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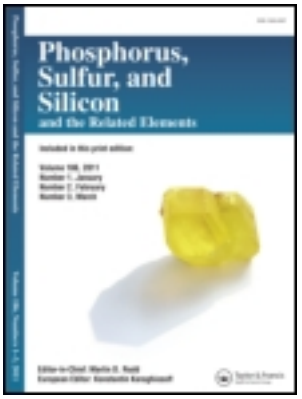

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Synthesis and Spectral Characterization of Some New Thiazolyl-Pyrazolines Bearing 1,2,4-Triazole Moiety

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SYNTHESIS AND SPECTRAL CHARACTERIZATION OF SOME NEW THIAZOLYL-PYRAZOLINES BEARING 1,2,4-TRIAZOLE MOIETY

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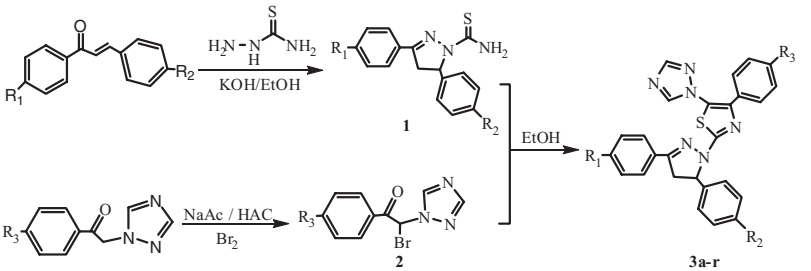
GRAPHICAL ABSTRACT



Abstract *In this study, several new 1-(4-aryl-5-triazolyl-2-thiazolyl)-3,5-diaryl-2-pyrazolines (**3a–r**) were synthesized by reacting 3,5-diaryl1-thiocarbamoyl-2-pyrazolines (**1**) with 2-bromo-1-aryl-2-(1H-1,2,4-triazol-1-yl) ethanones (**2**) in boiling ethanol. The chemical structures of the compounds were verified by IR, ^{1}H NMR, ESI-MS spectroscopic data, and elemental analyses.*

Keywords Carbothioamide; pyrazoline; thiazole; 1,2,4-triazole

INTRODUCTION

1,2,4-Triazole derivatives are of great interest in the medicinal and agricultural fields due to their broad spectrum pharmacological activities, such as insecticidal, antifungal, antibacterial, anti-HIV, COX-2 inhibitor, anticonvulsant, and plant growth regulative properties,[1–6] and quite a few of them have become commercial products (i.e., fluconazole, itraconazole).[7,8] Similarly, thiazole and pyrazoline derivatives are also well known nitrogen-containing heterocyclic compounds, which exhibit a large number of biological activities.[9–18] It was therefore of interest to combine these potential biologically active units to give fused derivatives.

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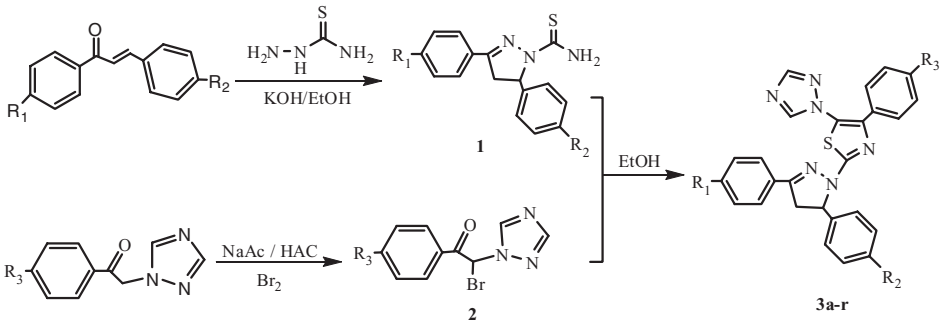
A literature survey showed that thiazole compounds containing both the 1,2,4-triazole group and the pyrazoline group in a single molecule have not been reported so far. Encouraged by these facts, and in continuation of our interest in the synthesis of chemically and biologically important heterocycles containing the 1,2,4-triazole moiety,[19] a series of novel heterocyclic compounds including 1,2,4-triazole, pyrazoline, and thiazole (**3a–r**) were synthesized, which might exhibit enhanced activities owing to the incorporation of different pharmacophores into their structures.

