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Ligand enabled none-oxidative decarbonylation of aliphatic aldehydes

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ABSTRACT

Decarbonylation of aldehydes is a basic organic transformation, which has been developed for more than six-decade. However, as comparing to well-studied aromatic aldehydes, fewer examples for catalytic decarbonylation of aliphatic aldehydes were reported, mainly on simple or special substrates. For α-bulky or highly functionalized ones, stoichiometric Rh(I) were usually required for decent yields. Herein, we present a rare example of Ir(I)-catalyzed direct decarbonylation of α-quaternary aldehydes with broad substrate scope and good functional group compatibility *via* judicious selection of ligand. The α-chirality is memorized in this decarbonylation process. In addition, we report a broad-spectrum decarbonylation of α-secondary and α-tertiary aldehydes containing multifunctional groups with an improved Rh(I)/DPPP recipe. Finally, we realized selective decarbonylation of α-tertiary aldehydes in the presence of α-quaternary one *via* the reactivity differences.

Transition-metal-catalyzed decarbonylation of aldehydes is a basic transformation that direct removes formyl group and releases CO [1–4]. Although it is a functionality degrading transformation, this reaction is a useful strategy for tuning reactivity of molecules since diverse reactivities of aldehyde group, its α-position, or α,β-unsaturated aldehydes *etc*. These reactivities offer opportunities to use aldehyde group as a powerful traceless transient directing group for C–H activation [5–11] and alkene functionalization reactions [12–14], traceless activating group for Diels-Alder reaction [15,16] or asymmetric α- or β-functionalization and so on [17–31]. In addition, the decarbonylative coupling of aldehydes with unsaturated hydrocarbons is a useful tool to use aldehydes as inexpensive coupling partner [32–44].

Since the first discovery of the activity with $Pd(OH)_2/BaSO_4$ by Eschinazi and Pines [45], and later on Wilkinson's catalyst $[Rh(PPh_3)_3Cl]$ [46] by Tsuji and Ohno [47,48], many catalytic systems, such as Ni [49,50], Pd [51–56], Rh [57–59] have been developed for direct none-oxidative decarbonylation of aldehydes, most of which are aromatic and alkenyl aldehydes. For more challenging aliphatic aldehydes, Rh has been proved to be effective catalysts [60–68], together with Pd [69], Ru [70] and Ir (Fig. 1A) [71–73]. However, for the application in complex nature product synthesis, stoichiometric amount of transition metal salt [1-2 equiv. of Rh(I)] were still needed for almost all examples [74–85], except for a few molecules with simple functionalities [86–89]. In addition, there is a significant rate decreasing for the decarbonylation of α-substituted bulky aldehydes [90], and α-quaternary aldehydes was proved to be very challenging even at 180–215 °C [2,16]. Many catalysts that exhibit good reactivity in other aldehydes failed in α-quaternary ones [64,71], except for a few special substrates or with stoichiometric Rh(I) (Figs. 1B–D) [2,48,69,91,92]. This challenging is probably from that bulky aldehyde may hinder the contact with coordinated metal catalysis, and that the migratory extrusion of CO might be impaired by the bulky and electron rich α-quaternary group, which can usually accelerate the migratory insertion of CO [93]. So, it is still desirable to develop highly efficient method for decarbonylation of aliphatic aldehydes, especially for the substrates with complex structures, multiple function groups, or with a sterically bulky α-position. Herein, we established a highly efficient catalytic decarbonylation of α-secondary and α-tertiary aldehydes that applicable to complex molecule transformations based on a known recipe. And we would also report a novel Ir(I) catalyzed decarbonylation of α-quaternary aldehydes with broad substrate scope *via* judicious selection of diphosphine ligand (Fig. 1E).

