Chapter II
Experimental methods

Synthesis of metallodyes

In this chapter, the materials employed at various stages of investigation, experimental procedure adopted to synthesis some of the starting compounds and methods of purification of solvents have been indicated. It also deals with a brief account of different physico-chemical techniques employed for the characterization of various complexes synthesis in this study.

Materials employed

Metal salts

1. Lead (II) acetate monohydrate, BDH product.

2. Cobalt (II) acetate tetra hydrate, SD product.

3. Nickel (II) acetate tetra hydrate, BDH product.

4. Copper (II) acetate monohydrate, BDH product.

Dyes

The dyes Amaranth, Methylene blue, Remazol red B and Golden yellow HER were analytical grade samples and directly used.

Solvents

Solvents like methanol, dimethylformamide and dimethylsulfoxide.used in the preparation of various complexes were of AR grade and were purified by the standard procedures.

2.1 Synthesis of lead complexes

2.1.1 Synthesis of lead complex of Amaranth

15.12 g (1 m mol) of ligand Amaranth was dissolved in about 25 cm^3 distilled water and 3.80 g (0.5 m mol) of lead acetate was dissolved in 20 cm^3 of distilled water. The ligand Amaranth solution (25 cm^3) was added slowly to the lead acetate solution with constant stirring and the pH of the mixture was raised to 7 by the addition of 10% alcoholic ammonia solution. The mixture was reflexes for about 3 h in order to complete the reaction upon slow evaporation of the solvent crude remained which was collected by filtration, washed several times with methanol then dried over P_2O_5[1-10].

2.1.2 Synthesis of lead complex of Methylene blue

8.00 g (1m mol) of ligand Methylene blue was dissolved in about 25 cm^3 distilled water and 3.80 g (0.5m mol) of lead acetate was dissolved in 20 cm^3 of distilled water. The ligand Methylene blue solution (25 cm^3) was added slowly to the lead acetate solution with constant stirring and the pH of the mixture was raised to 7 by the addition of 10% alcoholic ammonia solution. The mixture was reflexes for about 3 h in order to Complete the reaction upon slow evaporation of the solvent crude remained which was collected by filtration, washed several times with methanol then dried over P_2O_5.

2.1.3 Synthesis of lead complex of Remazol red B

9.62 g (1 m mol) of ligand Remazol red B was dissolved in about 25 cm^3 distilled water and 3.80 g (0.5 m mol) of lead acetate was dissolved in 20 cm^3 of distilled water. The ligand Remazol red B solution (25 cm^3) was added slowly to the

lead acetate solution with constant stirring and the pH of the mixture was raised to 7 by the addition of 10% alcoholic ammonia solution. The mixture was reflexes for about 3 h in order to complete the reaction upon slow evaporation of the solvent crude remained which was collected by filtration, washed several times with methanol then dried over P_2O_5.

2.1.4 Synthesis of lead complex of Golden yellow HER

8.14 g (1 m mol) of ligand Golden yellow HER was dissolved in about 25 cm^3 distilled water and 3.80 g (0.5 m mol) of lead acetate was dissolved in 20 cm^3 of distilled water. The ligand Golden yellow HER solution (25 cm^3) was added slowly to the lead acetate solution with constant stirring and the pH of the mixture was raised to 7 by the addition of 10% alcoholic ammonia solution. The mixture was reflexes for about 3 h in order to complete the reaction upon slow evaporation of the solvent crude remained which was collected by filtration, washed several times with methanol then dried over P_2O_5.

2.2 Synthesis of cobalt complexes

2.2.1 Synthesis of cobalt complex of Amaranth

15.12 g (1 m mol) of ligand Amaranth was dissolved in about 25 cm^3 distilled water and 2.95 g (0.5 m mol) of cobalt acetate was dissolved in 20 cm^3 of distilled water. The ligand Amaranth solution (25 cm^3) was added slowly to the cobalt acetate solution with constant stirring and the pH of the mixture was raised to 7 by the addition of 10% alcoholic ammonia solution. The mixture was reflexes for about 3 h in order to complete the reaction upon slow evaporation of the solvent crude remained which was collected by filtration, washed several times with methanol then dried over P_2O_5.

