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Efficient one-pot three-component synthesis of thiazolyl pyrazole derivatives under conventional method

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ABSTRACT

A series of 4-substituted-N-((1,3-diaryl-1H-pyrazol-4-yl)methylene)thiazol-2-amines were synthesized by the one-pot three component condensation of substituted phenacyl bromide / 3-(2-bromoacetyl)-2H-chromen-2-one with 1,3-diaryl-1H-pyrazole-4-carbaldehyde and thiourea under conventional heating in absolute ethanol using catalytic amount of acetic acid with good yields. The structure of the compounds was established by their spectral studies.

Keywords: Conventional heating, 1,3-Diaryl-1*H*-pyrazole-4-carbaldehyde, One-pot three component condensation, 4-Substituted-*N*-((1,3-diaryl-1*H*-pyrazol-4-yl)methylene)-thiazol-2-amines.

INTRODUCTION

In recent times, multicomponent reactions (MCRs) have attracted the researchers worldwide, because of their superior synthetic strategy over conventional organic reactions in the preparation of complex molecules with high efficiency and atom economy in minimum time from the readily available substrates in a single synthetic operation [1,2].

Literature survey revealed that the thiazole and their synthetic analogs were found to possess various biological and therapeutic properties such as antimicrobial [3,4,], anti-pseudomonal [5], antiallergic [6], antihypertensive [7], anti-inflammatory [8], anti-HIV [9], antiviral [10], antitubercular [11], antifilarial [12], urokinase inhibitors [13], fibrinogen receptor antagonists with antithrombotic [14] activities. On the other hand pyrazole derivatives were also possess antimicrobial [15-17], antioxidant [18,19], anti-inflammatory [20-23], cytotoxic [24], antitumor [25], antimalarial [26], estrogen receptor-α-selective agonists [27]. Because of the pharmacological importance of these moieties, several methods have been reported for the synthesis of thiazolyl pyrazole derivatives such as under conventional heating [28,29] and ultrasound irradiation [30] methods. Many of these reported methods suffer from one or several drawbacks such as low yield, long reaction time, multistep process and harsh reaction conditions. To overcome the above limitations, and for continuation of our studies toward the synthesis of biologically potent heterocyclic compounds [30-34]. Herein, we are reporting the highly efficient protocol for the synthesis of thiazolyl pyrazole derivatives under conventional heating *via* multicomponent approach in ethanol using catalytic amount of acetic acid in good yields.

MATERIALS AND METHODS

Experimental

All the melting points were determined in open capillaries using Stuart SMP30 melting point apparatus, and are uncorrected. The progress of the reaction was monitored by TLC and visualized with UV light and iodine vapors. IR spectra were recorded on Perkin–Elmer 100S spectrophotometer using KBr pellet, values are expressed in cm^{-1}. The C, H and N analysis of the compounds were done on a Carlo Erba modal EA1108, ^1H NMR spectra were recorded on Brucker 300-MHz spectrometer using TMS as an internal standard and chemical shifts were expressed in ppm. Mass spectra were recorded on a Jeol JMSD-300 spectrometer.

General procedure for the synthesis of thiazolyl pyrazole derivatives (7a-l):

A mixture of substituted phenacyl bromide/3-(2-bromoacetyl)-2*H*-chromen-2-one (1 mmol), thiourea (1 mmol) and 3-disubstituted-1*H*-pyrazole-4-carbaldehyde (1 mmol) were taken in 10 mL of absolute ethanol in catalytic amount of acetic acid and heated at refluxing temperature for 6-8 hrs. Monitored the reaction by TLC and poured the contents in an ice cold water, the solid obtained was filtered, dried and recrystallized in ethanol.

Characterization data

4-(4-Chlorophenyl)-*N*-((1,3-diphenyl-1*H*-pyrazol-4-yl)methylene)thiazol-2-amine (7a)

White solid; mp. 259-261 ^oC; IR (KBr, cm^{-1}) υ_{max}: 1622 (C=N), 1578 (C=C), 861(C-Cl); ^1H NMR (300MHz, DMSO-d_6): δ 7.15-7.77 (m, 14H), 7.92 (s, 1H), 7.94 (s, 1H), 8.49 (s, 1H); MS (ESI), 70 eV, *m/z*: 441 (M+H); Anal. Calcd. for $C_{25}H_{17}ClN_4S$: C, 68.10; H, 3.89; N, 12.71; Found: C, 68.23; H, 3.95; N, 12.62.

