



Design, Synthesis, and Spectral Characterization of Novel Benzothiazole-Based Heterocycles Containing Oxadiazole, Triazole, and Pyrazole Moieties

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ABSTRACT

Benzothiazole derivatives are an important class of heterocyclic compounds that have attracted considerable attention due to their diverse pharmacological properties and potential therapeutic applications. This study presents the synthesis of a series of benzothiazole-based compounds through multi-step reactions starting from 4-hydroxybenzaldehyde and 2-aminothiophenol.

The synthesized compounds were characterized using spectral techniques including IR and NMR (^1H and ^{13}C). The structural elucidation confirms the successful formation of functionalized benzothiazole frameworks, including oxadiazole, triazole, and pyrazole derivatives. These structures offer valuable scaffolds for further pharmaceutical development, particularly in the design of antimicrobial and anticancer agents.

After preparing the benzothiazole-2-phenol ring, ethyl acetate was loaded onto phenol using alpha-bromo ethyl acetate. The ester was then converted to hydrazide, which underwent a series of subsequent reactions with carbon disulfide and basic medium was converted to oxadiazole-2-thiol, where the oxygen atom was replaced by the hydrazide group to give the triazole ring. The hydrazide was further converted to pyrazole by reaction with acetylacetone and ethyl acetoacetate. Finally, the disubstituted triazole ring was prepared by melting and slowly cooling the hydrazide.

Keywords: Benzothiazole derivatives, 1,3,4-Oxadiazole, 1,2,4-Triazole, Pyrazole, Multi-step synthesis

INTRODUCTION

Heterocyclic compounds, commonly known as heterocycles, are organic compounds with ring structures containing one or more heteroatoms (Qadir *et al.* 2022). Oxygen, nitrogen, and sulfur are among the most common heteroatoms. There are also heterocycles containing other atoms, such as iron, phosphorus, magnesium, and selenium (Kabir and Uzzaman 2022). These compounds may consist of tri-, tetra-, and pentameric rings, etc. These rings can be separate or connected (Abd Alrazzak, 2018). The most common heterocyclic compounds are those with pentameric and hexameric rings, which contain heteroatoms. The most famous of these simple compounds are pyridine (1), pyrrole (2), furan (3), and thiophene (4) (Jaiswal, 2019). Heterocyclic atoms give heterocyclic compounds diverse physical and chemical properties (Patnayak and Optimization 2024).

These compounds have attracted the attention of researchers, particularly in the medical field, due to their tremendous therapeutic potential (Barbuceanu and Olaru, 2025). Heterocyclic compounds are widely distributed in nature and have important and diverse uses, including medical and industrial applications. These compounds are involved in the synthesis of sugars and their derivatives, as well as enzymes, proteins, and nucleic acids (Muhammad *et al.* 2024). They are also used in copolymers, disinfectants, antioxidants, corrosion inhibitors, and dyes (Qadir *et al.* 2022).

Benzothiazole is a heterocyclic compound consisting of a thiazole fused to benzene. The thiazole is a five-membered ring containing a sulfur and nitrogen atom at the (C3, C1) position (Haider *et al.* 2022). Benzothiazole was originally discovered in natural marine molecules, as well as terrestrial samples (Broudic *et al.* 2022). It is found in many biologically important natural products, and unsubstituted benzothiazole was first isolated in 1967 from the volatile matter of American cranberries and French oak wood (Palchykov 2025). Benzothiazole derivatives have been extensively studied for their biological activities (Yadav *et al.* 2023), and they hold a special and important place due to their pharmaceutical applications. Over time, benzothiazole derivatives have demonstrated a wide range of pharmacological effects, including anti-inflammatory, antiviral, antibacterial, analgesic, anticancer, and antioxidant properties (Yadav *et al.* 2023). Recently, benzothiazole derivatives have been shown to have potential uses as diagnostic tools for neurological diseases, orexin receptor agonists, L-coenzyme inhibitors, and antihistamines (Badgujar *et al.* 2024). The present work focuses on the synthesis of novel benzothiazole-based heterocycles containing oxadiazole, triazole, and pyrazole moieties through successive functional-group transformations. The synthesized compounds were characterized using FT-IR, ^1H NMR, and ^{13}C NMR spectroscopy, with selected reactions also examined using a nano-catalyzed approach.

MATERIALS AND METHODS

Materials

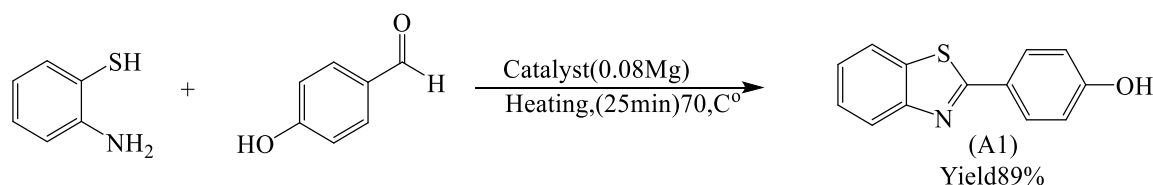
All chemicals and solvents were of analytical grade and used without further purification. Reaction progress was monitored by TLC where required. Melting points were determined in $^{\circ}\text{C}$, while FT-IR, ^1H NMR, and ^{13}C NMR spectra were used for structural characterization. Solvents were removed under reduced pressure using a rotary evaporator, and the magnetic nano-catalyst was separated using an external magnet.

Methods

Synthesis of 4-(benzo[de]thiazol-2-yl) phenol (A1) (Aouad *et al.*, 2024; Saleh *et al.*, 2022)

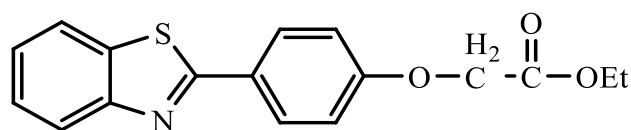
Method 1: 1 mol of 4-hydroxybenzaldehyde was dissolved in 15 ml of absolute ethanol, to which 1 mol of 2-aminothiophene was added. 2-3 drops of glacial acetic acid were added to the reaction mixture, and the mixture was heated for (8) hours. The mixture was then cooled, and the solvent was evaporated under vacuum. The resulting precipitate was recrystallized using ethanol, yielding a yellow precipitate with a melting point of 225-227 $^{\circ}\text{C}$, and a yield of 60%.

