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Rhodium-catalyzed phenylation of *N*-arylsulfonyl aldimines with sodium tetraphenylborate or trimethyl(phenyl)stannane

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Abstract

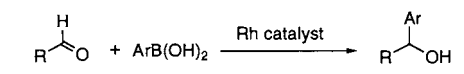
The rhodium-catalyzed addition reactions of trimethyl(phenyl)stannane and sodium tetraphenylborate to *N*-phenylsulfonyl aldimines $RCH{=}NSO_2Ph$ (R = alkyl and aryl) provided $R(Ph)CHNHSO_2Ph$ in high yields. A cationic and free phosphine complex $[Rh(cod)(MeCN)_2]BF_4$ was found to be an efficient catalyst for the addition of $PhSnMe_3$, whereas $[Rh(cod)(MeCN)_2]BF_4$/dppb catalyzed the addition of Ph_4BNa to various *N*-sulfonyl aldimines in dioxane at 90°C. The scope and limitations of the reactions, as well as the effect of varying the reaction conditions, are discussed. © 2000 Elsevier Science S.A. All rights reserved.

Keywords: Rhodium-catalyzed phenylation; *N*-arylsulfonyl aldimines; Sodium tetraphenylborate; Trimethyl(phenyl)stannane

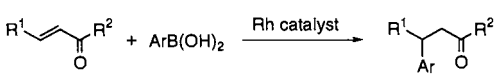
1. Introduction

The addition of the metal–carbon bond to the carbon–heteroatom double bond is a very popular reaction in main metal reagents of lithium and magnesium [1], but not much attention has been focused on the corresponding reaction of transition-metal compounds. However, the metal-catalyzed reactions, involving an insertion of the carbon–heteroatom bond to the carbon–transition metal bond, were recently extensively studied because the reactions are of interest due to their potential application to asymmetric synthesis. The additions of arylstannanes [2,3] and arylsilanes [4] to aldehydes, ketones or imines are catalyzed by a rhodium complex, $Ni(acac)_2/PPh_3$ catalyzes the methylation of aldehydes with trimethylaluminum [5], and allylstannanes [6,7] and allylsilanes [8] add to aldehydes and imines in the presence of $PdCl_2(PPh_3)_2$ or $PtCl_2(PPh_3)_2$.

Organoboron compounds are highly electrophilic, but the organic groups on boron are weakly nucleophilic, thus limiting the use of organoboron reagents for the ionic reactions. However, organoboron compounds, even organoboronic acids and esters, readily transmetalate to other metals, thus allowing the metal-catalyzed carbon–carbon bond forming reactions. For example, the cross-coupling reaction of organoboron compounds involving the transmetalation to palladium(II) halides has proved to be a general technique for a wide range of selective carbon–carbon bond-formations [9]. Although the metal-catalyzed addition reaction of organoboron compounds has not yet been well developed, the conjugate 1,4-addition of aryl- and 1-alkenylboronic acids to α,β-unsaturated ketones [10,11] and 1,2-addition to aldehydes [12] were recently found to be efficiently catalyzed by a rhodium(I) complex having a bidentate phosphine ligand with a large bite angle (Eqs. (1) and (2)).



R= alkyl, aryl (1)



(2)

The reaction proceeds in protic media or even in an aqueous solution because both the B–C and the Rh–C bonds are stable to water and alcohols. The asymmetric 1,4-additions using a chiral phosphine/rhodium complex were also demonstrated [11]. Here we report our first attempts to extend the protocol to the addition of

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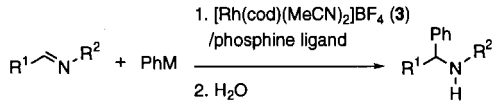
sodium tetraphenylborate and trimethyl(phenyl)stannane to aldimines [13] (Eq. (3)).



