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Palladium-catalyzed desulfitative arylation of sulfonamides with sodium arylsulfinates

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ABSTRACT

A Pd(II)-catalyzed desulfitative arylation protocol between sulfonamides and sodium arylsulfinates was herein reported. The direct arylation reaction was successfully achieved by a Pd(II)/Ag(I)-mediated system without participation of any external ligands with a release of SO₂. And different *N*-aryl sulfonamides were obtained readily in up to 86% yields, exhibiting good functional groups tolerance (25 examples).

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KEYWORDS

Arylation; desulfitative; palladium catalysis; sodium arylsulfinates; sulfonamides

GRAPHICAL ABSTRACT



Introduction

Novel methodologies for direct *N*-arylation of sulfonamides have been widely studied to improve the diversification of the significant compounds. Various aryl donators, including aryl halides,^[1] aryl boronic acids,^[2] aryl siloxanes,^[3] aryl nonaflates,^[4] even cyclohexanones,^[5] and so on^[6] have been successfully utilized for the straightforward arylation of sulfonamides with the assistance of various transition metal catalysts, such as Pd(II), Ni(II), and Cu(II) and etc. As far as we are aware, sodium arylsufinates, which have been frequently employed as efficient sulfonylation^[7] and sulfuration^[8] reagents, came to our consciousness as a direct and good sacrifice of aryl groups in different transition metallic catalysis in a desulfitative pattern, releasing gaseous SO₂. Traditionally, involvement of the reagent sodium sulfinates renders the desulfitative arylation means enjoy the advantages like stability and easy-handling work-up procedure. Up to date, desulfitative protocols for the constructions of C–C bonds^[9] have been well established on the substrates of different types, like arylmethyl chlorides,^[10] aldehydes,^[11] aryl triflates,^[12] polyfluoroarenes,^[13] olefins,^[14] alkynes.^[15] While formations of C–N bonds^[16] have been successfully achieved on the hetero compounds including thiophenol, indoles, azoles,

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Supplemental data (full experimental details, ¹H and ¹³C NMR spectra) can be accessed on the publisher's website.
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caffeines, coumarins, and even $CuCN^{[17]}$ have also been readily arylated in the same manner. Very recently, applications of potassium iodide and dialkyl phosphites for the formations of $C-I^{[18]}$ and C-P bonds^[19] mediated by a Pd(II)-catalyst were well-illustrated in a similar manner.

Cheng has disclosed a desulfitative synthesis of carboxylic acids in a liganded Cu(I)mediated system.^[20] Inspired by An's description, CuI was capable to make the desulfitative arylations of sulfonamides took place in the absence of any external organic ligands.^[21] Herein, we would like to unveil a Pd(II)-catalyzed method for arylation of sulfonamides with sodium arylsulfinates with the assistance a silver oxidant in the absence of any external ligands (Scheme 1).

Discussion

Firstly, reactions between S-phenyl sulfonamide (1a) and sodium phenylsulfinate (2a) were conducted to obtain the optimal conditions, which were summarized in Table 1. In the presence of palladium diacetate (5 mol%) and silver acetate (2.0 equiv.), the arylation reaction took place readily in 1,4-dioxane, but in low efficiency, for only 32% of **3aa** was isolated after column seperation (entry 1). Other simple Pd(II) catalysts, such as Pd(TFA)₂ (TFA for trifluoroacetic acid) and PdCl₂ offered superior performance to that Pd(OAc)₂ did. For 45 and 42% yields of **3aa** were obtained in the system (entries 2 and 3). Disappointingly, phosphine-liganded palladium catalysts, such as Pd(PPh₃)₂Cl₂ and Pd(dppf)₂Cl₂ (dppf stands for diphenylphosphino ferrocene) were incapable of making the desulfitative transformation happen for only trace of the desired product **3aa** was detected after 24 h (entries 4 and 5). By contrast, nitriles-coordinated Pd(II)-catalysts, which were exemplified by Pd(MeCN)₂Cl₂ and Pd(PhCN)₂Cl₂ rendered the coupling of **1a** and **2a** easily, leading to the formation of **3aa** in medium yields, up to 62% (entries 6 and 7). However, Pd(PPh₃)₄ failed to make the desufitative arylation happen, for only trace of **3aa** was found over the consumption of **2a** as monitored by TLC (entry 8). Other

