An efficient reactions for synthesis of functionalized pyrazoles

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Research Article

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Abstract

The functionalized pyrazole derivatives produced from the reactions of hydrazonoyl chloride with ethyl 3-oxo-3-phenylpropanoate, acetylacetone, ethyl cyanoacetate, methyl cyanoacetate, or malononitrile in the presence of inexpensive sodium methoxide as base in DMF under catalyst-free condition in good yields.

Introduction

Biologically active nitrogen-containing heterocyclic compounds are ubiquitous in natural products and have found broad applications in functional materials and pharmaceuticals [1–3]. Among various *N*-heterocycles, the pyrazole nucleus, particularly with various substitutions, confers a wide range of applications across technology, medicine, and agriculture [4–8]. Pyrazole derivatives have exhibited various biological activities in humans, including antiviral [9], antitumour [10, 11], antibacterial [12, 13], anti-amoebic [14], anti-inflammatory [15], and selective enzyme inhibition [16, 17]. This heterocycle is also found in numerous well-established drugs across different therapeutic categories [18]. Additionally, certain pyrazoles are employed in supramolecular and polymer chemistry, the food industry, and as cosmetic colorant [19].

Over the years, several methods have been reported for the synthesis of pyrazole derivatives. The most widely used strategies include: (i) condensation reaction between 1,3-dipolar compounds and various hydrazines [20–22], (ii) 1,3-dipolar cycloaddition reactions of diazo compound with olefins and alkynes [23–27], and (iii) more recent approach approaches involving the catalytic activity of CuO/ZrO_2 [28], ionic liquid [29], DABCO [30], and $NiFe_2O_4$ nanoparticle [31], in combination with suitably pre-functionalized substrates. Despite these advances, major challenges associated with these routes include harsh reaction condition, limited substrate scope and the requirement of pre-functionalized starting materials. Therefore, the development of efficient synthetic strategies under mild condition and with broad substrate compatibility remains highly desirable. The synthesis of substituted pyrazoles with structural diversity continues to be of great interest in modern synthetic organic chemistry, as functionalization of the pyrazole nucleus at different positions has yielded multifunctional pharmacologically active molecules in drug discovery.

Inspired by the significant role of pyrazole ring systems in medicinal chemistry and our ongoing interest in bioactive heterocyclic compounds, we became interested in synthesizing *N*-aryl pyrazoles via a simple and efficient reaction of hydrazonoyl chlorides using inexpensive sodium methoxide as a base in DMF under catalyst-free conditions.

Result And Discussion

We began our studies using the hydrazonoyl chloride derivatives **2** as our substrate as there in precedent for preparation of functionalized *N*-phenyl pyrazoles. Treatment this compounds with ethyl 3-oxo-3-phenylpropanoate or Acetylacetone **1** in presence of sodium methoxide in DMF as solvent at 50°C

temperature for 5 h afforded target productes in high yields of 78–89% (scheme 1). All the compounds are stable solids whose structure is fully supported by IR, elemental analyses, mass, and high-field ^{1}H NMR and ^{13}C NMR spectroscopy spectrometry data. The IR spectrum of new compound **3a** clearly shows resonance characteristic of ester carbonyl group at 1706 cm^{-1}. the ^{1}H NMR spectrum of **3a** exhibited ethoxy group at 4.10 as a quartet (^{3}J = 7.0 Hz) and methyl group at 2.43 as singlet. The ^{13}C resonance signals of the aromatic moiety were observed at 112.1-153.3 whereas the carbonyl group were seen at δ 163.8 ppm.

During the studies on exploring the scope of the reactions of hydrazonoyl chloride by using nucleophiles, we treated the reaction of ethyl cyanoacetate, methyl cyanoacetate, or malononitrile **4** with hydrazonoyl chloride **2** in polar aprotic solvent of DMF at 50°C temperature in the presence of MeONa to provide the 5-amino-1-phenyl-3aryl-1H-pyrazole-4-carbonitrile **5a**–**f** in 78–88% under catalyst-free condition (Scheme 2).

In an extension of our previous studies [32], At first, we have further investigated the reaction of sodium arylsulfinates with hydrazonoyl chloride in the presence triethylamine at room temperature. This reaction led to the synthesis of 1-((4-chlorophenyl)(tosyl)methylene)-2-phenylhydrazine **7** in high yield (Scheme 3). Then, a solution of hydrazonoyl chloride **2** and acetylenic esters **7** in dimethylformamide (DMF) were reacted with a stioichiometric amount of sodium arylsulfinates **6** at r.t for 12 h. Purification of the reaction mixture by column chromathography afforded the functionalized amino pyrazoles **9**. Product structures and isolated yields are summarized in Scheme 4.

