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Report

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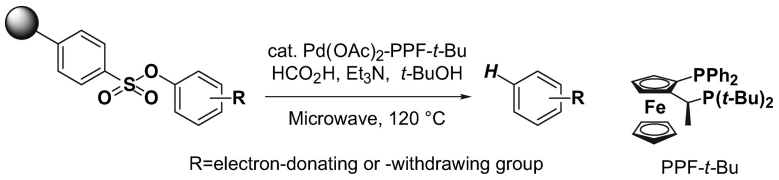

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Revisiting Benzenesulfonyl Linker for the Deoxygenation and Multifunctionalization of Phenols

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Arenes and heteroarenes are important classes in the field of pharmaceutical and material sciences. Discovery of the arenes possessing interesting biological activities and functions should require enormous time and labor. Solid-phase synthesis based on combinatorial chemistry has enabled rapid preparation of a lot of aromatic compounds and promoted the discovery process. A key component in solid-phase synthesis is the linker that is used to attach the molecules to the solid support. Traceless linkers represent an exciting aspect of solid-phase organic synthesis due to the desire to make molecules lacking any extraneous functionality.[1] Although several traceless linkers for attachment of the arenes have been developed, they have their own limitations. Group 14 metal-based linkers[2] and triazene-type linkers[3] require preactivation of the initial building block prior to attachment to the resin, i.e., metalation of aryl halides and formation of diazonium salts from the parent anilines, respectively. On the other hand, hydrazide[4] and boronate[5] linkers are restricted to classes of starting materials with relatively few commercial members. Benzenesulfonate linker[6−8] gets rid of these limitations where the direct and facile coupling of commercially available polystyrene sulfonyl chloride (PS-SO_2Cl)[9] and also commercially available phenols was possible without prior modification. However, only electron-deficient phenols could be deoxygenated under $Pd(OAc)_2$/1,3-bis(diphenylphosphino)propane (dppp) catalysis due to the poor activating ability of the benzenesulfonyl group. To improve the poor reactivity, some electron-deficient 'triflate-like' linkers[10] have been developed, but their preparation is now required.

The commercially availability and chemical stability of the benzenesulfonyl linker were enough attractive to drive us to reinvestigate the Pd-catalyzed reductive cleavage conditions applicable to the sulfonates of electron-rich phenols. Recent advance in Pd-catalyzed carbon−carbon and carbon−nitrogen bond formation using aryl benzenesulfonates reported by Hartwig's[11] and Buchwald's[12] groups illustrates that phosphine ligands coordinated to the Pd(0) plays an important role to overcome the poor reactivity. We now wish to describe the ligand effect on the Pd-catalyzed reductive cleavage of *p*-toluenesulfonates and resin-bound benzenesulfonates of electron-rich phenols. We also expand application of this linker to a strategy whereby additional functionality can be appended to the aryl ring during the cleavage step.

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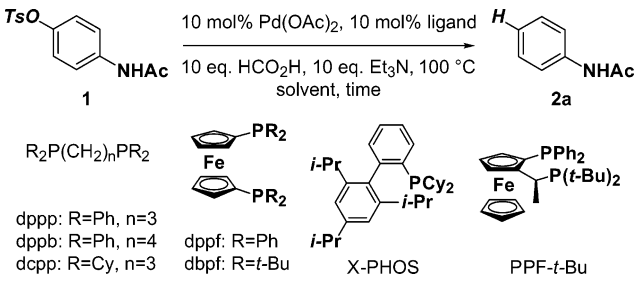
Table 1. Ligand Effect on Pd-Catalyzed Reductive Cleavage of **1**



entry	ligand	time (h)	solvent	yield[a] (**1**:**2a**)[b]
1	dppp	12	DMF	0%
2	dppb	12	DMF	0%
3	dppf	12	DMF	0%
4	dcpp	12	DMF	− (64:36)
5	dbpf	12	DMF	− (88:12)
6	X-PHOS[c]	12	DMF	− (99:1)
7	PPF-*t*-Bu	12	DMF	83%[d]
8	PPF-*t*-Bu	12	*t*-BuOH	quant.
9	PPF-*t*-Bu[e]	12	*t*-BuOH	− (12:88)
10[f]	PPF-*t*-Bu	2	*t*-BuOH	quant.
11[f,g]	PPF-*t*-Bu	4	*t*-BuOH	94%
12[f,h]	PPF-*t*-Bu	1	*t*-BuOH	quant.

[a] Isolated yield. [b] The ratio was determined by ^1H NMR analysis. [c] 20 mol % X-PHOS was used. [d] **1** was recovered in 13% yield. [e] 10 mol % $PdCl_2$(PPF-*t*-Bu) was used as a catalyst. [f] Reaction with microwave irradiation. [g] Reaction with 5 mol % catalyst. [h] Reaction at 120 °C.

