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Ag(I)/persulfate-catalyzed decarboxylative coupling of α -oxocarboxylates with organotrifluoroborates in water under room temperature†

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The decarboxylative coupling reaction of α -oxocarboxylates and organotrifluoroborates was carried out smoothly in the presence of catalytic AgNO₃ using K₂S₂O₈ as oxidant to generate diarylketone products in high yields. The method is novel, simple, safe and efficient. Both aryl substituted potassium α -oxocarboxylates and organotrifluoroborates proceeded smoothly in water under room temperature. The utilization of α -oxocarboxylates as acylating agent presents some elements of interest.

Introduction

Arylketone is a common functionality in many types of organic compounds, for example, pharmaceuticals and natural products.¹ For organic chemists, it is also an extremely useful functional group for further manipulations. Consequently, it continues to inspire the development of methods for the construction of this structural element. Classical routes to synthesize aryl ketones mainly rely on the Friedel–Crafts acylation of aromatic compounds² and the oxidation of the corresponding secondary alcohols.³ Transition-metal-catalyzed reactions provide many opportunities for the synthesis of aryl ketones. The coupling reaction of acylating agents with aryl halides (or aryl metallic compounds and their equivalents) has been developed under the catalysis of transition metals.⁴

Transition metal catalyzed decarboxylative coupling reactions have received much attention for their applications in the construction of C–C and C–hetero bonds in recent years.⁵ Among various types of carboxylic acids (carboxylate), α -oxocarboxylates have emerged as a novel class of acyl surrogates after releasing molecular CO₂.⁶ Theoretically, there are three types of acyl reagents from the decarboxylation of α -oxocarboxylates: acyl anions, acyl cations and acyl radicals.⁷ The direct decarboxylative acylation of potassium α -oxocarboxylates with aryl bromides/chlorides have been demonstrated by

Gooßen's group, with the Pd(II)/Cu(I) catalytic system being particularly efficient with the assistance of P-based and N-based ligands.^{8a–8c} Then they demonstrated a one-pot three-component decarboxylative coupling with α -oxocarboxylates, amines and aryl bromides for the synthesis of azomethines.^{8d} In 2014, Ji *et al.* reported a palladium/copper-catalyzed decarboxylative coupling of aryl iodides with α -oxocarboxylates without the need of ligands.^{8e} α -Oxocarboxylates providing acyl anions with Pd/Cu catalysis by this decarboxylation type, to afford diaryl ketones under harsh conditions (or derivatives) (Scheme 1, I). Recently, You and co-workers reported a transition-metal-free decarboxylation of α -oxocarboxylates with α -bromo-acetophenone.^{9a} In 2017, the room-temperature coupling/decarboxylation reaction between *in situ* generated α -oxocarboxylates with α -bromoketones was described by their group. The transformation shows excellent regioselectivity for 1,2- and 1,3-diketones by solvent-controlled.^{9b} Acyl cations were generated in this decarboxylation manner, with the coupling of α -oxocarboxylates with sp³ carbon center (Scheme 1, II). Acyl radicals were formed *via* Ag(I)/persulfate-catalysis of potassium α -oxocarboxylates by the third decarboxylation type. Lei's group described the first example for realizing isocyanide insertion by using an acyl radical *via* the oxidative radical decarboxylation, to construct 6-acyl phenanthridine structure (Scheme 1, III).^{10a} Synthesis of oxazoles by Ag(I)/persulfate-catalyzed oxidative decarboxylation–cyclization of α -oxocarboxylates and isocyanides was reported by them for the next year.^{10b} Therefore, it is still necessary to develop more Ag(I)/persulfate-catalyzed oxidative decarboxylation of potassium α -oxocarboxylates.

