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Synthesis, Characterization and Biological activity of Novel Tetrazole Compounds Derived from Azo Schiff bases

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Abstract

Tetrazole and its derivatives are one of the most common five-membered heterocyclic compounds that receive great attention from researchers. Their importance is due to their multiple applications in various fields, such as industrial, medical, and agricultural applications. The aim of the current study is to obtain a new nucleus for the active compound 4- aminoantipyrene with an active group, which is the formyl group, then link it with different amines to form compounds (Schiff bases) that contain the active group (C=N) azomethine and convert them to the heterogeneous pentacyclic system called tetrazoles which is expected to have promising biological effectiveness. In this study, (E)-3-((1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-yl) diazenyl)-4-hydroxybenzaldehyde (AZ1) was prepared by the reaction of 4-aminoantipyrine Diaz onium salts and 4- hydroxybenzaldehyde under base conditions. Schiff bases (2-6) were synthesized by the condensation of compound (1) and aromatic amines such as (4-aminoaniline, , 3-nitroaniline, 4-amino pyridine, 4-aminoantipyrin and 2,4-dinitroaniline). A novel of five member-rings of Tetrazoles (A_7-A_{11}) were created through the interaction of Schiff bases and sodium azide. IR as well as NMR spectrum investigations validated the structure of the resulted compounds example (A10 and A5) compounds

Introduction

Tetrazole compounds are very helpful and useful in the manufacturing of drugs and in other medical fields [1]. these compounds have shown therapeutic effects against anti-carcinogenic [2], six tetrazole derivatives were synthesized by multicomponent reactions (MCR) and their anticancer effects were evaluated in vitro [3], anti-inflammatory, antipain-causing[4,5] , germs, bacterial, asthmatic [6] and useing ligands in coordination chemistry[7]. Heterocyclic rings with five members have four nitrogen atoms in them (via aromatic properties) and one carbon atom [8]. Tetrazole compounds are considered one of most important types of heterocyclic compounds for toward medication [9,10]. Therefore this composition has a substantial effect on the physical and chemical properties of Tetrazoles[11], and the ring is used to create novel drugs because it's the analogue of the bioisosteric of cis-amide and the carboxylic acid [6]. Furthermore, tetrazoles react with both

electron with drawing and electron-donating groups [12]. Tetrazoles were prepared by the reaction of hydrogen cyanide and hydrazoic acid under pressure [13]. Therefore, these compounds have attracted considerable attention by researchers as potential multiple applications in the both fields of medicine and biochemistry [11][14]. According to the earlier mentioned importance of these compounds, this research employed the cyclization reaction to prepare novel tetrazole derivatives via Schiff bases. so, some of these compounds prepared against bacteria were studied, and some of them were proven effective against two types of bacteria. The manufacture of a number of these compounds or their derivatives may contribute to the development of a new drug with high effectiveness

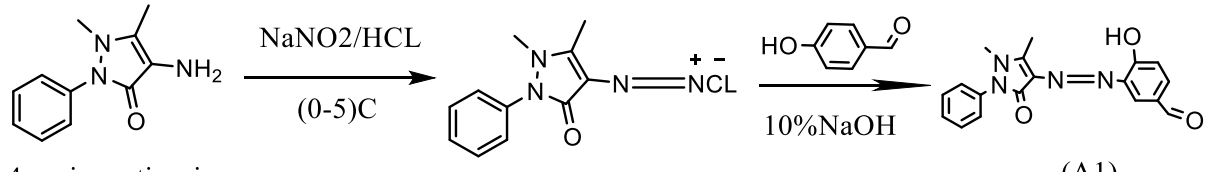
Material and Methods

The melting point of the compounds was measured using (melting point device type Smp.30) supplied by the British company (STURART). Infrared spectra were reported utilizing IR Affinity-1S, Fourier transform infrared spectrophotometer, Shimadzu type device at (4000-600 cm-1) in terms of wavenumber.

.^{1}H.NMR were reported by using Bruker 500MHz/Topspin nmrsu 66 apparatus, used TMS as an internal standard and DMSO-d6 as a solvent. Every chemical was supplied via Aldrich Sigma, Companies BHD and Fluka. Thin layer chromatography (TLC) have been implemented on a freshly prepared silica gel (120 mash) coated plates (1 × 10).

Prepared of general procedure for coupling diazotized 4- amino antipyrin to 4-hydroxy benzaldehyde to form 3-((1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-yl) diazenyl) -4 -hydroxybenzaldehyde (A_1) : [15][16]