In light of highly efficient cationic rhodium coordinated with two 1,3-bis(diphenylphosphino)propane (DPPP) in decarbonylation of aliphatic aldehydes [16,60,61,63–65,67], we started our study from optimizing this recipe in order to develop general conditions that could be applicable to complex molecules. After brief survey of solvents, temperature with model substrate **1-5** in the presence of easily available $[Rh(COD)Cl]_2$ and DPPP, the reaction in xylenes

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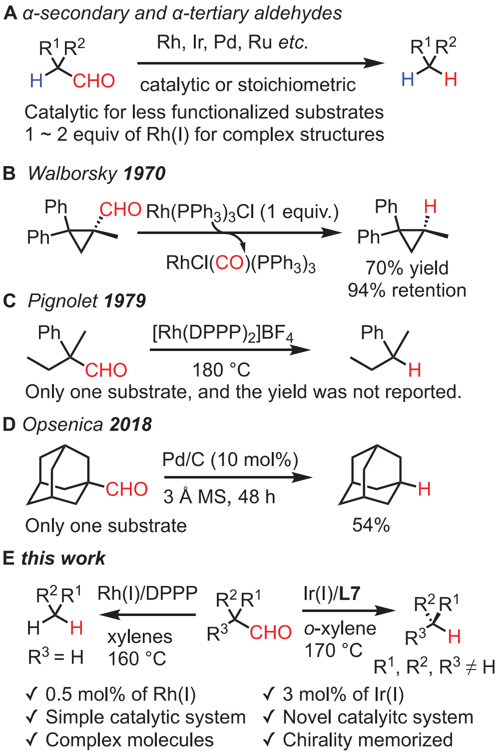
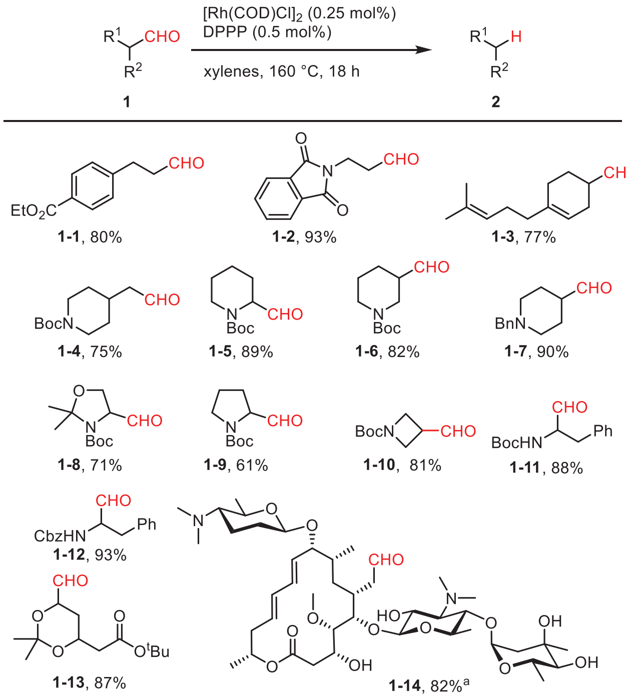


Fig. 1. Decarbonylation of aliphatic aldehydes. (A) Decarbonylation of α-secondary and α-tertiary aldehydes; (B–E) Decarbonylation of α-quaternary aldehydes.

at 160 °C is optimal, and the catalyst loading can be as low as 0.5 mol% of Rh atom. The Rh/DPPP ratio was also evaluated, and both 1:1 or 1:2 producing excellent yields ($>$90% by ^{1}H NMR) of **2-5**, which indicates that neutral rhodium also shows high reactivity in decarbonylation reactions (Page S4 in Supporting information). We then studied the compatibility of this reaction conditions (Scheme 1). Both α-secondary and α-tertiary aldehydes work well and all producing good to excellent yields (**1-1**∼**1-14**). The reaction also exhibits good functional group tolerance, proceeding smoothly with ester (**1-1**, **1-13**), alkene (**1-3**) functionalities. Protecting group such as Bn (**1-7**), Boc (**1-4**∼**1-6**, **1-8**∼**1-11**), Cbz (**1-12**), ketal (**1-13**), and phthaloyl (**1-2**) all survived in this reaction. Various protected heterocycles such as piperidine (**1-4**∼**1-7**), pyrrolidine (**1-9**), oxazolidine (**1-8**) and azetidine (**1-10**) were tested, and all performed well. To test the applicability of this reaction to the complex molecule synthesis, spiramycin (**1-14**) [94] with multiple function group like ester, ether, free amine, hydroxyl, sensitive moieties like conjugated diene and glycosides, proceeded cleanly in this condition and afforded good yield of decarbonylation product. It should be noting that no β-H elimination product was observed in all these examples [51,71,90,94–97]. In addition, this method can be applied to the decarbonylation of aromatic, heteroaromatic or alkenyl aldehydes with various function groups such as hydroxyl, carboxyl, and alkynyl, and producing high yields of decarbonylation products (Page S5 and Table S3 in Supporting information).

