

Ag-Catalyzed Oxidative *ipso*-Cyclization via Decarboxylative Acylation/Alkylation: Access to 3-Acyl/Alkyl-spiro[4.5]trienones

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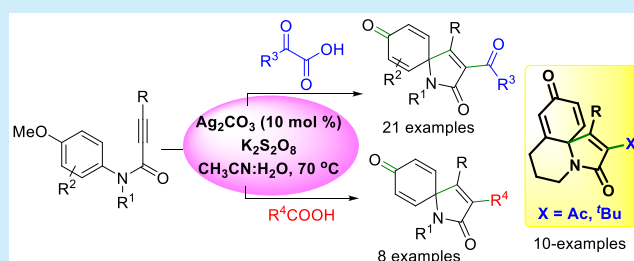
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ABSTRACT: A strategy to functionalized spiro[4.5]trienones, by domino silver-catalyzed decarboxylative acylation or alkylation/*ipso*-cyclization of *N*-arylpropiolamides with α -keto acids/alkyl carboxylic acids, is presented. This transformation offers a wide range of substituted 3-acyl/alkyl-spiro[4.5]trienones in high yields with a broad substrate scope. The approach was further extended to access fused tricyclic frameworks, 6,7-dihydro-3*H*-pyrrolo[2,1-*j*]quinoline-3,9(5*H*)-diones.



Aspirocyclohexadienones are an important molecular motif widely present in natural products (I) and biologically active compounds (II, Figure 1).¹ Indeed,

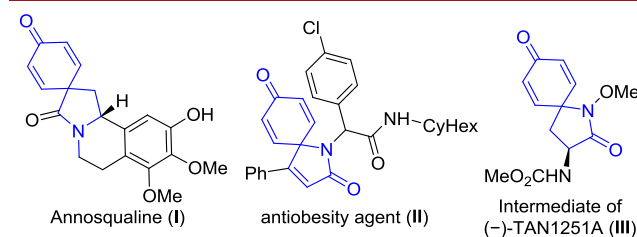


Figure 1. Representative aspirocyclohexadienones

azaspiro[4.5]dienones have been broadly utilized as a versatile intermediate (III) for constructing important alkaloids and related products.² Consequently, considerable efforts have been dedicated to construct azaspiro[4.5]trienones and several methods thereby developed.^{3–6} The electrophilic or radical cascade *ipso*-cyclization has emerged as a powerful tool for difunctionalization and dearomatization of the alkyne based aromatic compounds to provide diverse azaspiro[4.5]trienones.^{4,5} To date, two acylative *ipso*-cyclizations⁶ and one alkylative *ipso*-cyclization⁷ are known in the literature along with a few other *ipso*-carbocyclizations.⁸ In 2014, Li's group reported an oxidative *ipso*-carboacylation of *N*-arylpropiolamides with aldehydes for the preparation of 3-acylspiro[4.5]trienones at 110 °C.^{6a} Recently, Liu and Tang et al. have developed visible-light-mediated *ipso*-carboacylation of alkynes with aryl chlorides to access 3-arylspiro[4.5]trienones at 100 °C.^{6b} In 2016, Li and co-workers reported a copper-catalyzed oxidative coupling followed by *ipso*-cyclization of *N*-arylpropiolamides with unactivated alkanes (mostly cycloalkanes) for

the synthesis of 3-alkyl spiro[4.5]trienones using the TBHP as an oxidant.⁷ Regardless of their merits, the current strategies suffer from disadvantages such as high reaction temperature, limited substrate scope, and/or regioselectivity. Therefore, more general and mild approaches for the construction of diversely functionalized spiro[4.5]trienones are still of great interest.

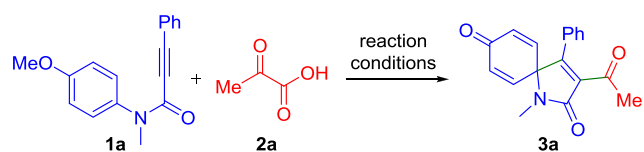
On the other hand, silver-catalyzed decarboxylative acylation/alkylation reactions are gaining prominence for the introduction of the acyl or alkyl groups, starting from α -keto acids^{3a} or alkyl carboxylic acids producing CO₂ as the waste.^{9–12} Among them, the incorporation of an in situ generated acyl or alkyl radical on to a functional group and subsequent radical cyclizations represent the best direct synthesis of corresponding cyclic frameworks.^{11,12} Nonetheless, the decarboxylative cascade addition of an acyl or alkyl radical on the alkyne followed by *ipso*-cyclization is unexplored. Our interest toward construction of valuable heterocyclic compounds via difunctionalization of alkyne¹³ provoked us to explore the above approach for the synthesis of corresponding spiro[4.5]trienones. Herein, we disclose silver-catalyzed decarboxylative *ipso*-carboacylation/alkylation of *N*-arylpropiolamide with α -keto acids/alkyl carboxylic acids to synthesize 3-acyl/alkyl spiro[4.5]trienones. The reaction strategy involves the generation of radical via decarboxylation of acids, which initiate the cascade reactions such as acylation/