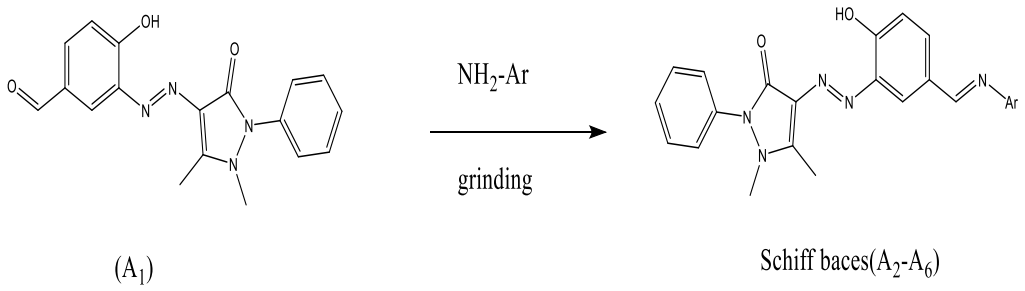
4-aminoantipyrin (7.58 gm, 0.058 mol) has been added in a mixture of (16mL) of each conc. HCl and water in the conical flask. The resulted solution was cooled using crushed ice at (0-5◦C), then sodium nitrite (4 gm, 0.08 mol) has been added in 20 ml water drop by drop using a separating funnel with stirring. Keep the solution stirred slowly for (5 minutes) in the ice bath. Then, 4-hydroxybenzaldehyde (5.05 gm, 0.054 mol) was placed to (45 ml ,10% NaOH) via mixing it to form phenolic solution. The crude solution was stirred vigorously under (5◦C) by adding more crushed ice immediately. The cold diazonium salt solution (with few amounts of crushed ice) was added in a separating funnel and placed drop to the stirred phenolic solution, the orang colure will instantly appear. After the formation of diazonium salt, the solution was stood away in an ice tube for (30 min), followed by filtration and washing thoroughly with three times cold water. After drying the precipitate and recrystallizing it with ethanol and water in a ratio of 2:1, orange-colored crystals were formed with a 95% product., m.p:169-168 ◦C, (monitored by TLC using (4:1) benzene: methanol, Rf:6.56.



4-aminoantipyrin (A1)

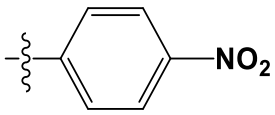
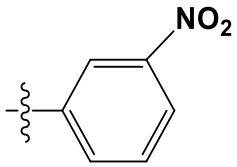
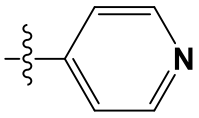
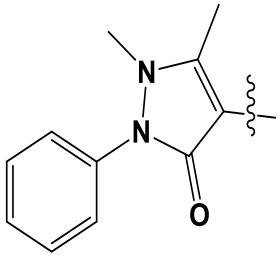
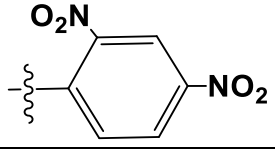
Prepared of Schiff bases derivatives 4-((E)-(5-((E)- (argioimino) methyl)-2-hydroxyphenyl)diazenyl)-1,5-dimethyl-2-phenyl-1,2-dihydro-3H-pyrazol-3-one (A_2-A_6) compounds via grinding technique: [17][18]

A rapid green Chemistry technique was used for the free conditions Schiff base synthesis, with great yields and excellent reactions. The azo Schiff bases were synthesized by grinding the aldehyde and different amines in a mortar for a minute and then maintaining the mixture at 25◦C for 1.5h. The resulted water was removed under vacuum at 70◦C. This reaction was stood at room temperature with gaining a good yield. The reaction was checked by TLC and the physical characteristics of substances (2-6) were included in the table 1.



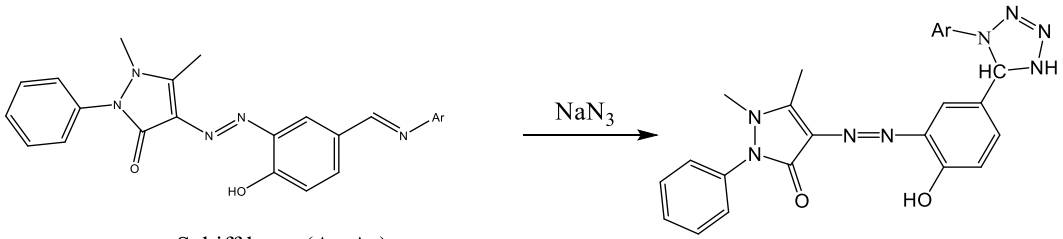
(A_1) Schiff baces(A_2-A_6)

Table 1: The physical characteristics constants of substances (A_2-A_6)

Comp No.	Structure of Ar	Molecular formula and M.Wt g/mol	M.P (^{0}c)	TLC	Yield %	Color
A_2		$C_{24}H_{20}N_6O_4$ 456	130-132	0.45	70	Orange crystal
A_3		$C_{24}H_{20}N_6O_4$ 456	155-153	0.53	72	White crystal
A_4		$C_{23}H_{20}N6O2$ 412	228-231	0.35	60	Orange powder
A_5		$C_{29}H_{27}N_7O_3$ 521	226-228	0.54	64	Orange powder
A_6		$C_{24}H_{19}N_7O_5$ 485	178 – 179	0.62	70	Orange powder

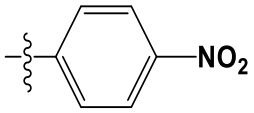
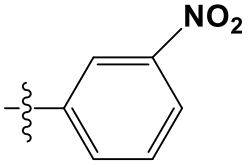
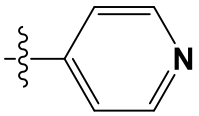
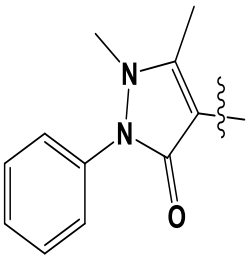
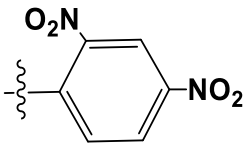
Prepared of 4-((5-(1-argio-4,5-dihydro-1H-tetrazol-5-yl)-2-hydroxyphenyl)diazenyl)-1,5-dimethyl-2-phenyl-1,2-dihydro-3H-pyrazol-3-one(A_7-A_{11}): [19]

A blend of sodium azide (0.01 mole , 0.65gm) as well (0.01 mole) of Schiff base in 15ml DMF was stirred for about four hours then let it cool. As the precipitate Filtered and then recrystallized from petroleum ether to afford the tetrazole derivatives (7-11).
The physical properties were listed within table 2 and the finalization of reaction was checked by TLC.



