Neat Reaction Technology and Conventional Method for the Synthesis of 4-Oxo-Thiazolidines and their 5-Arylidene Derivatives and its Antimicrobial Activity

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Abstract

Several new 4-arylideneamino-3-mercapto-5-[(phenothiazin-10-yl) methyl]-1,2,4-triazoles **5a-h**, 2-aryl-3-(3-mercapto-5-phenothiazin-10-ylmethyl-[1,2,4]-triazol-4-yl)-thiazolidin-4-one **6a-h** and 5-arylidene-2-aryl-3-(3-mercapto-5-phenothiazin-10-ylmethyl-[1,2,4]-triazol-4-yl)-thiazolidin-4-one **7a-h** were synthesized under both conventional as well as microwave irradiation technique. The structures of all synthesized compounds were confirmed by IR, ^{1}H NMR, ^{13}C NMR, FAB- mass spectra and by microanalytical data. All synthesized compounds were evaluated for their antibacterial activity against *Escherichia coli*, *Salmonella typhi* and *Bacillus subtilis* and antifungal activity against *Aspergillus flavus, Fusarium oxysporum* and *Penicillium citrinum*. Some of the compounds have shown significant antibacterial and antifungal activities.

Keywords: Phenothiazine; Thiazolidinone; Conventional and Microwave synthesis; Antimicrobial activity.

Introduction

Phenothiazine form an important class of heterocyclic compounds possessing wide spectrum diverse of biological activities such as antitumor, antimalarial, antipsychotic and anti-inflammatory[1-3] . The phenothiazine contain a heterocyclic ring skeleton with two carbocyclic aromatic rings connected to each other via a sulfide and an imino bridge which facilitates several types of reactions such as substitution at the nitrogen, electrophilic substitution on the aromatic rings, N-oxidation and photochemical reactions[4-6]. 4-thiazolidinones derivatives also exhibit antibacterial[7], antifungal[8], antitubercular[9-11], antiviral[12-14], anticancer[15,16], and so on properties. The incorporation of 4-oxo-thiazolidine moiety in phenothiazine through 1,2,4-triazole framework has been found to enhance the activity. In view of these we have synthesized some new 4-arylideneamino-3-mercapto-5-[(phenothiazin-10-yl) methyl]-1,2,4-triazoles **5a-h**, 2-aryl-3-(3-mercapto-5-phenothiazin-10-ylmethyl-[1,2,4]-triazol-4-yl)-thiazolidin-4-one **6a-h** and 5-arylidene-2-aryl-3-(3-mercapto-5-phenothiazin-10-ylmethyl-[1,2,4]-triazol-4-yl)-thiazolidin-4-one **7a-h** under both conventional as well as microwave irradiation technique. The application of microwave irradiation is used for carrying out chemical transformations, which are eco-friendly. Commercial microwave

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oven is used as a convenient source of heat in laboratory. The microwave reactions occur more rapidly, safely and with higher chemical yields [17]. Thus, render the microwave method superior to conventional method [18-21].

Materials and Methods

Melting points were taken in open capillary tubes. Formation of the compounds was routinely checked by TLC using silica gel 'G' and the spots were exposed to iodine vapours for visualization. IR spectra were recorded on Shimadzu 8201 PC spectrophotometer. The ^{1}H NMR spectra were recorded at 400 MHz and ^{13}C NMR spectra were recorded at 100 MHz on Bruker DRX-300 in $CDCl_3$ using TMS as an internal standard on δ scale. The FAB mass spectra were recorded on a Jeol SX 102 mass spectrometer. Microwave assisted reactions were carried out in Microwave oven (Bajaj 2100 ETC, 800W, 2450 MHz). All compounds gave satisfactory C, H and N percentage within the experimental limit.

Compounds 1, 2, 3 and 4 are known compounds and prepared according to a known procedure[22].

*Procedure for the synthesis of ethyl (phenothiazin-10-yl) acetate **1,** according to Microwave method*: A mixture of phenothiazine (35 g, 0.17 mol) and ethyl chloroacetate (21.55 g, 0.17 mol) was taken in 250 ml beaker, mixed well and irradiated in microwave oven 800W for 1 min. The completion of the reaction was monitored by TLC. After completion of the reaction the beaker was removed from the oven and the mixture was allowed to cool at room temperature. The product was recrystallized from acetone to furnish compound **1.**

*Procedure for the Synthesis of 2-(phenothiazin-10-yl) acetohydrazide **2,** according to Microwave method:* A mixture of compound **1** (32 g, 0.11 mol) and hydrazine hydrate (5.62 g, 0.11 mol) was taken in 250 ml beaker, mixed well and irradiated in microwave oven 800W for 1 min and worked up as usual.

*Procedure for the Synthesis of 2-mercapto-5-[(phenothiazin-10-yl) methyl]-1,3,4-oxadiazole **3,** according to Microwave method*: A mixture of compound **2** (29 g, 0.11 mol) and carbondisulfide (8.15 g, 0.11 mol) in the presence of potassium hydroxide (6.0 g, 0.11 mol) was taken in 250 ml beaker, mixed well and irradiated in microwave oven 800W for 1 min and worked up as usual.

*Procedure for the Synthesis of 4-amino-3-mercapto-5-[(phenothiazin-10-yl) methyl]-1,2,4-triazole **4,** according to Microwave method:* A mixture of compound **3** (25 g, 0.08 mol) and hydrazine hydrate (4.0 g, 0.08 mol) was taken in 250 ml beaker, mixed well and irradiated in microwave oven 800W for 1.5 min and worked up as usual.

*Procedure for the Synthesis of 4-benzylideneamino-3-mercapto-5-[(phenothiazin-10-yl) methyl]-1,2,4-triazole **5a.***

Conventional method: A mixture of the compound **4** (1.8 g, 0.005 mol) and benzaldehyde (0.58 g, 0.005 mol) in dioxane (30 ml) was refluxed for about 1 hr. on a water bath. The solvent was removed in vacuo and the residue thus obtained was

purified over the column of silica gel and recrystallized from acetone to furnish compound **5a.**

Other compounds **5b-h** were prepared in a similar manner using different carbonyl compounds.

Microwave method: A mixture of the compound **4** (1.8 g, 0.005 mol) and benzaldehyde (0.58 g, 0.005 mol) was taken in 250 ml beaker, mixed well and irradiated in microwave oven 800W for 1 min and worked up as usual, 5a.

Other compounds 5b-h were prepared similarly by microwave method using different carbonyl compounds.

