Experimental Procedures

Experiment: 1 **Date: 20/03/2020;**
Lab No: 4 **Time: 9:00a.m. to 1:00p.m.**

Preparation of *p*-chlorobenzaldehyde

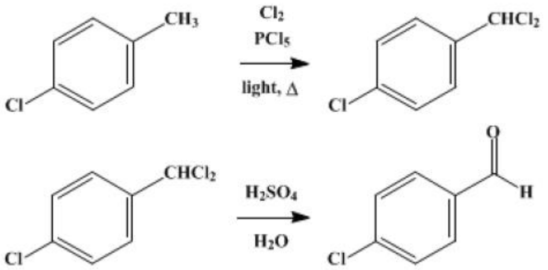

Figure 1 synthesis of *p*-chlorobenzaldehyde

Chemicals Required:

p-chlorotoluene	:6.30gm
Phosphorus pentachloride	:1.9gm
Suphuric Acid	:2 mL

Procedure:

A 100 ml two-necked, round-bottomed flask is provided with an air-cooled reflux condenser. Chlorine is to be introduced by means of a 4-mm. glass tube, inserted through a cork in a neck of the flask, extending close to the bottom of the flask and provided with a small bulb with fine perforations to break up the gas stream into small bubbles. The large quantities of hydrogen chloride formed can be disposed of by means of a gas absorption trap. Into the tared flask are placed 6.30 gm (0.05 mole) of *p*-chlorotoluene and 1.9 gm of phosphorus pentachloride. The flask is heated in a bath kept at 160–170°, and while illuminated with direct sunlight a rapid stream of chlorine is introduced directly from a cylinder until the gain in weight. The pale yellow or yellow-green product is then transferred to a 1 litre. wide-mouthed bottle containing 2 ml of concentrated sulfuric acid, and stirred vigorously (Hood) for five hours. The viscous mixture is then transferred to a separatory funnel and allowed to stand overnight, after which the lower layer is run slowly, with stirring, into a 1 litre beaker three-quarters filled with cracked ice. The cream-colored solid obtained when the ice has melted is filtered by suction, washed with water, pressed dry on the funnel, and divided into three equal parts. Each portion is dissolved in a minimum of ether, and the ether solution is repeatedly shaken with 2 per cent sodium hydroxide solution until acidification of the washings gives no precipitate of *p*-chlorobenzoic acid. After removal of the ether by distillation on a steam bath, the residue is distilled under diminished pressure and got the product.

M.P. 46-47°C

Yield: 4.55gm (65%)

Experiment: 2 **Date: 24/03/2020;**
Lab No: 5 **Time: 9:00a.m. to 1:00p.m.**

Preparation of malachite green

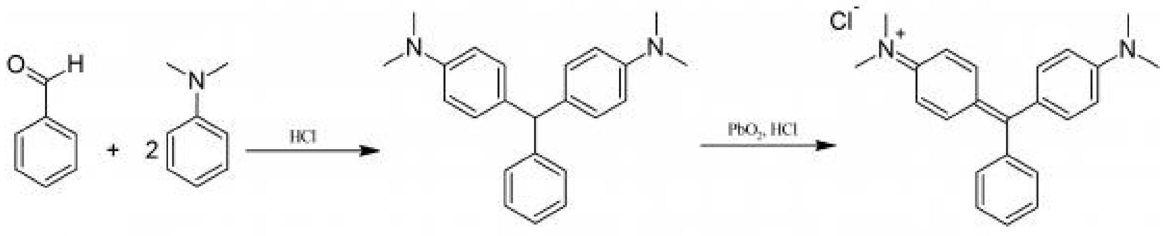

Figure 2 Synthesis of malachite green

Chemicals Required:

N,N-Dimethylaniline	:3.5gm
Benzaldehyde	:1.4gm
Conc. hydrochloric acid	:3.15 mL

Procedure:

3.5gm of dimethylaniline and 1.4gm of benzaldehyde are mixed with 3.15mL of concentrated hydrochloric acid and the mixture heated, under a reflux condenser, for 10 hours, at 100° C. The reaction mass is made alkaline with sodium hydroxide, after which traces of benzaldehyde and of dimethylaniline are removed by steam distillation. On pouring into 1 liter of water the leuco base separates in the form of hard granules. It is filtered off, washed free from alkali. The leuco base is oxidized as follows: 10 g of leuco base (dry weight) are melted by heating and a mixture of 2.7 g of hydrochloric acid and 1.0 grams of acetic acid in 250-300 ml of water are added, and a thin paste containing 1.0 grams of pure lead dioxide is allowed to flow in, with stirring, which is continued for 2 hours after the addition is complete. Unchanged lead dioxide is then filtered off, the filtrate is heated to boiling point and treated with sodium sulfate, to remove the lead. After filtration the solution is again raised to boiling point and the base is precipitated with the solution of sodium hydroxide. After cooling, it is filtered off, washed, dried, and purified by being dissolved in light petroleum and filtered from impurities, after which the petrol is distilled by evaporation yielding pure malachite green.

