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Iodine(III)-Mediated Oxidative Cross-Coupling of Enamines and Propargylamines under Metal-Free Conditions: An Alternative Way to Prepare Highly Substituted 3-Pyrrolines

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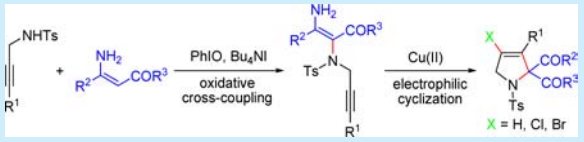
Supporting Information

ABSTRACT: A PhIO/Bu_4NI-mediated oxidative cross-coupling reaction between enamines and propargylamines under metal-free conditions has been developed. Bu_4NI works as an activator of PhIO. The resulting coupling products are ready to undergo copper(II)-mediated electrophilic cyclization to form highly substituted 3-pyrrolines.



Oxidative amination of an sp^2 C−H bond of arenes or alkenes provides a straightforward and efficient approach to prepare nitrogen-containing compounds.[1] Recently, due to the benign environmental character and versatility of polyvalent iodine compounds,[2] iodine(III)-mediated oxidative C(sp^2)−H amination under metal-free conditions has attracted considerable interest. For instance, the groups of Chang,[3] DeBoef,[4] and Antonchick[5] have developed intermolecular oxidative amination of simple arenes with a variety of nitrogen nucleophiles by using (diacetoxyiodo)benzene (DIB) or the in situ generated iodine-(III) compound as a promoter. Under similar conditions, the intramolecular oxidative amination of aromatic C−H bonds has also been developed to construct a range of heterocyclic compounds such as carbazoles,[6] benzimidazoles,[7] indeno-1,4-diazepinones,[8] benzimidazolin-2-ones,[9] benzooxazolones,[10] and indoles.[11] These elegant tactics have become good alternatives to the transition-metal-catalyzed oxidative amination reaction.[12,13] However, compared with the extensive studies on the oxidative amination of aromatic C−H bonds, iodine(III)-mediated oxidative cross-coupling reaction between alkenes and nitrogenous compounds has received much less scrutiny. For a medicinal chemistry project, we were curious to investigate the oxidative cross-coupling reaction of enamines and propargylamines. We envisaged that the resulting coupling product might be a potential precursor to highly substituted 3-pyrrolines (Scheme 1), which are present as key substructure in a number of pharmaceutically and biologically active compounds.[14,15]

To begin our study, *N*-Ts-protected propargylamine **1** and enamine **2** were used as the standard substrates to search for suitable coupling conditions. Preliminary tests using (diacetoxyiodo)benzene, [bis(trifluoroacetoxy)iodo]benzene (BTI), [hydroxy(tosyloxy)iodo]benzene (HTIB), or iodosylbenzene as a promoter did not lead to the formation of the coupling product (Table 1, entries 1−4). In our previous works,[16] we found that the addition of a bromide or an iodide salt could promote the iodine(III)-mediated oxidative cyclization reaction. Therefore, we examined the reaction using a combination of an iodine(III) compound and a salt. When $PhI(OAc)_2$ was used together with 2 equiv of Bu_4NI, although the reaction was still complex, the desired product **3** was isolated in 7% yield (Table 1, entry 5). To our delight, the yield was improved to 45% when PhIO was used instead of $PhI(OAc)_2$ (Table 1, entry 7). The formation of compound **3** was not observed when Bu_4NI was replaced by Bu_4NBr, Bu_4NCl, KI, or NaI (Table 1, entries 8−11). Very recently, Zhao and Du reported a Bu_4NI/TBHP-mediated oxidative coupling of enamines and electron-deficient amines.[17] However, the reaction of propargylamine **1** and enamine **2** under the reported conditions did not give rise to compound **3** (Table 1, entry 12). By screening solvents, temperatures, and the ratios of substrates and reagents, the optimized reaction conditions were established, and the isolated yield of the coupling product was increased to 87% (Table 1, entry 22).

