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# Novel isoxazoline ligand with ferrocene backbone: preparation and application in Heck reaction with water as solvent

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Two novel isoxazoline *N*,*N*-bidentate ligands with ferrocene backbone have been synthesized and employed for the palladium-catalyzed Heck coupling reaction. Among them, 1,3-bis-(5-ferrocenylisoxazoline-3-yl)benzene was found to be thermally stable and a highly effective ligand for Heck coupling reaction in neat water without N<sub>2</sub> protection, affording the desired coupling products in good to excellent yield with high diastereoselectivity. The developed catalytic system was also well workable for 1,2-disubstituted alkenes, which were less involved in the Heck reaction for its larger steric hindrance. Copyright © 2014 John Wiley & Sons, Ltd.

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Keywords: ferrocene; bisoxazoline; Heck reaction; water

#### Introduction

During the last four decades, the chemistry of ferrocenyl complexes has attracted much attention owing to their chemical versatility combined with high thermal stability.<sup>[1]</sup> Since found, they have been widely applied in electrochemistry, material science, synthesis and catalysis.<sup>[2]</sup> Besides, the ferrocenyl ligand represents one of the most important achiral and chiral ligands in transition-metal catalyzed transformations for its two crucial roles in catalytic cycle: (a) as a backbone or substituent in ligands providing a special and unique geometry; and (b) via its redox activity and the potential control of the reactivity of a coordinated, catalytically active metal center.<sup>[3]</sup> Among these, the ferrocenyl-oxazol(in)e ligands constitute an important group which have been extensively and successfully applied in palladium-catalyzed allylic substitutions and alkylations of aldehydes.<sup>[4]</sup> Contrary to the abundant literature on ferrocenyloxazol(in)es, the isoxazol(in)e ligands containing ferrocene backbone have been less explored. The isoxazol(in)e ring, which has the same rigid five-membered ring containing two heteroatoms (nitrogen and oxygen) as oxazol(in)e analogues, seems promising as a ligand for transition-metal promoted reactions.<sup>[5]</sup> Recently, Sasai's group reported their successful preparation of novel bis (isoxazoline) compounds containing a spiro backbone (SPRIXs) and further illustrated their versatile utility in various transitionmetal catalyzed asymmetric synthesis.<sup>[6]</sup>

Prompted by the potential performance of isoxazol(in)e ligand and the unique character of ferrocenyl substituent in the catalytic cycle, our group have initiated a program dealing with ferrocenylisoxazole derivatives since 2006, and have reported the synthesis of a series of ferrocenyl-substituted isoxazole compounds. Further explorations have demonstrated their satisfactory performance as ligands in palladium-catalyzed C–C bond formation reactions under phosphine-free conditions.<sup>[7]</sup> As part of our ongoing quest to find new ferrocene ligands, we herein wish to report the synthesis of ferrocenylisoxazoline ligands and further explored their catalytic utility in palladium-catalyzed Heck reactions.

#### **Results and Discussion**

#### Synthesis of Isoxazoline Ligands 4, 5 and 6

Novel isoxazoline ligands with a ferrocene backbone were synthesized following a method reported by our group previously,<sup>[7]</sup> and the procedure for 1,3-bis-(5-ferrocenylisoxazoline-3-yl)benzene **4** is shown in Scheme 1 as representative'. Its structure was unambiguously confirmed by X-ray diffraction analysis, which was in accordance with NMR, IR and mass spectra. Notably, no diastereomers were observed in the formation of ligand **4**, indicating that 1,3-dipolar cycloaddition proceeded with excellent diasteroselectivity. Following a similar procedure, its monoferrocenyl analogue **5** and another *N*,*N*-bidentate ligand, 1,3-bis-(5-phenylisoxazoline-3-yl)benzene **6**, were obtained with the corresponding aldehydes and terminal alkenes as starting materials in good yields (Fig. 1).

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Scheme 1. Synthesis of 1,3-bis-(5-ferrocenylisoxazoline-3-yl) benzene 4.



Figure 1. Another two isoxazoline ligands.

