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Palladium-catalyzed Hiyama coupling reaction of arylsulfonyl hydrazides under oxygen†

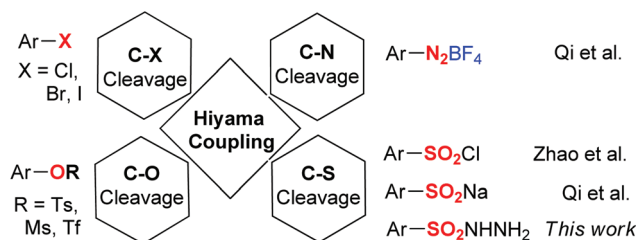Hui Miao, Fenhua Wang, Shuangliu Zhou,* Guangchao Zhang and Yang Li

Palladium-catalyzed Hiyama cross-coupling reactions of various arylsulfonyl hydrazides with a wide variety of aryl silanes have been achieved in good to excellent yields under simple conditions. The newly developed catalytic system does not require the use of expensive silver- or copper-based stoichiometric oxidants and can be accelerated by the addition of TBAT under an atmosphere of oxygen. The reported Hiyama-type coupling reactions are tolerant to common functional groups, making these transformations attractive alternatives to the traditional cross-coupling approaches.

Transition-metal-catalyzed cross-coupling reactions of aryl halides reagents to aryl silanes (Hiyama coupling) have attracted much attention and have provided great progress in the construction of new Ar–Ar bonds for the preparation of useful biaryl motifs.[1] The Hiyama cross-coupling reaction is one of the effective methods for constructing asymmetrical biaryls that are structural components of various pharmaceuticals and other functional materials.[2] In comparison with Stille coupling[3] using toxic tin reagents and Suzuki coupling[4] with problems in purifying the boron reagents, the silylated molecules used in Hiyama coupling are low toxicity, low cost, readily availability and have high chemical stability, making the coupling reactions more attractive from the environmental point of view.[5]

In the past few years, various arylating procedures other than dehalogenation have been explored for Hiyama coupling, and aryl halides can be replaced by a few other types of aryl sources such as aryl tosylates/mesylates *via* deoxidation.[6] Arenediazonium salts have been applied in cross-coupling reactions along with the release of nitrogen under mild conditions for a long time.[7] In 2011, Qi *et al.* reported the first example of a practical denitrogenative coupling procedure for

Hiyama cross-coupling reactions using arenediazonium salts as the electrophilic reagents.[8] In fact, the past few years have witnessed the application of several closely related $ArSO_2X$ (X = Cl and Na) compounds as a source of aryl group in oxidative C–C coupling reactions *via* desulfitation.[9] Recently, Hiyama cross-coupling reactions *via* desulfitation has been emerged. Qi *et al.*[10] reported their results on Pd-catalyzed Hiyama-type cross-coupling reactions of various arenesulfinates with different organosilicon reagents in the absence of bases and ligands under mild reaction conditions (Scheme 1). Since then arylsulfonyl chlorides have been applied as convenient arylating reagents by Zhao's group.[11]

Although significant progress has been made in this area, the development of an air-stable, readily available, and inexpensive arylating reagent is still in high demand. One attractive strategy is the combination of desulfitative and denitrificative coupling with arylsulfonyl hydrazides. Arylsulfonyl hydrazides are readily accessible and stable synthetic intermediates which can be simply prepared from easily available arylsulfonyl chlorides and hydrazine hydrates in one step. They have been widely employed as sulfone or thioether sources by means of N–S and/or S–O bond cleavage,[12] and more significantly, they could also serve as aryl sources by means of C–S bond cleavage.[13] In the presence of palladium catalysts and carbonyl compounds, arylsulfonyl hydrazides can readily convert into very active diazo compounds which are found to be very important cross-coupling partners.[14]

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†Electronic supplementary information (ESI) available. See DOI: 10.1039/c5ob00139k

Scheme 1 Different types of Hiyama coupling.

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Org. Biomol. Chem., 2015, **13**, 4647–4651 | **4647**

Table 1 The influence of additive to the reaction[a]
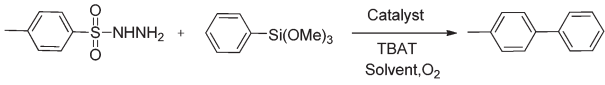
Entry	Additive	Yield[b] (%)
1	—	—
2	AgF	44
3	NH$_4$F	13
4	LiF	20
5	NaF	63
6	KF	61
7	CsF	77
8	TBAF (in THF)	82
9	TBAF (75%, in water)	64
10	TBAF·3H$_2$O	84
11	TMAF·4H$_2$O	81
12	TEAF·2H$_2$O	85
13	TASF	87
14	TBAT	93

[a] Reaction conditions: TsNHNH$_2$ (0.5 mmol), PhSi(OMe)$_3$ (0.6 mmol), Pd(TFA)$_2$ (5 mol%), additive (0.6 mmol), DMI (1.0 ml) at 60 °C for 12 hours under O$_2$ unless otherwise indicated. [b] Isolated coupling yield.