Method 2: A mixture of equimolar moles (10 mmol) of 2-aminothiophenol and 10 mmol of 4-hydroxybenzaldehyde was heated in the presence of 0.08 mg of catalyst at 70°C for 25 minutes. The resulting product was confirmed using thin-layer chromatography (TLC). After the reaction was complete, the mixture was cooled and 5 ml of ethyl acetate was added. The catalyst was then isolated using an external magnet, washed with ethanol, and dried at 60°C for reuse. The ethyl acetate was then evaporated under vacuum to produce a yellow precipitate with a melting point of 224-229°C. m°) with a yield of (89%)



Synthesis of ethyl 2-(4-benzo[d]thiazol-2-yl) phenoxyacetate (A2) (Abdu-Rahem *et al.*, 2021)

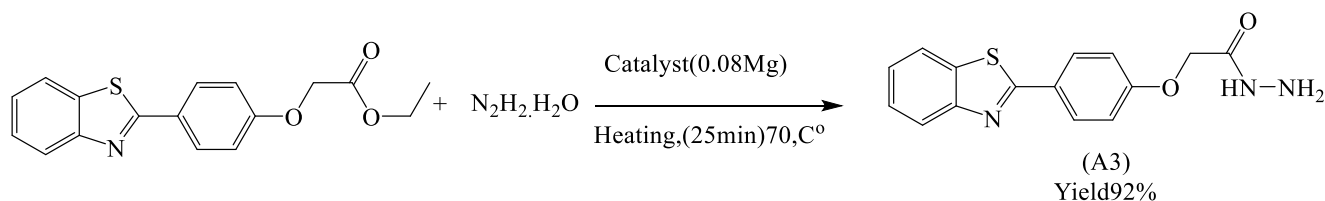
0.02 mol, 4.54 g of the phenolic compound (A1) was dissolved in 50 ml of absolute ethanol. 0.02 mol, 3.34 g of ethyl alpha-bromoacetate and 3 g of sodium bicarbonate (NaHCO₃) were added to the solution. The reaction mixture was then heated for 8 hours, and the solution was concentrated under vacuum to one third. The resulting precipitate was filtered, dried, and recrystallized with ethanol, yielding a white precipitate with a melting point of 91-93 °C, yielding a 60% yield.



Synthesis of 2-(4-(benzo(d)-2-yl) phenoxy) acetohydrazide (A3) (Sa'ad *et al.*, 2022; Sadeek *et al.*, 2023)

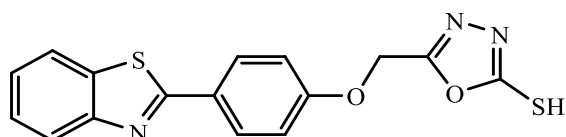
Method 1: (0.01 mol, 4.14 g of ester (A2) was dissolved in 100 ml of ethanol, then 0.05 mol, 2.5 ml of 80% aqueous hydrazine was added. The reaction mixture was heated for 6 hours. The solution was then concentrated under vacuum to one-third. The resulting precipitate was filtered, dried, and recrystallized with ethanol, yielding a white precipitate with a melting point of 248-250 °C and a yield of 84%.

Method 2: A mixture of 10 mmol of the ester (A2) with 80% aqueous hydrazine in the presence of 0.08 mg of catalyst was heated at 70°C for 25 minutes. The resulting product was confirmed using thin-layer chromatography (TLC). After the reaction was complete, the mixture was cooled and 5 ml of ethyl acetate was added. The catalyst was then isolated using an external magnet, washed with ethanol, and dried at 60°C for reuse. The ethyl acetate was then evaporated under vacuum. To give a white precipitate with a melting point of (89-91°C) and a yield of (92%).



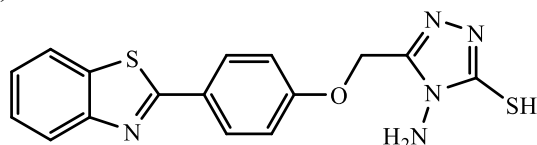
Synthesis of 5-((4-(benzo[d]thiazol-2-yl) phenoxy) methyl)-1,3,4-oxadiazole-2-thiol (A4) (Pandya *et al.*, 2021)

Dissolve (0.005 mol, 4.19 g) of hydrazide (A3) in a solution of potassium hydroxide (0.01 mol, 0.561 g) in (100 ml) of absolute ethanol. Then, gradually add (0.05 mol, 3 ml) of (CS₂) to the reaction mixture while stirring. The reaction mixture was then heated for (24) hours until the emission of (H₂S) gas ceased (as indicated by the blackening of a paper moistened with lead acetate solution). Then the solvent is evaporated under pressure to a third and the remaining volume is added to (20 g) of crushed ice, then the mixture is acidified with concentrated hydrochloric acid, then the precipitate is left for (24) hours to complete the precipitation. The resulting precipitate is filtered, dried and recrystallized from ethanol, to give a solid precipitate with a dark brown color and a melting point of (280-282 °C), with a yield percentage of (77%).



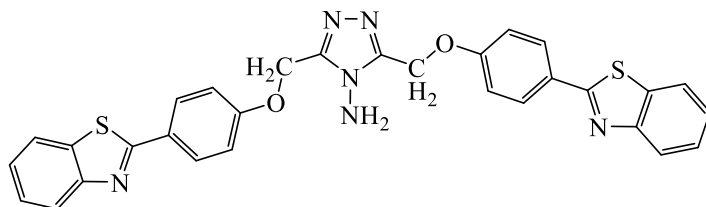
Synthesis of 5-((4-(benzo[d]thiazol-2-yl) phenoxy) methyl)-4-amino-1,2,4-triazole-3-thiol (A5)
(Abdellatif *et al.*, 2021)

(0.01 mol, 0.341 g) of compound (A4) was dissolved in (25 ml) of ethanol, then (0.005 mol, 2.5 ml) of (80%) aqueous hydrazine was added, then the mixture was sublimated for (6) hours, after which the solution was concentrated in a rotary evaporator to one third, then (10 g) of crushed ice was added to the solution to complete the precipitation, then the resulting precipitate was filtered and returned. It was crystallized using ethanol, to give a white precipitate with a melting point of (250-252 °C), and a yield of (74%).



Synthesis of 2-(4-(benzo[d]thiazol-2-yl) phenoxy)-1-(3,5-dimethyl-1H-pyrazol-1-yl) ethan-1-one (A6)
(Ahmed, 2006)

Heat (0.002 mol, 0.598 g) of hydrazide compound (A3) in a sand bath at (260-270 °C) for (60) minutes, then add (50 ml) of distilled water, then submerge for (15) minutes, then filter the hot solution, take the precipitate and discard the filtrate, to give a light brown precipitate with a melting point of (213-215 m), with a yield of (80%) crystalline.



Synthesis of 2-(4-(benzo[d]thiazol-2-yl) phenoxy)-1-(3,5-dimethyl-1H-pyrazol-1-yl) ethane-1-one (A7-A8) (Abd *et al.*, 2020)

Equivalent moles (0.002 mol, 0.598 g) of hydrazide (A3) and (0.002 mol) of (acetyl ethyl acetate, acetal acetone) were dissolved in 25 ml of absolute ethanol. The mixture was then heated for 12 hours. The solution was concentrated under vacuum to one-third. The resulting product was

filtered, dried, and recrystallized using ethanol, the physical constants of the two compounds are shown in (Table 1) below.

