration of CaSO_4 in the sample mixture is then calculated to be

$$\text{percent CaSO}_4 = \frac{\text{mass CaSO}_4}{\text{mass sample}} \times 100\%$$

$$\frac{0.3597 \text{ g}}{0.4550 \text{ g}} \times 100\% = 79.05\%$$

Volatilization gravimetry

Volatilization gravimetry involves separating components of our mixture by heating or chemically decomposing the sample. The heating or chemical decomposition separates out any volatile compounds, which results in a change in mass that we can measure.

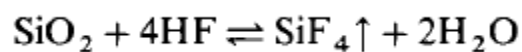
Volatilization gravimetry involves **separating components of our mixture by heating or chemically decomposing the sample**. The heating or chemical decomposition separates out any volatile compounds, which results in a change in mass that we can measure

The process of heating or chemical decomposition that separates out volatile compounds, which results in a change in mass that we can measure, is termed as Volatilization

The determination of carbon dioxide in carbonate-containing materials may be effected by treating the sample with excess of acid and absorbing the carbon dioxide in an alkaline absorbent, such as soda-lime or 'Carbosorb'.* The gas is completely expelled by heating the solution and by passing a current of purified air through the apparatus; it is, of course, led through a drying agent to remove water vapour before

passing to the carbon dioxide absorption apparatus. The gain in weight of the latter is due to carbon dioxide

Another example is the determination of pure silica in an impure ignited silica residue. The latter is treated in a platinum crucible with a mixture of sulphuric and hydrofluoric acids; the silica is converted into the volatile silicon tetrafluoride:



The residue consists of the impurities, and the loss in weight of the crucible gives the amount of pure silica present, provided that the contaminants are in the same form before and after the hydrofluoric acid treatment and are not volatilised in the operation. Although silicon is not the only element that forms a volatile fluoride, it is by far the most abundant and most often encountered element; consequently, the volatilisation method of separation is generally satisfactory.

Summary

Qualitative Analysis it is an analysis carried out to determine only the identity of a pure analyte, the identity of an analyte in a matrix.

Quantitative Analysis is any method used for determining the amount of a chemical in a sample (amount of analyte).

In order to recognize a substance we change it, usually with the help of another substance of known nature (reagent), into a new compound which possesses distinctive properties.

A reagent is a substance added to a system to test or determine the presence or absence of a specific chemical substance.

Wet chemistry is the ordinary chemical reaction. It is a chemical reaction that occurs when a liquid reagent and a sample are added to a reaction vessel and mixed. **A dry chemistry analyzer** is a process in which a liquid test sample is added directly to a dry reagent strip manufactured specifically for each project.

The analysis is a process that provides chemical or physical information about the constituents in the sample. The components of a sample that are to be identified or determined are often referred to as **analytes** and the word for the material in which the analyte is found is called the **matrix** of the analyte.

Determination is an analysis of a sample to find the identity, concentration, or properties of the analyte

Characterizing: A combination of the qualitative and quantitative analysis of a material or matrix is sometimes called **characterizing** the material.

Standardization is an analytical technique used to find an unknown concentration using a primary or a secondary standard solution. The most commonly used technique for standardization of a solution is titration.

A primary standard is a compound which is stable, of high purity, highly soluble in water and of a high molar mass to allow for accurate weighing.

Some examples of primary standards for titration of solutions, based on their high purity, are provided

- **Potassium bromate** (KBrO_3)
- **Potassium hydrogen phthalate** (usually called KHP) for standardization of aqueous base
- **Sodium carbonate** for standardization of aqueous acids

A **standard solution** is a solution of accurately known concentration prepared from a primary standard that is weighed accurately and made up to a fixed volume.

Calculating **mass of solute** in a molar or standard solution **in grams**

$$\text{Molarity of solution} = \frac{\text{moles of solute}}{\text{volume of solution}}$$

$$\text{Moles of solute (n)} = \text{Molarity of solution(M)} \times \text{volume of solution(V)}$$

Example

Calculate mass (grams) of NaOH that would be required to produce a 0.5M solution with a volume of 200 ml?