(3)

Table 1
Addition of trimethyl(phenyl)stannane (**2a**) to aldimines [a]

Entry	Imine	Catalyst	Yield (%) [b]
1	$PhCH{=}NSO_2Ph$	**3**	71
2		**3**/2PPh_3	50
3		**3**/dppp [c]	64
4		**3**/dppb [d]	42
5		**3**/dppf [e]	44
6		**3**/(*S*)-binap [f]	57
7	$4\text{-}MeOC_6H_4CH{=}NSO_2Ph$	**3**	(72)
8	$4\text{-}FC_6H_4CH{=}NSO_2Ph$	**3**	(89)
9	PhCH=NPh	**3**	(56)

[a] A mixture of imine (1 mmol), $PhSnMe_2$ (1.3 mmol), and catalyst (0.03 mmol, 3 mol %) in THF was stirred for 16 h at 60°C.
[b] GC yields and isolated yields are in parentheses.
[c] 1,3-bis(diphenylphosphino)propane.
[d] 1,4-bis(diphenylphosphino)butane.
[e] 1,1′-bis(diphenylphosphino)ferrocene.
[f] (*S*)-2,2′-bis(diphenylphosphino)-1,1′-binaphthyl.

Table 2
Reaction conditions in the addtion of Ph_4BNa (**2b**) to $PhCH{=}NSO_2Ph$ [a]

Entry	**2b** (equivalents)	Catalyst	Time (h)	Yield (%) [b]
1	1.3	$[Rh(acac)(C_2H_4)_2]$/dppb [c]	16	40
2		$[Rh(cod)_2]BF_4$/dppb	16	50
3		**3**	16	77
4		**3**/2PPh_3	16	97
5		**3**/dppe[c]	16	79
6		**3**/dppp	16	76
7		**3**/dppb	3	99 (89)
8		**3**/dppf	16	87
9		**3**/(*S*)-binap	16	61
10	0.25	**3**/dppb	16	85

[a] A mixture of $PhCH{=}NSO_2Ph$ (1 mmol), Ph_4BNa (0.5–1.3 mmol), and catalyst (0.03 mmol, 3 mol %) in dioxane was stirred at 90°C.
[b] GC yields and isolated yields are in parentheses.
[c] 1,2-bis(diphenylphosphino)ethane.

2. Results and discussion

2.1. Addition of $PhSnMe_3$ to aldimines

Various organometallic reagents which can transmetalate to rhodium(I) complexes will undergo the 1,2-addition to the C–N double bond. Among the reagents examined, trimethyl(phenyl)stannane (**2a**) was found to be an excellent reagent for phenylation of the *N*-sulfonyl aldimine in the presence of a cationic rhodium complex $[Rh(cod)(MeCN)_2]BF_4$ (**3**), but the reaction was very slow with neutral rhodium complexes such as $Rh(acac)(CO)_2$ and $Rh(acac)(CH_2{=}CH_2)_2$, or their combination with phosphine ligands. The additions of **2a** to aldimines are summarized in Table 1.

The reaction readily proceeded in THF at 60°C in the presence of **3** (3 mol%), but the addition of a phosphine ligand slowed down the reaction significantly (entries 2–6). Higher yields were easily realized for *N*-sulfonyl aldimines, but *N*-phenyl aldimine gave 58% of the addition product (entry 9), the results of which were in contrast to the similar reaction of Ph_4BNa discussed below (Table 3). The reaction has broad applicability for various aldimines under aprotic conditions, but the formation of toxic by-products may limit the use of organostannanes for organic synthesis.

2.2. Addition of Ph_4BNa to N-sulfonyl aldimines

The 1,2-addition of organoboronic acids to aldehydes [12] and the 1,4-addition to α,β-unsaturated ketones [10] afforded good results in an aqueous solvent (Eqs. (1) and (2)). However, a similar addition reaction of phenylboronic acid to *N*-sulfonyl aldimine **1** ($R^1 = Ph$, $R^2 = SO_2Ph$) at 80°C in DME/H_2O (6/1) resulted in the formation of diphenylmethanol (45%) through a sequence of hydrolysis of **1** with water and phenylation of resulting benzaldehyde (Eq. (1)). Thus, sodium tetraphenylborate **2b** was chosen as the arylation reagent of imines to carry out the reaction under anhydrous conditions.