The *N*-protecting group of propargylamine played a significant role in the oxidative cross-coupling reaction (Scheme 2). The reaction of *N*-Ms protected propargylamine gave rise to product

Scheme 1. Construction of 3-Pyrrolines via Iodine(III)-Mediated Oxidative Coupling and Subsequent Cyclization

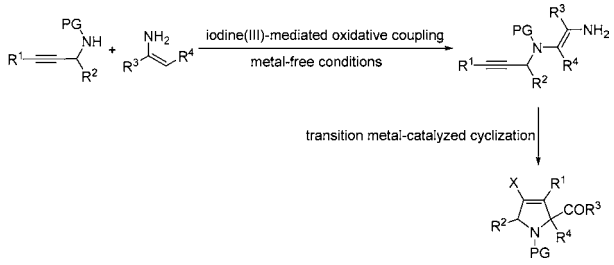


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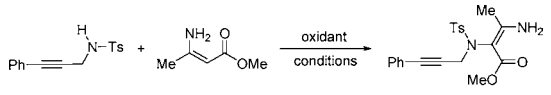
Table 1. Evaluation of Conditions

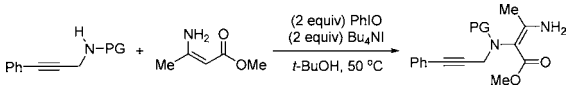


1 2 3

entry	oxidant (equiv)	conditions	3[a] (%)
1	$PhI(OAc)_2$ (2)	CH_3CN, 30 °C	0
2	$PhI(OCOCF_3)_2$ (2)	CH_3CN, 30 °C	0
3	PhI(OH)OTs (2)	CH_3CN, 30 °C	0
4	PhIO (2)	CH_3CN, 30 °C	0
5	$PhI(OAc)_2$ (2), Bu_4NI (2)	CH_3CN, 30 °C	7
6	$PhI(OCOCF_3)_2$ (2), Bu_4NI (2)	CH_3CN, 30 °C	0
7	PhIO (2), Bu_4NI (2)	CH_3CN, 30 °C	45
8	PhIO (2), Bu_4NBr (2)	CH_3CN, 30 °C	0
9	PhIO (2), Bu_4NCl (2)	CH_3CN, 30 °C	0
10	PhIO (2), NaI (2)	CH_3CN, 30 °C	0
11	PhIO (2), KI (2)	CH_3CN, 30 °C	0
12	Bu_4NI (0.3), TBHP (5)	DMF, 100 °C	0
13	PhIO (2), Bu_4NI (2)	$Cl(CH_2)_2Cl$, 30 °C	47
14	PhIO (2), Bu_4NI (2)	toluene, 30 °C	40
15	PhIO (2), Bu_4NI (2)	THF, 30 °C	33
16	PhIO (2), Bu_4NI (2)	DMF, 30 °C	39
17	PhIO (2), Bu_4NI (2)	CF_3CH_2OH, 30 °C	0
18	PhIO (2), Bu_4NI (2)	*t*-BuOH, 30 °C	54
19	PhIO (2), Bu_4NI (2)	*t*-BuOH, 50 °C	63
20	PhIO (2), Bu_4NI (2)	*t*-BuOH, reflux	51
21[b]	PhIO (2), Bu_4NI (2)	*t*-BuOH, 50 °C	75
22[b,c]	PhIO (1.5 + 0.5), Bu_4NI (1.5 + 0.5)	*t*-BuOH, 50 °C	87

[a]Isolated yields. [b]3 equiv of enamine **2** was used. [c]0.5 equiv of PhIO and 0.5 equiv of Bu_4NI were added after 2 h.

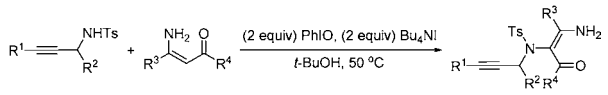
Scheme 2



PG = Ts **3** (87%)
PG = MS **4** (55%)
PG = Ac **5** (0%)
PG = $COCF_3$ **6** (0%)
PG = Bz **7** (0%)
PG = Bn **8** (0%)

4 in 55% yield. When *N*-acetyl-, *N*-benzoyl-, or *N*-benzyl-protected propargylamine was used as substrate, the reaction under the optimized conditions did not afford the expected coupling product. The scope of this PhIO/Bu_4NI-mediated oxidative cross-coupling reaction was then investigated, and the representative results are summarized in Table 2. The reaction of propargylamines could tolerate a range of substituted groups at the C-3 position involving electron-rich and electron-poor aryl groups, 2-thienyl group, ethyl group, or a hydrogen atom (Table 2, entries 1−12). When propargylamine bears a methyl or a phenyl group at the C-1 position, the corresponding coupling product was not obtained (Table 2, entries 13 and 14). The failure of the coupling reaction might result from the steric hindrance of the R^2 group. The methyl group of enamines was not indispensible for the cross-coupling reaction. The R^3 substituent could be an aryl group (Table 2, entries 15−17). When enamine derived from 4,4,4-trifluoro-3-oxobutanoate was used as the reaction partner, no coupling product was formed (Table 2, entry 18). The electron-withdrawing group of enamines could be not only a methoxycarbonyl group but also be acyl group (Table 2, entries 19−21). For enamine-bearing two