Yield: 6.0gm (50%)

Experiment: 3 **Date: 27/03/2020;**
Lab No: 4 **Time: 9:00a.m. to 1:00p.m.**

Preparation of Phenylazo-β-Naphthol

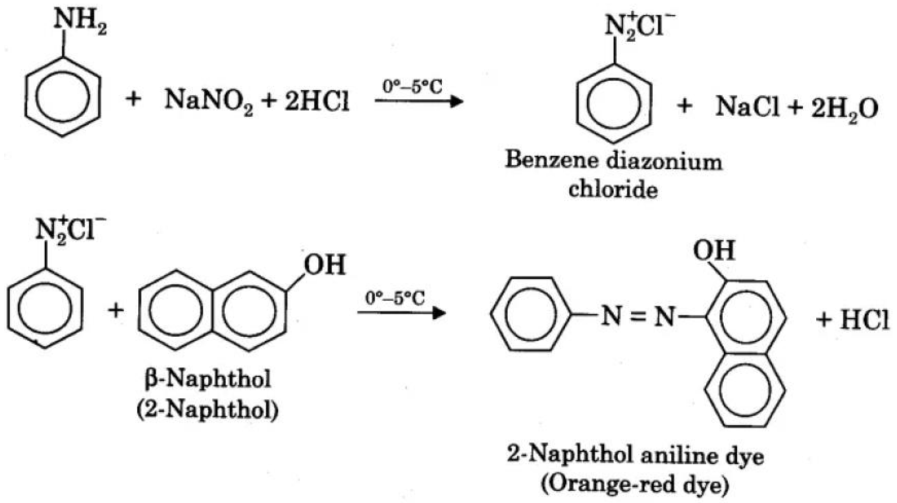

Figure 3 Synthesis of Phenylazo-β-Naphthol

Chemicals Required:

Aniline	:4.5mL
Sodium nitrite	:4.0gm
Conc. hydrochloric acid	:10 mL
2-naphthol	:7.0gm
10% NaOH solution	:60mL

Procedure:

Take a 100 ml conical flask and add 4.5 ml of aniline, 10 ml of cone. HCl and 20 ml of water. Cool this solution to 5°C by placing the conical flask in a trough containing ice- cold water. In a 100 ml beaker dissolve 4 g of sodium nitrite in 20 ml of water and cool this solution also to 5°C. Now slowly add sodium nitrite solution to the solution of aniline in cone. HCl. Dissolve 7.0 g of 2-naphthol in 60 ml of 10% NaOH solution taken in a 250 ml beaker and cool this solution to 5°C by placing in an ice bath. Some crushed ice may be added directly to fecilitate cooling. Now add the diazotised solution very slowly to the naphthol solution with constant stirring. The mixed solutions immediately develop a red colour and the phenyl-azo-β- naphthol rapidly separates as orange-red crystals. When the addition of diazo solution is complete, allow the mixture to stand in ice-salt mixture for 30 minutes, with occasional stirring. Filter the solution through a buchner funnel under suction from the pump. Wash the phenyl-azo-β-naphthol with water and dry the crystals obtained by pressing between the folds of filter paper. Recrystallise the product from glacial acetic acid. Filter the crystals obtained at the pump. Wash with a few ml of ethanol to remove acetic acid. Phenyl-azo-β-naphthol is obtained as orange-red crystals.

M.P. 133°C

Yield: 6.0gm (50%)

Experiment: 4 **Date: 31/03/2020;**
Lab No: 5 **Time: 9:00a.m. to 1:00p.m.**

Preparation of 4-methyl-7-methoxycoumarin

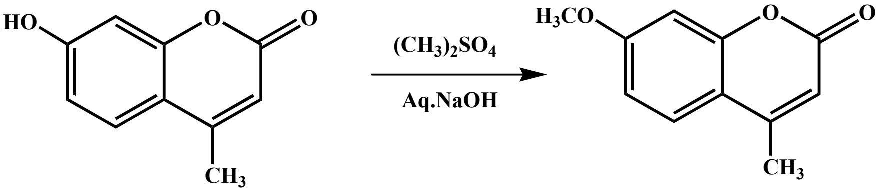

Figure 4 synthesis of 4-methyl-7-methoxycoumarin

Chemicals Required:

4-methyl-7-hydroxycoumarin	:2.75mL
Dry acetone	:40mL
Dimethyl sulphate	:1.8 mL
Ignited potassium carbonate	:6.0gm
Hot acetone	:15mL

Procedure

4-methyl-7-hydroxycoumarin (2.75g, 0.16mol) is dissolve in dry acetone (40ml) and reflux with dimethyl sulphate (1.8ml, 0.17mol) and ignited potassium carbonate (6.0g) for 3 hours under anhydrous conditions. The acetone solution is filtered and the inorganic salts washed with hot acetone (15ml). The combined acetone solution is distilled and the oily residue is macerated with ice. the solid product is filtered, washed with water and crystallized from methanol.

M.P. 160-163°C

Yield: 2.8gm (90%)