Procedure for the Synthesis of 2-phenyl-3-(3-mercapto-5-phenothiazin-10-ylmethyl-[1,2,4]-triazol-4-yl)-thiazolidin-4-one ***6a.***

Conventional method: A mixture of the compound **5a** (1.5 g, 0.003 mol) and thioglycolic acid (0.33 g, 0.003 mol) with a pinch of anhydrous $ZnCl_2$ in acetone (25ml) was refluxed for about 2 hr. on a water bath. The solvent was removed in vacuo and the residue thus obtained was purified over the column of silica gel and recrystallized from chloroform to furnish compound **6a**.

Other compounds **6b-h** were prepared in a similar manner using **5b-h.**

Microwave method: A mixture of the compound **5a** (1.5 g, 0.005 mol) and thioglycolic acid (0.33 g, 0.003 mol) with a pinch of anhydrous $ZnCl_2$ was taken in 250 ml beaker, mixed well and irradiated in microwave oven 800W for 1 min and worked up as usual, 6a.

Other compounds 6b-h were prepared similarly by microwave method using 5b-h.

Procedure for the Synthesis of 5-benzylidene-2-phenyl-3-(3-mercapto-5-phenothiazin-10-ylmethyl-[1,2,4]-triazol-4-yl)-thiazolidin-4-one ***7a.***

Conventional method: A mixture of the compound **6a** (1 g, 0.002 mol) and benzaldehyde (0.22 g, 0.002 mol) in dioxane (20ml) in the presence of sodium ethoxide was refluxed for about 2 hr. on a water bath. The solvent was removed in vacuo and the residue thus obtained was purified over the column of silica gel and recrystallized from chloroform to furnish compound **7a.**

Other compounds **7b-h** were prepared in a similar manner using **6b-h** and different carbonyl compounds.

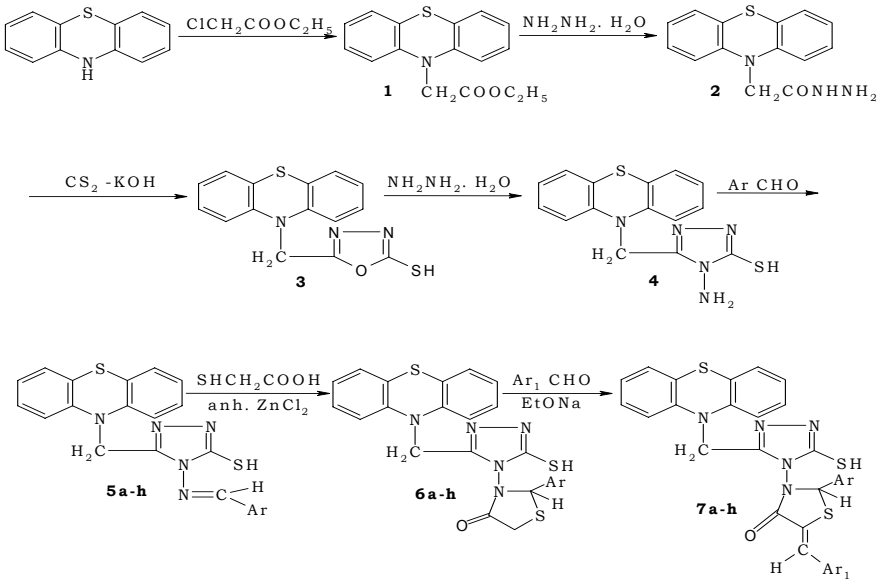
Microwave method: A mixture of the compound **6a** (1 g, 0.002 mol) and benzaldehyde (0.22 g, 0.002 mol) in the presence of sodium ethoxide was taken in 250 ml beaker, mixed well and irradiated in microwave oven 800W for 1 min and worked up as usual, 7a.

Other compounds 7b-h were prepared similarly by microwave method using 6b-h and different carbonyl compounds.

Results and Discussion

Compound **4** reacted with various aromatic aldehydes to afford new heterocyclic products 4-arylideneamino-3-mercapto-5-[(phenothiazin-10-yl) methyl]-1,2,4-triazoles

5a-h. In IR spectra the bands at 1575-1586 cm^{-1} were observed due to N=CH-Ar. In ^1H NMR spectra peaks at δ 8.61-8.72 ppm were observed due to N=CH-Ar. Furthermore in ^{13}C NMR spectra peaks at δ 143.0-144.1 ppm were observed due to N=CH-Ar confirmed the formation of compounds **5a-h.** Compounds **5a-h** upon underwent dehydrative annulation reaction with thioglycolic acid afforded 2-aryl-3-(3-mercapto-5-phenothiazin-10-ylmethyl-[1,2,4]-triazol-4-yl)-thiazolidin-4-one **6a-h.** In IR spectra the band at 1707-1730 cm^{-1} was assigned to >C=O, cyclic. In ^1H NMR spectra peaks at δ 4.01- 4.24 ppm were displayed due to N-CH-S and peaks at δ 3.41- 3.52 ppm due to S-CH_2. Furthermore in ^{13}C NMR spectra peaks at δ 40.0 - 42.3 ppm were displayed due to N-CH-S and peaks at δ 43.0 - 44.1 ppm were appeared due to –SCH_2 and peaks at 171.7-174.2 ppm were observed due to >C=O, cyclic confirmed the formation of compounds **6a-h.** Compounds **6a-h** reacted with various aromatic aldehydes to yield the final products 5-arylidene-2-aryl-3-(3-mercapto-5-phenothiazin-10-ylmethyl-[1,2,4]-triazol-4-yl)-thiazolidin-4-one **7a-h.** In IR spectra the bands at 1632-1643 cm^{-1} were observed due to >C=CH-Ar. In ^1H NMR spectra peaks at δ 5.25–5.36 ppm were appeared due to >C=CH-Ar. Furthermore in ^{13}C NMR spectra peaks at δ 125.4-126.3 ppm and δ 129.1-130.3 ppm due to >C=CH-Ar confirmed the formation of compounds **7a-h**.



Where Ar an Ar_1

a = C_6H_5; b = 2-OHC_6H_4; c = 2-ClC_6H_4; d = 2-Br C_6H_4;
e = 2-$NO_2C_6H_4$; f = 2-$CH_3OC_6H_4$; g = 4-$CH_3C_6H_4$; h = 4-N-$(CH_3)_2$ C_6H_4

Scheme-1

All reactions under microwave irradiation were completed within 1-3 min giving excellent yields whereas similar reactions under conventional heating (steam bath) under reflux for 1-4 hrs gave good yields. The impact of microwave irradiation and conventional heating for the synthesis of compounds **5a-h, 6a-h** and **7a-h** have been compared. The characterization and spectral data of the synthesized compounds are summarized in tables 1 and 2.

