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# The Synthesis and Electropolymerization of Porphyrin-substituted Ethylenedioxythiophene (EDOT) Monomers

Benjamin D. McIntyre  
*Hope College*

Joshua A. Hawkins  
*Hope College*

Kenneth L. Brown  
*Hope College*

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# The Synthesis of Porphyrin-Based Redox Mediators for Sensor Applications

*Benjamin D. McIntyre, Joshua A. Hawkins, Dr. Kenneth L. Brown, Dr. Elizabeth M. Sanford\*,  
 Chemistry Department, Hope College, Holland, MI*

## Background

- The goal of this project is to make an electrochemical glucose sensor for diabetes monitoring applications.
- This sensor would be made from attaching the enzyme glucose oxidase (Gox) to the surface of an electrode.
  - As the enzyme oxidizes glucose, the electrode will detect the reaction and give a signal, allowing us to monitor glucose levels.
- However, a redox mediator is needed in between the enzyme and the electrode in order to facilitate the electron movement.

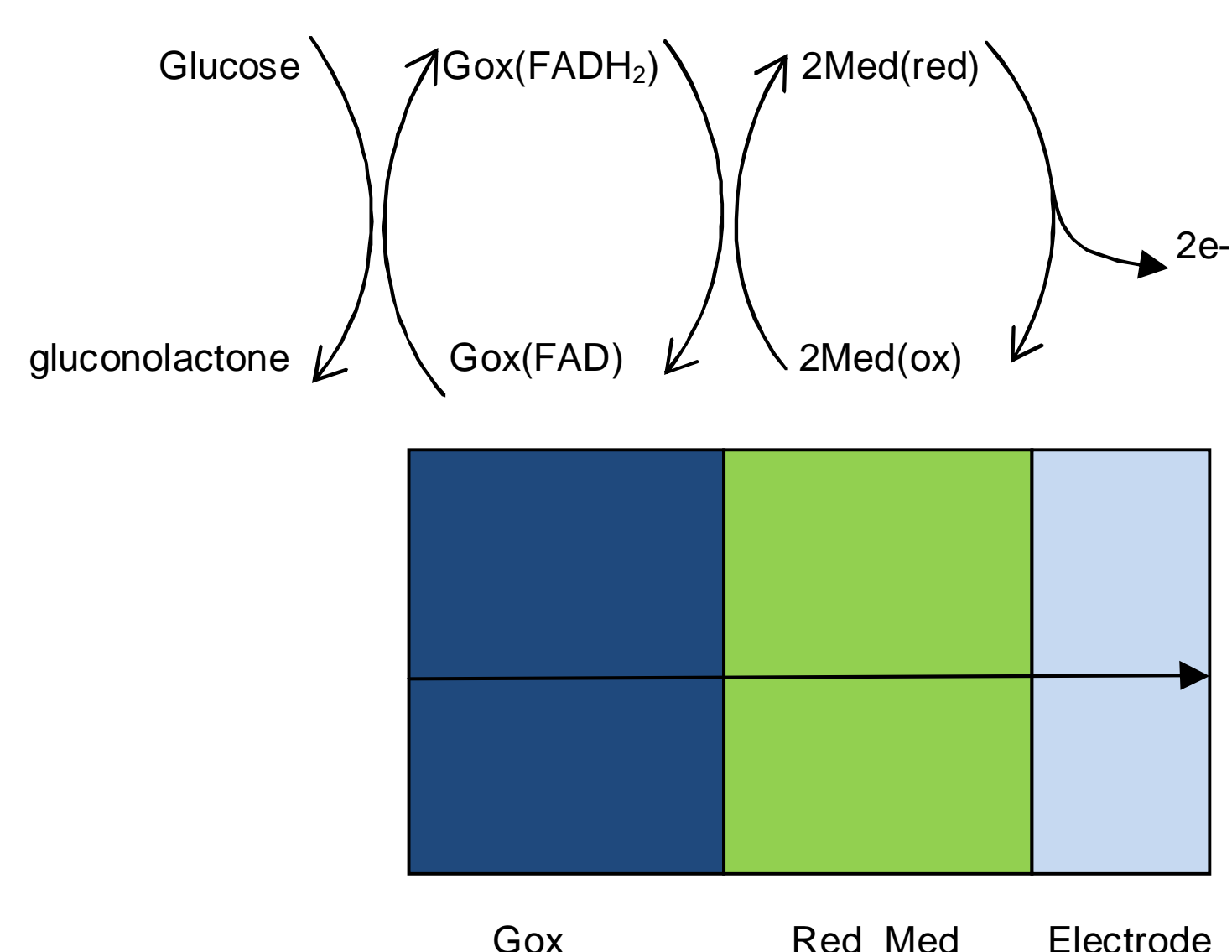


Figure 1. Components of an electrochemical glucose sensor (Gox = glucose oxidase, Red Med = redox mediator)

## Design Criteria

- The redox mediator must be composed of a monomer that can electropolymerize.
- It must form a conjugated polymer.
- And it must have side chain variability via the "R" group.

