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Journal of Organometallic Chemistry 691 (2006) 2686–2690
Journal of Organometallic Chemistry
www.elsevier.com/locate/jorganchem

Synthesis and biological activities of new 1*H*-1,2,4-triazole derivatives containing ferrocenyl moiety

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Received 8 November 2005; received in revised form 20 January 2006; accepted 2 February 2006
Available online 3 March 2006

Abstract

Some new 1*H*-1,2,4-triazole derivatives containing ferrocenyl moiety were synthesized in various yields by the condensation of ferrocenecarboxaldehyde with 1-(1*H*-1,2,4-triazol-1-yl)-3-aryl-2-one in toluene. Their structures of all these new compounds have been confirmed with ^{1}H NMR, IR, MS and elemental analysis. Their results of bioassay showed that some title compounds exhibited some degree of antifungal and plant growth regulatory activities.

Keywords: 1*H*-1,2,4-triazole; Ferrocenecarboxaldehyde; Biological activity; Synthesis

1. Introduction

During the latest few decades, much attention has been paid to the synthesis of 1*H*-1,2,4-triazole derivatives which possess anti-inflammatory [1], antiviral [2], antimicrobial [3], antitumorial [4], anticonvulsant [2], analgesic [5], antihypotensive [6], antiparasitic, fungicidal, insecticidal, herbicidal and plant growth regulatory activities [7]. Although it was indicated that replacement of aromatic groups by the ferrocenyl moiety has increased the antibacterial activities of penicillin and cephalosporin antibacterials [8], the broad application of ferrocenyl moiety to biologically active compounds has not gained much attention. Encouraged by our previous reported results [9], we designed and synthesized some new 1*H*-1,2,4-triazole derivatives containing ferrocenyl moiety, which have been characterized by 1H NMR, IR, MS spectra, together with elemental analysis and diffraction analysis. Preliminary bioassay showed in vitro biological activities and plant growth regulatory activities.

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doi:10.1016/j.jorganchem.2006.02.035

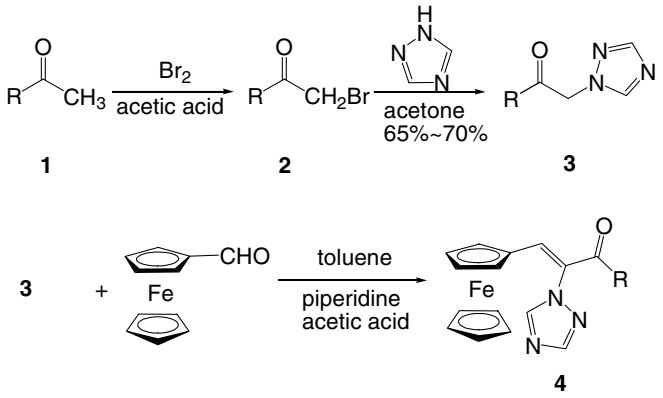
2. Results and discussions

2.1. Synthesis of 2-bromo-1-aryl-ethanone (**2**) and 1-aryl-2-(1H-1,2,4-triazol-1-yl)-ethanones (**3**)

Most of the 2-bromo-1-arylethanone derivatives **2** were synthesized [10] by the reaction of ketones with bromine in acetic acid with yields of 65–70%, pyridinium tribromide was used for the bromination of 2-acetylpyridine and 3-acetylpyridine [11]. Without being purified, the intermediates **2** can be converted to 1-aryl-2-(1*H*-1,2,4-triazol-1-yl)-ethanones **3** [12] (Scheme 1) in the next step.

2.2. Synthesis of 1-ferrocenyl-3-aryl-2-(1H-1,2,4-triazol-1-yl)-prop-2-en-1-one derivatives (**4**)

The condensation of ferrocenecarboxaldehyde with 1-aryl-2-(1*H*-1,2,4-triazol-1-yl)-ethanones (**3**) were carried out under nitrogen atmosphere in toluene using piperidine and acetic acid as catalyst, sodium carbonate, potassium hydroxide, and triethylamine were also used as the catalysts, but the yields were lower. The reason may be that the formation of enamines by piperidine and 1-aryl-2-(1*H*-1,2,4-trizol-1-yl)-ethanones (**3**) improved the



Scheme 1.

nucleophilicity of α-carbon atom of 1-aryl-2-(1*H*-1,2,4-triazol-1-yl)-ethanones **3** (Scheme 1).

