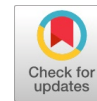




A Green Approach: Eosin Y-Facilitated Visible-Light Preparation of a Functionalized 1,2,3-Triazole Compound

Singh Disha, Srivastava Nidhi



Abstract: A key goal of contemporary organic chemistry is developing environmentally acceptable, sustainable methods for synthesising nitrogen-containing heterocycles. Because of their broad biological activities and uses in medicinal chemistry, 1,2,3-triazoles have garnered considerable attention among these heterocycles. In this study, we devised a visible-light-induced process for synthesising (4-hexyl-2H-1,2,3-triazole-2-yl) methanol using Eosin Y as an organic photocatalyst. The reaction is a three-component coupling of formaldehyde, sodium azide, and terminal alkynes that occurs under visible light at ambient temperature. Eosin Y (5 mol%) in an H₂O/DMSO (1:1) solvent system under blue LED irradiation yields the required triazole derivatives in good yield when reaction parameters are optimised. Control studies validated the crucial roles of visible light and the photocatalyst in the reaction. Without a transition-metal catalyst, the transformation proceeds under mild conditions, demonstrating the protocol's environmental friendliness. We propose a viable photoredox process that generates reactive intermediates and activates Eosin Y under visible light. This process offers a straightforward, effective, and environmentally friendly way to create functionalized 1,2,3-triazole derivatives.

Keywords: Eosin Y, Heterocyclic Compounds, Visible Light Photo-Redox Catalyst, Green Synthesis, and 1,2,3-Triazoles.

Nomenclature:

CuAAC: Catalysed Azide-Alkyne Cycloaddition

DMSO: Dimethyl Sulfoxide

THF: Tetrahydrofuran

TLC: Thin-Layer Chromatography

PDA: Potato Dextrose Agar

I. INTRODUCTION

Pharmaceuticals, agrochemicals, and functional materials all contain nitrogen-containing heterocycles, a significant class of organic compounds. [1,2]. Among these heterocyclic frameworks, 1,2,3-triazole derivatives have attracted significant interest due to their various pharmacological and biological characteristics, including antifungal, antibacterial, antiviral, and anticancer activities [3,4,5]. In addition to them

broad biological relevance, triazole moieties exhibit high chemical stability, strong dipole moments, and the capacity to engage in hydrogen-bonding interactions, making them valuable scaffolds in medicinal chemistry and drug design [6,7]. Owing to these unique structural and biological characteristics, triazole-containing compounds have emerged as important synthetic targets in contemporary organic and medicinal chemistry.

The thermal Huisgen 1,3-dipolar cycloaddition process between organic azides and alkynes has traditionally produced 1,2,3-triazoles. However, the conventional thermal Huisgen cycloaddition typically requires high temperatures and long reaction times and often yields a mixture of 1,4- and 1,5-disubstituted regioisomers. [8,9,10]. The discovery and development of copper(I)-catalysed azide-alkyne cycloaddition (CuAAC), which enables the formation of 1,4-disubstituted 1,2,3-triazoles under relatively mild conditions, significantly changed triazole synthesis. CuAAC reactions are generally performed at or near room temperature in aqueous or mixed green solvents and often provide excellent yields with minimal by-products [11,12]. As a result, CuAAC has emerged as one of the most potent and often used transformations in heterocyclic synthesis and click chemistry.

Despite these advantages, using copper and other transition-metal catalysts in triazole synthesis raises concerns about metal contamination, catalyst toxicity, environmental impact, and tedious purification procedures, particularly in pharmaceutical and biologically relevant applications. In this regard, prior research has documented the synthesis of hydroxymethyl-substituted 1,2,3-triazoles via multicomponent reactions involving aldehydes, sodium azide, and terminal alkynes. [13,14]. For example, Kaliski and colleagues reported a three-component, one-pot method for producing N-hydroxymethyl-1,2,3-triazoles under mild conditions using terminal alkynes, sodium azide, and formaldehyde in the presence of CuSO₄ and sodium ascorbate. Similar CuSO₄/ascorbate systems and related Cu(I/II)-catalysed methodologies have subsequently been applied to a variety of terminal alkynes to prepare functionalized triazole derivatives. Although these methods demonstrate high efficiency and operational simplicity, they still rely on transition-metal catalysts and reducing agents, which may raise environmental concerns and necessitate additional purification steps to remove residual metals [15,16,17].