Convert volume in litres $200 \text{ ml} / 1000 = 0.2 \text{ L}$

$$\text{Moles of solute (n)} = \text{Molarity of solution(M)} \times \text{volume of solution(V)}$$

$$n = 0.5 \text{ mol/L} \times 0.2 \text{ L} = 0.1 \text{ mol}$$

Convert 0.1mol into grams:

$$\text{Mass (g)} = \text{moles of solute (n)} \times \text{molar mass (mm)}$$

$$0.1 \text{ mol} \times 40 \text{ g/mol} = 4 \text{ g}$$

Note

200 ml of 0.5M of sodium hydroxide solution **contains 4g** of NaOH

More calculation with different volumes and molarity

Molarity of NaOH	Volume of NaOH	Mass (g) of NaOH
0.5 M	200 ml	4 g
0.5 M	400 ml	8 g
0.4 M	1000 ml	16 g
0.4M	500 ml	8 g
0.8 M	250 ml	8 g
1M	500 ml	20 g

Making Molar Solutions from Concentrated Aqueous Acids

Calculating number of milliliters of 35% by mass of hydrochloric acid that contains 1M mol or 36.5g of hydrochloric acid, Density of hydrochloric acid 1.18g/ml and molar mass =36.5g/mol

$$D = \frac{\text{Mass}}{\text{Vol}} \quad V = \frac{M}{D} \quad \text{Vol} = \frac{36.5\text{g/mol}}{1.18\text{g/ml}} = 30.9 \text{ ml}$$

Number of millimeters that contains 1M of HCl or 36.5gmol =

$$\frac{30.9 \times 100}{35} = 88 \text{ ml}$$

Checking that 36.5g = 1 mol HCl is in 88 ml

$$88 \text{ ml} \times 1.18 \text{ g/ml} = 104\text{g}$$

$$\frac{104\text{g} \times 35}{100} = 36.5 \text{ g}$$

Calculating different concentration of HCl

concentration		Number of millimeters
1M	1 M x 88 ml	88 ml
0.1 M	0.1 x 88 ml	8.8 ml
0.5 M	0.5 x 88 ml	44 ml

Example 2

Given

- 1) Molar mass of sulphuric acid = 98g/mol
- 2) Specific gravity = 1.84g/ml
- 3) 96% by mass H₂SO₄

$$D = \frac{\text{Mass}}{\text{Vol}} \quad V = \frac{M}{D} = \frac{98\text{g}}{1.84 \text{ g/ml}} = 53.26 \text{ ml}$$

number of milliliters of 96% m/m of sulphuric acid that contains: 1 mole
or 98g of sulphuric acid :

$$\frac{53.26 \times 100}{96} = 55.5 \text{ ml}$$

Calculating the molarity of concentrated acids

Calculate the molarity of 60% by mass of Perchloric acid it has the density of 1.54g/ml

Solution

1) Calculate the mass of acid solution

No matter how much of concentrated acid you have, use volume of 1000 ml

$$\text{Mass} = \text{density} \times \text{volume} \quad \text{Mass} = 1.54\text{g/ml} \times 1000 \text{ ml} = 1540\text{g}$$

2) Calculate mass of Perchloric acid

$$\text{Mass of perchloric acid} = \frac{1540\text{g} \times 60}{100} = 924\text{g HClO}_4$$

3) Find moles of Perchloric acid

Molar mass of Perchloric acid $\text{HClO}_4 = 100.5\text{g/mol}$

$$\text{Moles of HClO}_4 = \frac{\text{Mass of HClO}_4}{\text{Molar mass}} = \frac{924\text{ g}}{100.5\text{g/mol}} = 9.2\text{ mol}$$

4) Calculate Volume (ml) required to make 1000 ml of acid solution (1M)

$$\text{Vol} = \frac{1000\text{ ml}}{\text{moles of acid}} = \frac{1000\text{ ml}}{9.2\text{ mol}} = 108.7\text{ ml/mol}$$

Slowly add 108.7 ml of **Perchloric acid** to the water. Add more water until 1000ml mark

