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The Ionic Palladium Porphyrin as a Highly Efficient and Recyclable Catalyst for Heck Reaction in Ionic Liquid Solution Under Aerobic Conditions

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Abstract The ionic palladium porphyrin featured with pyridinium tags at four *meso*-positions, palladium tetrakis-(*N*-methyl-4-pyridinium)-porphyrin iodide ([Pd(II)TM-Py)P][I]$_4$ (**3**), embedded in the similar structured ionic liquid of *N*-butyl pyridinium tetrafluoroborate ([Bpy]BF$_4$) was found to be a highly efficient and recyclable catalytic system for the Heck cross-coupling of iodobenzene (derivatives) and ethyl acylate, with high turnover frequency of 56,000 h^{-1} under aerobic conditions. The active charge transfer transition occurred in **3**-[Bpy]BF$_4$ system during the Heck reaction could account for the efficiency in the catalytic coupling, which was observed in the UV–visible spectra.

Keywords Palladium porphyrin · Heck reactions · Ionic liquids · UV–visible spectrophotometer

1 Introduction

The Heck reaction, a palladium-catalyzed carbon–carbon bond formation between aryl halides and olefins, is one of the most important tools for the synthesis of elaborated styrene derivatives due to its wide variety of functional groups on both reactants [1, 2]. In the past decades, the considerable progresses have been made in palladium-catalyzed Heck reactions. [3–5] The development of homogeneous/heterogeneous catalysts led to a variety of different ligand systems, among which phosphine-containing palladacycles [6–9], bulky and electron-rich monodentate phosphine [10], and carbene donors like *N*-heterocyclic carbenes (NHCs) which are strong σ-donors with negligible π-accepting ability [11–15], are exemplarily attractive owing to the high activities of the derived Pd catalysts. The N-containing ligands recently became preferable in considerations of non-toxicity and insensitivity to oxygen/moisture, having the advantage of performing the reactions under aerobic conditions; however the rapid deactivation of the derived palladium catalysts in the recycling use was still a frustrated problem [16–19]. Recently, the phosphine-free systems of the CNC-pincer palladium complexes have been found to be the highly efficient and recyclable catalysts for the Suzuki reaction [20].

Porphyrin free bases are important *N*-pincer ligands to which most of the metal elements in the periodic table can coordinate. Palladium porphyrins have found applications as luminescent markers [21], oxygen sensor [22], sensitizers for singlet oxygen formation [23], and photo-induced protein cross-linking agents [24], due to their high yield of intersystem crossing and long lifetime of the resulting triplet state in diverse media [25, 26]. Anyway, the examples of palladium porphyrins as phosphine-free catalysts in C–C coupling reactions were few, except for the first case in the Suzuki reaction reported very recently [27].

In contrast to the common N-ligand, the porphyrin base as a tetradentate pincer ligand can provide more stable Pd complexes suitable for Heck reactions and thereby avoid the use of exogenous ligands and high loadings of palladium. However, the conventional palladium porphyrins give poor solubility even in polar organic solvents (DMF, DMSO) limited their use in homogeneous catalysis, which is a highly efficient process but with disadvantage of difficult separation from the catalyst with the reaction mixture

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for reuse. In recent years, the use of ionic liquids is of much interest topic in homogeneous catalysis. Ionic liquids (ILs) can dissolve many organic compounds and metal complexes but they are in turn immiscible in some apolar organic solvents such as alkanes or ethers, able to develop a liquid system in which the reaction is carried out in the ionic liquid media, and the reaction products are removed by simple liquid–liquid extraction using alkanes or ether for the catalyst reuse. In order to increase the compatibility of the metal complexes (catalysts) with the IL and avoid the metal catalysts leaching out of the IL phase, the efforts have been made to enhance the ionopholicity of the metal catalysts through incorporating imidazolium or pyridinium tags into them [28, 29].

Highlighted by the good coordinating ability of the porphyrin as a tetradentate ligand with characters of non-toxicity and insensitivity to moisture/oxygen, and the advantages of ILs as the ideal reaction media, an ionic palladium porphyrin featured with four pyridinium tags, palladium tetrakis-(*N*-methyl-4-pyridinium)-porphyrin iodide ([Pd(II)TMPy)P]][I]$_4$ (**3**), was synthesized and applied as the catalyst in the Heck reaction of ethyl acrylate with aryl iodides under aerobic conditions, along with the IL of *N*-butyl pyridinium tetrafluoroborate ([Bpy]BF$_4$) as the solvent in consideration of the matched ionophilicity and compatibility. The behavior of the palladium catalyst (**3**) in [Bpy]BF$_4$ was investigated by a UV–visible technique to elucidate the catalytic performance of **3**. To our knowledge, this is the first case to use the ionic palladium porphyrin in the IL solution to catalyze the Heck reaction.