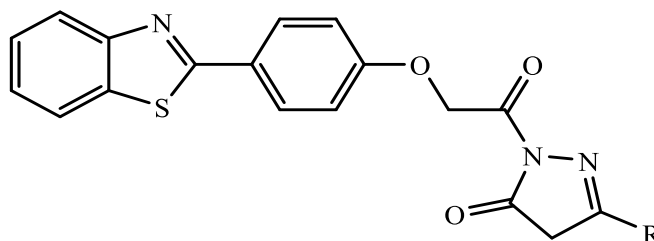


Table (1): Physical properties of compounds (A9 - A10)

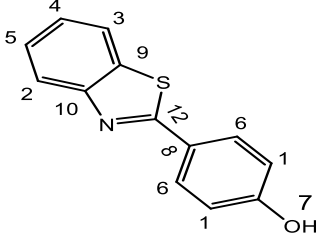
Comp No.	R	Molecular formula	m.p.(°C)	Yield (%)	Color
A7		C ₁₉ H ₁₅ N ₃ O ₃ S ₁	275-277	68	White
A8		C ₂₀ H ₁₇ N ₃ O ₂ S ₁	287-289	63	Pall yellow

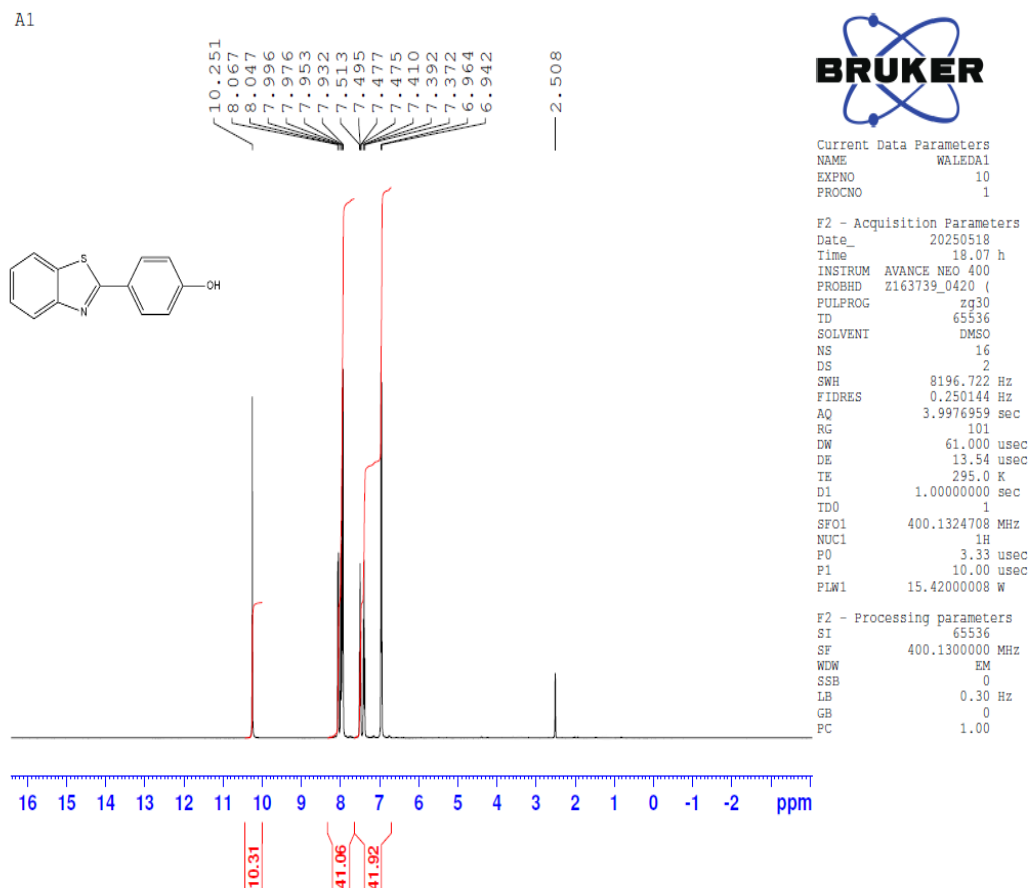
All synthesized compounds described in this work are summarized in Scheme 4, which presents the overall synthetic pathway and illustrates the sequence of reactions leading to the final benzothiazole-based heterocyclic derivatives.

RESULTS AND DISCUSSION

The benzothiazole compound substituted with a phenol group at position (2) was prepared by reacting ortho-aminothiophenol with para-hydroxybenzaldehyde in the presence of glacial acetic acid. The mixture was refluxed for 8 hours and the product was identified by spectral bands, giving Compound 4-(benzo(d)thiazol-2-yl) phenol (A1) show scheme (1) identified using the infrared (IR) spectrum, which showed a broad band at 3350 cm⁻¹, corresponding to the (O-H) group, and bands in the range of 1602-1453 cm⁻¹, corresponding to the (C=C) group, structural vibrations of the aromatic ring, in addition to the (C-O) bond stretching band at 1282 cm⁻¹. The spectrum also showed the aromatic (C-H) bond stretching band at a frequency of 3045 cm⁻¹. The compound (A1) was also identified based on the nuclear magnetic resonance spectrum (¹H-NMR), which showed a single band of the (OH) group at (12.25 ppm), while the resonance spectrum (¹³C-NMR) showed a band of the carbon atom linked to the (OH) group at (161 ppm), as shown in Fig. (1) and (2) and the (Table 2) below between the spectral properties of the compound (A1).

Table (2): The spectral properties of compound (A1)

Structure	¹ H NMR (DMSO-d ₆), ppm
	1:6.94 (d, H), 2:7.41(d, H),3:7.49(d, H),4-5:7.93-7.99(M,2H),6:8.04 (d, H),7:10.25(S, H),
	¹³ C NMR (DMSO-d ₆), ppm
	1:116.56, 2:122.54, 3:122.97, 4:124.53, 5:125.34, 6:129.25, 8:129.52, 9:134.59, 10:154.21, 11:161.01, 12:167.93