Concentrated Acid	Density	Molarity (M)	Volume (ml) required to make 1000 ml of acid solution (1M)
Perchloric acid 60%	1.54	9.2	108.7
Perchloric acid 70%	1.67	11.6	86.2
Hydrochloric acid 35%	1.18	11.3	88
Sulfuric acid 96%	1.84	18	55.5

Making diluted solutions from a more concentrated stock solution

Diluting Concentrated Solutions Equation:

$$C_1V_1 = C_2V_2$$

Where C_1 = concentration of starting (stock) solution

V_1 = volume to use of the stock solution to make diluted solution

C_2 = desired concentration of diluted solution

V_2 = desired volume of the diluted sample

Example

You have a stock solution of HCl that is 12M. Your lab requires 500 ml of a 0.1M HCl solution. How many milliliters of the stock will you use to prepare the solution?

You have a stock solution of HCl that is 12M (**C₁**). Your lab requires 500 ml (**V₂**) of a 0.1M (**C₂**) HCl solution. How many milliliters (**V₁**) of the stock will you use to prepare the solution?

Solution

$$C_1V_1 = C_2V_2 \quad \text{or} \quad M_1 \times V_1 = M_2 \times V_2$$

Calculating V₁ (volume to use of the stock solution to make diluted solution)

Convert to ml to L 500 ml = 0.5 L

$$V_1 = \frac{C_2 \times V_2}{C_1} = \frac{0.1 \times 0.5}{12} = 0.004\text{L}$$

Convert L to ml = 0.004 x 1000 ml = 4 ml

Answer: V₁ = 4 ml

Note

Add 4 ml of 12M HCl to water to make 500 ml of 0.1M HCl

Stoichiometry (/stɔɪki'ɒmɪtri/) is a section of chemistry that involves using relationships between reactants and products in a chemical reaction to determine desired quantitative data.

Law of Conservation of Mass

The Law of Conservation of Mass states that matter is neither created nor destroyed. In a chemical reaction, every chemical reaction has the same elements in its reactants and products.

Types of chemical reactions

1) Combination (synthesis)

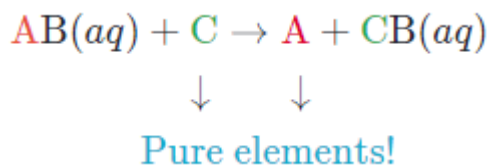
Reactions in which two or more reactants combine to give one product are called combination reactions for example the formation of HCl from the reaction of a chemical and H₂ and Cl₂

2) Decomposition reaction

In decomposition reaction, one reactant breaks down in two or more products for example copper (ii) carbonate decomposes when heated into copper oxide and carbon dioxide.

3) Single Displacement Reaction

A **single replacement reaction**, sometimes called a **single displacement reaction**, is a reaction in which one element is substituted for another element in a compound. The starting materials are always pure elements, such as a pure zinc metal or hydrogen gas, plus an aqueous compound. When a replacement reaction occurs, a new aqueous compound and a different pure element will be generated as products. The general pattern of a single replacement reaction is shown below



Examples

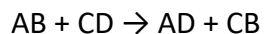
Zinc metal reacts with HCl producing an aqueous zinc chloride and hydrogen gas

Or sodium metal react with water forming sodium hydroxide and hydrogen gas

4) Double Displacement

A double displacement reaction is a **type of reaction in which two reactants exchange ions to form two new compounds**. Double displacement reactions typically result in the formation of a product that is a precipitate

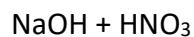
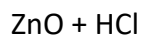
Double displacement reactions take the form:



- A double displacement reaction is a type of chemical reaction in which the reactant ions exchange places to form new products.
- Usually, a double displacement reaction results in precipitate formation.
- A double displacement reaction is also called a double replacement reaction, salt metathesis reaction, or double decomposition.

5) Acid-Base

Acid- base reaction is a chemical reaction in which acid and a base react quantitatively with each other. Neutral substances are formed such as water and salts.



6) Combustion

Combustion is the formation of CO_2 and H_2O from the reaction of a chemical and oxygen O_2

Example

Burning methane with oxygen gives CO_2 and H_2O