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Synthesis, characterization, and antimicrobial activity of some thiazole derivatives

Tahseen Saddam Fandi Al-Mathkuri[a]*, Asmaa B.Sabti[b] and Ali. A. Raheem[c]

Department of Chemistry, College of Science, University of Misan, Misan, 62001. Iraq

Abstract

Thiazole derivatives **2a-c** were synthesized via reaction of 2-chloro-*N*-phenylacetamide (**1**) and thiourea, thiosemicarbazide or thioacetamide in alcoholic potassium hydroxide. Also, substituted benzaldehyde reacted with thiosemicarbazide at reflux temperature in ethanol to afford hydrazone compounds **3a-k**. Under microwave irradiation (M.I., 100W) with neat for 20sec; arylketones, thiosemicarbazide and chloroacetylchloride were reacted to afford thiazole derivatives **4a-j**. Compounds are characterized by physical measurements, NMR and infrared spectra. Antibacterial activity of new compounds was evaluated with Gram Negative *Serratiamarcesence, Proteus vulgaris, Escherichia coli and Psudomonas aeruginosa* and Gram Positive; *Staphylococcus aureus* in comparison with Norfloxacin and Ciprofloxacin as standard drugs.

Keywords: Hydrazone, thiazole, Microwave, Antibacterial.

Introduction

Several heterocyclic sulfur and nitrogen-containing compounds have been studied. Compounds containing the thiazole core have attracted great interest due to their presentation as antibacterial, [1, 2] antifungal, [3] anticancer, [4] antimalarial, [5] antiviral, [6] analgesic, [7] and diuretic. [8] Activities (Fig. 1). Besides, it is also known that heterocyclic compounds bearing thiazoles, which are part of their molecular structure, possess a wide range of biological potential [9]. In the same way, hydrazones have antimicrobial activity with a wide spectrum of action, effective against Gram-positive and some Gram-negative bacteria. [10] Modifications to the heterocyclic core have resulted in a large number of compounds with diverse pharmacological activities. Therefore, all the current works aim at the synthesis of a new series of heterocyclic cycles, containing thiazole and hydrazine derivatives and also the in vitro antibacterial activity against pathogenic bacteria strains has been studied.

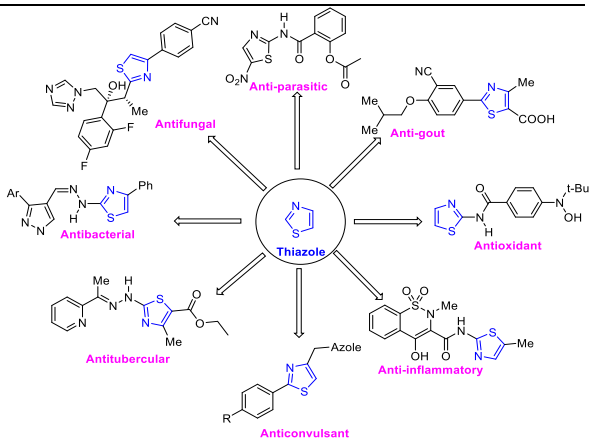

Figure 1: Biologically active compounds that contain thiazole moiety

Experimental

Chemistry

All chemicals were supplied by either Fluka or Aldrich chemical companies and were used without further purification. All melting points are uncorrected and were taken in open capillary tubes using Electrothermal apparatus 9100. **FT-IR** spectra were recorded with a Perkin-Elmer Frontier. Routine **NMR** spectra were recorded at room temperature on a Bruker Avance TM 400 spectrometer as solutions in dimethyl sulfoxide (DMSO) All chemical shifts

*Corresponding author e-mail: tahseensaddam@uomisan.edu.iq(Tahseen Saddam Fandi).
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are quoted in δ relative to the trace resonance of protonateddimethyl. Follow up of the reactions and checking the purity of the compounds were made by Thin Layer Chromatography (TLC) on silica gel-precoatedaluminum sheets (Type 60, F 254, Merck, Darmstadt, Germany)with eluent of methanol: chloroform (9:1). The prepared compounds were purified using suitable solvents.

Synthesis of 2-chloro-*N*-phenylacetamide (1)

Aniline (0.12 mol) and chloroacetyl chloride (0.12 mol) were reacted at room temperature in a clean state for 1 hour to obtain compound 1 in a good yield and recrystallized from ethanol.

Synthesis of thiazole derivatives 2a-c

An equimolar 2-chloro-*N*-phenylacetamide (**1**) and thiourea, thiosemicarbazide or thioacetamide with reflux in ethanol for 5h (in KOH). The reaction mixture is poured onto ice grits for precipitation, then dried and recrystallized in a suitable solvent to afford compounds **2a-c**.Some Physical properties of compounds **2a-c** were subjected at Table (1).