Table 1. Characterization data of synthesized compounds **1-4**, **5a-h, 6a-h** and **7a-h.**

Comp. No.	Compound name	Yield (%)		M.P.(°C)	Mol. Formula	Elemental analysis Found% (Calcd%)		
		CM (h)	MWT (min)			C	H	N
1	Ethyl (phenothiazin-10-yl) acetate	75 (1)	95 (1)	199-200	$C_{16}H_{15}NO_2S$	67.34 (67.37)	5.21 (5.26)	4.86 (4.91)
2	2-(phenothiazin-10-yl) acetohydrazide	80 (1)	96 (1)	178-179	$C_{14}H_{13}N_3OS$	61.93 (61.99)	4.76 (4.80)	15.45 (15.50)
3	2-mercapto-5-[(phenothiazin-10-yl) methyl]-1,3,4-oxadiazole	71 (1)	87 (1)	185-186	$C_{15}H_{11}N_3OS_2$	57.48 (57.51)	3.47 (3.51)	13.39 (13.42)
4	4-amino-3-mercapto-5-[(phenothiazin-10-yl) methyl]-1,2,4-triazole	74 (2)	89 (1.5)	218-220	$C_{15}H_{13}N_5S_2$	55.01 (55.04)	3.92 (3.97)	21.38 (21.41)
5a	4-benzylideneamino-3-mercapto-5-[(phenothiazin-10-yl) methyl]-1,2,4-triazole	71 (1)	90 (1)	138-140	$C_{22}H_{17}N_5S_2$	63.56 (63.61)	4.06 (4.10)	16.83 (16.87)
5b	4-(2-hydroxy-benzylideneamino)-3-mercapto-5-[(phenothiazin-10-yl) methyl]-1,2,4-triazole	72 (2)	88 (1)	160-162	$C_{22}H_{17}N_5OS_2$	61.20 (61.25)	3.88 (3.94)	16.20 (16.24)
5c	4-(2-chloro-benzylideneamino)-3-mercapto-5-[(phenothiazin-10-yl) methyl]-1,2,4-triazole	73 (2.5)	87 (1.5)	175-176	$C_{22}H_{16}N_5S_2Cl$	58.69 (58.73)	3.52 (3.56)	15.53 (15.57)
5d	4-(2-bromo-benzylideneamino)-3-mercapto-5-[(phenothiazin-10-yl) methyl]-1,2,4-triazole	69 (1.5)	84 (1)	179-180	$C_{22}H_{16}N_5S_2Br$	53.40 (53.45)	3.19 (3.24)	14.12 (14.17)
5e	4-(2-nitro-benzylideneamino)-3-mercapto-5-[(phenothiazin-10-yl) methyl]-1,2,4-triazole	75 (3)	91 (2)	120-122	$C_{22}H_{16}N_6O_2S_2$	57.33 (57.39)	3.42 (3.48)	18.18 (18.26)
5f	4-(2-methoxy-benzylideneamino)-3-mercapto-5-[(phenothiazin-10-yl) methyl]-1,2,4-triazole	70 (1.5)	85 (1)	138-140	$C_{23}H_{19}N_5OS_2$	61.97 (62.02)	4.22 (4.27)	15.68 (15.73)
5g	4-(4-methyl-benzylideneamino)-3-mercapto-5-[(phenothiazin-10-yl) methyl]-1,2,4-triazole	69 (2.5)	86 (1)	180-182	$C_{23}H_{19}N_5S_2$	64.30 (64.34)	4.38 (4.43)	16.26 (16.31)
5h	4-(4-dimethylamino-benzylideneamino)-3-mercapto-5-[(phenothiazin-10-yl) methyl]-1,2,4-triazole	71 (1.5)	90 (1)	183-184	$C_{24}H_{22}N_6S_2$	62.82 (62.88)	4.76 (4.80)	18.29 (18.34)
6a	2-phenyl-3-(3-mercapto-5-phenothiazin-10-ylmethyl-[1,2,4]-triazol-4-yl)-thiazolidin-4-one	68 (2)	88 (1)	135-136	$C_{24}H_{19}N_5OS_3$	58.86 (58.90)	3.83 (3.89)	14.29 (14.31)
6b	2-(2-hydroxy-phenyl)-3-(3-mercapto-5-phenothiazin-10-ylmethyl-[1,2,4]-triazol-4-yl)-thiazolidin-4-one	65 (2)	84 (2)	155-157	$C_{24}H_{19}N_5O_2S_3$	56.97 (57.03)	3.70 (3.76)	13.81 (13.86)
6c	2-(2-chloro-phenyl)-3-(3-mercapto-5-phenothiazin-10-ylmethyl-[1,2,4]-triazol-4-yl)-thiazolidin-4-one	71 (1.5)	88 (1)	149-150	$C_{24}H_{18}N_5OS_3Cl$	54.96 (55.01)	3.40 (3.44)	13.31 (13.37)
6d	2-(2-bromo-phenyl)-3-(3-mercapto-5-phenothiazin-10-ylmethyl-[1,2,4]-triazol-4-yl)-thiazolidin-4-one	70 (3)	85 (2)	148-150	$C_{24}H_{18}N_5OS_3Br$	50.66 (50.71)	3.13 (3.17)	12.28 (12.33)
6e	2-(2-nitro-phenyl)-3-(3-mercapto-5-phenothiazin-10-ylmethyl-[1,2,4]-triazol-4-yl)-thiazolidin-4-one	73 (4)	91 (3)	165-166	$C_{24}H_{18}N_6O_3S_3$	53.88 (53.93)	3.34 (3.37)	15.70 (15.73)
6f	2-(2-methoxy-phenyl)-3-(3-mercapto-5-phenothiazin-10-ylmethyl-[1,2,4]-triazol-4-yl)-thiazolidin-4-one	65 (2)	86 (2)	132-133	$C_{25}H_{21}N_5O_2S_3$	57.76 (57.80)	4.01 (4.05)	13.44 (13.49)
6g	2-(4-methyl-phenyl)-3-(3-mercapto-5-phenothiazin-10-ylmethyl-[1,2,4]-triazol-4-yl)-thiazolidin-4-one	71 (1.5)	89 (1)	176-177	$C_{25}H_{21}N_5OS_3$	59.62 (59.64)	4.12 (4.17)	13.86 (13.92)
6h	2-(4-dimethylamino-phenyl)-3-(3-mercapto-5-phenothiazin-10-ylmethyl-[1,2,4]-triazol-4-yl)-thiazolidin-4-one	68 (1)	88 (1)	172-174	$C_{26}H_{24}N_6OS_3$	58.60 (58.65)	4.46 (4.51)	15.73 (15.79)
7a	5-benzylidene-2-phenyl-3-(3-mercapto-5-phenothiazin-10-ylmethyl-[1,2,4]-triazol-4-yl)-thiazolidin-4-one	67 (2)	85 (1)	131-132	$C_{31}H_{23}N_5OS_3$	64.45 (64.47)	3.96 (3.99)	12.09 (12.13)
7b	5-(2-hydroxy-benzylidene)-2-(2-hydroxy-phenyl)-3-(3-mercapto-5-phenothiazin-10-ylmethyl-[1,2,4]-triazol-4-yl)-thiazolidin-4-one	69 (2)	90 (1.5)	151-152	$C_{31}H_{23}N_5O_3S_3$	61.02 (61.08)	3.73 (3.78)	11.43 (11.49)

