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Palladium-catalyzed oxamidation of alkenes: A new approach to benzoxazolidines

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ABSTRACT

A novel palladium catalyzed protocol for the synthesis of benzoxazolidine by the reaction sulfamidophenol and terminal alkene was developed. This oxamidation process is simple and does not require any ligand, base or inert atmosphere for the overall transformation. From control experiments, it is apparent that the cross-coupling reaction proceeds with initial formation of enesulfonamide which undergoes nucleopalladation by the intramolecular annulation and subsequent protodepalladation by TsOH to afford the benzoxazolidine.

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1. Introduction

Five-membered heterocycles having 1,3-diheteroatoms, especially oxazolidines are belonging to a biologically significant class of scaffold present in several pharmaceuticals and natural products.[1] Substituted 1,3-oxazolidines are endowed as chiral auxiliaries in asymmetric synthesis.[2] These are also found applications in the synthesis of designed medicinal agents as prodrugs for 1,2-amino alcohols or carbonyl-containing pharmacophores.[3] The classical approaches to 1,3-oxazolidine motif include (i) acid-catalyzed condensation of aldehydes or ketones with amino alcohols[4]; (ii) cycloaddition reaction of aziridines or azomethine ylides with carbonyl compounds[5]; and (iii) phosphine-catalyzed double Michael addition of amino alcohols with terminal alkynes.[6] Very recently, an oxidative cross-coupling reaction of N-alkyl C–H bond with alkyl O–H for the construction of the functionalized oxazolidines by using TBAI in the presence of TBHP was disclosed.[7] Consequently, an alternative route involving the transition metal catalyzed C-N and C-O bond formation reaction has been emerged. For instance, the Pd-catalyzed carboamination of O-vinyl-1,2-amino alcohol derivatives leading to 1,3-oxazolidine was reported by Wolfe.[8a] Jarvo exploited the transition metal mediated reaction of butadiene monoxide and aryl imines for the enantioselective synthesis of 1,3-oxazolidines.[8b] Copper(II) as well as iron-catalyzed formal aminohydroxylation of the alkene to accomplish 1,3-oxazolidines from the reaction of styrenes and oxaziridines was described by Yoon and co-workers.[8c-f] Synthesis of oxazolidines by Pd-catalyzed aza-Wacker reaction of electron deficient terminal alkenes was reported by Lloyd-Jones and Booker-Milburn.[8g] Contrastingly, though the benzannulated five-membered rings with 1,3- diheteroatoms are found application in medicinal and material chemistry,[9] there appear to be very few precedents for benzoxazolidine synthesis and none that proceed by benzoxamination of alkenes. Gratifyingly, Surya Prakash and Olah[10] achieved one-pot synthesis of fluorinated benzoxazolidines through gallium(III) triflate mediated condensation of 2-aminophenol and carbonyl compounds (Scheme 1). Kwon[11] exploited base-catalyzed double-Michael reactions of allenes with sulfamidophenols to produce C2-functionalized benzoxazolidines. An unprecedented pyrrolidine catalyzed [4+1]-annulation of ynals with *N*-protected-2-aminophenols to produce benzoxazolidines was disclosed by Wang and co-workers.[12] In continuation to our research interest on transition metal-catalyzed heterocycles synthesis,[13] here we first report a Pd-catalyzed straightforward method for the synthesis of benzoxazolidines from the reaction of *N*-sulfonylamino phenols and electron deficient olefins under ligand-free conditions (Scheme 1).

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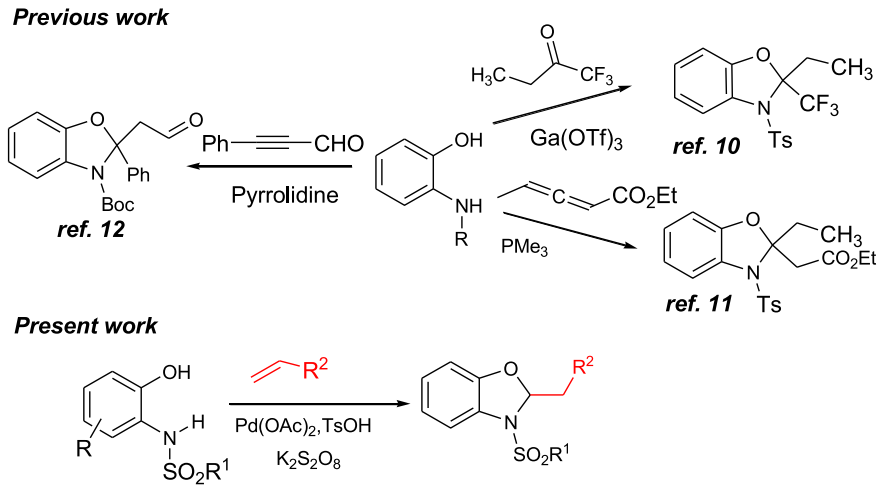
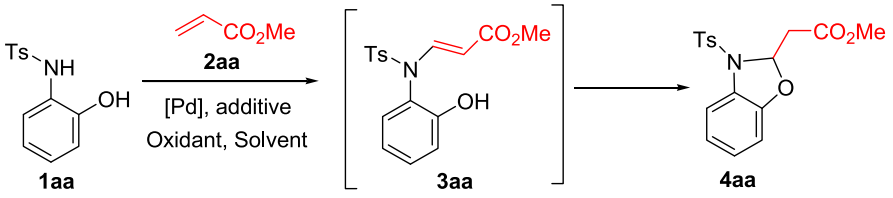


Scheme 1. Synthesis of benzoxazolidine.