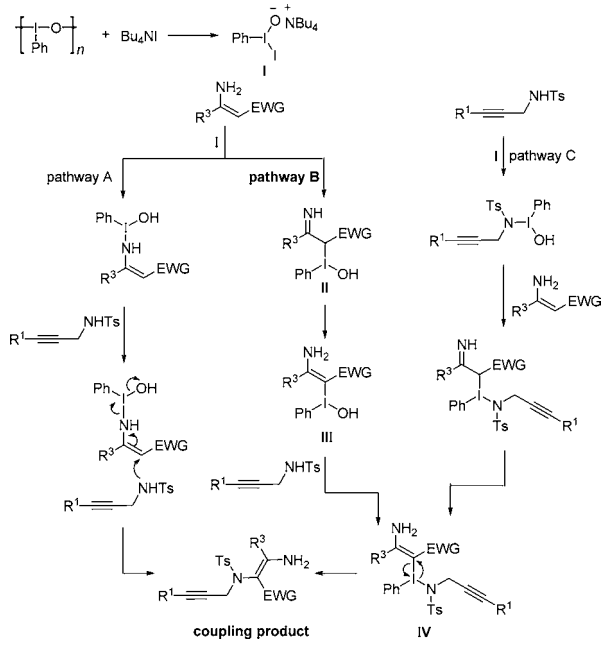
Table 2. Scope Investigation on Oxidative Cross-Coupling



entry	R^1	R^2	R^3	R^4	yield[a] (%)
1	Ph	H	Me	OMe	**3** (87)
2	4-MeC_6H_4	H	Me	OMe	**9** (78)
3	2-MeC_6H_4	H	Me	OMe	**10** (80)
4	4-MeOC_6H_4	H	Me	OMe	**11** (35)
5	4-ClC_6H_4	H	Me	OMe	**12** (88)
6	4-FC_6H_4	H	Me	OMe	**13** (76)
7	$4\text{-EtO}_2CC_6H_4$	H	Me	OMe	**14** (81)
8	4-CNC_6H_4	H	Me	OMe	**15** (58)
9	2-naphthyl	H	Me	OMe	**16** (84)
10	2-thiophene	H	Me	OMe	**17** (67)
11	Et	H	Me	OMe	**18** (59)
12	H	H	Me	OMe	**19** (75)
13	Ph	Me	Me	OMe	**20** (0)
14	Ph	Ph	Me	OMe	**21** (0)
15	Ph	H	Ph	OEt	**22** (67)
16	Ph	H	4-ClC_6H_4	OMe	**23** (68)
17	Ph	H	4-FC_6H_4	OMe	**24** (61)
18	Ph	Me	CF_3	OMe	**25** (0)
19	Ph	H	Me	Me	**26** (84)
20	Ph	H	Me	Ph	**27** (49)
21	Ph	H	Me	4-BrC_6H_4	**28** (51)
22	Ph	H	Ph	Ph	**29** (<5)

[a]Isolated yields.

Scheme 3. Mechanistic Hypothesis



phenyl groups, the reaction was sluggish under the same conditions (Table 2, entry 22). When *N*-Me- or *N*-Bn-protected enamine was employed, no coupling product was isolated.

To further demonstrate the scope of this iodine(III)-mediated oxidative coupling, *N*-Ts-protected benzylamine and butylamine were examined. The reaction of benzylamine gave rise to the corresponding product **30** in 37% yield, but the reaction of butylamine was complex. In addition, we also examined the

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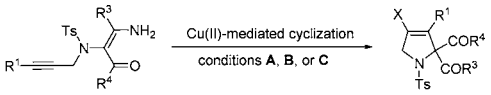
Table 3. Construction of Highly Substituted 3-Pyrrolines[a]



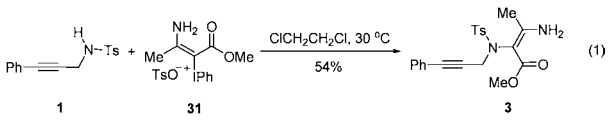
conditions **A**: (0.2 equiv) $Cu(hfacac)_2 \cdot H_2O$, $ClCH_2CH_2Cl$, 70 °C X = H
conditions **B**: (2 equiv) $CuCl_2$, $ClCH_2CH_2Cl$, 25 °C X = Cl
conditions **C**: (2 equiv) $CuBr_2$, $ClCH_2CH_2Cl$, 25 °C X = Br