4-(4-Bromophenyl)-*N*-((1,3-diphenyl-1*H*-pyrazol-4-yl)methylene)thiazol-2-amine (7b)

White solid; mp. 254-255 ^oC; IR (KBr, cm^{-1}) υ_{max}: 1625 (C=N), 1574 (C=C), 761 (C-Br); ^1H NMR (300MHz, DMSO-d_6): δ 7.23-7.81 (m, 14H), 7.91 (s, 1H), 8.02 (s, 1H), 8.34 (s, 1H); MS (ESI), 70 eV, *m/z*: 486 (M+H); Anal. Calcd. for $C_{25}H_{17}BrN_4S$: C, 61.86; H, 3.53; N, 11.54; Found: C, 61.71; H, 3.64; N, 11.69.

4-(4-Nitrophenyl)-*N*-((1,3-diphenyl-1*H*-pyrazol-4-yl)methylene)thiazol-2-amine (7c)

Yellow solid; mp. 251-253 ^oC; IR (KBr, cm^{-1}) υ_{max}: 1617 (C=N), 1574 (C=C), 1461 (NO_2); ^1H NMR (300MHz, DMSO-d_6): δ 7.09-7.88 (m, 14H), 7.90 (s, 1H), 7.98 (s, 1H), 8.28 (s, 1H); MS (ESI), 70 eV, *m/z*: 452 (M+H); Anal. Calcd. for $C_{25}H_{17}N_5O_2S$: C, 66.50; H, 3.80; N, 15.51; Found: C, 66.38; H, 3.92; N, 15.66.

***N*-((1,3-Diphenyl-1*H*-pyrazol-4-yl)methylene)-4-*p*-tolylthiazol-2-amine (7d)**

Yellow solid; mp. 152-154 ^oC; IR (KBr, cm^{-1}) υ_{max}: 1618 (C=N), 1581 (C=C); ^1H NMR (300MHz, DMSO-d_6): δ 1.90 (s, 3H), 7.12-7.77 (m, 14H), 7.91 (s, 1H), 7.97 (s, 1H), 8.25 (s, 1H); MS (ESI), 70 eV, *m/z*: 421 (M+H); Anal. Calcd. for $C_{26}H_{20}N_4S$: C, 74.26; H, 4.79; N, 13.32; Found: C, 74.35; H, 4.84; N, 13.18.

(4-Biphenyl-4-yl-thiazol-2-yl)-(1,3-diphenyl-1*H*-pyrazol-4-yl)methylene)amine (7e)

White solid; mp. 190-192 ^oC; IR (KBr, cm^{-1}) υ_{max}: 1620 (C=N), 1589 (C=C); ^1H NMR (300MHz, DMSO-d_6): δ 7.02-7.87 (m, 19H), 7.90 (s, 1H), 7.96 (s, 1H), 8.36 (s, 1H); MS (ESI), 70 eV, *m/z*: 483 (M+H); Anal. Calcd. for $C_{31}H_{22}N_4S$: C, 77.15; H, 4.59; N, 11.61; Found: C, 77.04; H, 4.70; N, 11.79.

3-(2-((1,3-Diphenyl-1*H*-pyrazol-4-yl)methyleneamino)thiazol-4yl)-2*H*-chromen-2-one (7f)

Light green solid; mp. 194-196 ^oC; IR (KBr, cm^{-1}) υ_{max}: 1733 (C=O), 1632 (C=N), 1579 (C=C); ^1H NMR (300MHz, DMSO-d_6): δ 7.02-7.87 (m, 14H), 7.91 (s, 1H), 7.95 (s, 1H), 8.12 (s, 1H), 8.28 (s, 1H); MS (ESI), 70 eV, *m/z*: 475 (M+H); Anal. Calcd. for $C_{28}H_{18}N_4O_2S$: C, 70.87; H, 3.82; N, 11.81; Found: C, 70.71; H, 3.98; N, 11.88.