Table 1: Physical features of substances 2a-c

Com.	color	Yield%	M.P. °C	R.F.	Solvent	Eluent: MeOH:$CHCl_3$
1	Brown	13	125-130	0.45	Ethanol	1:9
2a	yellow	71	160-165	0.60	Ethanol	1:9
2b	white	29	257-260	0.67	DMSO	1:9
2c	yellow	27.4	135-139	0.36	Acetone	1:9

Synthesize of hydrazone compounds 3a-k

0.01mol substituted benzaldehyde and 0.01mol thiosemicarbazide were refluxed in 20 mL ethanol at 80°C for 4h. Reaction was monitoring with TLC. Reaction mixture was cooled, recrystallized with proper solvent to afford compounds **3a-k** in good yield. SomePhysical properties of compounds **3a-k** were subjected at Table (2).

Table 2: Physical properties of compounds 3a-k

Com.	color	Yield%	M.P. °C	R.F.	crystallization solvent	Eluent: Hexan:EtOAc
3a	whitey	70	160	0.11	Ethanol	1:9
3b	yellow	46	210	0.75	Ethanol	1:9
3c	yellow	22	170	0.45	Ethanol	1:9
3d	yellow	58	220	0.11	Ethanol	1:9
3e	whitey	73	208	0.9	Ethanol	1:9
3f	Brown	30.8	175	0.8	Ethanol	1:9
3g	Off-white	24	175	0.25	Ethanol	1:9
3h	Orange	13.7	98	0.93	Ethanol	1:9
3i	Brown	44	160	0.81	DMSO	1:9
3j	whitey	61	183	0.6	Ethanol	1:9
3k	Brown	54	180	0.15	Ethanol	1:9

Synthesize of hydrazonyl thiazole (4a-j) [11]

Under microwave irradiation (M.I., 100W) with neat for 20sec; arylketones(0.01 mol), thiosemicarbazide (0.01mol) and chloroacetylchloride (0.01mol). The reaction was monitoring with TLC, then it was extracted andevaporated to afford compounds **4a-j** in pure yield with recrystallization from proper solvent. Physical properties of compounds **4a-j** were subjected at Table (3).

Table 3: Physical properties of compounds 6a-j

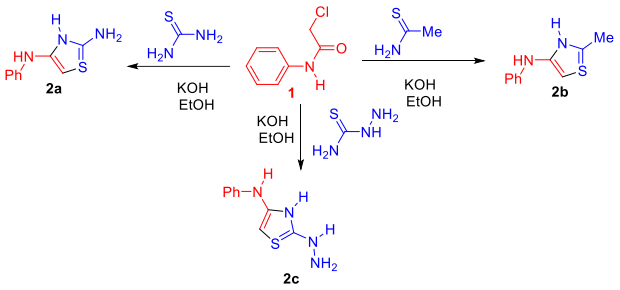
Com.	colour	Yield%	M.P. °C	R.F.	crystallization solvent	Eluent: MeOH:$CHCl_3$
4a	red	26	215-220	0.87	acetone	1:9
4b	orange	25	230-235	0.71	acetone	1:9
4c	yellow	35	205-210	0.32	DMF	1:9
4d	red	28	210-215	0.89	acetone	1:9
4e	Brown	20	240-242	0.81	acetone	1:9
4f	yellow	34	179-181	0.8	acetone	1:9
4g	orange	6	210-214	0.73	acetone	1:9
4h	Brown	15	185-189	0.6	acetone	1:9
4i	orange	40	_	0.5	acetone	1:9
4j	Brown	20	180-185	0.66	acetone	1:9

Antimicrobial Activity

The microbial activities [12, 13]were carried out using the diffusion plate method. A filter paper sterilized disk saturated with the measured quantity (25 μL) of the sample (1 mg/mL) was placed on a plate (9 cm diameter) containing a solid bacterial medium (nutrient agar) or a fungal medium (potato dextrose agar) that was seeded with the spore suspension of the test organism. After incubation at 37°C for 24 h for bacteria (in case of fungi, at 25°C for 72 h), the diameter of the clear zone of inhibition surrounding the sample is taken as a measure of the inhibitory power of the sample against the particular test organism (% inhibition = sample inhibition zone (cm)/plate diameter × 100). All measurements were done in DMSO as a solvent that has zero inhibition activity. The antimicrobial activity of the new compounds was examined against Gram Negative *Serratiamarcesence, Proteus vulgaris, Eshrichia coli and Psudomonas aeruginosa* and Gram Positive; *Staphylococcus aureus* in comparison with Norfloxacin and Ciprofloxacin as standard drugs.