Schiff baces(A_2-A_6) Tetrazole(A_7-A_{11})

Table 2: The physical properties for compounds (A_7-A_{11})

Comp No.	Structure of Ar	Molecular formula and M.Wt g/mol	M.P (^{0}c)	Yield %	TLC	Color
A_7		$C_{24}H_{21}N_6O_4$ 499	111-114	55	0.63	Orange crystal
A_8		$C_{24}H_{20}N_6O_4$ 499	96-98	63	0.54	White crystal
A_9		$C_{23}H_{20}N6O2$ 481	159-158	60	0.43	Brown Powder
A_{10}		$C_{29}H_{27}N_7O_3$ 504	196-193	55	0.66	Orange powder
A_{11}		$C_{24}H_{19}N_7O_5$ 560	150-149	70	0.71	Orange powder

Biological activity study: [20]

A culture medium from "Molar Hinton Agar" was used in accordance with the directives of the Indian producing business.", The preparation for the current study was performed as follows: dissolved (38gm) of the mentioned agar in one liter of distilled boiled water, then neutralized theacid function of the culture medium at (PH = 7). The produced solution was placed in the sterilization device (Autoclave), at (121) ºC with pressure (150 pounds/ang). After that, pour the culture-produced solution into sterilized glass dishes at a thickness of (0.5-1) for every dish, with decreasing the temperature to (50) Celsisus. To ensure that the medium is deprived of contamination, allow it to harden in the incubator at 37 Celsius for nearly 24 hours.

Numerous types of pathogenic laboratory-isolated bacteria undergo the application activity, diagnosed only four types of these bacteria which are (staphylococcus aurous is gram-positive bacteria, and gram-negative bacteria including (Proteus mirabili, Ecoli, Morganella Morgani) using the drilling method.

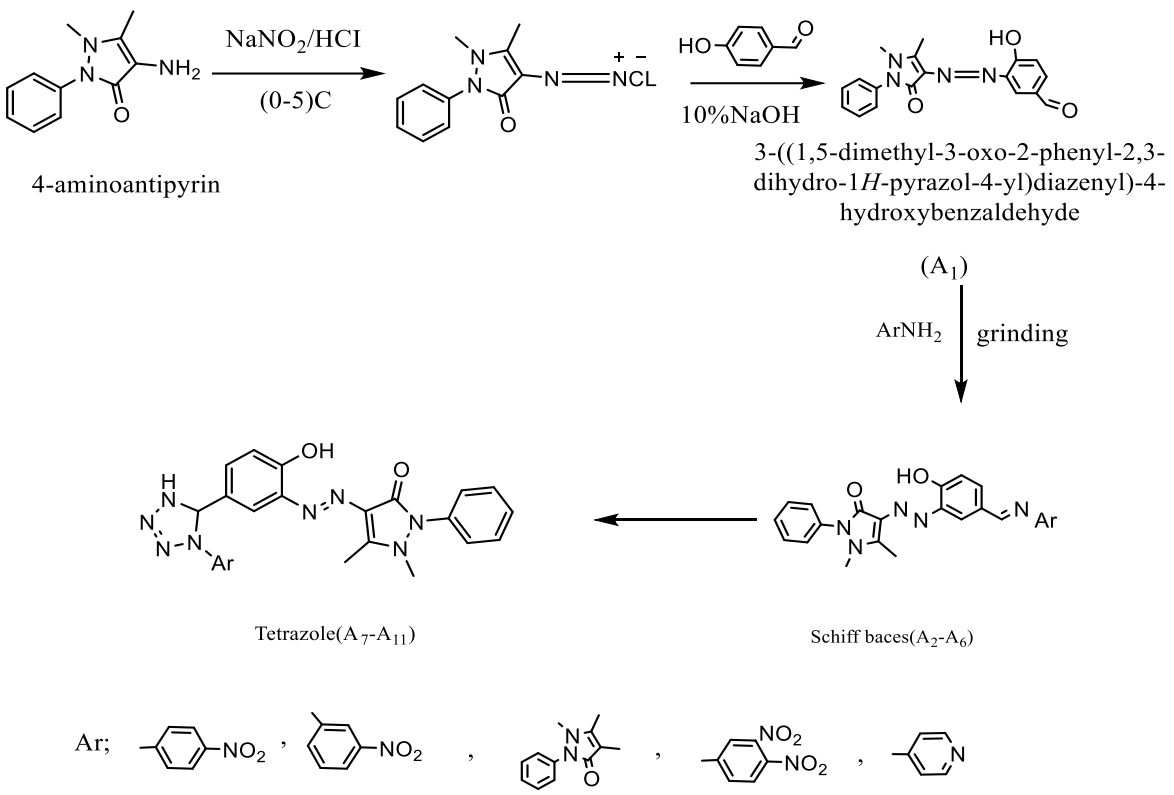
A sterilized cork drill was performed to induce medium holes and spread the selected bacteria on the surface of the dish with a sterile cotton swab utilizing a wiping method. The dishes are placed at a temperature of 37 degrees Celsius for 20 minutes. From the prepared chemical compounds, dissolve the prepared solutions in dimethyl sulfoxide solvent at (200 mg/1ml and 500 mg/1ml) two concentration. Put the produced solution inside the previously prepared holes in a medium with the closed tight dishes utilizing special tape.

