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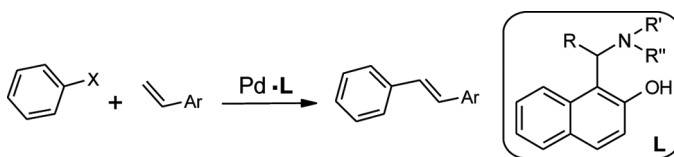
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APPLICATION OF 1-(α -AMINOBENZYL)-2-NAPHTHOLS AS AIR-STABLE LIGANDS FOR Pd-CATALYZED MIZOROKI–HECK COUPLING REACTION

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GRAPHICAL ABSTRACT



Abstract Air-stable, easily accessible Mannich bases, 1-(α -aminobenzyl)-2-naphthols, are used as ligands in palladium-catalyzed Mizoroki–Heck reaction on a variety of substrates. High turnover numbers are observed for both the reactions with aryl bromides and iodides, while aryl chlorides are inert.

Keywords 1-(α -Aminobenzyl)-2-naphthols; Mizoroki–Heck; phosphine-free ligands

INTRODUCTION

Palladium-catalyzed attachment of vinyl moiety to aryl halides, often referred as the Mizoroki–Heck reaction, is an extremely useful and routinely employed method of formation of carbon–carbon bonds.^[1,2] This exceptional reaction has been topic of extensive research in different laboratories of academic or industrial chemistry, and the results are accessible in a number of reviews.^[3–5] Most commonly, an appropriate aryl halide is treated with Pd catalyst, preferably as a complex with a suitable ligand,^[6–8] an olefin, and an appropriate base to furnish a substituted new alkene.

Most of the examples of Mizoroki–Heck reactions are usually carried out in the presence of phosphine ligands in an inert atmosphere. However, phosphine ligands are either expensive or difficult to prepare, are often toxic, and are air sensitive. For this reason, there is a need to find a phosphine-free and inexpensive ligand for these important reactions. In particular, there are some *N,N*-ligands reported in

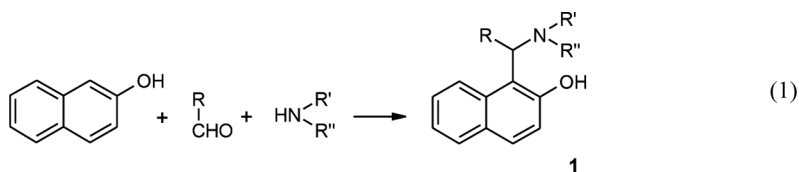
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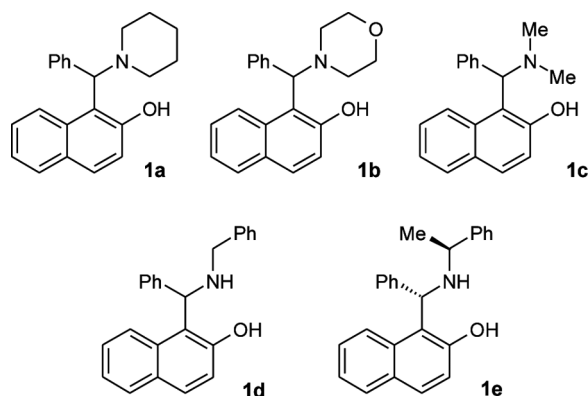
the recent literature, and these have shown varying degrees of activity as palladium catalyst system in the two reactions.^[9–14] There is a further scope to find more effective combination of simple *N,N*-ligands and palladium catalyst for this important reaction. In this communication, we present our findings where easily accessible 1-(α -aminobenzyl)-2-naphthols **1** were used as efficient phosphine-free ligands for this palladium-catalyzed reaction.

RESULTS AND DISCUSSION

Condensation of 1° or 2° amines with aldehydes and 2-naphthols similar to the classical Mannich reaction gives 1-(α -aminobenzyl)-2-naphthols **1** in good yields.^[15,16] The so-called Betti base is formed when ammonia (as the amine) is condensed with 2-naphthol and benzaldehyde.^[17,18] Some of the molecules with structures similar to **1** have been studied from different aspects and results are reported in the literature.^[19–21]



Compound **1** has an attractive arrangement of two heteroatoms (N and O) at a suitable distance to form a six-member stable chelate with metal ions, a prerequisite for applications as ligand in metal-catalyzed reactions, and few of them are available in the literature.^[22–27] In the present communication, we present our results when such ligands are used for palladium-catalyzed Heck reaction. A small group of these ligands **1a** to **1c** with 2° amines and **1d** and **1e** with 1° amines are prepared for this study; see Scheme 1.