Arylsulfonyl hydrazides could be employed as novel aryl sources, which would undergo loss of N$_2$ and SO$_2$ gas *in situ*. Herein, we have reported a novel protocol to construct unsymmetrical biaryls using arylsulfonyl hydrazides as aryl sources in Hiyama coupling.

We initiated our investigation by examining the feasibility of the Pd-catalyzed cross-coupling of arylsulfonyl hydrazides and aryl silanes (Table 1). We first carried out the reaction of 4-methylbenzenesulfonohydrazide with trimethoxy(phenyl)-silane under Pd(TFA)$_2$ catalysis in DMI (1,3-dimethyl-2-imid-azolidinone) at 60 °C under O$_2$, however, no desired product was detected (Table 1, entry 1). Then we started to investigate a variety of additives to favor the removal of silicon groups. In this reaction the influence of a fluoride resource seem to a play key role to promote the efficacy. To our delight, AgF served as an additive under oxygen to afford the desired cross-coupling product in 44% yield (Table 1, entry 2). Other fluoride salts such as NH$_4$F, LiF, NaF, KF and CsF were tested, and CsF gave much better results (77%) (Table 1, entries 3–7). The coupling reaction with the addition of TBAF (1 M, in THF) was explored, and 82% yield of desired product was isolated (Table 1, entry 8). However, the addition of TBAF (75% aqueous) only afforded 64% yield of product using DMI as the solvent (Table 1, entry 9). We added TBAF·3H$_2$O to this reaction under the standard conditions, 84% yield was obtained which showed that trace water did not hinder the transformation (Table 1, entry 10). Various activators were screened, TMAF (tetramethylammonium fluoride), TEAF (tetraethyl-ammonium fluoride) and TASF (tris(dimethylamino)sulfonium difluorotrimethylsilicate) afforded the desired cross-coupling products with 81%, 85% and 87% yields (Table 1, entries 11–13). Finally, we found that TBAT (tetrabutyl-ammoniumdifluorotriphenylsilicate) exhibited the best efficiency (Table 1, entry 14).

Next, we tested the catalytic effects of catalysts and solvents on this Hiyama coupling reaction and results are summarized

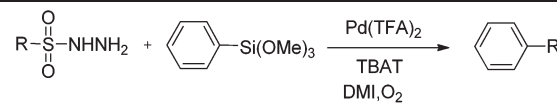**Table 2** Optimization of catalyst and solvent for the reaction[a]

Entry	Catalyst	Solvent	Yield[b] (%)
1	Pd(PPh$_3$)$_4$	DMI	—
2	Pd$_2$(dba)$_3$	DMI	—
3	Pd(OAc)$_2$	DMI	86
4	PdCl$_2$	DMI	75
5	PdCl$_2$ (PPh$_3$)$_3$	DMI	46
6	PdCl$_2$(dppf)	DMI	31
7	Pd(TFA)$_2$	DMI	93
8	Pd(TFA)$_2$	Xylene	10
9	Pd(TFA)$_2$	1,4-Dioxane	7
10	Pd(TFA)$_2$	CH$_3$CN	70
11	Pd(TFA)$_2$	DMSO	75
12	Pd(TFA)$_2$	DMF	83
13	Pd(TFA)$_2$	DMA	81
14	Pd(TFA)$_2$	NMP	86

[a] Reaction conditions: TsNHNH$_2$ (0.5 mmol), PhSi(OMe)$_3$ (0.6 mmol), catalyst (5 mol%), TBAT (1 mmol), solvent (1.0 ml) at 60 °C for 12 hours under oxygen unless otherwise indicated. [b] Isolated coupling yield.