**Fig. 1: ¹H NMR for compound A1**

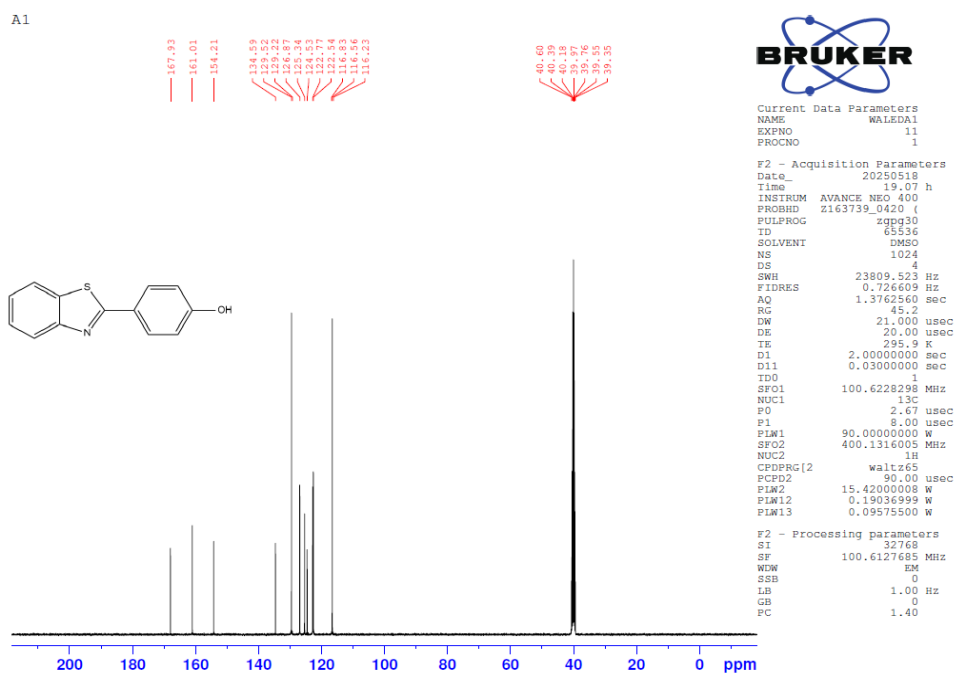
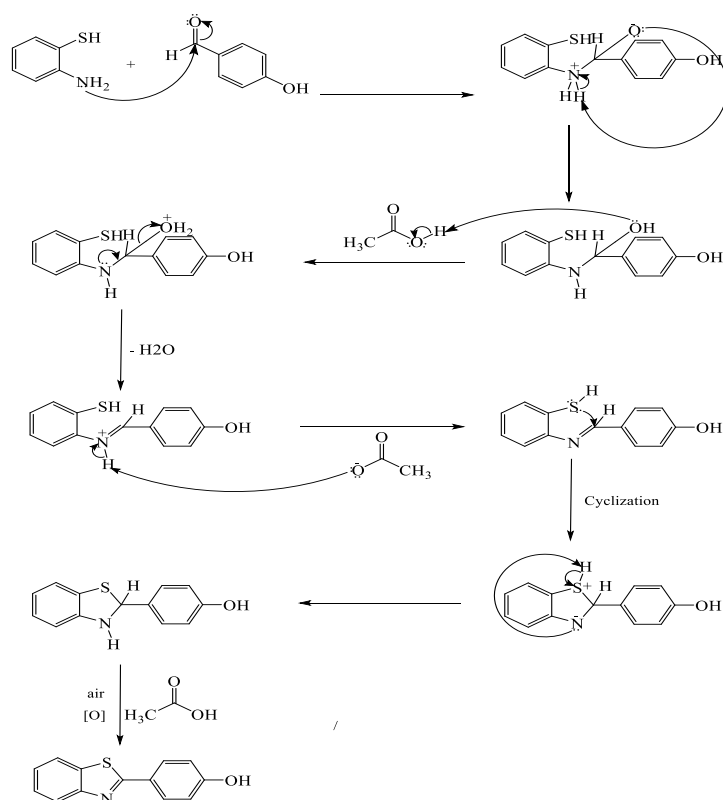


Fig. 2: Carbon 13 for compound A1



Scheme 1: Mechanism Synthesis reactions of compound (A1)

Also, in this work, it was possible to use a nano-catalyst to obtain a purer product, in a larger quantity, and in a faster time, as the iron-loaded nano-silica catalyst was used for this purpose.

Fig. 3: ^1H NMR for compound A2

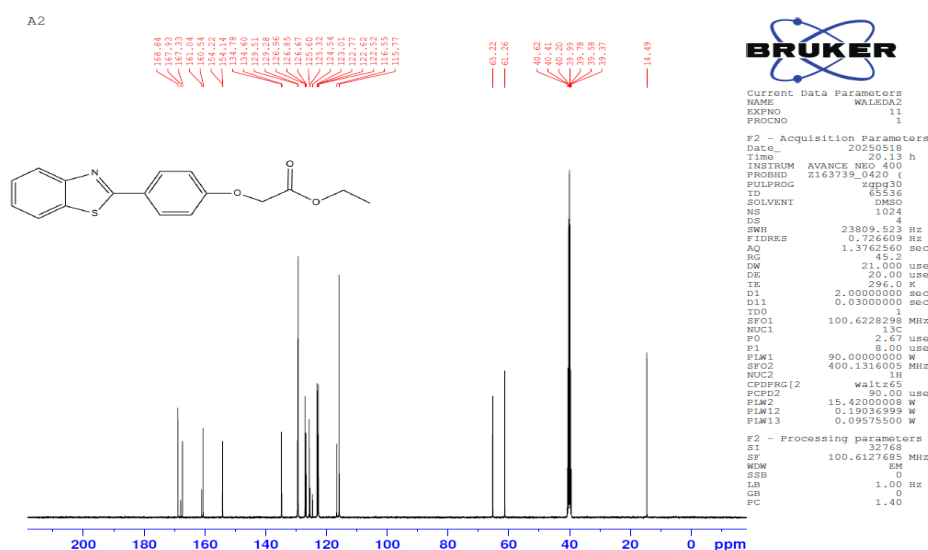
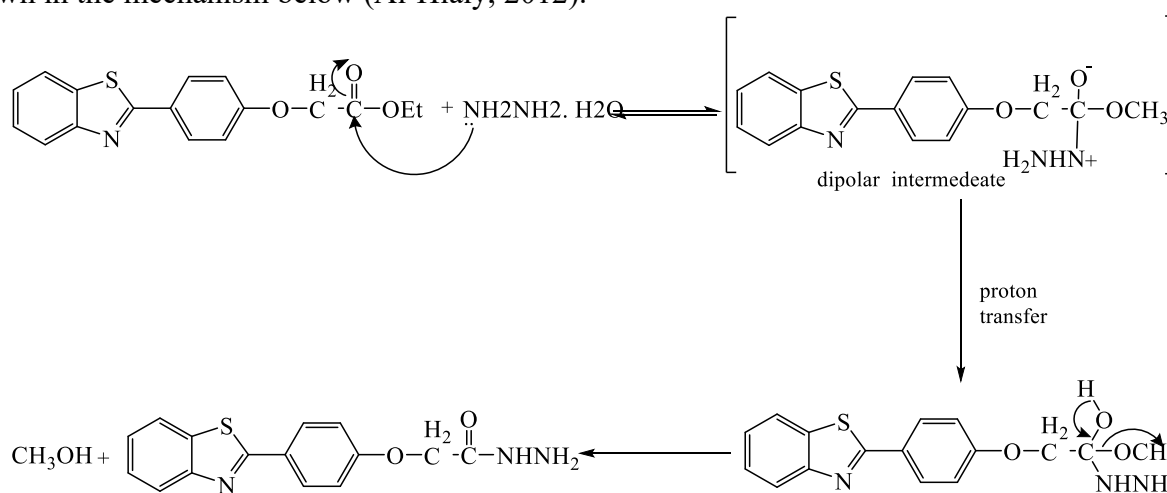


Fig. 4: Carbon 13 for compound A2

The ester group was converted to acid hydrazide by adding hydrazine water (80%) and sublimating the mixture in the presence of ethanol to give compound (3A) 4-(benzo(d)thiazol-2-yl)acetohydrazide was prepared by the reaction of the corresponding ester (A2) with hydrated hydrazine (80%) by thermal sublimation in ethyl alcohol, as shown in Scheme (2). The hydrazide preparation reaction is a tetrahedral nucleophilic substitution reaction on the carbonyl group, as shown in the mechanism below (Al-Hialy, 2012).