4-(4-Chlorophenyl)-*N*-((3-(4-chlorophenyl)-1-phenyl-1*H*-pyrazol-4-yl)methylene)thiazol-2-amine (7g)

Yellow solid; mp. 93-95 ^oC; IR (KBr, cm^{-1}) υ_{max}: 1622 (C=N), 1598 (C=C), 762 (C-Cl); ^1H NMR (300MHz, DMSO-d_6): δ 7.03-7.87 (m, 13H), 7.89 (s, 1H), 7.98 (s, 1H), 8.38 (s, 1H); MS (ESI), 70 eV, *m/z*: 476 (M+H); Anal. Calcd. for $C_{25}H_{16}Cl_2N_4S$: C, 63.16; H, 3.39; N, 11.79; Found: C, 63.30; H, 3.46; N, 11.53.

4-(4-Bromophenyl)-*N*-((3-(4-chlorophenyl)-1-phenyl-1*H*-pyrazol-4-yl)methylene)thiazol-2-amine (7h)

Yellow solid; mp. 80-82 ^oC; IR (KBr, cm^{-1}) υ_{max}: 1618 (C=N), 1598 (C=C), 762 (C-Cl), 651 (C-Br); ^1H NMR (300MHz, DMSO-d_6): δ 7.09-7.89 (m, 13H), 7.87 (s, 1H), 7.91 (s, 1H), 8.32 (s, 1H); MS (ESI), 70 eV, *m/z*: 520 (M+H); Anal. Calcd. for $C_{25}H_{16}BrClN_4S$: C, 57.76; H, 3.10; N, 10.78; Found: C, 57.87; H, 3.22; N, 10.61.

***N*-((3-(4-Chlorophenyl)-1-phenyl-1*H*-pyrazol-4-yl)methylene)-4-(4-nitrophenyl)thiazol-2-amine (7i)**
Orange solid; mp. 264-265 ^{o}C; IR (KBr, cm^{-1}) υ_{max}: 1613 (C=N), 1598 (C=C), 1459 (NO_2), 762 (C-Cl); ^{1}H NMR (300MHz, DMSO-d_6): δ 7.01-7.88 (m, 13H), 7.89 (s, 1H), 7.92 (s, 1H), 8.35 (s, 1H); MS (ESI), 70 eV, *m/z*: 486 (M+H); Anal. Calcd. for $C_{25}H_{16}ClN_5O_2S$: C, 61.79; H, 3.32; N, 14.41; Found: C, 61.93; H, 3.42; N, 14.27.

***N*-((3-(4-Chlorophenyl)-1-phenyl-1*H*-pyrazol-4-yl)methylene)-4-*p*-tolylthiazol-2-amine (7j)**
Yellow solid; mp. 90-92 ^{o}C; IR (KBr, cm^{-1}) υ_{max}: 1619 (C=N), 1586 (C=C), 762 (C-Cl); ^{1}H NMR (300MHz, DMSO-d_6): δ 1.98 (s, 3H), 6.99-7.87 (m, 13H), 7.88 (s, 1H), 7.89 (s, 1H), 8.33 (s, 1H); MS (ESI), 70 eV, *m/z*: 455 (M+H); Anal. Calcd. for $C_{26}H_{19}ClN_4S$: C, 68.64; H, 4.21; N, 12.31; Found: C, 68.54; H, 4.36; N, 12.49.

(4-Biphenyl-4-yl-thiazol-2-yl)-[3-(4-Chlorophenyl)-1-phenyl-1*H*-pyrazol-4-ylmethylene] amine (7k)
Yellow solid; mp. 189-190 ^{o}C; IR (KBr, cm^{-1}) υ_{max}: 1615 (C=N), 1579 (C=C), 764 (C-Cl); ^{1}H NMR (300MHz, DMSO-d_6): δ 7.02-7.87 (m, 18H), 7.91 (s, 1H), 7.95 (s, 1H), 8.29 (s, 1H); MS (ESI), 70 eV, *m/z*: 518 (M+H); Anal. Calcd. for $C_{31}H_{21}ClN_4S$: C, 72.01; H, 4.09; N, 10.84; Found: C, 72.33; H, 4.17; N, 10.62.