The reaction conditions for the addition of **2b** to aldimines are summarized in Table 2. The imines did not react with **2b** at 90°C in the absence of a catalyst. Both $Rh(acac)(CH_2{=}CH_2)_2$/dppb and $[Rh(cod)_2]BF_4$/dppb were ineffective (entries 1 and 2), but $[Rh(acac)(MeCN)_2]BF_4$ (**3**) or its combination with a phosphine ligand efficiently catalyzed the addition (entries 3–9). The reaction smoothly proceeded in toluene (76%) and dioxane (99%), but it was very slow in a donating solvent such as DMF (18%). The rhodium-catalyzed addition of organoboronic acids to aldehydes (Eq. (1)) exhibited a pronounced effect of the phosphine ligand preferring the large bite angle ($\angle$P–Rh–P) [12]. Although there was no large difference in yields between the representative ligands having various bite

Table 3
Rhodium-catalyzed addition of Ph_4BNa (**2b**) to aldimines[a]

Entry	Imine	Time (h)	Yield (%)[b]
1	PhCH=NC_4H_9	16	Trace
2	PhCH=NCH_2Ph	16	Trace
3	PhCH=NPh	16	8
4	PhCH=NCOPh	16	75
5	PhCH=NSO_2(4-ClC_6H_4)	16	95
6	PhCH=NSO_2Ph	3	89
7	PhCH=NSO_2(4-MeC_6H_5)	16	89
8	4-$MeOC_6H_4$CH=NSO_2Ph	3	94
9	4-FC_6H_4CH=NSO_2Ph	3	91
10	4-$CF_3C_6H_4$CH=NSO_2Ph	16	92
11	1-naphthylCH=NSO_2Ph	3	68
12	2-MeC_6H_4CH=NSO_2Ph	16	90
13	cyclo-C_6H_{11}CH=NSO_2Ph	3	60

[a] A mixture of an imine (1 mmol), Ph_4BNa (1.3 mmol), **3** and dppb (0.03 mmol, 3 mol %) in dioxane was stirred at 90°C.
[b] Isolated yields based on the imines.

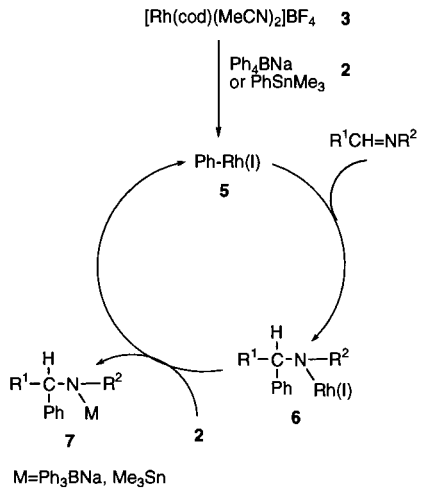


Fig. 1. Catalytic cycle.

angles, a combination of **3** and dppb achieved a quantitative yield within 3 h (entry 7). The ligand-accelerated reactions are of interest due to their potential application to asymmetric synthesis (entry 9). Although the reaction was studied in the presence of a stoichiometric amount of **2b**, all four phenyl groups in **2b** can participate in the catalytic cycle because the addition of 0.25 equivalents of **2b** achieved a comparable yield (entry 10).