Of all the title compounds **4** separated by silica gel column chromatography, *Z* isomers were the major ones except **4j** (R = 4-OCH_3, *E*-isomer), the ratio of **4i** (R = 4-OCH_3, *Z*-isomer) and **4j** (*E*-isomer) is 94:6. Due to the bulkiness of ferrocenyl unit, single crystal X-ray diffraction analysis of compound **4q** (Fig. 1) revealed that the substituted aryl group was spatially repulsed and swerved to nearby the triazole group, *Z*-isomers is the stable configuration of compounds **4**. It is worthy to note that in our previous work [14] only one analogous example was able to be separated on silica gel to give two pure *Z* and *E* isomers (Scheme 2).

2.3. Biological activities

Some selected title compounds **4** were screened for their biological activities in vitro against the *Gibberella zeae*, *Alternaria solani*, *Cercospora arachidicola*, *Physalospora piricola*, *Phomopsis asparagi*, *Cladosporium cucumerinum*, *Sclerotinia sclerotiorum*, and *Piricularia oryzae* at the concentration of 50 mg/L, and the relative inhibitory ratios (%) against these fungi were listed in Table 1.

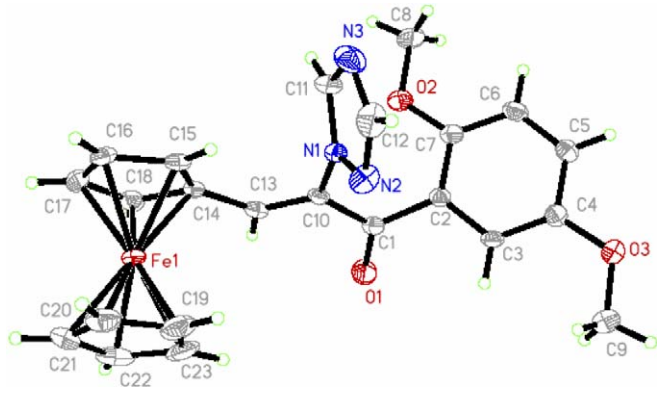


Fig. 1. Molecular structure and crystallographic numbering scheme for compounds **4q**. Selected bond lengths (Å): O(1)–C(1) 1.216(4); O(2)–C(7) 1.365(4); O(3)–C(4) 1.376(4); N(1)–C(10) 1.436 (4); N(2)–C(12) 1.312(4); O(3)–C(9) 1.405(4); O(2)–C(8) 1.430(4); C(10)–C(13) 1.332(4); C(13)–C(14) 1.445(4); C(14)–C(15) 1.426(4). Selected bond angles (°): C(11)–N(3)–C(12) 102.3(3); O(1)–C(1)–C(10) 120.2(3); O(1)–C(1)–C(2) 120.2(3); C(10)–C(1)–C(2) 119.5(3); C(7)–O(2)–C(8) 117.8(3); C(4)–O(3)–C(9) 1117.9(3); C(11)–N(1)–N(2) 109.5(3); C(11)–N(1)–C(10) 129.8(3); C(7)–C(2)–C(1) 121.9(3); C(3)–C(2)–C(1) 117.9(3); C(3)–C(4)–O(3) 124.5(3); O(3)–C(4)–C(5) 115.4(3); O(2)–C(7)–C(2) 116.1(3); O(2)–C(7)–C(6) 124.5(3).

The screening data revealed that all selected compounds **4** showed some antifungal activities, among which **4a**, **4b**, **4c** and **4f**, showed 100% inhibitory ratios against *P. oryzae*, *C. cucumerinum*, and *S. sclerotiorum*. Compared with known commercial antifungal analog (Triadimenfon), the antibacterial activities of most title compounds were not encouraging, although some compounds manifested certain antibacterial activity. To the best of our knowledge, a linkage between the triazole ring and the aryl group via a carbon–carbon single or double bond is essential for fungicidal activities. In addition, it has been proved that an extended carbon backbone linking the triazole cycle and the aryl group in an almost linear fashion possesses higher activity than a distorted backbone. The X-ray structure of (**4q**) shows that, because of the bulkiness of ferrocene, the triazole cycle and the aryl group are not connected in such a way, but via a bent linkage (Fig. 1). and most of this series of compounds display low fungicidal activity. This may imply that a bulky group close to the triazole cycle is not a wise choice for the generation of compounds with fungicidal activities.

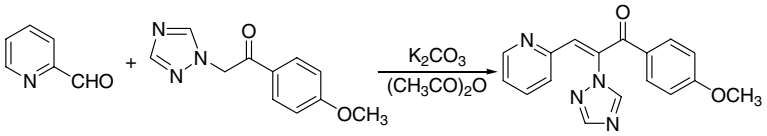
The plant growth regulatory activities of the title compounds were tested by wheat coleoptile and cucumber cotyledon test at the concentration of 10 mg/L. All of the compounds exhibited low inhibitory activities on the growth of wheat coleoptile and cucumber cotylendon, and the inhibitory ratio is −3.2% to −15.3%.