RESULTS AND DISCUSSION

The synthetic routes leading to the desired compounds are shown in Scheme 1. 1,3-Diaryl-2-propenones (chalcones) were readily obtained by the reaction of aromatic aldehydes and 1-arylethanones by a Claisen–Schmidt condensation reaction in good yield. The chalcones were subsequently reacted with thiosemicarbazide in the presence of sodium hydroxide in ethanol, to give 1-thiocarbamoyl-3,5-diphenyl-pyrazoline derivatives (**1**) according to the methods described in the literature.[15,20–22]



(3a) R_1=R_2=R_3=H
(3b) R_1=R_2=H,R_3=Cl
(3c) R_1=R_2=H,R_3=OCH_3
(3d) R_1=R_3=H,R_2=Cl
(3e) R_1=H,R_2=R_3=Cl
(3f) R_1=H,R_2=Cl,R_3=OCH3
(3g) R_1=R_3=H,R_2=OCH_3
(3h) R_1=H,R_2=OCH_3,R_3=Cl
(3e) R_1=H,R2=R_3=OCH_3
(3j) R_1=Cl,R_2=R_3=H
(3k) R_1=R_3=Cl,R_2=H
(3l) R_1=Cl,R_2=H,R_3=OCH_3
(3m) R_1=R_2=Cl,R_3=H
(3n) R_1=R_2=R_3=Cl
(3o) R_1=R_2=Cl,R_3=OCH3
(3p) R_1=Cl,R_2=OCH_3,R_3=H
(3q) R_1=R_3=Cl,R_2=OCH_3
(3r) R_1=Cl,R2=R_3=OCH_3

Scheme 1

2-Bromo-1-arylethanones, prepared by the reaction of 1-arylethanones with bromine in anhydrous ether, reacted with 1H-1,2,4-triazole in the presence of triethylamine in acetone to give 1-aryl-2-(1H-1,2,4-triazol-1-yl)-ethanone, which was brominated in the presence of sodium acetate/acetic acid to afford the corresponding 2-bromo-1-aryl-2-(1H-1,2,4-triazol-1-yl)-ethanones (**2**).[23,24]

The condensation of 2-bromo-1-arylethanones (**2**) with pyrazolines (**1**) in refluxing ethanol resulted in the formation of the desired cyclized products 2-(3,5-diaryl-4,5-dihydro-1H-pyrazol-1-yl)-4-aryl-5-(1H-1,2,4-triazol-1-yl) thiazoles (**3a–r**, Table 1) in 75–95%

Table 1 Physical data of compounds (**3a–r**)

(%) Analyses, Calcd./Found			Molecular formula	Yield (%)	Mp, °C	Compound no.
N	H	C				
18.74/18.78	4.49/4.48	69.62/69.57	$C_{26}H_{20}N_6S$	92	110–112	**3a**
17.40/17.34	3.97/4.01	64.66/64.69	$C_{26}H_{19}ClN_6S$	90	182–183	**3b**
17.56/17.52	4.63/4.57	67.76/67.79	$C_{27}H_{22}N_6OS$	94	170–171	**3c**
17.40/17.51	3.97/4.00	64.66/64.71	$C_{26}H_{19}ClN_6S$	80	176–177	**3d**
16.24/16.33	3.51/3.56	60.35/60.28	$C_{26}H_{18}Cl_2N_6S$	80	213–214	**3e**
16.38/16.45	4.13/4.03	63.21/63.33	$C_{27}H_{21}ClN_6OS$	89	116–117	**3f**
17.56/17.63	4.63/4.67	67.76/67.68	$C_{27}H_{22}N_6OS$	94	171–173	**3g**
16.38/16.43	4.13/4.08	63.21/63.32	$C_{27}H_{21}ClN_6OS$	90	176–177	**3h**
16.52/16.55	4.76/4.65	66.12/66.23	$C_{28}H_{24}N_6O_2S$	94	175–177	**3i**
17.40/17.44	3.97/3.88	64.66/64.72	$C_{26}H_{19}ClN_6S$	88	198–199	**3j**
16.24/16.20	3.51/3.54	60.35/60.32	$C_{26}H_{18}Cl_2N_6S$	81	213–214	**3k**
16.38/16.43	4.13/4.21	63.21/63.27	$C_{27}H_{21}ClN_6OS$	92	163–164	**3l**
16.24/16.27	3.51/3.62	60.35/60.42	$C_{26}H_{18}Cl_2N_6S$	80	204–205	**3m**
15.23/15.27	3.10/3.14	56.58/56.59	$C_{26}H_{17}Cl_3N_6S$	76	256–258	**3n**
15.35/15.33	3.68/3.72	59.24/59.41	$C_{27}H_{20}Cl_2N_6OS$	84	214–215	**3o**
16.38/16.44	4.13/4.12	63.21/63.28	$C_{27}H_{21}ClN_6OS$	90	192–193	**3p**
15.35/15.41	3.68/3.63	59.24/59.33	$C_{27}H_{20}Cl_2N_6OS$	84	208–210	**3q**
15.48/15.44	4.27/4.22	61.93/61.99	$C_{28}H_{23}ClN_6O_2S$	93	241–242	**3r**