Interestingly, there is a novel recognition that α -oxocarboxylic acids can serve as aryl radical precursors *via* oxidative silver-promoted carbon–carbon bond cleavage in recent years.¹¹ Ag(I)/persulfate catalytic systems have been shown to be effective for

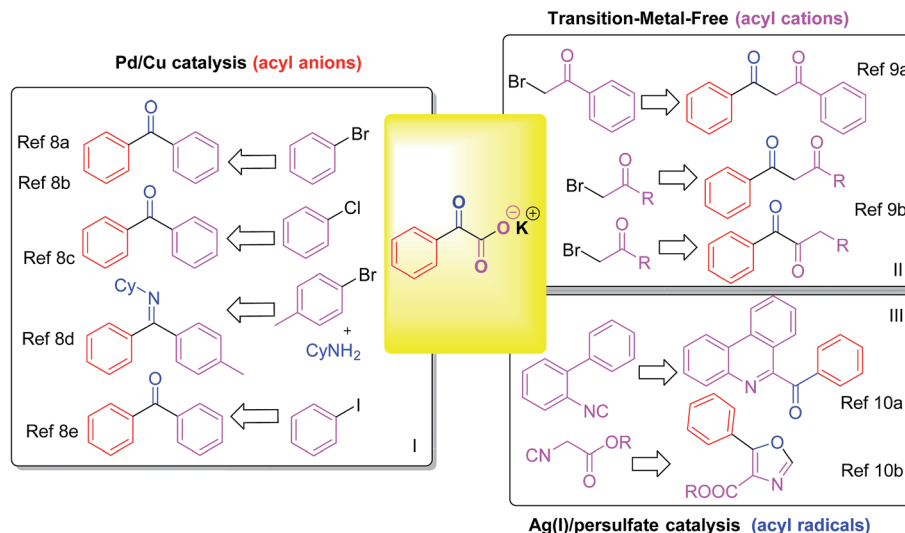
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Scheme 1 Decarboxylation coupling of α -oxocarboxylates.

this transformation, including direct C–H acylation of arenes¹² and oxidative radical decarboxylation–cyclization.¹³ In the presence of silver(I) salts, a persulfate anion disproportionates into a sulfate dianion and a sulfate radical anion, which could react with α -oxocarboxylates through a radical process, providing an acyl radical.

As continued effort on coupling of organoboronic reagents and carbonylative coupling,¹⁴ we report herein a novel method for the silver-catalyzed, decarboxylative acylation of aryltrifluoroborates to form the corresponding diarylketones. It has been successfully developed using α -oxocarboxylates as acylating reagents, as well as a catalytic amount of a silver salt and a cheap inorganic oxidant. The reaction demonstrates a broad substrate scope and excellent functional-group tolerance. This catalytic method should be valuable in the synthesis of aryl-carbonyl motifs.

Results and discussion

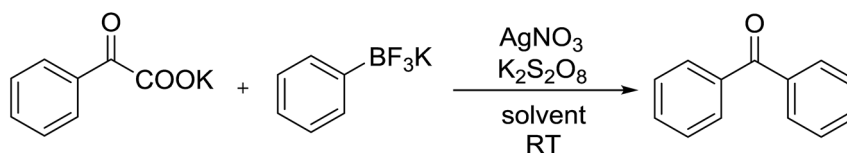
Initially, we optimized the reaction conditions using potassium oxophenylacetate and phenyltrifluoroborate as model substrate in acetone with 5 mol% AgNO_3 and 1.5 equiv. $\text{K}_2\text{S}_2\text{O}_8$ at room temperature for 1 h under air. Only trace amount of cross-coupling biphenyl ketone products was observed (Table 1, entry 1). Then we explored the silver-catalyzed reactions with a variety of reaction medium (Table 1), and 22% of yield were obtained when CH_3CN was used as a solvent (Table 1, entry 2). No better results were obtained with polar aprotic solvents such as DMF, DMSO, or NMP (*N*-methyl-2-pyrrolidone) (Table 1, entries 3–5). The solvents (DCE, DCM, CHCl_3 and CCl_4) were tested, and DCE gave the best result (Table 1, entries 6–9). Three additional ethereal solvents [1,4-dioxane, DME and THF] were surveyed without better results (Table 1, entries 10–12). However, the reaction did not proceed in toluene, EA or *t*-BuOH (Table 1, entries 13–15). Considered that the infusibility of both potassium α -oxocarboxylates and aryltrifluoroborate in organic