2 Experimental

2.1 Materials and Methods

Pyrrole was purified by re-distillation before use. Pyridine-4-carbaldehyde, methyl iodide (CH_3I), and the other reagents/solvents were of commercial grade and used as received. The ^{1}H NMR (500 MHz) spectra were recorded on a Bruker Avance 500 spectrometer. The IR spectra were recorded on a Nicolet NEXUS 670 spectrometer. The elemental analyses were performed by the Analytical Center in ECNU (Elementar Vario EL III). GC analyses were performed on a SHIMADZU-2014 chromatography equipped with Rtx-wax capillary column (30 m × 0.25 mm). GC-MS analyses were recorded on an Agilent 6890 instrument equipped with Agilent 5973 mass selective detector.

The UV–visible spectra were recorded on a SHIMADZU-UV 2550 spectrophotometer at ambient temperature (ca. 25 °C). The spectral resolution was about 1 nm. Upon completion, the left IL phase (0.05 mL) containing **3** and the formed salt of $Et_3N{\cdot}HI$ after extraction by diethyl ether was taken out and then diluted with 2 mL [Bpy]BF$_4$ (or other solvent) into a UV cuvette (3 mL) by single mixing. The reference sample is the same as the solvent used for the test sample.

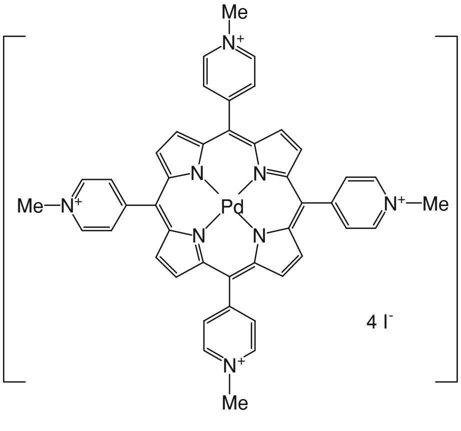
2.2 Synthesis

2.2.1 Palladium Tetrakis-(N-Methyl-4-Pyridinium)-Porphyrin iodide ([Pd(II)TMPy)P][I]$_4$ (3)

The neutral porphyrin free base of tetrakis(4-pyridyl)-porphyrin ([H$_2$TPyP], **1**) was prepared according to the reported work [30]. The ionic porphyrin free base of tetrakis(*N*-methyl-4-pyridinium)-porphyrin iodide ([H$_2$TMPyP][I]$_4$, **2**) was prepared according to our reported work [31]. The obtained **2** (0.5 mmol) and $PdCl_2$ (0.6 mmol) was added into 20 mL deionized water. The resultant mixture was refluxed until the absorbance peak at 425 nm (Soret band of **2**) blue-shifted to 416 nm (Soret band of **3**) without any change afterwards. The obtained black solids were washed with hot water thoroughly and ethanol to give **3** (Scheme 1) after dryness in vacuo, with yield of 89 mol%. ^{1}H NMR (500 MHz, DMSO-d_6, ppm): δ_H 9.5 (d, $J = 5$ Hz, 8H, pyridinium, C*H*N$^+$C*H*), 9.1 (s, 8H, pyrrolyl, *β-H*), 9.0 (d, $J = 5$, 8H, Hz, pyridinium, C*H*CC*H*). UV–vis (**3** in [Bpy]BF$_4$): $\lambda_{max} = 416$ (s, Soret band), 525 (w, Q band), 560 (Q band) nm.

