# BS 192: Chemistry Lab Report
# Experiment 5: Synthesis of Organic Dye and Chemiluminescence

Akshit Chhabra 24110026, Rayan Talukder 24110294, Rhythem Soni 24110296,
Rishi Soni 24110297, Roshia Shweta 24110304

***Chemiluminescence is the emission of light as a result of a chemical reaction with minimal heat generation. This phenomenon has significant applications in fields such as forensic science and bio-imaging. In this experiment, we explore chemiluminescence by synthesizing TCPO (1,2,3,4-Tetrachloro-6,7-dimethoxyisoquinoline) and observing its light-emitting reaction with hydrogen peroxide and a fluorescent dye, Diphenyl anthracene. The synthesis involves reacting 2,4,6-trichlorophenol with oxalyl chloride in the presence of triethylamine under anhydrous conditions. The resulting TCPO is then introduced into a solution containing Diphenyl anthracene and hydrogen peroxide in diethyl phthalate, initiating a reaction that produces a visible blue glow. This experiment demonstrates the principles of chemiluminescence and illustrates how chemical energy can be converted into visible light through a multi-step molecular energy transfer process.***

***Index Terms - Organic Synthesis, TCPO, Chemiluminescence, Fluorescence, Glow Stick***

## I. Introduction

Chemiluminescence is the process of emission of light which produces very less heat. In fluorescence and phosphorescence, light absorption and emission are separate processes, but chemiluminescence happens spontaneously when a chemical reaction releases energy in the form of visible light. It is also seen in fireflies and glow sticks. In this experiment, we will synthesize TCPO (1,2,3,4-Tetrachloro-6,7-dimethoxyisoquinoline) to study chemiluminescence. It produces light when mixed with hydrogen peroxide and a fluorescent dye, Diphenyl anthracene. In the reaction, hydrogen peroxide will break TCPO, which will generate an excited-state intermediate that will transfer energy to Diphenyl anthracene, which will glow it. The experiment contains two key steps:

1) Synthesis of TCPO - Reaction of 2,4,6-trichlorophenol with oxalyl chloride in the presence of triethylamine in a dry solvent.
2) Observation of Chemiluminescence - TCPO is introduced into a solution containing hydrogen peroxide and Diphenyl anthracene, which results in the emission of visible light.

This experiment helps us to understand how chemical reactions can produce light and tells us about the applications of chemiluminescence in various fields like forensic science and bio-imaging.

## II. Materials and Methods

### A. Materials Required

1) Test Tubes
2) Magnetic Bead
3) Rubber Septum
4) Two Metal Needles
5) 2,4,6-Trichlorophenol
6) Triethylamine
7) Dry Toluene
8) Oxalyl Chloride
9) Diphenyl Anthracene
10) Diethyl Phthalate
11) Hydrogen Peroxide
12) Methanol
13) Sodium Acetate
14) Balloons (filled with Nitrogen gas)

### B. Methods

1) **Part A - Synthesis of TCPO:**
   a) Place a 20 mL Test tube on a stirrer with a magnetic bead inside. Add 500mg (1 equivalent) of 2,4,6-trichlorophenol, cover with a septum, and purge with nitrogen gas.
   b) Dissolve the trichlorophenol in Dry Toluene using a metallic needle. Add 0.35mL (1 equivalent) of Triethylamine and stir for 5 minutes under nitrogen conditions.
   c) After stirring, add 0.1mL (0.5 equivalent) of Oxalyl chloride to the solution while in an ice bath. The solution will solidify.
   d) Remove the flask from the stirrer, vacuum filter the solution, and wash the solid with Methanol. The product, TCPO, will be a white powder.
2) **Part B - Let it glow:**
   a) Chemiluminescent Solution: Prepare a solution of Sodium acetate (25mg) and Diphenyl anthracene (25mg) (1:1) in Diethyl phthalate (2ml).
   b) Add 5 drops of Hydrogen peroxide to the solution.

c) Add a small amount of synthesized TCPO to the chemiluminescent solution. A blue chemiluminescent glow will be observed.
d) Observe the glow (chemiluminescence) in the dark.

## III. Results

First, we formed the TCPO (1,2,3,4-tetrachloro-6,7-dimethoxyisoquinoline) solution using the following reaction.

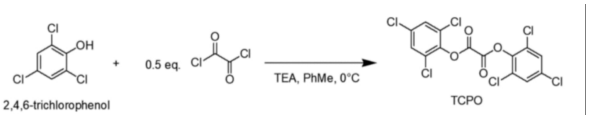


Fig. 1: Synthesis of TCPO.

Then, we reacted the formed TCPO with hydrogen peroxide and a high energy energy intermediate molecule was formed. We then use this energy from the molecule to light up our solution by using an organic dye, diphenyl anthracene.