2.2.2 Synthesis of cobalt complex of Methylene blue

8.00 g (1 m mol) of ligand Methylene blue was dissolved in about 25 cm^3 distilled water and 2.95 g (0.5 m mol) of cobalt acetate was dissolved in 20 cm^3 of distilled water. The ligand Methylene blue solution (25 cm^3) was added slowly to the cobalt acetate solution with constant stirring and the pH of the mixture was raised to 7 by the addition of 10% alcoholic ammonia solution. The mixture was reflexes for about 3 h in order to complete the reaction upon slow evaporation of the solvent crude remained which was collected by filtration, washed several times with methanol then dried over P_2O_5.

2.2.3 Synthesis of cobalt complex of Remazol red B

9.62 g (1 m mol) of ligand Remazol red B was dissolved in about 25 cm^3 distilled water and 2.95 g (0.5 m mol) of cobalt acetate was dissolved in 20 cm^3 of distilled water. The ligand Remazol red B solution (25 cm^3) was added slowly to the cobalt acetate solution with constant stirring and the pH of the mixture was raised to 7 by the addition of 10% alcoholic ammonia solution. The mixture was reflexes for about 3 h in order to complete the reaction upon slow evaporation of the solvent crude remained which was collected by filtration, washed several times with methanol then dried over P_2O_5.

2.2.4 Synthesis of cobalt complex of Golden yellow HER

8.14 g (1 m mol) of ligand Golden yellow HER was dissolved in about 25 cm^3 distilled water and 2.95 g (0.5 m mol) of cobalt acetate was dissolved in 20 cm^3 of distilled water. The ligand Golden yellow HER solution (25 cm^3) was added slowly to the cobalt acetate solution with constant stirring and the pH of the mixture was raised to 7 by the addition of 10% alcoholic ammonia solution. The mixture was reflexes for

about 3 h in order to complete the reaction upon slow evaporation of the solvent crude remained which was collected by filtration, washed several times with methanol then dried over P_2O_5.

2.3 Synthesis of nickel complexes

2.3.1 Synthesis of nickel complex of Amaranth

15.12 g (1 m mol) of ligand Amaranth was dissolved in about 25 cm^3 distilled water and 2.49 g (0.5 m mol) of nickel acetate was dissolved in 20 cm^3 of distilled water. The ligand Amaranth solution (25 cm^3) was added slowly to the nickel acetate solution with constant stirring and the pH of the mixture was raised to 7 by the addition of 10% alcoholic ammonia solution. The mixture was reflexes for about 3 h in order to complete the reaction upon slow evaporation of the solvent crude remained which was collected by filtration, washed several times with methanol then dried over P_2O_5.

2.3.2. Synthesis of nickel complex of Methylene blue

8.00 g (1 m mol) of ligand Methylene blue was dissolved in about 25 cm^3 distilled water and 2.49 g (0.5 m mol) of nickel acetate was dissolved in 20 cm^3 of distilled water. The ligand Methylene blue solution (25 cm^3) was added slowly to the nickel acetate solution with constant stirring and the pH of the mixture was raised to 7 by the addition of 10% alcoholic ammonia solution. The mixture was reflexes for about 3 h in order to complete the reaction upon slow evaporation of the solvent crude remained which was collected by filtration, washed several times with methanol then dried over P_2O_5.

2.3.3. Synthesis of nickel complex of Remazol red B

9.62 g (1 m mol) of ligand Remazol red B was dissolved in about 25 cm^3 distilled water and 2.49 g (0.5 m mol) of nickel acetate was dissolved in 20 cm^3 of distilled water.

The ligand Remazol red B solution (25 cm^3) was added slowly to the nickel acetate solution with constant stirring and the pH of the mixture was raised to 7 by the addition of 10% alcoholic ammonia solution. The mixture was reflexes for about 3 h in order to complete the reaction upon slow evaporation of the solvent crude remained which was collected by filtration, washed several times with methanol then dried over P_2O_5.

