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Acid-Promoted Expeditious Syntheses of Aminated Dibenzosultams via Palladium/Norbornene Cooperatively Catalysed C–H Amination/Arylation

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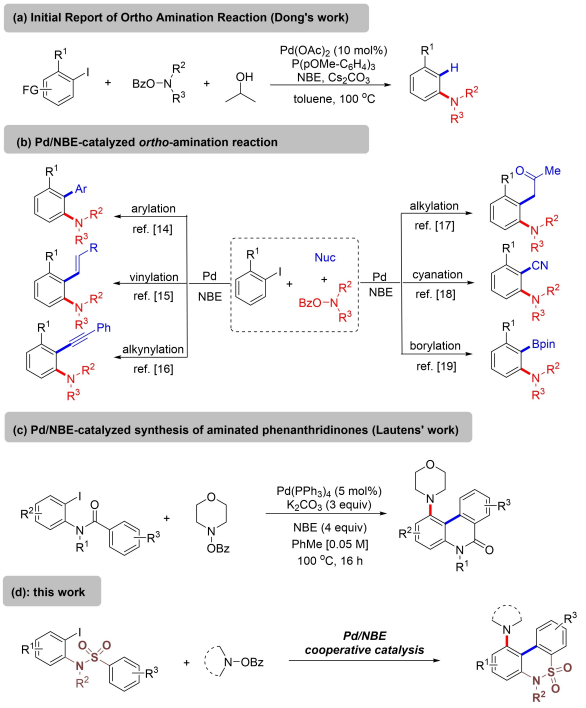
Abstract: Herein we reported a protocol to access aminated benzofused sultams through sequential *ortho*-amination, followed by *ipso*-arylation of aryl iodide bearing sulfonamide functional group via palladium/norbornene coorperative catalysis. This reaction has showcased a broad spectrum of substrate scope under mild conditions with moderate to good efficiency.

Keywords: palladium/norbornene; PivOH; *ortho*-amination; *ispo*-C(sp^2)–H arylation; benzosultams

Over the past few decades, transition-metal catalysed C–H bond functionalization has emerged as a powerful strategy for the construction of complex bioactive molecules, as such, many endeavours have been made and significant achievements have been achieved in this field.[1] The incorporation of C–H bond activation into different mechanistic pathways offers efficient avenues for molecule syntheses.[2] Conventional methodologies often capitalise on directing-groups which require pre-decoration of substrate molecule to achieve desired selectivity.[3] However, the multiple chemical steps involving removal of directing groups or functional group interconversion from the resulting product also mean low step economy and limit the application.[4] In contrast to chelated-assisted method, the more atom- and step-economical traceless or transient directing group strategies have gained increasing attention because of environmentally benign property.[5]

One well-established reaction harnessing the transient C–H activating reagent is the Catellani reaction, first introduced by the group of Catellani in 1997,[6] a powerful methodology for efficient preparation of multi-substituted arenes through chemoselective functionalization of both *ortho*- and *ipso*- positions of aryl halides in a single transformation.[7] A variety of functional groups can be appended at *ipso*-position, which is classified as classical palladium (0) cross-coupling.[8] By comparison, *ortho*-functionalization was restricted to alkylation,[9] arylation,[10] acylation,[11] and thiolation.[12] In 2013, Dong and co-workers first developed the *ortho* amination/*ipso* hydrogenation of aryl halide by exploiting *O*-benzoyl hydroxylamines as electrophile in Pd/NBE (norbornene) chemistry (Scheme 1a).[13] Since then, various termination process using different nucleophiles as quenching reagents based on *ortho* amination have been developed, including arylation,[14] vinylation,[15] alkynylation,[16] alkylation,[17] cyanation,[18] and borylation (Scheme 1b).[19] By virtue of this strategy, Luan[20] and Liang[21] disclosed several elegant Catellani-type annulations to access diversified benzofused rings. Very recently, Lautens group reported Pd/NBE-catalyzed synthesis of aminated phenanthridinones through *ortho* intermolecular amination followed by intramolecular C–H activation (Scheme 1c).[22] Despite these exquisite efforts, to significantly broaden the scope of this reaction, the discovery of novel reaction compatible with Pd/NBE catalysis is highly desirable.