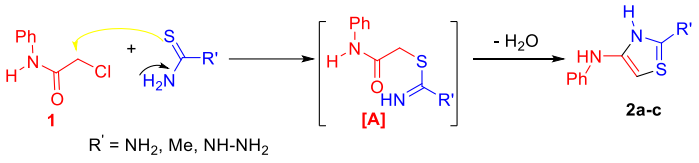
Results and Discussion

At room temperature, 2-chloro-*N*-phenylacetamide (**1**) was synthesized via reaction of aniline and 2-chloroacetyl chloride. Compound **1** reacted with different nucleophiles namely; thiourea, thiosemicarbazide, and thioactamide to afford N^4-phenyl-3*H*-λ^4-thiazole-2,4-diamine (**2a**) 2-hydrazineyl-N-phenyl-3*H*-λ^4--thiazol-4-amine (**2b**)and 2-methyl-N-phenyl-3H-λ^4-thiazol-4-amine (**2c**) in good yield (Scheme 1).



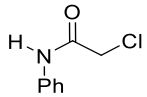
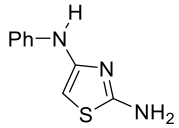
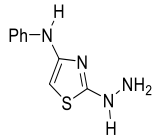
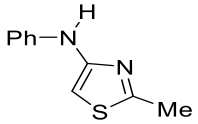
Scheme 1

The mechanism of formation of thiazole derivatives **2a-c** was depicted in Scheme 2; Nucleophilic attack of thiourea, thiosemicarbazide, or thioactamide to afford intermediate **A** which loss water molecule to get thiazole derivatives **2a-c** (Scheme 2). Structures of compounds **2a-c** were proved according to their spectroscopic analyses (Table 4).

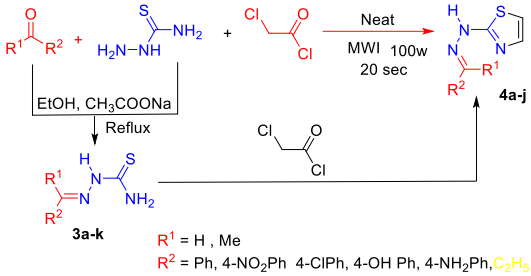


Scheme 2

Table (4)IR spectral data of compounds 1, 2a-c(cm^{-1})

Comp.	Structure	N-H	Ar.C-H	$^{\upsilon}$Ali,C-H	-C=C-	-C=N-	-C-N-	-C-S-	Other group
1		3265	3028	2947	1600 1556 1496	_	1249	_	C=O 1668 C-Cl 748
2a		3311	_	_	1589	1653	1113	1368	_
2b		3360	3025	_	1600	1650	1112	1383	_
2c		3284	3050	2922	1620 1595 1548	1668	1143	1334	_

On the other hand, hydrazone derivatives **3a-k** were synthesized through reaction of substituted benzaldehyde and thiosemicarbazide at reflux temperature in ethanol. Compounds **3a-k** reacted with chloracetylchloride to afford thiazole derivatives 4a-j in good yield (Scheme 3). Some spectral data was depicted in table 5.



cheme 3

In the same manner, benzaldehyde derivatives reacted with thiosemicarbazide and chloracetylchloride in one pot reaction under microwave irradiation for 20sec. to afford thiazole derivatives **4a-j** (Scheme 3). Some spectral data was depicted in Table 6.

Table (5)IR spectral data of compounds 3a-k(cm^{-1})

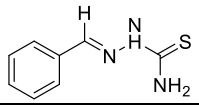
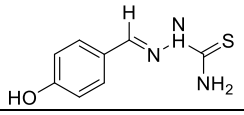
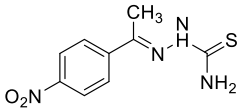
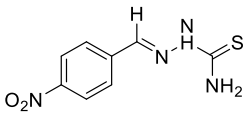
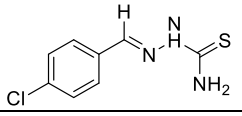
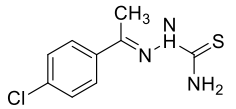
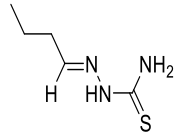
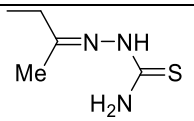
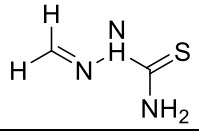
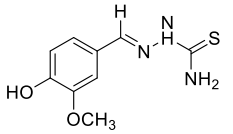
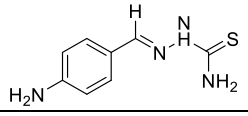
Comp.	Structure	-NH$_2$	Ar-C-H	Ali-C-H	Ar-C=C-	-C=N-	-C=S-	-N-N-	Other group
3a		3457 3345	3034	-	1602 1504	1663	1233	1367	_
3b		3433 3340	3021	-	1590 1558	1625	1256	1346	O-H 3523
3c		3468 3332	3012	2934	1595 1453	1635	1302	1387	N=O 1458 1359
3d		3491 3365	3018	–	1589 1504	1612	1286	1359	ᵞN=O 1452 1336
3e		3420 3315	3022	–	1601 1555	1618	1293	1319	C-Cl 730
3f		3437 3313	3037	2895	1589 1445	1634	1313	1354	C-Cl 780
3g		3369 3265	—	2995 2970	—	1663	1317	1363	—
3h		3448 3379	—	2970 2933 2906	—	1656	1292	1371	—
3i		3406 3267	–	2834	–	1645	1278	1325	–
3j		3435 3288	3057	2980	1587	1602	1343	1318	ᵞO-H 3524 ᵞC-O 1031
3k		3470 3374 3287	3030	–	1600 1556 1443	1618	1331	1358	–