This effort began by ligand screening for the Pd-catalyzed reductive cleavage of acetamide-substituted *p*-toluenesulfonate **1**[13] (Table 1). The reaction was carried out in DMF on heating at 100 °C with an excess of formic acid and triethylamine as reducing agents in the presence of a catalytic amount of $Pd(OAc)_2$ and a series of phosphine ligands. The electron-donating acetamide group in **1** hindered the cleavage with the palladium ligated with bis(diphenylphosphine) such as dppp, dppb, and dppf (entries 1−3).[14] The use of more σ-donating bis(dialkylphosphine) afforded a small amount of the reduction product **2a** (entries 4, 5). These results suggest oxidative addition of **1** to the Pd(0) complex should be a rate-determining step. While 2-(dicyclohexylphosphino)-2′,4′,6′-tri-*i*-propyl-1,1′-biphenyl (X-PHOS)[15] as a sterically hindered monophosphine developed by Buchwald[12] was less effective, PPF-*t*-Bu[15] as a sterically hindered bis(phosphine) employed by Hartwig[11] proved to be the most effective for the cleavage (entries 6, 7). Further optimization of reaction conditions revealed that *t*-BuOH as solvent completely converted **1** into **2a** (entry 8).[16] $PdCl_2$(PPF-*t*-Bu) complex[11b] could also be used for the reductive cleavage (entry 9). Microwave irradiation[17] accelerated the reduction and allowed decreasing the catalyst loading to 5 mol % (entries 10, 11). Furthermore, raising the reaction temperature to

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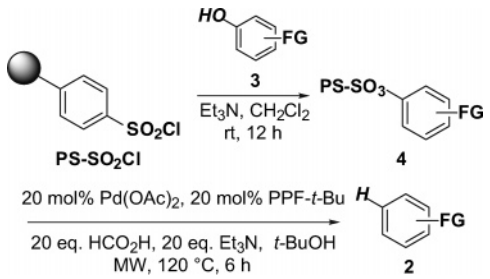
Table 2. Pd-Catalyzed Reductive Cleavage of Resin-Bound Benzenesulfonate **4**



entry	FG	product	overall yield (%)[a]
1	4-NHAc	**2a**	92 (91)[b]
2[c]	4-NHAc	**2a**	53 (97)[b]
3	2-NHAc	**2a**	quant.
4	3-NHAc	**2a**	89
5	2,4-diNHAc	**2d**	95
6[d]	4-OCH_2CO_2-*t*-Bu	**2e**	80
7	4-SCH_2CO_2-*t*-Bu	**2f**	82
8	4-$(CH_2)_2CMe_2OH$	**2g**	85
9	4-$CH_2CH(NHBoc)CO_2Me$	**2h**	68
10	2-OMe-4-CH_2OH	**2i**	64
11[e]	4-COPh	**2j**	93

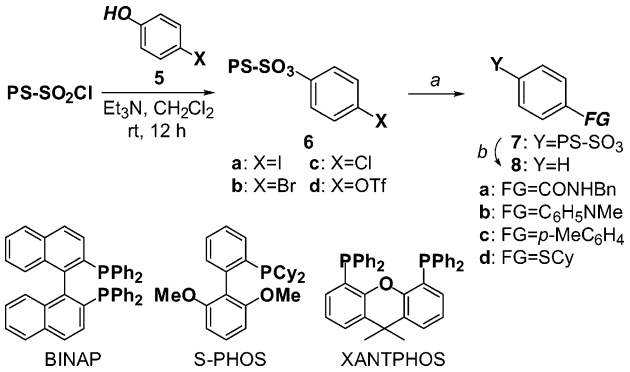
[a] Isolated yield based on the initial loading of the resin. [b] Purity of the crude material determined by reversed-phase HPLC with monitoring at 254 nm is shown in parentheses. [c] Reaction for 12 h with 10 mol % catalyst. [d] Reaction for 8 h. [e] Reaction for 2 h.

120 °C enabled the reduction to be completed within 1 h (entry 12).

The reductive cleavage of solid-supported benzenesulfonate **4a**, easily prepared by *p*-hydroxyacetanilide (**3a**) and PS-SO_2Cl in the presence of triethylamine, required a larger amount of reagents and a longer reaction time than that of **1** in the solution-phase reaction, but it provided acetanilide in high yield (Table 2, entry 1). Low catalyst loading in order to minimize impurities in the crude material after cleavage resulted in poor yield (entry 2). Phenols containing the acetamide group at the *ortho*- or *meta*-position and even the *ortho*-, *para*-disubstituted one could be deoxygenated equally (entries 3−5). Other electron-donating substituents including ether, thioether, and alkyl groups were also allowed (entries 6−10). An electron-withdrawing benzoyl group on the phenol ring enhanced the reductive cleavage (entry 11). Furthermore, the cleavage conditions are compatible with a wide variety of functional groups, i.e., ketone, ester, amide, and alcohol.