On the other hand, the benzosultams are important structural motif widely exist in many biologically active compounds. Compounds with the privileged scaffold usually demonstrate versatile inhibitory properties.[23] Divergent synthetic methods for assem-

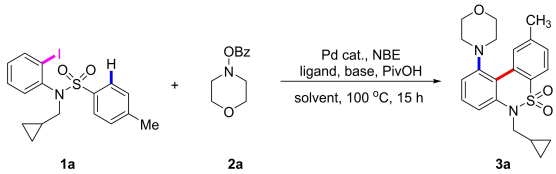


Scheme 1. *ortho* Amination/*ipso* Functionalizations of Aryl Iodides.

ble of sultams have been established by the group of Zhu,[24b] Wu,[25b] Ma,[25c] Kanai,[26b] and Lei,[26c] including transition-metal or lewis acid-catalyzed processes,[24] metal-free oxidative annulation strategy,[25] and intermolecular cyclization.[26] Inspired by these reports and in continuation of our research interests[11d,e] in Pd/NBE-catalyzed reaction, we are intrigued whether it would be possible to synthesize benzosultams motif through Catellani-type reaction using aryl halide with sulfoamide group as linking group, which has not been reported to date. Herein we reported our newly disclosed protocol to access aminated bensosultams through sequential *ortho*-amination/*ipso*-C–H bond arylation with sulfoamide-tethered aryl iodide via palladium/norbornene coorperative catalysis (scheme 1d).

For reaction optimization, we initiated our investigation by selecting sulfonamide tethered aryl iodide **1a** and *O*-benzoylhydroxylamine **2a** as model coupling partners in the presence of catalytic system composed of $Pd(OAc)_2$ (5 mol%) and $PCy_3{\cdot}HBF_4$ (15 mol%). To our delight, the desired benzosultam **3a** was afforded in yield of 39% (entry 1). Encouraged by this result, we next systematically examined the other reaction parameters as summarised in Table 1. Besides $Pd(OAc)_2$, other commonly used palladium precatalysts including $Pd_2(dba)_3$, $\{PdCl(C_3H_5)\}_2$, $PdCl_2(PPh_3)_2$, and $Pd(PPh_3)_4$ were tested (entries 2–5), and it was found $Pd(PPh_3)_4$ without external ligand was the best choice of catalyst, furnishing desired

Table 1. Reaction Condition Optimization.[a]



entry	[Pd]	ligand	base	solvent	Yield%
1	$Pd(OAc)_2$	$PCy_3{\cdot}HBF_4$	Cs_2CO_3	toluene	39
2	$Pd_2(dba)_3$	$PCy_3{\cdot}HBF_4$	Cs_2CO_3	toluene	20
3	$\{Pd(C_3H_5)Cl\}_2$	$PCy_3{\cdot}HBF_4$	Cs_2CO_3	toluene	5
4	$PdCl_2(PPh_3)_2$	–	Cs_2CO_3	toluene	63
5	$Pd(PPh_3)_4$	–	Cs_2CO_3	toluene	74
6	$Pd(PPh_3)_4$	RuPhos	Cs_2CO_3	toluene	72
7	$Pd(PPh_3)_4$	XantPhos	Cs_2CO_3	toluene	31
8	$Pd(PPh_3)_4$	TFP	Cs_2CO_3	toluene	90
9	$Pd(PPh_3)_4$	TFP	K_3PO_4	toluene	7
10	$Pd(PPh_3)_4$	TFP	K_2CO_3	toluene	6
11	$Pd(PPh_3)_4$	TFP	Na_2CO_3	toluene	trace
12	$Pd(PPh_3)_4$	TFP	KOAc	toluene	trace
13	$Pd(PPh_3)_4$	TFP	Cs_2CO_3	DMF	20
14	$Pd(PPh_3)_4$	TFP	Cs_2CO_3	MeCN	18
15	$Pd(PPh_3)_4$	TFP	Cs_2CO_3	dioxane	80
16[b]	$Pd(PPh_3)_4$	TFP	Cs_2CO_3	toluene	76
17[c]	$Pd(PPh_3)_4$	TFP	Cs_2CO_3	toluene	52
18	$Pd(OAc)_2$	TFP	Cs_2CO_3	toluene	73
19	$PdCl_2$	TFP	Cs_2CO_3	toluene	76
20	$\{Pd(C_3H_5)Cl\}_2$	TFP	Cs_2CO_3	toluene	80
21	$Pd_2(dba)_3$	TFP	Cs_2CO_3	toluene	78

