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Synthesis, Structures, and Biological Activities of New 1*H*-1,2,4-Triazole Derivatives Containing Pyridine Unit

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ABSTRACT: *Fourteen new 1*H-*1,2,4-triazole derivatives containing pyridine moiety were synthesized by condensation of 1-(pyridine-3-yl)-2-(1*H-*1,2,4-triazol-1-yl)ethanone with aryl aldehydes, and their reaction conditions were studied. The title compounds were screened for their antibacterial and plant growth regulatory activities. The screening data revealed that most of the compounds showed some antifungal and plant growth regulatory activities.* © 2007 Wiley Periodicals, Inc. Heteroatom Chem 18:376–380, 2007; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.20308

INTRODUCTION

The considerable biological importance of heterocyclic compounds has stimulated much work on them [1–4]. Traditionally, small heterocyclic molecules have been a reliable source for discovering novel biologically active molecules. Compared with the structurally complex natural compounds, these molecules are easily synthesized and their structural optimization usually leads to a feasible candidate compound. Of all the heterocyclic compounds, 1*H*-1,2,4-triazole derivatives due to their broad spectrum of biological activities, high activity, and high selectivity had attracted the attention of many chemists [5–9], and their various commercial compounds had been widely used in plant protection and medicines as fungicides triadimefon, triadimenol, flusilazole, bitertanol, cyproconazole, etc. and as clinical drugs fluconazole and itraconazole [10,11].

It is well known that pyridine unit is another important heterocyclic nucleus with regard to biological activity [12]. Incorporating pyridine ring into active compounds may usually improve their biological or physiological activities [13]. Many pyridine derivatives, such as pioglitazone, rosiglitazone, zolpidem, frowncide, etc. [14–16], have played an important role in agrochemistry and medical chemistry.

In our laboratory, various triazole derivatives have been synthesized and were found to be effective in fungicidal and plant growth regulatory activities [17,18]. To optimize them, we designed and synthesized 14 new *N*-heterocyclic compounds by incorporating pyridine ring into triazole compounds (Scheme 1), and evaluated their fungicidal and plant growth regulatory activities.

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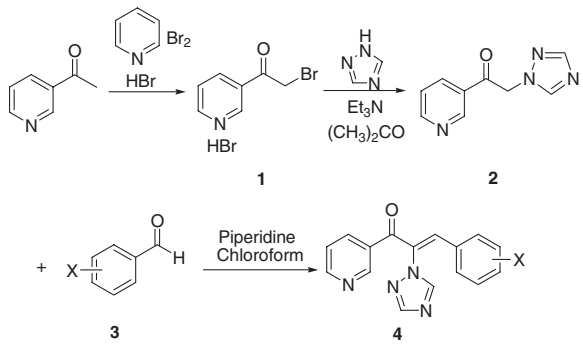
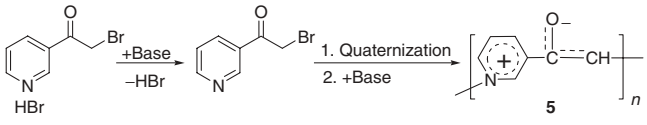


SCHEME 1



SCHEME 2

RESULTS AND DISCUSSION

*Synthesis of 1-(Pyridin-3-yl)-2-(1*H*-1,2,4-triazol-1-yl)ethanone (**2**)*

Condensation of 1*H*-1,2,4-triazole with compound **1** was performed in acetone using triethylamine (TEA) as a base, and the yield can be improved by up to 10% by refluxing a mixture of 1*H*-1,2,4-triazole, TEA, and acetone for 30 minutes before the addition of intermediate **1**. The yield of this reaction is lower than the similar reaction of 1*H*-1,2,4-triazole with 2-bromo-1-arylethanone [19]; the reason may be due to the conformation of aggregate **5** [20] (Scheme 2).