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yield. The progress of each reaction was monitored by thin layer chromatography. Compounds (**3a–r**) were characterized by IR, ^{1}H NMR spectroscopy, and mass spectrometry (Scheme 1).

The IR spectral, mass spectra, and ^{1}H NMR spectral data gave strong evidence for the structures of **3a–r** (Table 2). The IR spectra of **3a–r** exhibit bands at 1631–1632 cm^{-1}(C=N). In addition, their ^{1}H NMR spectra revealed a multiplet between δ 7.79–6.73 ppm due to the protons of aromatic rings, the signal for 1,2,4-triazole, and methoxyl appeared at δ 8.19–8.06 ppm and δ 3.82–3.74 ppm, respectively. Three distinct double of doublets of the ABX system (a CH proton and two anisochronous protons of a CH_2) appeared at δ 5.71–3.29 ppm, as has been observed in the pyrazoline ring.[15] The EI mass spectra of compounds **3a–r** revealed the existence of the molecular ion peaks and anticipated fragmentation peaks, which were in good agreement with the given structures of products. For example, the mass spectrum of **3a** had molecular ion peaks at m/z 448 (100%), 380 (M-$C_2H_2N_3$ 2%), 221 (M-$C_6H_5C_2H_2N_3C_3NS$ 78%), 117 ($C_6H_5CN_2$ 23%), 104 ($C_6H_5C_2H_3$ 77%), 77 (C6H5 49%), consistent with the molecular formula.

The chapped mechanism of the mass spectrum is showed in Figure 1. The IR spectrum of compound **3i** showed the stretching band of C=N at 1632 cm^{-1}, and its ^{1}H NMR spectrum showed the signals of H_a, H_b, H_x of pyrazoline ring as doublet of doublets in the regions 3.40–3.35 ppm (H_a) and 3.96–3.88 ppm (H_b). The CH (H_x) proton appeared as a doublet of doublets at 5.70–5.65 ppm due to vicinal coupling with the two magnetically nonequivalent protons of the methylene group at position 4 of pyrazoline ring ($J_{ab} = 17.60$, $J_{ax} = 12.01$, and $J_{bx} = 5.99$ Hz). Two sharp singlets showed at δ 3.81ppm and δ 3.76 ppm in the ^{1}H NMR spectrum due to its methoxyl protons. The remaining 13 aromatic protons resonated as multiplets in the range δ 7.78–6.73.[15]

Table 2 IR (cm^{-1}), ^1H NMR (δppm), and mass spectral data of compounds (**3a–r**)