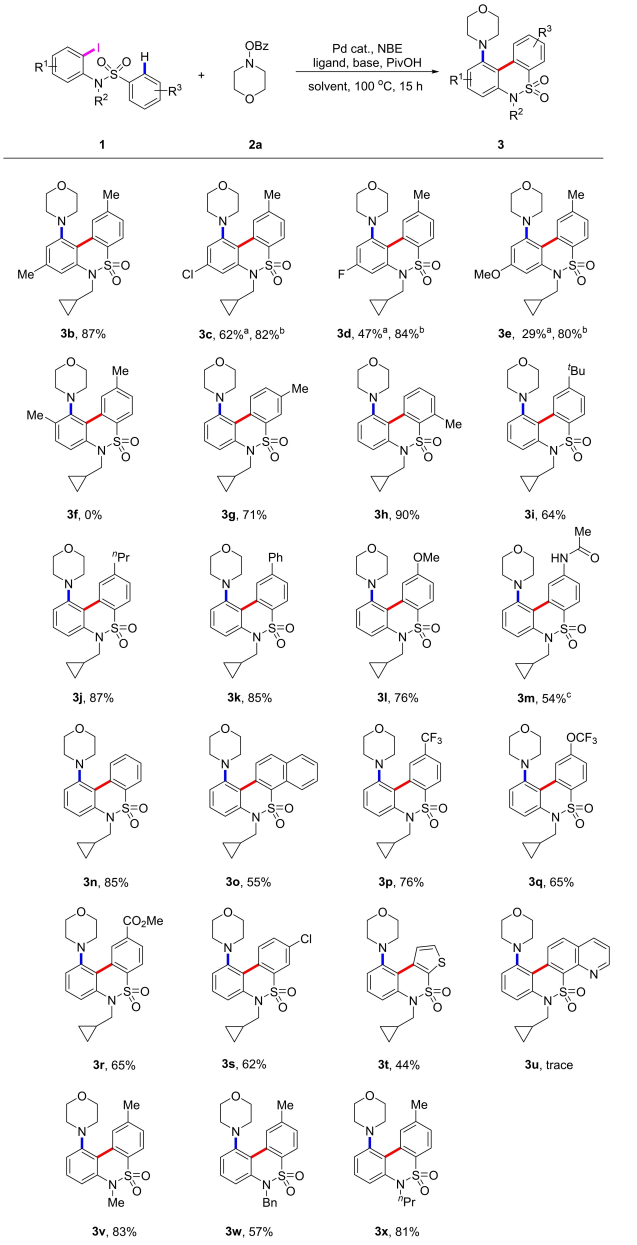
[a] Unless otherwise noted, all reactions were carried out using **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.3 mmol, 1.5 equiv.), PivOH (0.1 mmol, 0.5 equiv.), base (0.4 mmol, 2.0 equiv.), Pd catalyst (5.0 mol%), ligand (15.0 mol%), norbornene (0.6 mmol, 3.0 equiv.) in a solvent (2 mL) at 100 °C for 15 h under N_2 atmosphere.
[b] 10.0 mol% of TFP.
[c] without PivOH.