Table (6)IR spectral data of compounds 4a-k(cm^{-1})

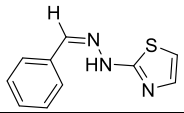
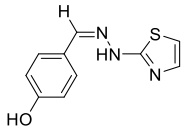
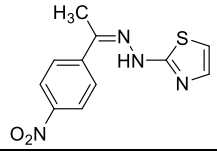
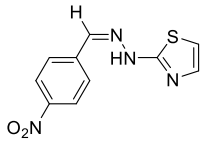
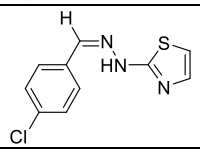
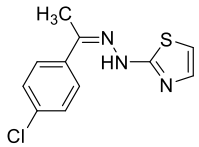
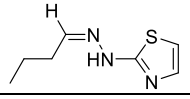
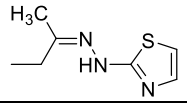
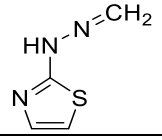
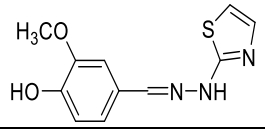
Comp.	Structure	- N-H	Ar-C-H	ali.C-H	-C=C	C=N-	-O-H	C-N-	-C-S-	Other group
4a		3183	3035	2812	1602 1514	1643	–	1215	1186	
4b		3120	3037	2958	1629 1593 1575 1512	1687	3200	1230	1163	
4c		3200	3055	2970	1598 1457	1623	–	1245	1145	NO_2 1430 1348
4d		3215	3061	2811	1593 1517	1651	–	1287	1180	NO_2 1467 1380
4e		3164	3032	2789	1617 1590 1453	1635	–	1220	1154	C-Cl 733
4f		1231	3042	2879	1623 1607 1489	1682	–	1267	1148	C-Cl 737
4g		3253	3073	2920 2815	1626	1653	–	1286	1164	
4h		3267	3079	2945 2830	1631	1650	–	1250	1196	
4i		3153	3112	2980 2926	1602 1558	1683	—	1232	1195	
4j		3160	3068	2962 2899	1635 1600 1510	1683	3200	1120	1166	

Table (7) spectral data of compounds (1, 2a-c)

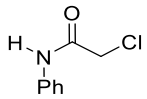
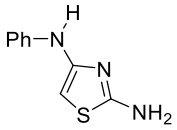
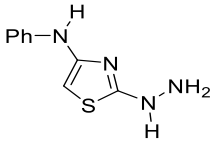
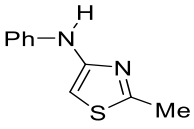
Comp.	Structure	Chemical shift ppm/ DMSO-d6
1		10.1(S, 1H, NH) , 7.60-7.08(m, 5H, Ar-H), 4.23(S,2H.CH_2)
2a		8.84 (S,1H,NH), 4.3(2H,NH_2), 7.78-7.4(m.5H,Ar-H), 6.04(S,1H,CH)
2b		8.55(S,1H,NH), , 7.77-7,00 (m,5H,Ar-H) , 6.25(S,1H,NH) 6,45(S,1H,CH), 4.13(S,2H,NH_2)
2c		9.02(S,1H,NH), 7.83-7.21(m,5H.Ar-H), 6.22(S,1H,CH) , 2,3(S,3H,CH_3)