*Synthesis of 3-Aryl-1-(pyridin-3-yl)-2-(1*H*-1,2,4-triazol-1-yl)prop-2-en-1-one*

The reaction conditions for aldol condensation have been well documented in literature, but the condensation of substituted aryl aldehyde with the intermediate **2** could not provide satisfactory results using these reported conditions. Traditionally, toluene, benzene, or cyclohexane was used as solvent and piperidine or piperidine and acetic acid as catalysts in the aldol condensation reaction. When we synthesized the title compounds **4a** (X = H), **4h** (X = 2-F-4-Br), and **4n** (X = 2-Cl) using the above conditions, the yield of **4a** is only 26.4%. Moreover, the yields of **4h** and **4n** were too low to be separated from the final products. Acetic anhydride both as a solvent and a water acceptor and anhydrous potassium carbonate as a catalyst were also investigated, but the yield was still lower than 20%. Among the other solvents investigated such as ethanol, acetonitrile, and chloroform, chloroform was found to be the best solvent in synthesizing the title compounds **4a–n**, and by using piperidine as catalyst, their yield was more than 30% (Table 1).

The Structures of Title Compounds

All title compounds were colorless solids and their structures were confirmed by ^{1}H NMR (Table 2) and elemental analyses (Table 1). The structure of compound **4g** was also confirmed by single crystal X-ray diffraction analysis [21] (Fig. 1). In the crystal cell, due to the bulkiness of pyridine ring, the substituted aryl group is spatially repulsed and swerves to the nearby triazole group.

TABLE 1 Physical Properties and Elemental Analysis Data for Compounds **4a–n**

				Elementary Analysis (Calcd, %)		
	X	*Yield* (%)	*mp (°C)*	*C*	*H*	*N*
4a	H	47.1	149–150	69.67 (69.55)	4.49 (4.38)	20.64 (20.28)
4b	4-Cl	32.2	119–121	61.76 (61.84)	3.69 (3.57)	17.96 (18.03)
4c	2-F	43.2	93–95	65.12 (65.30)	3.60 (3.77)	18.94 (19.04)
4d	3-F	32.3	120–122	65.28 (65.30)	3.78 (3.77)	18.95 (19.04)
4e	2-OMe	50.1	144–146	66.58 (66.66)	4.58 (4.61)	18.04 (18.29)
4f	2,4-Cl_2	47.4	156–158	55.74 (55.67)	2.86 (2.92)	16.33 (16.23)
4g	2,6-Cl_2	50.0	135–137	55.58 (55.67)	2.76 (2.92)	16.16 (16.23)
4h	2-F-4-Br	43.8	142–144	51.53 (51.50)	2.87 (2.70)	14.94 (15.01)
4i	2,4-Me_2	33.9	83–85	71.19 (71.04)	5.24 (5.30)	18.40 (18.41)
4j	3,4-Me_2	52.0	124–126	71.12 (71.04)	5.32 (5.30)	18.54 (18.41)
4k	2,4-$(OMe)_2$	30.8	121–123	64.25 (64.28)	4.82 (4.79)	16.66 (16.66)
4l	3,4-$(OMe)_2$	45.0	153–155	64.24 (64.28)	4.76 (4.79)	16.73 (16.66)
4m	3,4-(—OCH_2O—)	48.1	134–136	63.60 (63.75)	3.83 (3.78)	17.49 (17.49)
4n	2-Cl	54.8	101–103	62.07 (61.84)	3.78 (3.57)	17.77 (18.03)