2.3 General Procedures for the Heck Reaction and Recycling of the Catalyst

In a typical example, a solution of iodobenzene (5 mmol) with ethyl acrylate (6.0 mmol) in [Bpy]BF$_4$ (2 mL) was mixed with **3** and triethylamine (Et_3N, 7.5 mmol). The mixture sealed in the glass vials reacted at 100 °C for 1 h on an Advantage Series^{TM} 2410 Personal Screening



[Pd(II)TMPyP][I]$_4$ (**3**)

Scheme 1 The structure of the ionic palladium porphyrin (**3**)

Synthesizer (Argonaut Technologies Inc.). Upon completion, the reaction mixture was cooled to room temperature and then extracted with diethyl ether (2.5 mL × 3). The ether fractions were combined, and then analyzed by GC to determine the conversions (1-dodecane as internal standard) and the selectivities (normalization method). The structures of obtained products were further confirmed by GC-Mass. The remaining IL phase containing **3** and the formed salt of $Et_3N{\cdot}HI$ was dried in vacuo at room temperature and then used directly without further treatment for the next run. Due to the stoichiometric consumption of the base, Et_3N (7.5 mmol) was added additionally per pass.

All manipulations were conducted in air.

3 Results and Discussion

In UV-visible spectra (Fig. 1), the characteristic Soret bands of the free base **2** is observed at 425 nm along with 4 Q-bands at 517, 550, 590, 644 nm. As for the palladium porphyrin **3**, its Soret band blue-shifts to 416 nm. It was also noted that after Pd insertion, the spectrum pattern changes from a four Q-band spectrum, indicating D_{2h} symmetry for **2**, to a two Q-band spectrum (525, 560 nm), indicating D_{4h} symmetry, which is characteristic of porphyrin coordinated to a metal ion through the four *N*-heteronuclei [32]. The ^1H NMR spectra of **2** and **3** are nearly the same except for the high-field resonance signal at −3.08 ppm for N–H units in **2**.

3.1 Catalytic Performance of 3 in Heck Reaction

To evaluate the catalytic performance of **3** in Heck reaction, the coupling reaction of iodobenzene with ethyl acrylate has been chosen as a model reaction. The investigations on the solvents ($[Bmim]BF_4$, $[Bmim]PF_6$, $[Bpy]BF_4$, DMF) indicated that the activity of the fresh **3** was mostly favored by using $[Bpy]BF_4$ as a solvent. Under the mild reaction conditions (100 °C, 1 h), the coupling product of *trans*-ethyl cinnamate was obtained in excellent yield (100%, Table 1, No. 1). As shown in Table 2, even at the concentration of 0.00125 mol%, 70% yield of the coupling product was obtained with high TOF of 56,000 h^{-1} in **3**-$[Bpy]BF_4$ system (No. 5). Anyway, compared to $PdCl_2$, the mixture of the equivalent $PdCl_2$ and porphyrin free base **2** (or **1**) did not show obviously improved effect on the catalytic activity, implying that the palladium porphyrin complexes derived from the ligation of Pd(II) ion to the porphyrin free bases could not be formed in situ under the conditions of the coupling reactions in Table 2. Especially, for the mixture of $PdCl_2$-**1**, it was impossible to insert Pd(II) ion into **1** even after refluxing in PhCN for 48 h.

The recovery and reuse of catalysts are important in the practice of Heck reactions from an economic point of view

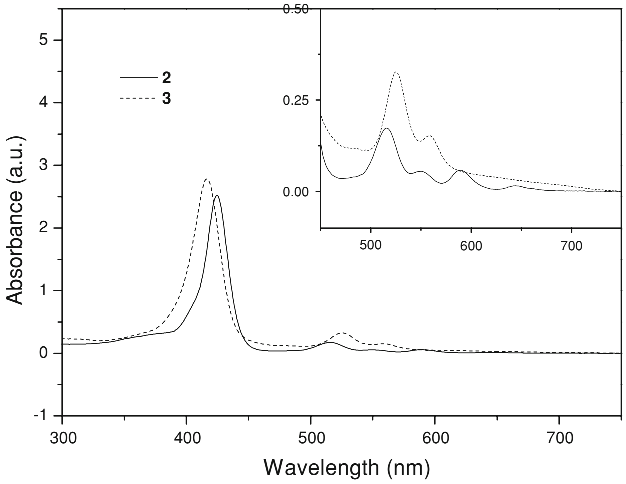


Fig. 1 The UV–visible spectra of free base **2** and ionic palladium porphyrin **3**

Table 1 Effects of the solvents on the catalytic performance of **3** for the Heck reaction

No.	Sol.	Conv. (%)	Sel. (%)[a]
1	$[BPy]BF_4$	100	100
2	$[BMIM]PF_6$	96	100
3	$[BMIM]BF_4$	74	100
4	DMF	94	100

3 0.02 mol%, Et_3N 7.5 mmol; PhI 5 mmol; ethyl acrylate 6 mmol; solvent 2 mL; reaction time 1 h, reaction temperature 100 °C