Table (8) spectral data of compounds(3a-3k

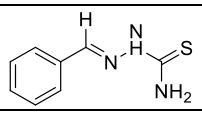
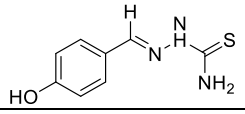
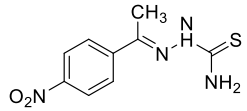
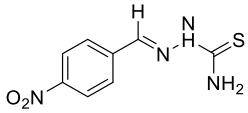
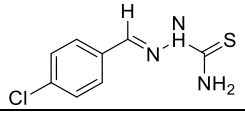
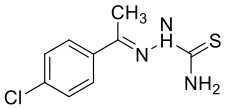
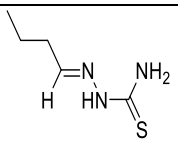
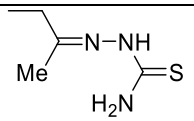
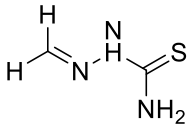
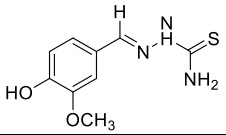
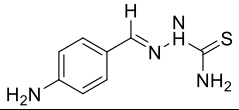
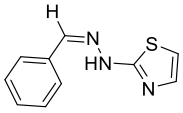
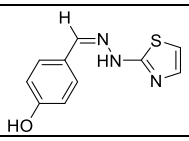
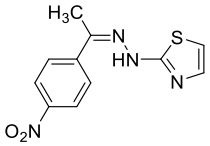
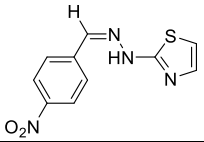
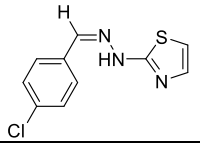
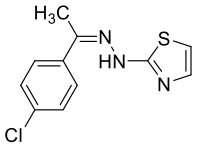
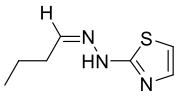
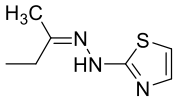
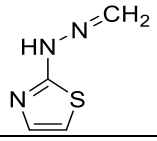
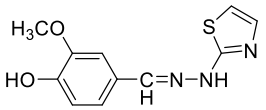
3a		11.67(S,1H,NH), 8.61(S,1H,N=CH), 7.89-7.45(m,5H, Ar-H), 6,37(S,2H,NH_2).
3b		11.72(S,1H,NH), 9.68(S,1H,OH) , 8,32(S,1H, N=CH), 7.60-8.65(m,4H,Ar-H), 6.2(S,2H,NH_2).
3c		11.29(S,1H,NH), 8.33-7.98(m,4H,Ar-H) , 7.20(S,2H,NH_2), 2.33(S,3H,CH_3)
3d		11.57(S,1H,NH), 8.38(S,1H,N=CH), 8.27-7.79(m,4H,Ar-H), 6,20(S,2H,NH_2)
3e		11,63(S,1H,NH) , 8.42(S,1H,N=CH) , 7.81-7.57(m,4H,Ar-H), 5.23(S,2H,NH_2)
3f		11.25(S,1H,NH), 7.98-7.65(m,4H,Ar-H), 6.13(S,2H,NH_2), 2.30(S,3H,CH_3)
3g		11.05(1H,NH), 8.66(S,1H, N=CH) , 7.58(S,2H, NH_2), 2.15(t,2H,CH_2), 1.48(m,2H,CH_2) , 0,88(t,3H,CH_3)
3h		11.29(S,1H,NH), 7.55(S,2H,NH_2) , 2.19(t,2H,CH_2), 1.94(S,3H,CH_3) , .83(t,3H,CH_3)
3i		11.67(S,1H,NH), 6.23(S,2H,NH_2), 5.13(S,1H, CH=N), 4.98(S,1H,CH=N)
3j		11.56(S,1H,NH), 9.55(S,1H,OH), 8.08(S,1H,N=CH) , 7.34-6.88(m,3H,Ar-H) , 6.21(S,2H,NH_2), 3.83(S,3H,OCH_3)
3k		11.67(S,1H,NH), 8.41(S,1H,N=CH) , 7.53-6.88(m,4H,Ar-H), 6.27(S,2H,NH_2), 5.48(S,2H,NH_2)

Table (9) spectral data of compounds(4a-4j)