To explain the mechanism for the formation of the product **3**, we propose a plausible reaction course, which is illustrated in scheme 5. The first step is the formation of the two intermediates of **10** and **11** from hydrazonoyl chloride **2** and ethyl 3-oxobutanoate or ethyl 3-oxo-3-phenylpropanoate **1** in the presence triethylamine and sodium methoxide as base. Nucleophilic addition of the intermediate **11** to positively charged carbon of intermediate **10**, which is converted to **13** by cyclization. Eventually, the elimination of hydroxide completes the reaction sequence leading to the final products **3**.

A mechanistic rationalization of the product **9** is provided in scheme 6. Presumably, the intermediate **14** formed from sodium arylsulfinate **6** and dialkyl acetylenedicarboxylate **8**, which undergoes further reaction with hydrazonoyl chloride **2** to generate **15**. This intermediate is converted into **16** via cyclization, which then undergoes elimination of sodium arylsulfinates to provided the final product **9**.

In conclution, we have devised novel reactions for the synthesis of a series of substituted pyrazole derivatives under free-catalyst conditions. The presence of transformable functionalities in these products makes them potentially valuable from the vantage point of further synthetic manipulations. It is noteworthy that pyrazole derivatives are often found useful as pharmaceuticals and agrochemicals.

Experimental

General

Electrothermal-9100 apparatus was used for melting points. The data of IR, NMR, Elemental analyses, and Mass were recorded by Shimadzu-IR-460 spectrometer, Bruker DRX-400, Vario EL III CHNOS, and Finnigan-MAT-8430EI-MS respectively.

General procedure for preparation of Substituted N-phenyl pyrazoles (3a-f)

To a magnetically stirred solution of ethyl 3-oxo-3-phenylpropanoate or Acetylacetone **1** (1 mmol) in DMF (4 ml) was added sodium methoxide (1 mmol) and triethylamine (1 mmol) at room temperature. The reaction mixture stirred for 10 min, followed by the addition of the hydrazonoyl chloride **2** (1 mmol) and the reaction stirred for 5 h at 50°C. The mixture was poured onto H_2O (20 ml), extracted with CH_2Cl_2 (20 ml), dried ($MgSO_4$), and the solvent was removed under reduced pressure. The residue was subjected to silica gel (0.063-0.200 size) column chromatography using hexane-ethyl acetate as eluent.

ethyl 1,5-diphenyl-3-(p-tolyl)-1H-pyrazole-4-carboxylate (3a). White powder, mp: 103–105ºC, yield: 0.32 g (85%). IR (KBr): 2953, 1706, 1612, 1594, 1493, 1177, 1145, 1072, 760, 659. ^{1}H NMR (400.1 MHz, $CDCl_3$): δ_H = 1.01 (t, J = 7.0 Hz, 3H); 2.43 (s, 3H); 4.10 (q, J = 7.0 Hz, 2H); 7.28–7.29 (m, 7H); 7.33–7.40 (m, 5H); 7.70 (d, J = 8.2 Hz, 2H). ^{13}C NMR (100.6 MHz, $CDCl_3$): δ_C = 13.7, 21.4, 60.2, 112.1, 125.4, 127.8, 128.0, 128.6, 128.8, 128.9, 129.0, 129.7, 129.75, 130.4, 138.2, 139.2, 146.2, 153.3, 163.8. MS: *m/z* (%) = 382 (M+, 100), 337 (80), 180 (14), 77 (19). Anal. calc. for $C_{25}H_{22}N_2O_2$ (382.46): C 78.51, H 5.80, N 7.32%; found: C 78.63, H 5.89, N 7.28%.

1-(3-(4-chlorophenyl)-5-methyl-1-phenyl-1H-pyrazol-4-yl)ethan-1-one (**3b**). White powder, mp: 132°C, yield: 0.26 g (84%). IR (KBr): 2957, 1721, 1653, 1594, 1515, 1452, 1375, 1267, 1194, 1085, 756, 690, 524. ^{1}H NMR (400.1 MHz, $CDCl_3$): δ_H = 2.15 (s, 3H); 2.54 (s, 3H); 7.41–7.53 (m, 9H). ^{13}C NMR (100.6 MHz, $CDCl_3$): δ_C = 12.9, 31.0, 120.6, 125.8, 128.7, 128.9, 129.4, 130.8, 132.2, 134.9, 138.5, 143.9, 152.1, 195.5. MS: *m/z* (%) = 310 (M^+, 46), 295 (100), 77 (23). Anal. calc. for $C_{18}H_{15}ClN_2O$ (310.78): C 69.57, H 4.87, N 9.01%; found: C 69.62, H 4.83, N 9.07%.