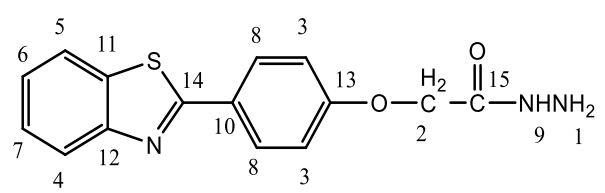
Scheme 2: Mechanism Synthesis reactions of compound (A2)

This reaction was also prepared using nano-catalyst, which gave a higher yield and good purity, in addition to the shortened time.

was characterized by the infrared (IR) spectrum, which showed a significant decrease in the frequency value of the amide carbonyl group (1661 cm^{-1}) compared to the frequency of the ester carbonyl group, in addition to the appearance of stretching bands of aliphatic and aromatic (C-H) groups at frequencies (2911) and (3028 cm^{-1}), respectively. The spectrum also showed stretching bands of amide amine groups at (3203 cm^{-1}) and the primary amine group at (3311 cm^{-1}), in addition

to the appearance of stretching bands of structural (C=C) groups belonging to the aromatic ring in the range (1602-1483) cm^{-1} . Compound (A3) was also identified based on its ^1H -NMR spectrum, which showed a single band at 4.37 ppm, corresponding to the primary amine group (NH_2), as well as the appearance of a single band for the proton of the amide group ($\text{O}=\text{C}-\text{NH}_2$) at 9.43 ppm. Meanwhile, the ^{13}C -NMR spectrum showed the normal carbon atom band of the amide carbonyl group at 168.83 ppm, while the carbon atom band of the ethoxy group of the corresponding ester disappeared (Table 4) below contains the spectral information for compound (A3).

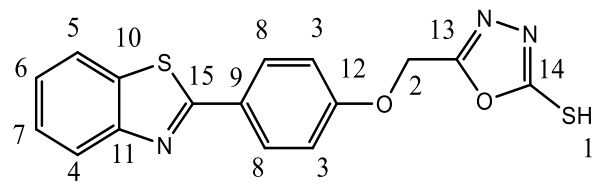
Table (4): The spectral properties of compound (A3)

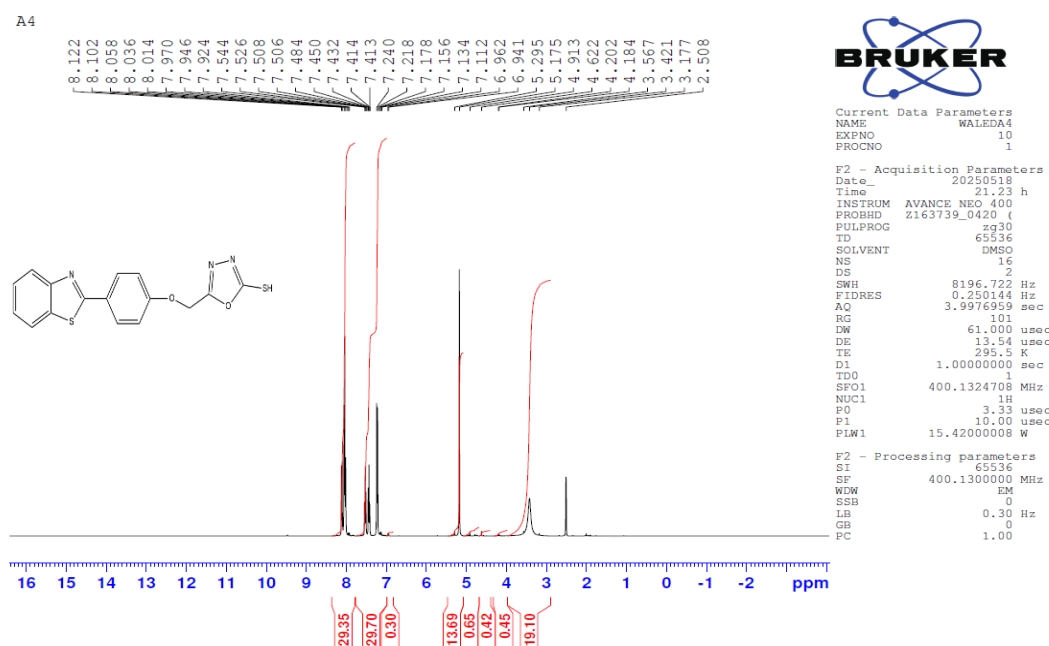
Structure	^1H NMR (DMSO- d_6), ppm
	1:4.37 (S, H), 2:4.61 (S, 2H), 3:7.13 (d, H), 4:7.41 (d, H), 5:7.50 (d, H), 6-7:7.92-8.05 (M, 2H), 8:8.09 (d, H), 9:9.43 (S, H).
	^{13}C NMR (DMSO- d_6), ppm
	2:65.22, 3:116.55, 4:122.62, 5:122.77, 6:124.54, 7:125.30, 8:129.28, 10:126.69, 11:134.78, 12:154.22, 13:161.04, 14:167.33, 15:168.84

The acid hydrazide was converted to oxadiazole by cyclization through the addition of carbon disulfide with potassium hydroxide and sublimation of the mixture (Saleh *et al.*, 2023), which gave compound (A4) was characterized by infrared (IR) spectrum, which showed the disappearance of bands from the primary amine and carbonyl amide (NH_2) groups of the hydrazide compound, and the appearance of (C=N) bond stretching bands at (1633 ppm) cm^{-1} , in addition to the appearance of (C-O-C) bond stretching bands in the range (1045-1102) cm^{-1} . The spectrum also showed stretching bands of aliphatic and aromatic (C-H) groups at frequencies (2945) and (3099) cm^{-1} , respectively, in addition to the appearance of a distinctive band at (912) cm^{-1} , which is due to the stretching of the (C-S) bond, which confirms the authenticity of the resulting compound, in addition to the appearance of stretching bands of groups The (C=C) structural pattern was identified in the range of 1602-1456 cm^{-1} .

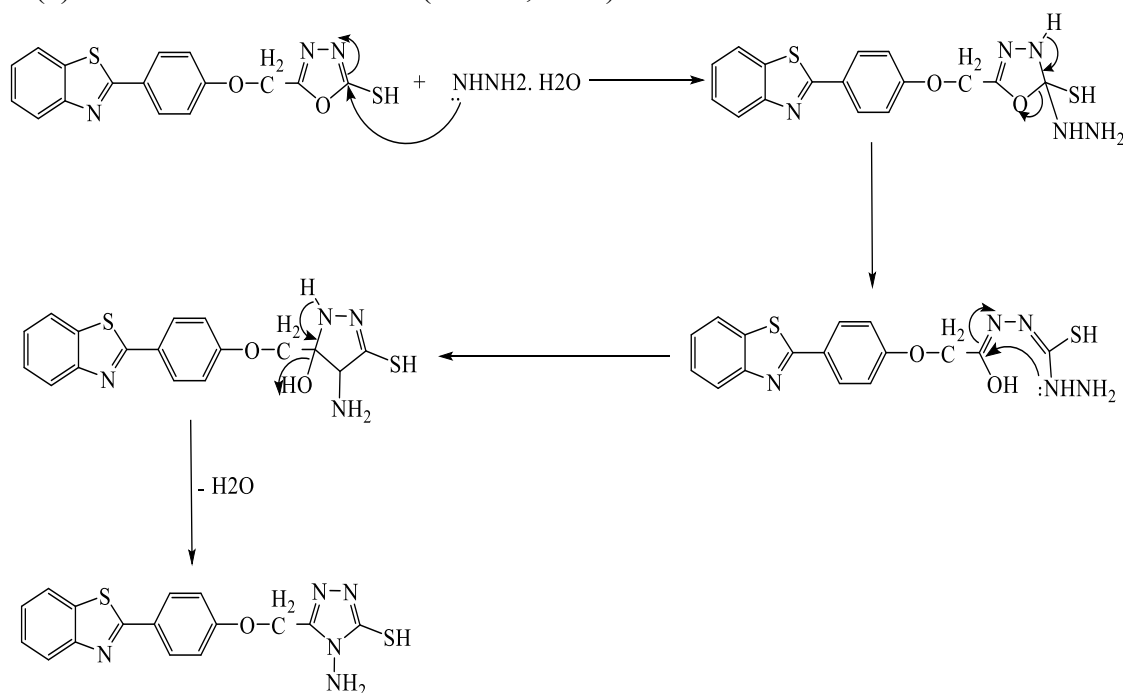
Compound (A4) was also identified based on its ^1H -NMR spectrum, which showed a single band at 3.42 ppm, corresponding to the (SH) group, as well as a single band corresponding to the (O- CH_2) group at 5.17 ppm. Meanwhile, the (^{13}C -NMR) spectrum showed a carbon atom band located within the oxadiazole ring at 158.12 ppm, as shown in Fig. (5) and (Table 5) below shows the spectral information for compound (A4).