Compound no.	IR (cm^{-1}) C=N	^1H NMR($CDCl_3$)	MS(EI): m/z(M^+)
3a	1631	δ 8.06,8.19(2H,2s,Triazole-H),6.96–7.73(15H,m,phenyl-H), 5.71 (dd,1H,C_5-H_x,J_{ax} = 12.0Hz,J_{bx} = 6.0Hz), 3.95 (dd,1H,C_4-H_a,J_{ax} = 12.0Hz,J_{ab} = 17.60Hz),3.39(dd,1H,C_4-H_b,J_{bx} = 6.0Hz,J_{ab} = 17.6Hz).	448
3b	1631	δ 8.06,8.19(2H,2s,Triazole-H),7.00–7.77(14H,m,phenyl-H), 5.69 (dd,1H,C_5-H_x,J_{ax} = 12.0Hz,J_{bx} = 6.3Hz), 3.93 (dd,1H,C_4-H_a,J_{ax} = 12.0Hz,J_{ab} = 17.6Hz),3.31(dd,1H,C_4-H_b,J_{bx} = 6.3Hz,J_{ab} = 17.6Hz).	482
3c	1632	δ 8.06,8.19(2H,2s,Triazole-H),7.03–7.79(14H,m,phenyl-H), 5.68 (dd,1H,C_5-H_x,J_{ax} = 12.0Hz,J_{bx} = 6.33Hz), 3.92 (dd,1H,C_4-H_a,J_{ax} = 12.0Hz,J_{ab} = 17.6Hz), 3.75(3H,s,CH_3O), 3.30(dd,1H,C_4-H_b,J_{bx} = 6.3Hz,J_{ab} = 17.6Hz).	478
3d	1631	δ 8.06,8.18(2H,2s,Triazole-H),7.01–7.70(14H,m,phenyl-H), 5.71 (dd,1H,C_5-H_x,J_{ax} = 11.9Hz,J_{bx} = 6.4Hz), 3.89 (dd,1H,C_4-H_a,J_{ax} = 11.9Hz,J_{ab} = 17.6Hz),3.38(dd,1H,C_4-H_b,J_{bx} = 6.42Hz,J_{ab} = 17.6Hz).	482
3e	1632	δ 8.06,8.18(2H,2s,Triazole-H),6.98–7.76(13H,m,phenyl-H), 5.70 (dd,1H,C_5-H_x,J_{ax} = 12.00Hz,J_{bx} = 6.4Hz), 3.90 (dd,1H,C_4-H_a,J_{ax} = 12.00Hz,J_{ab} = 17.6Hz),3.40(dd,1H,C_4-H_b,J_{bx} = 6.4Hz,J_{ab} = 17.60Hz).	516
3f	1632	δ 8.06,8.18(2H,2s,Triazole-H),7.00–7.77(13H,m,phenyl-H), 5.68 (dd,1H,C_5-H_x,J_{ax} = 12.0Hz,J_{bx} = 6.0Hz), 3.91 (dd,1H,C_4-H_a,J_{ax} = 12.0Hz,J_{ab} = 17.6Hz), 3.76(3H,s,CH_3O), 3.37(dd,1H,C_4-H_b,J_{bx} = 6.0Hz,J_{ab} = 17.6Hz).	512
3g	1631	δ 8.06,8.16(2H,2s,Triazole-H),6.74–7.79(14H,m,phenyl-H), 5.71 (dd,1H,C_5-H_x,J_{ax} = 11.9Hz,J_{bx} = 6.2Hz), 3.93 (dd,1H,C_4-H_a,J_{ax} = 11.9Hz,J_{ab} = 17.6Hz), 3.82(3H,s,CH_3O), 3.35(dd,1H,C_4-H_b,J_{bx} = 6.2Hz,J_{ab} = 17.6Hz).	478
3h	1631	δ 8.06,8.16(2H,2s,Triazole-H),7.00–7.79(13H,m,phenyl-H), 5.67 (dd,1H,C_5-H_x,J_{ax} = 11.9Hz,J_{bx} = 6.3Hz), 3.94 (dd,1H,C_4-H_a,J_{ax} = 11.9Hz,J_{ab} = 17.6Hz), 3.80(3H,s,CH_3O), 3.32(dd,1H,C_4-H_b,J_{bx} = 6.3Hz,J_{ab} = 17.6Hz).	512
3i	1632	δ 8.06,8.16(2H,2s,Triazole-H),6.73–7.78(13H,m,phenyl-H), 5.67 (dd,1H,C_5-H_x,J_{ax} = 12.0Hz,J_{bx} = 6.0Hz), 3.92 (dd,1H,C_4-H_a,J_{ax} = 12.0Hz,J_{ab} = 17.6Hz),3.81(3H,s,CH_3O), 3.76(3H,s,CH_3O),3.38 (dd,1H,C_4-H_b,J_{bx} = 6.0Hz, J_{ab} = 17.6Hz).	508
3j	1632	δ 8.06,8.19(2H,2s,Triazole-H),6.89–7.70(14H,m,phenyl-H), 5.68 (dd,1H,C_5-H_x,J_{ax} = 12.0Hz,J_{bx} = 6.0Hz), 3.95 (dd,1H,C_4-H_a,J_{ax} = 12.0Hz,J_{ab} = 17.6Hz),3.39(dd,1H,C_4-H_b,J_{bx} = 6.0Hz, J_{ab} = 17.6Hz).	482
3k	1631	δ 8.06,8.19(2H,2s,Triazole-H),7.00–7.70(13H,m,phenyl-H), 5.71 (dd,1H,C_5-H_x,J_{ax} = 11.9Hz,J_{bx} = 6.1Hz), 3.94 (dd,1H,C_4-H_a,J_{ax} = 11.9Hz,J_{ab} = 17.6Hz),3.37(dd,1H,C_4-H_b,J_{bx} = 6.1Hz,J_{ab} = 17.6Hz).	516
3l	1632	δ 8.06,8.19(2H,2s,Triazole-H),7.00–7.71(13H,m,phenyl-H), 5.66 (dd,1H,C_5-H_x,J_{ax} = 12.0Hz,J_{bx} = 6.2Hz), 3.91 (dd,1H,C_4-H_a,J_{ax} = 12.0Hz,J_{ab} = 17.6Hz), 3.74(3H,s,CH_3O), 3.38(dd,1H,C_4-H_b,J_{bx} = 6.2Hz,J_{ab} = 17.6Hz).	512
3m	1631	δ 8.06,8.16(2H,2s,Triazole-H),6.82–7.70(13H,m,phenyl-H) ,5.69 (dd,1H,C_5-H_x,J_{ax} = 11.9Hz,J_{bx} = 6.0Hz), 3.94 (dd,1H,C_4-H_a,J_{ax} = 11.9Hz,J_{ab} = 17.6Hz),3.32(dd,1H,C_4-H_b,J_{bx} = 6.0Hz,J_{ab} = 17.6Hz).	516
3n	1632	δ 8.06,8.16(2H,2s,Triazole-H),6.75–7.70(12H,m,phenyl-H) ,5.71 (dd,1H,C_5-H_x,J_{ax} = 12.0Hz,J_{bx} = 6.0Hz), 3.90 (dd,1H,C_4-H_a,J_{ax} = 12.0Hz,J_{ab} = 17.6Hz),3.29(dd,1H,C_4-H_b,J_{bx} = 6.0Hz,J_{ab} = 17.6Hz).	552
3o	1631	δ 8.06,8.16(2H,2s,Triazole-H),6.73–7.69(12H,m,phenyl-H), 5.70 (dd,1H,C_5-H_x,J_{ax} = 12.0Hz,J_{bx} = 6.4Hz), 3.92 (dd,1H,C_4-H_a,J_{ax} = 12.0Hz,J_{ab} = 17.6Hz), 3.76(3H,s,CH_3O), 3.30(dd,1H,C_4-H_b,J_{bx} = 6.4Hz,J_{ab} = 17.6Hz).	546

