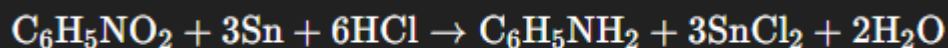


## 19. Laboratory Preparation of Aniline

**Aniline** is commonly prepared in the laboratory by the reduction of nitrobenzene. The standard procedure involves:

Mixing nitrobenzene with granulated tin (Sn) and concentrated hydrochloric acid (HCl) in a round-bottom flask.

The mixture is heated under reflux, which reduces nitrobenzene to aniline via the following reaction:



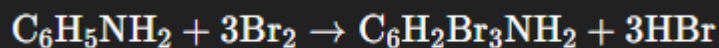
After the reaction, the mixture is made alkaline (using NaOH), and aniline is separated by steam distillation and purified by extraction and redistillation.

### Reactions of Aniline with Various Reagents

#### a) Reaction with Aqueous Bromine (aq. Br<sub>2</sub>)

**Observation:** When aniline is treated with aqueous bromine, a white precipitate forms, and the reddish-brown color of bromine water disappears.

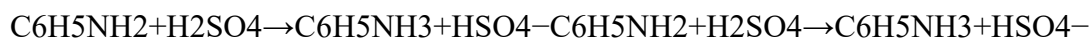
**Reaction:** Aniline undergoes rapid bromination at the ortho and para positions, yielding 2,4,6-tribromoaniline:



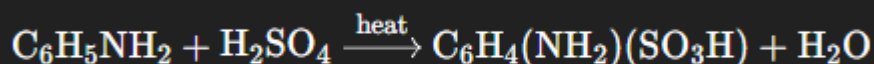
**Product:** 2,4,6-tribromoaniline (white precipitate).

#### b) Reaction with Concentrated Sulphuric Acid (conc. H<sub>2</sub>SO<sub>4</sub>)

**Step 1:** Aniline reacts with concentrated sulphuric acid to form anilinium hydrogen sulfate:



**Step 2:** Upon heating, this intermediate undergoes sulfonation to produce sulphanilic acid (p-aminobenzenesulfonic acid):



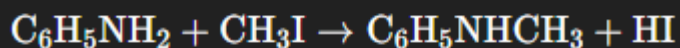
**Product:** Sulphanilic acid.

#### c) Reaction with Iodomethane (CH<sub>3</sub>I)

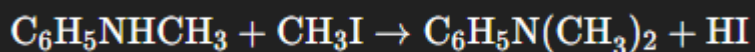
**Process:** Aniline reacts with iodomethane (methyl iodide) in an alkylation reaction.

**Reaction:** The nitrogen atom in aniline attacks the methyl group of iodomethane, first forming N-methylaniline. With excess  $\text{CH}_3\text{I}$ , further alkylation can occur, producing N,N-dimethylaniline and, with even more  $\text{CH}_3\text{I}$ , the quaternary salt (trimethylanilinium iodide):

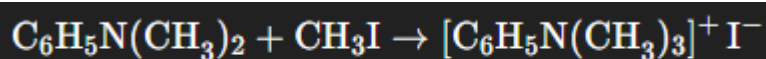
First step:



Further methylation:



With excess methyl iodide and base:



**Products:** N-methylaniline, N,N-dimethylaniline, and finally, trimethylanilinium iodide (quaternary salt) depending on the amount of  $\text{CH}_3\text{I}$  used.

20.

### Faraday's Laws of Electrolysis

Michael Faraday formulated two fundamental laws that describe the quantitative relationship between the amount of substance altered at an electrode during electrolysis and the quantity of electricity passed through the electrolyte.

#### Faraday's First Law of Electrolysis

##### Statement:

The mass ( $m$ ) of a substance deposited or liberated at an electrode during electrolysis is directly proportional to the quantity of electricity ( $Q$ ) passed through the electrolyte.

##### Mathematical Expression:

$$m \propto Q \Rightarrow m = Z \cdot Q$$

Where:

$m$  = mass of substance deposited (in grams)

$Q$  = total electric charge passed (in coulombs)

$Z$  = electrochemical equivalent (ECE) of the substance (grams per coulomb)

Since  $Q = I \times t$  (where  $I$  is current in amperes and  $t$  is time in seconds), the formula can also be written as:

$$m = Z \cdot I \cdot t$$

**Explanation:**

This law means that the greater the amount of electricity passed, the more substance will be deposited or liberated at the electrode. The proportionality constant  $Z$  is specific to each substance and represents the mass deposited by one coulomb of charge.

**Faraday's Second Law of Electrolysis****Statement:**

When the same quantity of electricity is passed through different electrolytes, the masses of substances deposited or liberated at the electrodes are directly proportional to their respective chemical equivalents (equivalent weights).