Comp.	Structure	**Chemical shift ppm/ DMSO-d6**
4a		11.72(S,1H,NH), 8,32(S,1H, N=CH), 8.07(S,1H, N-CH=CH) 7.60-7.25 (m,5H,Ar-H), 7.03(S,1H,S-CH=CH)
4b		10.06(S,1H, NH), 8.57(S,1H, OH) , 8.36(S,1H, N-CH=CH), 8.27(S,1H, S-CH=CH), 7.71-6.83(m,4H, Ar-H)
4c		10.57(S,1H, NH), 8.38-8.07 (m,4H, Ar-H), 7,77(S,1H, N-CH=CH), 7,03(S,1H, S-CH=CH), 2.34(S,3H, CH_3)
4d		10.66(S,1H, NH), 8.31-8.07(m,4H, Ar-H), 7.92(S,1H, N=CH), 7,47(S,1H, N-CH=CH), 6,83(S,1H, S-CH=CH)
4e		10.85(S,1H,NH), 8.03(S,1H, N=CH) 7.88-7.60(m,4H,Ar-H), 7,40(S,1H, N-CH=CH), 6,63(S,1H, S-CH=CH)
4f		11.65(1H,NH), 8.08-7.90(m,4H,Ar-H), 7,53(S,1H, N-CH=CH), 6,73(S,1H, S-CH=CH)
4g		11.88(S,1H,NH),7,53(S,1H, N-CH=CH), 7.03(S,1H, N=CH), 6,90(S,1H, S-CH=CH)
4h		11.53(S,1H,NH), 7,23(S,1H, N-CH=CH), 6,90(S,1H, S-CH=CH), 2.21(q,2H, CH_2), 2,09(S,3H, CH_3), 0.89(t,3H, CH_3)
4i		11.96(S,1H,NH), 7,25(d,1H, N-CH=CH), 6,69(d,1H, S-CH=CH)
4j		11.85(S,1H,NH), 9.65(S,1H, OH), 8.06(S,1H, N=CH) 7.37-6.92(m,4H,Ar-H), 7.00(S,1H, N-CH=CH), 6,67(S,1H, S-CH=CH), 3.92(S,3H, OCH_3)