The additions of **2b** to the representative aldimines in the presence of **3**/dppb (3 mol %) are summarized in Table 3. The insertion of imines into the carbon–rhodium bond may involve the nucleophilic attack of the aryl group to the C–N double bond. Thus, the reaction was markedly slow for the *N*-butyl, *N*-benzyl and *N*-phenylimine derivatives (entries 1–3), whereas the addition to activated aldimines with *N*-sulfonyl and *N*-benzoyl groups afforded high yields of the addition products (entries 4–13). The reaction was not significantly affected by the electronic effect of the substituents because both electron-rich and -deficient aldimines gave similarly good results (entries 8–10), which is in sharp contrast to the high electronic effect observed in the addition of arylboronic acids to aromatic aldehydes [12]. The *ortho*-substituents retarded the reaction, but it achieved 90% on prolongation the reaction time to 16 h (entry 12). The addition to alkylimines resulted in relatively low yields, but they were not improved on prolongation of reaction time (entry 13).

2.3. Reaction mechanism

Many transition-metal complexes catalyze the addition reactions of main metal reagents, but their mechanism has not yet been studied in detail. However, the transmetalation between the main metal reagents and transition-metal complexes has been proposed as a key step of the catalytic cycle [2–12]. Arylstannanes [14] and sodium tetraarylborate [15,16] have been used for the cross-coupling reaction with organic electrophiles since they readily transmetalate to various transition-metal halides such as palladium, platinum, and rhodium. Thus, the present addition reaction to imines should involve the transmetalation to the rhodium(I) complex, which can be explained as follows: (i) the transfer of the phenyl group of **2** to **3** produces phenylrhodium(I) species **5**, (ii) the insertion of the imine double bond into the carbon–rhodium bond yields a rhodium(I) amide **6**, and finally, (iii) the transmetalation between **2** and **6** produces **5** again (Fig. 1).

The arylrhodium(I) complexes are unstable such as to preclude isolation in pure form [17], but they apparently are involved in various coupling reactions with organic halides [18] and the addition to alkenes and alkynes [19] since they are stable to some extent in solution. The insertion of imine into the carbon–rhodium bond and the subsequent transmetalation between arylboron compounds and the rhodium amide **6** have not yet been previously studied, but the assumed pathway may be consistent with other related addition reactions of organostannanes and organosilanes [2–8]. The cationic rhodium complex **3** exhibited high catalyst efficiency over the neutral complexes, probably due to its high reactivity on the transmetalation with **2** or by high Lewis acidity on the coordination of imine to the rhodium metal center.

3. Experimental

All the experiments were carried out under argon atmosphere. ^{1}H-NMR spectra were recorded in $CDCl_3$ by a Jeol JNM-A400II (400 MHz) spectrometer using

Me_4Si as an internal standard. Mass spectra were obtained with a Finnigan ITD 800 for the GC–MS analyses and Jeol FABmate and Jeol HX110 for the high-resolution analyses. GC analyses were performed using a Hitachi G-5000 equipped with a glass column (OV-17 on Uniport B, 2 m).

3.1. Materials and reagents

N-Arylsulfonyl aldimines [20,21] and *N*-(phenylmethylene)benzamide [22,23] were prepared by the literture procedures. Ph_4BNa was dried in vacuo for 24 h at 110°C. The literature procedure gave $[Rh(cod)(MeCN)_2]BF_4$ [24].

3.2. General procedure for the addition of $PhSnMe_3$ (Table 1)

The flask was charged with $[Rh(cod)(MeCN)_2]BF_4$(0.015 mmol) and imine (0.5 mmol), and flushed with argon. THF (2 ml) and $PhSnMe_3$ (0.65 mmol) were successively added to the flask. After being stirred at 60°C for 16 h, the reaction was quenched by addition of water (2 ml). The product was extracted with benzene, washed with water, and finally dried over $MgSO_4$. Chromatography over silica gel gave the corresponding amines.

3.3. General procedure for the addition of Ph_4BNa (Tables 2 and 3)

The flask charged with $[Rh(cod)(MeCN)_2]BF_4$ (0.015 mmol), ligand ($2PPh_3$, dppf, dppe, dppb, or dppb) (0.015 mmol), *N*-arylsolfonylaldomine (0.5 mmol), and Ph_4BNa (0.65 mmol) was flushed with argon. Dioxane (3 ml) was then added. After being stirred for 16 h at 90°C, water (2 ml) was added at room temperature. The product was extracted with benzene, washed with water, and dried over $MgSO_4$. Chromatography over silicagel gave the following amines.