solvent, water could be much better medium candidate for this transformation. Water is an easily available, nontoxic, nonflammable, and renewable solvent. The unusual properties of water can lead to unusual reactivity that is not seen in traditional organic solvents. Consequently, the use of water as a solvent in synthetic organic chemistry and materials science has spread throughout the chemical community. The combination of water and metal catalysis has led in recent years to the development of a huge number of new and greener synthetic methodologies. It also provides opportunities for alternative reactivity and simplified product isolation compared to organic solvents. To our delight, sharply increase was observed with the application of water (Table 1, entry 16).

We began to screen many oxidants for the assistance of the regeneration of Ag(I) . Many oxidants have been tested and showed different influence on the reaction (Table 2, entries 1–10). The cross-coupling could not proceed with Cu(OAc)_2 , oxygen and DDQ (Table 2, entries 2–4). PhI(OAc)_2 and BPO (dibenzoyl peroxide) can enhance the efficiency to about 26–34% (Table 2, entries 5–6). In addition, inorganic peroxide are efficient to the cross-coupling. Oxone and $(\text{NH}_4)_2\text{S}_2\text{O}_8$ can afford diphenylketone with the yield of 70% and 74% (Table 2, entries 7–8), then $\text{Na}_2\text{S}_2\text{O}_8$ performed better and $\text{K}_2\text{S}_2\text{O}_8$ performed best (86%) (Table 2, entries 9–10). Then the effect of silver salts was investigated, and many silver(I) compounds such as AgOAc , Ag_2CO_3 , and AgOTf were also applicable for this reaction, albeit in the slightly decreased yield (Table 2, entries 11–13). AgBF_4 , AgOTf and Ag_2O were inferior to AgOTf (Table 2, entries 14–16). Meanwhile, the use of Ag_2SO_4 , AgSbF_6 and AgF as catalysts gave no desired products (Table 2, entries 17–19). And 86% of yield were obtained when AgNO_3 was used as a catalyst (Table 2, entry 20). Therefore, $\text{AgNO}_3/\text{K}_2\text{S}_2\text{O}_8$ in water at room temperature under air for 1 hour was chosen as the optimal conditions for the synthesis of diarylketone from α -oxocarboxylates and aryltrifluoroborate.

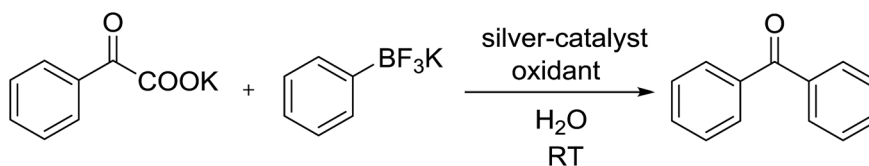
With optimal conditions in hand, the scope of this decarboxylative coupling reaction method was evaluated next (Tables



Table 1 Solvent effect of cross-coupling reaction^a

Entry	Solvent	Yield ^b (%)	Entry	Solvent	Yield ^b (%)
1	Acetone	Trace	9	CCl ₄	31
2	CH ₃ CN	22	10	1,4-Dioxane	26
3	DMF	15	11	DME	38
4	DMSO	20	12	THF	41
5	NMP	12	13	Toluene	Trace
6	DCE	58	14	EA	Trace
7	DCM	46	15	<i>t</i> -BuOH	Trace
8	CHCl ₃	53	16	H ₂ O	86

^a Reaction conditions: potassium oxophenylacetate (1.0 mmol), phenyltrifluoroborate (1.05 mmol), AgNO₃ (0.05 mmol), K₂S₂O₈ (1.5 mmol), solvent (2 mL), 25 °C, 1 h. ^b Isolated yields.

