**Exp.7: Preparation of Aniline**

**Principle:**

Aniline is also known as aminobenzene or phenylamine. Many reducing agents

have been introduced to reduce aromatic nitro compounds. The most commonly

used reducing agents are Zn, Sn or Fe in acid and catalytic hydrogenation.

(i) Aniline formation:

In the laboratory, when nitrobenzene is heated with tin and HCl, aniline

is formed. The reaction of tin (Sn) and conc HCl produce nascent

hydrogen and SnCl2 or SnCl4, the nascent hydrogen reduces – NO2

group to – NH2 group. The produced aniline further reacts with HCl

forming anilinium hydrochloride salt.



(ii) **Separation of aniline:**

When an alkali solution is added to this salt, aniline is separated and

remains floating forming a layer of a dark brown oil.



**Physical properties of aniline:**

* Aniline is colourless oily liquid having boiling point 1840C and melting

point – 60C.

* It has a characteristic unpleasant smell.
* It is sparingly soluble (insoluble in water but soluble in organic solvents

like ethanol, ether and benzene.

* It is steam volatile (and hence can be steam distilled from a mixture

containing inorganic substances).

* It is highly toxic in nature.

**Aniline is used:**

* For preparing dyes and dye intermediates.
* For produce of antioxidants and vulcanization accelerators in rubber

production.

* For a mixture of drugs, particularly sulpha drugs.
* For making isocyanates required for polyurethane plastics used for

insulation.

**Method of preparation:**

1. 10 ml nitrobenzene and 20 gm of granulated tin are placed in the 250

ml round bottom flask fitted with a reflux condenser.

2. 50 ml of conc. HCl is added gradually with constant shaking. After each

addition, the round bottom flask is cooled so that the temperature may not

go above 900C.

3. Then the reaction mixture is heated on a boiling water bath for about

one hour until the reaction is completed which is indicated by the smell

of nitrobenzene, the disappearance of smell indicates the completion of

the reaction.

4. The flask is then cooled and a crystalline solid mass of double salt is

separated out.

5. The crystalline solid mass is then treated with conc. NaOH until the

solution is cleared and becomes strongly alkaline. Aniline is separated

out and is floated on the surface as dark brown oil.

6. The mixture obtained is then subjected to the process of steam

distillation until clear distillate is obtained.

7. Aniline is extracted by shaking the distillate several times with ether.

The ethereal layer is separated each time with the help of the separating

funnel. Now, the ethereal aniline is placed for evaporation whereas

ether evaporates out. Aniline thus obtained is finally purified by

redistillation at 182-1840C.



