RESEARCH ARTICLE

Synthesis and Characterization of Some New (Tetrazole, Thiazolidin-4-one) compounds derived from Drugs and Evaluation of their Biological Activities

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ABSTRACT

In this paper a new series of substituted tetrazole and Thiazolidin-4-one compound were synthesized by three steps. The first step involved the reaction of *p*-hydroxy benzaldehyde with dichloro ethane to result compound (1) . The second step includes reaction of compound (1) with various amino drugs producing the corresponding Schiff bases (2-7) , whereas the third step, involved preparation new tetrazole (8-13) and Thiazolidin-4-one (14-19) derivatives through reaction of the Schiff bases with sodium azide , mercaptoacetic acid respectively. The prepared compounds were characterized by FT-IR, ^{1}H-NMR spectroscopy and their physical properties in addition of study the biological effect for some of them .

Key words: Derivatives, Schiff bases, Tetrazole, Thiazolidine.

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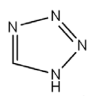
Conflict of interest: None.

INTRODUCTION

Tetrazole (Tetrazacyclopentadiene, 1-*H* Tetrazole) are type of synthetic organic heterocyclic compounds consisting five-member ring of four nitrogen atoms and one carbon atom (plus H). The simplest formula of tetrazole is (CN_4H_2) as shown below:

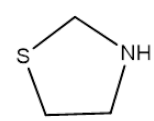


H-tetrazole

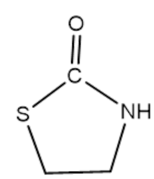
Tetrazole is white to pale yellow solid crystalline, soluble in water and alcohole and acidic in nature belong to presence of five nitrogen atoms.[1,2] Tetrazole and its derivatives are used for biological activities such as: antiviral, antifungal, antibacterial, anti-inflammatory, antitubercolous, antinociceptive, cyclo-oxygenase inhibitors, hypoglycemic and anticancer activities.[3]

Thiazolidines and thiazolidinones are five member ring heterocyclic compounds[4] contain sulfur and nitrogen atoms and three carbon atoms and non-aromatic, they have structure shown below[5]:

The existence of interactive unsaturated ketone group in thiazolidin-4-ones is accountable for their antibacterial, antitubercular, anticonvulsant, analgesic,[6-8] antioxidant, antiparkinson and non- narcotic analgesic activity.[9-11]



Thiazolidine



Thiazolidinone

Accordingly, we wish to report herein the synthesis of compounds which possesses a chemically significant nitrogen heterocyclic nucleus tetrazole and thiazolidine-4-one.

MATERIALS AND METHODS

All chemicals utilized were of analytical degree and used without further purification.

Instrumentation

Melting points were registered with Stuart Melting Point apparatus. Infrared spectra Fourier-transform infrared spectroscopy (FTIR) were recorded on (Shimadzu-8300 spectrophotometer) in Ibn Sina State Company (ISSC). hydrogen-1 nuclear magnetic resonance (^{1}HNMR) spectra were recorded on a (Bruker-400 MHz) by using tetra methyl silane (TMS) as inner standard in (DMSO-d_6 solvent), AL-albayt University-Jordon. C.H.N.S. micro elemental analysis

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was measured by using a device (Euro Vectro-3000A Element Analyzer)/Ibn Al-Haitham College, University of Baghdad. The biological study was measured in Central Environmental Laboratory, College of Science, University of Baghdad, Baghdad, Iraq.

Methods

Synthesis of [4.4-ethane-1.2-diyl-1-bis(oxy)-dibenzaldehyde] (1)[12]

A mixture of *p*-hydroxy benzaldehyde (4gm, 0.033mole) and dichloro ethane (12.4gm, 0.066mole) was mixed in (20mL) absolute ethanol then anhydrous sodium carbonate (7gm,0.066mole) added, the mixture was refluxed with stirring for 3hours. The mixture was cooled and filtered, the resulting precipitate was dried and recrystallized from ethanol.

Synthesis of Schiff Bases (2–7)[13]