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alkylation, *ipso*-annulation, dearomatization, and dehydration in a single operation.

At the onset of our investigations, the decarboxylative *ipso*-annulation of *N*-(4-methoxyphenyl)-*N*-methyl-3-phenyl-propiolamide (**1a**) with pyruvic acid (**2a**) was chosen as the model reaction to find the optimal reaction conditions (Table 1). To our delight, the initial experiment using AgNO₃ (10 mol

Table 1. Optimization of Reaction Conditions^a



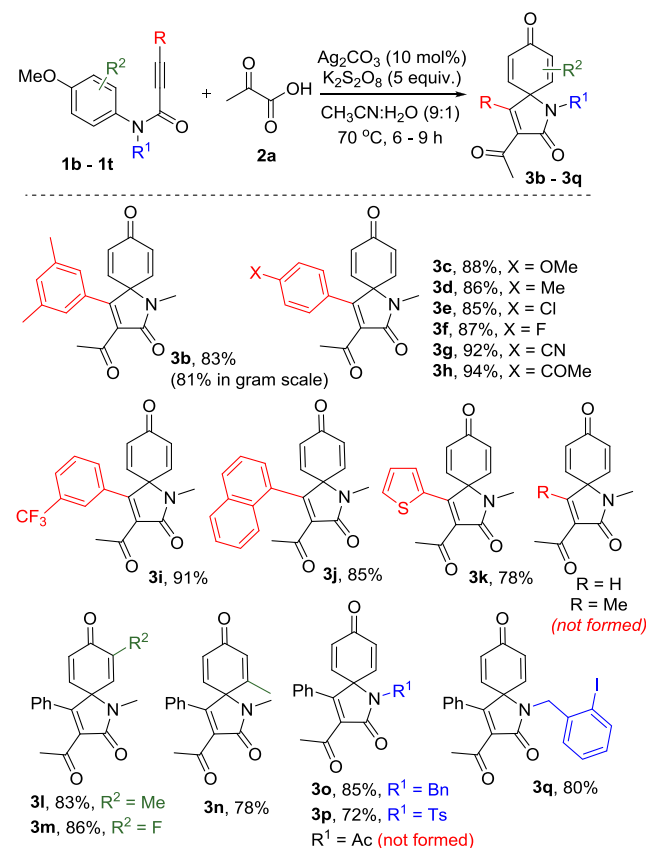
entry	catalyst (mol %)	oxidant	solvent	yield (%) ^b
1	AgNO ₃ (10)	K ₂ S ₂ O ₈	CH ₃ CN/H ₂ O	74
2	AgNO ₃ (10)	Na ₂ S ₂ O ₈	CH ₃ CN/H ₂ O	63
3	AgNO ₃ (10)	(NH ₄) ₂ S ₂ O ₈	CH ₃ CN/H ₂ O	38
4	AgNO ₃ (10)	TBHP	CH ₃ CN/H ₂ O	trace
5	AgOAc (10)	K ₂ S ₂ O ₈	CH ₃ CN/H ₂ O	46
6	AgOTf (10)	K ₂ S ₂ O ₈	CH ₃ CN/H ₂ O	42
7	Ag ₂ CO ₃ (10)	K ₂ S ₂ O ₈	CH ₃ CN/H ₂ O	94
8	Ag ₂ CO ₃ (5)	K ₂ S ₂ O ₈	CH ₃ CN/H ₂ O	81
9 ^c	Ag ₂ CO ₃ (10)	K ₂ S ₂ O ₈	CH ₃ CN/H ₂ O	52
10	None	K ₂ S ₂ O ₈	CH ₃ CN/H ₂ O	0
11	Ag ₂ CO ₃ (10)	K ₂ S ₂ O ₈	CH ₃ CN	23
12 ^d	Ag ₂ CO ₃ (10)	K ₂ S ₂ O ₈	CH ₃ CN/H ₂ O	0

^aUnless otherwise stated, all the reactions were performed using **1a** (0.3 mmol) with **2a** (0.6 mmol). K₂S₂O₈ (1.5 mmol) and catalyst in 3 mL of solvent mixture (9:1). ^bIsolated yield. ^c0.9 mmol of K₂S₂O₈ used. ^dReaction in the absence of **2a**.