#### Heck Coupling Reaction Catalyzed by Pd(OAc)<sub>2</sub>/Ligand 4

With the desired isoxazoline ligands in hand, their catalytic properties were evaluated in Heck coupling reactions, and the coupling of iodobenzene and butyl acrylate was chosen as a model reaction for optimization of conditions.

As indicated in Table 1, the bisoxazoline ligand **4** was found to be most efficient for the coupling reaction, affording the desired coupling alkene in 87% yield with excellent stereoselectivity under low catalyst loading (only 0.2 mol% Pd(OAc)<sub>2</sub> and 0.4 mol% ligand were used). The best result obtained with ligand **4** 

revealed the necessity for the ferrocenyl group and the advantage of two isoxazoline rings (entries 1-3). Although various bases could afford this transformation, NaOAc proved to be the best choice (entries 1, 4 and 5). Replacement of organic solvent DMF with neat water did not bring any detriment to this transformation (entries 5-7). Water is undoubtedly the ideal solvent for its safety, non-toxicity and economical availability. Although the coupling product could be obtained under ligand-free conditions, an incomplete conversion and palladium black were observed in the flask. A temperature of 80°C was suitable for this transformation (entries 5, 7 and 8). Lowering the ratio of ligand would decrease the yield (entry 11). Tetra-n-butylam-

monium bromide (TBAB) was helpful.

Contrary to traditional palladium-catalyzed Heck coupling in the presence of phosphane ligands, this new catalytic system has no need for the exclusion of air, indicating the high thermal stability of ferreocenylisoxazoline ligand (entries 9 and 13). Based on the investigation of various parameters, the optimal reaction condition was performed with 2.0 equiv. NaOAc in the presence of 0.2 mol% Pd(OAc)<sub>2</sub> and 0.4 mol% ligand **4** in H<sub>2</sub>O at 80°C under air atmosphere (Table 1, entry 13).

With the optimal conditions in hand, various aryl halides and alkenes were used to test the versatility of the catalytic system (Table 2). Various terminal olefins were tested first and satisfactory

Table 1. Optimization of coupling reaction conditions												
✓ I + COO <sup>n</sup> Bu → COO <sup>n</sup> Bu → COO <sup>n</sup> Bu												
Entry	Ligand	Solvent	Base	Additive	<i>T</i> (°C)	Time	Yield <sup>a,b</sup>					
1	4	DMF	K <sub>2</sub> CO <sub>3</sub>	TBAB	100	6 h	87%					
2	5	DMF	K <sub>2</sub> CO <sub>3</sub>	TBAB	100	6 h	70%					
3	6	DMF	K <sub>2</sub> CO <sub>3</sub>	TBAB	100	6 h	77%					
4	4	DMF	Pyridine	TBAB	100	6 h	83%					
5	4	DMF	NaOAc	TBAB	100	6 h	90%					
6	4	DMF/H <sub>2</sub> O	NaOAc	TBAB	100	6 h	89%					
7	4	H <sub>2</sub> O	NaOAc	TBAB	100	6 h	90%					
8	_	H <sub>2</sub> O	NaOAc	TBAB	100	6 h	54%					
9	4	H <sub>2</sub> O	NaOAc	TBAB	80	4 h	91%					
10	4	H <sub>2</sub> O	NaOAc	TBAB	60	4 h	70%					
11 <sup>c</sup>	4	H <sub>2</sub> O	NaOAc	TBAB	80	4 h	79%					
12	4	H <sub>2</sub> O	NaOAc	—	80	4 h	83%					
13 <sup>d</sup>	4	H <sub>2</sub> O	NaOAc	TBAB	80	4 h	90%					

<sup>a</sup>Unless mentioned, the reaction was performed under the following conditions: iodobenzene(1 mmol), butyl acylate (1.5 mmol), Pd(OAc)<sub>2</sub> (0.2 mol%), ligand (0.4 mol%), base (2 mmol), TBAB (0.5 mmol) under N<sub>2</sub>.

<sup>b</sup>lsolated yield after flash chromatography.

<sup>c</sup>Pd(OAc)<sub>2</sub> (0.2 mol%), ligand (0.2 mol%).

