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COMMUNICATION

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NCC Pincer Ni (II) Complexes Catalyzed Hydrophosphination of Nitroalkenes with Diphenylphosphine

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An efficient NCC pincer Ni (II)-catalyzed hydrophosphination of nitroalkenes with diphenylphosphine has been developed. Under the optimized conditions, both (hetero)aromatic and aliphatic nitroalkenes were well tolerated, irrespective of electronic effect, to provide the corresponding products in up to 99% yield.

KEYWORDS
Hydrophosphination, NCC pincer Ni (II) complexes, Nitroalkenes

1 | INTRODUCTION

Pincer complexes are important class of well-defined structures supported by tridentate ligands, which exhibits superior performance in catalytic transformations via suitable choice of transition metals as well as fine modifications of steric and electronic effects of ligands.[1] Up to now, a series of NCN, PCP, NNC, PNP, PNN, and other tridentate metal complexes have been successfully synthesized through variations of donor moieties.[2] In special, imidazolines have been recognized as representative nitrogen donor moieties,[3] which have been utilized in Morita-Baylis-Hillman reactions, alkynylation of trifluoropyruvates, alkylations of phthalan, and sequential hydroboration/hydrogenation of internal alkynes.[4] Recently, N-heterocyclic carbenes (NHCs) have also demonstrated superior σ-donating ability to form stable metal-NHC complexes.[5] Consequently, the combination of imidazoline and NHC moieties around metal center enables generation of efficient and stable catalysts with superior catalytic reactivity.

Organophosphorus compounds are versatile building blocks in organic synthesis, pharmaceuticals, and materials science.[6] Meanwhile, they served as important ligands in transition-metal catalyzed and organocatalytic reactions.[7] Moreover, phosphorus moieties could also be utilized as ortho-directing groups to facilitate regioselective C-H activation.[8] Recently, great effort has been made to prepare P-containing compounds,[9] mainly including metal catalyzed cross-coupling of pre-functionalized arenes,[10] C-H functionalization of aromatics and other substrates,[11] and difunctionalization of alkenes[12] and alkynes[13] with various phosphorus precursors. Among these categories, metal-catalyzed hydrophosphination of alkeneand alkynes represents an

efficient methodology for the preparation of functionalized phosphorus products.[14–16] Specially, pincer complexes catalyzed hydrophosphination of electron-deficient alkenes with secondary phosphines has been well established by the group of Duan, Leung, Pullarkat, Zhang, and Song groups.[17–21] Despite the above elegant work, most of the reported systems rely on precious palladium metal. It is thus highly desirable to develop earth-abundant metal catalyzed hydrophosphination.

Our group has been interested in synthesizing novel pincer-type complexes and exploring the corresponding catalytic activity.[17c,21] Recently, we have also developed imidazoline- and imidazopyridine-based pincer complexes, which exhibited good activity in Friedel-Crafts alkylation, Suzuki-Miyaura reaction, transfer hydrogenations, and other applications.[22,23] As the continuation of our previous work, we herein firstly developed NCC pincer Ni (II) complex **2a** catalyzed hydrophosphination of nitroalkenes (Scheme 1).

2 | EXPERIMENTAL

2.1 | Synthesis of complexes Ni (II) complex 2 g

In a 25 ml two-necked Schlenk tube were added NCC ligand precursor (0.25 g, 0.44 mmol), NaOAc (0.36 g, 4.40 mmol), and $NiCl_2$ (0.11 g, 0.77 mmol) in dry DMAc (10 ml). The mixture was refluxed under an Ar atmosphere for 48 hr. After removal of organic solvent, the residue was purified by passing through a short column containing a layer of Celite and a layer of silica with dichloromethane as the eluent. The desired products **2 g** were purified once again by preparative TLC on silica gel plates with CH_2Cl_2/EtOAc = 1/1 as the eluent.

