Synthesis of K27 Derivatives

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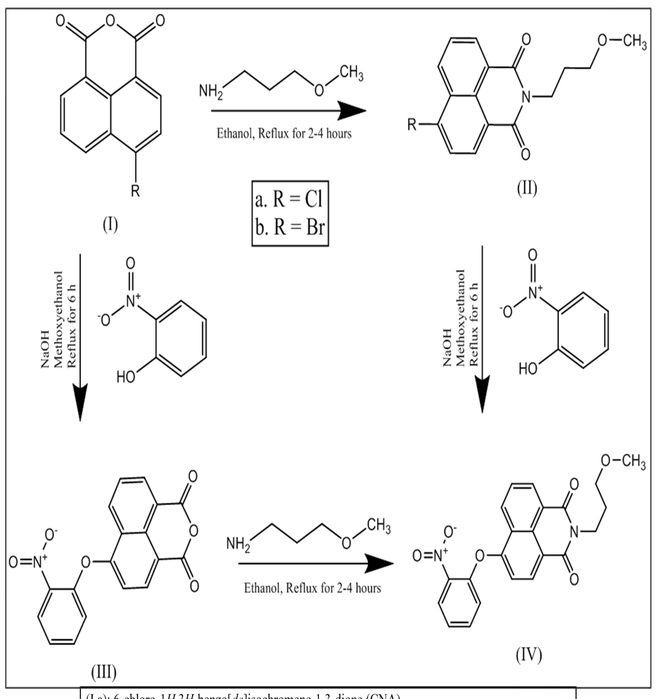
Introduction

Wavelength Shifting Optical (WLS) Fibers are an essential tool in plastic scintillation detectors. Plastic scintillators interact with ionizing radiation resulting in the emission of photons which are absorbed by the WLS fibers and re-emitted at longer wavelengths. This light is then converted to an electrical signal using a photodetector.

In this study, we researched the preparation of fluorescent compounds used in the green-emitting WLS fibers, namely K27 derivatives. Their syntheses require four separate steps to completion. We focused on the first two reactions as shown in Figure 1. Once synthesized, the compounds can be studied for future use and development of WLS fibers as well as scintillation detector production.



(I.a): 6-chloro-1*H*,3*H*-benzo[*de*]isochromene-1,3-dione (CNA)
(I.b): 6-bromo-1*H*,3*H*-benzo[*de*]isochromene-1,3-dione (BNA)
(II.a): 6-chloro-2-(3-methoxypropyl)-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione (CMD)
(II.b): 6-bromo-2-(3-methoxypropyl)-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione (BMD)
(III): 6-(2-nitrophenoxy)-1*H*,3*H*-benzo[*de*]isochromene-1,3-dione (NPNA)
(IV): 2-(3-methoxypropyl)-6-(2-nitrophenoxy)-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione (NPMD)

Figure 1: Scheme of reactions to synthesize NPMD (IV) from napthalic anhydrides (I).

Procedure of Condensation Reaction[1,2]

CNA (I.a) and 3-methoxypropylamine were stirred at reflux in ethanol for 2-6 hours (Fig. 2). After cooling, the precipitate was filtered and recrystallized with ethanol to yield pure CMD (II.a) crystals. A similar method would be used to synthesize BMD (II.b) from BNA (I.b) or NPMD (IV) from NPNA (III). Figures 3 and 4 show data gathered during the characterization process for product BMD (II.b).

Procedure of Nucleophilic Substitution[1,3]

2-Nitrophenol and NaOH were dissolved and stirred in hot methoxyethanol (MOE) to produce the nitrophenoxy ion. CNA (I.a) is dissolved in hot MOE and stirred with the ion. Solution is set to reflux for 6 h. After cooling, the formed precipitate was filtered and the reaction crude was extracted with ether. A similar method would be used to synthesize NPNA (III) from BNA (I.b) or NPMD (IV) from CMD (II.a) or BMD (II.b).