Then the prepared dishes are placed inside the incubator at a temperature of 37 degrees Celsius for nearly 24 hours. The inhibition of bacterial growth was measured depending on the Prescott method, using an electronic measuring ruler that is used to measure damping diameter.

Results and Discussion

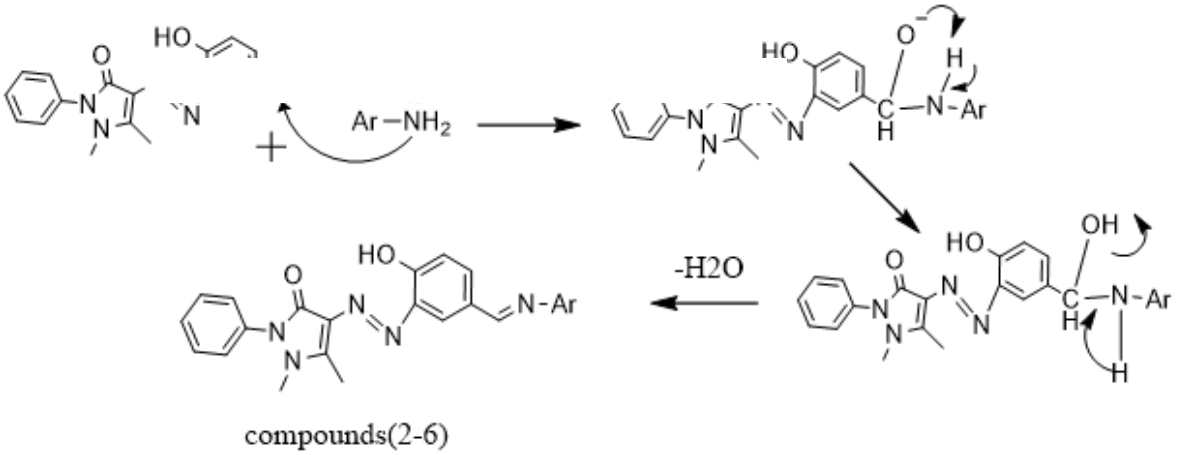
Unusual substituted benzaldehyde (1) was employed as a starting material used to prepare a novel Schiff bases (2-6). The stimulating substance has an excellent functional aldehyde group that may easily react to produce products. [18][20]

Compound (1) was created using the 4-aminoantipyrin reaction and 4-hydroxybenzaldehyde under base condition. All compounds were synthesized through the following synthetic Scheme 1:



Scheme 1: Synthetic pathway for compounds (A_1-A_{11}).

Firstly, Schiff bases (A_2-A_6) were prepared according to the actual suggested Mechanism, as seen in Scheme 2 :[21] [22]

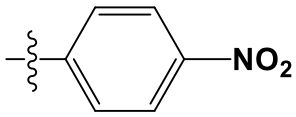
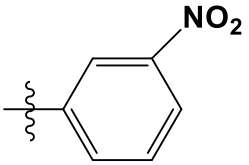
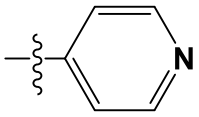
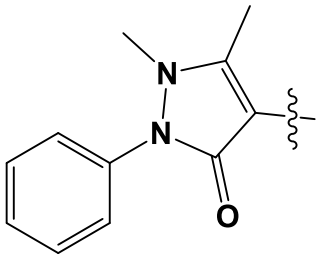
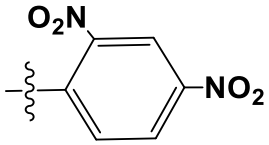


Scheme 2: Mechanism of preparation of Schiff bases.

The spectrum in FT-IR for compound (A_2) indicated an absorption showed up 1422cm^{-1} for N=N azo group moreover, there is an absorption band popped up at 1681 cm^{-1} related to aldehyde carbonyl group as well OH group in phenol showed a peak 3236 cm^{-1}. The resulting material was determined by FT-IR spectrums for Schiff bases (A_2-A_6) gave peaks at (3383 cm^{-1}) for the OH group, N=N azo group (1493- cm^{-1}) and C-N imin (1626- cm^{-1}).[23][24].

All spectroscopic information as shown in table 3 and figures 1 and 2.

Table 3: FTIR spectum for compounds (A_2-A_6)

IR in KBr, γ (cm−1)							
Comp No	Ar	OH	C=C	C=N	N=N	C-O	Other
A_2		3217	1614	1597	1493	1054	NO2 (sym(1383-1337 asym(1566-1512)
A_3		3319	1614	1598	1492	1055	NO2 (sym(1383-1343 asym(1555-1511)
A_4		3383	1560	1626	1489	1068	
A_5		3331	1601	1577	1456	1074	
A_6		3331	1614	1598	1423	1058	NO2 (sym(1331-1384) asym(1531-1512)