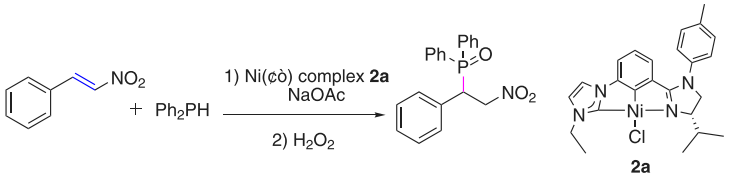
Yield: 17% (0.043 g). $[\alpha]^{20}_D$ = +160° (*c* 0.25, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (d, J = 7.3 Hz, 2H), 7.42–7.27 (m, 7H), 7.21 (d, J = 1.9 Hz, 1H), 7.07 (d, J = 7.7 Hz, 2H), 7.01–6.90 (m, 2H), 6.72 (q, J = 7.5 Hz, 2H), 6.76 (d, J = 1.9 Hz, 1H), 6.27 (dd, J = 6.7, 1.6 Hz, 1H), 5.23 (d, J = 4.4 Hz, 1H), 4.70 (d, J = 4.5 Hz, 1H), 4.67 (m, 1H), 4.46 (m, 1H), 2.33 (s, 3H), 1.39 (t, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.7, 168.3, 144.3, 141.0, 137.8, 136.1, 135.1, 129.9, 129.1, 128.7, 127.3, 127.0, 126.6126.3, 123.2, 121.0, 120.5, 113.1, 110.5, 80.2, 72.1, 44.6, 21.1, 16.8. HRMS (positive ESI): $[M - Cl]^+$ calcd for $C_{33}H_{29}N_4Ni$ 539.1746, found 539.1735.

2.2 | General procedure for the synthesis of nitroalkenes (3a-r)[24]

To a stirred mixture of aldehyde derivatives (10 mmol) and nitromethane (11 mmol) in methanol (5 ml) at 0 °C was added an aqueous solution of sodium hydroxide (5 M, 4 ml) over a period of 30 min. The reaction mixture was stirred at 0 °C to 5 °C for another 30 min. After warmed to room temperature, the reaction was completed in 1–12 hr, which was monitored by TLC. Next, the reaction mixture was mixed with ice water (2 ml) and poured over crushed ice containing concentrated HCl (5 M, 10 ml). The yellow precipitation was filtered, dried in a vacuum desiccator, and crystallized from hot EtOH to give the corresponding niroalkenes **3**.

2.3 | General procedure for Hydrophosphination

To a mixture of KOAc (1.96 mg, 10 μmol) and **2a** (4.5 mg, 10 μmol) in 1,2,3-trichloropropane (2 ml) was added diphenylphosphine **4a** (17.0 μL, 0.10 mmol) and the resulting solution was stirred at −20 °C for 30 min. After addition of trans-β-nitrostyrene **3** (0.15 mmol), the solution was stirred at −20 °C for 12 hr, followed by directly oxidation by H_2O_2 aqueous solution (30%, 40 μL). The reaction mixture was warmed to room temperature and stirred for another 2 hr. Then saturated NaCl aqueous solution (0.15 ml) was added and the solution was extracted with dichloromethane. The organic phase was collected and dried over anhydrous Na_2SO_4. After removal of organic solvent, the residue was purified by silica gel chromatography using CH_2Cl_2/EA = 2/1 as eluent to afford the product **5**.



