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FULL PAPER

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Kryptofix 5 as an inexpensive and efficient ligand for the palladium-catalyzed Mizoroki-Heck reaction

Barahman Movassagh 💿 | Shabnam Ranjbari

Department of Chemistry, K.N. Toosi University of Technology, P.O. Box 16315-1618, Tehran, Iran

Correspondence

Barahman Movassagh, Department of Chemistry, K.N. Toosi University of Technology, P.O. Box 16315-1618, Tehran, Iran Email: bmovass1178@yahoo.com A novel complex of $PdCl_2$ with a multidentate cryptand ligand, Kryptofix 5, has been prepared and characterized by various techniques including ¹H- and ¹³C NMR spectroscopy, Fourier transform infrared (FT-IR), Raman, ultraviolet and visible (UV-VIS) spectroscopy, inductively coupled plasma (ICP), CHN elemental and energy dispersive X-ray analysis (EDX). This heat- and air-stable complex was utilized as a highly active catalyst for the Mizoroki-Heck reaction of aryl halides with various olefins. Interestingly, it was found that aryl bromides as well as aryl iodides were efficiently cross-coupled with terminal alkenes at 130 °C in 10 min. Furthermore, the least reactive aryl chlorides reacted with styrene to obtain the desired products in acceptable yields.

KEYWORDS

aryl halides, cross-coupling, Heck reaction, Kryptofix 5, $PdCl_2$ -K 5 complex

1 | INTRODUCTION

Transition metal-catalyzed cross-coupling reaction to create a new carbon-carbon $(C_{sp2}-C_{sp2})$ bond have played a crucial role in contemporary organic synthesis.^[1] The palladium-catalyzed coupling of aryl halides and terminal alkenes, the Mizoroki-Heck reaction, is a versatile and powerful tool for the preparation of various substituted olefins, dienes, and precursors of conjugated polymers.^[2,3] The Mizoroki-Heck reaction has received considerable attention due to its functional group tolerance and its widespread application in the synthesis of agrochemicals,^[4] pharmaceuticals,^[5] and in enantioselective synthesis of natural products.^[6] Unlike other carbon-carbon bond forming reactions that involve a polar addition, the Mizoroki-Heck reaction can be performed in the presence of sensitive functional groups such as aldehyde, ketone, hydroxyl, amino, cyano, ester, carboxy, or nitro groups, thus avoiding the need for protection and deprotection of functional groups during organic transformation.^[7] Therefore, to find industrial application, several goals have to be met, such as the use of stable and inexpensive ligands, the use of cheap starting materials, and achievement of high

turnover numbers (TON's) with less reactive aryl bromides and chlorides. The catalytic system for an efficient Heck reaction consists of a palladium source, ligand, base and solvent. Usually, suitable ligand is required to stabilize palladium catalyst precursor. Many catalytic systems have been developed for this reaction using different palladium catalysts together with phosphane^[8]</sup> and phosphorous ligands,^[9] as they play an important role in stabilization and in situ generation of Pd(0) species from Pd(II) complexes. However, due to the high cost, toxicity and thermal instability of phosphine ligands, various phosphane-free catalytic systems^[10] have been developed as less complicated and environmentally more desirable alternatives to the original Pd-phosphane catalysts. Therefore, the design of new ligands and their palladium complexes that can catalyze the Mizoroki-Heck reaction of less reactive aryl bromides and chlorides with high efficiency is in high demand.