<sup>d</sup>The reaction was performed in air.

TBAB, tetra-*n*-butylammonium bromide.

Table 2. Examination of reaction scope <sup>a</sup>										
R <sup>1</sup>	X R <sup>2</sup>	∕∼ <sub>R³</sub>	Pd(OA Ligand	c) <sub>2</sub> (0.2mol%), t <b>4</b> (0.4 mol%) , TBAB, H <sub>2</sub> O, 80		R <sup>2</sup> R <sup>3</sup>				
					K.					
Entry	R <sup>1</sup>	Х	R <sup>2</sup>	R <sup>3</sup>	Time	Yield (%) <sup>b</sup>				
1	2-NO <sub>2</sub>	I	Н	Ph	4	93				
2	2-NO <sub>2</sub>	I	Н	Fc	6	88				
3	2-NO <sub>2</sub>	I	Н	CHO	3	94				
4	2-NO <sub>2</sub>	I	Н	COO <sup>n</sup> Bu	3	95				
5	4-NO <sub>2</sub>	Ι	Н	COO <sup>n</sup> Bu	3	95				
6	4-NO <sub>2</sub>	I	Н	COOCH <sub>3</sub>	3	94				
7	4-COOH	I	Н	COO <sup>n</sup> Bu	4	94				
8	$4-COOC_2H_5$	I	Н	COO <sup>n</sup> Bu	4	94				
9	4-Cl	I	Н	COO <sup>n</sup> Bu	4	95				
10	Н	I	Н	COO <sup>n</sup> Bu	4	90				
11	Н	I	Н	Ph	4	94				
12	4-CH <sub>3</sub>	I	Н	COO <sup>n</sup> Bu	4	87				
13	4-OCH <sub>3</sub>	Ι	Н	COO <sup>n</sup> Bu	6	89				
14	4-OH	Ι	Н	COO <sup>n</sup> Bu	6	86				
15	2-NO <sub>2</sub>	Br	Н	COO <sup>n</sup> Bu	6	90				
16	4-Br	Br	Н	COO <sup>n</sup> Bu	6	92 <sup>d</sup>				
17	4-CHO	Br	Н	$COOC_2H_5$	6	91				
18	Н	Br	Н	COO <sup>n</sup> Bu	4	88				
19	Н	Cl	Н	COO <sup>n</sup> Bu	6	46 <sup>e</sup>				
20	4-CH <sub>3</sub>	Ι	Ph	COOPh	6	83 <sup>e</sup>				
21	4-CH <sub>3</sub>	I	Ph	Ph	6	86 <sup>e</sup>				
22	4-CH <sub>3</sub>	I	$R^2$ -	$+R^3 = C_4H_8$	6	80 <sup>e</sup>				
23	4-NO <sub>2</sub>	I	Ph	COOPh	6	83 <sup>e</sup>				
24	4-COOC <sub>2</sub> H <sub>5</sub>	Ι	Ph	COOPh	6	78 <sup>e</sup>				
<sup>a</sup> General reaction conditions: aryl halide (1 mmol), olefin (1.5 mmol) NaOAc (2 mmol) TBAB (0.5 mmol) Pd(OAc), (0.2 mol										

mmol), NaOAc (2 mmol), TBAB (0.5 mmol), Pd(OAc)<sub>2</sub> (0.2 mol %), ligand **4** (0.4 mol%), H<sub>2</sub>O (4 ml) in air. <sup>b</sup>Isolated yield after flash chromatography.

<sup>c</sup>The product is *E*-isomer.

<sup>d</sup>In order to obtained double-coupling product, the amount of olefin was increased to 2.4 mmol.

