Tetrahedron Letters 57 (2016) 1239–1242

Contents lists available at ScienceDirect

Tetrahedron Letters

journal homepage: www.elsevier.com/locate/tetlet

A metal-free addition/dearomatization–cyclization/decarboxylation cascade of aryl 3-arylpropiolate toward the synthesis of 1,1-diaryl-2-alkyl-substituted ethylenes

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ARTICLE INFO

Article history:
Received 1 December 2015
Revised 29 January 2016
Accepted 3 February 2016
Available online 4 February 2016

Keywords:
Tandem reaction
1,1,2-Trisubstituted ethylenes
Aryl 3-arylpropiolate
Decarboxylation

ABSTRACT

A radical-participated addition/dearomatization–cyclization/decarboxylation cascade between aryl 3-arylpropiolate and ethers (cycloalkane) has been developed to give a series of 1,1,2-trisubstituted ethylenes in moderate to good yields.

The synthesis of multisubstituted alkenes is an evergreen topic in the field of synthetic chemistry as these types of structural moieties broadly exist in a number of bioactive molecules and important synthetic intermediates such as Tamoxifen, Claravis, Tamiflu, and Sutent (Fig. 1). Different from the synthesis of mono- or di-substituted alkenes, whose synthetic methodologies have been well established, developing new methods for the synthesis of tri- or tetra-substituted alkenes is still far more underdeveloped as there is lack of general methods for this problem.[1] After years of efforts of synthetic chemists, three common strategies, elimination from proper alkanes, addition to the alkynes, and substitution of alkenes have been successfully applied to this subject. Especially, under the stimulation of the C–H functionalization, a number of methods for the construction of this type of moiety have been developed through the direct functionalization of carbon–carbon double/triple bond.[2,3] Nevertheless, it is still desirable to further investigate this topic.

In recent years, difunctionalization reactions of carbon–carbon double/triple bond through an addition/cyclization cascade have received considerable attention of organic chemistry community.[4–7] In particular, for those involving the activated carbon–carbon triple bond of aryl 3-arylpropiolate type of substrate, corresponding studies have led to the construction of two major types of molecular skeletons, that is, chroman-2-one and 1-oxaspiro[4.5]deca-3,6,9-triene-2,8-dione (Scheme 1, Eqs. 1 and 2).[6,7] As for the former one (Scheme 1, Eq. 1), produced from a better studied reaction mode, a series of reagents have been successfully applied to it under either transition metal catalysis or metal-free conditions.[6] Different from the first reaction mode, the involvement of an *ipso*-cyclization would afford the products with an oxa-spiro skeleton through a dearomatization process (Scheme 1, Eq. 2).[7] Regarding to the second type of reaction, only limited number of reagents like tetrahydrofuran, the Langlois reagent, halonium reagent, and bisiodonium salts were used in the transformation. And most of them need the presence of a transition metal catalyst. In connection with our interest in the difunctionalization reaction of carbon–carbon double or triple bond,[8] our initial goal was to develop a metal-free version of the reaction between aryl 3-arylpropiolate and 1,4-dioxane with the use of proper oxidant. Different from what we expected, a trisubstituted alkene was obtained in moderate yield when $PhI(OAc)_2$ was used as the oxidant for the reaction between phenyl 3-phenylpropiolate (**1a**) and 1,4-dioxane (**2a**) (Scheme 1, Eq. 3). In our opinion, this transformation represents a new reaction mode for this type of substrate and might bring an alternative way for the synthesis of related alkenes.[9] Herein, we present the results for this tandem