product in 74% yield. We then studied the effect of ligand and found the use of bulky biphosphine ligand XantPhos resulted in the dramatic decrease on yield (entry 7), while the addition of RuPhos had negligible influence on the reaction (entry 6). Gratifyingly, the best yield of **3a**, 90%, was achieved using tri-2-furylphosphine (TFP) as ligand (entry 8). Replacing Cs_2CO_3 with other weaker base, such as, K_3PO_4, K_2CO_3 (entries 9–12), revealed only K_3PO_4 and K_2CO_3 could promote this transformation; however, trace amount of product was detected in the case of Na_2CO_3 and KOAc as base. The nature of the solvent had a crucial role on this reaction, as demonstrated the use of polar solvents such as DMF, acetonitrile, and dioxane resulted in suppressing the reactivity (entries 13–15). Attempt to improve the yield by decreasing the loading of TFP was also not successful (entry 16). Control experiments indicated the PivOH as additive to

facilitate C–H bond activation was indispensable since a remarkable deleterious result was obtained without PivOH (entry 17), and the combination of other palladium salts including $Pd(OAc)_2$, $PdCl_2$, $\{Pd(C_3H_5)Cl\}_2$, $Pd_2(dba)_3$ with TFP led to decreased yields to some extent, evidencing the unique role of this dual ligands system, though the reason remains unclear (entries 18–21). Finally, the reaction condition listed in entry 8 was selected as the optimum conditions.

Having identified the optimal conditions, we first explored potential arylsulfonamides **1** that were compatible in this Pd/NBE cooperatively catalysed process by coupling with *O*-benzoylhydroxylmorpholine **2a** as shown in Table 2. Generally, a broad range of aryl iodide tethered arylsulfonamides was proven to be competent substrates, furnishing corresponding sultam products in moderate to good yields. Electron-donating substituent, methyl, at *para*-position of aryl iodide moiety reacted smoothly to give **3b** in synthetically useful yield. Furthermore, electron-withdrawing groups including chlorine **3c** and fluorine **3d**, which could serve as points for further derivatization, at *para*-position were also amenable to this transformation, albeit moderate results obtained. However, only 29% yield of **3e** was afforded along with 56% recovery of substrate **1e** under initial reaction conditions. To our delight, using $PdCl_2$ instead of $Pd(PPh_3)_4$ as precatalyst could greatly increase the formation of **3c**, **3d** and **3e**. Reaction with *meta*-methyl substituted substrate **3f** was unsuccessful, presumably due to the strong steric hinderance. Substituents on sulfonamide moiety were next examined. Substrates bearing electron-donating functionalities at *ortho*- (**3h**), *meta*- (**3g**), and *para*- position (**3i**–**3l**) proceeded readily to furnish target products in satisfactory yields. Notably, substrate with amide group was viable to afford final product **3m**. Utilizing the unsubstituted phenyl and 1-naphthyl sulfonamides gave desired coupling products **3n** and **3o** in 85% and 55% yields, respectively. In addition to electro-rich substrates, electron-deficient sulphonamides bearing biological relevant trifluoromethyl, trifluoromethoxyl or ester groups could also be applicable to the catalytic system, with corresponding products (**3p, 3q** and **3r**) obtained in decent yields. Chlorinated sulfonamide was also viable coupling partner to give a 62% yield under standard conditions (**3s**). It was noteworthy that C–H activation of heterocycle could be feasible as exemplified in the case of **3t**, albeit the target product isolated in moderate yield. However, substrate bearing quinoline (**3u**) proved to be challenging, furnishing trace amount of product. Finally, the survey of various substituents at nitrogen indicated methyl (**3v**) and *n*-propyl (**3x**) were all suitable for this transformation and afforded products in good yields. Good selectivity was observed in **3w**, since C–H activation event selectively took place at sulfonyl-tethered aryl moiety with benzyl intact.