Comp. No.	Compound name	Yield (%)		M.P.(^{O}C)	Mol. Formula	Elemental analysis Found% (Calcd%)		
		CM (h)	MWT (min)			C	H	N
7c	5-(2-chloro-benzylidene)-2-(2-chloro-phenyl)-3-(3-mercapto-5-phenothiazin-10-ylmethyl-[1,2,4]-triazol-4-yl)-thiazolidin-4-one	68 (2.5)	88 (1.5)	160-161	$C_{31}H_{21}N_5OS_3Cl_2$	57.53 (57.59)	3.20 (3.25)	10.79 (10.83)
7d	5-(2-bromo-benzylidene)-2-(2-bromo-phenyl)-3-(3-mercapto-5-phenothiazin-10-ylmethyl-[1,2,4]-triazol-4-yl)-thiazolidin-4-one	72 (1)	87 (1)	178-180	$C_{31}H_{21}N_5OS_3Br_2$	50.58 (50.63)	2.82 (2.86)	9.47 (9.52)
7e	5-(2-nitro-benzylidene)-2-(2-nitro-phenyl)-3-(3-mercapto-5-phenothiazin-10-ylmethyl-[1,2,4]-triazol-4-yl)-thiazolidin-4-one	73 (2.5)	92 (1)	153-154	$C_{31}H_{21}N_7O_5S_3$	55.72 (55.77)	3.11 (3.15)	14.65 (14.69)
7f	5-(2-methoxy-benzylidene)-2-(2-methoxy-phenyl)-3-(3-mercapto-5-phenothiazin-10-ylmethyl-[1,2,4]-triazol-4-yl)-thiazolidin-4-one	70 (1.5)	90 (1)	129-130	$C_{33}H_{27}N_5O_3S_3$	62.12 (62.17)	4.20 (4.24)	10.94 (10.99)
7g	5-(4-methyl-benzylidene)-2-(4-methyl-phenyl)-3-(3-mercapto-5-phenothiazin-10-ylmethyl-[1,2,4]-triazol-4-yl)-thiazolidin-4-one	74 (1)	89 (1)	173-174	$C_{33}H_{27}N_5OS_3$	65.41 (65.45)	4.41 (4.46)	11.52 (11.57)
7h	5-(4-dimethylamino-benzylidene)-2-(4-dimethylamino-phenyl)-3-(3-mercapto-5-phenothiazin-10-ylmethyl-[1,2,4]-triazol-4-yl)-thiazolidin-4-one	71 (1)	88 (1)	172-173	$C_{35}H_{33}N_7OS_3$	63.30 (63.35)	4.95 (4.98)	14.75 (14.78)

CM = Conventional Method, MWT = Microwave assisted technique, h= hours, min = minute

Table 2. Spectral data of synthesized compounds **1-4, 5a-h, 6a-h** and **7a-h.**

Comp. No.	IR [KBr, ν, cm^{-1}]	^{1}HNMR [δ, ppm]	^{13}CNMR [δ, ppm]	Mass (m/z)
1	3040 (aromatic ring), 1735 (>C=O, ester), 1435 (N-CH_2), 645 (C-S-C)	6.53-7.24 (m, 8H, Ar-H), 2.79 (s, 2H, N-CH_2), 4.22 (q, 2H, J=7 Hz, $COOCH_2CH_3$), 1.20 (t, 3H, J=7 Hz, $COOCH_2CH_3$)	168.5 (>C=O, ester), 114.43-127.32 (C of aromatic ring), 61.1 ($COO\underline{CH_2}CH_3$), 37.41 (N-CH_2), 14.1 ($COOCH_2\underline{CH_3}$)	285 [M^+], 212, 198, 166
2	3255, 3382 (-$NHNH_2$), 3042 (aromatic ring), 1683 (>C=O, amide), 1436 (N-CH_2), 647 (C-S-C)	8.22 (s, 1H, CONH), 6.55-7.26 (m, 8H, Ar-H), 2.82 (s, 2H, N-CH_2), 4.47 (s, 2H, NH_2)	166.5 (CONH), 114.44-127.34 (C of aromatic ring), 37.43 (N-CH_2)	271 [M^+], 212, 198, 166
3	3041 (aromatic ring), 2580 (-SH), 1600 (>C=N), 1438 (N-CH_2), 1268, 1058 (C-O-C), 649 (C-S-C)	13.59 (s, 1H, SH), 6.54 -7.25 (m, 8H, Ar-H) , 2.80 (s, 2H, N-CH_2)	175.84 (1,3,4-Oxadiazole,C-5), 158.68 (1,3,4-Oxadiazole,C-2), 114.46 - 127.36 (C of aromatic ring), 37.42 (N-CH_2)	313 [M^+], 280, 252, 212, 198, 166
4	3308, 3200 (-NH_2), 3044 (aromatic ring), 2581 (-SH), 1598 (>C=N), 1440 (N-CH_2), 646 (C-S-C)	13.60 (s, 1H, SH), 6.56 -7.27 (m, 8H, Ar-H) , 5.52 (s, 2H, NH_2), 2.81 (s, 2H, N-CH_2)	152.70 (1,2,4-triazole,C-5), 145.60 (1,2,4-triazole,C-3), 114.47 - 127.37 (C of aromatic ring), 37.45 (N-CH_2)	327 [M^+], 294, 212, 198, 166, 115
5a	3045 (aromatic ring), 2583 (-SH), 1596 (>C=N), 1575 (N=CH-Ar), 1437 (N-CH_2), 648 (C-S-C)	13.62 (s, 1H, SH), 8.61 (s, 1H, N=CH-Ar), 6.58 -7.29 (m, 13H, Ar-H) , 2.83 (s, 2H, N-CH_2)	152.72 (1,2,4-triazole,C-5), 145.62 (1,2,4-triazole,C-3), 143.0 (N=CH-Ar), 114.45 - 127.35 (C of aromatic ring), 37.46 (N-CH_2)	415 [M^+], 382, 212, 203, 198, 166
5b	3248 (Ar-OH), 3043 (aromatic ring), 2584 (-SH), 1592 (>C=N), 1577 (N=CH-Ar), 1439 (N-CH_2), 650 (C-S-C)	13.64 (s, 1H, SH), 11.91 (s, 1H, Ar-OH), 8.63 (s, 1H, N=CH-Ar), 6.57 -7.28 (m, 12H, Ar-H) , 2.86 (s, 2H, N-CH_2)	165.5 (C-OH, aromatic), 152.71 (1,2,4-triazole,C-5), 145.63 (1,2,4-triazole,C-3), 143.1 (N=CH-Ar), 114.48 - 127.38 (C of aromatic ring), 37.44 (N-CH_2)	431 [M^+], 398, 219, 212, 198, 166
5c	3046 (aromatic ring), 2582 (-SH), 1591 (>C=N), 1576 (N=CH-Ar), 1441 (N-CH_2), 756 (Ar-Cl), 652 (C-S-C)	13.61 (s, 1H, SH), 8.62 (s, 1H, N=CH-Ar), 6.60 -7.31 (m, 12H, Ar-H) , 2.84 (s, 2H, N-CH_2)	152.74 (1,2,4-triazole,C-5), 145.61 (1,2,4-triazole,C-3), 143.3 (N=CH-Ar), 134.5 (C-Cl, aromatic), 114.50 - 127.40 (C of aromatic ring), 37.47 (N-CH_2)	449.5 [M^+], 417, 238, 212, 198, 166
5d	3049 (aromatic ring), 2586 (-SH), 1594 (>C=N), 1579 (N=CH-Ar), 1444 (N-CH_2), 651 (C-S-C), 620 (Ar-Br)	13.65 (s, 1H, SH), 8.66 (s, 1H, N=CH-Ar), 6.61 -7.32 (m, 12H, Ar-H) , 2.87 (s, 2H, N-CH_2)	152.73 (1,2,4-triazole,C-5), 145.67 (1,2,4-triazole,C-3), 143.4 (N=CH-Ar), 121.2 (C-Br, aromatic), 114.51 - 127.41 (C of aromatic ring), 37.48 (N-CH_2)	494 [M^+], 461, 282, 212, 198, 166