TABLE 2 ^{1}H NMR Spectral Data for Compounds **4a–n**

	X	*^{1}H NMR spectral data (δ)*
4a	H	9.05 (s, 1H, PyH), 8.85 (d, *J* = 3.3 Hz, 1H, PyH), 8.17 (s, 1H, TrH), 8.23 (s, 1H, TrH), 8.10 (d, *J* = 7.8 Hz, 1H, PyH), 7.64 (s, 1H, CH═), 7.45–7.49 (m, 1H, PyH), 7.33–7.45 (m, 3H, phH), 6.72 (d, *J* = 7.2 Hz, 2H, phH)
4b	4-Cl	9.02 (s, 1H, PyH), 8.84 (d, *J* = 3.6 Hz, 1H, PyH), 8.17 (s, 1H, TrH), 8.22 (s, 1H, TrH), 8.08 (d, *J* = 7.8 Hz, 1H, PyH), 7.58 (s, 1H, CH═), 7.47 (d d, *J* = 4.8 Hz, PyH), 7.30 (d, *J* = 8.4 Hz, 2H, phH), 6.88 (d, *J* = 8.7 Hz, 2H, phH)
4c	2-F	9.05 (d, *J* = 1.5 Hz, 1H, PyH), 8.84 (d d, *J* = 1.8 Hz, 1H, PyH), 8.17 (s, 1H, TrH), 8.20 (s, 1H, TrH), 8.09–8.12 (m, 1H, PyH), 7.78 (s, 1H, CH═), 7.45–7.50 (m, 1H, PyH), 7.40–7.46 (m, 1H, phH), 7.00–7.15 (m, 2H, phH), 6.58–6.64 (m, 1H, phH)
4d	3-F	9.04 (s, 1H, PyH), 8.85 (d, *J* = 3.6 Hz, 1H, PyH), 8.18 (s, 1H, TrH), 8.23 (s, 1H, TrH), 8.09 (d, *J* = 7.8 Hz, 1H, PyH), 7.59 (s, 1H, CH═), 7.10–7.40 (m, 2H, PyH), 6.78 (d, *J* = 7.5 Hz, 1H, phH), 6.65 (d, *J* = 9.6 Hz, 1H, phH)
4e	2-OMe	9.03 (d, *J* = 2.1 Hz, 1H, PyH), 8.83 (d d, *J* = 1.6 Hz, 1H, PyH), 8.14 (s, 1H, TrH), 8.15 (s, 1H, TrH), 8.10–8.12 (m, 1H, PyH), 7.98 (s, 1H, CH═), 7.44–7.49 (m, 1H, PyH), 7.40–7.41 (m, 1H, phH), 6.91 (d, *J* = 8.4 Hz, 1H, phH), 6.79 (t, *J* = 7.8 Hz, phH), 6.55 (d d, *J* = 1.5 Hz, phH), 3.81 (s, 3H, OCH_3)
4f	2,4-Cl_2	9.06 (d, *J* = 1.5 Hz, 1H, PyH), 8.83 (d, *J* = 1.2 Hz, 1H, PyH), 8.11(s, 1H, TrH), 8.20 (s, 1H, TrH), 8.14 (t, *J* = 1.5 Hz, 1H, PyH), 7.76 (s, 1H, CH═), 7.46–7.50 (m, 2H, PyH, phH), 7.12 (d d, *J* = 2.1 Hz, phH), 6.63 (d, *J* = 8.7 Hz, 1H, phH)
4g	2,6-Cl_2	9.20 (d, *J* = 1.8 Hz, 1H, PyH), 8.88 (d, *J* = 1.5 Hz, 1H, PyH), 8.28 (d, *J* = 8.1 Hz, 1H, PyH), 8.23 (s, 1H, TrH), 7.90 (s, 1H, TrH), 7.51 (d d, *J* = 4.8 Hz, 1H, PyH), 7.37 (d, *J* = 2.4 Hz, 1H, phH), 7.34(s, 1H, CH═), 7.24–7.30 (m, 2H, phH)