<sup>e</sup>The reaction was performed at 100°C.

results were obtained (entries 1-4). Notably, acrolein could be well tolerated in this transformation, making this method more favorable for synthetic applications owing to the high reactivity of acrolein (entry 3). The hindrance of substituents in aryl halides seemed to have little effect on this reaction. The coupling of stereo-hindered 2-nitroiodobenzene could generate a parallel result with 4-nitroiodobenzene (entries 4 and 5). Compared to the weak steric effect of substituents, the electron effect seemed to be more sensitive. In general, the reactions of aryl halides with electron-withdrawing groups (such as  $-NO_{2}$ , -COOH,  $-COOC_{2}H_{5}$ ) could be accomplished in a shorter time and with higher yields than those with electron-releasing groups (entries 5-9 vs. 12-14). The coupling of any bromides gave slightly lower but still satisfactory yields (entries 15-18). In the case of inactive aryl chloride, the desired product could be obtained in moderate yield with higher temperature and longer time (entry 19). It is worth noting that only E-isomers were collected in all the reactions. To our delight, the new developed catalytic system was equally workable for the coupling of disubstituted alkenes, which were less introduced as

coupling partners in Heck reactions for their steric hindrance. In our method, the reactions of disubstituted alkenes with aryl iodides proceeded smoothly to produce the desired trisubstituted alkenes with satisfactory yield at a slightly higher temperature (entries 20–24). Compared to the ferrocenylisoxazole ligand reported by our group previously,<sup>7c</sup> the newly developed catalytic system offers a much milder approach, with a broader substrate scope, giving coupling compounds with higher yields under much lower catalyst loading.

# Conclusions

In summary, a thermally stable bisoxazoline ligand with ferrocene backbone, namely 1,3-bis-(5-ferrocenylisoxazoline-3-yl)benzene **4**, has been easily synthesized. Further investigation revealed its excellent catalytic property for palladium-catalyzed Heck coupling reactions in neat water under mild conditions. The developed catalytic system is workable for a broad spectrum of substrates including more hindrance-disubstituted alkenes, affording the desired coupling products in good to excellent yield. A variety of functional groups such as methoxyl, hydroxyl, formyl, nitro and ester groups were well tolerated. Notably, further application of the ferrocenylisoxazoline ligand in other metal-catalyzed reactions and its asymmetric version is under study in our laboratory.

## **Experimental**

#### General

All chemicals were obtained from commercial suppliers and used without further purification. Melting points were measured using a WC-1 microscopic apparatus. IR spectra were recorded on a PerkinElmer 983 FT-IR spectrometer. <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra were recorded on a Bruker Avance DMX300 instrument. Chemical shifts are reported in ppm with tetramethylsilane (TMS, 0.00 ppm) as internal standard. Mass spectra (EI) were performed on an HP-5989 instrument with ionization energy maintained at 70 eV. Elemental analyses were carried out on an EA-1110 elemental analyzer. X-ray crystallographic data were made on a Rigaku Mercury CCDC X-ray diffractometer (3 kV, sealed tube) at 193 K by using graphite monochromated Mo- $K_a$  (K= 0.71070 AX).

# General Procedure for 1,3-Bis-(5-ferrocenylisoxazoline-3-yl) benzene 4

Aldoxime **2** was prepared according to a previously described procedure.<sup>7b</sup> Vinylferrocene **3** was synthesized by dehydration of 1-(ferrocenyl)ethanol following Wang's method.<sup>[8]</sup>

Aldoxime **2** (1 mmol) and chlorosuccinimide (2 mmol) were stirred in 3 ml dry dichloromethane (DCM) and 1.5 mL DMF at 30°C for about 3 hours. After the complete conversion of aldoxime **2**, vinylferrocene **3** (2 mmol) was added followed by dropwise addition of 0.28 ml triethylamine in 3 ml DCM over 1.5 h and the mixture was stirred overnight at room temperature. The solid thus formed was filtrated and washed with DCM to give gold crystals of compound **4** with 90% yield; m.p. 237–238°C; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  = 8.03 (s, 1H, H<sup>2</sup>), 7.78 (d, *J* = 7.8 Hz, 2H, H<sup>4</sup> and H<sup>6</sup>), 7.48 (t, *J* = 7.8 Hz, 1H, H<sup>5</sup>), 5.58 (t, *J* = 9.0 Hz, 2H, H<sup>9</sup> and H<sup>9'</sup>), 4.20–4.48 (m, 18H, H<sup>11</sup>–H<sup>19</sup> and H<sup>11′–</sup>H<sup>19'</sup>), 3.68 (dd, *J* = 9.0 Hz, 10.8 Hz, 2H, H<sup>8a</sup> and H<sup>8a'</sup>), 3.42 (dd, *J* = 9.0 Hz, 10.8 Hz, 2H, H<sup>8b</sup> and H<sup>8b'</sup>) ppm; <sup>13</sup>C NMR