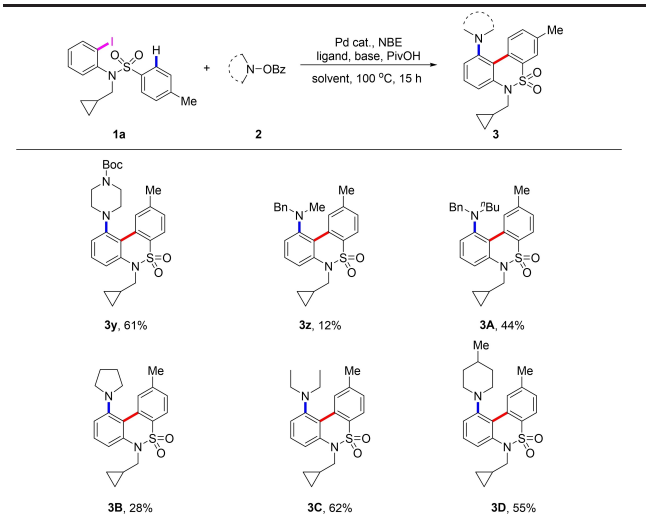
Table 2. Substrate Scope with Respect to Aryl Iodides.[a]



[a] Unless otherwise noted, all reactions were carried out using **1** (0.2 mmol, 1.0 equiv.), **2a** (0.3 mmol, 1.5 equiv.), PivOH (0.1 mmol, 0.5 equiv.), Cs_2CO_3 (0.4 mmol, 2.0 equiv.), $Pd(PPh_3)_4$ (5.0 mol%), TFP (15.0 mol%), norbornene (0.6 mmol, 3.0 equiv.) in toluene (2 mL) at 100 °C for 15 h under N_2 atmosphere.
[b] $PdCl_2$ (5.0 mol%) was used.
[c] Inseparable impurities were contained.

Next, the substrate scope with respect to various electrophilic amination reagents **2** was investigated as shown in Table 3. Various structurally distinct *O*-benzoylhydroxylamines could be applied to this trans-

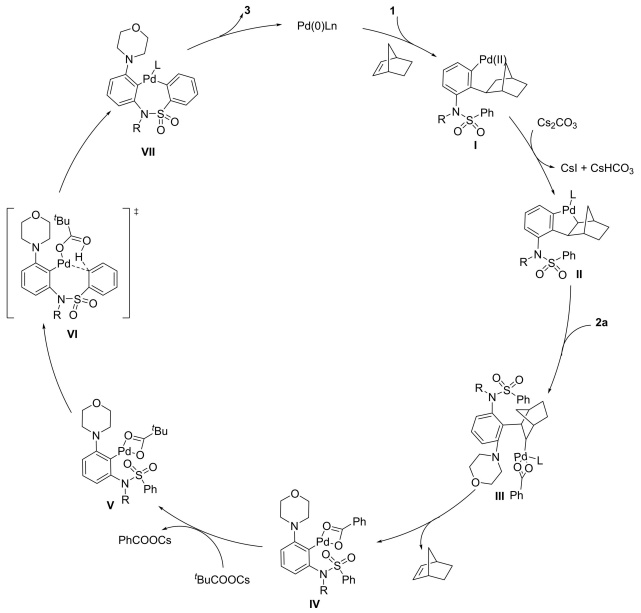
Table 3. Substrate Scope of *O*-Benzoylhydroxylamines.[a]



[a] Unless otherwise noted, all reactions were carried out using **1a** (0.2 mmol, 1.0 equiv.), **2** (0.3 mmol, 1.5 equiv.), PivOH (0.1 mmol, 0.5 equiv.), Cs_2CO_3 (0.4 mmol, 2.0 equiv.), Pd$(PPh_3)_4$ (5.0 mol%), TFP (15.0 mol%), norbornene (0.6 mmol, 3.0 equiv.) in toluene (2 mL) at 100 °C for 15 h under N_2 atmosphere.