Scheme 1. List of ligands prepared and scanned.

Table 1. Search for suitable conditions for Heck reaction with 1-(α -aminobenzyl)-2-naphthols **1** as ligand

No.	ArX	Ligand/Pd(OAc) ₂ (mol% ratio)	Solvent	Yield (%) of 4 (TON) ^c
1	2a	1a (1.2/1.0) ^a	DMA	No reaction [—]
2	2b	1a (0.12/0.10)	DMA	60 [597]
3	2c	1a (0.55/0.50)	DMA	96 [194]
4	2c	1b (0.55/0.50)	DMA	93 [187]
5	2c	1c (0.55/0.50)	DMA	87 [175]
6	2c	1d (0.55/0.50)	DMA	84 [170]
7	2c	1e (0.55/0.50)	DMA	86 [173]
8	2c	1a (0.55/0.50)	DMF	89 [180]
9	2c	1a (0.55/0.50)	NMP	69 [139]
10	2c	1a (0.12/0.10)	DMA	97 [975]
11	2c	1a (0.012/0.010)	DMA	79 [7890]
12	2c	1a (0.006/0.005)	DMA	68 [13594]
13	2c	1a (0.12/0.10) ^b	DMA	89 [889]
14	2c	1a (0.55/0.25)	DMA	97 [390]

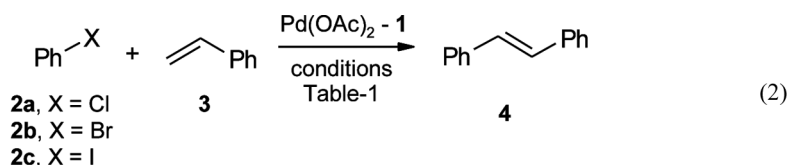
Note. Compound **4** (Ar & Ar' = Ph). All reactions run with K₂CO₃ (2.5 eq.) at 140 °C for 40 h, except entry 13.

^aWith TBAB (0.25 eq.).

^bWith NEt₃ (2.5 eq.).

^cTON—turn over number.

The series of ligands we are then tested for standard Heck conditions, and the results are presented in Table 1:



The present ligands prepared either from 1° or 2° amines are very effective for the Heck reaction with either K₂CO₃ or with organic amine Et₃N, and the stilbene is obtained almost as a single *trans* isomer. As expected, chlorobenzene **2a** was inert even in the presence of tetrabutylammonium bromide (TBAB) as additive for this reaction, while iodobenzene **2c** is more reactive than bromobenzene **2b**. We varied the amount of catalyst to as low as 0.005 (mol% Pd), and stilbene was isolated in moderate yield with excellent turnover number.

Solvent study indicated dimethylacetamide (DMA) or dimethylformamide (DMF) to be good solvents, and all the ligands were almost equally efficient in the reaction. The effect of the ligand is ascertained by a separate experiment with relatively less-reactive bromobenzene **2b**. The reaction with ligand **1a** under controlled conditions and without any ligand with Pd(OAc)₂ under identical conditions clearly shows increase in the conversion and yield of *trans*-stilbene in the former (see Fig 1).

A number of different derivatives **5** to **9** of stilbene are prepared by using ligand–Pd ratio (**1a**/Pd(OAc)₂ = 0.12/0.10 mol%) while for **10** a greater ratio of styrene (6 eq.) and catalyst was needed (1.65/1.5 mol%). It is noteworthy that we were able to prepare 1,3,5-tris(styryl)-benzenes **10** in good yields compared to other olefination methods^[28–35] (Scheme 2).