In recent years, visible-light photoredox catalysis has gained popularity as a

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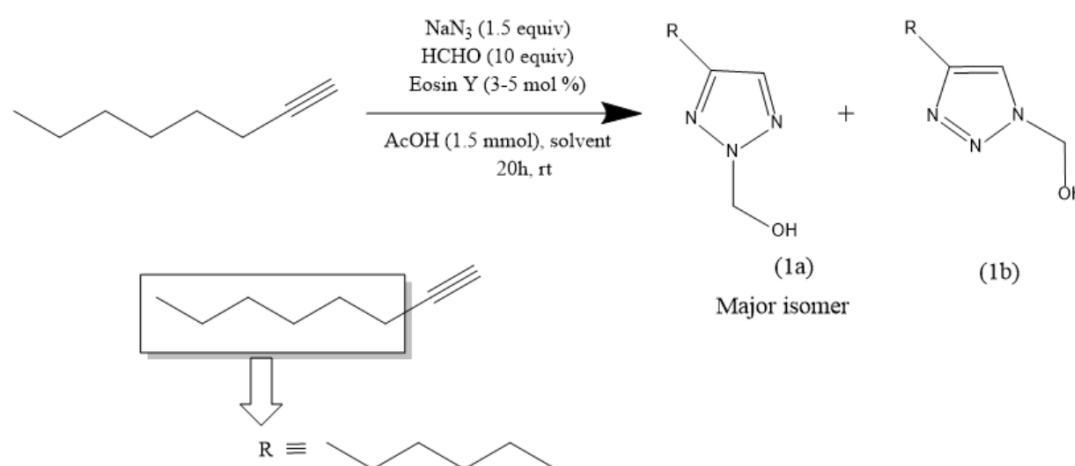
sustainable technique for a range of organic transformations. [18,19,20].

Photochemical reactions driven by visible light offer several advantages, including mild reaction conditions, reduced energy consumption, improved atom economy, and enhanced environmental compatibility. Among the various photocatalysts employed in visible-light-mediated synthesis, organic dyes such as Eosin Y have attracted considerable attention for their low toxicity, ready availability, cost-effectiveness, and excellent photophysical properties [21,22,23]. Furthermore, visible-light photoredox catalysis has rapidly emerged as a potent tool for synthesising heterocyclic compounds, enabling the formation of nitrogen-containing frameworks via radical pathways under remarkably mild conditions. [24,25,26,27,28]. These methodologies align strongly with the principles of green chemistry, which emphasise sustainable synthetic routes,

environmentally benign reaction conditions, and reduced chemical waste generation [29,30,31].

Therefore, in modern organic synthesis, the development of metal-free, environmentally safe processes for synthesising triazole derivatives has received increasing attention. [32,33,34]. Using Eosin Y as an organic photocatalyst, we report the visible-light-induced synthesis of (4-hexyl-2H-1,2,3-triazol-2-yl) methanol (1a) from terminal alkynes, formaldehyde, and sodium azide. This reaction enables the synthesis of functionalized triazole derivatives with potential biological significance in an environmentally friendly, straightforward manner, proceeding effectively under mild conditions at room temperature. The present methodology is also consistent with recently reported Eosin Y-catalysed metal-free syntheses of heterocyclic compounds under visible-light irradiation [35,36,37,38].

A. Reaction Scheme



Scheme 1. Visible-Light-Induced Synthesis of Compound 1a

Upon exposure to visible light, terminal alkyne (R-C≡CH) reacts with formaldehyde and sodium azide (NaN₃) in the presence of Eosin Y, an organic photocatalyst, to give compound 1a.

B. Reaction Conditions

Terminal alkyne (1.0 equiv), NaN₃ (1.5 equiv), formaldehyde (10 equiv), acetic acid (1.5 equiv), Eosin Y (5 mol%), solvent (H₂O/DMSO, 1:1), exposure to visible light, room temperature, 18–24 hours.

II. THE EXPERIMENTAL PART

A. Materials

Unless otherwise specified, we used all analytical-grade reagents and solvents as received from commercial suppliers without further purification. We obtained terminal alkynes, sodium azide (NaN₃), formaldehyde solution (37% in water), Eosin Y, and acetic acid from commercial chemical suppliers. We used dimethyl sulfoxide (DMSO), tetrahydrofuran (THF), and distilled water as solvents.

Before use, all reaction glassware was oven-dried. Unless otherwise noted, we conducted the reactions under ambient conditions.

