



Synthesis and Characterization of New 1,2,4-Triazole Derivative Form Methylbenzoate with Sulfur as Bridge Links

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Schiff base, triazole derivatives, benzylidene amine, hydrazide.

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ABSTRACT

The basic nucleus 4-(benzylideneamino-5-phenyl-4*H*-1,2,4-triazol-3-yl)-2-(4-benzylideneamino-5-phenyl-4*H*-1,2,4-triazol-3-yl)-thioethanethioate T.4 was produced using various steps, from the cyclization of T.2 with carbon disulfide/potassium hydroxide using ethanol as a solvent followed by the addition of hydrazine hydrate for 12 h while experiencing reflux. To create the Schiff base T.3, we exposed its successful synthesis via reflux T.2 with 1,4-dicarbalddehyde and reacted T.3 with chemicals for condensation with chloroacetyl chloride. Physical characteristics, such as the melting point, and spectroscopic analysis was used to confirm the compounds (Fourier transform infrared spectroscopy and proton nuclear magnetic resonance).

Introduction

Hugo Schiff, a German scientist, created Schiff bases (imines) for the first time in 1869. Schiff bases (imines) are typically derived from primary amines and carbonyl substances (aldehydes or ketones) [1]. An imine or ketimine is the term for the bond created when a ketone reacts with it, and the connection created by an aldehyde reaction is known as an azomethine or an aldimine. The general formula when R and R1 is alkyl is $RCH = NR$. However, alkyl substituents can be observed instead [2]. Given their extremely diverse structural characteristics, several imines have been studied and used as chelating ligands in coordination chemistry [3-7]. The availability of Schiff bases in numerous fields, including biological systems, medicine, and new technologies, may be credited for this interest [8-10].

In addition, imines are important starting materials and serve as versatile intermediates for the synthesis of numerous reactions, including Mannich bases [11, 12], indoles [13, 14], β -lactam [15, 16], and pyrimidine derivatives [17].

Given their biological activity, compounds created from the reactions involving Schiff bases have been applied to several types of illnesses [18]. The synthesis of imines has enabled the utilization of a variety of chemicals, including Lewis's acids [19-22], metal complex [23], circumstances without metals [24], [25], those promoted by radiation from microwaves [26], and ultrasonic radiation [27]. Triazoles are heterocyclic organic compounds and comprise rings consisting of five members and three nitrogen atoms. Two different types of triazoles, namely, 1,2,3- and 1,2,4-triazoles, exist, and their chemistry has been extensively studied in literature [28, 29]. Given their practical use in medicine, the chemistry of triazole derivatives has garnered interest [30] in agriculture and industry [31]. In addition, a portion of these triazoles is used as reagents for analysis [32] and in the creation of polymers, dyes, photographic chemicals, and other substances [33]. Bladin created the first 1,2,4-triazole derivative in 1885. Studies reported the combination of numerous triazole derivatives [34]. Alkinson and Polya created 1,3-diphenyl-1,2,4-triazole. Klingsberg prepared triaryl-s-triazoles from

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diaroylhydrazines. Kurzer and Canelle created three-amino-5-mercapto-1,2,4-triazoles with four substituents [28], who cited the production of 3-(1,2,4-triazole-4-yl)-5-amino 1,2,4-triazole [35]. Reimlinger Peiren and colleagues investigated the synthesis and characterization of four isomeric oxodihydro s-triazolo pyrimidines. [36]. Moreover, numerous workers have reported the synthesis of various novel triazoles [37-39]. Molina *et al.* documented the production and certain responsiveness of mesoionic 1,2,4-triazolo-[4,3-b]-1,2,4-triazole compounds in some cases [28]. 1,5-Diaryl-3-alkylthio-1H-1,2,4-triazoles was recently discovered, and the corresponding sulfoxides and sulfones have been prepared [40]. Reid and Heindal reacted aryl acid hydrazide with CS₂/KOH and hydrazine hydrate to create triazoles [41].

In this study, an effective multistep synthesis was used to create Schiff bases with a 1,2,4-triazole motif. Various spectroscopic methods (Fourier transform infrared spectroscopy (FT-IR)) and (proton nuclear magnetic resonance (¹H-NMR)) were utilized to verify the formation of the specified Schiff base ligands.