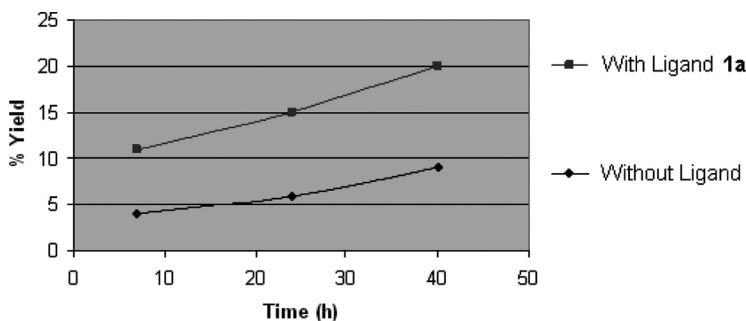
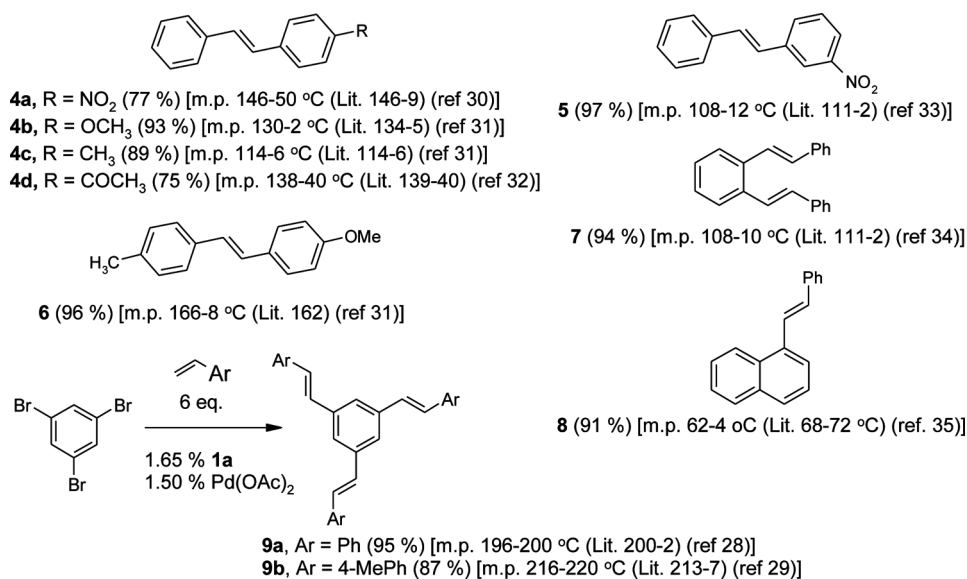


Figure 1. Effect of ligand on the Mizoroki–Heck reaction of **2b**. Pd(OAc)₂ (0.1%), **1a** (0.12%), K₂CO₃ (2.5 eq.), styrene (1.5 eq.) in DMA at 100 °C for the specified time. Yields are isolated ones.



Scheme 2. Examples of Mizoroki–Heck reaction.

The well-established catalytic cycle of the Heck reaction starts with reduction of Pd(II) to Pd(0), often referred to as preactivation, followed by oxidative addition of arylhalide, insertion of olefin, and finally reductive elimination.^[5,6] In the case of phosphine-free reaction conditions, it is possible that the initial reduction of palladium during preactivation is assisted by nucleophilic amine of the ligands rather from alkene.

EXPERIMENTAL

All reactions were carried out using standard bench-top laboratory techniques and using commercial-grade reagents and solvents. Solvents such as DMA, DMF,

and N-methyl-2-pyrrolidone (NMP) were purified according to the standard procedure. ^1H NMR spectra were recorded in chloroform- D_3 at 400 MHz using Bruker Avance-II spectrometer. Infrared (IR) spectra were recorded as KBr pallet on a Perkin Elmer RXI spectrometer. Purification of reaction products was carried out by column chromatography using silica gel (60–120 mesh) using light petroleum ether and ethyl acetate as eluent. Thin-layer chromatography (TLC) was performed on silica gel 60 F₂₅₄ (Merck), and melting points were recorded in capillary tubes heated slowly in paraffin oil and are uncorrected. All the products of Mizoroki–Heck reaction are known compounds, and their structures were established by comparing the mp and mass spectra or NMR spectra with the reported values.

Method A for the Preparation of **1a**

A solution of β -naphthol (1.0 g, 6.93 mmol) and benzaldehyde (0.74 g, 6.93 mmol) was prepared in absolute alcohol (2 mL). Piperidine (0.59 g, 6.93 mmol) was slowly added to this solution, and the mixture was stirred for 48 h at room temperature. The solid was filtered off and washed twice with cold ethyl alcohol (2×3 mL). It was recrystallized from a mixture of petroleum ether and ethyl acetate to get colorless crystals of **1a** (1.02 g, 46%), mp = 195–198 °C (lit.^[16] 198–199 °C).

Method B for the Preparation of **1a**

Alternatively, this mixture of β -naphthol, benzaldehyde, and piperidine (same quantities as A) was stirred at 60 °C for 24 h under a nitrogen atmosphere. The reaction mixture was dispersed at room temperature with ethanol (5 mL). The white solid was separated, collected, and washed with cold ethanol (2×3 mL). The crystalline white residue was purified by recrystallization from a mixture of petroleum ether and ethyl acetate to get **1a** (1.37 g, 62%); mp = 196–198 °C (lit.^[16] 198–199 °C).