in Table 2. Firstly, various Pd precursors were screened for the reaction in DMI under O$_2$ at 60 °C in the presence of TBAT. Unfortunately, with the use of Pd(0) precursors such as Pd(PPh$_3$)$_4$ and Pd$_2$(dba)$_3$, only a trace amount of the desired product was detected (Table 2, entries 1–2). The reactivity of Pd(OAc)$_2$ is more than that of PdCl$_2$, while PdCl$_2$ (PPh$_3$)$_3$ did not exhibit good catalytic activity (Table 2, entries 3–5). When 5 mol% of PdCl$_2$(dppf) was used, only 31% yield of 4-methyl-biphenyl was obtained (Table 2, entry 6). Among the tested palladium catalysts, Pd(TFA)$_2$ showed the highest catalytic reactivity and the desired product was isolated in 93% yield (Table 2, entry 7). The influence of the solvent was also studied. When other solvents, such as xylene and 1,4-dioxane, were applied instead of DMI, only a trace amount of the desired product was observed (Table 2, entries 8–9). Protic sol-vents were tested, and the application of MeCN and DMSO were much more efficient (Table 2, entries 10–11). Indeed, the yield of 4-methyl-biphenyls were improved to 83%, 81% and 86% when the reaction was carried out in DMF, DMA (*N*,*N*-di-methylacetamide) and NMP (*N*-methyl-2-pyrrolidone) (Table 2, entries 12–14). Various solvents were screened and we found that DMI exhibited the best efficiency.

We used the reaction conditions of entry 7 in Table 2 to first examine the substrate scope of this reaction with respect to the palladium-catalyzed Hiyama coupling reaction of aryl-sulfonyl hydrazides. As summarized in Table 3, we set out to test different arylsulfonyl hydrazides as coupling partners. A range of arylsulfonyl hydrazides containing both electron-rich and electron-deficient groups smoothly underwent cross-coupling with trimethoxy(phenyl)silane, delivering a series of biaryls in moderate to excellent yields. For example, methoxyl, fluoro, chloro, bromo, cyano, and nitro substituents on the phenyl rings survived and the desired products were obtained

Table 3 Pd-catalyzed Hiyama cross-coupling reactions of various aryl-sulfonyl hydrazides with trimethoxy(phenyl)silane[a]
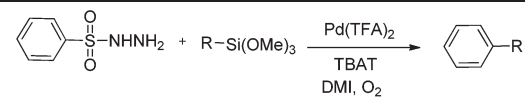
Entry	R	Yield[b] (%)
1	4-CH$_3$OC$_6$H$_4$	95
2	4-FC$_6$H$_4$	88
3	4-ClC$_6$H$_4$	92
4	4-BrC$_6$H$_4$	87
5	4-NCC$_6$H$_4$	83
6	4-NO$_2$C$_6$H$_4$	85
7	4-IC$_6$H$_4$	90
8	3-CH$_3$C$_6$H$_4$	87
9	2-CH$_3$C$_6$H$_4$	86
10	2-Naphthyl	89
11	1-Naphthyl	82
12	4-HOC$_6$H$_4$	76
13	4-H$_2$NC$_6$H$_4$	80
14	1H-Tetrazole-5-yl	72
15	1H-Imidazole-2-yl	79
16	1-Methyl-1H-indole-2-yl	83
17	1H-Indole-2-yl	78

[a] Reaction conditions: RSO$_2$NHNH$_2$ (0.5 mmol), PhSi(OMe)$_3$ (0.6 mmol), Pd(TFA)$_2$ (5 mol%), TBAT (1 mmol), DMI (1.0 ml) at 60 °C for 12 hours under oxygen unless otherwise indicated. [b] Isolated coupling yield.

Table 4 Pd-catalyzed Hiyama cross-coupling reactions of phenylsulfo-nyl hydrazides with a wide variety of aryl silanes[a]

Entry	R	Yield[b] (%)
1	4-CH$_3$OC$_6$H$_4$	88
2	4-ClC$_6$H$_4$	90
3	4-FC$_6$H$_4$	94
4	4-NO$_2$C$_6$H$_4$	91
5	4-CH$_3$C$_6$H$_4$	94
6	3-CH$_3$C$_6$H$_4$	89
7	2-CH$_3$C$_6$H$_4$	81
8	2-Naphthyl	86
9	1-Naphthyl	84
10	2-Thienyl	78
11	Pyridin-2-yl	74
12	Pyridin-3-yl	79

[a] Reaction conditions: PhSO$_2$NHNH$_2$ (0.5 mmol), RSi(OMe)$_3$ (0.6 mmol), Pd(TFA)$_2$ (5 mol%), TBAT (1 mmol), DMI (1.0 ml) at 60 °C for 12 hours under oxygen unless otherwise indicated. [b] Isolated coupling yield.