Materials and Methods

Scheme 1 depicts the synthesis method considered in this work. Without any additional purification, all the reagents, starting ingredients, and solvents were obtained from commercial sources. Melting point values were obtained from open glass capillaries. The absorption infrared spectra were obtained using dimethyl sulfoxide (DMS). DMS served as the solvent and internal standard, and an NMR spectrophotometer Hitachi R-600 (60 MHz) was used to determine the ¹HNMR spectra. For the recording, an FT-IR Shimadzu 8300 spectrophotometer was utilized.

Synthesis of Benzoic acid hydrazide (T.1)

A mixture containing methyl benzoate (0.079 mol., 10 mL) and hydrazine hydrate (5 mL) was refluxed for 4 h. Then, 8 mL ethanol were added, and the mixture was refluxed for 1 h. After cooling, the product was filtered out, cleaned with ether, and allowed to dry in preparation for the next stage [41].

FT-IR: NH₂ stretching at 3498 cm⁻¹, C=O of amide at 1690 cm⁻¹, stretching of C-H of aromatic ring at 3055 cm⁻¹, and C=C of aromatic ring at 1469 cm⁻¹.

Synthesis of 4-amino-5-phenyl-4H-1,2,4-triazole-3-thiole (T.2)

Benzohydrazide (3.4 g, 0.024 mol) was eliminated in 30 mL 100% ethanol and potassium hydroxide (2 g, 0.035 mol), and the mixture was stirred for 15 min. After the addition of a tiny amount of carbon disulfide (2 mL, 0.033 mol), the mixture was agitated for a whole day to achieve homogeneity. Then, 10 mL hydrazine hydrate was added, the mixture was refluxed with stirring, and potassium xanthate product was added. These conditions resulted in a homogeneous solution and hydrogen sulfide evolution (paper acetate of lead). The reaction ended when hydrogen sulfide evolution stopped. The reaction mixture was filtered, added with strong hydrochloric acid, and diluted with 10 mL cold water, which produced a white precipitate [41].

FT-IR measurements included S-H, 2376 cm⁻¹, C-H stretching of the aromatic ring 3002 cm⁻¹, C=C of the aromatic ring 1498 cm⁻¹, and N-H stretching 3338 cm⁻¹.

Synthesis of 4-benzylideneamino-5-phenyl-4H-1,2,4-triazole-3-thiol-4-benzylideneamino-5-phenyl-4H-1,2,4-triazole-3-thiol (T.3)

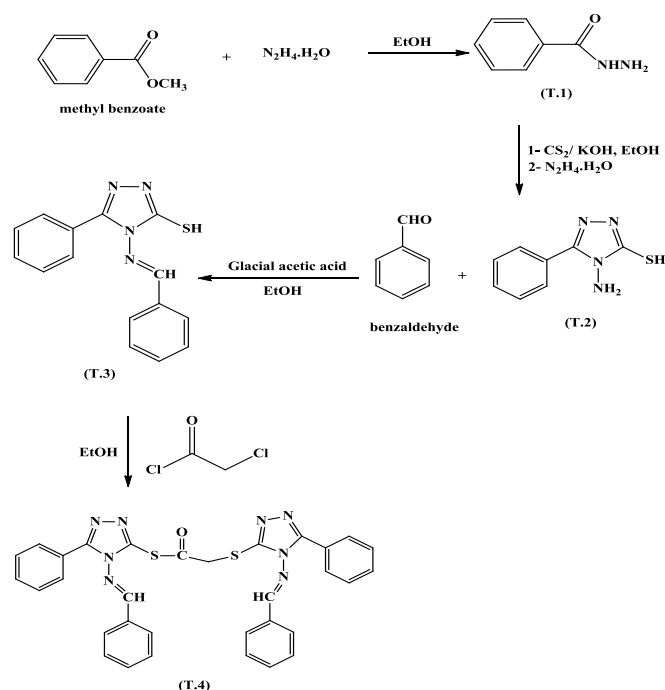
For a full 12 h period, a mixture consisting of compound T.2 (1 g, 0.005 mol.), benzaldehyde (0.5 g, 0.005 mol), and a mixture of 25 mL ethanol and a pair of glacial acetic acid droplets was used to induce reflux. This concoction was chilled to produce an ethanol-derived precipitate and recrystallization [42].