%) as a catalyst and K₂S₂O₈ as the oxidant in CH₃CN:H₂O (9:1) led to the desired product **3a** in 74% yield (Table 1, entry 1). Other oxidants, including Na₂S₂O₈, (NH₄)₂S₂O₈, and TBHP, gave a reduced yield (Table 1, entries 2 to 4) of **3a** and recovery of **1a**. This might be due to the superior solubility of potassium salt in organic solvent compared to the other oxidants used.^{3b} The employment of other catalysts such as AgOAc and AgOTf resulted in the formation of product in low yields (Table 1, entries 5 and 6) along with the unreacted **1a**. Interestingly, the yield of the desired product **3a** was dramatically improved to 94%, when we switched the catalyst to 10 mol % of Ag₂CO₃ (entry 7). Diminishing yield of the product was observed with either lowering the catalyst loading to 5 mol % of Ag₂CO₃ (entry 8) or reducing the oxidant molar ratio to 3 equiv (entry 9). Notably, the spirocyclization did not happen without silver catalyst, demonstrating the significant role of the catalyst in this transformation (entry 10). In the absence of water, only 23% of **3a** was isolated, revealing that water is essential (entry 11). The failure of *ipso*-annulation of **1a** in the absence of **2a** suggests that the acyl radical is initiating the reaction (entry 12).

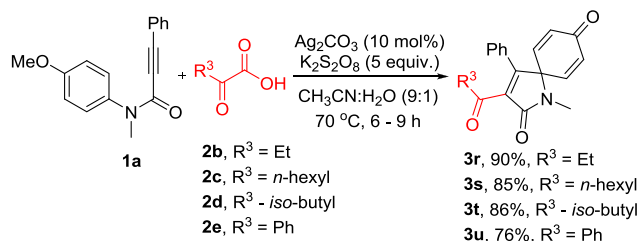
With the optimized reaction conditions in hand, the scope of this Ag-catalyzed decarboxylative dearomatization reaction was investigated using a series of substituted *N*-arylpropiolamides with **2a** (Scheme 1). First, the effect of the substituents on the alkynyl moiety was studied. *N*-(4-Methoxyphenyl)-propiolamides bearing the electron-donating groups such as 3,5-dimethyl (**1b**), OMe (**1c**), and Me (**1d**) led to spiro[4,5]-trienones **3b** to **3d** in 83–88% yields. The reactions of propiolamides containing electron-deactivating groups, such as

Scheme 1. Scope of *N*-Arylpropiolamides



4-chloro (**1e**) and 4-fluoro (**1f**), as well as electron-withdrawing groups, such as 4-cyano (**1g**) and 4-COMe (**1h**), on the alkynyl moiety were well tolerated under the typical reaction conditions to give **3e** to **3h** in very good yields. In addition, the reaction of (3-(trifluoromethyl)phenyl)-propiolamide **1i** with **2a** furnished **3i** in 91% yield. Interestingly, naphthyl substituted propiolamide **1j** and thiophenyl propiolamide **1k** were also found to be suitable to provide the 3-acylspiro[4,5]trienones **3j** and **3k** in 85% and 78% yield, respectively. However, the reactions of propiolamides having a terminal alkyne (**1l**) and methyl substituted alkyne (**1m**) were futile under the optimal conditions. Notably, the substrates having substitutions on the *N*-(4-methoxyphenyl) ring, such as 3-Me (**1n**), 3-F (**1o**), and 2-Me (**1p**) groups, were found to be reliable in reaction with **2a** to afford the products **3l** (83%), **3m** (86%), and **3n** (78%), respectively. The *ipso*-cyclization of *N*-arylpropiolamides having various *N*-protecting groups such as benzyl (**1q**), tosyl (**1r**), and 2-iodobenzyl (**1t**) were productive in giving the corresponding spirocycles **3o** (85%), **3p** (72%), and **3q** (80%), respectively. But, *N*-acetylpropiolamide **1s** failed to give the desired product. A gram-scale reaction of **1b** (3.4 mmol) with **2a** proceeded effectively to deliver the corresponding spiro[4,5]-trienone **3b** in 81% yield, which showcases the scalability of the method.