B. General Procedure for Visible Light-Induced Synthesis of Compound 1a

We added terminal alkyne (1 mmol) and sodium azide (1.5 mmol) to a 25 mL round-bottom flask equipped with a magnetic stir bar. The reaction mixture was then supplemented with formaldehyde solution (37% in water, 10 mmol) and acetic acid (1.5 mmol).

Eosin Y (5 mol%) was added as an organic photocatalyst, and the reaction mixture was stirred in H₂O/DMSO (1:1, 6 mL) under blue LED irradiation at room temperature.

A blue LED source (450 nm) was used to magnetically swirl the reaction mixture while it was exposed to visible light at room temperature. Thin-layer chromatography (TLC) was used to monitor the reaction progress periodically.

After stirring the reaction mixture for 18 to 24 hours and exposing it to visible light, TLC analysis showed that the reaction was complete, and the desired hydroxymethyl-substituted 1,2,3-triazole derivative formed under optimal reaction conditions.

C. Monitoring of Reaction Progress





Thin-layer chromatography (TLC) on silica gel plates was utilised to monitor the reaction's progress using an appropriate solvent system as the mobile phase.

Under UV light, the TLC patches were visible. The reaction was proven complete when the starting materials disappeared, and a new product matching the target triazole derivative appeared.

D. Purification

A rotary evaporator was used to remove the solvent under reduced pressure after the reaction was complete. Ethyl acetate was used to extract the reaction mixture after dilution with water. The mixed organic layers were filtered, concentrated under reduced pressure, and dried over anhydrous sodium sulphate. Using a suitable hexane/ethyl acetate mixture as the eluent, column chromatography on silica gel afforded the desired hydroxymethyl-substituted 1,2,3-triazole derivative in good isolated yield.

E. Characterization Data

The synthesised compound 1a was characterised using standard spectroscopic techniques, including ^1H NMR, ^{13}C NMR, and IR spectroscopy. ^1H NMR, ^{13}C NMR and IR spectroscopy confirmed the structure of compound 1a. The obtained spectral data were consistent with the proposed structure.

Characteristic absorption bands in the IR spectra and corresponding ^1H and ^{13}C NMR signals confirmed successful production of the intended product.

Table-I: Reaction Parameter Optimization for Compound 1a Synthesis under Visible Light Irradiation

S.No.	Photocatalytic Condition	Reaction in Solvent	LED Light	Time in hour	Product Yield (%)
1	None	H ₂ O/DMSO	Blue LED	24	Trace
2	Eosin Y (4 mol%)	H ₂ O/DMSO (1:1)	Blue LED	24	85
3	Rose Bengal	H ₂ O/DMSO	Blue LED	24	63
4	Eosin Y	H ₂ O/DMSO	Green LED	24	70
5	Eosin Y	H ₂ O/DMSO	Dark	24	Trace
6	Eosin Y (3 mol%)	H ₂ O/DMSO	Blue LED	24	72
7	Eosin Y (4 mol%)	H ₂ O/DMSO	Blue LED	18	80
8	Eosin Y (4 mol%)	H ₂ O	Blue LED	24	42
9	Eosin Y (4 mol%)	MeCN	Blue LED	24	55
10	Eosin Y (5 mol%)	THF	Blue LED	24	61

Reaction optimization for the synthesis of compound 1a from terminal alkyne, sodium azide and formaldehyde under visible-light irradiation.

III. ANTIFUNGAL SCREENING PROCEDURE

Using the poisoned food approach on potato dextrose agar (PDA) medium, we evaluated the antifungal activity of compound 1a against *Aspergillus niger*. We dissolved

compound 1a in a 2:8 (v/v) acetone–water mixture and evaluated it at different concentrations (1000, 100, and 10 ppm).

We added PDA medium containing the test chemicals to pre-sterilised Petri dishes. A 5 mm fungal disk from the edge of a seven-day-old *Aspergillus niger* culture was used to inoculate each Petri plate. The plates were then incubated for 96 hours at $28 \pm 1^\circ\text{C}$.

Antifungal activity was evaluated by measuring the fungal colony's radial growth. The percentage suppression of fungal growth was calculated using the following formula:

$$\% \text{ Inhibition} = \frac{C - T}{C} \times 100$$

where T is the fungal growth in the treated plate, and C is the fungal growth in the control plate.

[39,40,41]

IV. RESULTS AND DISCUSSION

We first studied the reaction involving terminal alkynes, sodium azide, and formaldehyde under visible-light irradiation to develop an effective methodology for synthesising compound 1a. Table I summarises the outcomes of adjusting the photocatalyst, solvent system, photocatalyst loading, and light source to optimise reaction conditions.