Synthesis of 4-(benzylideneamino-5-phenyl-4H-1,2,4-triazol-3-yl)-2-(4-benzylideneamino-5-phenyl-4H-1,2,4-triazol-3-yl)-thioethanethioate (T.4)

In 25 mL pure ethanol, a mixture of compound T.3 (1 g, 0.0035 mol) and KOH (0.2 g, 0.0035 mol) was dissolved. After the addition of chloroacetyl chloride (0.08 mL, 0.0007 mol), the suspension mixture was refluxed for 12 h with stirring. The reaction mixture was chilled to produce the precipitate and recrystallize ethanol [43],[44].

Melting point: 176 °C–178 °C. FT-IR included the following measurements: Ar-H stretching 3101 cm⁻¹, -N=CH 1603 cm⁻¹, C=O 1725 cm⁻¹, C-H (2928-2885 cm⁻¹, and C-S 719 cm⁻¹.

¹H-NMR revealed ppm values of 7.21-8.07 (m, 20H, Ar-H), 4.13 (s, 2H, CH₂), and 9.75 (s, 2H, -N=CH).



Scheme 1: Synthesis route of compounds T1–T4.

Results and Discussion

Using the proper solvents, all the produced final compounds were initially refined through serial recrystallization. Melting point measurements were used to verify the purity of the synthesized chemicals. Afterward, spectral analyses, including those of FT-IR and 1H -NMR spectra, were performed on the synthesized compounds to verify their structures. Every spectrum was consistent in relation to the structures. Using pure alcohol, benzoic acid hydrazide (T.1) was created from methyl benzoate and hydrazine hydrate. Potassium dithiocarbazinate can be possibly created from the chemical benzoic acid hydrazide. In addition, compounds T.2, T.3, and T.4 were created.

FT-IR spectra revealed that the $-N=CH$ imino group was located at 1603 cm^{-1} and C-S stretching at 719 cm^{-1} .

Compound T.4 was synthesized through the nucleophilical substitution of with chloroacetyl chloride, and KOH was used to prepare a compound that contain sulfur bridge links. The mercapto group and $-N-N-C$ -moiety both imparted activities. Moreover, the entry of aromatic/heterocyclic rings into the central nervous system was facilitated. Given that they have an imine

($-N=CH-$) structure, Schiff bases constitute an important class of flexible compounds that are similar to naturally occurring biological materials.

FT-IR Spectroscopy

FT-IR spectroscopy was used to characterize the produced chemicals. Figure 1.1 shows the FT-IR spectra of compound (T.4) The C-H stretching of aromatic ring 3101 cm^{-1} , C-H stretching of the aliphatic group (2908 – 2885 cm^{-1}), C-S, 719 cm^{-1} , imine group ($C=N$) at 1603 cm^{-1} . Bands of C=N triazole ring at 1507 cm^{-1} .

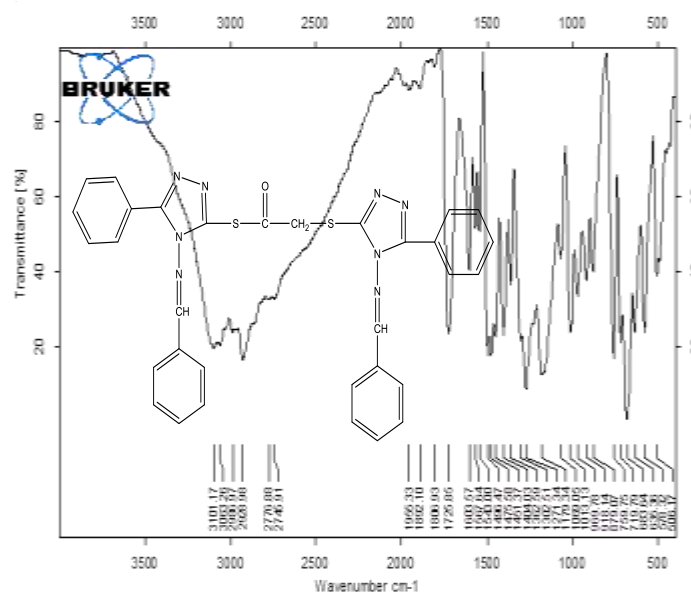


Figure 1. FT-IR spectrum of compound (T.4).