Comp. No.	IR [KBr, ν, cm^{-1}]	^{1}HNMR [δ, ppm]	^{13}CNMR [δ, ppm]	Mass (m/z)
5e	3050 (aromatic ring), 2589 (-SH), 1597 (>C=N), 1582 (N=CH-Ar), 1532,1340 ($ArNO_2$), 1443 (N-CH_2), 655 (C-S-C)	13.68 (s, 1H, SH), 8.67 (s, 1H, N=CH-Ar), 6.65 -7.36 (m, 12H, Ar-H) , 2.88 (s, 2H, N-CH_2)	152.77 (1,2,4-triazole,C-5), 145.66 (1,2,4-triazole,C-3), 143.8 (N=CH-Ar), 140.2 (C-NO_2, aromatic), 114.52 - 127.42 (C of aromatic ring), 37.52 (N-CH_2)	460 [M^+], 427, 248, 212, 198, 166
5f	3051 (aromatic ring), 2828 (Ar-OCH_3), 2590 (-SH), 1601 (>C=N), 1583 (N=CH-Ar), 1448 (N-CH_2), 656 (C-S-C)	13.67 (s, 1H, SH), 8.70 (s, 1H, N=CH-Ar), 6.64 -7.35 (m, 12H, Ar-H) , 2.92 (s, 2H, N-CH_2), 3.99 (s, 3H, OCH_3)	152.78 (1,2,4-triazole,C-5), 145.70 (1,2,4-triazole,C-3), 143.7 (N=CH-Ar), 114.56 - 127.46 (C of aromatic ring), 55.6 (OCH_3), 37.51 (N-CH_2)	445 [M^+], 412, 233, 212, 198, 166
5g	3055 (aromatic ring), 2922 (Ar-CH_3), 2594 (-SH), 1602 (>C=N), 1584 (N=CH-Ar), 1449 (N-CH_2), 659 (C-S-C)	13.71 (s, 1H, SH), 8.71 (s, 1H, N=CH-Ar), 6.69 -7.40 (m, 12H, Ar-H) , 2.91 (s, 2H, N-CH_2), 2.38 (s, 3H, Ar-CH_3)	152.83 (1,2,4-triazole,C-5), 145.69 (1,2,4-triazole,C-3), 144.0 (N=CH-Ar), 114.57 - 127.47 (C of aromatic ring), 37.56 (N-CH_2), 22.6 (CH_3)	429 [M^+], 396, 217, 212, 198, 166
5h	3056 (aromatic ring), 2912, 2853 (N-CH_3), 2592 (-SH), 1604 (>C=N), 1586 (N=CH-Ar), 1450 (N-CH_2), 660 (C-S-C)	13.73 (s, 1H, SH), 8.72 (s, 1H, N=CH-Ar), 6.67 -7.38 (m, 12H, Ar-H) , 2.93 (s, 2H, N-CH_2), 3.14 (s, 6H, 2×CH_3)	152.81 (1,2,4-triazole,C-5), 145.72 (1,2,4-triazole,C-3), 144.1 (N=CH-Ar), 114.58 - 127.48 (C of aromatic ring), 37.55 (N-CH_2), 23.5 (NCH_3)	458 [M^+], 425, 246, 212, 198, 166
6a	3053 (aromatic ring), 2976 (N-CH-S), 2595 (-SH), 1707 (>C=O, cyclic), 1608 (>C=N), 1452 (N-CH_2), 662 (C-S-C)	13.72 (s, 1H, SH), 6.70 - 7.41 (m, 13H, Ar-H) , 2.95 (s, 2H, N-CH_2), 4.01 (s,1H, -N-CH-), 3.41 (s, 2H, S-CH_2)	171.7 (>C=O, cyclic), 152.84 (1,2,4-triazole,C-5), 145.75 (1,2,4-triazole,C-3), 114.60 - 127.50 (C of aromatic ring), 43.0 (S-CH_2), 40.0 (N-CH-S), 37.57 (N-CH_2)	489 [M^+], 456, 311, 277, 212, 198, 178, 166
6b	3250 (Ar-OH), 3057 (aromatic ring), 2978 (N-CH-S), 2593 (-SH), 1708 (>C=O, cyclic), 1606 (>C=N), 1451 (N-CH_2), 663 (C-S-C)	13.75 (s, 1H, SH), 11.93 (s, 1H, Ar-OH), 6.68 - 7.39 (m, 12H, Ar-H) , 2.96 (s, 2H, N-CH_2), 4.02 (s,1H, -N-CH-), 3.43 (s, 2H, S-CH_2)	171.9 (>C=O, cyclic), 165.7 (C-OH, aromatic), 152.82 (1,2,4-triazole,C-5), 145.73 (1,2,4-triazole,C-3), 114.62 - 127.52 (C of aromatic ring), 43.2 (S-CH_2), 40.1 (N-CH-S), 37.59 (N-CH_2)	505 [M^+], 472, 311, 293, 212, 198, 194, 166