SCHEME 1 Ni (ll) complex 3a catalyzed hydrophosphination of nitroalkenes

3 | RESULTS AND DISCUSSION

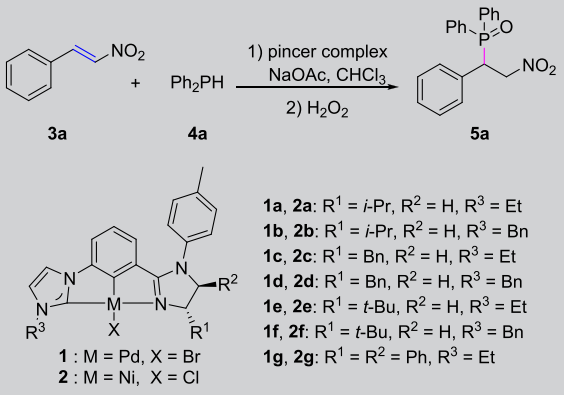
3.1 | Catalytic investigation

Pincer Pd and Ni complexes **1** and **2** were prepared according to previous report by our group.[22a] In addition, complex **2 g** was new compound and fully characterized. Our initial investigation started with hydrophosphination of trans-β-nitrostyrene **3a** with diphenylphosphine **4a** in the presence of Pd and Ni complexes (Table 1). To our delight, the desire product **5a** could be obtained in the range of 65–94% yield when different catalyst was employed. In general, Pd (II) complexes **1a**-**g** demonstrates superior reactivity compared with Ni (II) complexes **2a**-**g**. For Pd (II) complexes, **1b** exhibited the best performance to afford **5a** in 94% yield (Table 1, entry 2), while Ni (II) complex **2a** could also provide **5a** in 91% yield (Table 1, entry 8). Considering the low toxicity and inexpensiveness of Ni complex, catalyst **2a** was chosen for further evaluation. Meanwhile, H_2O_2 was added at the work-up step to obtain an oxidephosphine complex due to the oxygen sensitivity of the phosphine group.

Subsequently, the effect of base, solvent, temperature, and molar ratio was systematically evaluated (Table 2). Initially, various base, such as *t*-BuONa, KOAc, NaOAc, and Na_2CO_3, were employed, indicating NaOAc is the best choice (Table 2, entries 1–4). Either increasing or decreasing temperature is detrimental to reaction efficiency (Table 2, entries 5 and 6). When the reaction was performed under air, no corresponding product could be detected, probably due to the oxygen sensitivity of the phosphine group (Table 2, entry 7).[18b] Next, various solvent, including CH_2Cl_2, 1,2,3-trichloropropane, acetone, THF, CH_3CN, and toluene, were examined (Table 2, entries 8–13). When 1,2,3-trichloropropane was utilized, product **5a** could be obtained in up to 99% yield (Table 2, entry 9). Finally, the molar ratio of **3a**/**4a** was investigated. Decreasing the molar ratio of **3a**/**4a** to 1/1 led to low reaction efficiency (Table 2, entry 14), while increasing the molar ration of **3a**/**4a** to 2/1 gave no increased efficiency (Table 2, entry 15). More details of hydrophosphination investigation are provided in Supporting Information (Tables S1-S4).

With the optimized conditions in hand, the substrate scope of nitroalkenes and phosphines was investigated (Scheme 2). Initially, aromatic nitroalkenes with substituents at the *para*-, *meta*-, and *ortho*-positions were examined. Both electron-rich (OMe and Me) and electron-deficient (F, Cl, and Br) substrates were well

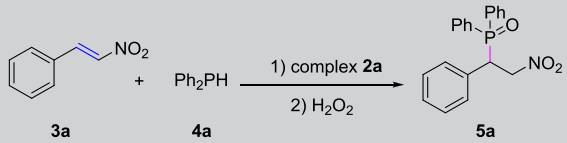
TABLE 1 Optimization of reaction conditions[a]



Entry	Complex	Yield (%)	Entry	Complex	Yield (%)
1	**1a**	92	**8**	**2a**	**91**
2	**1b**	94	9	**2b**	75
3	**1c**	91	10	**2c**	64
4	**1d**	92	11	**2d**	77
5	**1e**	81	12	**2e**	65
6	**1f**	89	13	**2f**	71
7	**1 g**	93	14	**2 g**	78

[a]Reaction conditions: **3a** (0.15 mmol), **4a** (0.10 mmol), pincer complex (10 mol%), NaOAc (10 mol%), $CHCl_3$, −20 °C, 12 hr; then H_2O_2 (2.5 equiv), −20 °C to r.t., 2 hr. Isolated yields.