Table 2 The screening of various oxidants and silver salts^a

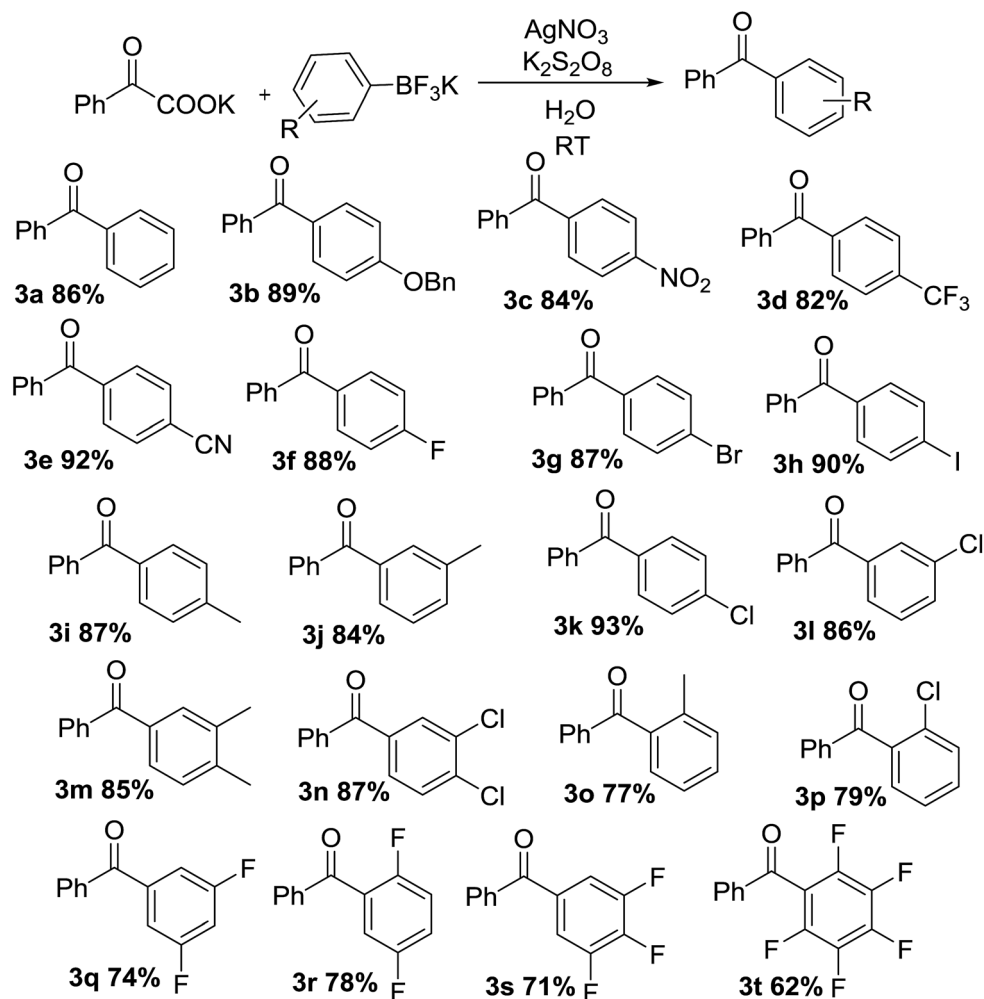
Entry	Oxidant	Yield ^b (%)	Entry	Silver salts	Yield ^c (%)
1	—	—	11	AgOAc	84
2	Cu(OAc) ₂	Trace	12	Ag ₂ CO ₃	81
3	O ₂ (1 atm)	Trace	13	AgOTf	79
4	DDQ	Trace	14	AgBF ₄	43
5	PhI(OAc) ₂	26	15	AgOTf	57
6	BPO	34	16	Ag ₂ O	59
7	Oxone	77	17	Ag ₂ SO ₄	—
8	(NH ₄) ₂ S ₂ O ₈	74	18	AgSbF ₆	—
9	Na ₂ S ₂ O ₈	82	19	AgF	—
10	K ₂ S ₂ O ₈	86	20	AgNO ₃	86