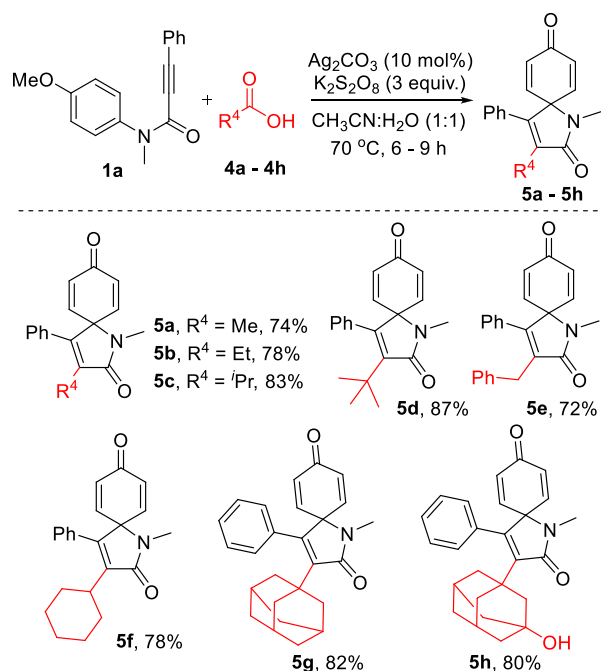
Motivated by the above results, we next set out to explore the scope of various α -keto acids in the current decarboxylative *ipso*-carbocyclization strategy to install diverse carbonyl groups onto the azaspiro[4.5]trienone framework (Scheme 2). To our delight, different aliphatic carbonyls could be successfully introduced by employing the reaction of **1a** with aliphatic α -

Scheme 2. Scope of α -Keto Acids

keto acids **2b–2d** to obtain the spirocycles **3r** to **3t** in 85–90% yield. Gratifyingly, the reaction has expanded to phenylketo acid **2e** to attain the 3-benzoylspiro[4,5]trienone **3u** in 76% yield.

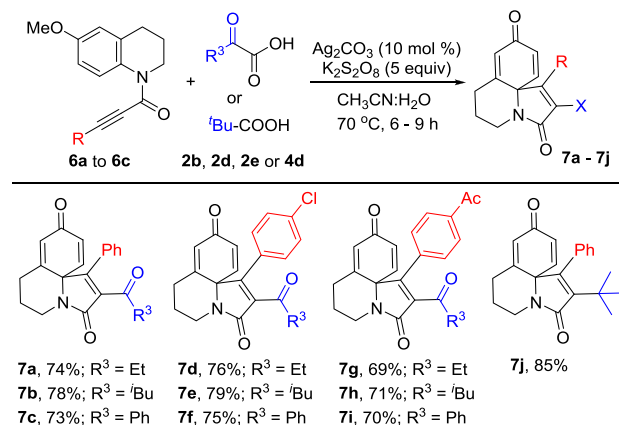
To expand the scope of this domino approach, we have verified the possibility of using carboxylic acids as an alkyl radical source with respect to the formation of a novel class of spiro[4,5]trienones (Scheme 3). Delightfully, the reaction of

Scheme 3. Synthesis of 3-Alkylspiro[4,5]trienones



1a with acetic acid (**4a**) under the optimized conditions (see Supporting Information) led to 3-methylspiro[4,5]trienone **5a** via domino decarboxylative alkylation/*ipso*-cyclization. Likewise, other carboxylic acids such as propionic acid (**4b**), isobutyric acid (**4c**), pivalic acid (**4d**), and 2-phenylacetic acid (**4e**) are well suited to provide **5b** (78%), **5c** (83%), **5d** (87%), and **5e** (72%), respectively. The cyclic secondary alkylation was also realized using cyclohexyl (**4f**) carboxylic acids, affording the product **5f** in 78% yield. Sterically hindered 1-adamantane-carboxylic acid (**4g**) and 3-hydroxyadamantane-1-carboxylic acid (**4h**) were also effective in reacting with **1a** to produce tertiary alkylated products **5g** and **5h** in 82% and 80% yields, respectively.