NMR Spectroscopy

Compound T.4, 1H -NMR confirmed the values of 7.21–8.07 (m, 20H, Ar-H), 4.13 (s, 2H, CH_2), and 9.75 (s, 2H, $-N=CH$) ppm.

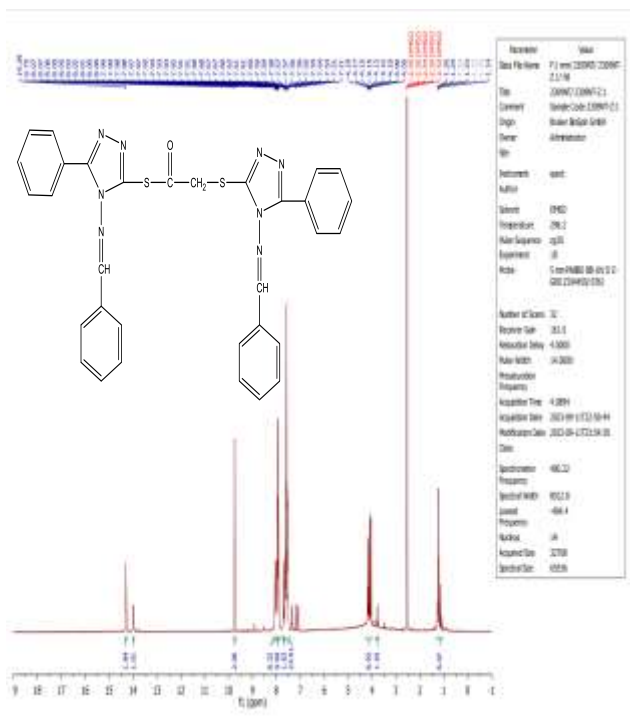


Figure 2. ¹H-NMR spectrum of compound (T.4).

Conclusions

Schiff bases (T.3 and T.4) were synthesized in multiple phases. Carbon disulfide with potassium hydroxide was treated methyl benzoate then with hydrazine hydrate to yield triazole T.2. Compound T.3 was condensed with various aromatic aldehydes to produce the final derivatives in good yields. The creation of T.4 led us to believe in the importance of such compounds for a wide range of uses in industry, agriculture, and medicine

Acknowledgments

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تحضير وتشخيص مشتق 4,2,1-تريازول من بنزوات المثل بروابط جسرية كبريتية

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الخلاصة:

النواة الأساسية 4-(benzylideneamino-5-phenyl-4H-1,2,4-triazol-3-yl)-2-(4-benzylideneamino-5-phenyl-4H-1,2,4-triazol-3-yl)-thioethanethioate T.4 تم تحضيرها بعدة خطوات، بالغلق الحلقي للمركب T.2 باستخدام الإيثانول كمذيب ووجود ثنائي كبريتيد الكربون و هيدروكسيد البوتاسيوم متبوعا باضافة الهيدرازين المائي بالتصعيد لمدة 12 ساعة. للحصول على قاعدة شف T.3، تم مفاعلة المركب T.2 بتفاعل تكثيف بعملية التصعيد مع 4,1-ثنائي كاربالدهيد ثم مفاعلة T.3 مع كلوريد الكلوروأسيثيل. تم استخدام الخصائص الفيزيائية مثل درجة الانصهار والتحليل الطيفي لتأكيد المركبات باستخدام طيف الأشعة فوق البنفسجية وطيف الرنين النووي للبروتون. الكلمات المفتاحية: قاعدة شف، مشتقات التريازول، بنزايدين امين، هيدرازيد.