(75 MHz, CDCl<sub>3</sub>)  $\delta$  155.6 (2C, C<sup>7</sup> and C<sup>7'</sup>), 130.4 (2C, C<sup>1</sup> and C<sup>3</sup>), 129.2 (1C, C<sup>2</sup>), 128.0 (2C, C<sup>4</sup> and C<sup>6</sup>), 124.7 (1C, C<sup>5</sup>), 86.6 (2C, C<sup>9</sup> and C<sup>9'</sup>), 80.2 (2C, C<sup>10</sup> and C<sup>10'</sup>), 68.8 (5C, C<sup>15</sup>–C<sup>19</sup>), 68.7(5C, C<sup>15'</sup>–C<sup>19'</sup>), 67.5 (4C, C<sup>11</sup>, C<sup>14</sup>, C<sup>11'</sup>and C<sup>14'</sup>), 66.1 (4C, C<sup>12</sup>, C<sup>13</sup>, C<sup>12'</sup> and C<sup>13'</sup>), 41.2 (2C, C<sup>8</sup> and C<sup>8'</sup>) ppm; IR: 3001, 2380, 1643, 1250, 1048, 803, 680 cm<sup>-1</sup>; El-MS: *m/z*=607 [M+Na] <sup>+</sup>; Anal. calcd for C<sub>32</sub>H<sub>28</sub>Fe<sub>2</sub>N<sub>2</sub>O<sub>2</sub>: C, 65.78; H, 4.83; N, 4.79; found: C, 65.69; H, 4.64; N, 4.72.

#### 3-Pyridyl-5-ferrocenyl-isoxazoline 5

83% yield; light-yellow solid; m.p. 203–205°C; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  = 8.64 (s, 1H, H<sup>1</sup>), 8.06 (d, *J* = 7.8 Hz, 1H, H<sup>4</sup>), 7.73 (t, *J* = 7.8 Hz, 1H, H<sup>3</sup>), 7.26–7.32 (m, 1H, H<sup>2</sup>), 5.59 (t, *J* = 9.9 Hz, 1H, H<sup>8</sup>), 4.18–4.25 (m, 9H, H<sup>10</sup>–H<sup>13</sup> and H<sup>14</sup>–H<sup>18</sup>), 3.80 (dd, *J* = 17.5 Hz, 10.8 Hz, 1H, H<sup>7a</sup>), 3.61 (dd, *J* = 9.0 Hz, 17.5 Hz, 1H, H<sup>7b</sup>) ppm; <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  156.9 (1C, C<sup>6</sup>), 148.5 (1C, C<sup>5</sup>), 148.3 (1C, C<sup>1</sup>), 135.4 (1C, C<sup>4</sup>), 123.2 (1C, C<sup>2</sup>), 120.7 (1C, C<sup>3</sup>), 85.1 (1C, C<sup>8</sup>), 79.8 (1C, C<sup>9</sup>), 67.8 (7C, C<sup>11</sup>, C<sup>12</sup> and C<sup>14</sup>–C<sup>18</sup>), 67.1 (1C, C<sup>10</sup>), 65.0 (1C, C<sup>13</sup>), 39.4 (1C, C<sup>7</sup>) ppm; IR (cm<sup>-1</sup>): 3100, 1651, 1450, 1274, 1000, 930, 817, 703; El-MS: *m/z* = 333 [M+H] <sup>+</sup>; Anal. calcd for C<sub>18</sub>H<sub>16</sub>FeN<sub>2</sub>O: C, 65.08; H, 4.85; N, 8.43; found: C, 65.19; H, 4.95; N, 8.51.

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