KI
$$\frac{\text{ArSO}_{2}\text{Na}/[\text{Pd}]}{[\text{Ref 18}]} \text{Ar}-I$$

$$\stackrel{O}{\text{Ro}}_{P}-H \xrightarrow{\text{ArSO}_{2}\text{Na}/[\text{Pd}]}{[\text{Ref 18}]} \text{RO}_{P}-\text{Ar}$$

$$\stackrel{O}{\text{Ro}}_{R'} \xrightarrow{\text{ArSO}_{2}\text{Na}/[\text{Pd}]}{[\text{Ref 19}]} \text{RO}_{R'} \xrightarrow{\text{Ar}}{RO}$$

$$\frac{\text{ArSO}_{2}\text{Na}/[\text{Cu}]}{[\text{Ref 20}]} \text{Ar}-\text{COOH}$$

$$\stackrel{O}{\text{R'}} \xrightarrow{\text{O}}_{NH_{2}} \xrightarrow{\text{ArSO}_{2}\text{Na}/[\text{Cu}]}{[\text{Ref 21}]} \xrightarrow{\text{O}}_{R'} \xrightarrow{\text{O}}_{N} \xrightarrow{\text{Ar}}{Ar}$$

$$\stackrel{O}{\text{R'}} \xrightarrow{\text{O}}_{NH_{2}} \xrightarrow{\text{ArSO}_{2}\text{Na}/[\text{Pd}]}{\frac{\text{ArSO}_{2}\text{Na}/[\text{Pd}]}{This work}} \xrightarrow{\text{O}}_{R'} \xrightarrow{\text{O}}_{N} \xrightarrow{\text{Ar}}{Ar}$$

Scheme 1. Desulfitative arylation methodologies.





^aReaction conditions: **1a** (0.3 mmol), **2a** (0.36 mmol), [Pd] (5 mol%), [O] (2.0 equiv.), under argon atmosphere or noted in sol. (2.0 mL) at 120 °C for 24 h.

^bIsolated yields.

Stands for not detected.

^dThe ratio is 9:1 by volume.

^eThe yield in the parentheses was obtained in 0.5 mmol scale.

DCE, dichloroethane; DCP, dicumyl peroxide; DMSO, dimethyl sulfoxide; DTBP, di-tert-butyl peroxide; THF, tetrahydrofuran.

oxidants were also screened in the Pd-mediated protocol. Except Ag_2CO_3 , other silver salts, like AgNO₃, Ag₂O, and AgOTf did not offer any positive results for trace or maximum 55% of **3aa** was checked in the systems (entries 9–12). Furthermore, other organic oxidants such as $K_2S_2O_8$ (entry 13), dicumyl peroxide (entry 14), di-*tert*-butyl peroxide (entry 15), and clean oxidant molecular dioxygen (entry 16) proved to be totally ineffective to the arylation system for no reaction was detected in the Pd-Ag-mediated system after 24 h. For solvents, pure solvents including tetrahydrofuran (entry 17), toluene (entry 18), dichloroethane (entry 19), and dimethyl sulfoxide (DMSO for entry 20) gave inferior yields to that 1,4-dioxane did in the existence of the combination of Pd(MeCN)₂Cl₂ and AgOAc. However, beyond our expectations, the addition of DMSO as co-solvent of 1,4-dioxane (ratio 9:1) raised the yield of **3aa** dramatically up to a 82% ratio (entry 21). This probably was due to the better solubility of **2a** in the mixture of the solvents. Meanwhile, Pd(MeCN)₂Cl₂ was proved crucial to the transformation for no reaction was detected in the absence of only AgOAc (entry 22).

With the optimal conditions in hands, the scope and limitations of the substrates on the sulfonamides were evaluated in the Pd-Ag-mediated system, which was summarized in Table 2. First, *para*-methylphenyl sulfonamide (**1b**) coupled with **2a** successfully, forming

	0 S ⁰ + R ^{NH} 2 +	O Š Ph ONa	Pd(MeCN) ₂ Cl ₂ AgOAc 1,4-Dioxane/DMSO	O、O R ^{´S} N [−] Ph	
	1b - 1n	2a	(9:1), 120 °C, 24 h	3ba - 3na	
Entry	1		R	3	Yield ^b (%)
1	1b	4-C	H ₃ C ₆ H ₄	3ba	85
2	1c	2-C	$H_3C_6H_4$	3ca	78
3	1d	4-C	H ₃ OC ₆ H ₄	3da	82
4	1e	4- <i>t</i> E	BuC ₆ H ₄	3ea	79
5	1f	4-F(C ₆ H ₄	3fa	72
6	1g	4-C	C ₆ H ₄	3ga	78
7	1h	4-B	rC ₆ H ₄	3ha	81
8	1i	4-N	$O_2C_6H_4$	3ia	68
9	1j	4-C	F ₃ C ₆ H ₄	3ja	76
10	1k	2-N	aphthyl	3ka	80
11	11	2-TI	niophenyl	3la	72
12	1m	CH3		3ma	48
13	1n	CH3	CH ₂	3na	45

Table 2. Substrates scope of the sulfonamides.^a

^aReaction conditions: 1 (0.5 mmol), 2a (0.6 mmol), Pd(MeCN)₂Cl₂ (5.0 mol%), AgOAc (1.0 mmol), in 1,4-dioxane/DMSO (3.5 mL in 9:1 ratio) at 120 °C for 24 h.