In the absence of a photocatalyst, we initially carried out the reaction in an H₂O/DMSO solvent mixture under blue LED irradiation. Under these conditions, only a tiny amount of the intended product was observed, suggesting that the transformation requires a photocatalyst. To improve reaction efficiency, we examined various photocatalysts. Of all the photocatalysts tested, Eosin Y was the most efficient and afforded the highest yield of compound 1a, which was obtained as the major regioisomer under the optimised reaction conditions.

Additionally, we examined the impact of the solvent system on the reaction outcome. When the reaction was carried out in water alone, it gave a moderate product yield. Among the solvent systems examined, H₂O/DMSO (1:1) provided the highest yield (85%). In contrast, acetonitrile and tetrahydrofuran afforded comparatively lower yields. This mixed solvent system likely improves the solubility of both organic and inorganic reaction components, facilitating efficient interaction between the reactants under visible-light irradiation.

By reducing the Eosin Y concentration from 5 mol% to 3 mol%, we further investigated the effect of photocatalyst loading. We observed a clear drop in product yield, suggesting that 5 mol% Eosin Y is optimal for effective reaction progress. Furthermore, we assessed the impact of various light sources. Green LED irradiation produced lower efficiency, while blue LED irradiation produced the highest output yield. Crucially, we observed only trace product when the reaction was conducted in the absence of light. These findings unequivocally demonstrate the importance of visible-light irradiation in accelerating the process.

Under ideal conditions, the reaction proceeded smoothly



and afforded compound 1a in excellent isolated yield, using Eosin Y (5 mol%), H₂O/DMSO (1:1) as the solvent system, and blue LED irradiation at room temperature. The methodology proved environmentally benign, efficient, and operationally straightforward, aligning strongly with green chemistry principles.

¹H NMR, ¹³C NMR, and IR spectroscopy were employed to characterize compound 1a. The obtained spectroscopic data were consistent with the proposed structure.

Eosin Y is activated by visible light to produce an excited-state photocatalyst that can stimulate radical production, a probable reaction mechanism. The excited photocatalyst facilitates the formation of reactive intermediates from sodium azide and terminal alkynes, which then undergo cycloaddition to form the triazole ring. Compound 1a forms in the presence of formaldehyde.

Overall, this visible-light-mediated approach offers a productive and sustainable pathway for synthesising functionalized triazole compounds. This approach is especially appealing for synthesising clinically significant heterocyclic compounds, as it avoids transition-metal catalysts, uses an organic photocatalyst such as Eosin Y, and enables mild reaction conditions.

V. CONCLUSION

In conclusion, we developed a visible-light-induced method to efficiently synthesise compound 1a from terminal alkynes, sodium azide, and formaldehyde using Eosin Y as an organic photocatalyst. The reaction proceeded smoothly under mild conditions at room temperature with visible-light irradiation, without transition-metal catalysts. Optimisation studies showed that Eosin Y (5 mol%) in H₂O/DMSO (1:1) under blue LED irradiation gave the highest yield of compound 1a.

¹H NMR, ¹³C NMR, and IR spectroscopic analyses confirmed the structure of compound 1a. Operational simplicity, mild reaction conditions, good isolated yields, and environmentally benign reaction conditions are just a few benefits of the established technology. Additionally, the suggested photoredox mechanism implies that photoexcitation of Eosin Y initiates a radical pathway that leads to the transformation.

Overall, the developed protocol provides a practical, environmentally friendly, and sustainable approach to synthesising functionalized 1,2,3-triazole derivatives with potential applications in medicinal chemistry and the development of bioactive heterocyclic compounds. Future studies may expand the substrate scope and evaluate the biological activities of the synthesised derivatives.

DECLARATION STATEMENT

As the article's author, I must verify the accuracy of the following information after aggregating input from all authors.

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- **Funding Support:** This article has not been funded by any organizations or agencies. This independence

ensures that the research is conducted objectively and without external influence.

- **Ethical Approval and Consent to Participate:** The content of this article does not necessitate ethical approval or consent to participate, with supporting documentation.
- **Data Access Statement and Material Availability:** The data and materials in this article are publicly accessible.
- **Author's Contributions:** The authorship of this article is contributed equally to all participating individuals.

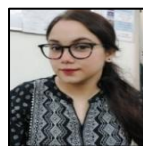
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