[a] To *trans*-ethyl cinnamate

Table 2 Comparison of the catalytic performance of **3**, $PdCl_2$-**2**, and $PdCl_2$-**1** for the Heck reaction

No.	Cat.	Conc. of Pd (mol%)	Conv. (%)	Sel. (%)[a]	TOF (h^{-1})
1	**3**	0.0200	100	100	5,000
2	**3**	0.0100	100	100	10,000
3	**3**	0.0050	98	100	19,600
4	**3**	0.0025	94	100	37,200
5	**3**	0.00125	70	100	56,000
6	$PdCl_2$-**2**[b]	0.0050	58	100	13,600
7	$PdCl_2$-**1**[b]	0.0050	57	100	12,000
8	$PdCl_2$	0.0050	54	100	12,200

PhI 5 mmol, ethyl acrylate 6 mmol, Et_3N 7.5 mmol, $[Bpy]BF_4$ 2 mL, temperature 100 °C, time 1 h

[a] To *trans*-ethyl cinnamate

[b] Free base **2** (or **1**) and $PdCl_2$ were mixed and used directly for the reaction

Table 3 The recycling use of **3** for the Heck reaction in [Bpy]BF_4

Run	1	2	3	4	5	6	7
Conv./Sel.[a]	94/100	98/100	94/99	90/99	87/100	84/99	81/99

3 0.0025 mol% (0.125 μmol), PhI 5 mmol; ethyl acrylate 6 mmol, Et_3N 7.5 mmol, [Bpy]BF_4 2 mL, reaction time 1 h, temperature 100 °C

[a] To *trans*-ethyl cinnamate

due to high cost of palladium compounds and ligands, as well as for preventing contamination of final products by toxic metals. The recycling uses of **3** in [Bpy]BF_4 were examined in Table 3. At concentration of 0.0025 mol%, **3** exhibited very good activity and stability even after 7 runs without any precipitation of Pd black. Anyway, the cumulative slurry ammonium salts ($Et_3N{\cdot}HI$) showed degraded impacted on the activity of **3**, mainly due to the mass transfer limitation. In each run, the pure product of ethyl cinnamate was obtained in good yield by extracting with diethyl ether and concentrating under vacuum. The ICP (inductive coupled plasma emission spectrometer) analysis indicated that the leaching of Pd into organic phase was below the detection limit (<0.1 μg/g). Due to the porphyrin ligand **2** is non-sensitive to oxygen and moisture, all the manipulations for performing the reaction, separating the product from the IL phase, and recycling the IL mixture, could be handled without N_2 protection, which facilitated the workup greatly.

3.2 UV–Visible Analysis of 3 in the Different Solvents after Used in the Heck Reaction

In order to understand the behaviors of **3** in different solvents in Table 1, the resultant mixtures after performing the reaction were analyzed by the UV–visible spectrophotometer. Figure 2 showed that, in the solvents of [Bmim]BF_4, [Bmim]PF_4, or DMF, the characteristic absorbance of **3** was observed unexceptionally. Surprisingly in [Bpy]BF_4, the typical Soret band at ca. 416 nm of **3** was completely overwhelmed by an intensive absorbance at ca. 350–450 nm.

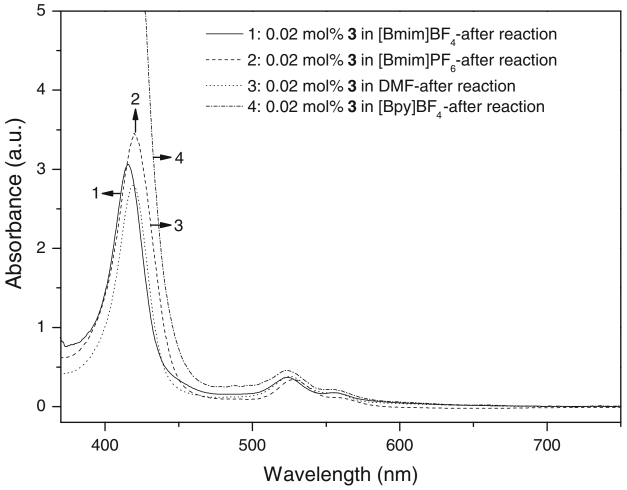


Fig. 2 The UV–visible spectra of **3** in different solvents after 1st used in the Heck reaction as presented in Table 1 [0.05 mL remaining mixture after ether extraction was diluted into 2 mL corresponding solvent]