2 | RESULTS AND DISCUSSION

Palladium catalysts are known to aggregate easily and form palladium black, which may lead to a considerable

loss of catalytic activity.^[11] To overcome this intrinsic problem of homogeneous Pd catalysis, we explored a new class of palladium catalyst system based on cryptands. In cross-coupling reactions, amines are generally used as bases, but they can also serve to stabilize the reactive palladium intermediates.^[12] Because of their stronger σ -donation, which favors both the oxidative addition step and the slow reductive elimination step in the catalytic cycle, ligands of the N,N-type show superior properties to P,P-type ligands in terms of both palladium complexation and catalysis of cross-coupling reactions.^[13] Many N-based ligands are stable and have been reported to be efficient ligands for the Pd-catalyzed Heck and Suzuki coupling reactions.^[14] Among these, the N- and O-donor cryptands represent an important family of multidentate ligands which forms a three-dimentional cavity for metal cations.^[15] A rather intriguing ligand of this type is Kryptofix 5. As part of our ongoing investigation to find a robust and easily prepared system to catalyze C-C bond forming reactions,^[10d, e, 16] we found that a Pd(II) catalyst system based on a commercially available Kryptofix 5 [1,13-bis(8-quinolyl)-1,4,7,10,13-pentaoxatridecane, here abbreviated with K 5] is a very active homogeneous catalyst under aerobic condition. This ligand consist of a crown ether moiety and two guinoline substituent. The metal cation (Pd^{2+}) can be coordinated to the crown ether unit as well as to the quinoline chromophores (Figure 1). This catalyst was prepared in our laboratory for the first time by an easy method.

The Pd(II)-Kryptofix 5 complex has been fully characterized by elemental analysis, NMR, FT-IR, Raman, UV-VIS, inductively coupled plasma (ICP), and energy dispersive X-ray analysis (EDX) techniques. The loading value of Pd(II) in the catalyst, as determined by ICP analysis, was found to be 16.61% (1.56 mmol.g⁻¹) which is in good agreement with the calculated value (17.00%). The elemental composition of the complex was confirmed using EDX and elemental analyses (Figure 2); it revealed the presence of C, N, O, Cl, and Pd elements. Also, the ratio of N/Pd in the catalyst was found to be 2.03.

The IR spectrum of the K 5 ligand shows a strong C-O-C stretching frequency at 1101 cm⁻¹; this band appears at 1112 cm⁻¹ for the PdCl₂–K 5 complex. Also, Raman spectroscopy of the complex showed two prominent bands at 478 and 361 cm⁻¹ which are attributed to



FIGURE 1 PdCl₂-K 5 catalyst



FIGURE 2 The EDX spectrum of PdCl₂-K 5 complex

the stretching bands of the nitrogen-palladium and palladium-chlorine bonds, respectively (Figure 3).

The UV-visible spectra of Kryptofix 5 and its Pd(II) complex were carried out in CH₂Cl₂ at room temperature (Figure 4); The spectrum of ligand, Kryptofix 5, displayed a strong absorption band $(\pi \rightarrow \pi^*)$ at 239 and a weak absorption at 304 nm. The absorption spectrum of the complex, Pd(II)-K 5, showed its first band at 244 nm and the weak band in the range 310-320 nm which might be due to metal to ligand charge transfer (MLCT) bands from the *d*-orbitals of the palladium metal to the π^* of the aryl group.^[17]



FIGURE 3 Raman spectrum of PdCl₂-K 5 complex



FIGURE 4 Absorption spectra of Kryptofix 5 and its Pd(II) complex

To evaluate the usefulness of this multidentate, phosphine-free Pd(II) complex in C–C bond forming reaction, we investigated its application to the Heck reaction of aryl halides. To optimize the reaction conditions with respect to base, solvent, temperature, and the catalyst loading, the coupling reaction between iodobenzene (1 equiv) with styrene (1.1 equiv), and a base (1.1 equiv) was performed as a model reaction under aerobic condition (Table 1). Specifically, five bases and five solvents were surveyed where they were compatible.