IR (KBr) ν : 3459, 3065, 2971, 2939, 1599, 1519, 1491, 1238, 1155, 1035, 717 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ 1.70–2.17 (m, 8H), 2.68 (br s, 1H), 3.34 (br s, 1H), 5.10 (s, 1H), 7.17–7.38 (m, 6H), 7.56 (d, 6.1 Hz, 2H), 7.66 (d, 8.8 Hz, 1H), 7.68–7.77 (dd, 8.08 & 0.8 Hz, 1H), 7.83 (d, 8.6 Hz, 1H), 13.84 (br s, 1H).

Important Analytical Data for Other Ligands

Compound 1b. Prepared by procedure A: 46.4% yield mp = 176–179 °C (lit.^[16] 181–183 °C).

IR (KBr) ν : 3465, 3057, 2971, 2841, 1618, 1593, 1515, 1411, 1315, 1238, 1157, 1028, 869, 824, 746 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ 2.30–2.45 (m, 3H), 3.13 (br s, 1H), 3.6–3.82 (m, 4H), 5.14 (s, 1H), 7.16 (d, 8.84 Hz, 1H), 7.19–7.44 (m, 5H), 7.58 (d, 7.64 Hz, 2H), 7.66–7.71 (m, 2H), 7.84 (d, 8.56 Hz, 1H), 13.07 (s, 1H).

Compound 1c. Prepared by procedure A: 71.4% yield mp = 162–164 °C (lit.^[15] 164–164.5 °C).

IR (KBr) ν 3270, 3057, 2991, 2953, 2858, 1599, 1517, 1409, 1374, 1351, 1238, 1161, 1033, 870, 704 cm^{-1} . ^1H NMR (500 MHz, CDCl_3) δ : 2.34 (br s, 6H), 4.84 (s, 1H), 7.15–7.16 (d, 8.7 Hz, 1H), 7.18–7.28 (m, 4H), 7.34–7.39 (m, 1H), 7.58 (d,

7.5 Hz, 2H), 7.66 (d, 8.9 Hz, 1H), 7.68–7.70 (d, 7.95 Hz, 1H), 7.85 (d, 8.6 Hz, 1H), 13.76 (s, 1H).

Compound 1d. Prepared by procedure B: 30% yield mp = 134–136 °C (lit.^[18] 136–137 °C).

IR (KBr) ν : 3325, 3025, 1619, 1598, 1515, 1413, 1384, 1344, 1270, 1239, 1156, 1073, 1027, 861, 827 cm^{-1} . H NMR (400 MHz, CDCl_3) δ 3.83 (d, 12.9 Hz, 1H), 4.05 (d, 12.9 Hz, 1H), 5.76 (s, 1H), 7.19–7.39 (m, 13H), 7.66–7.69 (d, 8.52 Hz, 1H), 7.73–7.76 (m, 2H), 13.26 (br s, 1H).

Compound 1e. Prepared by procedure B: 81.2% yield mp = 154–156 °C (lit.^[19] 155–156 °C).

IR (KBr) ν 3275, 3059, 3025, 2963, 1622, 1414, 1379, 1270, 1237, 1077, 743, 699 cm^{-1} . H NMR (400 MHz, CDCl_3) δ : 1.5 (d, 6.8 Hz, 3H), 2.28 (br m, 1H), 3.89 (q, 6.8 Hz, 1H), 5.45 (s, 1H), 7.17–7.75 (m, 16H), 13.72 (s, 1H).

Typical Procedure for the Heck Reaction (Table 1, No. 11)

A solution of palladium acetate (0.055 mg, 0.000245 mmol) and ligand **1a** (0.093 mg, 0.000294 mmol) was made in dry dimethylacetamide (2 mL) under the nitrogen atmosphere. This was sonicated for 2–3 min to degas and to make the solution homogeneous. In another two-necked flask, a mixture of iodobenzene (0.5 g, 2.45 mmol), dry potassium carbonate (0.85 g, 6.13 mmol), and dry dimethylacetamide (5 mL) was made and heated under a nitrogen atmosphere. When the temperature attained 60 °C, styrene (0.38 g, 3.7 mmol) was slowly introduced by syringe. The mixture was then heated to 100 °C and the previously prepared catalyst solution was added. The temperature was further raised to 140 °C and continued for another 40 h. The reaction mixture was quenched with water, neutralized using aqueous HCl (6 N, 5 mL), and extracted with dichloromethane (3 \times 25 mL). The combined organic phase was washed with water and dried over anhydrous sodium sulfate. Solvent was removed in vacuum, and the crude product was purified by column chromatography on silica gel to afford *trans*-stilbene (0.35 g, 79%). mp = 120–122 °C.

CONCLUSION

We report novel use of readily available and air-stable 1-(α -aminobenzyl)-2-naphthols as phosphine-free ligands for efficient palladium-catalyzed Mizoroki–Heck reaction of aryl bromides/iodides.

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