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http://dx.doi.org/10.1016/j.tetlet.2016.02.013
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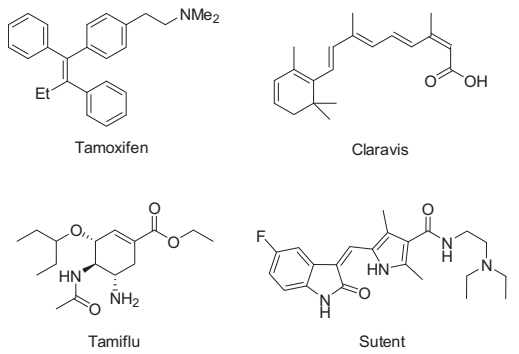
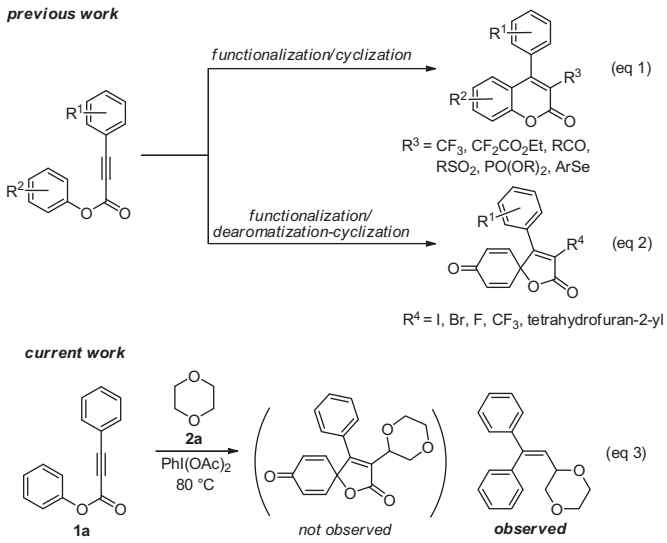


Figure 1. Selected molecules with multisubstituted double bond.

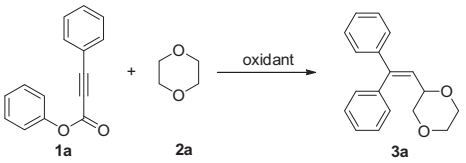
reaction, in which an addition/dearomatization–cyclization/decarboxylation cascade was involved.

After observing the initial result, we began the reaction condition optimization of this tandem transformation with phenyl 3-phenylpropiolate (**1a**) and 1,4-dioxane (**2a**) as the mode substrates. Accordingly, a series of oxidants were screened to promote this reaction at 80 °C (Table 1, entries 1–7), and most of them could afford the expected product **3a**. Among them, the use of benzoyl peroxide (BPO) could give the expected product **3a** in the highest yield of 48% (Table 1, entry 7), while no desired product could be detected in the presence of $K_2S_2O_8$ (Table 1, entry 3). Next, the reaction was carried out at different temperatures (Table 1, entries 8–11), through which the yield of the product **3a** was further increased to 64% when the transformation was performed at 100 °C (Table 1, entry 10). Having the above results, we further optimized the reaction conditions by varying the amount of the BPO (Table 1, entries 12–18). It was found that the best result was obtained in the presence of 1 equiv of BPO (Table 1, entry 17). Increasing the amount of BPO did not give a clear improvement of the reaction result (Table 1, entry 18), and the yield of **3a** was clearly decreased by reducing the amount of BPO (Table 1, entries 15 and 16). Besides, it should be noted that carrying the reaction for 2 h was enough to afford the best result (Table 1, entry 17), and no product **3a** was obtained without the use of BPO (Table 1, entry 19). The reaction was also performed under air or with less amount of 1,4-dioxane, but all these attempts led to the decrease of the reaction yield (Table 1, entries 20–22).



Scheme 1. Addition/cyclization of aryl 3-arylpropiolate.

Table 1
Optimization of reaction conditions[a]