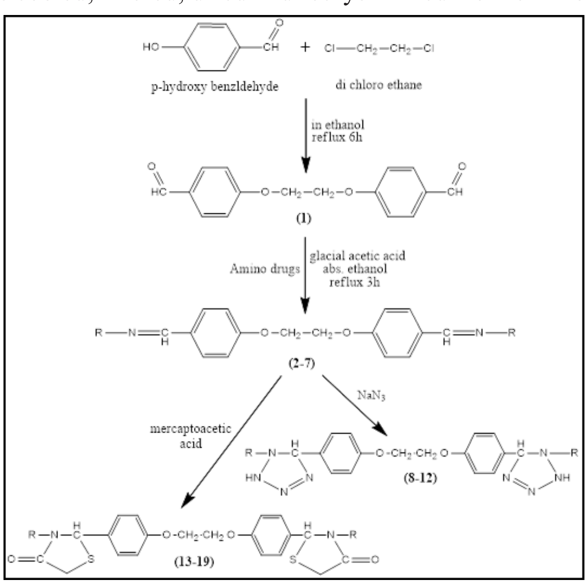
Compound (1) (0.29gm, 0.001mole) and (0.001mole) from various amino drugs {2-amino benzothiozole, Trimethoprim, Metoclopramide, Sulfamethoxazole, 4-amino antipyrine (Ampyrone)} was mixed and dissolved in (15mL) absolute ethanol, (3) drops of glacial acetic acid was added then refluxed for 6 hours. The resulting precipitate was cooled, filtered, dried and recrystallized from ethanol.

Synthesis of Tetrazole derivatives (8–12)[14]

Schiff bases (1.06gm, 0.002mole) was dissolved in (20ml) tetrahydrofuran and mixed with (0.26gm, 0.004mole) sodium azide. The mixture refluxed in water path at 50–60°C for 8–12 hours. The precipitate was cooled, filtered, dried and recrystallized from ethanol.

Synthesis of Thiazolidin-4-one derivatives (13–19)[15]

To a solution of Schiff bases (0.53gm, 0.001mole) in (15mL) tetrahydrofuran (THF); (0.13mL, 0.002mole) mercaptoacetic acid and a pinch of anhydrous zinc chloride ($ZnCl_2$) added and refluxed in water path for 16-18 hours. The separated precipitate was cooled, filtered, dried and recrystallized from ethanol.



Scheme 1: Synthetic route of preparation compounds

RESULT AND DISCUSSION

In the present work novel substituted tetrazole and thiazolidine-4-one compounds was prepared.The new derivatives following the reaction sequence depicted in Scheme 1, and was characterized and screened for their biological activity.

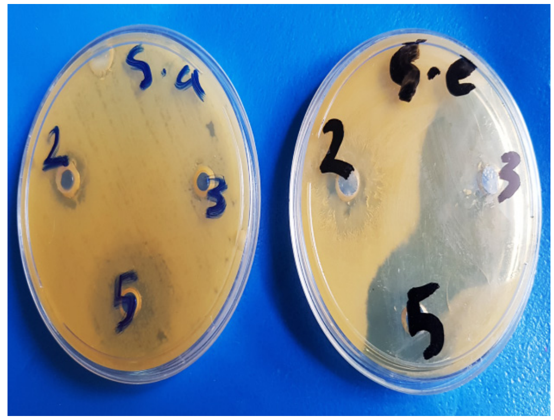


Figure 1: The effect of (C_2,C_3,C_5) on *S. aureus* and *S. epidermidis*

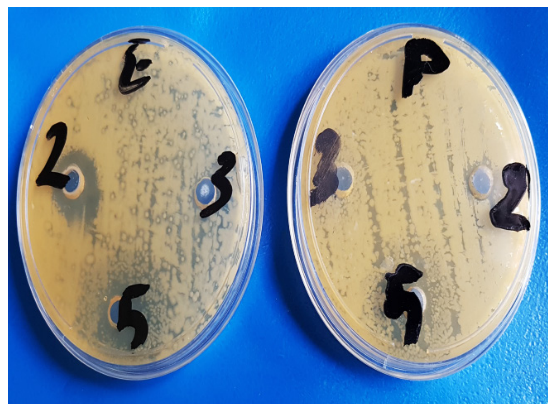
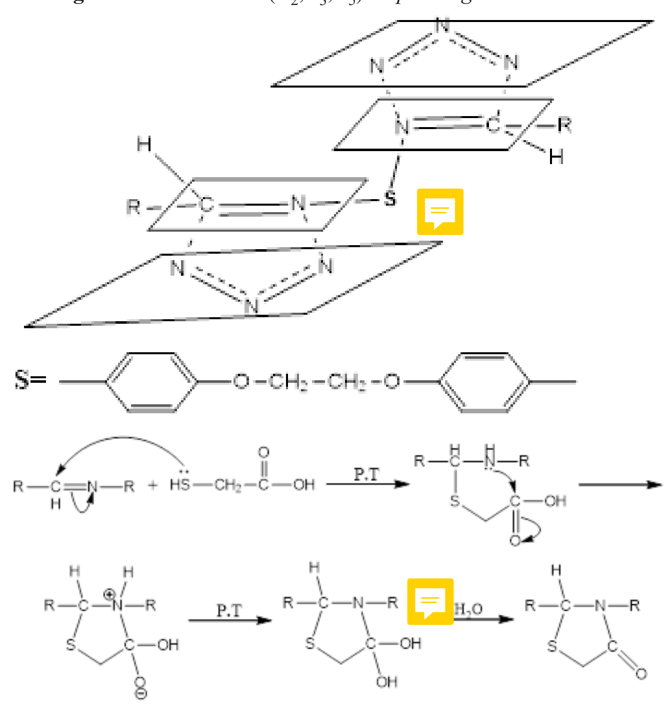