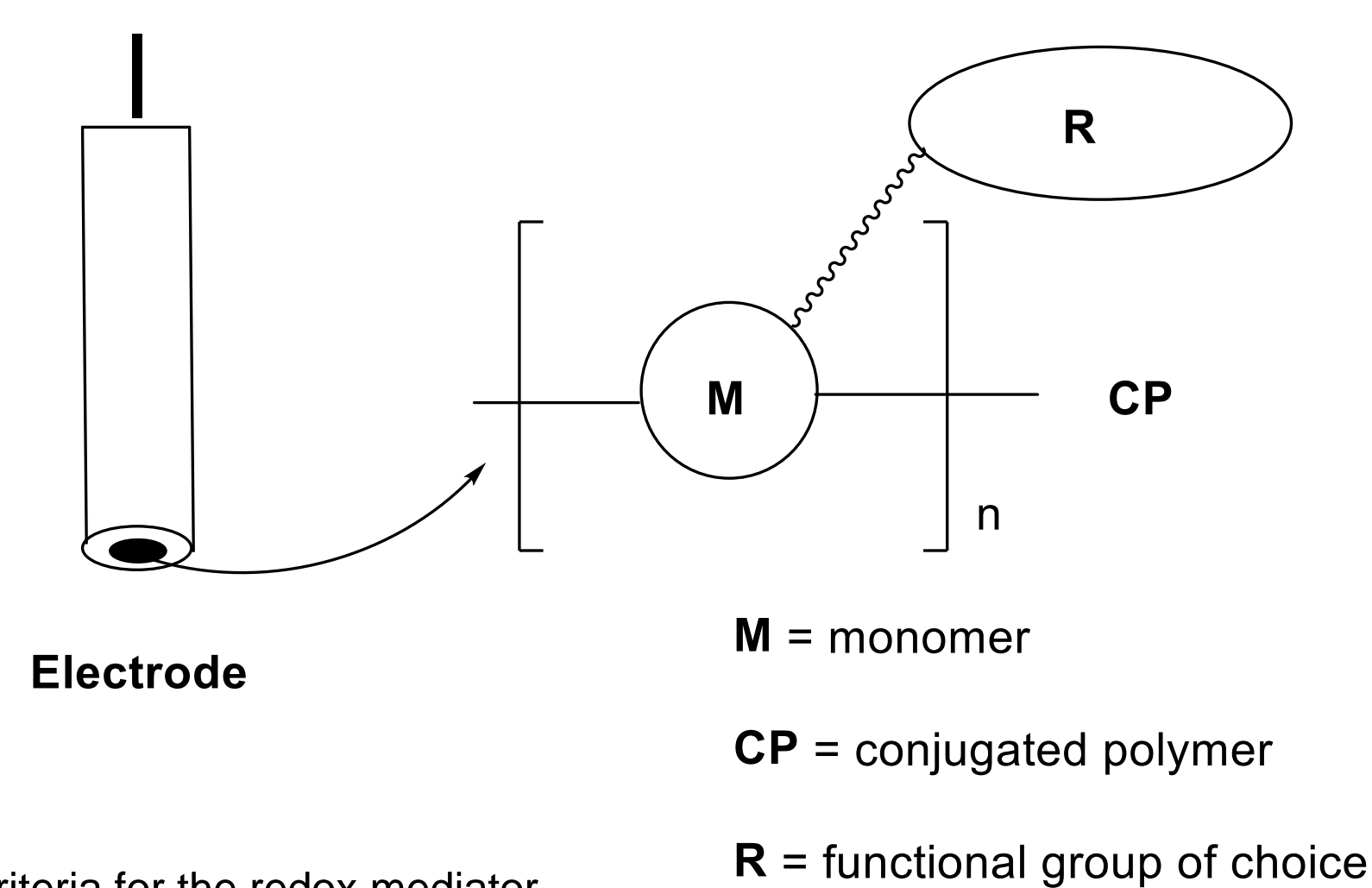


Figure 2. The basic design criteria for the redox mediator

- We chose to use ethylene dioxythiophene as the monomer.
  - It can electropolymerize and has good electronic properties.
- The "R" group will be an iron porphyrin.
  - This will function as a metal-based redox center.