**Mathematical Expression:**

$$\frac{m_1}{m_2} = \frac{E_1}{E_2}$$

Where:

$m_1, m_2$  = masses of substances deposited

$E_1, E_2$  = equivalent weights of the substances

The equivalent weight ( $E$ ) is calculated as:

$E = \frac{\text{Atomic (or molar) mass}}{\text{Valency}}$      $E = \frac{\text{Valency}}{\text{Atomic (or molar) mass}}$

**Explanation:**

If the same amount of charge is passed through solutions of different electrolytes, the ratio of the masses deposited is equal to the ratio of their equivalent weights. This law allows comparison of deposition for different substances under identical electrical conditions.

**Key Points**

**One Faraday (F):** The quantity of electricity required to deposit or liberate one gram equivalent of a substance. Numerically,  $1 F = 96,500$  coulombs.

**Electrochemical Equivalent (Z):** The mass of a substance deposited by one coulomb of electricity.

21.

### Laboratory Preparation of Hydrogen

**Principle:** Hydrogen gas is commonly prepared in the laboratory by reacting granulated zinc with dilute acids such as hydrochloric acid (HCl) or sulphuric acid (H<sub>2</sub>SO<sub>4</sub>).

**Procedure:**

Place zinc granules in a flask.

Add dilute HCl or H<sub>2</sub>SO<sub>4</sub> through a thistle funnel.

The reaction produces hydrogen gas, which is collected over water using a delivery tube, as hydrogen is less dense than water and only slightly soluble in it.

**Chemical Reactions:**

With hydrochloric acid:  
$$\text{Zn(s)} + 2\text{HCl(aq)} \rightarrow \text{ZnCl}_2\text{(aq)} + \text{H}_2\text{(g)}$$

With sulphuric acid:  
$$\text{Zn(s)} + \text{H}_2\text{SO}_4\text{(aq)} \rightarrow \text{ZnSO}_4\text{(aq)} + \text{H}_2\text{(g)}$$

**Precautions:** Ensure all air is expelled from the apparatus before collecting hydrogen, as hydrogen mixed with air is explosive. Test for hydrogen by bringing a burning splint near the mouth of the jar; a 'pop' sound confirms its presence.

### Allotropes of Carbon

Allotropes are different forms of the same element, where atoms are bonded in different ways, resulting in distinct physical and chemical properties.

**Major Allotropes of Carbon:**

**Diamond:** Each carbon atom is tetrahedrally bonded to four others, making it extremely hard and transparent. It is an excellent thermal conductor but a poor electrical conductor.

**Graphite:** Carbon atoms form layers of hexagonal rings. Each atom is bonded to three others, with delocalized electrons allowing graphite to conduct electricity and act as a lubricant.

**Fullerenes:** Molecules like C<sub>60</sub> (buckminsterfullerene) are spherical or tubular. They have unique properties and applications in nanotechnology.

**Graphene:** A single layer of carbon atoms arranged in a hexagonal lattice. It is strong, flexible, and an excellent conductor.

**Carbon Nanotubes:** Cylindrical structures with remarkable strength and electrical conductivity.

**Amorphous Carbon:** Lacks a crystalline structure (e.g., charcoal, soot).

**Properties Table:**

Allotrope	Structure	Key Properties
Diamond	Tetrahedral 3D network	Hardest, transparent, insulator
Graphite	Layered hexagonal	Soft, black, conductor, lubricant
Fullerenes	Spherical/tubular	Unique, used in nanotechnology
Graphene	Single-layer hexagonal	Strong, flexible, good conductor
Nanotubes	Cylindrical	Strong, conductive
Amorphous	No definite structure	Variable, used in inks, pigments

These allotropes differ in their atomic arrangements, leading to a wide range of uses from jewelry (diamond) to lubricants (graphite) and advanced materials (graphene, nanotubes).

**Laboratory Preparation of Sulphur Dioxide (SO<sub>2</sub>)**

**Principle:** SO<sub>2</sub> gas is prepared by reacting a metal sulphite (such as sodium sulphite, Na<sub>2</sub>SO<sub>3</sub>) with dilute sulphuric acid or by heating copper turnings with concentrated sulphuric acid.

**Method 1: Using Sodium Sulphite**

Place sodium sulphite in a flask.

Add dilute H<sub>2</sub>SO<sub>4</sub> slowly.

SO<sub>2</sub> gas evolves and is collected by upward displacement of air (since it is denser than air).

Reaction:



**Method 2: Using Copper and Concentrated Sulphuric Acid**

Place copper turnings in a flask.

Add concentrated H<sub>2</sub>SO<sub>4</sub> and heat gently.

SO<sub>2</sub> gas is liberated and collected as above.

Reaction:



**Precautions:** SO<sub>2</sub> is a toxic, choking gas; the experiment should be conducted in a well-ventilated area or fume hood. The gas can be dried by passing it through concentrated sulphuric acid before collection