Figure 2: The effect of (C_2,C_3,C_5) on *p. aeruginosa* and *E. coli*



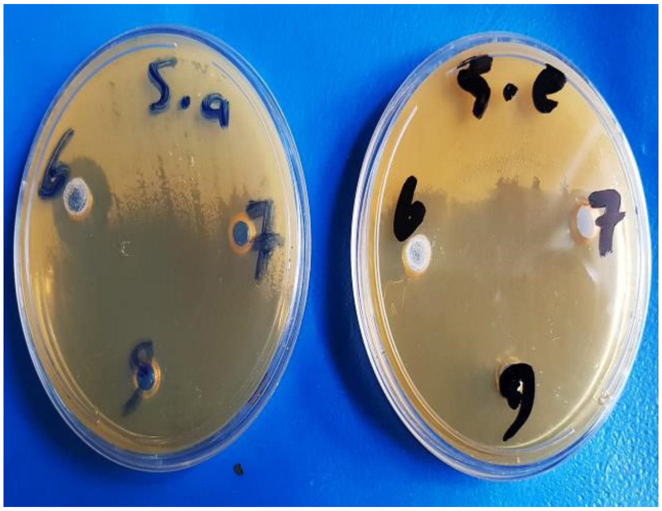


Figure 3: The effect of (C_6,C_7,C_9) on *S. aureus* and *S. epidermidis*

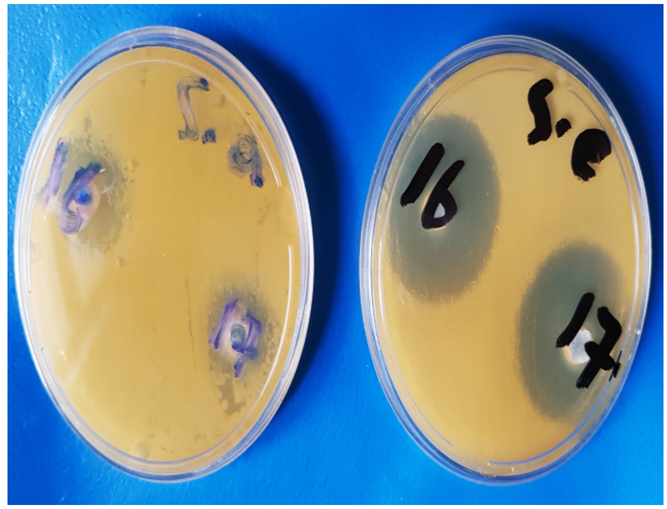
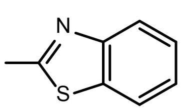
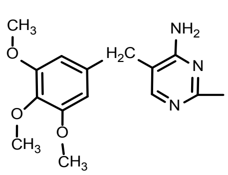
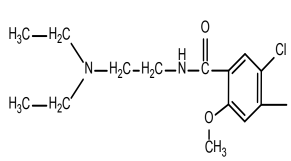


Figure 5: The effect of (C_{16},C_{17}) on *S. aureus* and *S. epidermidis*

Figure 4: The effect of (C_6,C_7,C_9) on *p. aeruginosa* and *E. coli*

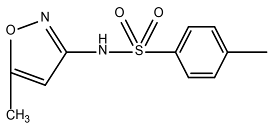
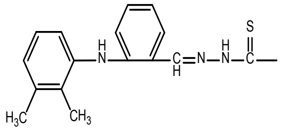
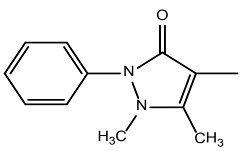
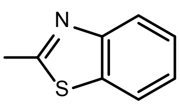
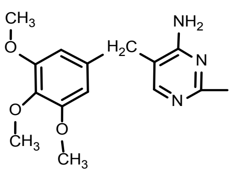
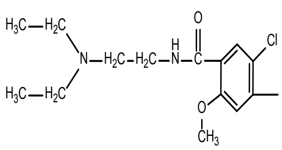
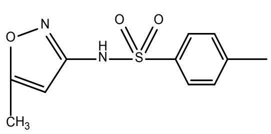
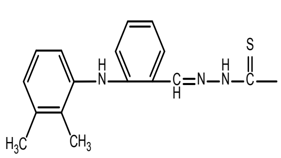
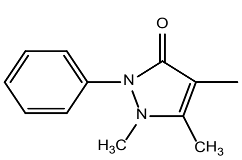
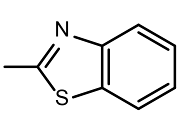
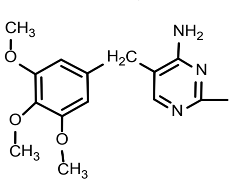
Figure 6: The effect of (C_{16},C_{17}) on *p. aeruginosa* and *E. coli*

Table 1: Molecular formula, physical properties and elemental analysis for all compounds.