6c	3060 (aromatic ring), 2977 (N-CH-S), 2596 (-SH), 1710 (>C=O, cyclic), 1607 (>C=N), 1453 (N-CH_2), 758 (Ar-Cl), 661 (C-S-C)	13.74 (s, 1H, SH), 6.72 - 7.43 (m, 12H, Ar-H) , 2.98 (s, 2H, N-CH_2), 4.03 (s,1H, -N-CH-), 3.42 (s, 2H, S-CH_2)	172.0 (>C=O, cyclic), 152.86 (1,2,4-triazole,C-5), 145.76 (1,2,4-triazole,C-3), 134.7 (C-Cl, aromatic), 114.64 - 127.54 (C of aromatic ring), 43.1 (S-CH_2), 40.2 (N-CH-S), 37.58 (N-CH_2)	523.5 [M^+], 491, 312, 311, 213, 212, 198, 166
6d	3059 (aromatic ring), 2980 (N-CH-S), 2597 (-SH), 1711 (>C=O, cyclic), 1610 (>C=N), 1455 (N-CH_2), 665 (C-S-C), 622 (Ar-Br)	13.77 (s, 1H, SH), 6.71 - 7.42 (m, 12H, Ar-H) , 2.97 (s, 2H, N-CH_2), 4.04 (s,1H, -N-CH-), 3.45 (s, 2H, S-CH_2)	172.1 (>C=O, cyclic), 152.87 (1,2,4-triazole,C-5), 145.77 (1,2,4-triazole,C-3), 121.3 (C-Br, aromatic), 114.65 - 127.55 (C of aromatic ring), 43.3 (S-CH_2), 40.3 (N-CH-S), 37.63 (N-CH_2)	568 [M^+], 535, 356, 311, 257, 212, 198, 166
6e	3063 (aromatic ring), 2982 (N-CH-S), 2601 (-SH), 1715 (>C=O, cyclic), 1611 (>C=N), 1533,1342 ($ArNO_2$), 1456 (N-CH_2), 666 (C-S-C)	13.80 (s, 1H, SH), 6.75 - 7.46 (m, 12H, Ar-H) , 2.90 (s, 2H, N-CH_2), 4.07 (s,1H, -N-CH-), 3.46 (s, 2H, S-CH_2)	172.2 (>C=O, cyclic), 152.88 (1,2,4-triazole,C-5), 145.78 (1,2,4-triazole,C-3), 140.5 (C-NO_2, aromatic), 114.68 - 127.58 (C of aromatic ring), 43.6 (S-CH_2), 40.6 (N-CH-S), 37.64 (N-CH_2)	534 [M^+], 501, 322, 311, 223, 212, 198, 166
6f	3064 (aromatic ring), 2985 (N-CH-S), 2829 (Ar-OCH_3), 2602 (-SH), 1714 (>C=O, cyclic), 1614 (>C=N), 1459 (N-CH_2), 670 (C-S-C)	13.81 (s, 1H, SH), 6.78 - 7.49 (m, 12H, Ar-H) , 2.88 (s, 2H, N-CH_2), 4.08 (s,1H, -N-CH-), 3.97 (s, 3H, OCH_3), 3.50 (s, 2H, S-CH_2)	172.5 (>C=O, cyclic), 152.89 (1,2,4-triazole,C-5), 145.81 (1,2,4-triazole,C-3), 114.69 - 127.59 (C of aromatic ring), 55.8 (OCH_3), 43.9 (S-CH_2), 40.9 (N-CH-S), 37.65 (N-CH_2)	519 [M^+], 486, 311, 307, 212, 208, 198, 166
6g	3067 (aromatic ring), 2986 (N-CH-S), 2924 (Ar-CH_3), 2603 (-SH), 1718 (>C=O, cyclic), 1615 (>C=N), 1461 (N-CH_2), 673 (C-S-C)	13.84 (s, 1H, SH), 6.80 - 7.51 (m, 12H, Ar-H) , 2.86 (s, 2H, N-CH_2), 4.12 (s,1H, -N-CH-), 3.51 (s, 2H, S-CH_2) , 2.39 (s, 3H, Ar-CH_3)	172.8 (>C=O, cyclic), 152.93 (1,2,4-triazole,C-5), 145.82 (1,2,4-triazole,C-3), 114.72 - 127.62 (C of aromatic ring), 43.8 (S-CH_2), 41.0 (N-CH-S), 37.68 (N-CH_2), 22.7 (CH_3)	503 [M^+], 470, 311, 291, 212, 198, 192, 166
6h	3066 (aromatic ring), 2988 (N-CH-S), 2914, 2854 (N-CH_3), 2605 (-SH), 1720 (>C=O, cyclic), 1618 (>C=N), 1463 (N-CH_2), 672 (C-S-C)	13.85 (s, 1H, SH), 6.79 - 7.50 (m, 12H, Ar-H) , 2.82 (s, 2H, N-CH_2), 4.11 (s,1H, -N-CH-), 3.52 (s, 2H, S-CH_2), 3.15 (s, 6H, 2×CH_3)	173.0 (>C=O, cyclic), 152.95 (1,2,4-triazole,C-5), 145.85 (1,2,4-triazole,C-3), 114.71 - 127.61 (C of aromatic ring), 44.1 (S-CH_2), 41.2 (N-CH-S), 37.67 (N-CH_2), 23.7 (NCH_3)	532 [M^+], 499, 320, 311, 221, 212, 198, 166