TABLE 2 Optimization of reaction conditions[a]



Entry	Solvent	Base	T [°C]	Yield (%)
1	$CHCl_3$	*t*-BuONa	−20	91
2	$CHCl_3$	KOAc	−20	91
3	$CHCl_3$	NaOAc	−20	93
4	$CHCl_3$	Na_2CO_3	−20	66
5	$CHCl_3$	NaOAc	30	57
6	$CHCl_3$	NaOAc	−40	82
7[b]	$CHCl_3$	NaOAc	−20	N.R.
8	CH_2Cl_2	NaOAc	−20	90
9	**1,2,3-Trichloropropane**	**NaOAc**	**−20**	**99**
10	Acetone	NaOAc	−20	77
11	THF	NaOAc	−20	61
12	CH_3CN	NaOAc	−20	79
13	Toluene	NaOAc	−20	59
14[c]	1,2,3-Trichloropropane	NaOAc	−20	58
15[d]	1,2,3-Trichloropropane	NaOAc	−20	99

[a]Reaction conditions: **3a** (0.15 mmol), **4a** (0.10 mmol), complex **2a** (10 mol%), base (10 mol%), 12 hr; then H_2O_2 (2.5 equiv), −20 °C to r.t., 2 hr. Isolated yields.
[b]in the air.
[c]**3a** (0.10 mmol), **4a** (0.10 mmol). [d] **3a** (0.20 mmol), **4a** (0.10 mmol).

tolerated to afford the corresponding products **5a**-**l** in 73–99% yields. The molecular structure of product **5 k** was further confirmed by X-ray analysis, which is provided in Supporting Information (Figure S1 and Table S5). Also, dimethoxy-substituted nitroalkene could react with diphenylphosphine to provide **5 m** in 77% yield. Next, heteroaromatic and 2-naphthyl-substituted nitroalkenes were also employed, which generated products **5n**-**p** in 80–94% yield. Moreover, the current protocol could be applied to aliphatic nitroalkenes to deliver products **5q** and **5r** in 81% and 87% yields, respectively. Finally, bis (adamant-1-yl)phosphine was also proved to be a suitable substrate, which reacted with nitroalkenes to furnish the corresponding products **5 s**-**v** in 40–80% yields.

On the basis of previous literatures,[17–21] a plausible mechanism was proposed (Scheme 3). Initially, replacement of chloride in Ni (II) complex **2a** by OAc would afford Ni-OAc complex **I**, which underwent transphosphination reaction with Ph_2PH **4a** to give Ni-PPh_2 complex **II**. Subsequently, nucleophilic attack of nickel phosphide to nitroalkene **3a**[25] would generate a neutral nitro-nickel intermediate **III** with increased reactivity,[26] which might equilibrate with the zwitterionic Ni-phosphine complex **IV**. Finally, the protonolysis of intermediate **IV** in the presence of HOAc would provide the desired product **5a** accompanied with regeneration of reactive Ni species **I**.

4 | CONCLUSIONS

In conclusion, we have developed pincer Ni (II)-catalyzed of 1,4-addition of diphenylphosphine to nitroalkenes, generating the corresponding phosphinated products in good to excellent yield. The current protocol exhibits several characteristics, including earth-abundant transition-metal, broad substrate scope, and high efficiency.