4h	2-F-4-Br	9.03 (s, 1H, PyH), 8.86 (d, *J* = 3.6 Hz, 1H, PyH), 8.23 (s, 1H, TrH), 8.17 (s, 1H, TrH), 8.10 (d, *J* = 3.6 Hz, 1H, PyH), 7.67 (s, 1H, CH═), 7.48 (d d, *J* = 4.8 Hz, 1H, PyH), 7.31 (t, *J* = 9.6 Hz, 1H, phH), 7.20 (d, *J* = 8.4 Hz, 1H, phH), 7.31 (t, *J* = 7.8 Hz, 1H, phH)
4i	2,4-Me_2	9.01 (s, 1H, PyH), 8.83 (d, *J* = 3.9 Hz, 1H, PyH), 8.23 (s, 1H, TrH), 8.15 (s, 1H, TrH), 8.07 (d, *J* = 7.8 Hz, 1H, PyH), 7.60 (s, 1H, CH═), 7.46 (d d, *J* = 4.8 Hz, 1H, PyH), 7.07 (d, *J* = 8.1 Hz, 1H, phH), 7.05 (s, 1H, phH), 6.85 (d, *J* = 7.8 Hz, 1H, phH), 6.49 (d, *J* = 7.8 Hz, 1H, phH), 2.30 (s, 6H, CH_3)
4j	3,4-Me_2	9.02 (s, 1H, PyH), 8.81 (d, *J* = 4.5 Hz, 1H, PyH), 8.11 (s, 1H, TrH), 8.08 (t, *J* = 1.8 Hz, 1H, PyH), 8.06 (s, 1H, TrH), 7.76 (s, 1H, CH═), 7.44 (d d, *J* = 4.8 Hz, 1H, PyH), 7.07 (s, 1H, phH), 6.85 (d, *J* = 7.8 Hz, 1H, phH), 6.61 (d, *J* = 7.8 Hz, 1H, phH), 2.25 (s, 6H, CH_3).
4k	2,4-$(OMe)_2$	9.01 (d, *J* = 1.8 Hz, 1H, PyH), 8.10 (d d, *J* = 1.5 Hz, 1H, PyH), 8.20 (s, 1H, TrH), 8.16 (s, 1H, TrH), 8.05–8.10 (m, 1H, PyH), 8.04 (s, 1H, CH═), 7.45 (d d, *J* = 4.8 Hz, 1H, PyH), 6.30–6.41(m, 3H, phH), 3.79 (s, 3H, OCH_3), 3.81 (s, 3H, OCH_3).
4l	3,4-$(OMe)_2$	9.03 (s, 1H, PyH), 8.83 (d, *J* = 3.6 Hz, 1H, PyH), 8.27 (s, 1H, TrH), 8.21 (s, 1H, TrH), 8.08 (d, *J* = 7.5 Hz, 1H, PyH), 7.63 (s, 1H, CH═), 7.46 (d d, *J* = 4.2 Hz, 1H, PyH), 6.81 (d, *J* = 8.1 Hz, 1H, phH), 6.75 (d, *J* = 8.4 Hz, 1H, phH), 6.14 (s, 1H, phH), 3.90 (s, 3H, OCH_3), 3.64 (s, 3H, OCH_3)
4m	3,4-(—OCH_2O—)	8.99 (d, *J* = 1.5 Hz, 1H, PyH), 8.82 (d, *J* = 4.2 Hz, 1H, PyH), 8.25 (s, 1H, TrH), 8.18 (s, 1H, TrH), 8.05 (d, *J* = 7.8 Hz, 1H, PyH), 7.58 (s, 1H, CH═), 7.46 (d d, *J* = 4.8 Hz, 1H, PyH), 6.78 (d, *J* = 8.1 Hz, 1H, phH), 6.71(d, *J* = 8.1 Hz, 1H, phH), 6.01 (s, 1H, phH), 2.41 (s, 2H, OCH_2O)
4n	2-Cl	9.08 (d, *J* = 1.5 Hz, 1H, PyH), 8.86 (d d, *J* = 1.8 Hz, 1H, PyH), 8.13–8.18 (m, 2H, PyH, TrH), 8.11 (s, 1H, TrH), 7.84 (s, 1H, CH═), 7.45–7.51 (m, 2H, PyH, phH), 7.31–7.36 (m, 1H, phH), 7.10–7.16 (m, 1H, phH), 6.70 (d d, *J* = 1.2 Hz, 1H, phH)