Comp. No.	IR [KBr, ν, cm^{-1}]	^{1}HNMR [δ, ppm]	^{13}CNMR [δ, ppm]	Mass (m/z)
7a	3069 (aromatic ring), 2987 (N-CH-S), 2607 (-SH), 1717 (>C=O, cyclic), 1632 (>C=CH-Ar), 1617 (>C=N), 1462 (N-CH_2), 674 (C-S-C)	13.86 (s, 1H, SH), 6.82 - 7.53 (m, 18H, Ar-H) , 5.25 (s, 1H, >C=CH-Ar), 2.89 (s, 2H, N-CH_2), 4.13 (s,1H, -N-CH-)	172.7 (>C=O, cyclic), 152.94 (1,2,4-triazole,C-5), 145.84 (1,2,4-triazole,C-3), 129.1, 125.4 (>C=CH-Ar), 114.75 - 127.65 (C of aromatic ring), 41.1 (N-CH-S), 37.69 (N-CH_2)	577 [M^+], 544, 516, 365, 311, 266, 212, 198, 166
7b	3252 (Ar-OH), 3068 (aromatic ring), 2990 (N-CH-S), 2606 (-SH), 1719 (>C=O, cyclic), 1634 (>C=CH-Ar), 1620 (>C=N), 1464 (N-CH_2), 676 (C-S-C)	13.88 (s, 1H, SH), 11.94 (s, 1H, Ar-OH), 6.81 - 7.52 (m, 16H, Ar-H) , 5.27 (s, 1H, >C=CH-Ar), 2.81 (s, 2H, N-CH_2), 4.14 (s,1H, -N-CH-)	172.9 (>C=O, cyclic), 165.8 (C-OH, aromatic), 152.96 (1,2,4-triazole,C-5), 145.86 (1,2,4-triazole,C-3), 129.3, 125.6 (>C=CH-Ar), 114.73 - 127.63 (C of aromatic ring), 41.3 (N-CH-S), 37.70 (N-CH_2)	609 [M^+], 576, 397, 311, 298, 212, 198, 166
7c	3070 (aromatic ring), 2989 (N-CH-S), 2608 (-SH), 1721 (>C=O, cyclic), 1635 (>C=CH-Ar), 1619 (>C=N), 1466 (N-CH_2), 759 (Ar-Cl), 677 (C-S-C)	13.87 (s, 1H, SH), 6.84 - 7.55 (m, 16H, Ar-H) , 5.26 (s, 1H, >C=CH-Ar), 2.90 (s, 2H, N-CH_2), 4.16 (s,1H, -N-CH-)	173.1 (>C=O, cyclic), 152.99 (1,2,4-triazole,C-5), 145.88 (1,2,4-triazole,C-3), 134.9 (C-Cl, aromatic), 129.5, 125.7 (>C=CH-Ar), 114.76 - 127.66 (C of aromatic ring), 41.5 (N-CH-S), 37.72 (N-CH_2)	646 [M^+], 613, 434, 335, 311, 212, 198, 166
7d	3074 (aromatic ring), 2992 (N-CH-S), 2611 (-SH), 1722 (>C=O, cyclic), 1636 (>C=CH-Ar), 1622 (>C=N), 1467 (N-CH_2), 678 (C-S-C), 624 (Ar-Br)	13.91 (s, 1H, SH), 6.85 - 7.56 (m, 16H, Ar-H) , 5.29 (s, 1H, >C=CH-Ar), 2.93 (s, 2H, N-CH_2), 4.17 (s,1H, -N-CH-)	173.2 (>C=O, cyclic), 152.98 (1,2,4-triazole,C-5), 145.90 (1,2,4-triazole,C-3), 129.4, 125.3 (>C=CH-Ar), 121.4 (C-Br, aromatic), 114.77 - 127.67 (C of aromatic ring), 41.6 (N-CH-S), 37.73 (N-CH_2)	735 [M^+], 702, 523, 495, 424, 311, 212, 198, 166
7e	3075 (aromatic ring), 2993 (N-CH-S), 2612 (-SH), 1724 (>C=O, cyclic), 1637 (>C=CH-Ar), 1623 (>C=N), 1534,1345 (ArNO_2), 1468 (N-CH_2), 679 (C-S-C)	13.92 (s, 1H, SH), 6.86 - 7.57 (m, 16H, Ar-H) , 5.30 (s, 1H, >C=CH-Ar), 2.88 (s, 2H, N-CH_2), 4.20 (s,1H, -N-CH-)	173.3 (>C=O, cyclic), 153.02 (1,2,4-triazole,C-5), 145.93 (1,2,4-triazole,C-3), 140.6 (C-NO_2, aromatic), 129.6, 125.8 (>C=CH-Ar), 114.78 - 127.68 (C of aromatic ring), 41.7 (N-CH-S), 37.74 (N-CH_2)	667 [M^+], 634, 455, 356, 311, 212, 198, 166
7f	3076 (aromatic ring), 2994 (N-CH-S), 2830 (Ar-OCH_3), 2615 (-SH), 1728 (>C=O, cyclic), 1641 (>C=CH-Ar), 1625 (>C=N), 1471 (N-CH_2), 682 (C-S-C)	13.96 (s, 1H, SH), 6.90 - 7.61 (m, 16H, Ar-H) , 5.34 (s, 1H, >C=CH-Ar), 2.87 (s, 2H, N-CH_2), 4.22 (s,1H, -N-CH-), 3.96 (s, 3H, OCH_3)	173.8 (>C=O, cyclic), 153.06 (1,2,4-triazole,C-5), 145.94 (1,2,4-triazole,C-3), 130.0, 126.1 (>C=CH-Ar), 114.83 - 127.73 (C of aromatic ring), 55.9 (OCH_3), 42.0 (N-CH-S), 37.77 (N-CH_2)	637 [M^+], 604, 425, 326, 311, 212, 198, 166
7g	3077 (aromatic ring), 2998 (N-CH-S), 2926 (Ar-CH_3), 2616 (-SH), 1729 (>C=O, cyclic), 1642 (>C=CH-Ar), 1628 (>C=N), 1472 (N-CH_2), 683 (C-S-C)	13.97 (s, 1H, SH), 6.92 - 7.63 (m, 16H, Ar-H) , 5.35 (s, 1H, >C=CH-Ar), 2.88 (s, 2H, N-CH_2), 4.23 (s,1H, -N-CH-), 2.37 (s, 3H, Ar-CH_3)	174.0 (>C=O, cyclic), 153.03 (1,2,4-triazole,C-5), 145.95 (1,2,4-triazole,C-3), 130.1, 126.2 (>C=CH-Ar), 114.84 - 127.74 (C of aromatic ring), 42.1 (N-CH-S), 37.78 (N-CH_2), 22.8 (CH_3)	605 [M^+], 572, 393, 311, 294, 212, 198, 166
7h	3079 (aromatic ring), 2999 (N-CH-S), 2915, 2856 (N-CH_3), 2617 (-SH), 1730 (>C=O, cyclic), 1643 (>C=CH-Ar), 1627 (>C=N), 1474 (N-CH_2), 685 (C-S-C)	13.98 (s, 1H, SH), 6.91 - 7.62 (m, 16H, Ar-H) , 5.36 (s, 1H, >C=CH-Ar), 2.79 (s, 2H, N-CH_2), 4.24 (s,1H, -N-CH-), 3.12 (s, 6H, 2×CH_3)	174.2 (>C=O, cyclic), 153.07 (1,2,4-triazole,C-5), 145.97 (1,2,4-triazole,C-3), 130.3, 126.3, (>C=CH-Ar), 114.82 - 127.72 (C of aromatic ring), 42.3 (N-CH-S), 37.80 (N-CH_2), 23.8 (NCH_3)	663 [M^+], 630, 451, 352, 311, 212, 198, 166