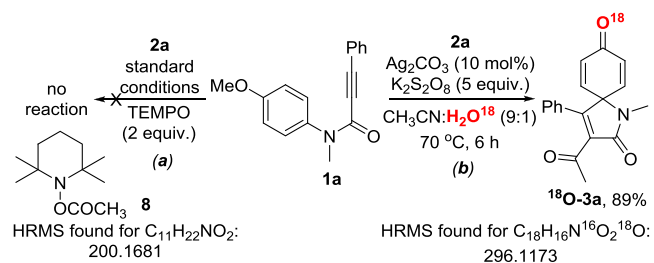
To further accentuate the compatibility of this approach, we examined the tolerance of *N*-(alkynoyl)-6-methoxytetrahydroquinolines to obtain pyrrolo-[2,1-*j*]-quinolone, a challenging framework found in tricyclic marine alkaloids^{3c} (Scheme 4). The domino acylative *ipso*-cyclization of quinoline-based

Scheme 4. Synthesis of Pyrrolo-[2,1-*j*]-quinolones

propiolamide **6a** with **2b** ensued efficiently, giving the acylated pyrrolo-[2,1-*j*]-quinolone **7a** in 74% yield. Other α -keto acids **2d** and **2e** smoothly underwent reaction with **6a**, furnishing tricyclic products **7b** (78%) and **7c** (73%), respectively. Replacing the phenyl group on the alkyne with 4-Cl-Ph (**6b**) and 4-Ac-Ph (**6c**) had no deleterious electronic effect on the reaction, providing the acyl-trienones **7d** to **7i**. The alkylative *ipso*-cyclization of **6a** with **4d** was also equally progressed to afford the *tert*-butyl-pyrrolo-[2,1-*j*]-quinolone **7j** in 85% yield.

To shed light on the possible reaction mechanism, a couple of control experiments were performed. The addition of a radical scavenger (TEMPO) into the reaction suppressed the *ipso*-annulation (by forming TEMPO adduct **8**),¹⁴ indicating that the reaction seemingly proceeds via a radical pathway (Scheme 5a). Besides, to know the source of oxygen, **1a** was

Scheme 5. Control Experiments

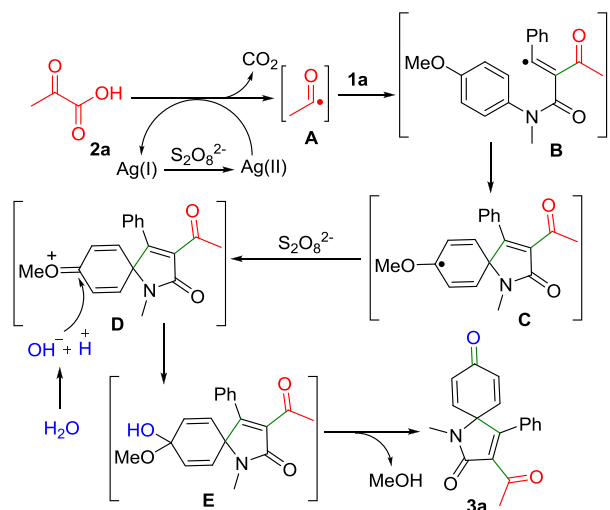


treated in CH₃CN/H₂O¹⁸, which provided the ¹⁸O-integrated spiro[4,5]trienone **18O-3a** (Scheme 5b). This supports the source of oxygen is H₂O during the oxidative dearomatization.

Based on the aforesaid outcomes and relevant literature precedents,^{6b,15} a possible reaction pathway is proposed (Scheme 6). Initially, Ag(I) is oxidized with stoichiometric K₂S₂O₈ to give Ag(II), which engages the α -keto acid **2a** to generate the acyl radical **A** involving decarboxylation. Next, addition of the acyl radical **A** onto alkyne **1a** produces the vinyl radical **B**, which undergoes intramolecular *ipso*-cyclization and dearomatization to give the intermediate **C**. Afterward, oxidation of **C** to oxocarbenium **D** followed by nucleophilic attack of hydroxide (generated from H₂O) provides the intermediate **E**. Finally, hydrolysis of hemiacetal **E** delivers the product **3a**.

In summary, an unprecedented approach, for the synthesis of 3-acyl/alkyl-spiro[4.5]trienones from *N*-arylpropiolamides, has been developed involving silver-catalyzed decarboxylative

Scheme 6. Plausible Mechanism



acylation/alkylation followed by oxidative *ipso*-cyclization. Readily available α -keto acids and alkyl carboxylic acids were used as acyl and alkyl agents, respectively. Additionally, the reaction has been extended for the first time to access unique acyl/alkyl tricyclic pyrrolo-[2,1-*j*]-quinolones. The ease of operation, wide substrate scope, and readily accessible starting materials are the salient features to make the current method valuable.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acs.orglett.0c01588>.

Experimental procedures, characterization details, and ^1H and ^{13}C NMR spectra of new compounds (PDF)

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Notes

The authors declare no competing financial interest.

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