in good to excellent yields (Table 3, entries 1–6). The results implied that the electronic effect is not critical for this transformation. Furthermore, a selective C–S bond cleavage of arylsulfonyl hydrazides with bromo and iodo substitution was observed in the reaction without the detection of C–X coupling by-products under the current reaction conditions, providing the sole products 4-bromo-biphenyl and 4-iodo-biphenyl in 87% and 90% yields respectively (Table 3, entries 4 and 7). The positions of the substituents on the phenyl rings had a negligible effect on the transformation. Whether the substituents were installed in the *meta-* or *ortho*-position on the arenes, arylsulfonyl hydrazides could be converted into the desired biaryls (Table 3, entries 8–9). Moreover, when the phenyl ring was replaced by a naphthyl group, the reaction also afforded the target molecules in 89% and 82% yield (Table 3, entries 10–11). It should be noted that the yields of desired products were decreased with the introduction of hydroxyl and amino groups (Table 3, entries 12–13). When 1H-tetrazole-5-yl and 1H-imidazole-2-yl reacted with trimethoxy-(phenyl)silane, the desired cross-coupling products were obtained in 72% and 79% yields respectively (Table 3, entries 14–15). To our delight, indolyl sulfonyl hydrazides were efficiently coupled with trimethoxy(phenyl)silane, no matter whether there is a methyl group on the N-1 position or not (Table 3, entries 16–17).

We next investigated the scope of the reaction with a series of aryl silanes and phenylsulfonyl hydrazides. As shown in Table 4, this reaction was compatible with methoxyl, chloro, fluoro and nitro substituents, and furnished the desired pro-
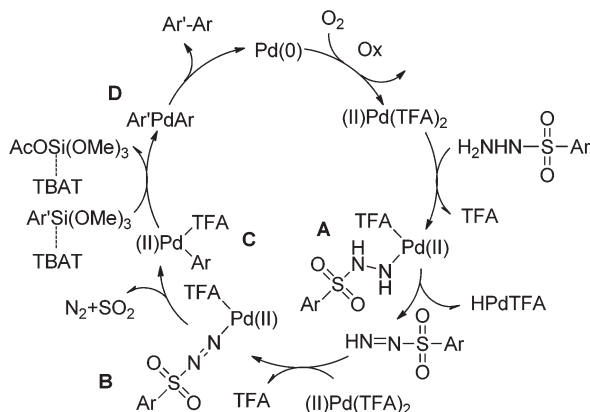
ducts in good yields (Table 4, entries 1–4). Meanwhile, phenyl trimethoxysilane, bearing a sterically hindered group, such as methyl at their *ortho-*/*meta*-positions, also underwent the reaction with phenylsulfonyl hydrazide smoothly, providing the corresponding products in slightly lower yields (81–89%) compared with their corresponding *para*-substituted ones (Table 4, entries 5–7). However, a weak *ortho* position effect was observed in the reaction with naphthyl trimethoxysilane (Table 4, entries 8–9). In addition, 2-thienyl, pyridin-2-yl and pyridin-3-yl also underwent the reaction with phenylsulfonyl hydrazide to generate desired products in 78%, 74% and 79% yields, respectively (Table 4, entries 10–12).

We examined this Pd-catalyzed cross-coupling reaction under different atmospheres. No cross-coupling product was detected under nitrogen gas protection, and the cross-coupling product yield was found to be only about 39% under air. A proposed mechanism for the palladium-catalyzed Hiyama reaction of arylsulfonyl hydrazides with aryl silanes *via* direct desulfitative and denitrificative coupling in the presence of TBAT is shown in Scheme 2. The reaction probably proceeds involving the formation of Pd(II) intermediate A by the displacement of Pd(TFA)$_2$ catalyst by arylsulfonyl hydrazide. Intermediate A undergoes β-hydride elimination to generate (arylsulfonyl)diazene and release HPd(TFA) at the same time. Subsequent displacement of Pd(TFA)$_2$ with (arylsulfonyl)-diazene provides reactive Pd(II) intermediate B, and arylpalla-dium(II) intermediate C is formed through successive liberation of N$_2$ and SO$_2$ gas.[13b,c] The transmetallation of aryl silanes with intermediate C with the assistance of TBAT gives arylpalladium(II) species D. Reductive elimination of D affords the cross-coupling product. Pd(II) species are then regenerated by the oxidation of the Pd(0) species with oxygen for next run.

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Scheme 2 Proposed mechanism for the reaction.

Conclusions

In summary, we have developed a Pd-catalyzed Hiyama coupling reaction with arylsulfonyl hydrazides, which is an alternative approach to the coupling of aryl silanes with an aryl halide surrogate. In this approach, widely accessible arylsulfonyl hydrazide derivatives function as the coupling reagents on the basis of C–S cleavage, and a wide range of biaryls could be efficiently synthesized under this desulfitative and denitrificative reaction. Further optimization and expansion of the scope of this reaction as well as mechanistic studies are currently under way.

Acknowledgements

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