Antimicrobial activity

All newly synthesized compounds were screened for their antibacterial activity against gram positive bacterium i.e. *Bacillus subtilis* and gram negative bacteria i.e. *Escherichia coli, Salmonella typhi* using Nutrient agar medium and antifungal activity against *Aspergillus flavus*, *P.citrinum* and *Fusarium oxysporum* using Potato dextrose agar medium at 50 and 100 µg/mL concentrations by filter paper disk technique[22]. Standard antibacterial Streptomycin and antifungal Griseofulvin were also tested under similar conditions for comparison. The results are summarized in tables 3 and 4.

Table 3. Antibacterial data of the compounds **1-4, 5a-h, 6a-h** and **7a-h.**

Comp.	*E. coli*		*B. subtilis*		*S. typhi*	
	50 µg ml^{-1}	100 µg ml^{-1}	50 µg ml^{-1}	100 µg ml^{-1}	50 µg ml^{-1}	100 µg ml^{-1}
1	4	8	1	4	2	5
2	6	10	2	6	3	7
3	9	12	5	8	7	10
4	7	11	3	6	5	8
5a	12.8	16.9	10	14	11.5	15
5b	14.5	18	11.5	15	13	16
5c	17.5	20	15	17.5	16.2	19
5d	15.5	18.9	13	16.6	14.1	17.5
5e	19.4	22	17	20	18.2	21
5f	11.8	15.9	9	13	10.7	14
5g	10	14	7	10	9	12
5h	11	15	8	12	10	13
6a	17.5	22.1	15	18.4	16	20
6b	20	24.3	16.8	19.5	18	23
6c	24	27.5	19.9	23	23.2	26
6d	22.7	26	18.8	22	22	25
6e	25	29	21	25	24.3	27
6f	16	20	14	17	15	18.5
6g	13.5	17	11	14	12	15.4
6h	15	18	13	16	13.9	17
7a	17	21.1	13.5	16	15.5	18.5
7b	18	22	15.4	16.9	17	19.6
7c	20.3	23.6	17.9	20	19	22
7d	19.5	22.9	16.4	18.5	18	21
7e	22.6	25.8	19	22	21	24
7f	15.8	19	12.5	14.4	14	16.8
7g	12	15	9.4	12.5	10.9	13.5
7h	13	17	11	13.5	12	15
SM[a]	26	30	23	27	25	28

a Streptomycin
Zone of inhibition in mm.

Table 4. Antifungal data of the compounds **1-4, 5a-h, 6a-h** and **7a-h.**

Comp.	*A. flavus*		*P. citrinum*		*F. oxysporum*	
	50 µg ml^{-1}	100 µg ml^{-1}	50 µg ml^{-1}	100 µg ml^{-1}	50 µg ml^{-1}	100 µg ml^{-1}
1	3	6	2	5	5.2	8.5
2	4.5	8	3	6	7.4	10.8
3	7.8	11	6	10	10	13
4	6	9	4	7	9	12
5a	12.5	16	11	14.5	14	17.9
5b	13.4	17	12.2	16	15.6	18.8
5c	17	21	16.5	19.9	18.3	22
5d	15	18.4	14	17	16.4	20
5e	19	22	18.1	21	20	23
5f	11.8	15	10.1	13.9	12.8	16.9
5g	10.2	13	8	11	11.4	15
5h	11	14	9	13	12	16.2
6a	16.9	21	15.7	19	19	22
6b	19	23	17	20	21	25
6c	24	27	21	25	26	29
6d	22	26.1	20	23	24.5	27.4
6e	25	28	22	26	27	30
6f	16.2	20	15	18.1	17	20.8
6g	13	16.8	12.1	15	14.6	18
6h	15	18	13.5	16.8	16	19.2
7a	16.2	20	14.1	16.7	18	21
7b	18.1	21	16	19	19	22.5
7c	19.6	23	18.1	21	21.9	25.9
7d	18.9	22	17	20	21	24
7e	21.8	24.5	20	23	23.1	27
7f	14.3	18	13.5	16	16.5	20
7g	11.5	14	10	13	13	16
7h	13	16	12	14.6	14	18
GR[a]	27	30	25	28	28	32

a Griseofulvin
Zone of inhibition in mm.

The investigation of antimicrobial screening data revealed that all compounds showed considerable and varied activity against the pathogenic bacteria and fungi. Compound **6e** showed the maximum antibacterial activity against the *E.coli* and antifungal activity against *F.oxysporum* which may be attributed to the aromatic ring and nitro group. Compounds **5e, 6c, 6d, 7c** and **7e** have shown significant antimicrobial activity while compounds **5c, 5d, 6a, 6b, 6f, 6h, 7a, 7b** and **7d** have shown good antimicrobial activity, the rest of the compounds have shown moderate activity. However, the activities of the synthesized compounds are less than that of standard antibacterial and antifungal drugs.

Conclusion

A simple and efficient method was developed for the synthesis of 4-oxo-thiazolidines and their 5-arylidene derivatives having a phenothiazine nucleus. It is believed that the procedural simplicity, efficiency and the easy accessibility of the reaction patterns could give access to a wide array of heterocyclic frameworks equipped with a phenothiazine unit. Microwave irradiation technique was used. This is a solvent free reaction that leads to considerable saving in the reaction time and energetically profitable. The solvent free condition contributes to saving in cost and diminishes the waste disposal problem. Some compounds have shown significant antibacterial and antifungal activities.

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