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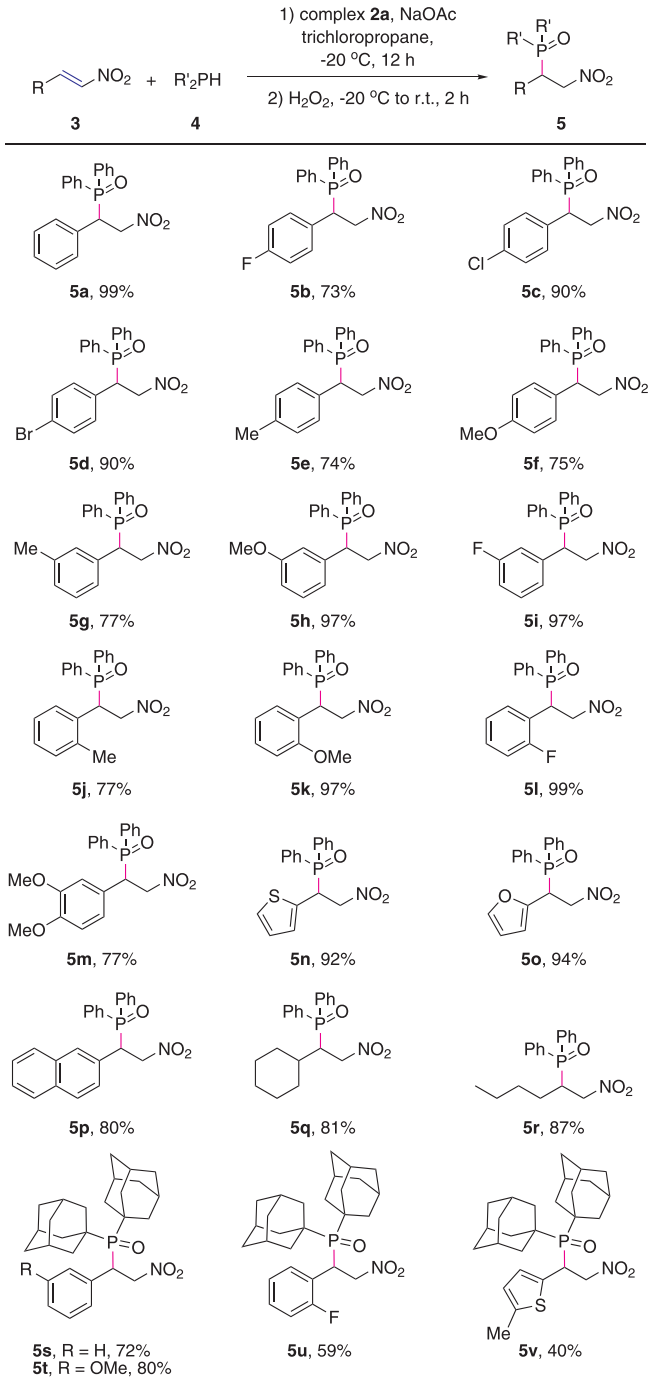
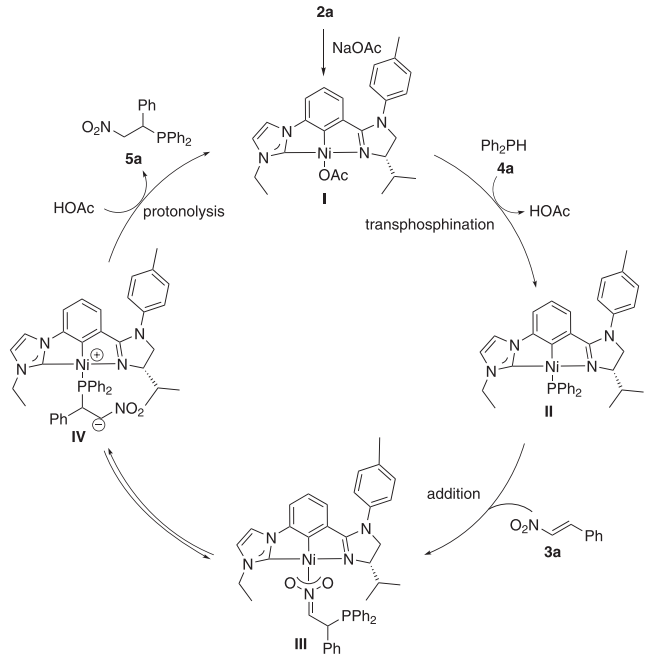


SCHEME 2 Substrate scope of nitroalkenes, [a] Reaction conditions 3 (0.15 mmol), 4(0.10 mmol), complex 2a (10 mol%), 1,2,3-trichloropropane, -20C, 12 hr, then H_2O_2 (2.5 equiv), −20 C to r.t., 2 hr Isolated yields



SCHEME 3 Proposed mechanism

AUTHOR CONTRIBUTIONS

Jing Yan: Investigation. **Yan-Bing Wang:** Data curation. **Senyao Hou:** Data curation. **XQ Hao:** Conceptualization; methodology. **Mao-Ping Song:** Funding acquisition; supervision.

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SUPPORTING INFORMATION

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