Comp. No.	*-R*	*Molecular Formula M.wt.(gm/mol)*	*Color*	*M.P°C*	*Yield %*	*Elemental micro analysis found (Calc.) %*			
						C%	*H%*	*N%*	*S%*
1	-	$C_{16}H_{14}O_4$ 270.28	Beige	280–282	71	71.32 (71.10)	5.70 (5.22)	-	-
2		$C_{30}H_{22}N_4O_2S_2$ 534.65	Yellow	110–112	85	67.70 (67.39)	4.45 (4.15)	10.22 (10.48)	12.07 (11.99)
3		$C_{44}H_{46}N_8O_8$ 814.88	Beige	177–179	56	64.98 (64.85)	5.85 (5.69)	13.88 (13.75)	-
4		$C_{42}H_{50}Cl_2N_6O_4$ 773.79	Yellow	118–120	65	65.41 (65.19)	6.73 (6.51)	11.03 (10.86)	-

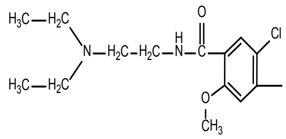
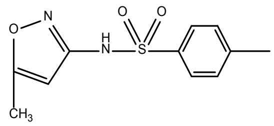
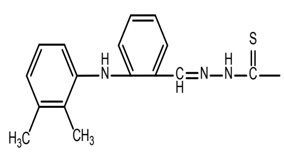
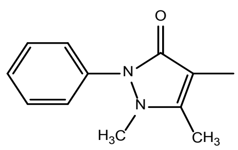
Cont.

Cont.

5		$C_{36}H_{32}N_6O_8S_2$ 740.80	Yellow	202–204	82	58.62 (58.37)	4.57 (4.35)	11.55 (11.34)	8.82 (8.66)
6		$C_{48}H_{46}N_8O_2S_2$ 831.06	Yellow	160–162	83	69.44 (69.37)	5.70 (5.58)	13.64 (13.48)	7.95 7.72
7		$C_{38}H_{36}N_6O_4$ 640.73	Yellow	203–205	64	70.02 (71.23)	5.81 (5.66)	12.94 (13.12)	-
8		$C_{30}H_{24}N_{10}O_2S_2$ 620.71	White	304–306	72	58.33 (58.05)	4.07 (3.90)	22.42 (22.57)	10.40 (10.33)
9		$C_{44}H_{48}N_{14}O_8$ 900.94	White	321–323	60	58.81 (58.66)	5.55 (5.37)	21.90 (21.77)	-
10		$C_{42}H_{52}Cl_2N_{12}O_4$ 859.85	White	285–287	69	58,80 (58.67)	6.26 (6.10)	19.67 (19.55)	-
11		$C_{36}H_{34}N_{12}O_8S_2$ 826.86	Biege	226–228	73	52.39 (52.29)	4.22 (4.14)	20.50 (20.33)	7.81 (7.76)
12		$C_{48}H_{50}N_{20}O_2S_2$ 1003.17	Biege	331–333	70	57.60 (57.47)	5.13 (5.02)	28.06 (27.92)	6.55 (6.39)
13		$C_{38}H_{38}N_{12}O_4$ 726.79	White	320-322	67	62.95 (62.80)	5.33 (5.27)	23.30 (23.13)	-
14		$C_{34}H_{26}N_4O_4S_4$ 682.85	White	241–243	74	59.92 (59.80)	4.01 (3.84)	8.39 (8.20)	18.90 (18.78)
15		$C_{48}H_{50}N_8O_{10}S_2$ 963.09	Beige	240–242	75	60.04 (59.86)	5.37 (5.23)	11.71 (11.63)	6.79 (6.66)

Cont.

Cont.