Various organic and inorganic bases such as, Et₃N, diisopropylethylamine (DIPEA), K₂CO₃, Na₂CO₃, and KO^tBu were screened. The yield of the Heck product was reduced when inorganic bases were used (Table 1, entries 1-4). A considerable increase in product formation is observed in the presence of triethylamine, in DMF at a high temperature, 130 °C (Table 1, entry 5). Several other solvents, including dimethylsulfoxide (DMSO), Nmethylpyrrolidin-2-one (NMP), dimethylacetamide (DMAc), and toluene were also surveyed under similar conditions, but compared with DMF all gave inferior results (Table 1, entries 6-9). Also, reducing the temperature to 120 °C and 110 °C resulted in the formation of the target product in 76% and 51% yields, respectively (Table 1, entries 10 and 11). Then, various amounts of catalyst between 0.6 and 0.9 mol% were investigated for the reaction (Table 1, entries 5 and 12-14). Among the

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different amounts, 0.9 mol% of the catalyst is found to be the best (Table 1, entry 5). Therefore, it was decided to use Et_3N as the base and DMF as solvent at 130 °C in the presence of 0.9 mol% of the catalyst as the optimal condition in further studies. To find out if the *in situ* formation of PdCl₂/K 5 in the reaction vessel would drive the reaction, the process was also conducted under the same reaction conditions, by adding a mixture of iodobenzene and styrene in DMF, to a test tube containing PdCl₂ (0.9 mol%), Kryptofix 5 (0.9 mol%), and Et_3N ; stirring the reaction mixture for 1.5 h produced the corresponding product in 68% yield.

To examine the general scope of this coupling reaction, the catalyst was then applied to other cross-coupling reactions of aryl halides carrying either electron-withdrawing or electron-donating substituents in aromatic rings with various vinylic substrates (styrenes, acrylates, acrylonitrile, and acrylamide) (Table 2). Only *trans* products were obtained selectively in all the cases. The results clearly show that the PdCl₂-K 5 catalytic system is highly active and tolerates a wide range of functional groups. As expected, the reaction of aryl iodides with terminal olefins all proceeded smoothly within 10 min to give the desired products in 71-95% isolated yields (Table 2, entries 1-14). However, the reaction of sterically hindered 2iodotoluene with styrene and methyl acrylate gave lower yields (Table 2, entries 8 and 9). Electron-donating

	C	- +	PdCl ₂ -K 5 (cat.) Solvent, Base 10 min		
Entry	Base	2a Solvent	Temp (°C)	sa Catalyst (mol%)	Yield ^b (%)
1	K ₂ CO ₃	DMF	130	0.9	56
2	Na ₂ CO ₃	DMF	130	0.9	42
3	KO ^t Bu	DMF	130	0.9	33
4	DIPEA	DMF	130	0.9	63
5	Et ₃ N	DMF	130	0.9	95
6	Et ₃ N	DMSO	130	0.9	77
7	Et ₃ N	NMP	130	0.9	64
8	Et ₃ N	DMAc	130	0.9	53
9	Et ₃ N	PhCH ₃	130	0.9	48
10	Et ₃ N	DMF	120	0.9	76
11	Et ₃ N	DMF	110	0.9	51
12	Et ₃ N	DMF	130	0.8	83
13	Et ₃ N	DMF	130	0.7	74
14	Et ₃ N	DMF	130	0.6	65

^aReaction conditions: iodobenzene (1 mmol), styrene (1.1 mmol), base (1.1 mmol), solvent (1 ml), 10 min, under air. ^bIsolated yields.