2.3.4. Synthesis of nickel complex of Golden yellow HER

8.14 g (1 m mol) of ligand Golden yellow HER was dissolved in about 25 cm^3 distilled water and 2.49 g (0.5 m mol) of nickel acetate was dissolved in 20 cm^3 of distilled water. The ligand Golden yellow HER solution (25 cm^3) was added slowly to the nickel acetate solution with constant stirring and the pH of the mixture was raised to 7 by the addition of 10% alcoholic ammonia solution. The mixture was reflexes for about 3 h in order to complete the reaction upon slow evaporation of the solvent crude remained which was collected by filtration, washed several times with methanol then dried over P_2O_5.

2.4. Synthesis of copper complexes

2.4.1 Synthesis of copper complex of Amaranth

15.12 g (1 m mol) of ligand Amaranth was dissolved in about 25 cm^3 distilled water and 2.00g (0.5 m mol) of copper acetate was dissolved in 20 cm^3 of distilled water. The ligand Amaranth solution (25 cm^3) was added slowly to the copper acetate solution with constant stirring and the pH of the mixture was raised to 7 by the addition of 10% alcoholic ammonia solution. The mixture was reflexes for about 3 h in order to complete the reaction upon slow evaporation of the solvent crude remained which was collected by filtration, washed several times with methanol then dried over P_2O_5.

2.4.2 Synthesis of copper complex of Methylene blue

8.00 g (1 m mol) of ligand Methylene blue was dissolved in about 25 cm^3 distilled water and 2.00 g (0.5 m mol) of copper acetate was dissolved in 20 cm^3 of distilled water. The ligand Methylene blue solution (25 cm^3) was added slowly to the copper acetate solution with constant stirring and the pH of the mixture was raised to 7 by the addition of 10% alcoholic ammonia solution. The mixture was reflexes for about 3 h in order to complete the reaction upon slow evaporation of the solvent crude remained which was collected by filtration, washed several times with methanol then dried over P_2O_5.

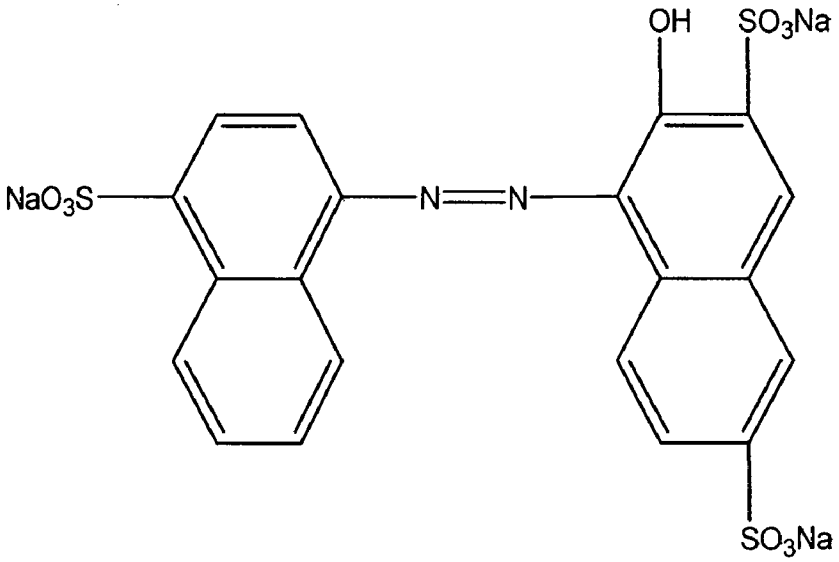
2.4.3 Synthesis of copper complex of Remazol red B

9.62 g (1 m mol) of ligand Remazol red B was dissolved in about 25 cm^3 distilled water and 2.00 g (0.5 m mol) of copper acetate was dissolved in 20 cm^3 of