16		$C_{46}H_{54}C_{12}N_6O_6S_2$ 921.99	Beige	230–232	80	60.12 (59.92)	6.08 (5.90)	8.96 (9.12)	7.07 (6.96)
17		$C_{40}H_{36}N_6O_{10}S_4$ 889.01	White	221–223	68	54.22 (54.04)	4.19 (4.08)	9.52 (9.45)	14.60 (14.43)
18		$C_{56}H_{54}N_8O_6S_6$ 1127.47	White	234–236	72	59.78 (59.66)	4.95 (4.83)	9.97 (9.94)	17.15 (17.06)
19		$C_{42}H_{40}N_6O_6S_2$ 788.93	Beige	217–219	84	64.10 (63.94)	5.22 (5.11)	10.74 (10.65)	8.25 (8.13)

The FTIR spectrum of compound 1 shows the frequency of (C=O) groups at 1654 cm^{-1}, 1681 cm^{-1},and absorption band at (3062)cm^{-1} due to stretching vibration of (C-H) aromatic ring, other bands are shown in Table 2.

Reaction of compound 1 with diverse amino drugs leads to obtain Schiff bases (2–7).

The FTIR spectrum of Schiff base (3), Figure 7, shows absorption band at (3425, 3448) cm^{-1} due to the asymmetric and symmetric stretching vibration of the (NH_2) group and appearance band at (1662)cm^{-1} for (C=N) group. These bands and other compounds bands are shown in Table 2.

Tetrazole derivatives (8-12) was obtained by refluxing of Schiff bases with sodium azide. The mechanism for this reaction is a cyclo addition that called [1-3 dipolar cyclo addition reaction]. It is include the addition of unsaturated group (dipolarphiles) to 1-3 dipoles , a molecule handling resonance contributors that a positive and negative charge were placed in (1,3-position) relative to each other, as shown below:[16]

Compound (10), Figure 10, shows characteristic band at: (2129)cm^{-1} due to (=N-N=C-) azide group and other absorption bands at: (1639)cm^{-1} for (C=N),(640)cm^{-1} for (C-Cl) and (1558) cm^{-1} for (C=C) aromatic ring, these are listed in Table 2.

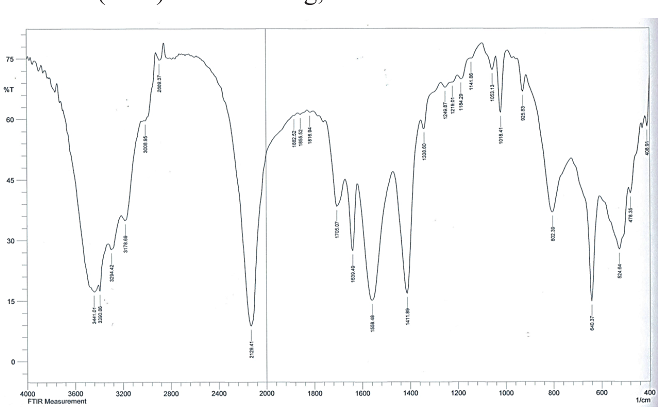


Figure 7: The FTIR spectrum of (C_3)

The other route, reaction of Schiff bases with mercaptoacetic acid in (THF) produced Thiazolidin-4-one compounds. The proposed mechanism of this reacton is shown below:

The FTIR spectrum of thiazolidine compound,[17] Figure 11 shows multiple bands at : (1226)cm^{-1} for (C=S) , (1705) cm^{-1} for (C=O) amide and at (3417, 3468) cm^{-1} due to the stretching vibration for NH_2 group.

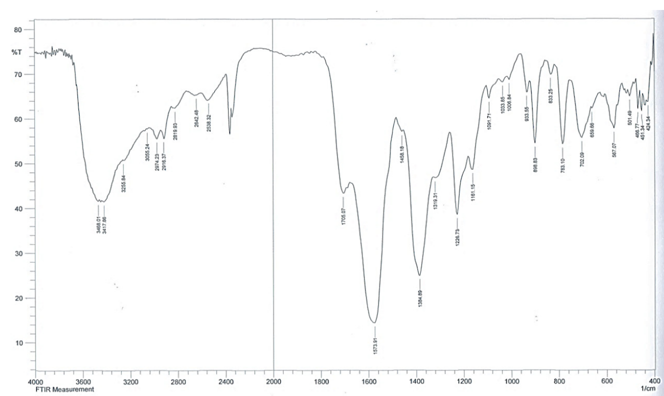


Figure 8: The FTIR spectrum of (C_4)