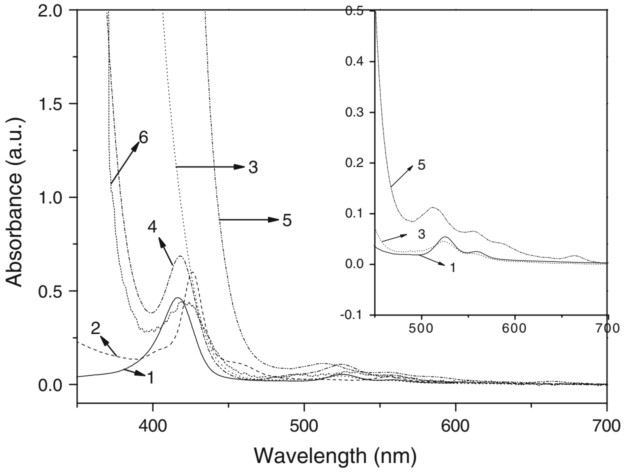


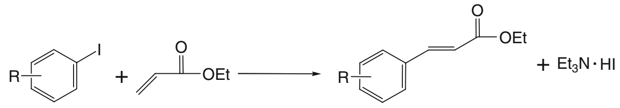
Fig. 3 The UV–vis spectra recorded at room temperature (25 °C) for **3** in [Bpy]BF_4 under different conditions. (1) Fresh **3** in [Bpy]BF_4 before used in the Heck reaction. (2) The IL mixture of **3** in [Bpy]BF_4 treated with ethyl acrylate and Et_3N at 100 °C for 1 h, dissolved in [Bpy]BF_4. (3) the IL mixture of **3** in [Bpy]BF_4 after 1st use (Table 3, Run 1), dissolved in [Bpy]BF_4. (4) The IL mixture of **3** in [Bpy]BF_4 after 1st use (Table 3, Run 1), dissolved in H_2O. (5) The IL mixture of **3** in [Bpy]BF_4 after 7th use (Table 3, Run 7), dissolved in [Bpy]BF_4. (6) The IL mixture of **3** in [Bpy]BF_4 after 7th use (Table 3, Run 7), dissolved in H_2O [0.05 mL remaining IL mixture after ether extraction was added into a UV cuvette (3 mL) which was dissolved in 2 mL [Bpy]BF_4 (or H_2O) with the final concentration of **3** at ca. 6×10^{-6} M]

Highlighted by the appearance of the intensive absorbance at ca. 350–450 nm, the detailed investigations on **3**-[Bpy]BF_4 system for the Heck reaction was carried out as shown in Fig. 3. Comparatively, the fresh **3** in [Bpy]BF_4, without performing the Heck reaction, exhibited the typical Soret band and Q bands (line 1). Even when **3** in [Bpy]BF_4 was treated with ethyl acrylate and Et_3N at 100 °C for 1 h, its characteristic Soret and Q bands were observed normally (line 2), but with the slight red-shift Soret band compared to that in line 1. Only when **3** in [Bpy]BF_4 was treated with PhI, Et_3N, and ethyl acrylate at 100 °C for 1 h to proceed the Heck reaction, did the intensive absorbance at 350–450 nm appear, which completely overlaid the characteristic Soret band of **3** similar to line 4 in Fig. 2. At the first sight of such intensive absorbance, it was supposed that a new palladium complex probably was formed derived from **3**-[Bpy]BF_4 system during the Heck reaction. However, the further analysis on **3**-[Bpy]BF_4 system indicated that the intensive absorbance at ca. 350–450 nm could not be ascribed to a new palladium complex, but still responsible to the palladium porphyrin **3** or its derivative. Firstly, the characteristic spectrum pattern of the two Q-bands at 525 and 560 nm revealed that the palladium porphyrin **3** remained intact after the first use in the Heck reaction in [Bpy]BF_4 (line 3). Secondly, when water was used to dilute the mixture of **3**-[Bpy]BF_4 corresponding to line 3 in Fig. 3, the intensive absorbance at 350–450 nm was subsided, and then the Soret band of **3** at 418 nm was observed clearly (line 4), confirmatively indicating the intactness of **3** after the Heck reaction. Similarly, even after the 7th use of **3** in [Bpy]BF_4, the typical Q bands of **3** were clearly observed (line 5), but with indication of more intensive band at 350–450 nm, from which the Soret peak (420 nm) could be released again after treated with water (line 6).