^a Reaction conditions: potassium oxophenylacetate (1.0 mmol), phenyltrifluoroborate (1.05 mmol), silver salts (0.05 mmol), oxidant (1.5 mmol), H₂O (2 mL), 25 °C, 1 h. ^b Isolated yields using AgNO₃ as the silver salt. ^c Isolated yields using K₂S₂O₈ as the oxidant.

3 and 4). As shown, the reaction exhibits a broad substrate scope, with respect to both of the α -oxocarboxylates and the organotrifluoroborates coupling partners. Importantly, the reaction is compatible with electron-neutral (3a), electron-rich (3b), and electron-deficient organotrifluoroborate precursors (3c–f), furnishing the products in good yields. The mild reaction conditions are compatible with a range of sensitive functional groups, such as nitro-(3c), trifluoromethyl-(3d), cyan-(3e), and even halides (fluoro-, bromo-, iodo-) (3f–h), affording diaryl ketone products with a bouquet of functional handles poised for further functionalization. Although, the organotrifluoroborate component bearing functional groups at

different positions, such as methyl and chloro at 3- or 4-position of the phenyl ring were obtained in good yields (3i–l). Moreover, *meta*, *para*-di-methyl phenyltrifluoroborate and *meta*, *para*-di-chloro phenyltrifluoroborate (3m and 3n) underwent the desired cross-coupling in high yields. Substrates having *ortho* methyl (3o) or *ortho*-chloro (3p) substituents at phenyl moiety gave slightly lower reactivity, presumably due to steric reason. Finally, we were pleased to find that the reaction conditions could be readily extended to the arylation of a range of poly-fluorinated substrates (3q–t). Importantly, polyfluorinated biaryl building blocks are of great interest in materials chemistry, because of favorable physicochemical properties.



Table 3 The scope of various organotrifluoroborate^{a,b}

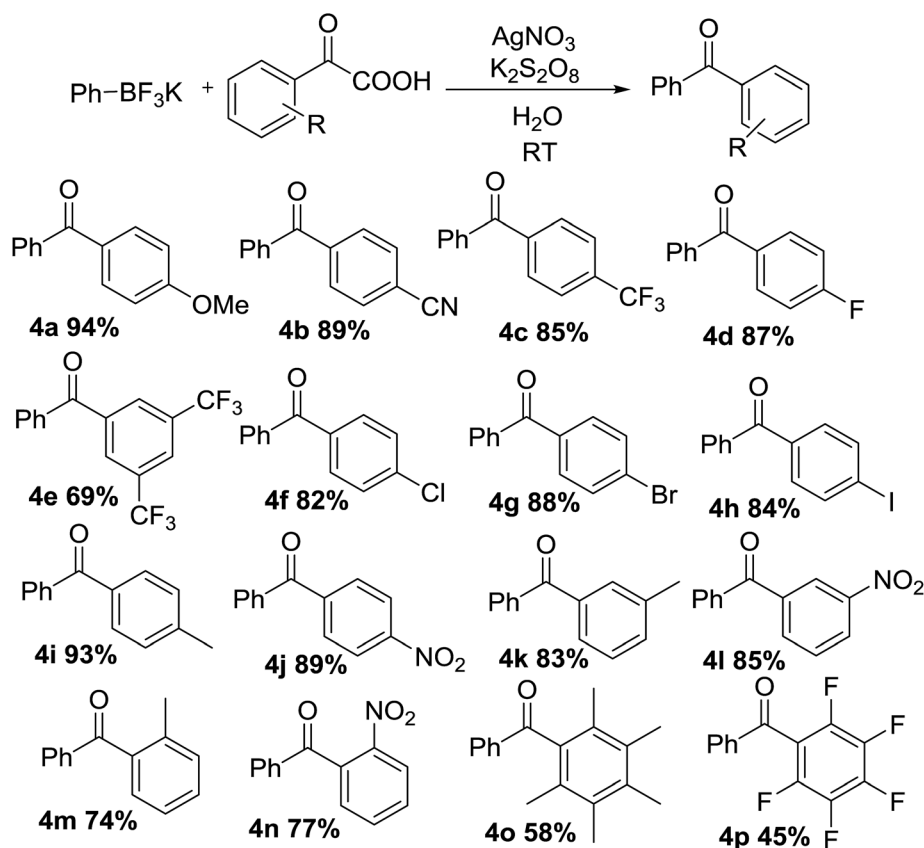
^a Reaction conditions: potassium oxophenylacetate (1.0 mmol), aryltrifluoroborate (1.05 mmol), AgNO_3 (0.05 mmol), $\text{K}_2\text{S}_2\text{O}_8$ (1.5 mmol), H_2O (2 mL), 25 °C, 1 h. ^b Isolated yields.