TABLE 1Screening of the reaction conditions^a

TABLE 2	Palladium(II)) chloride-Kry	otofix 5-catalyz	ed Mizoroki-Heck	reaction of ar	yl halides with olefins ^a
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	Ar-X +	PdCl ₂ -K 5 (cat. DMF, Et ₃ N, 130) °C Ar		
	1	2	3		
Entry	Aryl halide	R	Product	Time	Yield ^b (%)
1	PhI	Ph	3a	10 min	95
2	PhI	$4-MeOC_6H_4$	3b	10 min	81
3	PhI	CN	3c	10 min	91
4	PhI	CONH ₂	3d	10 min	86
5	PhI	CO ₂ Me	3e	10 min	91
6	$4-MeC_6H_4I$	Ph	3f	10 min	90
7	$4-MeC_6H_4I$	CO ₂ Me	3g	10 min	87
8	2-MeC ₆ H ₄ I	Ph	3h	10 min	76
9	2-MeOC ₆ H ₄ I	CO ₂ Me	3i	10 min	71
10	$2-MeC_6H_4I$	CO ₂ Me	3j	10 min	82
11	1-iodonaphthalene	Ph	3k	10 min	86
12	4-MeCOC ₆ H ₄ I	CO ₂ Me	31	10 min	94
13	4-BrC ₆ H ₄ I	Ph	3m	10 min	81
14	2-iodothiophene	CO ₂ Bu	3n	10 min	91
15	PhBr	Ph	3a	24 h	16
16	PhBr	Ph	3a ^c	10 min	70
17	PhBr	Ph	3a ^d	10 min	74
18	4-MeOC ₆ H ₄ Br	Ph	3b ^c	10 min	74
19	4-MeCOC ₆ H ₄ Br	Ph	30 ^c	10 min	90
20	4-BrC ₆ H ₄ CHO	Ph	3p ^c	10 min	87
21	$4-BrC_6H_4NO_2$	Ph	3q ^c	10 min	91
22	4-BrC ₆ H ₄ CN	Ph	3r ^c	10 min	88
23	PhCl	Ph	3a	36 h	8
24	PhCl	Ph	3a ^c	6 h	45
25	PhCl	Ph	3a ^c	24 h	49
26	4-MeCOC ₆ H ₄ Cl	Ph	30 ^c	4 h	54
27	4-ClC ₆ H ₄ CHO	CO ₂ Me	3s ^c	1 h	51
28	4-ClC ₆ H ₄ CN	Ph	3r ^c	2 h	58

^aReaction conditions: aryl halide (1 mmol), olefin (1.1 mmol), Et₃N (1.1 mmol), catalyst (0.9 mol%), in DMF (1 ml) at 130 °C.

^bIsolated yields.

 $^{\mathrm{c}}\mathrm{TBAB}$ (1 mmol) and catalyst (0.9 mol%) were used.

^dTBAB (1 mmol) and catalyst (1.5 mol%) were used.

substituent (4-methoxy) on styrene gave lower yield when compared to that of styrene (Table 2, entries 1 and 2). It was also observed that for a particular olefin, activated electron-deficient aryl iodide (4-iodo acetophenone) could effectively couple with the olefin (methyl acrylate) providing the corresponding product in excellent yield after 10 min (Table 2, entry 12). The non-activated electronneutral iodobenzene and deactivated electron-rich substrates (4-iodotoluene and 4-iodoanisol) gave lower yields when coupling with methyl acrylate (91, 87, and 82%, respectively) (Table 2, entries 5, 7, and 10).

Chemoselectivity of the procedure was also studied using 4-bromo-1-iodobenzene (Table 2, entry 13). In this reaction, iodine acted as a better leaving group. Under the same reaction conditions, coupling of bromobenzene and chlorobenzene with styrene gave very poor yields (16% and 8%, respectively) after 24 h and 36 h (Table 2, entries 15 and 23). Changing the amount of the catalyst from 0.9 mol% to 1.5 mol% in the case of bromobenzene, or increasing reaction time to 24 h in the case of chlorobenzene had little effect on the product yields (Table 2, entries 17 and 25); therefore, tetra-nbutylammonium bromide (TBAB, 1 equiv) was used as an additive for the cross-coupling of aryl bromides or chlorides with olefins. Several aryl bromides, possessing electron-rich (deactivated) or electrondeficient (activated) substituents, worked well with styrene to give the corresponding stilbenes. The reactivity of aryl bromides with electron-deficient substituent was higher than that of aryl bromides with electron-rich substituent (Table 2, entries 18-22). Compared with the corresponding bromo analogues, the reaction of the chloro derivatives gave lower yields and required longer times (Table 2, entries 23-28).