entry	R^1	R^3	R^4	X	.product[b] (%)
1	Ph	Me	OMe	H	**32** (85)
2	Ph	Me	OMe	Cl	**33** (61)
3	Ph	Me	OMe	Br	**34** (95)
4	4-MeC_6H_4	Me	OMe	H	**35** (78)
5	4-MeC_6H_4	Me	OMe	Cl	**36** (50)
6	4-MeC_6H_4	Me	OMe	Br	**37** (79)
7	4-ClC_6H_4	Me	OMe	Cl	**38** (65)
8	4-ClC_6H_4	Me	OMe	Br	**39** (96)
9	4-FC_6H_4	Me	OMe	H	**40** (73)
10	4-FC_6H_4	Me	OMe	Br	**41** (72)
11	4-$EtO_2CC_6H_4$	Me	OMe	H	**42** (81)
12	4-$EtO_2CC_6H_4$	Me	OMe	Cl	**43** (84)
13	4-$EtO_2CC_6H_4$	Me	OMe	Br	**44** (83)
14	4-CNC_6H_4	Me	OMe	Br	**45** (88)
15	2-naphthyl	Me	OMe	H	**46** (93)
16	2-thiophene	Me	OMe	Br	**47** (69)
17	Et	Me	OMe	H	**48** (65)
18	H	Me	OMe	H	**49** (88)
19	Ph	Ph	OEt	Br	**50** (81)
20	Ph	4-ClC_6H_4	OMe	Br	**51** (89)
21	Ph	4-FC_6H_4	OMe	Br	**52** (87)
22	Ph	Me	Me	Br	**53** (75)
23	Ph	Me	Me	H	**54** (83)
24	Ph	Me	Me	Cl	**55** (76)
25	Ph	Me	Ph	Br	**56** (75)
26	Ph	Me	4-BrC_6H_4	Br	**57** (69)

[a]$Cu(hfacac)_2 \cdot H_2O$, hfacac = hexafluoroacetylacetonate. [b]Isolated yields.

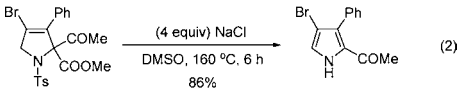
reactions of phthalimide and succinimide, which have proved to be suitable partners in Bu_4NI/TBHP-mediated oxidative coupling. No reaction was observed under the standard conditions.

At least three reaction pathways are plausible for this PhIO/Bu_4NI-mediated oxidative cross-coupling reaction (Scheme 3). With the aid of tetrabutylammonium iodide, polymeric iodosobenzene is depolymerized leading to the formation of a new iodine(III) species **I**. This iodine(III) compound might be highly reactive in the cross-coupling reaction because its oxygen anion can act as a base to promote the deprotonation of substrate and the iodide is a good leaving group. To understand the reaction pathway, we prepared an alkenyl iodonium salt **31**.[18] The reaction of this salt with propargylamine **1** in dichloroethane at 50 °C gave rise to compound **3** in 54% yield (eq 1). This result indicated that the oxidative cross-coupling reaction might proceed through the pathway **B**. The ligand-exchange reaction of the in situ generated iodine(III) compound **I** with enamines produces an alkenyl iodonium salt **III**. The nucleophilic attack of propargylamine to this intermediate forms an intermediate **IV**. After a reductive elimination, the expected coupling product was formed.



1 **31** **3**

As shown in Table 3, treating the resulting cross-coupling product with 0.2 equiv of $Cu(hfacac)_2 \cdot H_2O$ in dichloroethane at 70 °C led to the conversion to 3-pyrrolines (X = H). Moreover, when 2 equiv of copper(II) chloride or copper(II) bromide was used as promoter, the electrophilic cyclization was followed by a reductive elimination, and the reaction gave rise to 3-chloro- or 3-bromo-substituted 3-pyrrolines in moderate to excellent yields. When the obtained 3-pyrroline **34** was mixed with 4 equiv of sodium chloride in dimethyl sulfoxide at 160 °C for 6 h,[19] it was converted to trisubstituted pyrrole **58** in 86% yield (eq 2).



34 **58**

In conclusion, we have developed an iodine(III)-mediated oxidative cross-coupling reaction of enamines and propargylamines under metal-free conditions. Bu_4NI works as an activator of PhIO. Subsequent copper(II)-mediated cyclization of the resulting cross-coupling products affords an alternative way to prepare highly substituted 3-pyrrolines. Detailed studies on the reaction mechanism and extension of the reaction scope are now underway.

■ ASSOCIATED CONTENT

Supporting Information

Experimental procedures, characterization data, and copies of ^1H NMR and ^{13}C NMR of new compounds. This material is available free of charge via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

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