3. Experimental

All reactions were carried out under N_2 atmosphere with the exclusion of moisture and monitored by TLC. ^{1}H NMR spectra were obtained at a Brucker AC-300 spectrometer in $CDCl_3$ solution with TMS as internal standard, and the Chemical shift values (δ) were given in ppm. All melting points were determined at a Yanaco-241 apparatus and thermometer was uncorrected. Elemental analyses were determined at a Yanaco CHN Corder elemental analyzer. X-ray diffraction data were recorded at 293 K at a Bruker Smart 1000 diffractionmeter (graphitemonochromatized Mo Kα radiation $\lambda = 0.7103$ Å). Ferrocenecarboxaldehyde was synthesized in yield of 70% [13], 2-bromo-1-(pyridine-2-yl)ethanone and 2-bromo-1-(pyridine-3-yl)ethanone were prepared in yield of 92% [11]. The biological activity of the title compounds were assayed at the Biological Assay Centre, Nankai University according to procedures described previously [15].

3.1. General procedure for the synthesis of 2, 3

To a vigorous stirred suspension of 1*H*-1,2,4-triazole (5.52 g, 0.08 mol) and 2-bromo-1-arylethanones **2**



Scheme 2.

Table 1
Fungicidal activity of some compounds **4**

Compound	R	Relative inhibitory ratio (%) (50 mg/L)							
		G. zeae	*A. solani*	*C. ara.*	*P. piri.*	*P. asp.*	*C. cucum.*	*S. scler.*	*P. oryzae*
4a	Ph	18.9	22.8	0	18.7	22.6	72.5	77.9	100
4b	*p*-ClPh	12.3	23.9	16.2	16.6	19.6	86.9	100	51.7
4c	*p*-BrPh	16.5	25.4	0	13.5	15.3	100	69.8	38.5
4f	*m*-CH_3Ph	15.2	11.1	0	0	13.8	79.8	100	85.1
4j	2-Pyridine	12.3	12.5	0	15.5	24.4	66.3	85.6	61.2
4k	3-Pyridine	11.7	10.5	0	22.5	0	54.9	46.8	47.3
4l	2.4-F_2Ph	17.7	13.7	0	0	0	34.2	23.5	31.9
4m	2.4-Cl_2Ph	16.7	30.8	23.1	18.9	14.6	26.8	52.2	66.2
4o	3.4-Cl_2Ph	18.1	21.1	0	0	0	37.7	49.7	32.1
4p	4-FPh	17.6	29.8	0	23.6	24.9	55.5	60.0	19.4
4q	2.5-Cl_2Ph	10.0	24.6	0	15.9	34.2	66.6	66.8	52.1

Table 2
physical properties and elemental analysis data for compounds **4**

Entry	R	Yield (%)	m.p. (°C)	Elemental analysis (found/calcd.) (%)		
				C	H	N
4a	Ph	82.6	155–156	65.95 (65.80)	4.58 (4.44)	10.78 (10.97)
4b	*p*-ClPh	81.8	149–150	60.34 (60.36)	3.98 (3.83)	10.03 (10.06)
4c	*p*-BrPh	81.5	127–128	54.48 (54.54)	3.41 (3.46)	8.97 (9.09)
4d	*m*-BrPh	79.6	199–200	54.56 (54.54)	3.47 (3.46)	9.05 (9.09)
4e	*m*-NO_2Ph	80.7	178–179	58.60 (58.88)	3.83 (3.74)	13.06 (13.08)
4f	*m*-CH_3Ph	40.2	151–154	66.46 (66.50)	4.80 (4.78)	10.35 (10.58)
4g	*o*-CH_3Ph	41.9	151–152	66.50 (66.50)	4.75 (4.78)	10.40 (10.58)
4h	*p*-CH_3OPh(*Z*)	70.8	130–132	63.96 (63.92)	4.69 (4.60)	10.25 (10.17)
4i	*p*-CH_3OPh(*E*)	4.5	156–159	63.99 (63.92)	4.60 (4.60)	10.10 (10.17)
4j	2-Pyridine	36.9	146–147	62.65 (62.68)	4.06 (3.94)	14.45 (14.62)
4k	3-Pyridine	35.8	165–166	62.78 (62.68)	4.10 (3.94)	14.72 (14.62)
4l	2.4-F_2Ph	79.6	155–156	60.12 (60.17)	3.59 (3.61)	10.13 (10.02)
4m	2.4-Cl_2Ph	78.8	127–129	55.57 (55.75)	3.35 (3.35)	59.29 (9.29)
4n	3.4-Cl_2Ph	73.9	172–173	55.86 (55.75)	3.34 (3.35)	9.28 (9.29)
4o	4-FPh	79.4	150–152	62.58 (62.86)	4.28 (4.02)	10.55 (10.47)
4p	2.5-Cl_2Ph	76.7	152–154	55.74 (55.75)	3.48 (3.35)	9.28 (9.29)
4q	2.5-$(CH_3O)_2$Ph	58.7	191–193	62.49 (62.32)	4.87 (4.78)	9.57 (9.48)