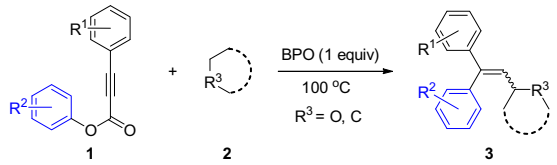


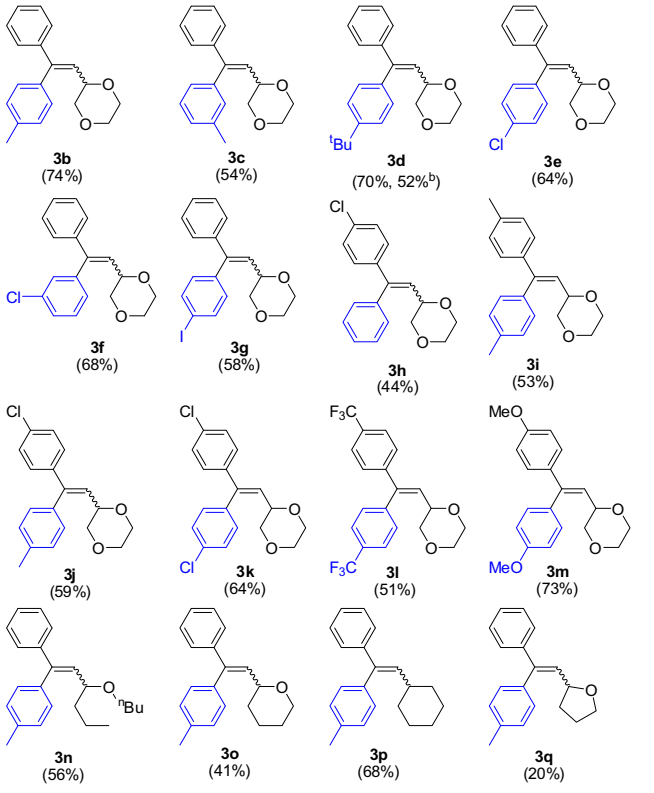
Entry	Oxidant (equiv)	Temp (°C)	*t* (h)	Yield[b] (%)
1	$PhI(OAc)_2$ (1.2)	80	12	34
2	DTBP[c] (1.2)	80	12	27
3	$K_2S_2O_8$ (1.2)	80	12	n.d.[d]
4	H_2O_2 (1.2)	80	12	16
5	TBPB[e] (1.2)	80	12	24
6	CHP[f] (1.2)	80	12	26
7	BPO (1.2)	80	12	48
8	BPO (1.2)	60	12	25
9	BPO (1.2)	90	12	59
10	BPO (1.2)	100	12	64
11	BPO (1.2)	110	12	60
12	BPO (1.2)	100	1	45
13	BPO (1.2)	100	2	63
14	BPO (1.2)	100	5	64
15	BPO (0.5)	100	3	38
16	BPO (0.8)	100	2	62
17	BPO (1)	100	2	74
18	BPO (2)	100	2	69
19	—	100	2	n.d.[d]
20[g]	BPO (1)	100	2	55
21[h]	BPO (1)	100	2	50
22[i]	BPO (1)	100	2	30

[a] Reaction conditions: without other notifications, all the reactions were performed with phenyl 3-phenylpropiolate (50 mg, 0.23 mmol) in 1,4-dioxane (1 mL) under Ar.
[b] Isolated yield.
[c] DTBP = di-*tert*-butyl peroxide.
[d] n.d. = not detected.
[e] TBPB = *tert*-butyl peroxybenzoate.
[f] CHP = cumyl hydroperoxide.
[g] The reaction was carried out under air.
[h] 1,4-Dioxane (0.5 mL) was used.
[i] 1,4-Dioxane (0.25 mL) was used.

With the optimized reaction conditions in hand (Table 1, entry 17), we further evaluated the generality of this transformation. As shown in Table 2, most of the substrates tested could afford the expected products in moderate to good yields. From the results, it is clear that the position of substituent of R^2 affected the yield of corresponding products. Taking methyl as an example, when it was at *para*-position, the expected product **3b** could be produced in 74% yield. And a lower yield of 54% was obtained when the same group was at *meta*-position. In contrast, no desired product could be produced with a change of the methyl group to the *ortho*-position. Furthermore, there is no clear electron effect of the substituent R^2. Besides the methyl group, when the R^2 group was *para*-^{t}Bu, *para*-chloro, *para*-iodo, *para*-trifluoromethyl, or *para*-methoxyl group, the substrates all gave the expected products in good yield (Table 2, products **3d**, **3e**, **3g**, **3l**, and **3m**). And the presence of the halogen atom in the products would provide an additional position for further derivatization. The substrates with different R^1 groups were also tested with 1,4-dioxane under the optimal reaction conditions, and the corresponding products were formed in moderate yields (Table 2, products **3h**–**3m**). Next, we attempted some other reagents that have been used in radical-involved reactions. Among them, the use of 1-butoxybutane, tetrahydropyran (THP), and cyclohexane all led to the desired products in moderate to good yields (Table 2, products **3n**–**3p**), and the use of tetrahydrofuran only afforded the expected product **3q** in a lower yield of 20%. While no desired products were

Table 2
BPO-promoted addition/*ipso*-cyclization/decarboxylation of aryl 3-arylpropiolate[a]



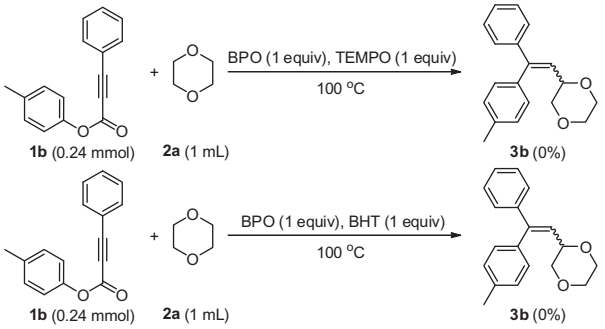


[a] Reaction conditions: without other notifications, all of the reactions were performed with **1** (0.23 mmol) in **2** (1 mL) in the presence of BPO (1 equiv) under Ar. The yields are isolated yield.
[b] The reaction was carried out with 1d (1 g, 3.6 mmol) in 1,4-dioxane (15 mL) with BPO (1 equiv) under Ar. The yield is isolated yield.