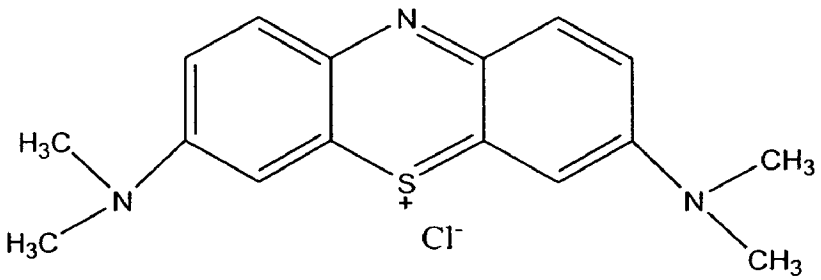
distilled water. The ligand Remazol red B solution (25 cm^3) was added slowly to the copper 0acetate solution with constant stirring and the pH of the mixture was raised to 7 by the addition of 10% alcoholic ammonia solution. The mixture was reflexes for about 3 h in order to complete the reaction upon slow evaporation of the solvent crude remained which was collected by filtration, washed several times with methanol then dried over P_2O_5.

2.4.4. Synthesis of copper complex of Golden yellow HER

8.14 g (1 m mol) of ligand Golden yellow HER was dissolved in about 25 cm^3 distilled water and 2.00 g (0.5 m mol) of copper acetate was dissolved in 20 cm^3 of distilled water. The ligand Golden yellow HER solution (25 cm^3) was added slowly to the copper acetate solution with constant stirring and the pH of the mixture was raised to 7 by the addition of 10% alcoholic ammonia solution. The mixture was refluxed for about 3 h in order to complete the reaction upon slow evaporation of the solvent crude remained which was collected by filtration, washed several times with methanol then dried over P_2O_5. The structure of all the ligand in fig.2.1



Amaranth



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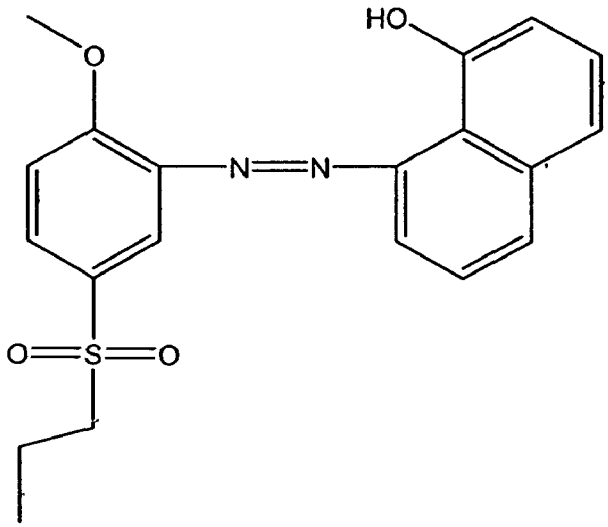
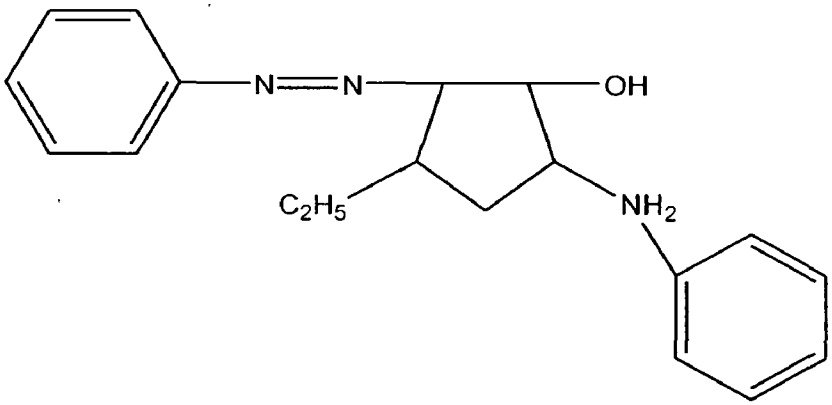


Remazol red B



Golden yellow HER

Fig.2.1

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