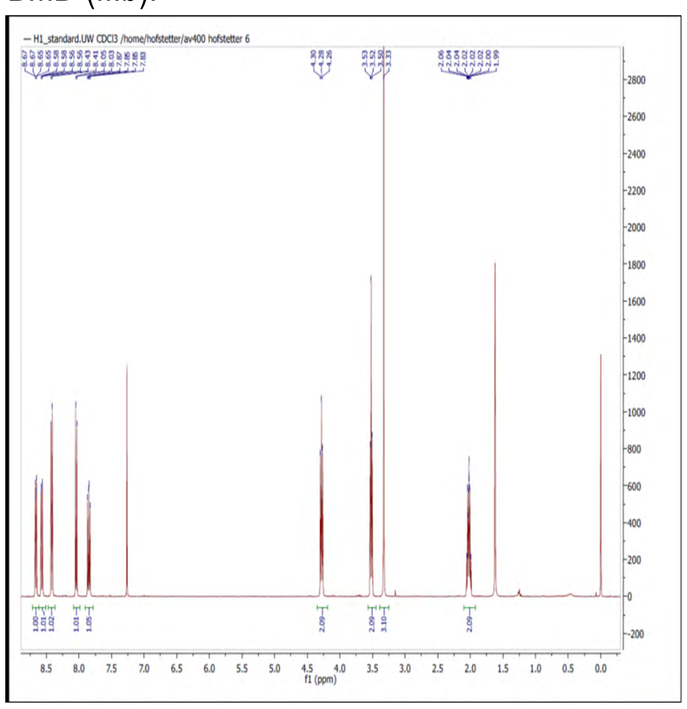


Fig. 3: Proton Nuclear Magnetic Resonance Spectrum of Synthesized BMD (II.b)

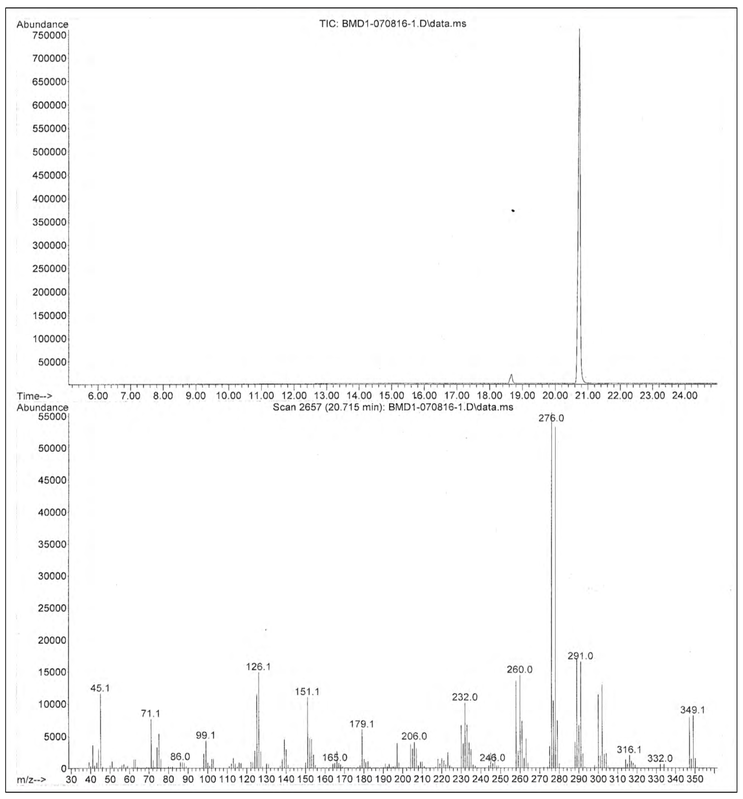


Fig. 4: Gas Chromatogram and Mass Spectrum of Synthesized BMD (II.b)

Product	Starting Compound	Yield	Melting Point of Product (°C)
CMD	CNA	>51%	82-84
BMD	BNA	>41%	82-86
NPMD	BMD	No Yield	N/A
NPNA	CNA or BNA	No Yield	N/A

Conclusions and Future Work

The condensation reaction to synthesize (II.a) and (II.b) is successful with very high purity and a fair yield (>40%).

Despite previous work supporting the nucleophilic substitution reaction, the starting material does not react. We assume this is due to the solvent, methoxyethanol, which is different from the previous studies of the substitution reactions.

In the future, more solvents will be studied for compatibility with the nucleophilic substitution reaction. Work will continue to synthesize a K27 derivative (Fig. 5). Once achieved, the compound will be studied to improve scintillator detectors and their development.

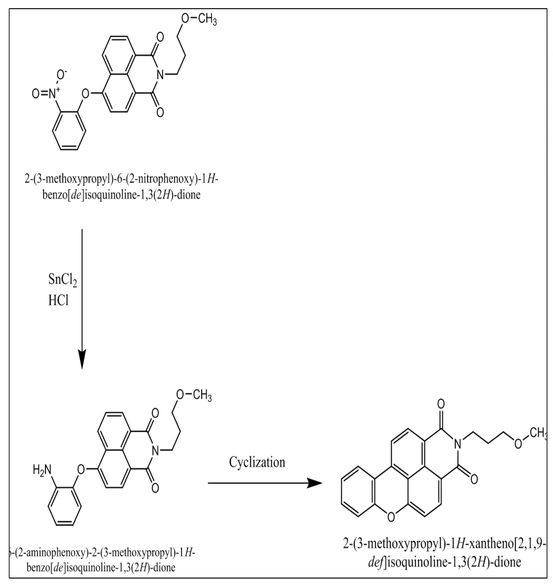


Fig. 5: Scheme showing remaining reaction steps to produce a K27 derivative from NPMD (IV).

References

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