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OLED production:

Electroluminescence with
Polyvinyl carbazide (PVC) as hole conductor

Experiment execution:

Two different OLEDs are to be produced in this practical trial. The components required for this (phosphors, substrate and hole conductor) must be synthesized and then combined using various processes.

1. Aluminium tris(8-hydroxyquinoline) (Alq3) (Day 1)

a) Preparation of the necessary solutions

- 1% PVK in 10 ml chlorobenzene. The solution must be filtered through a 2 µm filter paper.
- Ammonium acetate solution 2 mol/l
- Precipitation reagent: Dissolve 2.5 g 8-hydroxyquinoline in 6 ml conc. acetic acid and dilute with distilled water. Fill up to 50 ml with water.

b) Preparation of the complex

Dissolve 1.853 mmol $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ in a 400 ml beaker and fill up to 200 ml. Then add 4 ml conc. acetic acid and heat to 70 °C. Add 30 ml of the previously prepared precipitant to the sample solution and add as much ammonium acetate solution as necessary to form a permanent yellow-green precipitate. Then add further 20 ml of ammonium acetate solution. Wait an hour and check that the precipitate is complete by adding a few drops of precipitant. The resulting complex is vacuumed off and washed with H_2O . Dry the precipitate at 120 °C.

2. Preparation of $\text{Eu}(\text{tfa})_3\text{phen}$ (1,10-phenanthroline)-tris(2-thenoyltrifluoroacetato)europium

a) Production of $\text{Eu}(\text{tfa})_3$ (Day 1)

To prepare 0.8 g of product, dissolve a stoichiometric amount of thenoyltrifluoroacetylacetone in 50 ml ethanol/ H_2O (90/10) and add the corresponding volume of a Eu^{3+} solution. Adjust the pH to 8–9 with diluted ammonia. The $\text{Eu}(\text{tfa})_3$ is obtained by careful evaporation until dry at the rotary evaporator. Solve the dried product in 50 ml tert. Butyl methyl ether and extract twice with 25 ml H_2O in the separating funnel. Remove the water residues by adding MgSO_4 as desiccant. After 30 minutes, the MgSO_4 is filtered off and the filtrate is evaporated at the rotary evaporator, whereby the final product precipitates. Dry the fluorescent in the drying cabinet at 50 °C.

b) Production of $\text{Eu}(\text{tfa})_3\text{phen}$ (Day 2)

The addition of phenanthroline further improves the luminescence properties.

Therefore, dissolve the complex in ethanol and add the phenanthroline in a molar ratio of 1:1. The $\text{Eu}(\text{tfa})_3\text{phen}$ can be isolated by evaporation (rotary evaporator) and drying (50 °C).

Purify the product by recrystallization in toluene.

3. preparation Eu(ttfa)₃phen (synthesis pathway 2) (day 1-3) **(1,10-Phenanthroline)-tris(2-thenoyltrifluoroacetato)europium**

First, dissolve 2 mmol $\text{EuCl}_3 \cdot 6\text{H}_2\text{O}$ in 25 ml dist. H_2O and transferred into a dropping funnel. Then 6 mmol of thenoyltrifluoroacetone and 2 mmol of phenanthroline are dissolved in 50 ml of ethanol and adjusted to a pH between 8 and 9 with 1 molar NH_3 solution. With continuous stirring, the EuCl_3 solution is slowly dropped to the ligand solution, a yellowish precipitate should form. After completion of the precipitation, the resulting suspension is stirred overnight.

The next day, the precipitate is sucked off and dried at 60 °C in a vacuum drying oven.

The product is purified by recrystallisation in water. For this purpose, it is dissolved in a little acetone (approx. 30 ml) under heat and slowly dropped into water while stirring. The resulting suspension is stirred for 30 minutes, then filtered, washed with cold water and dried at 60 °C (overnight if necessary).

4. Preparation of the glass substrate coated with indium tin oxide (ITO) **(Day 2)**

a) Cleaning ITO glass (substrate) before etching and spin coating:

1. Cleaning with microfiber cloth and ethanol
2. Place the glass in a beaker and cover with methanol. Cover the beaker with a watch glass and treat for 10 minutes in an ultrasonic bath.

b) Etch:

Cover the clean and dry substrate by spraying with photoresist and dry it in the dark (5 minutes in air, 15 minutes in the drying cabinet at 70 °C).

Cover the dried substrate with the mask and irradiate with a 366 nm UV lamp for 5 minutes.

To remove the photoresist, dip the exposed substrate into a beaker containing 0.7 % NaOH and treat for about two minutes. Rinse with distilled water.

To remove the ITO layer from the exposed areas, place the rinsed substrate in a beaker with 60 °C hot, semi-concentrated HCl and treated for about two minutes. Rinse with distilled water and dry the substrate with cellulose.

Remove the remaining photoresist carefully with acetone.

Check the successful etching with the conductivity meter.

c) Thin film application via spincoating

Place the previously etched substrate carefully in the middle of the spincoater disc and close the lid.

Apply approx. 1 ml of the PVC solution with a Pasteur pipette in the middle.

The centrifuge is started (300 rpm, 2 minutes).

At the end of the program, carefully lift off the substrate.

If necessary, clean the backside with cellulose and toluene.

Use the UV lamp to test the homogeneity of the coating.

5. Apply thin film with the high vacuum evaporation system (Day 3+4)

a) Phosphors:

Thickness 10 – 200 nm (depending on formulation)

Apply mask and attach substrate to target.

Mix sample with same amount of quartz sand and fill into boats.

Evaporate the phosphor in a high vacuum. (approx. $1.5 - 2.5 \cdot 10^{-5}$ mbar)

b) Al electrode:

Thickness approx. 200 – 300 nm

Place the mask on the target and fix it in place.

Fill shuttle with Al wire pieces (total length = 15 cm, length of wire pieces < 2 mm).

Evaporate the Al in a high vacuum. (approx. $1.5 - 2.5 \cdot 10^{-5}$ mbar)

6. OLEDs to be manufactured:

- ITO| 1 % PVK Lsg.| 30 nm Alq₃| 200 nm Al
- ITO| 1 % PVK Lsg.| 10 nm Eu(tffa)₃phen| 15 – 20 nm Alq₃| 200 nm Al