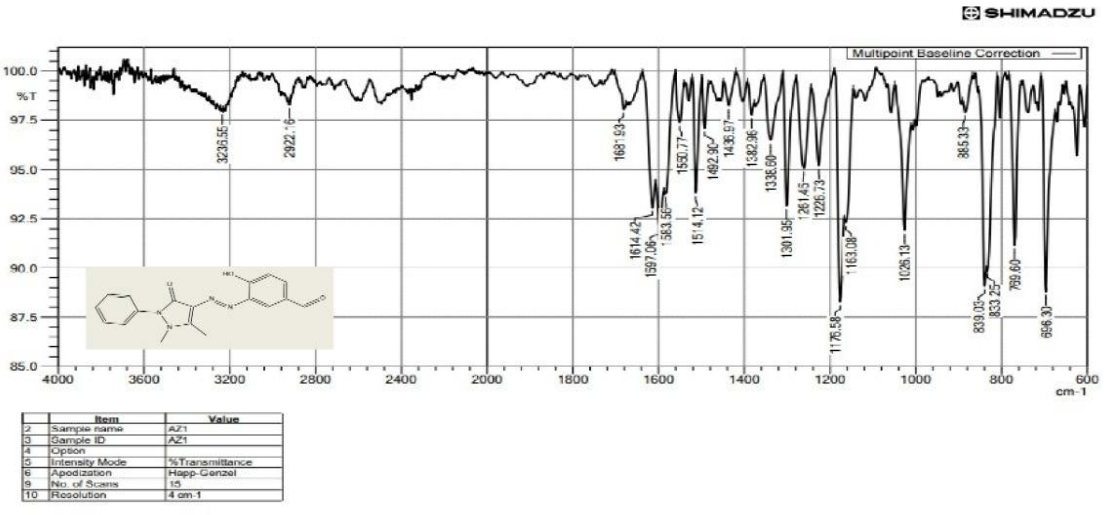


Figure 1: F.TIR spectrum for compound (A_1)

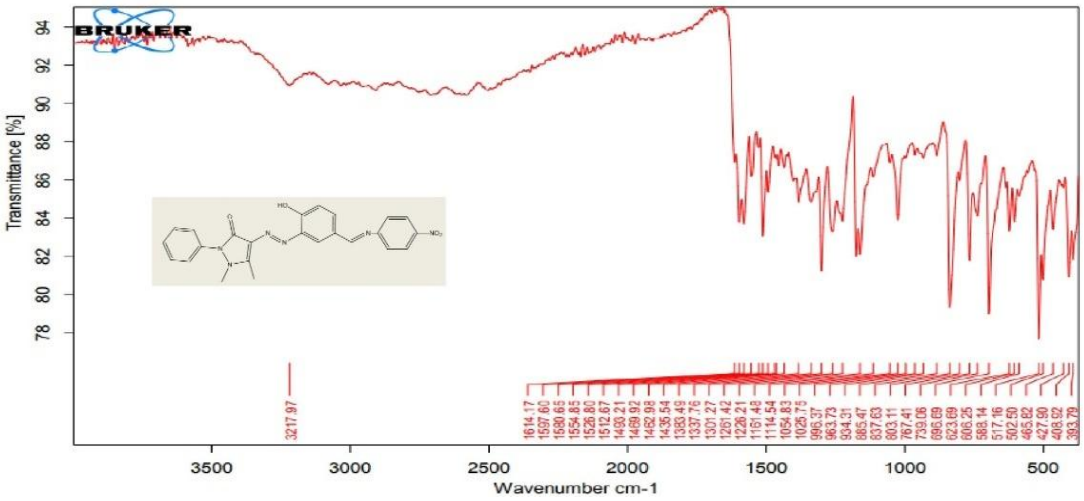
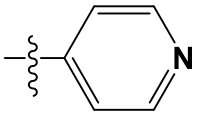
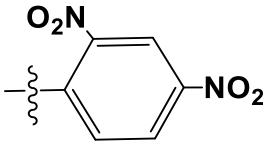


Figure 2: F.TIR spectrum for compound (A_2)

On the other hand, the 1H-NMR spectrum for (A_2) compound showed peaks at 2.44 ppm (s, CH_3, 3H for CH_3 group), 3.15 ppm (s, CH_3, 3H for N-CH_3), 7.35-7.71 ppm (m,8H, at Aromatic ring), 9.44 ppm (s,1H, C=O group), 9.82 ppm (s,1H, OH group) as shown in figure 3 with spectroscopic information in table 4. (Figure 4).

Table 4: 1H-NMR spectra for compounds (A_4-A_6)

Comp. No	Ar	H^1NMR(PPM)-DMSO-d6
A_4		2.44 (s, CH3, 3H for CH3), 3.17 (s, CH_3, 3H for N-CH3), 6.92 (d, CH, 1H for phenol ring), 7.42 (m , CH, 6H for aromatic ring), 7.66 (S,CH,1H for aromatic ring), 8.43 (d, CH, 2H for pyridine ring), 8.88 (s, CH, 1H for imin group), 9.86 (s, OH, 1H for phenol)
A_6		2.44 (s, CH_3,3H for CH3), 3.15 (s, CH_3, N-CH_3), 6.92-7.52 (m, CH,7H for aromatic ring), , 8.45 (d, CH,1H for phenyl ring), 8.87 (s, CH, 1H for Imin group), 9.77 (s, OH,1H for phenol)

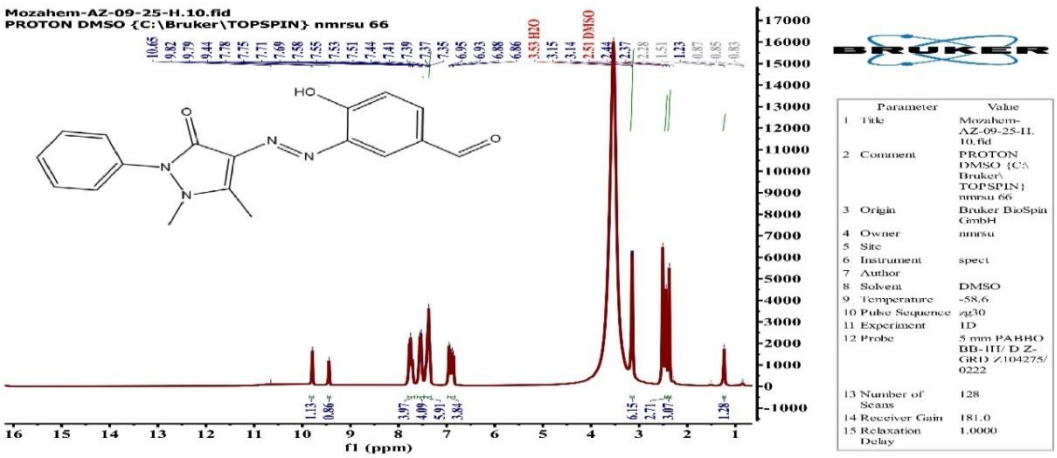


Figure 3: ^{1}H.NMR spectrum for compound (A_1)