As can been seen in Figs. 2 and 3, besides the typical Pd-porphyrin Soret and Q absorbances, **3** dissolved in [Bpy]BF_4 after performing the Heck reaction shows an unique strong and broad absorbance band in the region of 350–450 nm, which was tentatively contributed to a charge transfer (CT) transition originated from the interaction between the palladium porphyrin and [Bpy]BF_4 according to the reported work [33–39]. Although the CT transition had been universally observed in metalloporphyrin catalyzed oxidations and metal-ligand coordination compounds [38, 39], it was firstly observed in palladium porphyrin catalyzed Heck reaction herein. The formation of this low-lying and long-lived CT complex could be dramatically affected by "environmental tuning" [37] like the polar solvent of [Bpy]BF_4, due to the strong cation-π/π-π interaction [40, 41], which was favored by the structural similarity and preferential symmetry between $[Bpy]^+$ cations and **3**. The active electron transfer between the palladium porphyrin and [Bpy]BF_4 through orbital mixing resulted in the corresponding CT transition [37]. Whereas, with replacement of [Bpy]BF_4 by [Bmim]PF_6, [Bmim]BF_4 or MDF as the solvent of **3**, the CT transition with intensive absorbance at ca. 350–450 nm could not be observed (Fig. 2, line 2). Anyway, the built-up cation-π/π-π interaction between [Bpy]BF_4 and **3** could be collapsed by the intrusion of the third water molecule [42], and then the corresponding intensive CT transition was dramatically suppressed due to the destruction of the CT complex, as shown in Fig. 3 (lines 4, 6) after treatment by water.

As we know, the essence of the redox process is the intermolecular or intramolecular electron transfer. The essence of CT transition is characterized by the electron transfer [37]. From this point of view, the active CT transition occurred in the system of **3**-[Bpy]BF_4 during the Heck reaction is correlated to the rapid redox process happened on the palladium porphyrin [Pd(II)↔Pd(0)] with the super-conjugated and electron-reservoir characters, which could account for the catalytic efficiency for the Heck reaction [43].

Table 4 The generality of **3** for different iodobenzene derivatives in the Heck reaction



No.	R	Conv. (%)	Sel. (%)[a]
1	*p*-CH_3	100	99
2	*m*-CH_3	87	99
3	*o*-CH_3	40	100
4	*p*-OCH_3	100	100
5	*m*-OCH_3	100	100
6	*o*-OCH_3	71	100
7	*p*-NO_2	100	100
8	*m*-NO_2	100	100
9	*o*-NO_2	–	–
10	*p*-CF_3	100	99
11	*m*-CF_3	100	100
12	*o*-CF_3	43	100

3 0.0025 mol%; substrate 5 mmol; Et_3N 7.5 mmol; ethyl acrylate 6 mmol; [Bpy]BF_4 2 mL; temperature 100 °C, reaction time 2 h

[a] To *trans*-coupling product which was confirmed by GC-Mass analysis

3.3 Generality of 3-[Bpy]BF_4 for Heck Reaction

The scope of the Heck reaction was investigated on different iodobenzene derivatives with different electronic and steric effects. It was indicated that, due to the high reactivity of the aryl iodides, the *trans*-cross-coupling products were all obtained in high yields (86–100%) under mild conditions (100 °C, 2 h) without obvious discrimination of the electronic nature of the substituents. The steric effect could only be observed when the *o*-substituting group involved (Nos. 3, 6, 12). However, with the activated *o*-nitroiodobenzene no reaction took place (No. 9). As to the activation of aryl bromides, no acceptable conversions were obtained under low concentration of **3** and the mild conditions (100 °C, 2 h) (Table 4).

4 Conclusions

In conclusion, the Heck reaction of iodobenezen (derivatives) and ethyl acrylate could be successfully catalyzed by ionic palladium porphyrin **3** feature with four pyridinium tags, along with the similar structured IL of [Bpy]BF_4 as the solvent under aerobic conditions. The combination of ionic porphyrin **3** with the IL of [Bpy]BF_4 not only gave rise to the high activity, but also endow the homogeneous catalyst **3** with recyclability. The formation of the CT complex in **3**-[Bpy]BF_4 system during the Heck reaction was favorably allowed due to the compatible ionopholicity, symmetry, and the strong cation-π/π-π interaction, which could account for the high catalytic efficiency, and was firstly observed in this study.

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