(0.05 mol) in 30 ml of acetone, was added triethylamine (8.1 g, 0.05 mol) dropwise over a period of 1 h with the temperature below 0 °C, and the reaction mixture was stirred for another 30 min at room temperature, Then the mixture was filtered to move triethylamine hydrobromide salt precipitates, the precipitates was washed with 3×10 ml of acetone, and the filtrate was evaporated under reduced pressure, and the residues was dissolved in 50 ml of chloroform, and washed with 2×25 ml of water. After evaporation of chloroform, the yellow solid was recrystallized with 2-propanol, and 1-aryl-2-(1*H*-1,2,4-triazol-1-yl)-ethanones (**3**) were obtained as white solids with yields of 35.8–81.8%.

3.2. General procedure for the synthesis of *4*

To a stirred solution of 1-aryl-2-(1*H*-1,2,4-triazol-1-yl)-ethanones (**3**) (0.01 mol), 2.36 g (0.011 mol) ferrocenecarboxaldehyde in dry toluene (50 ml), was added five drops of piperidine and five drops of glacial acetic acid at room temperature under nitrogen atmosphere. The mixture was then heated to reflux and kept at this temperature until completion of the reaction in 4–6 h, meanwhile, while the water generated was evaporated off. The toluene was evaporated off in vacuum and the residue was purified by chromatography on silica gel with the solvent system of ethyl acetate and petroleum ether (60–90 °C) to afford desired