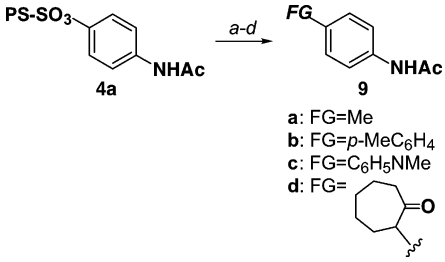
The benzenesulfonate linker would be expected to survive Pd-catalyzed carbon−carbon or carbon−heteroatom bond formation of aryl iodides, bromides, chlorides, and triflates due to its low reactivity. To demonstrate its stability, *p*-halophenols **5a**−**c** and *p*-(trifluoromethanesulfonyloxy)-phenol (**5d**) were attached to the PS-SO_2Cl resin, which were subjected to their own Pd-catalyzed transformations (Scheme 1). Carbonylation[18] of resin-bound aryl iodide **6a**, amination[19,20] of the bromide **6b**, Suzuki−Miyaura cross-coupling reaction[21] of the chloride **6c**, and thioetherification[22] of the triflate **6d** followed by reductive cleavage provided functionalized arenes **8a**−**d** in moderate to excellent yield. Because these palladium-catalyzed reactions are widely used,

Scheme 1. Pd-Catalyzed Transformations on Benzenesulfonate Linker[a]



[a] Reagents and conditions: (*a*) 10 equiv $BnNH_2$, 10 equiv DBU, 10 mol % $[PdCl(C_3H_5)]_2$, 40 mol % PPh_3, THF, CO (1 atm), rt, 14 h for **6a**; 10 equiv C_6H_5NHMe, 13 equiv Cs_2CO_3, 20 mol % $Pd(OAc)_2$, 20 mol % BINAP, toluene, 100 °C, 24 h for **6b**; 6 equiv *p*-$MeC_6H_4B(OH)_2$, 12 equiv $K_3PO_4 \cdot nH_2O$, 15 mol % $Pd(OAc)_2$, 38 mol % S-PHOS, THF, rt, 24 h for **6c**; 10 equiv CySH, 20 equiv *i*-Pr_2NEt, 10 mol % Pd_2dba_3, 22 mol % XANTPHOS, 1,4-dioxane, 100 °C, 24 h for **6d**. (*b*) 20 equiv HCO_2H, 20 equiv Et_3N, 20 mol % $Pd(OAc)_2$, 20 mol % PPF-*t*-Bu, *t*-BuOH, MW, 120 °C, 6 h. Overall yield based on the initial loading of the resin: 95% for **8a**; 58% for **8b**; quant. for **8c**; 89% for **8d**.

Scheme 2. Pd- and Ni-Catalyzed Multifunctional Cleavage of **4a**[a]



[a] Reagents and conditions: (*a*) 10 equiv $MeB(OH)_2$, 15 equiv $K_3PO_4 \cdot nH_2O$, 20 mol % $Pd(OAc)_2$, 50 mol % X-PHOS, THF, MW, 120 °C, 5 h, 60%. (*b*) 10 equiv *p*-$MeC_6H_4B(OH)_2$, 13 equiv $K_3PO_4 \cdot nH_2O$, 30 mol % $NiCl_2(PCy_3)_2$, 120 mol % PCy_3, 1,4-dioxane, 130 °C, 24 h, 65%. (*c*) 10 equiv C_6H_5NHMe, 15 equiv Cs_2CO_3, 20 mol % $Pd(OAc)_2$, 50 mol % X-PHOS, *t*-BuOH, MW, 120 °C, 8 h, 83%. (*d*) 10 equiv cycloheptanone, 15 equiv Cs_2CO_3, 20 mol % $Pd(OAc)_2$, 50 mol % X-PHOS, *t*-BuOH, MW, 120 °C, 8 h, 43%. Yields are based on the initial loading of the resin.

especially for solid-phase synthesis, the benzenesulfonyl linker has an advantage over the activated 'triflate-like' linker.

Finally, multifunctional cleavage of the benzenesulfonyl linker in **4a** using other nucleophiles was examined (Scheme 2). In contrast to the reductive cleavage, the $Pd(OAc)_2$/X-PHOS catalytic system in combination with microwave irradiation was suitable for Suzuki−Miyaura cross-coupling reaction[12b] with methylboronic acid, amination[12a] with *N*-methylaniline, and α-arylation[12b] of cycloheptanone to give functionalized acetanilides **9a**, **9c**, and **9d**. It is noteworthy that the Suzuki−Miyaura reaction with arylboronic acid under the same condition did not provide the coupling product **9b**, but promoted self-coupling reaction of the boronic acid to form biaryl. Instead of the Pd catalyst, $Ni(PCy_3)_2Cl_2/PCy_3$[23] catalyzed the cross-coupling reaction efficiently without microwave irradiation.

In conclusion, we have discovered the Pd-catalyzed reductive cleavage conditions for resin-bound benzenesulfonates of electron-rich phenols as well as an electron-deficient one. Commercial availability and stability of the

linker to the Pd-catalyzed transformation of aryl halides and triflate must be an advantage over the activated 'triflate-like' linker. The multifunctional cleavage of the linker was also achieved under palladium or nickel catalysis in the presence of a wide variety of nucleophiles. Solid-phase synthesis of biologically active arenes using the benzenesulfonate linker is underway in our laboratory.

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Supporting Information Available. Experimental procedures and compound characterization data (PDF). This material is available free of charge via the Internet at http://pubs.acs.org.

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