Table (5): shows the spectral properties of compound (A4)

Structure	^1H NMR (DMSO- d_6), ppm
	1:3.42 (S, H), 2:5.17 (S, 2H), 3:7.21 (d, H), 4:7.43 (d, H), 5:7.50 (d, H), 6-7:7.92-8.05 (M, 2H), 8:8.10 (S, H).
	^{13}C NMR (DMSO- d_6), ppm
	1:181.16, 2:60.60, 3:116.01, 4:122.70, 5:123.01, 6:125.65, 7:126.64, 8:129.32, 9:127.01, 10:134.77, 11:154.12, 12:158.12, 13:160.63, 14:167.38

Fig. 5: ¹HNMR for compound A5

Compound (A5) was prepared by the condensation of hydrazine (80%) with 1,3,4-oxadiazol-2-thiol (A4) in ethanol. This reaction resulted in the loss of a water molecule, as shown in Scheme (3) and the mechanism below (Ahmed, 2013).

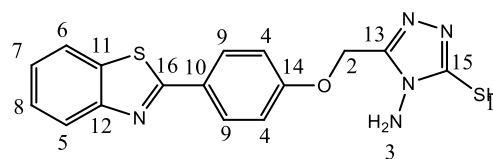


Scheme 3: Mechanism Synthesis reactions of compound (A5)

Compound (A5) was characterized by infrared (IR) spectroscopy, which showed a distinct band at 3318 cm^{-1} due to the stretching of the primary amine group, as well as a stretching band for the (C=N) bond, which appeared at 1630 cm^{-1} , and a stretching band for the thione group (HN-C=S) at 1221 cm^{-1} . The spectrum also showed stretching bands for the aliphatic and aromatic (C-H) groups at positions 2970 and 3048 cm^{-1} , respectively, in addition to the appearance of stretching bands for

the structural (C=C) groups belonging to the aromatic ring at the range of 1601-1453 cm^{-1} . Compound (A5) was identified based on its ^1H -NMR spectrum, which showed a single band at 5.70 ppm, which is attributed to the primary amine group (NH_2). The ^{13}C -NMR spectrum also showed a carbon atom band ($\text{O}-\text{CH}_2$) at 59.86 ppm, and a carbon atom band associated with the (SH) group at 148.27 ppm, as shown in Fig. (6) and (Table 6) below shows the spectral properties of compound (A5).

Table (6): shows the spectral properties of compound (A5)

Structure	^1H NMR ($\text{DMSO}-d_6$), ppm
	1:4.61 (S, H), 2:5.32 (S, 2H), 3:5.70 (S, 2H), 4:7.23 (d, H), 5:7.46 (d, H), 6:7.53 (d, H), 7-8:7.97-8.07 (M, 2H), 9:8.11 (d, H).
	^{13}C NMR ($\text{DMSO}-d_6$), ppm 2:59.86, 4:116.19, 5:122.73, 6:123.03, 7:125.69, 8:126.82, 9:129.34, 10:127.04, 11:134.77, 12:154.11, 13:148.27, 14:160.58, 15:167.24, 16:167.33

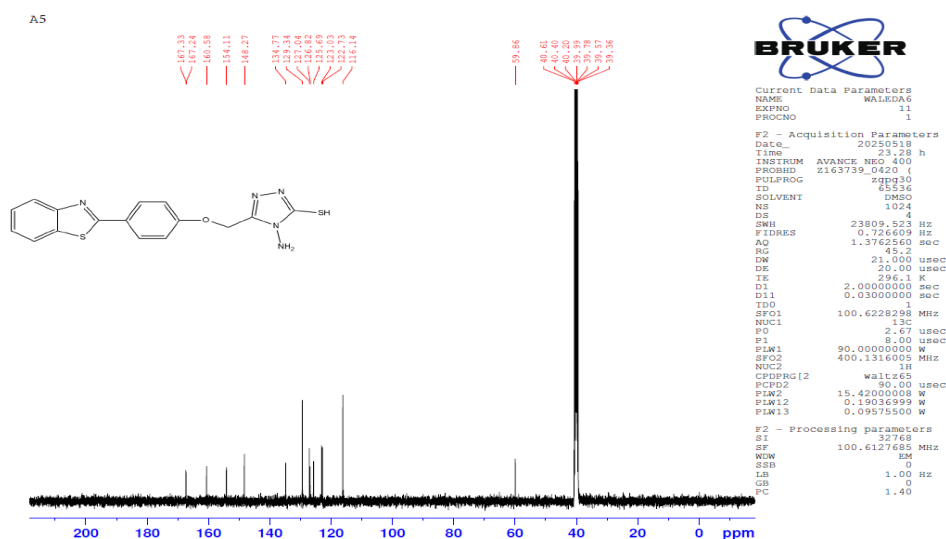
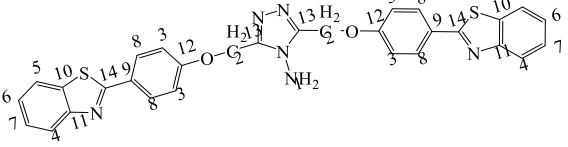


Fig. (6): Carbon 13 for compound A1

By melting the acid hydrazide and heating it at its melting point, the triazole compound substituted with an amine group was obtained after cooling the melt and adding water to it, which gave compound (A7) was identified by infrared (IR) spectroscopy, which showed stretching bands of the primary amine group at 3328 cm^{-1} , as well as a distinct band at 1654 cm^{-1} , which is due to the stretching of the ($\text{N}=\text{C}$) bond. The spectrum also showed stretching bands of the aliphatic and aromatic (C-H) groups at frequencies of 2981 and 3031 cm^{-1} , respectively, as well as stretching bands of the structural (C=C) groups of the aromatic ring at 1484-1604 cm^{-1} .