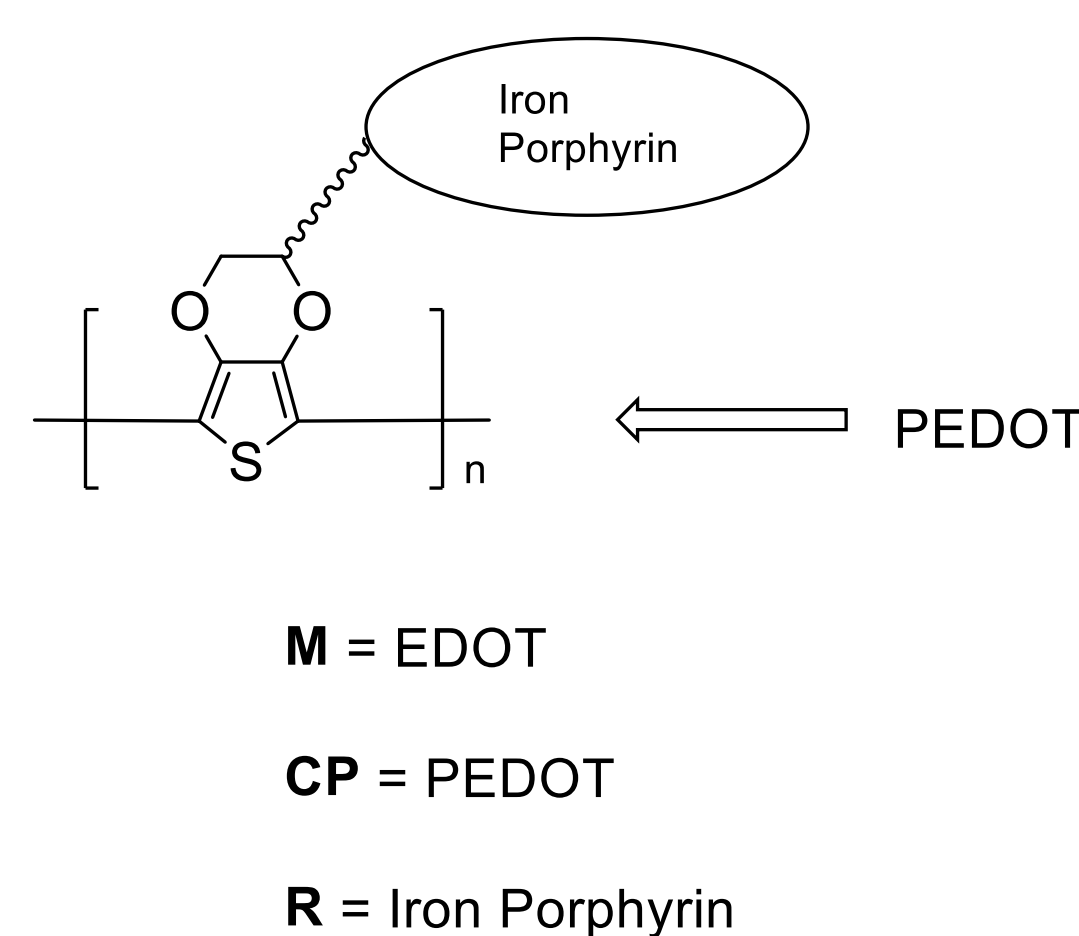


Figure 3. A diagram of the redox mediator design

## Synthesis of Fe EDOT-Substituted Porphyrin

We start with the tosylation of hydroxymethyl-EDOT (**1**) to give tosylated EDOT (**2**). We then react this product with 4-hydroxybenzaldehyde (**3**), which undergoes a Williamson ether synthesis, yielding EDOT aldehyde (**4**). A condensation reaction with pyrrole under acidic conditions will give the free porphyrin (**5**). After purification, a final metalation with FeCl<sub>2</sub> results in the desired FeEDOT-substituted porphyrin (**6**).