2. Results and discussion

Our work initiated with the oxamidation of an electron deficient olefin i.e., methyl acrylate (**2aa**) by sulfamidophenol (**1aa**) in the presence of $Pd(CH_3CN)Cl_2$ catalyst by following a similar procedure reported by Lloyd-Jones and Booker-Milburn[8g] for the synthesis of oxazolidine. Though, it appears to be the logical extension of earlier work; unfortunately, no benzoxazolidine (**4aa**) was obtained with complete recovery of starting material (Table 1, entry 1). This failure is probably due to the poor nucleophilicity of sulfamidophenol (**1aa**).

Table 1
Optimization of reaction conditions.[a]



Entry	catalyst	oxidant	additive	solvent	% yield
1	$Pd(CH_3CN)_2Cl_2$	BQ (1 equiv)	TsOH	DME	n.r.
2	$Pd(OAc)_2$	$K_2S_2O_8$(1 equiv)	TsOH	toluene	<10
3	$Pd(OAc)_2$	AgOAc(1 equiv)	TsOH	toluene	n.r.
4	$Pd(OAc)_2$	Ag_2CO_3(1 equiv)	TsOH	toluene	n.r.
5	$Pd(OAc)_2$	$Cu(OAc)_2$(1 equiv)	TsOH	toluene	n.r.
6	$Pd(OAc)_2$	*m*CPBA(1 equiv)	TsOH	toluene	n.r.
7	$Pd(OAc)_2$	$K_2S_2O_8$ (3 equiv)	TsOH	toluene	40
8	$Pd(OAc)_2$	BQ (3 equiv)	TsOH	toluene	n.r.
9	$Pd(OAc)_2$	Oxone (3 equiv)	TsOH	toluene	31
10	$Pd(OAc)_2$	$KBrO_3$(3 equiv)	TsOH	toluene	18
11	$Pd(OAc)_2$	$NaIO_4$(3 equiv)	TsOH	toluene	22
12	$Pd(OAc)_2$	$K_2S_2O_8$ (5 equiv)	TsOH	toluene	48
13	**$Pd(OAc)_2$**	**$K_2S_2O_8$(10 equiv)**	**TsOH**	**toluene**	**78**
14	$Pd(OAc)_2$	$K_2S_2O_8$(10 equiv)	AcOH	toluene	n.r.
15	$Pd(OAc)_2$	$K_2S_2O_8$(10 equiv)	CF_3CO_2H	toluene	0
16	$Pd(OAc)_2$	$K_2S_2O_8$(10 equiv)	PivOH	toluene	0
17	$Pd(OAc)_2$	$K_2S_2O_8$(10 equiv)	MsOH	toluene	trace
18	$Pd(OAc)_2$	$K_2S_2O_8$(10 equiv)	NaOAc	toluene	n.r.
19	$Pd(PPh_3)_4$	$K_2S_2O_8$(10 equiv)	TsOH	toluene	trace
20	Pd/C	$K_2S_2O_8$(10 equiv)	TsOH	toluene	n.r.
21	$Pd(CH_3CN)_2Cl_2$	$K_2S_2O_8$(10 equiv)	TsOH	toluene	35
22	$PdCl_2$	$K_2S_2O_8$(10 equiv)	TsOH	toluene	52
23	$Pd(OAc)_2$	$K_2S_2O_8$(10 equiv)	TsOH	DCE	60
24	$Pd(OAc)_2$	$K_2S_2O_8$ (10 equiv)	TsOH	DMF	0
25	$Pd(OAc)_2$	$K_2S_2O_8$ (10 equiv)	TsOH	DMSO	0
26	$Pd(OAc)_2$	$K_2S_2O_8$ (10 equiv)	TsOH	dioxane	trace
27	$Pd(OAc)_2$	$K_2S_2O_8$ (10 equiv)	TsOH	CH_3CN	0
28	$Pd(OAc)_2$	$K_2S_2O_8$ (10 equiv)	TsOH	DME	trace

[a] *Reaction conditions*: **1a** (50 mg, 0.19 mmol), methyl acrylate (49 mg, 0.57 mmol), catalyst (10 mol%), oxidant, additive (3 equiv.), solvent (3 mL), 60 °C, 16 h. n. r. = no reaction.

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