3.3.1. N-Phenylsulfonyl diphenylmethylamine

IR (Nujol): 3230, 2340, 1350, 1160 cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 5.01 (d, $J = 7.2$ Hz, 1H), 5.61 (d, $J = 7.2$ Hz, 1H), 7.08–7.11 (m, 4H), 7.20–7.22 (m, 6H), 7.35 (t, $J = 7.8$ Hz, 2H), 7.48 (t, $J = 7.8$ Hz, 1H), 7.68 (d, $J = 7.8$ Hz, 2H). Calc. for $C_{19}H_{17}O_2NS$ 323.0960, found 323.0961.

3.3.2. N-Phenyldiphenylmethylamine

IR (neat): 3400, 3060, 3030, 2450, 1700, 1605, 1503, 1455, 1415, 1313, 1265, 1205 cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 5.49 (s, 1H), 6.54 (d, $J = 8.5$ Hz, 2H), 6.69 (t, $J = 7.3$ Hz, 1H), 7.11 (dd, $J = 7.3$ and 8.5 Hz, 2H), 7.24–7.27 (m, 4H), 7.30–7.37 (m, 6H). Calc. for $C_{19}H_{17}N$ 259.1361, found 259.1358.

3.3.3. N-Benzoyldiphenylmethylamine

IR (Nujol): 3320, 2350, 1638, 1515, 1473 cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 6.46 (d, $J = 7.8$ Hz, 1H), 6.65 (d, $J = 7.8$Hz, 1H), 7.27–7.37 (m, 10H), 7.44 (t, $J = 7.8$ Hz, 2H), 6.51 (t, $J = 7.8$ Hz, 1H), 7.82 (d, $J = 7.8$ Hz, 2H). Calc. for $C_{20}H_{17}NO$ 288.1388, found 288.1376.

3.3.4. N-p-Chlorophenylsulfonyldiphenylmethylamine

IR (Nujol): 3240, 2345, 1308, 1166, 1162 cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 5.13 (d, $J = 7.3$ Hz, 1H), 5.62 (d, $J = 7.3$ Hz, 1H), 7.09–7.11 (m, 4H), 7.21–7.23 (m, 6H), 7.27 (d, $J = 8.5$ Hz, 2H), 7.55 (d, $J = 8.5$ Hz, 2H). Calc. for $C_{19}H_{16}NO_2SClNa$ 380.0488, found 380.0469 (FAB, *m*-nitrobenzyl alcohol, added NaI).

3.3.5. N-p-Methylphenylsulfonyl diphenylmethylamine

IR (Nujol): 3240, 2350, 1306, 1161 cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 2.38 (s, 3H), 5.02 (d, $J = 7.1$ Hz, 1H), 5.56 (d, $J = 7.1$ Hz, 1H), 7.09–7.11 (m, 4H), 7.14 (d, $J = 8.3$ Hz, 2H), 7.20–7.23 (m, 6H), 7.56 (d, $J = 8.3$ Hz, 2H). Calc. for $C_{20}H_{20}NO_2S$ 338.1215, found 338.1236.

3.3.6. N-Phenylsufonyl-p-methoxyphenyl(phenyl)-methylamine

IR (Nujol): 3240, 2995, 2305, 1492, 1295, 1228, 1156, 1127 cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 3.75 (s, 3H), 4.99 (d, $J = 7.1$ Hz, 1H), 5.56 (d, $J = 7.1$ Hz, 1H), 6.74 (d, $J = 8.8$ Hz, 2H), 6.99 (d, $J = 8.8$ Hz, 2H), 7.08–7.11 (m, 2H), 7.17–7.22 (m, 3H), 7.35 (dd, $J =$ 7.2, 8.3 Hz, 2H), 7.48 (t, $J = 7.2$ Hz, 1H), 7.67 (d, $J = 8.3$ Hz, 2H). Calc. for $C_{20}H_{19}NO_3S$ 353.1085, found 353.1090.