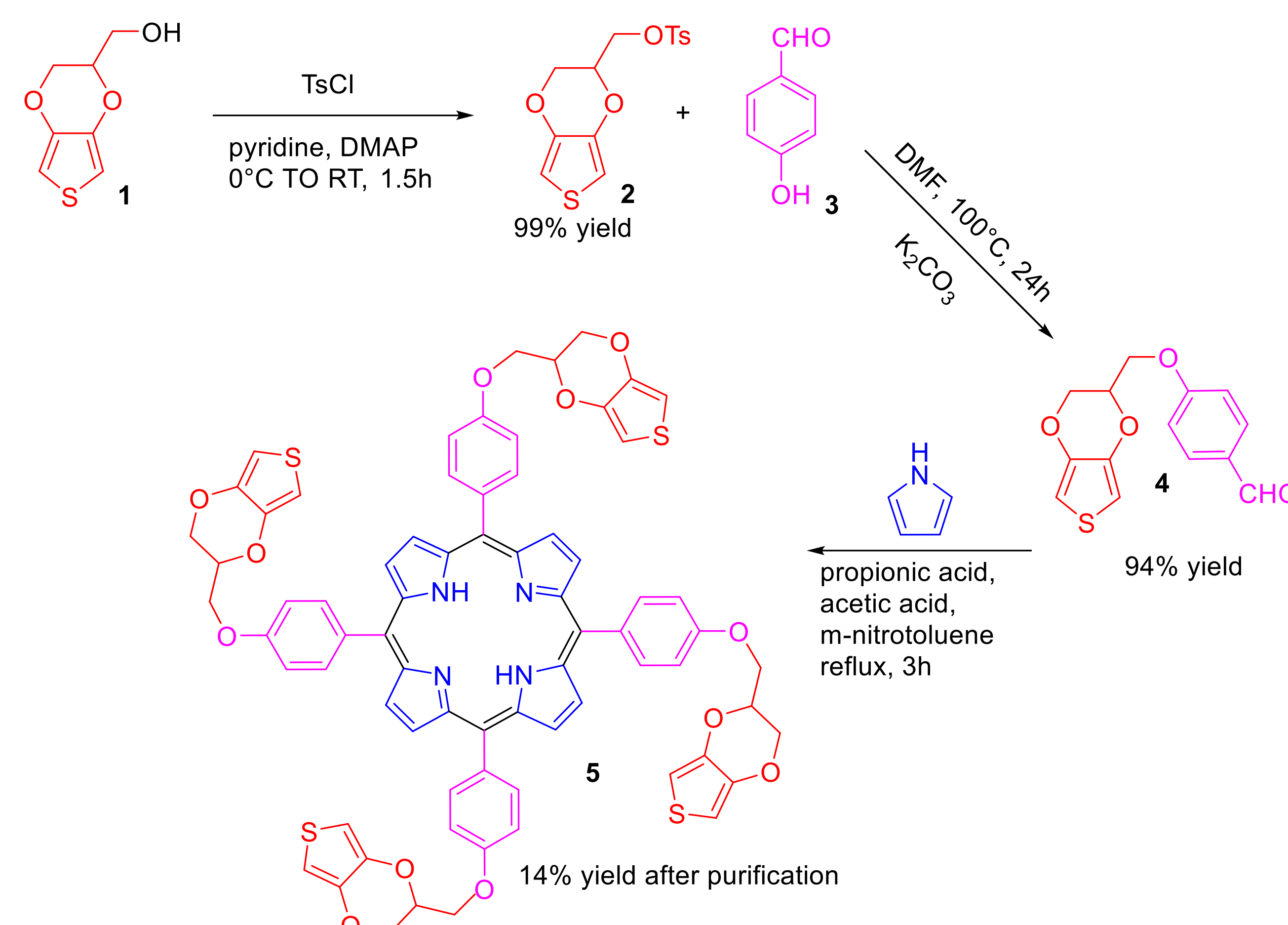


Figure 4. Synthesis of EDOT porphyrin

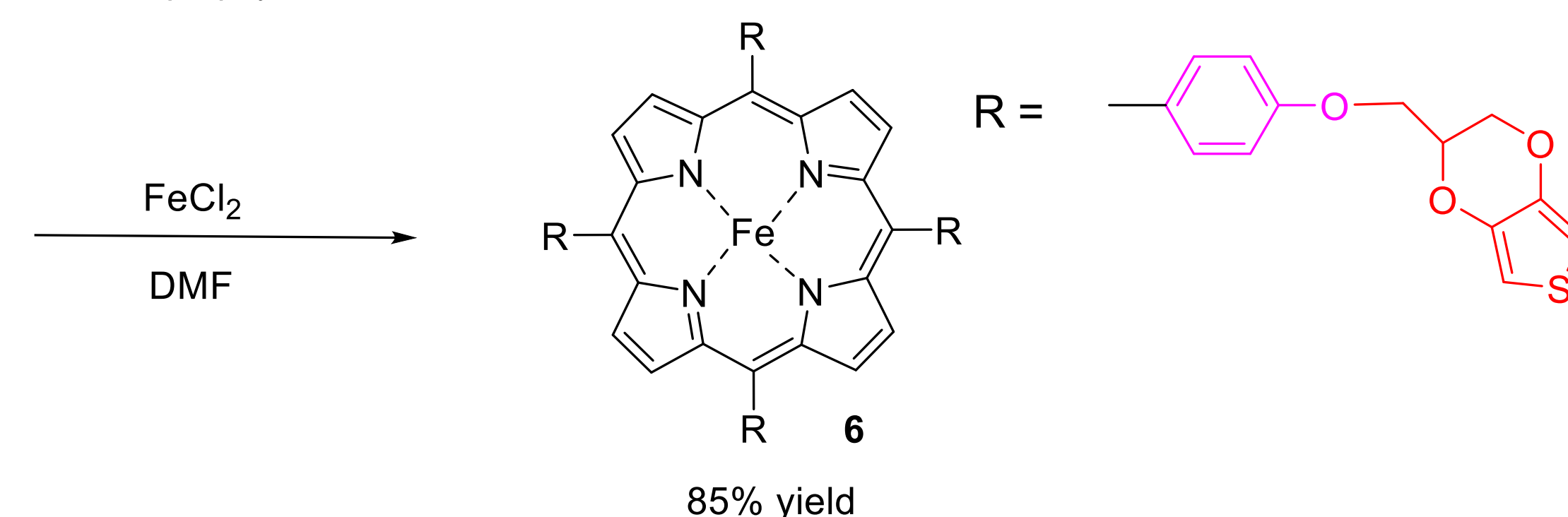
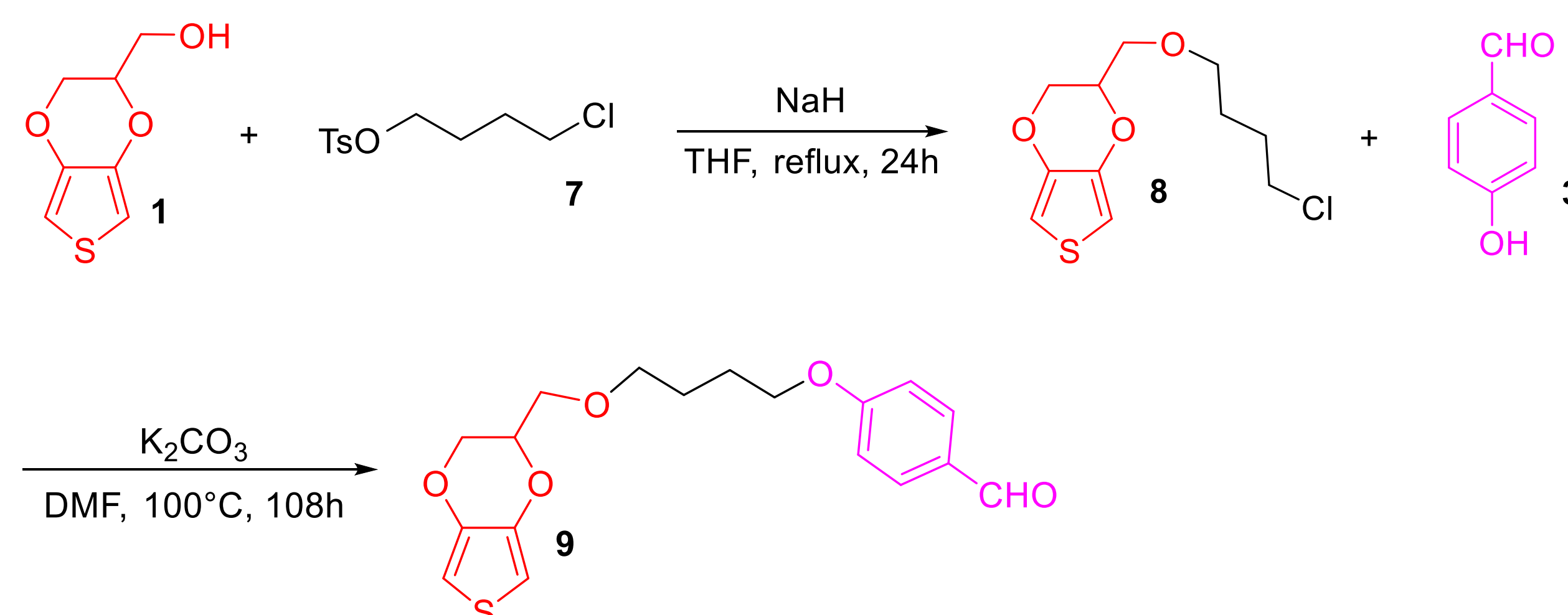


Figure 5. Metalation of EDOT porphyrin

We have recently started synthesizing long-chain (LC) porphyrin because it is less sterically congested than the short-chain porphyrin for electropolymerization. The LC linker (**7**) reacted with hydroxymethyl-EDOT (**1**) to give LC-tosylated EDOT (**8**). We then react this product with 4-hydroxybenzaldehyde (**3**), which undergoes a Williamson ether synthesis, yielding LC-EDOT aldehyde (**9**). We are currently working to perform a condensation reaction with pyrrole under acidic conditions to produce a free LC-porphyrin for future metalation.



## Cyclic Voltammetry

- With monomer **6** in hand, we use cyclic voltammetry to polymerize it onto the surface of a glassy carbon electrode.