1-(5-methyl-1-phenyl-3-(p-tolyl)-1H-pyrazol-4-yl)ethan-1-one (3c). White powder, mp: 128ºC, yield: 0.23 g (80%). IR (KBr): 2837, 1705, 1687, 1569, 1532, 1467, 1412, 1367, 1294, 1121, 984, 783, 564. ^{1}H NMR (400.1 MHz, $CDCl_3$): δ_H = 2.26 (s, 3H); 2.41 (s, 3H); 2.57 (s, 3H); 7.26–7.28 (m, 2H); 7.46–7.61 (m, 5H); 7.90 (d, J = 8.1 Hz, 2H). ^{13}C NMR (100.6 MHz, $CDCl_3$): δ_C = 12.4, 21.4, 30.5, 120.8, 125.7, 128.4, 128.7, 128.77, 129.2, 129.4, 133.6, 138.5, 143.8, 153.8, 196.3. MS: *m/z* (%) = 290 (M+, 100), 180 (18), 77 (35). Anal. calc. for $C_{19}H_{18}N_2O$ (290.36): C 78.59, H 6.25, N 9.65%; found: C 78.84, H 6.17, N 9.58%.

ethyl 3-(4-chlorophenyl)-1,5-diphenyl-1H-pyrazole-4-carboxylate (**3d**). White powder, mp: 192°C, yield: 0.31 g (78%). IR (KBr): 2914, 1712, 1627, 1493, 1449, 1430, 1306, 1142, 1087, 1011, 835, 756, 656. ^{1}H NMR (400.1 MHz, $CDCl_3$): δ_H = 1.01 (t, *J* = 7.1 Hz, 3H); 4.65 (q, *J* = 7.1 Hz, 2H); 7.39–7.33 (m, 5H); 7.35–

7.37 (m, 5H); 7.43 (d, J = 8.4 Hz, 2H), 7.77 (d, J = 8.4 Hz, 2H). ^{13}C NMR (100.6 MHz, $CDCl_3$): δ_C = 13.7, 60.3, 112.0, 119.9, 125.4, 128.0, 128.1, 128.2, 128.8, 129.1, 130.4, 130.6, 131.2, 134.4, 139.0, 146.6, 152.2, 163.5. MS: *m/z* (%) = 402 (M^+, 100), 357 (92), 180 (14), 77 (28). Anal. calc. for $C_{24}H_{19}ClN_2O_2$ (402.87): C 71.55, H 4.75, N 6.95%; found: C 71.62, H 4.70, N 6.89%.

1-(5-methyl-1,3-diphenyl-1H-pyrazol-4-yl)ethan-1-one (**3e**).White powder, yield: 0.22 g (82%). ^{1}H NMR (400.1 MHz, $CDCl_3$): δ_H = 2.13 (s, 3H); 2.56 (s, 3H); 7.46–7.43 (m, 4H); 7.52–7.48 (m, 4H); 7.56–7.54 (m, 2H). ^{13}C NMR (100.6 MHz, $CDCl_3$): δ_C = 12.8, 30.8, 120.5, 125.7, 127.7, 128.4, 128.7, 129.2, 129.4, 133.7, 138.5, 143.7, 153.3, 195.9.

ethyl 1,3,5-triphenyl-1H-pyrazole-4-carboxylate (**3f**). White powder, yield: 0.25 g (86%). ^{1}H NMR (400.1 MHz, $CDCl_3$): δ_H = 0.90 (t, J = 7.0 Hz, 3H); 4.00 (J = 7.0 Hz, 2H); 7.30–7.20 (m, 10H); 7.35–7.38 (m, 3H); 7.70 (m, d, J = 7.5 Hz, 2H). ^{13}C NMR (100.6 MHz, $CDCl_3$): δ_C = 13.7, 60.3, 112.2, 125.4, 127.8, 127.9, 128.1, 128.4, 128.8, 129.0, 129.2, 129.6, 130.4, 132.7, 139.2, 146.3, 153.3, 163.8.