3.3.7. N-Phenylsulfonyl-p-fluorophenyl(phenyl-)methylamine

IR (Nujol): 3275, 3240, 2340, 1305, 1230, 1160 cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 5.11 (d, $J = 7.,1$ Hz, 1H), 5.58 (d, $J = 7.1$ Hz, 1H), 6.89 (t, $J = 8.5$ Hz, 2H), 7.04 (m, 4H), 7.20–7.24 (m, 4H), 7.36 (dd, $J = 7.3$, 7.9Hz, 2H), 7.49 (t, $J = 7.3$ Hz, 1H), 7.67 (d, $J = 7.9$Hz, 2H). Calc. for $C_{19}H_{16}NO_2SFNa$ 364.0784, found 364.0764 (FAB, *m*-nitrobenzyl alcohol, added NaI).

3.3.8. N-Phenylsulfonyl-p-trifluoromethylphenyl-(phenyl)methylamine

IR (Nujol): 3250, 2340, 1320, 1158, 1130, 760 cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 5.30 (d, $J = 7.1$ Hz, 1H), 5.63 (d, $J = 7.1$ Hz, 1h), 7.02–7.04 (m, 2H), 7.21–7.28 (m, 4H), 7.33–7.38 (m, 3H), 7.47 (t, $J = 8.0$ Hz, 2H), 7.67 (d, $J = 8.0$ Hz, 2H). Calc. for $C_{20}H_{16}F_3NO_2SNa$ 414.0752, found 414.0765 (FAB, *m*-nitrobenzyl alcohol, added NaI).

3.3.9. N-Phenylsulfonyl-1-napthylphenylmethylamine

IR (Nujol): 3245, 2340, 1308, 1155 cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 5.12 (d, $J = 7.3$ Hz, 1H), 6.35 (d, $J = 7.3$ Hz, 1H), 7.127.47 (m, 12H), 7.64 (d, $J = 7.3$ Hz, 2H), 7.73 (d, $J = 8.1$ Hz, 1H), 7.83 (d, $J = 9.0$ Hz, 2H). Calc. for $C_{23}H_{19}NO_2S$ 373.1136, found 373.1134.

3.3.10. N-Phenylsufonyl-o-methylphenyl(phenyl)-methylamine

IR (Nujol): 3250, 2340, 1318, 1302, 1147 cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 2.17 (s, 3H), 4.99 (d, $J = 6.8$ Hz, 1H), 5.83 (d, $J = 6.8$ Hz, 1H), 7.03–7.09 (m, 6H), 7.19–7.20 (m, 3H), 7.33 (t, $J = 7.8$ Hz, 2H), 7.45 (t, $J = 7.8$ Hz, 1H), 7.66 (d, $J = 7.8$ Hz, 2H). Calc. for $C_{20}H_{18}NO_2S$ 336.1058, found 336.1065(M − 1).

3.3.11. N-Phenylsufonyl-cyclohexyl(phenyl)-methylamine

IR (Nujol): 3270, 2375, 1320, 1160 cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 1.05–1.17 (m, 3H), 1.28 (d, $J = 12.7$ Hz, 1H), 1.54–1.59 (m, 3H), 1.73 (d, $J = 12.7$ Hz, 1H), 1.95 (d, $J = 12.7$ Hz, 1H), 4.06 (t, $J = 8.1$ Hz, 1H), 6.88–6.90 (m, 2H), 7.07–7.09 (m, 3H), 7.23 (d, $J = 7.6$ Hz, 2H), 7.36 (dd, $J = 7.3$, 7.6 Hz, 1H), 7.56 (d, $J = 7.3$ Hz, 2H). Calc. for $C_{19}H_{23}NO_2S$ 329.1449, found 329.1469.

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