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Table 2 IR (cm^{-1}), ^1H NMR (δppm), and mass spectral data of compounds (**3a–r**) *(Continued)*

Compound no.	IR(cm^{-1}) C=N	^1H NMR($CDCl_3$)	MS(EI): m/z(M^+)
3p	1631	δ 8.06,8.17(2H,2s,Triazole-H),6.83–7.69(13H,m,phenyl-H), 5.71 (dd,1H,C_5-H_x,J_{ax} = 12.0Hz,J_{bx} = 6.4Hz), 3.89 (dd,1H,C_4-H_a,J_{ax} = 12.0Hz,J_{ab} = 17.6Hz), 3.80(3H,s,CH_3O), 3.29(dd,1H,C_4-H_b,J_{bx} = 6.4Hz,J_{ab} = 17.6Hz).	512
3q	1632	δ 8.06,8.17(2H,2s,Triazole-H),6.89–7.70(12H,m,phenyl-H), 5.67 (dd,1H,C_5-H_x,J_{ax} = 12.0Hz,J_{bx} = 6.0Hz), 3.90 (dd,1H,C_4-H_a,J_{ax} = 12.0Hz,J_{ab} = 17.6Hz), 3.81(3H,s,CH_3O), 3.35(dd,1H,C_4-H_b,J_{bx} = 6.0Hz,J_{ab} = 17.6Hz).	546
3r	1631	δ 8.06,8.17(2H,2s,Triazole-H),6.81–7.69(12H,m,phenyl-H), 5.68 (dd,1H,C_5-H_x,J_{ax} = 11.9Hz,J_{bx} = 6.0Hz), 3.91 (dd,1H,C_4-H_a,J_{ax} = 11.9Hz,J_{ab} = 17.6Hz),3.82(3H,s,CH_3O), 3.74(3H,s,CH_3O),3.34(dd,1H,C_4-H_b,J_{bx} = 6.0Hz,J_{ab} = 17.6Hz).	542