Biological Activities

The assessment of in vitro fungicidal activities for compounds **4a–n** were performed against six selected fungi including *G. zeae, A. solani, P. asparagi, P. piricola, C. rachidicola*, and *C. cucumerinum*. Their relative inhibitory ratios (%) were determined, and the results of such studies are listed in Table 3.

The screening data revealed that most of the compounds **4a–n** showed some degree of antifungal activities. But the relative inhibitory ratio was present on the low side. To the best of our knowledge, a linkage between the triazole ring and substituted aryl group via no more than two single or double bonds is essential for their fungicidal activities, and an extended carbon backbone linking the triazole ring and aryl group in an almost linear fashion possesses higher activity than a distorted one [18]. Because the triazole ring and aryl groups of the compounds **4a–n** are connected in a distorted fashion, they do not display predominant fungicidal activity.

The plant growth regulator activities of compounds **4a–n** were also tested by wheat coleoptile and cucumber cotyledon tests at a concentration

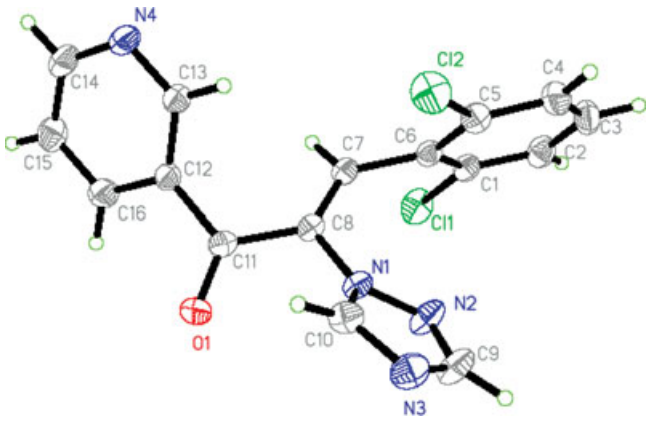


FIGURE 1 Molecular structure and crystallographic numbering for compound **4g**. Selected bond lengths (Å): Cl (1)—C (1) 1.7292 (17); Cl (2)—C (5) 1.7373 (17); O (1)—C (11) 1.212 (2); N (1)—C (10) 1.339 (2); N (1)—N (2) 1.3608 (19); N (1)—C (8) 1.418 (2); N (3)—C (10) 1.306 (2); N (4)—C (13) 1.330 (2); C (5)—C (6) 1.392 (2); C (6)—C (7) 1.475 (2); C (7)—C (8) 1.328 (2). Selected angles (°): O (1)—C (11)— C(8) 120.44 (15); O (1)—C (11)—C (12) 120.06 (15); C (12)—C (11)—C (8) 119.49 (14); C (7)—C (8)—N (1) 121.81 (15); C (7)—C (8)—C (11) 123.08 (15); N (1)—C (8)—C (11) 114.82 (13); C (6)—C (7)—C (8) 126.28 (15); C (10)—N (1)—N (2) 108.82 (14); C (10)—N (1)—C (8) 129.33 (14); N (2)—N (1)—C (8) 121.83 (13); C (10)—N (3)—C (9) 101.71 (15); C (13)—N (4)—C (14) 116.31 (16); N (4)—C (14)—C (15) 124.48 (18).

of 10 mg/L. All of the compounds exhibited low inhibitory activities on the growth of wheat coleoptile and cucumber cotyledon, and the inhibitory ratio (%) are listed in Table 4.

EXPERIMENTAL

All reactions were carried out under nitrogen and monitored by thin-layer chromatography. The ^{1}H NMR spectra were measured on a Bruker AC 300, using tetramethylsilane and deuterochloroform as the internal standard and solvent, respectively. Elemental analyses were determined with a Yanaco CHN Corder MT-3 elemental analyzer. Melting points were determined with X-4 digital melting point apparatus, and the thermometer was uncorrected. The intermediate compound **1** was synthesized according to the literature [22], and was used in the next step without further purification.

*Synthesis of 1-(Pyridin-3-yl)-2-(1*H*-1,2,4-triazol-1-yl)ethanone (**2**)*

To a vigorous stirred suspension of 1*H*-1,2,4-triazole (4.08 g, 0.059 mol) in 80 mL acetone, TEA (11.38 g, 0.112 mol) was added in a single portion. The mixture was refluxed for 30 minutes, and then cooled to room temperature. After cooling to −5°C to −10°C in an ice–salt bath, the intermediate compound **1** (15.02 g, 0.053 mol) was added in portions. The mixture was reacted for another 6 h at this temperature, and then refluxed for 30 minutes. The reaction mixture was cooled to room temperature and filtered, and the filtrate was evaporated under reduced pressure. The residue was purified by column chromatography on silica gel (100–200 mesh) with petroleum ether/ethyl acetate (1:1 v/v) as eluant to