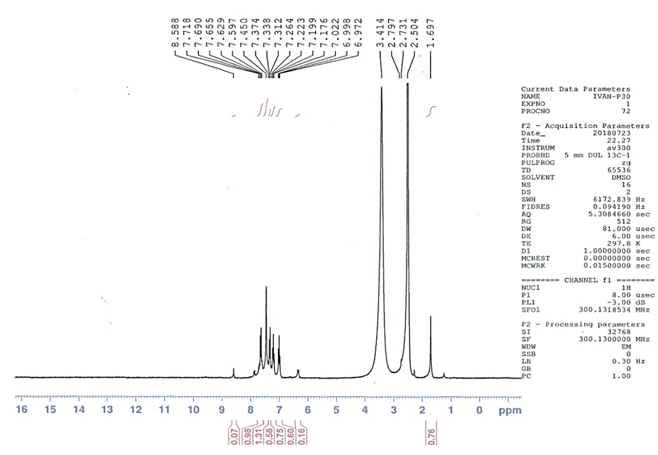


Figure 9: The FTIR spectrum of (C_7)

Table 2: FTIR spectral data of all compounds.

Comp. No.	*υ(C=O) cm^{-1}*	*υ(C=N) cm^{-1}*	*υ(C=C) cm^{-1}*	*υ(C-H)cm^{-1} aliphatic*	*υ(C-H)cm^{-1} aromatic*	*υ(C-N) cm^{-1}*	*υ(C-O) cm^{-1}*	*Other Band cm^{-1}*
1	1681 1654	-	1543 1593	2831 3062	3236	-	1300,1315 1373,1388	-
2	-	1643	1531 1562	2835 2900	(3128- 3275)	1415,1446	1284 1311	υ2(C-S) 721, 740
3	-	1662	1508 1593	(2831- 2958)	3155	1423,1469	1238 1334	υ(NH_2) 3425, 3448
4	1681	1635	1558 1585	(2800- 2974)	3224	1411 1446	1253 1292	υ(C-Cl) 817 υ2(-NH) 3325, 3402
5	-	1681	(1504- 1597)	(2754- 3008)	(3143- 3298)	1415 1469	1265 1311	υ2(-NH) 3379, 3468 υ2(-SO_2) (1145-1157), (1365-1366) υ2(C-S) 640,682
6	-	1651	(1512- 1577)	(2738- 2820)	(3159- 3294)	1412 1450	1257 1334	υ2(C=S) 1018, 1161 υ2(-NH) 3398, 3487
7	1708	1647	1585	(2808- 2989)	(3047- 3182)	1411 1496	1276 1357	υ(N-N)
8	-	1639	1562	2889	(3012- 3174)	1411	1246 1338	υ(-NH) 3390, 3437 υ(C-S) 640 υ(=N-N=C-)azide 2133
9	-	1640	1566	2877	(3024- 3189)	1415 1446	1288 1334	υ(NH_2) 3390, 3452 υ(=N-N=C-)azide 2122
10	1705	1639	1558	2889	(3008- 3178)	1411	1249 1338	υ(-NH) 3390,3441 υ(C-Cl) 640 υ(=N-N=C-)azide 2129
11	1693 1705	1639	1562	2893	(3039- 3182)	1420	1243 1330	υ(-NH) 3392, 3451 υ(C-S) 642 υ(-SO_2) (1155-1360) υ(=N-N=C-)azide 2130
12	1691	-	1577	2889	3120	1434	1219 1350	υ(-NH) 3466 υ(N-N) 1525 υ(C=S) 1039
13	1685	1636	1581	2935	(3010- 3170)	1415	1253 1332	υ(-NH) 3390, 3448 υ(N-N) 1510 υ(=N-N=C-)azide 2110
14	1680	1620	1572	2890	3122	1426	1330	υ(C-S) 630
15	1719	1641	1560	2932	3152	1440	1343	υ(NH_2) 3460 υ(C-S) 640
16	1723	-	1545	2988	3165	1430	1322	υ(C-Cl) 790 υ(C-S) 589
17	1705	-	1573	2819-2974	3055	1458	1226 1319	υ(-NH) 3417,3468 υ(-SO_2) 1161,1384 υ(C-S) 702
18	1690	-	1551	2821-2940	3112	1447	1360	υ(-NH) 3455 υ(C=S) 1050 υ(C-S) 665
19	1715	-	1589	2890	3151	1421	1333	υ(N-N) 1550 υ(C-S) 680

^{1}HNMR spectral data of Schiff base (2) showed signals at (δppm): (1.7) due to (CH) imine, (3.5) due to (CH_2-O) and multiple peak (6.4-7.9) due to protons of aromatic rings as shown in Figure 12.

Figure 13 for Schiff base (3) shows the following characteristic chemical shifts at (δppm): (1.9) due to (CH_2), (3.5) due to (CH) imine , (3.7) for (CH_2-O) , (3.8) due to (CH_3-O), (5.8) due to (NH_2) and finally multiple peak at (6.2–7.5) for protons of aromatic rings.