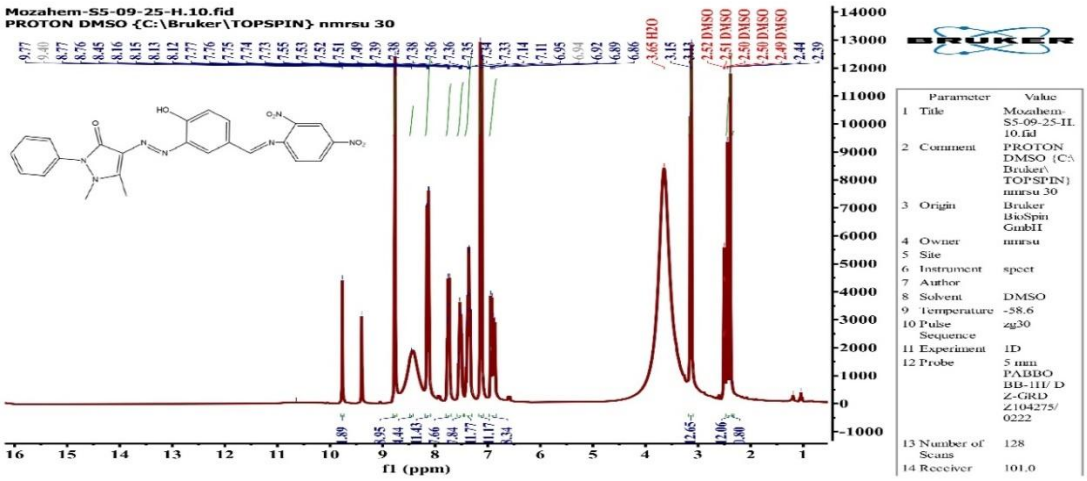
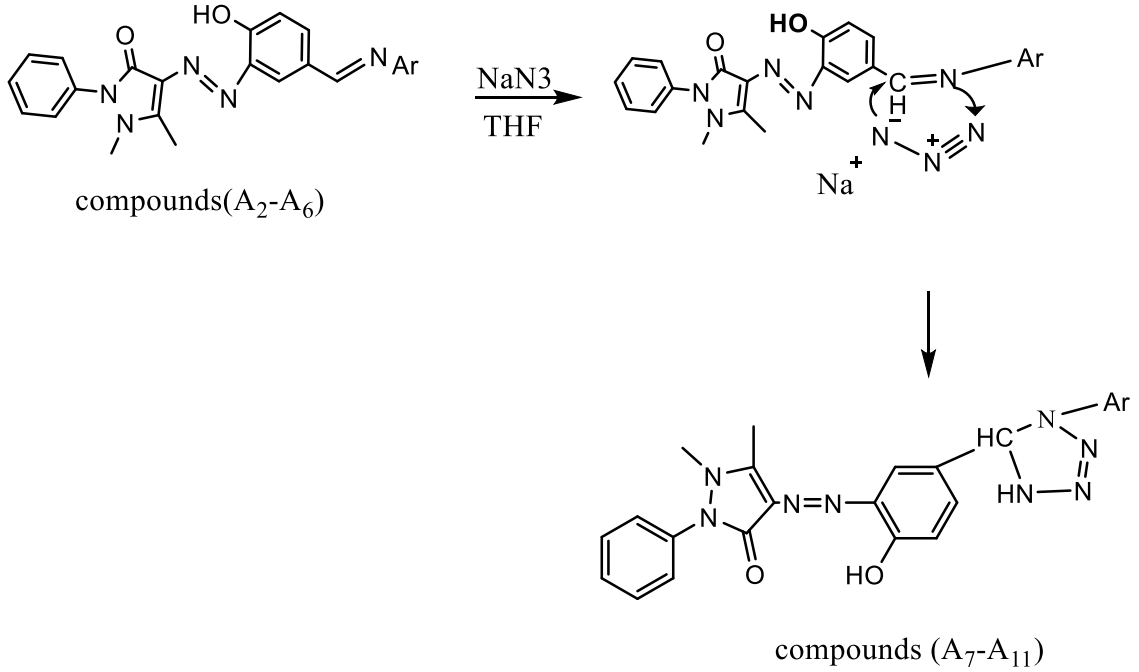


Figure 4: ^{1}H.NMR spectrum for compound (A_6)

Sodium azide was used to convert C=N bond in Schiff bases (A_2-A_6) to tetrazole compounds (7-11) in DMF solvent and reflux for 4 hours under the predicted mechanism as shown in scheme 3 [25].