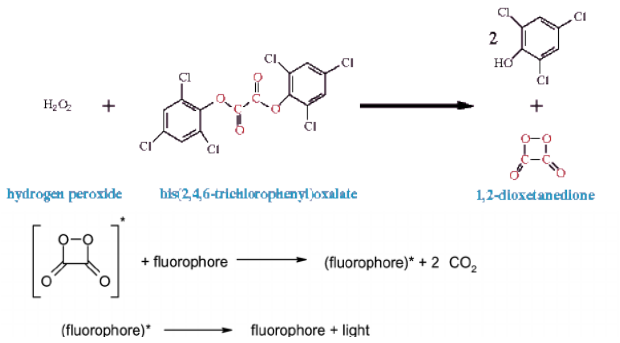


Fig. 2: Emission of light on reaction of TCPO with hydrogen peroxide and diphenyl anthracene.

Thus, we obtain a blue-colored glowing liquid. It glows due to the chemical reactions that take place inside and the glow is best viewed under dark conditions. Transfer of energy takes place between the reaction intermediates and the fluorescent compound which emits the observed light.

In this exothermic reaction, instead of releasing most of its energy as heat, it releases it in the form of visible light (chemiluminescence). There is still some minimal production of heat production, but it is negligible compared to the emitted light.

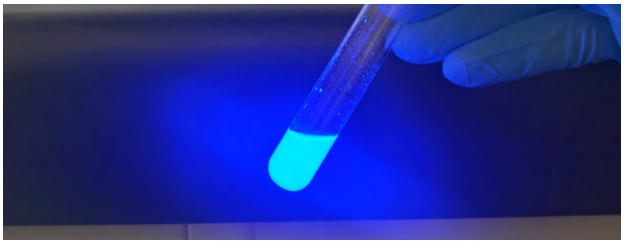

Fig. 3: Observed glow in front of a dark background.

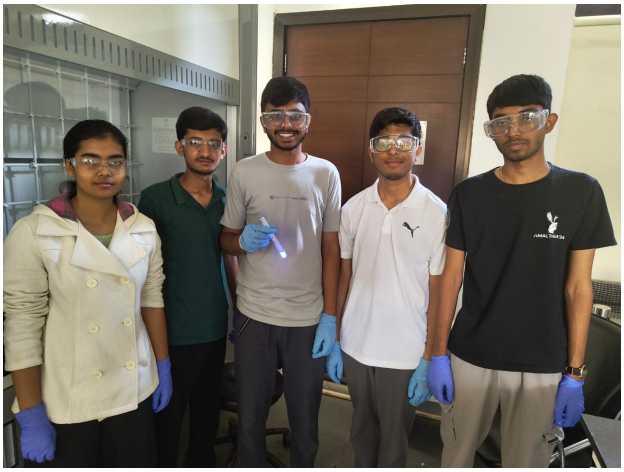

Fig. 4: Group photo with the glowing solution

## IV. Conclusion

In this experiment, we successfully synthesized TCPO (1,2,3,4-Tetrachloro-6,7-methoxy isoquinoline), a essential compound in chemiluminescent reactions, through the reaction of 2,4,6-trichlorophenol with oxalyl chloride under nitrogen atmosphere and dissolve it in dry toluene through metallic needle. The resulting product, TCPO, was isolated as a white powder after filtration and washing with methanol. This confirmed that the synthesis was successful.

Following the synthesis, we prepared the chemiluminescent solution by combining sodium acetate, diphenyl anthracene, and diethyl phthalate. The addition of hydrogen peroxide to this solution, along with the synthesized TCPO powder, resulted in the expected chemiluminescent glow, emitting blue light in the dark.The test tube emits a glow resembling that of a glow stick. This demonstrated the successful conversion of chemical energy into visible light through the process of chemiluminescence.

## V. Author Contributions

1) Akshit Chhabra, 24110026
   - Performed the experiment and assisted in synthesis of TCPO.
   - Completed the 'Abstract' and 'Author Contributions' sections and compiled, formatted the report on LaTeX.
2) Rayan Talukder, 24110294
   - Performed the experiment and assisted in synthesis of TCPO.
   - Completed the 'Results' section.
3) Rhythem Soni, 24110296
   - Performed the experiment and assisted in "let it glow".
   - Completed the 'Introduction' section and assisted in the 'Materials and Methods' section.
4) Rishi Soni, 24110297
   - Performed the experiment and assisted in synthesis of TCPO.

- Completed the 'Conclusion' section.

5) Roshia Shweta, 24110304
   - Performed the experiment and assisted in "let it glow".
   - Assisted in the 'Materials and Methods' section.

## REFERENCES

[1] BS 192 Chemistry Lab Manual