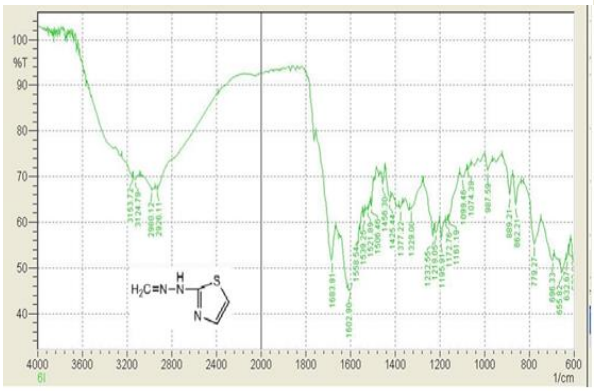


Fig. (4): FT-IR spectrum of 4i

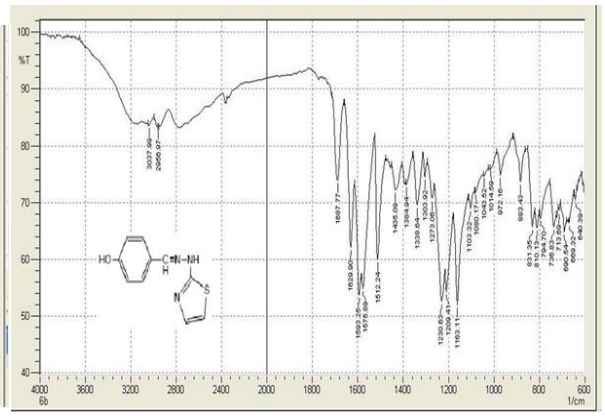


Fig. (5): FT-IR spectrum of 4b

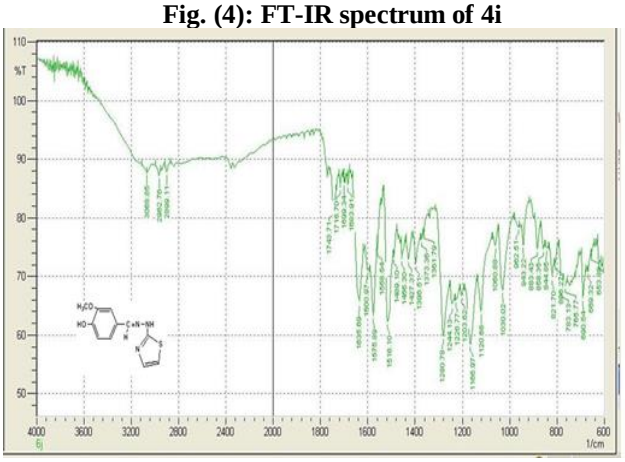


Fig. (6): FT-IR spectrum of 4j

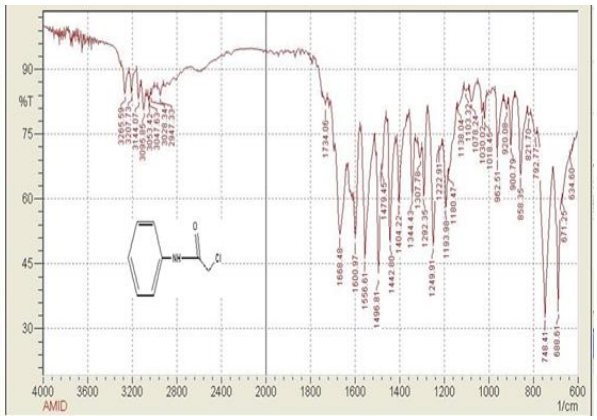


Fig. (7): FT-IR spectrum of 1

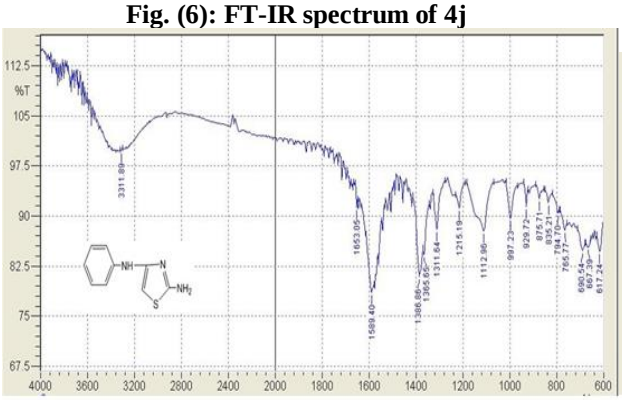


Fig. (8): FT-IR spectrum of 2a

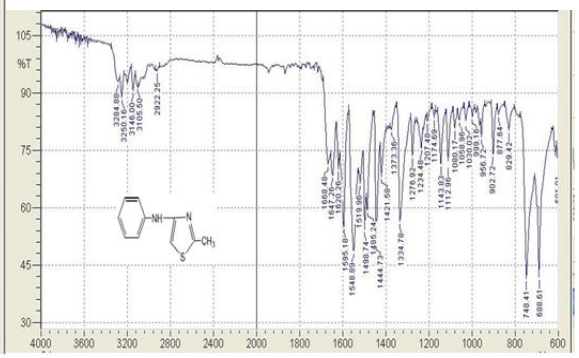


Fig. (9): FT-IR spectrum of 2c

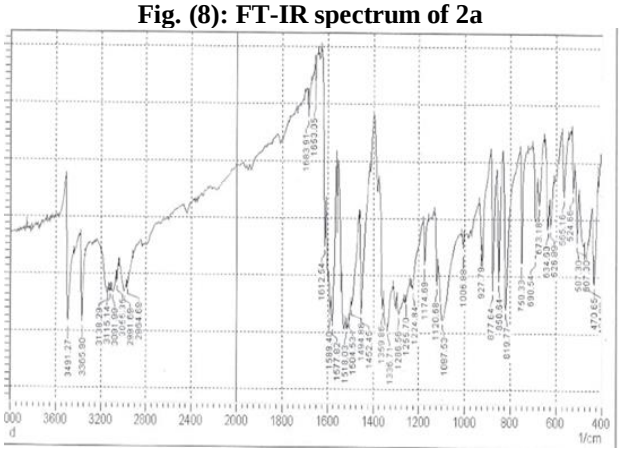


Fig. (10): FT-IR spectrum of 3d

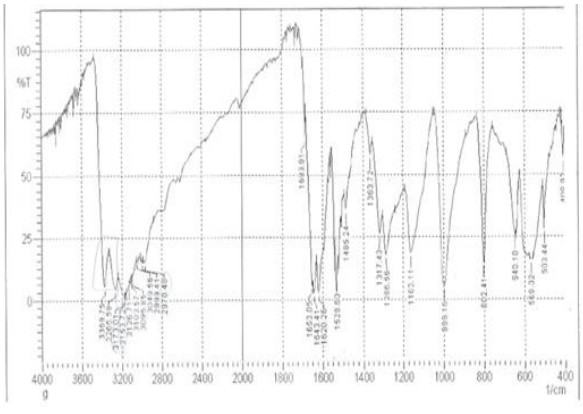


Fig. (11): FT-IR spectrum of 3g

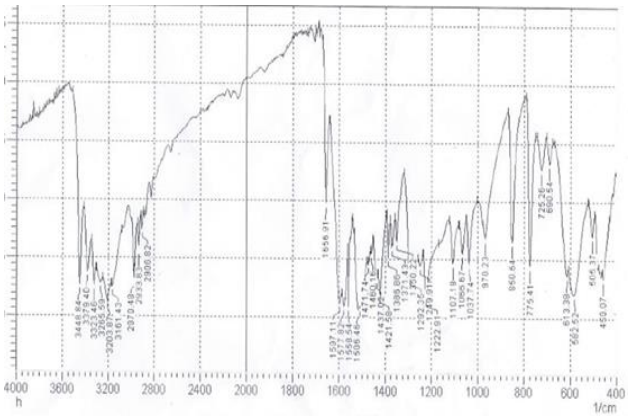


Fig. (12): FT-IR spectrum of 3h

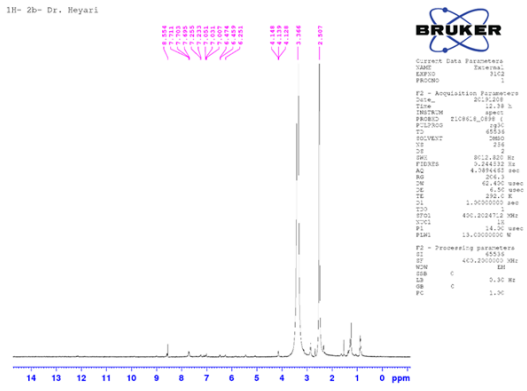


Fig. (13): 1H NMR spectrum of 2b

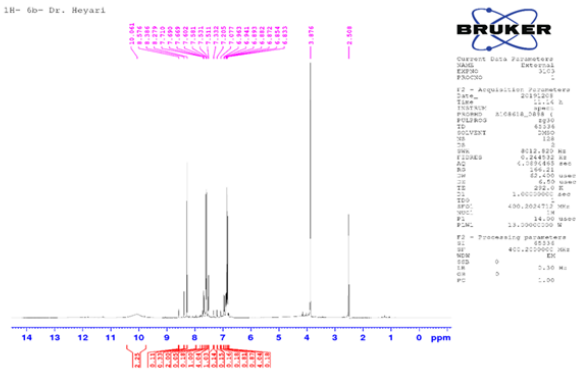


Fig. (14): 1H NMR spectrum of 3g

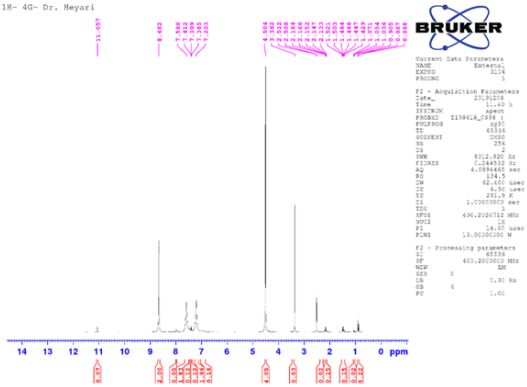


Fig. (15): 1H NMR spectrum of 4b

Antibacterial activity

Ten new compounds were screened to determine their antibacterial activity against Gram Negative *Serratiamarcesence, Proteus vulgaris, Eshrichia coli and Psudomonas aeruginosa* and Gram Positive; *Staphylococcus aureus* in comparison with Norfloxacin and Ciprofloxacin as standard drugs.

The agar diffusion technique was used to conduct antibacterial tests. On the basis of the observed zone of inhibition value, antimicrobial activities were assessed by measuring the diameter of the zone of inhibition against test organisms after incubation at 37°C for a 24-hour [14].