General procedure for preparation of Substituted 5-amino-1-phenyl-3aryl-1H-pyrazole-4-carbonitrile (5a-f)

To a magnetically stirred solution of ethyl cyanoacetate, methyl cyanoacetate, or malononitrile **4** (1 mmol) in DMF (4 ml) was added sodium methoxide (1 mmol) and triethylamine (1 mmol) at room temperature. The reaction mixture stirred for 10 min, followed by the addition of the hydrazonoyl chloride **2** (1 mmol) and the reaction stirred for 5 h at 50°C. The mixture was poured onto H_2O (20 ml), extracted with CH_2Cl_2 (20 ml), dried ($MgSO_4$), and the solvent was removed under reduced pressure. The residue was subjected to silica gel (0.063-0.200 size) column chromatography using hexane-ethyl acetate as eluent.

5-amino-3-(4-chlorophenyl)-1-phenyl-1H-pyrazole-4-carbonitrile (**5a**). White powder, yield: 0.24 g (84%). ^{1}H NMR (400.1 MHz, $CDCl_3$): δ_H = 4.75 (s, 2H); 7.40 (d, J = 8.0 Hz, 2H); 7.49–7.50 (m, 1H); 7.57–7.58 (m, 4H); 7.93 (d, J = 8.0 Hz, 2H). ^{13}C NMR (100.6 MHz, $CDCl_3$): δ_C = 73.9, 115.1, 124.4, 127.8, 129.1, 129.2, 129.5, 130.1, 135.3, 136.9, 150.3, 151.5.

5-amino-1,3-diphenyl-1H-pyrazole-4-carbonitrile (**5b**). White powder, yield: 0.32 g (78%). ^{1}H NMR (400.1 MHz, $CDCl_3$): δ_H = 4.71 (s, 2H); 7.43–7.49 (m, 4H); 7.55–7.61 (m, 4H); 7.99 (d, J = 6.6 Hz, 2H). ^{13}C NMR (100.6 MHz, $CDCl_3$): δ_C = 74.0, 115.2, 124.3, 126.4, 128.7, 128.9, 129.3, 130.0, 130.9, 137.0, 151.3, 151.4.

5-amino-1-phenyl-3-(p-tolyl)-1H-pyrazole-4-carbonitrile (**5c**). Pink powder, yield: 0.24 g (88%). ^{1}H NMR (400.1 MHz, $CDCl_3$): δ_H = 2.41 (s, 3H); 4.65 (s, 2H); 7.26–7.28 (m, 2H); 7.46–7.49 (m, 1H); 7.54–7.61 (m, 4H), 7.90 (d, J = 8.1 Hz, 2H). ^{13}C NMR (100.6 MHz, $CDCl_3$): δ_C = 21.4, 115.3, 124.3, 126.3, 128.1, 128.8, 129.4, 130.0, 137.0, 139.4, 141.5, 151.2, 151.5.

methyl 5-amino-3-(4-chlorophenyl)-1-phenyl-1H-pyrazole-4-carboxylate (**5d**). Red powder, mp: 109–111°C, yield: 0.28 g (88%). IR (KBr): 3433, 3331, 2917, 1668, 1592,1519, 1340, 1277, 1138, 756, 696. ^{1}H NMR (400.1 MHz, $CDCl_3$): δ_H = 3.77 (s, 3H); 5.53 (s, 2H); 7.38–7.46 (m, 3H); 7.52–7.67 (m, 6H). ^{13}C NMR (100.6 MHz, $CDCl_3$): δ_C = 50.9, 93.4, 124.2, 127.9, 128.8, 129.8, 130.7, 131.6, 134.3, 137.2, 150.7, 151.7, 165.0. MS: *m/z* (%) = 327 (M^+, 58), 295 (100), 91 (36), 77 (22). Anal. calc. for $C_{17}H_{14}ClN_3O_2$ (327.76): C 62.30, H 4.31, N 12.82%; found: C 62.46, H 4.27, N 12.73%.

methyl 5-amino-1,3-diphenyl-1H-pyrazole-4-carboxylate (**5e**). Red powder, mp: 153°C, yield: 0.24 g (85%). IR (KBr): 3430, 3327, 2914, 1687, 1568,1509, 1323, 1297, 1141, 724. ^{1}H NMR (400.1 MHz, $CDCl_3$): δ_H = 3.01 (s, 3H); 4.43 (s, 2H); 7.43–7.49 (m, 4H); 7.51–7.61 (m, 4H); 7.90 (d, *J* = 6.6 Hz, 2H). ^{13}C NMR (100.6 MHz, $CDCl_3$): δ_C = 50.0, 93.9, 124.3, 126.4, 128.7, 128.9, 129.3, 130.0, 130.9, 137.0, 151.3, 151.4, 165.4. EI-MS: 293 (M^+, 61), 295 (100), 91 (43), 77 (28). Anal. calc. for $C_{17}H_{15}N_3O_2$ (293.32): C 69.61, H 5.15, N 14.33%; found: C 69.31, H 5.10, N 14.37%.