EXPERIMENTAL

Melting points were recorded on a Mettler FP-5 capillary melting point apparatus and are uncorrected. Elemental analyses were recorded on a Perkin-Elmer 2400 elemental analyzer. The IR spectra were determined as potassium bromide pellets on a Bruker Equinox 55 FT-IR spectrophotometer. The ^1H NMR spectra were recorded on a Varian Inova-400

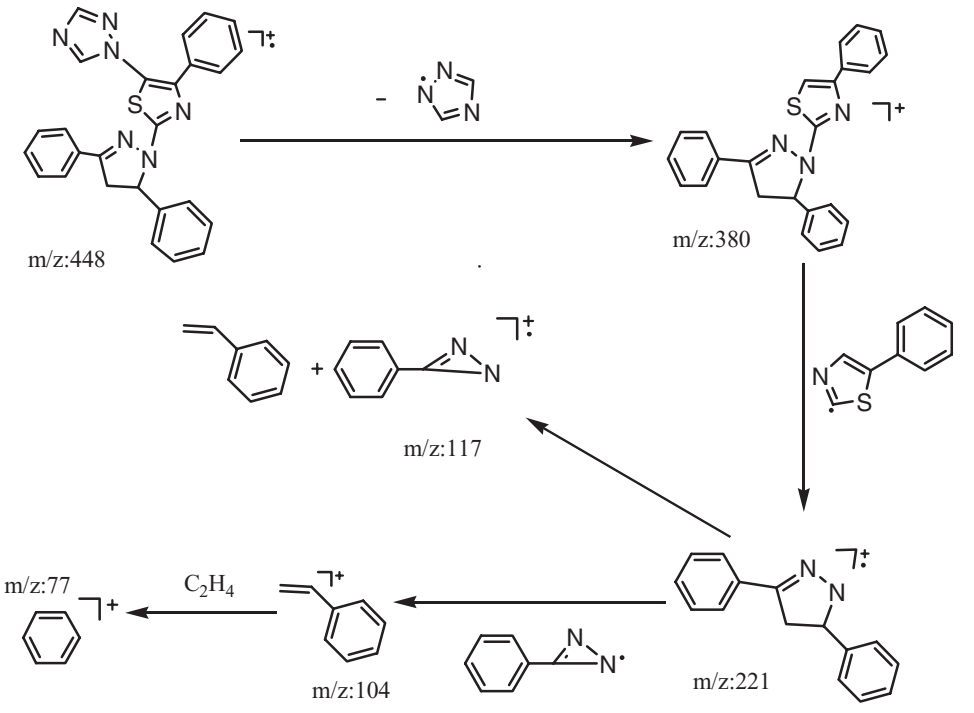

Figure 1 Synthesis of compound **3a**.

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spectrophotometer using TMS as an internal standard. EI-MS spectra were obtained with an Agilent 5975 apparatus.

1-Thiocarbamoyl-3,5-diphenyl-pyrazoline derivatives (**1**) were prepared according to the reported methods.[15,20–22] Triazolylethanones and bromine substituted triazolylethanones (**2**) were prepared according to the reported methods.[23,24]

General Procedure (3a–3r): Synthesis of 2-(3,5-Diphenyl-4,5-dihydro-1H-pyrazol-1-yl)-4-phenyl-5-(1H-1,2,4-triazol-1-yl)-thiazole (3a)

A mixture of 3,5-diphenyl-4,5-dihydro-1H-pyrazole-1-carbothioamide (0.5 mmol) and 2-bromo-1-phenyl-2-(1H-1,2,4-triazol-1-yl) ethanone (0.5 mmol) in ethanol (30 mL) was heated under reflux for 0.5 h. Upon standing the reaction mixture at 25°C, the crude product (**3a**) separated, which was filtered, washed with cold ethanol, dried, and recrystallized From ethanol.

Compounds (**3b–r**) were obtained using the same procedure (Scheme 1). The physical data of the compounds prepared (**3a–r**) are shown in Table 1.

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