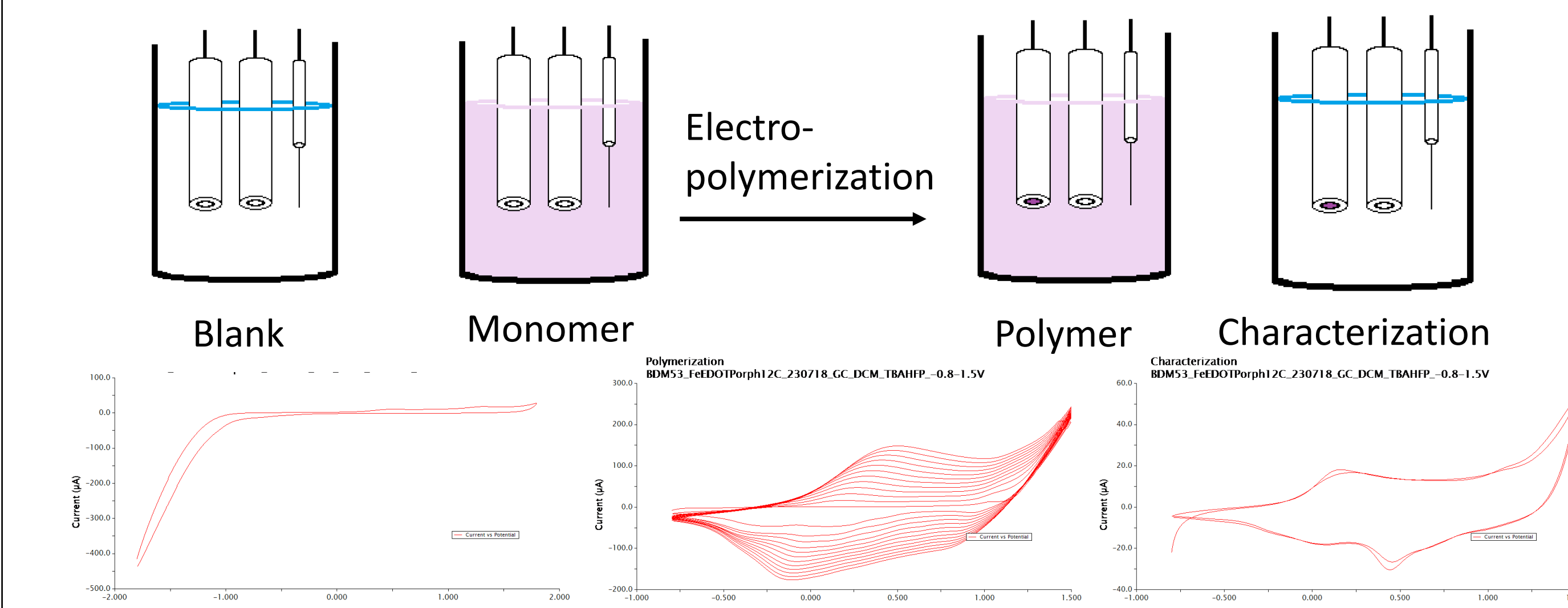


Figure 7. Diagram of a typical electropolymerization set up using cyclic voltammetry (simulated results)

- A glassy carbon working, platinum counter, and Ag/AgCl reference electrode are put into a blank solution and potential is cycled while the current is measured.
- If the blank is clean, the electrodes are put into a monomer solution and cycled 10-20 times, polymerizing the monomer directly onto the surface of the working electrode.
- A characterization is done to ensure successful polymerization of a redox active film.
- Once a successful polymerization on the electrode is achieved, we will focus on characterizing the film and then testing the redox mediation film for glucose detection.

## Conclusion

FeEDOT-substituted porphyrin (**6**) was synthesized. The polymerization of FeEDOT-substituted porphyrin (**6**) on a glassy carbon electrode is possible using cyclic voltammetry. However, polymerization does not consistently proceed with increasing current with each cycle, so we will have to continue to develop cyclic voltammeter parameters. Tetra substitution with EDOT causes significant steric congestion. Current exploration with LC-EDOT substituted porphyrin may ease this congestion and aid in electropolymerization.

## Acknowledgements

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