formation, delivering target products in moderate yields. *N*-Boc protected piperazinyl could be successfully appended at *ortho*-position, with aminated product **3y** obtained in 61% isolated yield. *O*-benzoyl-*N*,*N*-diethylhydroxylamine and 4-methylpiperidinyl benzoate exhibited comparable efficiency, giving corresponding products **3C** and **3D** in 62% and 55% yields, respectively. As for **3A**, the reaction proceeded smoothly with slightly decreased yield obtained. Unfortunately, attempts to synthesize *N*-benzyl-*N*-methyl and pyrrolidinyl products **3z** and **3B** were challenging, and desired products were obtained in relatively low yields.

Based on the previous work as well as our own experimental results, the following plausible mechanistic scenario is proposed (Scheme 2). Substrate **1** undergoes oxidative addition to generate aryl-Pd(II) species **I**, which is followed by carbopalladation of norbornene, and subsequent Cs_2CO_3-mediated *ortho* C–H bond activation to generate the crucial aryl-norbornyl-palladacycle **II** (ANP) intermediate. Next, two possible pathways might occur to form C–N bond: either a Pd(IV) process arising from oxidative addition of *N*-benzoyloxyamine **2** with the ANP intermediate **II** and then reductive elemination or via direct electrophilic amination of *N*-benzoyloxyamine **2** with ANP intermediate **II**, both affording aminated arene **III**. After extrusion of norbornene via β-carbon elimination of intermediate **III**, the κ^2 – benzoic acid intermediate **IV** is formed. A pivalic acid ligated intermediate **V** might be delivered via a rapid acid exchange in which



Scheme 2. Proposed Reaction Mechanism.

benzoic acid is replaced by cesium pivalate generated from the reaction of PivOH with Cs_2CO_3. A subsequent acid promoted concerted metalaltion deprotonation (CMD)[27] process via transition state **VI** takes place, furnishing intermediate **VII**, which proceeds through reductive elimination to give rise to the desired product **3** and regenerate Pd(0).

In conclusion, we have developed a practical and efficient method for the construction of bioactive aminated benzosultams under mild conditions through *ortho*-amination/*ispo*-C(sp^2)–H arylation reaction via Palladium/norbornene cooperative catalysis. This protocol has a wide substrate scope in both coupling partners and exhibits a high functional group tolerance. Control experiment reveals pivalic acid as an additive serves as essential elements to effectively promote the C–H activation in the termination step. Further application of the gained benzosultams in organic synthetic chemistry is underway in our laboratory.

Experimental Section

Typical experiment procedure for isolated products: To a 25 mL of Schlenk tube were added the sulfonamide **1** (0.20 mmol, 1.0 equiv.), *O*-benzoylhydroxylamine **2** (0.30 mmol, 1.5 equiv.), TFP (0.03 mmol, 15.0 mol%), Cs_2CO_3 (0.40 mmol, 2.0 equiv.), Pd$(PPh_3)_4$ (0.01 mmol, 5.0 mol%). The mixture was evacuated and backfilled with N_2 for three times, NBE (0.60 mmol, 3.0 equiv.), PivOH (0.10 mmol, 0.5 equiv.), and toluene (2 mL) were then added. The Schlenk tube was screw capped and put into a preheated oil bath (100 °C). After stirring for 15 h, the reaction mixture was cooled to room temperature. The reaction was diluted with EtOAc (40 mL). After washed with water (2× 15 mL) and brine (15 mL), the organic layer was dried over

anhydrous Na_2SO_4, filtered, and then concentrated under reduced pressure. The residue was purified with column chromatography on silica gel to give pure product **3**.

Acknowledgements

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