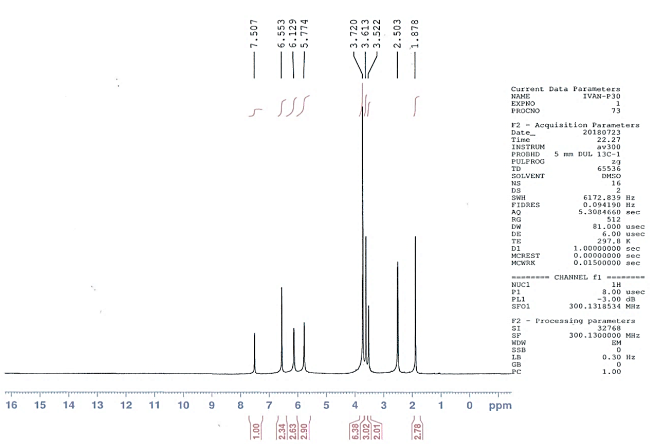


Figure 10: The FTIR spectrum of (C_{10})

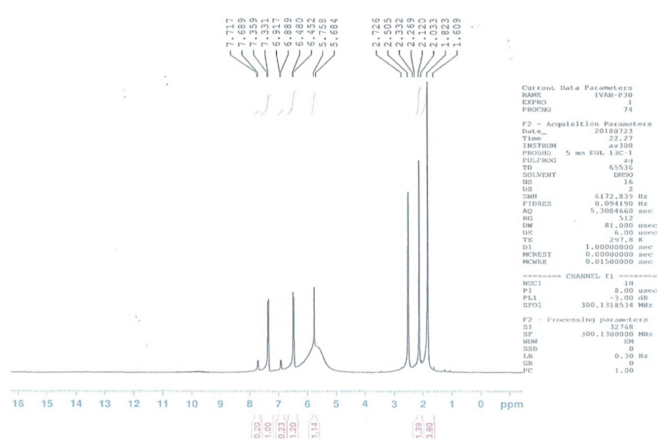


Figure 11: The FTIR spectrum of (C_{17})

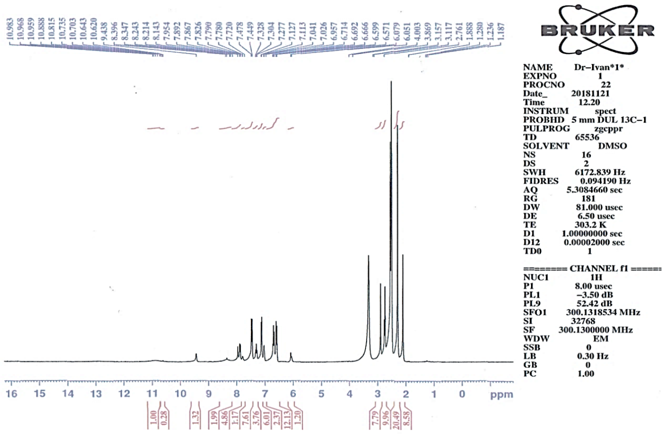


Figure 12: The^{1}HNMR spectrum of (C_2)

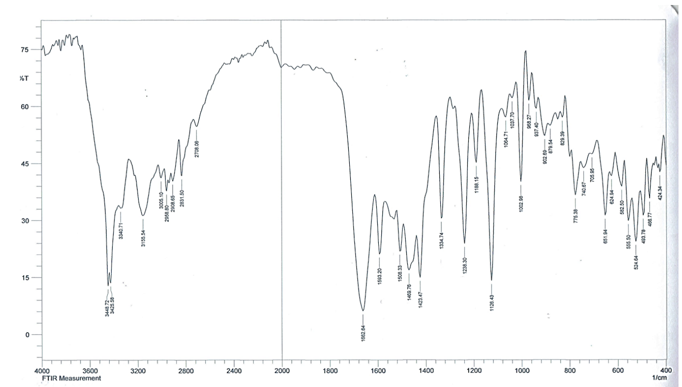


Figure 13: The^{1}HNMR spectrum of (C_3)

The ^{1}HNMR spectrum of Schiff base (5) showed in Figure 14.

^{1}HNMR spectrum of thiazolidine compound,[16] Figure 15 showed signals at (δppm): 2.1 belongs to (CH) thiazolidin-4-one ring, 2.3 for 2(CH_3), 2.6 due to 2(N-CH_2), 2.8 for (CH_2-N), 2.9 for(CH_2) near amide group, 3.4 belong to (O-CH_2), 6.1 for (NH) and (6.5-7.9) for protons of aromatic rings.

Table 3 shows ^{1}HNMR spectral data for some compounds mentioned earlier.