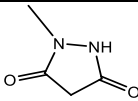
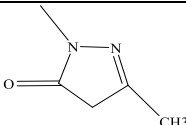
Compound (A6) was identified by nuclear magnetic resonance (^1H -NMR) spectroscopy, which showed a single band of the primary amine (NH_2) group at 3.37 cm^{-1} . ppm), and the resonance spectrum (^{13}C -NMR) showed a carbon atom band ($\text{O}-\text{CH}_2$) at (66.79 ppm), (Table 7) below shows the spectral properties of compound (A6).

Table (7): The spectral properties of compound (A6)

Structure	¹ H NMR (DMSO-d ₆), ppm
	1:3.37 (s, 2H), 2:4.78 (s, H), 3:7.20 (d, H), 4:7.45 (d, H), 5:7.52 (d, H), 6-7:7.92-8.06 (m, 2H), 8:8.11 (d, H).
	¹³ C NMR (DMSO-d ₆), ppm
	2:66.79, 3:116.56, 4:122.69, 5:122.76, 6:124.51, 7:125.64, 8:129.27, 9:127.01, 10:134.77, 11:154.12, 12:160.62, 13:160.00, 14:167.37

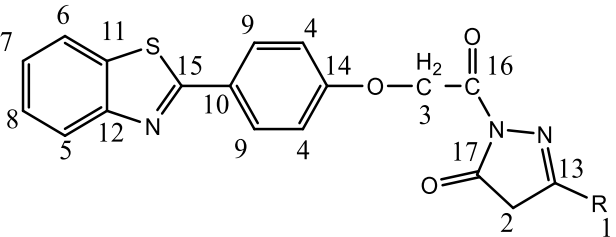
Addition of ethyl acetoacetate and acetyl acetone to acid hydrazide gives substituted pyrazole compounds as in the two compounds (A7-8) were identified by spectroscopic measurements. The infrared (IR) spectrum of compound (A7) showed the appearance of a stretching band of the carbonyl lactam group (O=C) at a frequency of (1705) cm⁻¹, in addition to the appearance of a stretching band of the amide (N-H) bond at (3311) cm⁻¹. The disappearance of the stretching band of the primary amine group of the hydrazide confirms the validity of the reaction. The spectrum also showed stretching bands of the aliphatic and aromatic (H-C) groups at positions (2973) and (3057) cm⁻¹, respectively, in addition to the appearance of stretching bands of the structural (C=C) groups belonging to the aromatic ring at the range (1602-1459) cm⁻¹, as shown in (Table 8).

Table (8): The spectral properties of compound (A7-8)

Comp No.	R	C=O Lactum	C=N	C=C	C-H Ar	C-H Alp
A ₈		1705	1623	1602-1459	3057	2973
A ₉		1702	1440-1613	1440-1583	3090	2955

The compound (A8) was also identified based on the nuclear magnetic resonance spectrum (¹H-NMR), as the spectrum showed the disappearance of the bands of the primary amine group (NH₂) and the proton of the amide group, in contrast to the appearance of a single band of the (CH₃) group at (2.13 ppm), while the resonance spectrum (¹³C-NMR) showed a lactam carbon atom band at (168.08 ppm), in addition to a carbon atom band (CH₃) at position (14.08 ppm), as shown in Fig. (7), and the (Table 9) below shows the spectral information for the compound (A7).

Table (9): Shows the spectral properties of compound (A7)

Structure	¹ H NMR (DMSO-d ₆), ppm
	1:2.13 (s, 3H), 2:3.34 (s, 2H), 3:5.61 (s, 2H), 4:7.16 (d, H), 5:7.43 (d, H), 6:7.53 (d, H), 7-8:8.01-8.04 (m, 2H), 9:8.10 (d, H).
	¹³ C NMR (DMSO-d ₆), ppm
	1:14.08, 2:58.07, 3:66.60, 4:115.88, 5:122.71, 6:123.00, 7:125.04, 8:126.53, 9:129.33, 10:127.01, 11:134.73, 12:154.14, 13:153.05, 14:160.77, 15:167.39, 16:168.08, 17:169.38