The scope, with respect to the α -oxocarboxylates component, is also very broad (Table 4), including electron-rich (4a), and electron-deficient (4b–d). The reaction was shown to tolerate a wide variety of functionality on the aryl ring including methoxy-, cyan-, fluoro- and trifluoromethyl- (4a–d). Yields were reduced when using highly electron deficient arenes with substituents such as two CF_3 (4e). The reaction could accommodate substrates containing a chloro, bromo or iodo substituent, which can later be applied to various cross-coupling reactions with metal catalysts (4f–h). Good yield was achieved using *para*-methyl and *para*-nitro substituents, to afford corresponding diaryl methane (4i–j). *Meta*-substituted examples such as 3-methyl and 3-nitro (4k–l) also gave desired products in excellent yield. The *ortho*-substituted example (4m–n) were also tolerated however in reduced yield for sterically hindered site-selectivity. Furthermore, the formation of sterically demanding per-methylated and per-fluorinated product was smoothly in this process (4o–4p). The high selectivity of Ag(I) /persulfate-catalyzed system across a range of sterically and

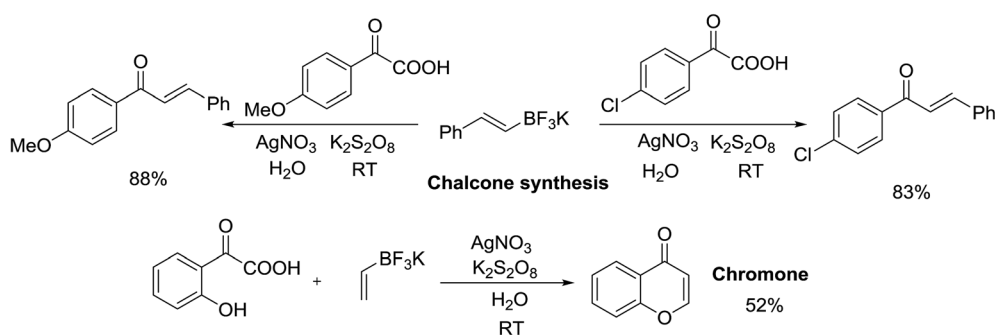
electronically diverse α -oxocarboxylates and organotrifluoroborate precursors is a particularly noteworthy feature of this reaction manifold.