^blsolated yields.

DMSO, dimethyl sulfoxide.

the desired arylated product **3ba** in a good 85% yield (entry 1). The variation of the methyl group on the para- or ortho-positions of the S-phenyl sulfonamide showed a slight influence on the efficiency of the transformation for 78% of 3ca was isolated successfully (entry 2). Other electron-donating groups, such as methoxy- (entry 3) and tert-butyl (entry 4) which occupied on the para-positions of S-phenyl sulfonamides, provided the desired products 3da and 3ea in 82 and 79% yields, respectively. S-Halophenyl sulfonamides, which were exemplified by S-(4-fluorophenyl) sulfonamide (1f for entry 5), S-(4-chlorophenyl) sulfonamide (1g for entry 6) and S-(4-bromophenyl) sulfonamide (1h for entry 7), expressed good capabilities in the coupling with sodium phenylsulfinate (2a), for the corresponding products 3fa-3ha were furnished in yields from 72 to 82%. Decreased yields were generally observed when electron-withdrawing groups were decorated on the phenyl sulfonamides. For instance, S-(4-nitrophenyl) sulfonamide (1i) and S-(4-trifluorophenyl) sulfonamide (1j) afforded the arylated products 3ia and 3ja in only 68 and 76% yields, separately (entries 8 and 9). The compatibilities of S-polyaryl or heteroaryl-decorated sulfonamides were also checked in the system and 2-naphthyl sulfonamide (1k for entry 10) and 2-thiophenyl sulfonamide (1l for entry 11) offered an easy access to the arylated products 3ka and 3la in yields of 80 and 72%. Surprisingly, alkyl sulfonamides like methyl sulfonamide (1m) and ethyl sulfonamide (1n) gave the desired arylated products 3ma and 3na in yields of 48 and 45% (entries 12 and 13).

Successively, the tolerance of the functional groups of sodium arylsulfinates was also checked in the system as shown in Table 3. First, sodium *p*-tolylsulfinate (**2b**) coupled with **1a** readily, forming the desired molecule **3ab** in 85% yield (entry 1). However, change of the position of the methyl groups on the sodium arylsulfinates affected the yield of the arylated products slightly for N-(*o*-methylphenylated)-*S*-phenyl sulfonamide **3ac** was provided in 78% yield (entry 2). Also, expected N-(*p*-methoxyphenylated) sulfonamides

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Table J.	Substrates scope of social alyisannates.					
	0,0	0=0	Pd(MeCN) ₂ Cl ₂ AgOAc	0,50		
	Ph [´] `NH ₂	Ar´`ONa	1,4-Dioxane/DMSO	Ph´N´ ^{Ar} H		
	1a	2b - 2l	(9:1), 120 °C, 24 h	3ab - 3al		
Entry	2		Ar	3	Yield ^b (%)	
1	2b	4-CH	$_{3}C_{6}H_{4}$	3ab	85	
2	2c	2-CH	₃ C ₆ H ₄	3ac	78	
3	2d	4-CH	3OC ₆ H ₄	3ad	86	
4	2e	4-FC _e	H ₄	3ae	79	
5	2f	4-CIC	₆ H ₄	3af	81	
6	2g	2-CIC	₆ H ₄	3ag	72	
7	2h	4-BrC	C_6H_4	3ah	80	
8	2i	2-BrC	C ₆ H ₄	3ai	70	
9	2j	4-CF	C ₆ H ₄	3aj	58	
10	2k	4-CN	C ₆ H ₄	3ak	62	
11	21	2-Na	ohthyl	3al	80	
12	2m	2-Thi	ophenyl	3am	n.d.	
13	2n	CH₃C	H ₂	3an	n.d.	

 Table 3.
 Substrates scope of sodium arylsulfinates.^a

^aReaction conditions: **1a** (0.5 mmol), **2** (0.6 mmol), Pd(MeCN)₂Cl₂ (5.0 mol%), AgOAc (1.0 mmol), in 1,4-dioxane/DMSO (3.5 mL in 9:1 ratio) at 120 °C for 24 h.