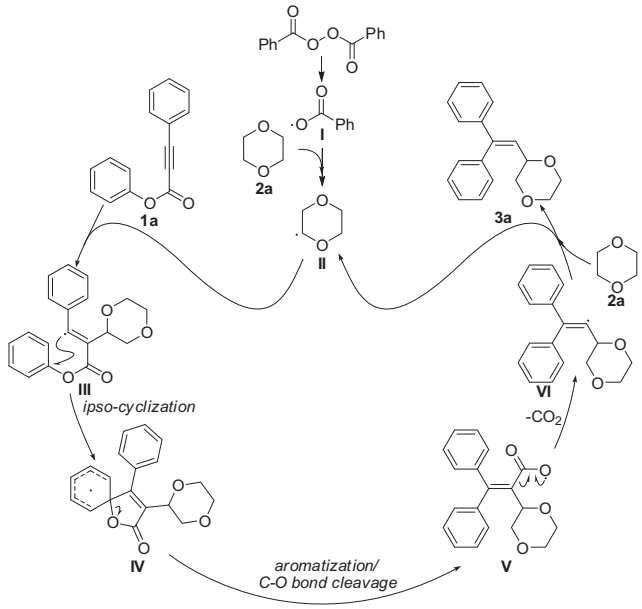
observed by using other nitrogen-containing reagents, that is, morpholine, 4-methylmorpholine, and *N*-methylpiperidine. Additionally, in order to support the practical utility of this transformation, we have carried out it in gram scale with substrate **1d** as an example, and obtained the product **3d** in a yield of 52%. It should be admitted that the stereoselectivity for the formation of carbon–carbon double bond of the desired products is not satisfactory, and the product with two different aromatic groups were all confirmed as a mixture of two *E*/*Z* isomers in about 1:1 ratio.

In order to better understand the reaction mechanism, some control experiments were carried out (Scheme 2). When the reaction between substrates **1b** and **2a** was performed in the presence of 1 equiv of TEMPO (2,2,6,6-tetramethyl-1-piperidinyloxy) or BHT (dibutylhydroxytoluene), this tandem reaction was totally inhibited. No product **3b** could be detected, and most of the **1b** was left unreacted. These experiments indicated that such a transformation might be a radical-involved process.

Based on the above experimental results, the mechanism for this reaction is proposed (Scheme 3). Using the reaction of **1a** and **2a** as an example, the reaction should be initiated by the thermal homolysis of BPO to give an oxygen-centered radical **I**,[10] which reacted with **2a** to afford radical intermediate **II** through a hydrogen abstraction process. The addition of **II** to the carbon–carbon triple bond led to the vinyl radical intermediate **III**, which would produce intermediate **IV** through an intramolecular dearomatization–cyclization. Next, the aromatization/C–O bond cleavage would afford the carboxyl radical intermediate **V**, which underwent a decarboxylation to form vinyl radical **VI**. Finally, another hydrogen abstraction of **2a** by intermediate **VI** would give the product **3a**.



Scheme 2. Preliminary mechanism studies.



Scheme 3. Plausible mechanism of the reaction.

Conclusions

In conclusion, a BPO-promoted tandem reaction between aryl 3-arylpropiolate and ether (cycloalkane) has been developed, in which a radical-participated addition/dearomatization–cyclization/decarboxylation cascade was involved. This procedure might provide an alternative strategy for the synthesis 1,1,2-trisubstituted ethylenes. Currently, further studies related to this type of transformation are on-going in the same group.

Acknowledgements

This work was supported by the NSFC (Nos. 21202073 and 21472077), the Fundamental Research Funds for the Central Universities (lzujbky-2015-k12), and the Opening Project of Key Laboratory of Drug Targeting and Drug Delivery System, Ministry of Education (Sichuan University).

Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.tetlet.2016.02.013.

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