To further explore the potential of this decarboxylative coupling method, several (*E*)-styryl potassium trifluoroborate derivatives were examined (Scheme 2). (*E*)-styryl potassium trifluoroborate underwent decarboxylative coupling with potassium 4-methoxy-oxophenylacetate in moderate yield. Decarboxylative coupling of (*E*)-styryl potassium trifluoroborate with potassium 4-chloro-oxophenylacetate also provided chalcone in good to excellent yields. It is worth noting that when the hydroxyl group was introduced to *ortho*-position of potassium oxophenylacetate, the decarboxylative coupling with vinyl potassium trifluoroborate gave acceptable conversion to afford chromone, showing the robustness of our protocol and demonstrating potential for practical applications. This synthetic method could further be extended to a large-scale decarboxylative acylation. The desired products (3a, 3i, 3k, 4f, 4i) were obtained with slightly decreased yields on 10 mmol



Table 4 The scope of various α -oxocarboxylates^{a,b}

^a Reaction conditions: α -oxocarboxylates (1.0 mmol), phenyltrifluoroborate (1.05 mmol), AgNO_3 (0.1 mmol), $\text{K}_2\text{S}_2\text{O}_8$ (1.0 mmol), H_2O (2 mL), 25 °C, 1 h. ^b Isolated yields.

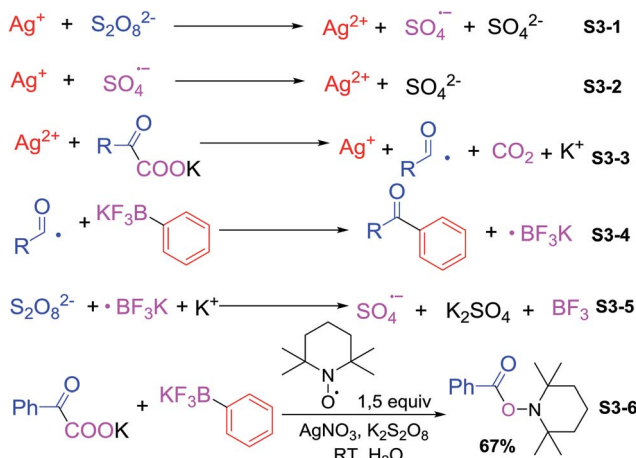
Scheme 2 Applications of Ag(I) /persulfate-catalyzed decarboxylative coupling.

scale (78% for 3a, 72% for 3i, 86% for 3k, 77% for 4f, and 81% for 4i).

With regard to our results and literature reports,¹⁰ we propose the following possible reaction mechanism (Scheme 3). Monovalent silver catalyst was oxidized by $\text{K}_2\text{S}_2\text{O}_8$ to persulfate anion radical and divalent silver ion (S3-1^\dagger) (the persulfate anion radical may also oxidize Ag(I) to Ag(II) (S3-2^\dagger)). Then the Ag(II) generated *in situ* reacted with the α -oxocarboxylates formed acyl radical complexes and CO_2 (S3-3^\dagger). Cross-coupling product obtained after the attack of acyl radical to

aryltrifluoroborate (S3-4^\dagger), which could be terminated by addition of radical scavengers such as hydroquinone or 2,6-di-*tert*-butylphenol, suggesting that the cross-coupling reactions proceed *via* a radical mechanism. Potassium trifluoroborate radical was finally quenched by oxidation of persulfate anion (S3-5^\dagger). Finally, 1.5 equiv. of TEMPO (2,2,6,6-tetramethyl-1-piperidinyloxy) was added to the reaction, and the acyl radical was trapped successfully by TEMPO, affording the corresponding adduct in 67% yields (Scheme 3, S3-6^\dagger).





Scheme 3 Possible mechanism.

Conclusions

In conclusion, we have demonstrated a general method for highly selective decarboxylative coupling of α -oxocarboxylates with aryltrifluoroborates enabled by versatile Ag(I)/persulfate-catalyzed system. The development of this coupling further highlights the beneficial use of inexpensive Ag(I) catalysts for decarboxylation, while exploiting $K_2S_2O_8$ promoter. We anticipate that this strategy will find widespread use as a practical alternative to the classic ketone construction in organic synthesis. Most notably, this new protocol features the synthesis of chalcone and chromone derivatives. Further mechanistic studies and extension to a variety of coupling partners are currently underway in our laboratories.

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

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