^blsolated yields.

DMSO, dimethyl sulfoxide.

3ad were isolated successfully in 86% yield (entry 3). Halogenated phenyl groups were also well-tolerated in the system for sodium 4-fluorophenyl (entry 4), 4-chlorophenyl (entry 5), 2-chlorophenyl (entry 6), 4-bromophenyl (entry 7), 2-bromophenyl (entry 8) sulfinates were compatible in the protocol and the desired products **3ae–3ai** were separated in yields of 70–81% range. Electron-withdrawing groups decorated arylsulfinates were also tested in the system and *N*-(4-trifluoromethylphenylated) and *N*-(4-cyanophenylated) products **3aj** and **3ak** were isolated in 58 and 62% yields, respectively (entries 9 and 10). Polyaryl group like sodium 2-naphthylsulfinate offered the desired 2-naphthylated product **3al** in 80% yield (entry 11). However, heteroaryl group like 2-thiophenyl showed a negative effective to the arylation protocol, as well as the alkylsulfinate did, for 2-thiophenylation and ethylation reactions were not observed in the Pa/Ag-mediated system (entries 12 and 13).

Based on the extensive literature explorations, the mechanism of the Pd/Ag-mediated protocol was proposed, which was illustrated by the reaction between **1a** and **2a** (Scheme 2). Firstly, Pd(II)-catalyst coordinated with the substrate **1a**, forming the liganded-palladium intermediate **A**. Successively, sodium phenylsulfinate (**2a**) attacked the intermediate **A** through the anionic interaction, forming another key intermediate **B**. Next, the key intermediate **B** was transformed into another key intermediate **C**, with a release of a molecular SO₂. Then reductive elimination made the formation of final product **3aa** with Pd(0), which was reoxided into Pd(II) in the presence of Ag⁺ for the completion of a catalytic circle.

Conclusion

In summary, we have successfully developed a general and efficient Pd/Ag-mediated transformation towards *N*-arylated sulfonamides with sodium arylsulfinates through a desulfitative pathway. The transformation enjoyed good functional group compatibility and high efficiency. The practical and facile methodology offered a promising avenue towards the compounds of great significance.



Scheme 2. Proposed mechanism.

Experimental

Typical synthetic procedure of N-aryl sulfonamides

Under the argon atmosphere, a Schlenk tube (15 mL) equipped with a magnetic bar was loaded with the sulfonamide 1 (0.5 mmol), sodium arylsulfinates 2 (0.6 mmol, 1.2 equiv.), $Pd(MeCN)_2Cl_2$ (6.5 mg, 5 mol%), and AgOAc (166.9 mg, 1.0 mmol) in one portion. Then, the mixture of 1,4-dioxane/DMSO (3.5 mL in a 9:1 ratio) was added to obtain a clear solution and the reaction mixture was allowed to stir at 120 °C for 24 h. After cooling to room temperature, the mixture was filtered through a short celite pad and washed with dichloromethane (15 mL \times 3). The filtrate was concentrated and the oily crude product was purified by column chromatography using silica gel (200–300 mesh) as stationary phase and a petroleum ether and ethyl acetate (3/1) as eluent to give the *N*-aryl sulfonamides 3 in noted yields.

Spectra data of N-aryl sulfonamides 3

N,S-diphenyl sulfonamide (3aa)

White solid, mp: 105–107 °C. Yield: 80%. ¹H NMR (400 MHz, CDCl₃) δ = 7.81 (d, J = 7.6 Hz, 2H), 7.57 (t, J = 7.3 Hz, 1H), 7.47 (t, J = 7.5 Hz, 2H), 7.29 (d, J = 8.0 Hz, 2H), 7.16 (t, J = 7.4 Hz, 1H), 7.11 (d, J = 7.8 Hz, 2H), 6.81 (br.s, NH) (ppm). ¹³C NMR (100 MHz, CDCl₃) δ = 139.1, 136.4, 133.18, 129.5, 129.2, 127.3, 125.7, 122.0 (ppm). IR (in KBr): v = 3257, 2961, 2362, 1727, 1599, 1496, 1463, 1410, 1337, 1264, 1158, 1094, 1022, 921, 803, 754, 697, 631, 599, 561 cm⁻¹. MS(EI) *m*/*z* (%): 233.0, 168.0, 141.0, 92.0, 77.0, 65.0. HRMS(EI) *m*/*z*: [M]⁺ Calcd for C₁₂H₁₁NO₂S: 233.0510; Found 233.0505.

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