3 | CONCLUSION

In conclusion, Pd(II) complex bearing Kryptofix 5 ligand has been synthesized with a simple method and characterized by various methods. Furthermore, the $PdCl_2-K$ 5 complex was found to be highly efficient homogeneous catalyst for the Mizoroki-Heck reactions of aryl halides with various terminal olefins. This new catalyst has the advantages of being thermally stable, readily prepared, inexpensive, easily handled, and operating under air. In addition, Kryptofix 5 might act as a ligand to stabilizing the Pd(0) species in the Mizoroki-Heck reaction.

4 | EXPERIMENTAL

All chemicals were purchased from Merck, Sigma and Acros chemical companies. Melting points were determined on a Büchi melting point B-540 apparatus. FT-IR spectra were recorded on an ABB Bomem model FTLA 2000 spectrophotometer. Nuclear magnetic resonance (NMR) spectra were recorded at 300 (^{1}H) and 75.4 (^{13}C) MHz, on a commercial Bruker AQS-300 instrument using CDCl₃ as solvent. The ICP analyses were carried out on a Varian vista-MPX instrument. Elemental analyses for C, H and N were performed using a Perkin Elmer 2400 series Rapid analyzer. The EDX for the elements' valuation was performed using Vega, TESCAN-Model instrument. UV-VIS spectra of the ligand and complex were carried out (10⁻⁵ M in DCM) using a 4802 UNICO UV/VIS double beam spectrophotometer. Raman spectrum of the complex was recorded on a Thermo Nicolet Almega Dispersive Raman Spectrometer with 2nd harmonic @532 nm of a Nd:YLF laser.

4.1 | Preparation of PdCl₂-K 5 complex

A RB-flask was charged with Kryptofix 5 (0.137 g, 0.31 mmol), PdCl₂ (0.054 g, 0.31 mmol), and CH₃CN (10 ml). The mixture was refluxed for 2 h, during which time the color of the mixture was turned from colorless to brown, and all the materials dissolved thoroughly. Then, the solvent was removed under reduced pressure, and to the resulting residue was added dichloromethane (25 ml), and filtered; diethyl ether (25 ml) was added to the filterate, and a light yellow precipitate formed. The precipitate was washed with Et₂O and air-dried to give the PdCl₂-K 5 complex (140 mg, 73 %). Mp 138-140 °C. ¹H NMR (300 MHz, CDCl₃): δ 9.96-10.09 (m, 2H), 8.00-8.19 (m, 2H), 7.01-7.61 (m, 8H), 3.52-5.64 (m, 16H); ¹³C NMR (75 MHz, CDCl₃): δ 155.0, 151.9, 139.1, 138.3, 131.0, 127.9, 121.7, 121.2, 112.9, 70.6, 70.3, 69.9. Anal. Calcd. for C₂₆H₂₈N₂O₅Pd.H₂O: C, 48.50; H, 4.69; N, 4.35. Found: C, 48.82; H, 4.61; N, 4.44.

4.2 | General procedure for the Mizoroki-Heck coupling reaction

A test tube was charged with aryl halide (1.0 mmol), olefin (1.1 mmol), PdCl₂-K 5 (5.6 mg, 0.9 mol%), Et₃N (1.1 mmol), DMF (1 ml). [For aryl bromides or chlorides, TBAB (1.0 mmol) was also added]. The mixture was then stirred at 130 °C under air for appropriate time. The progress of the reaction was monitored using TLC. Upon completion of the reaction, the mixture was cooled to room temperature, poured into H₂O (12 ml), and extracted with CH₂Cl₂ (3×8 ml). The combined organic extracts were dried (Na₂SO₄) and concentrated in vacuo to give the crude product, which was further purified by prep. TLC [silica gel; *n*-hexane or *n*-hexane-EtOAc (9:1)] to afford the desired pure product. The products were characterized using FT-IR, 1H- and 13C NMR spectroscopies (see supporting information).

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ORCID

Barahman Movassagh D http://orcid.org/0000-0001-8489-9962

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SUPPORTING INFORMATION

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