TABLE 3 Fungicidal Activities of Compounds **4a–n** (50 mg/L, Relative Inhibitory Ratio %)

	Ratio (%)													
	4a	***4b***	***4c***	***4d***	***4e***	***4f***	***4g***	***4h***	***4i***	***4j***	***4k***	***4l***	***4m***	***4n***
G. zeae	28.6	34.3	17.8	26.9	35.7	22.9	43.5	34.3	26.6	33.3	35.1	22.2	6.1	54.5
A. solani	19.5	41.5	31.7	35.2	18.1	43.9	22.9	48.7	33.1	7.9	34.1	18.4	15.2	34.9
P. asparagi	13.0	34.8	11.9	28.4	32.7	39.1	28.4	39.1	29.8	31.7	41.0	19.7	24.6	15.9
P. piricola	0	43.6	25.8	19.5	41.9	12.8	35.1	35.3	28.7	25.7	31.9	38.1	29.8	28.6
C. rachidicola	25.0	5.0	18.1	25.6	27.9	7.5	21.7	28.1	25.7	18.9	18.8	34.8	19.5	25.3
C. cucumerinum	15.0	15.0	26.8	28.6	35.7	25.0	33.3	30.0	42.6	19.4	29.5	34.1	41.9	29.1

TABLE 4 Plant Growth Regulator Activities of Compounds **4a–n** (10 mg/L)

	Ratio[a]													
	4a	***4b***	***4c***	***4d***	***4e***	***4f***	***4g***	***4h***	***4i***	***4j***	***4k***	***4l***	***4m***	***4n***
Growth of wheat coleoptile	−18.9	−26.7	−2.9	−36.7	−38.9	−40.5	−55.6	−51.8	−28.9	−36.7	−46.8	−48.9	−16.8	−28.8
Growth of cucumber cotyledon	−33.9	−60.9	−25.6	−19.8	−19.1	−9.1	−36.4	−37.9	−38.1	−45.5	−29.7	−19.1	−9.1	−18.9

[a]: inhibition %.

afford 6.39 g yellow solid (yield 63.5%), mp 114–116°C; ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 9.17 (s, 1H, TrH), 9.16 (s, 1H, TrH), 8.24 (s, 1H, PyH), 8.20 (d, $J = 1.8$ Hz, 1H, PyH), 7.97 (s, 1H, PyH), 7.46 (d d, $J = 4.8$ Hz, pyH), 5.64 (s, 2H, CH_2); for $C_9H_8N_4O$ anal, calcd: C 57.44, H 4.28, N 29.77; found: C 57.52, H 4.15, N 29.38.

*General Procedure for the Synthesis of Title Compounds **4a–n***

To a stirred solution of 1-(pyridin-3-yl)-2-(1*H*-1,2,4-triazol-1-yl)ethanone (1.00 g, 3.83 mmol), 4.60 mmol aryl aldehyde, and 30 ml dry chloroform, a few drops of piperidine was added at room temperature under nitrogen. The mixture was then heated under reflux for 4 h. The solvent was evaporated under reduced pressure, and then the residue was purified by column chromatography on silica gel (200–300 mesh) with the solvent system petroleum ether/ethyl acetate (4:1 v/v) to get the desired compounds **4a–n** in various yields.

Biological Activities

The title compounds **4a–n** were screened for their in vitro biological activities against the selected fungi *G. zeae, A. solani, P. asparagi, P. piricola, C. rachidicola*, and *C. cucumerinum* at a concentration of 50 mg/L, and the relative inhibitory ratios (%) against these fungi are listed in Table 3. The plant growth regulatory activities of the title compounds were tested by wheat coleoptile and cucumber cotyledon tests at a concentration of 10 mg/L (Table 4). The biological activities of the title compounds were assayed at the Biological Assay Centre, Nankai University, Tianjin, according to procedures described previously [18].

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