Table 3
^{1}H NMR spectra data for compounds **4**

Entry	R	^{1}H NMR(δ)
4a	Ph	3.88 (s, 2H, C_5H_4), 4.30 (s, 5H, C_5H_5), 4.56 (s, 2H, C_5H_4), 7.25 (s, 1H, =CH), 7.28–8.00 (m, 5H, Ar), 8.10 (s, 1H, TrH), 8.30 (s, 1H, TrH)
4b	*p*-ClPh	3.76 (s, 2H, C_5H_4), 4.16 (s, 5H, C_5H_5), 4.45 (s, 2H, C_5H_4), 7.18 (s, 1H, =CH), 7.33 (d, 2H, ArH), 7.52 (d, 2H, ArH), 7.86 (s, 1H, TrH), 8.21 (s, 1H, TrH)
4c	*p*-BrPh	3.86 (s, 2H, C_5H_4), 4.26 (s, 5H, C_5H_5), 4.56 (s, 2H, C_5H_4), 7.26 (s, 1H, =CH), 7.36 (d, 2H, ArH), 7.73 (d, 2H, ArH), 8.33 (s, 1H, TrH), 8.47 (s, 1H, TrH)
4d	m-BrPh	3.92 (s, 2H, C_5H_4), 4.33 (s, 5H, C_5H_5), 4.59 (s, 2H, C_5H_4), 7.36 (s, 1H, =CH), 7.76 (s, 4H, ArH), 7.56 (s, 1H, TrH), 8.02 (s, 1H, TrH)
4e	*m*-NO_2Ph	3.86 (s, 1H, C_5H_4), 4.26 (s, 5H, C_5H_5), 4.56 (s, 2H, C_5H_4), 7.26 (s, 1H, =CH), 7.52–8.13 (m, 4H, ArH), 8.33 (s, 1H, TrH), 8.67 (s, 1H, TrH),
4f	m-CH_3Ph	2.45 (s, 3H, CH_3), 3.89 (s, 2H, C_5H_4), 4.33 (s, 5H, C_5H_5), 4.59 (s, 2H, C_5H_4), 7.33–7.60 (m, 5H, =CH, ArH), 7.66 (s, 1H, TrH), 8.30 (s, 1H, TrH)
4g	*o*-CH_3Ph	3.86 (s, 3H, OCH_3), 3.89 (s, 2H, C_5H_4), 4.26 (s, 5H, C_5H_5), 4.53 (s, 2H, C_5H_4), 7.12 (d, 2H, ArH), 7.38 (s, 1H, =CH), 7.78 (d, 2H, ArH), 7.66 (s, 1H, TrH), 8.30 (s, 1H, TrH)
4h	*p*-CH_3O-Ph(Z)	3.86 (s, 3H, OCH_3), 3.89 (s, 2H, C_5H_4), 4.26 (s, 5H, C_5H_5), 4.53 (s, 2H, C_5H_4), 7.12 (d, 2H, ArH), 7.38 (s, 1H, =CH), 7.78 (d,2H, ArH), 7.66 (s, 1H, TrH), 8.30 (s, 1H, TrH)
4i	*p*-CH_3O-Ph(E)	2.50 (s, 3H, OCH_3), 3.89 (s, 2H, C_5H_4), 4.33 (s, 5H, C_5H_5), 4.59 (s, 2H, C_5H_4), 7.41 (s, 1H, =CH), 7.53 (s, 1H, ArH), 7.56 (s, 1H, TrH), 8.36 (s, 1H, TrH)
4j	2-Pydine	3.86 (s, 2H, C_5H_4), 4.28 (s, 5H, C_5H_5), 4.51 (s, 2H, C_5H_4), 7.84–7.93 (m, 3H, PyH) 8.635, 8.649 (s, 1H, PyH), 8.43 (s, 1H, =CH), 8.18 (s, 1H, TrH), 8.22 (s, 1H, TrH)
4k	3-Pydine	3.84 (s, 2H, C_5H_4), 4.25 (s, 5H, C_5H_5), 4.55 (s, 2H, C_5H_4), 7.71 (s, 1H, =CH), 7.415–8.00 (3H, m, PyH), 8.952 (s, 1H, PyH), 8.15 (s, 1H, TrH), 8.24 (s, 1H, TrH)
4l	2.4-F_2Ph	3.68 (s, 2H, C_5H_4), 4.16 (s, 5H, C_5H_5), 4.24 (s, 2H, C_5H_4), 7.25–7.53 (3H, m, ArH), 7.36 (s, 1H, =CH), 8.13 (s, 1H, TrH), 7.82 (s, 1H, TrH)
4m	2.4-Cl_2Ph	3.80 (s, 2H, C_5H_4), 4.54 (s, 5H, C_5H_5), 4.57 (s, 2H, C_5H_4), 7.38–7.82 (2H, m, ArH), 7.47 (s, 1H, ArH), 7.71 (s, 1H, =CH), 8.17 (s, 1H, TrH), 8.21 (s, 1H,TrH)
4n	3.4-Cl_2Ph	3.84 (s, 2H, C_5H_4), 4.45 (s, 5H, C_5H_5), 4.54 (s, 2H, C_5H_4), 7.50–7.58 (2H, m, ArH) 7.84 (s, 1H, ArH), 7.65 (s, 1H, =CH), 8.13 (s, 1H, TrH), 8.23 (s, 1H, TrH)
4o	4-FPh	3.83 (s, 2H, C_5H_4), 4.50 (s, 5H, C_5H_5), 4.52 (s, 2H, C_5H_4), 7.16–7.20 (m, 2H, ArH), 7.72–7.78 (m, 2H, ArH), 7.63 (s, 1H, =CH), 8.15 (s, 1H, TrH), 8.22 (s, 1H, TrH)
4p	2.5-Cl_2Ph	3.45 (s, 2H, C_5H_4), 4.25 (s, 5H, C_5H_5), 4.56 (s, 2H, C_5H_4), 7.26–7.42 (3H, m, ArH), 7.748 (s, 1H, =CH), 8.17 (s, 1H, TrH), 8.22 (s, 1H, TrH)
4q	2.5-(CH_3-O)$_2$Ph	3.79 (6H, m, OCH_3), 3.81 (s, 2H, C_5H_4), 4.21 (s, 5H, C_5H_5), 4.78 (s, 2H, C_5H_4), 6.88–7.01 (3H, m, ArH) 7.53 (s, 1H, =CH), 8.15 (s, 1H, TrH), 8.16 (s, 1H,TrH)

2-(1H-1,2,4-triazol-1-yl)1-ferrocenyl-3-aryl-prop-2-en-1-one derivatives **4** in various yields. The physical properties, elemental analysis data and ^{1}H NMR spectra of compounds **4** thus synthesized were listed in the Tables 2 and 3, respectively.

Acknowledgements

We gratefully acknowledge the National Natural Science Foundation of China (NNSFC) (No. 29872022, 20172030) and the Research Fund for the Doctoral program of High Education (RFDP) (No. 9805520) for financial support.

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