ethyl 5-amino-3-(4-chlorophenyl)-1-phenyl-1H-pyrazole-4-carboxylate (**5f**). Red powder, mp: 163°C, yield: 0.26 g (79%). IR (KBr): IR (KBr): 3435, 3314, 2908, 1697, 1574,1523, 1345, 1238, 1085, 761. ^{1}H NMR (400.1 MHz, $CDCl_3$): δ_H = 1.01 (t, *J* = 7.0 Hz, 3H); 4.12 (q, *J* = 7.0 Hz, 2H); 5.53 (s, 2H); 7.34–7.79 (m, 9H). ^{13}C NMR (100.6 MHz, $CDCl_3$): δ_C = 13.72, 60.33, 93.41, 124.7, 127.9, 128.8, 129.8, 130.7, 131.6, 134.3, 137.2, 150.7, 151.3, 165.7. EI-MS: 341 (M^+, 68), 295 (100), 91 (35), 77 (25). Anal. calc. for $C_{18}H_{16}ClN_3O_2$ (341.79): C 63.25, H 4.72, N 12.29%; found: C 63.32, H 4.79, N 12.23%.

General procedure for preparation of 1-((4-chlorophenyl)(tosyl)methylene)-2-phenylhydrazine (7).

To a magnetically stirred solution of sodium 4-methylbenzenesulfinate **6** (1 mmol) and 4-chloro-*N*-phenylbenzohydrazonoyl chloride **2** in DMF (4 ml) was added triethylamine (1 mmol) and the reaction mixture stirred for 8 h at room temperature. The mixture was poured onto H_2O (20 ml), extracted with CH_2Cl_2 (20 ml), dried ($MgSO_4$), and the solvent was removed under reduced pressure. The residue was subjected to silica gel (0.063-0.200 size) column chromatography using hexane-ethyl acetate as eluent.

1-((4-chlorophenyl)(tosyl)methylene)-2-phenylhydrazine (**7**). White powder, mp: 124–126°C, yield: 0.31 g (82%). IR (KBr): 2932, 1541, 1356, 1267, 1128, 1079, 743, 653. ^{1}H NMR (400.1 MHz, $CDCl_3$): δ_H = 2.47 (s, 3H); 6.95–6.97 (m, 3H); 7.23–7.35 (m, 6H); 7.51 (d, *J* = 7.0 Hz, 2H), 7.78 (d, *J* = 8.2 Hz, 2H), 7.82 (s, 1H). ^{13}C NMR (100.6 MHz, $CDCl_3$): δ_C = 21.8, 114.0, 122.7, 124.6, 129.0, 129.4, 129.7, 130.2, 131.4, 136.0, 137.4, 141.6, 142.2, 144.6. EI-MS: 384 (M^+, 100), 91 (46), 77 (38). Anal. calc. for $C_{20}H_{17}ClN_2O_2S$ (384.87): C 62.41, H 4.45, N 7.28, S 8.33%; found: C 62.32, H 4.46, N 7.23, S 8.27%.

General procedure for preparation of substituted 3-Aryl-1-phenyl-1H-pyrazoles (9a-e)

A solution of hydrazonoyl chloride **2** and sodium arylsulfinates **6** in DMF (3 ml) was cooled to 0–5°C. To this, a solution of dialkyl acetylenedicaboxylate **8** (1 mmol) in DMF (2 ml) was added and the reaction

mixture was stirred for 12 h at r.t. The mixture was poured onto H_2O (20 ml), extracted with CH_2Cl_2 (20 ml), dried ($MgSO_4$), and the solvent was removed under reduced pressure. The resulting residue was purified by silica gel column chromatography and recrystallized from diehyl ether to afford crystalline product.

Compounds 3a-d, 5d-f, and 7 are new compounds, while derivatives 9a-e [32], 5a-c [33], 3e-f [34, 35], have been reported in the literature.

Declarations

Acknowledgements

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Supplementary Material

Supporting Information: Full experimental detail, 1H and 13C NMR spectra. This material can be found via the "Supplementary Contenet" section of this article's webpage.

Notes

The authors declare on conflict of interest in connection with the manuscript.

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Schemes

Schemes 1 to 6 are available in the Supplementary Files section

Supplementary Files

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