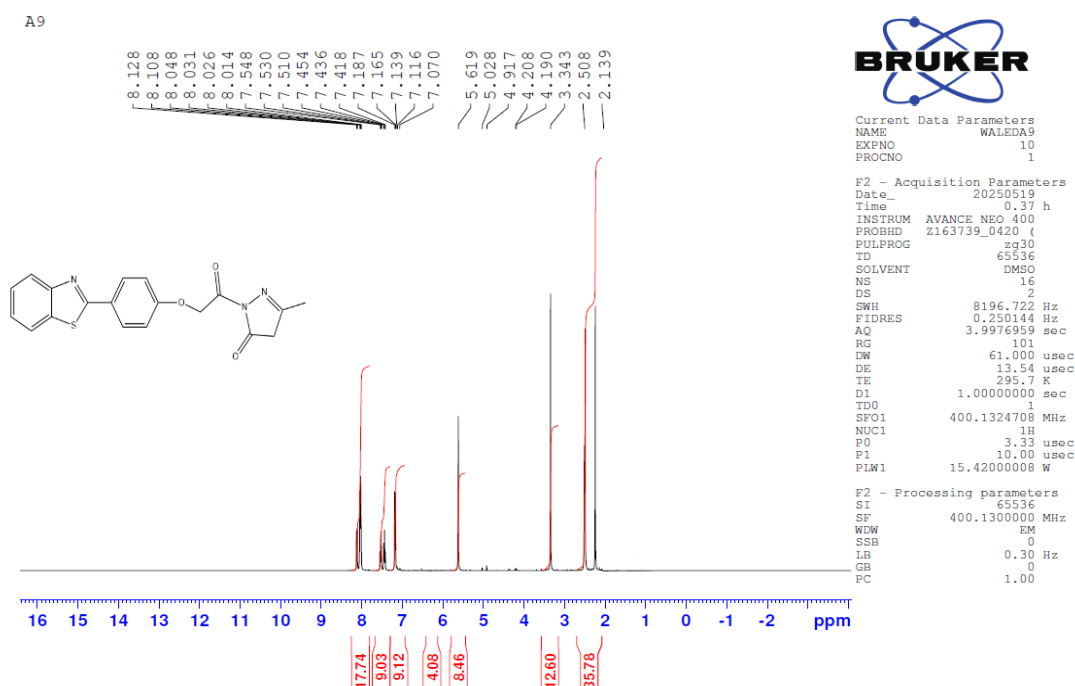


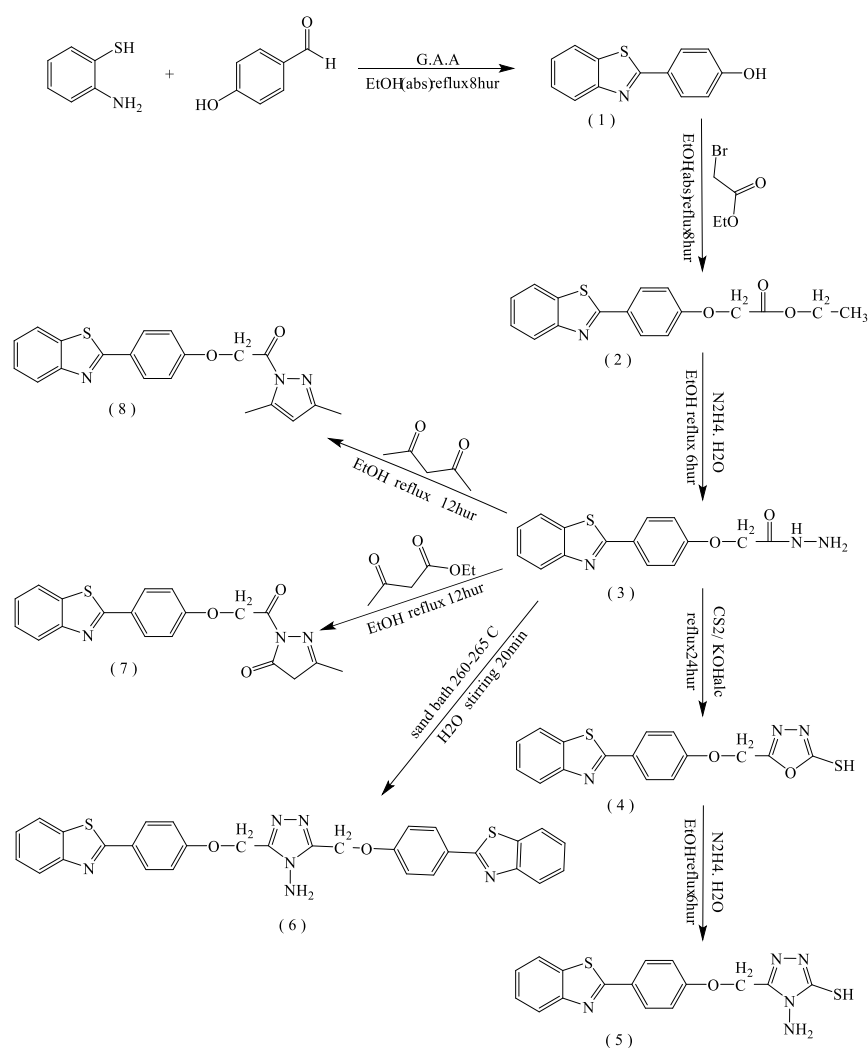
Fig. 7: ^1H NMR for compound A7

Compounds 2-(4-(benzo[d]thiazol-2-yl) phenoxy-(1-(3,5-dimethyl-H1-pyrazol-1-yl) ethane-1-one) (A8) was identified by spectroscopic measurements (Saleh and Saleh 2022). The infrared (IR) spectrum revealed the appearance of a stretching band of the carbonyl lactam group at 1693 cm^{-1} , in addition to the appearance of a (C=N) stretching band at 1621 cm^{-1} , and an amide (N-H) stretching band at 3311 cm^{-1} . The disappearance of the stretching band of the primary amine group of the hydrazide confirms the validity of the reaction. The spectrum also showed stretching bands of aliphatic and aromatic (H-C) groups at positions 2999 and 3092 cm^{-1} , respectively, in addition to the appearance of stretching bands of structural (C=C) groups belonging to the aromatic ring. At a range of $(1478\text{-}1602)\text{ cm}^{-1}$.

Compound (A8) was identified based on its ^1H -NMR spectrum. The spectrum revealed the disappearance of the primary amine (NH_2) and amide proton bands, while two single bands of (CH_3) groups appeared at 1.23 ppm and 2.00 ppm . The ^{13}C -NMR spectrum revealed a lactam carbon atom band at 169.93 ppm . (Table 10) below shows the spectral properties of compound (A8).

Table (10): The spectral properties of compound (A8)

Structure	^1H NMR (DMSO- d_6), ppm
	1:1.23 (S, 3H), 2:2.00 (S, 3H), 3:4.15 (S, 2H), 4:5.08 (S, H), 5:7.04 (d, H), 6:7.43 (d, H), 7:7.52 (d, H), 8-9:8.01-8.07 (M, 2H), 10:8.10 (d, H).
	^{13}C NMR (DMSO- d_6), ppm 1:14.54, 2:16.77, 3:65.47, 4:115.98, 5:115.68, 6:122.69, 7:122.96, 8:125.59, 9:126.16, 10:129.25, 11:127.00, 12:134.72, 13:154.14, 14:148.54, 15:153.22, 16:161.21, 17:169.38, 18:169.93



Scheme 4: Synthesis reactions of benzothiazole substitutes

CONCLUSIONS

A series of novel benzothiazole derivatives were successfully synthesized via efficient and reproducible synthetic routes. Spectroscopic data (IR, ¹H-NMR, and ¹³C-NMR) confirmed the structures of the synthesized compounds. The presence of different heterocyclic systems, such as 1,3,4-oxadiazole and 1,2,4-triazole rings, enhances the potential biological activity of the final products. These benzothiazole-based compounds could serve as promising candidates for further biological screening and drug development. The synthesis pathways used offer good yields, mild conditions, and potential for green chemistry through catalyst reuse and short reaction times.

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تصميم وتحضير والتشخيص الطيفي لمركبات حلقة غير متجانسة جديدة مشتقة من البنزوثيازول والمحتوية على وحدات الأوكساديازول والترايازول والبيرازول

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الملخص

تُعد مشتقات البنزوثيازول فئة مهمة من المركبات الحلقية غير المتجانسة التي حظيت باهتمام كبير بسبب تنوع خصائصها الدوائية وإمكاناتها الواعدة في التطبيقات العلاجية. تعرض هذه الدراسة تحضير سلسلة من المركبات المشتقة من البنزوثيازول من خلال تفاعلات متعددة الخطوات، بدءاً من 4-هيدروكسي بنزالديهايد و2-أمينوثيوفينول. شُخِّصت المركبات المحضرة باستخدام تقنيات التحليل الطيفي، بما في ذلك مطيافية الأشعة تحت الحمراء ومطيافية الرنين المغناطيسي النووي للبروتون والكربون-13. وأكد التعيين البنوي نجاح تكوين هياكل بنزوثيازول وظيفية والتي اشتملت على مشتقات الأوكساديازول والترايازول والبيرازول. وتمثل هذه التراكيب هياكل أساسية قيّمة يمكن الاستفادة منها في الدراسات الدوائية المستقبلية، ولا سيما في تصميم العوامل المضادة للميكروبات والمضادة للسرطان. بعد تحضير حلقة البنزوثيازول-2-فينول، أُدخلت مجموعة إيثيل أسيتات إلى مجموعة الفينول باستخدام ألفا-برومو إيثيل أسيتات. ثم حُوِّل الإستر إلى الهيدرازيد، الذي أخضع لسلسلة من التفاعلات اللاحقة مع ثنائي كبريتيد الكربون في وسط قاعدي لتكوين أوكساديازول-2-ثيول. وبعد ذلك، استُبدلت ذرة الأوكسجين بمجموعة الهيدرازيد لتكوين حلقة الترايازول. كما حُوِّل الهيدرازيد إلى مشتقات البيرازول من خلال تفاعله مع الأسيتيل أسيتون وإيثيل أسيتوأسيتات. وأخيراً، حُضِرَت حلقة الترايازول ثنائية الاستبدال من خلال صهر الهيدرازيد ثم تبريده ببطء.

الكلمات الدالة: مشتقات البنزوثيازول، 1، 3، 4 - أوكساديازول، 1، 2، 4 - ترايازول، البيرازول، التحضير متعدد الخطوات.