3-(2-((3-(4-Chlorophenyl)-1-phenyl-1*H*-pyrazol-4-yl)methyleneamino)thiazol-4yl)-2*H*-chromen-2-one (7l)
Yellow solid; mp. 107-109 ^{o}C; IR (KBr, cm^{-1}) υ_{max}: 1742 (C=O), 1624 (C=N), 1563 (C=C), 743 (C-Cl); ^{1}H NMR (300MHz, DMSO-d_6): δ 6.98-7.89 (m, 13H), 7.95 (s, 1H), 8.01 (s, 1H), 8.14 (s, 1H), 8.34 (s, 1H); MS (ESI), 70 eV, *m/z*: 509 (M+H); Anal. Calcd. for $C_{28}H_{17}ClN_4O_2S$: C, 66.07; H, 3.37; N, 11.01; Found: C, 66.15; H, 3.48; N, 11.20.

RESULTS AND DISCUSSION

1,3-Disubstituted-1*H*-pyrazole-4-carbaldehydes (**4a,b)** were prepared according to the literature procedure [35] **(Scheme-1)** by the double formylation of 2-aryl-1-(1-phenylethylidene)hydrazine (**3a,b)** with Vilsmayer-Hack reagent. The title compounds, thiazolyl pyrazole derivatives were synthesized *via* one-pot three component condensation of phenacyl bromides (**5a-e)** / 3-(2-bromoacetyl)-2*H*-chromen-2-one **(5f)** with thiourea (**6)** and 1,3-diaryl-1*H*-pyrazole-4-carbaldehydes (**4a,b)** under refluxing conditions in ethanol using catalytic amount of acetic acid **(Scheme-2)**.

To optimize the reaction conditions, initially the reaction of 4-chlorophenacyl bromide **(5a)** with thiourea **(6)** and 1,3-diphenyl-1*H*-pyrazole-4-carbaldehyde **(4a)** was carried out in different solvents like methanol, ethanol and acetonitrile at refluxing temperature, and observed the maximum yield (67%) of the product **7a** in ethanol. To improve the yield of the product **7a**, we tried the same the reaction using catalytic amount of acetic acid, and observed an improvement in the yield of the product (maximum yield: ethanol + catalytic amount of acetic acid, 78%). We also observed there was no change in the yield on the addition of excess acetic acid **(Table-1)**.

We under took the synthesis of thiazolyl pyrazole in ethanol and catalytic amount of acetic acid. The results were postulated in **Table-2**. All the synthesized compounds were confirmed by their analytical and spectroscopic data. The absence of N-H and C=O stretching frequencies in the range of 3250-3400 cm^{-1} and 1650-1750 cm^{-1} respectively from IR, aldehyde proton in the range of 11-13 ppm from ^{1}H NMR; Presence of C=N stretching frequency in the range of 1580-1635 cm^{-1} from IR, three singlets in the range 7.80-9.00 ppm (thiazole, pyrazole and imine CH protons) form the ^{1}H NMR and molecular ion peak from the mass spectra confirms the formation of the desired compound.

Table-1: Effect of solvents on yield of the product 7a[a].

Entry	Solvent	Time (h)	Yield[b] (%)
1	Methanol	12	63
2	Ethanol	12	67
3	Acetonitrile	12	60
4	Methanol + Catalytic Acetic acid	8	72
5	Ethanol + Catalytic Acetic acid	8	78
6	Acetonitrile + Catalytic Acetic acid	8	67

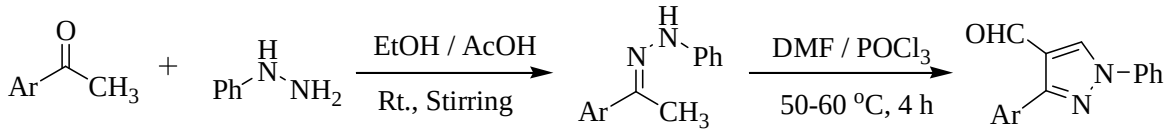
[a]***Reaction conditions:*** *4-chlorophenacyl bromide (1 mmol), thiourea (1 mmol) and 1,3-diphenyl-1H-pyrazole-4-carbaldehyde (1 mmol), Reflux.*
[b] *Yields refers to pure isolated product **7a**.*

Table-2: Synthesis of thiazolopyrazole derivatives.