Antibacterial activity study[17]

Some of new synthesized compounds were investigated according to the (disk diffusion method) against two strain gram +ve bacteria (*Staphylococcus aureus*, *Staphylococcus epidermidis*) and two strain gram -ve bacteria (*Pseudomonas aeruginosa*, *Escherichia coli*). From the results below we note that (C_9) has the highest activity against two strains of bacteria. Compound (C_2) has weak activity against *P. aeruginosa* and moderate activity against each of : *S. aureus*, *S. epidermidis* and *E. coli*, while (C_3) has activity just against *S. epidermidis*.

Each of (C_5) and (C_6) have moderate activity against strain gram –ve bacteria and high activity against strain gram +ve bacteria, while (C_7) has activity against just strain gram +ve bacteria.

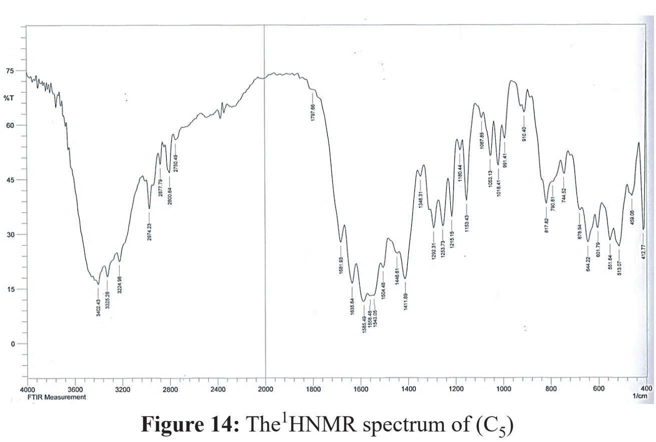


Figure 14: The^{1}HNMR spectrum of (C_5)

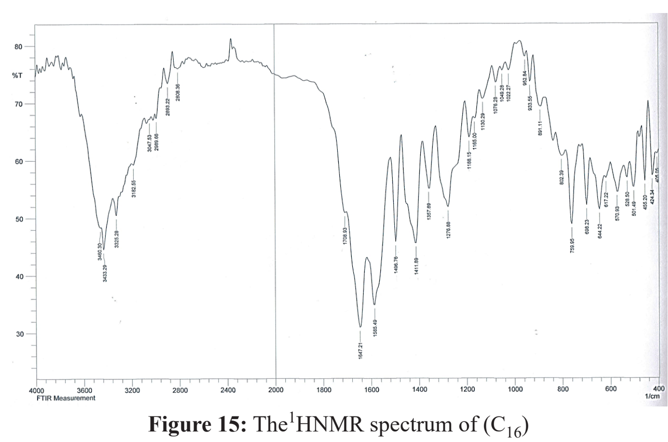


Figure 15: The^{1}HNMR spectrum of (C_{16})

Table 3: ^{1}HNMR data for compounds (C_2, C_3, C_5, C_{16}).

Comp. No.	*^{1}HNMR spectral data δ ppm*
2	1.7(s,1H,CH imine), 3.5(s,2H,CH_2-O), 6.4-7.9(m,8H,Ar-H)
3	1.9(s,1H,CH_2), 3.5(s,1H,CH imine), 3.7(s,2H,CH_2-O), 3.8(s,9H,CH_3-O), 5.8(s,2H,NH_2), 6.2-7.5(m,7H,Ar-H)
5	1.8(s,3H,CH3 imidazole), 2.5(s,2H,CH_2-O), 5.5(b.s,1H,NH), 6.4(s,1H,CH imine) 6.8-7.8(m,8H,Ar-H)
16	2.1(s,1H,CH thiazolidin-4-one ring), 2.3(s,6H,2CH_3), 2.6(s,4H,2N-CH_2), 2.8(s.2H,CH_2), 2.9(s,2H,CH_2 near amide group), 3.4(s,2H,CH_2-O),6.1(s,1H,NH), 6.5-7.9 (m,7H,Ar-H)

Table 4: Antibacterial activity of some prepared compounds.

	Gram positive		*Gram negative*	
Comp. No.	*Staphylococcus aureus*	*Staphylococcus epidermidis*	*Pseudomonas aeruginosa*	*Escherichia coli*
C_2	12	12	9	15
C_3	-	30	-	-
C_5	22	30	11	12
C_6	17	20	15	15
C_7	15	20	-	-
C_9	40	40	40	40
C_{16}	19	25	16	22
C_{17}	18	25	16	23

Compounds (C_{16}) and (C_{17}) have high activity against *S. epidermidis* and *E. coli* and moderate activity against *S. aureus* and *P. aeruginosa*.

The results of the examined compounds has been registered in Table 4.

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