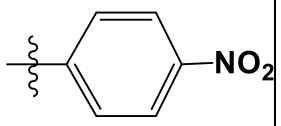
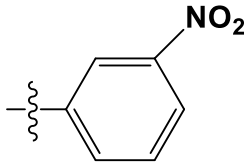
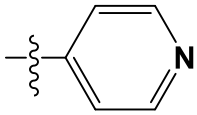
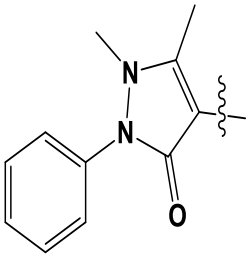
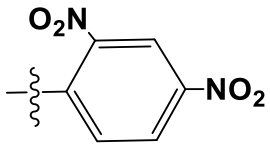
compounds(A_2-A_6)

compounds (A_7-A_{11})

Scheme 3: Mechanism of preparation of Tetrazole.

The resulted compounds were identified by Infrared Spectra which gave spectrum bands for compounds (A_7-A_{11}) at 3580-3253 cm^{-1} for the OH phenol group, N-N 1490-1451 cm^{-1} in tetrazole ring, and appeared band at (3901-3164 cm^{-1}) for N=H tetrazole group. The spectroscopic information is shown in table 5. The ^{1}H-NMR spectra for compounds (A_7,A_{11}) are shown in table 6 and figures 5 and 6.

Table 5: IR Spectrum for compounds (A_7-A_{11})

IR in KBr, γ (cm−1)									
Comp No	Ar	OH	C-Ar Aromatic	C-H Aliphatic	C=C	N=N	N-H Tetrazole	C=O	Other
A_7		3580	3015	2913	1600	1455	3164	1600	NO2 sym(1235-(1270 asym(1509-1523)
A_8		3253	3064	2940	1619	1413	3180	1640	NO2 sym(1226-((1272 asym(1512-1536)
A_9		3301	3084	2361	1626	1451	3901	1640	
A_{10}		3580	3020	2917	1601	1423	3290	1653	
A_{11}		3441	3020	2871	1605	1421	3331	1619	NO2 sym(1223-1260) asym(1511-1578)

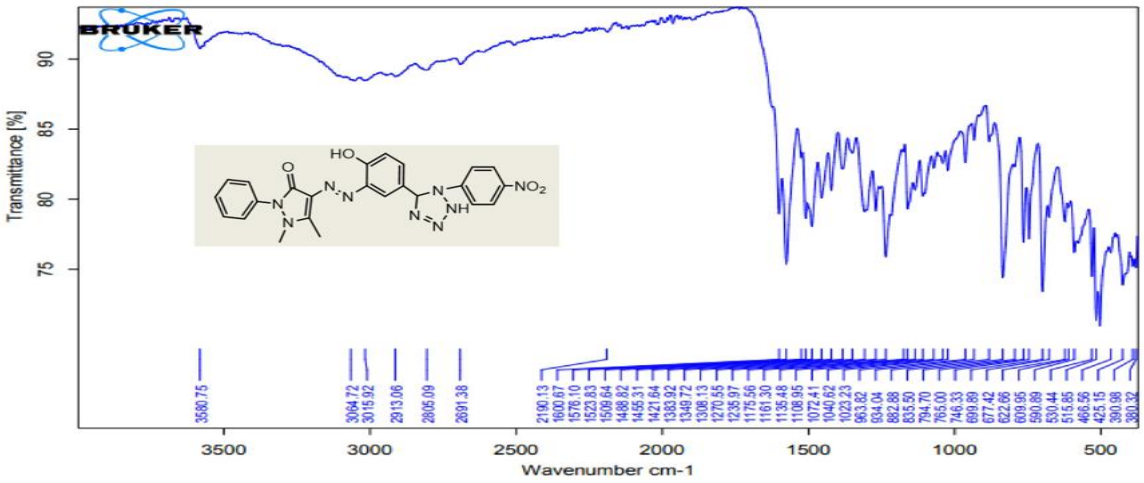
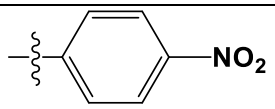
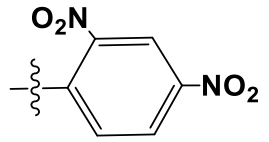


Figure 5: F.TIR spectrum for compound (A_7)

Table 6: ^{1}H-NMR spectra for compounds (A_7,A_{11}) .

Comp No	Ar	HNMR (PPM)-DMSO-d6
A_7		2.44 (s, CH_3, 3H for CH_3 for methyl group), 2.73 (s, CH_3, 3H for N- CH_3), 6.96 (m, CH,1H for phenol ring), 7.29-7,96 (m, CH, 9H for Aromatic ring, 9.97 (s, OH, 1H for phenol group)

		
A_{11}		2.15 (s, CH_3, 3H for CH_3 for methyl), 2.73 (s, CH_3, 3H for N-CH_3), 7.51-8-14 (m, CH, 6H for Aromatic ring), 6.28 (s, CH, 1H for methin group), 3.9 (s, NH, 1H for NH group), , 8.93 (d, CH, 2H for Aromatic ring), 9.45 (s, OH,1H for phenol ring)