Entry	R	Ar	Product[a]	Time (h)	Yield[b] (%)
1	4-ClC_6H_4	C_6H_5	7a	8	78
2	4-BrC_6H_4	C_6H_5	7b	8	78
3	4-$NO_2C_6H_4$	C_6H_5	7c	6	80
4	4-$CH_3C_6H_4$	C_6H_5	7d	8	75
5	4-$(C_6H_4)C_6H_4$	C_6H_5	7e	7	79
6	Coumarinyl	C_6H_5	7f	8	82
7	4-ClC_6H_4	4-ClC_6H_4	7g	6	76
8	4-BrC_6H_4	4-ClC_6H_4	7h	6	72
9	4-$NO_2C_6H_4$	4-ClC_6H_4	7i	7	72
10	4-$CH_3C_6H_4$	4-ClC_6H_4	7j	8	76
11	4-$(C_6H_4)C_6H_4$	4-ClC_6H_4	7k	8	81
12	Coumarinyl	4-ClC_6H_4	7l	7	78

[a]**Reaction conditions:** Phenacyl bromide/3-(2-bromoacetyl)-2*H*-chromen-2-one (1 mmol), thiourea (1 mmol) and 1,3-disubstituted-1*H*-pyrazole-4-carbaldehyde (1 mmol), ethanol + cat. acetic acid, reflux.

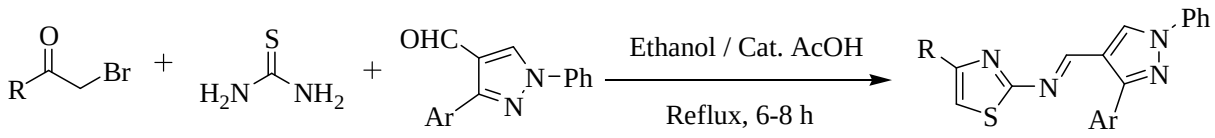
[b] Yields refers to isolated products.



1 (a,b) **2** **3 (a,b)** **4 (a,b)**

4a: Ar = C_6H_5; **4b:** Ar = 4-ClC_6H_4

Scheme-1: Synthesis of 1,3-diaryl-1*H*-pyrazole-4-carbaldehydes



5 (a-f) **6** **4 (a,b)** **7 (a-l)**

7a: Ar = C_6H_5; R = 4-ClC_6H_4
7b: Ar = C_6H_5; R = 4-BrC_6H_4
7c: Ar = C_6H_5; R = 4-$NO_2C_6H_4$
7d: Ar = C_6H_5; R = 4-$CH_3C_6H_4$
7e: Ar = C_6H_5; R = 4-$(C_6H_4)C_6H_4$
7f: Ar = C_6H_5; R = Coumarinyl
7g: Ar = 4-ClC_6H_4; R = 4-ClC_6H_4
7h: Ar = 4-ClC_6H_4; R = 4-BrC_6H_4
7i: Ar = 4-ClC_6H_4; R = 4-$NO_2C_6H_4$
7j: Ar = 4-ClC_6H_4; R = 4-$CH_3C_6H_4$
7k: Ar = 4-ClC_6H_4; R = 4-$(C_6H_4)C_6H_4$
7l: Ar = 4-ClC_6H_4; R = Coumarinyl

Scheme-2: Synthesis of thiazolopyrazole derivatives

CONCLUSION

In conclusion, we have synthesized a series of 4-substituted-*N*-((1,3-diaryl-1*H*-pyrazol-4-yl)methylene)thiazol-2-amines *via* one-pot three component condensation of phenacyl bromide / 3-(2-bromoacetyl)-2*H*-chromen-2-one, thiourea and 1,3-diaryl-1*H*-pyrazole-4-carbaldehydes in ethanol utilizing catalytic amount of acetic acid. The present work is high yielding in shorter reaction times and we believe that this method is superior to the existed methods.

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