Based on the diameters of the inhibition zones tabulated in Table (**4**), it could be investigated that most of the evaluated thiazole and hydrazone compounds produced moderate to significant broad spectrum antimicrobial activity comparing to the used reference drugs. The hydrazine derivatives **2c,d** exhibited moderate antibacterial activity, while its conversion to thiazole **3b-j** increases the potency. It has been found that thiazole derivatives **4a-k** exhibited lower potent antibacterial activity in comparison to the reference drug,

Furthermore, the formation of the cyclized analogue thiazole **4a-k** enhanced lower potency against the tested bacterial strains exhibiting inhibition zones with (5-10) mm. Further modification and optimization are needed to get new candidates of more significant antimicrobial activity against various types of bacteria and fungi.

Table (10): inhibition zone diameter in mm

Compound	*Gram negative*				*Gram positive*
	Serratiamarcesence	***Proteus vulgaris***	***Escherichia coli***	***Psudomonas aeruginosa***	***Staphylococcus aureus***
2c	++	++	++	++	++
2d	++	++	+	+	++
3b	++	++	++	++	++
3d	++	++	++	++	++
3e	++	++	++	++	++
3j	++	++	++	++	++
4a	+	+	+	+	+
4c	+	+	+	+	+

4d	+	+	+	+	+
4e	+	+	+	+	+
4h	+	+	+	+	+
Norfloxacin	_	_	++	+++	+++
ciprofloxacin	++	_	+	+++	++

-, + repression area diameter: **- <5 mm; + (5-10) mm; ++ (10-20) mm; +++ > 20**

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