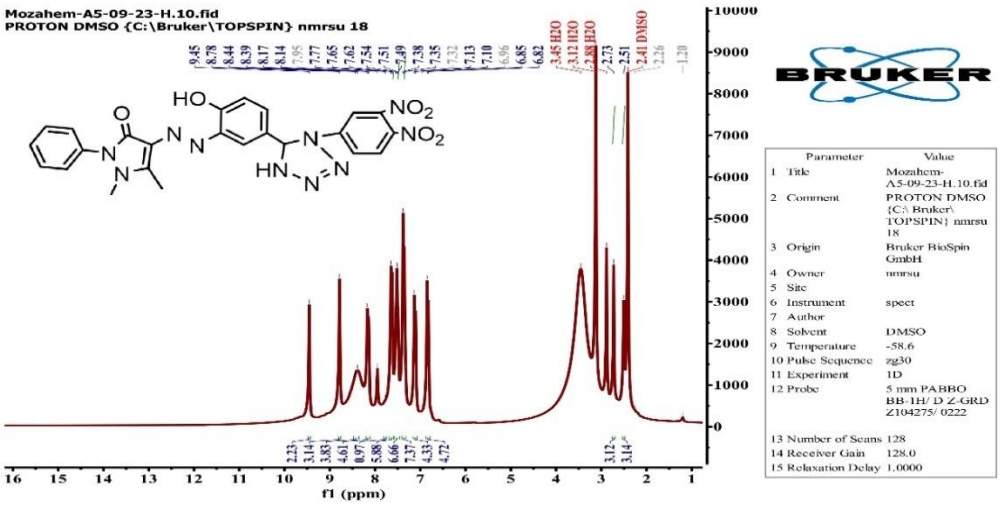


Figure 6: ^{1}H.NMR spectra for compound (A_{11})

Biological activity

The results of the present study demonstrated the increase of various developed types of produced bacteria as an increase in bacterial spread.

The produced bacteria changed their shape to many forms to enable them to prevent the penetration of antibiotics. However, bacterial resistance to antibiotics is considered an important problem that must be studied to develop solutions. Hence the idea came to manufacture several chemical compounds or their derivatives that may have important biological activity that may contribute to the development of a promising drug [26].

In the current study, the biological effectiveness of several prepared compounds (A_1, A_3,A_5, A_9,A_{10}) under study were tested. The activity included several isolated bacteria those were laboratory-diagnosed as pathogenic, namely (staphylococcus aurous), gram-positive bacteria, and gram-negative bacteria, where three types were isolated. Including (Proteus mirabilis, Ecoli, Morganella Morgani) using the drilling method.

The results of the current study revealed that the two compounds (A_{10} and A_5) at 500 mg/1ml were effective against some types of bacteria, in varying degrees, while the other compounds did not have any effectiveness against bacteria. Compound A_{10} registered that the "15" inhibition zone diameters against completely against the E. coli bacteria and it was not effective against other types.

On the other hand, the compound (A_5) was administered with a diameter of 26 against the Gram-positive bacteria Staphylococcus aurous, there was no significant effectiveness was obtained against the other types, the prepared compounds did not show any effectiveness at a concentration of 200 mg /1ml the figures below illustrate this.

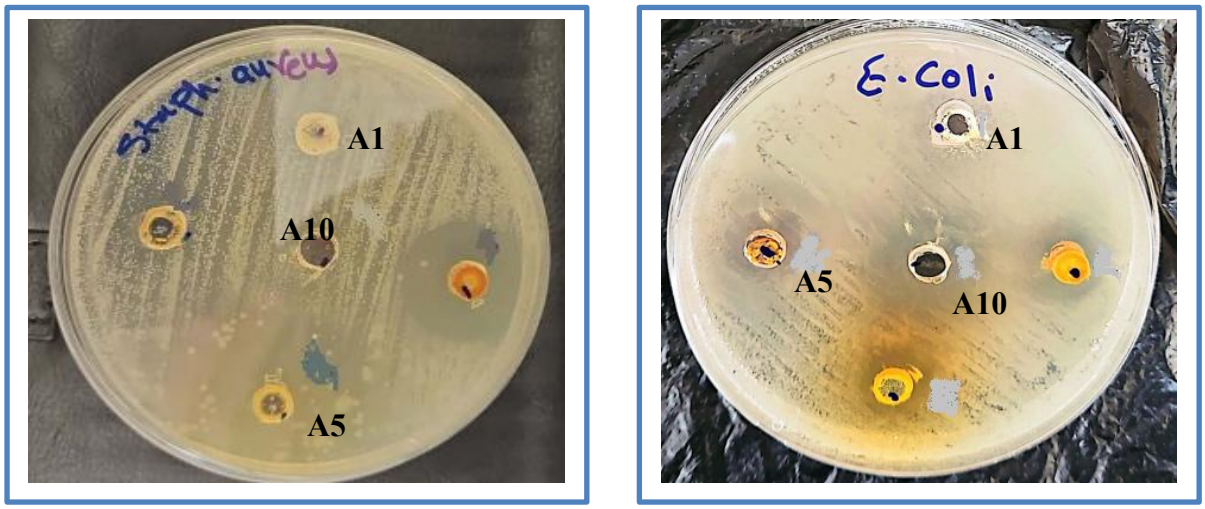


Figure 7: The antibacterial activity of compounds (4-5)

Conclusions

In order to prepare new derivatives of the tetrazole moiety, the current study dealt with several important spectroscopic measurements and studied the biological properties of some of them. The aim of this was to compile the literature, as the researchers reported before, and to present the latest developments on the tetrazole moiety. Through that study, compounds A5 and A10 showed good results againstTwo types of bacteria that cause diseases to humans, namely E.coli and Staph, respectively, as shown in Figure (7). It is possible to continue the biological study of tetrazole compounds active on animals for the purpose of studying their use in studies as promising drugs, and to prepare other derivatives from these lines and study them biologically as well.

Conflicts of interest

No Conflicted interest.

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تحضير وتشخيص والفعالية البايلوجية لمركبات تيترازول جديدة مشتقة من قواعد شيف الآزو

البحث مستل من رسالة ماجستير الباحث الاول

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