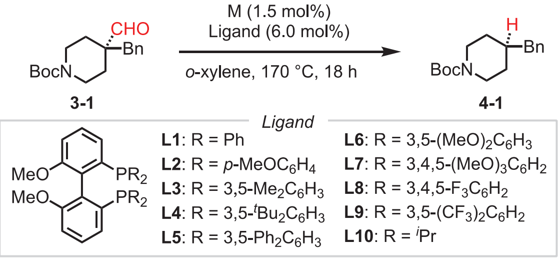
Next, we tested this Rh(I)/DPPP catalyst in the reaction of the α-quaternary aldehydes. Unfortunately, no product was formed except for some decomposition of the aldehyde (Table 1, entry 11). After systematic evaluation of the common used ligands with combination of Ir(I) (Tables S6 and S7, Pages S7 and S8 in Supporting information) [71,72], as well as other catalytic systems (Table 1, entries 12–16), biphenylphosphine (MeO-BIPHEP) **L1** stood out as the most promising ligand skeleton with 64% NMR yield and 97% conversion (Table 1, entry 1). Then, a number of MeO-BIPHEP



Scheme 1. Substrates scope of Rh(I)-DPPP catalyzed decarbonylation of aldehydes. Conditions: **1** (1.0 mmol), $[Rh(COD)Cl]_2$ (0.0025 mmol), DPPP (0.005 mmol), xylenes (1.0 mL), 160 °C, 18 h. [a] 2 mL of xylenes was used, and the reaction time is 10 h.

Table 1

Ligands evaluation in Ir(I) catalyzed decarbonylation of α-quaternary aldehyde **3-1** and some control experiments.[a]



Entry	M	Ligand	Conversion (%)	Yield (%)
1	$[Ir(COD)Cl]_2$	**L1** (*R*)	76	68
2	$[Ir(COD)Cl]_2$	**L2** (*rac*)	70	61
3	$[Ir(COD)Cl]_2$	**L3** (*rac*)	97	85
4	$[Ir(COD)Cl]_2$	**L4** (*S*)	65	60
5	$[Ir(COD)Cl]_2$	**L5** (*rac*)	95	73
6	$[Ir(COD)Cl]_2$	**L6** (*rac*)	97	88
7	$[Ir(COD)Cl]_2$	**L7** (*rac*)	98	93
8	$[Ir(COD)Cl]_2$	**L8** (*rac*)	27	23
9	$[Ir(COD)Cl]_2$	**L9** (*rac*)	48	29
10	$[Ir(COD)Cl]_2$	**L10** (*R*)	49	21
11[b]	$[Rh(COD)Cl]_2$	DPPP	13	–
12[c]	$Rh(PPh_3)_3Cl$	–	33	7
13[c]	$[Rh(DPPP)_2]BF_4$	–	24	–
14[c,d]	Pd/C	–	36	12
15[d,e]	Pd/C	–	66	28
16[d,f]	Pd/C	–	100	31
17	$[Ir(COD)Cl]_2$	–	15	–
18	–	L7 (*rac*)	15	–

[a] Conditions: **3-1** (1.0 mmol), $[Ir(COD)Cl]_2$ (0.015 mmol), ligand (0.06 mmol) and *o*-xylene (1.5 mL) at 170 °C for 18 h. The yield was determined by ^{1}H NMR analysis of the crude reaction mixture with mesitylene as the internal standard.

[b] 1.5 mol% of $[Rh(COD)Cl]_2$, 6.0 mol% of DPPP.

[c] 3 mol% of metal catalyst.

[d] 150 mg 3 Å molecular sieve was added.

[e] 10 mol% of Pd/C.

[f] 50 mol% of Pd/C.

type ligands **L2~L10** [98–100] with fine-tuning steric and electronic properties of substitution groups on phosphine (entries 2–10) were examined, and the results indicate that **L7** is the optimal ligand screened. This reaction is generally benefited from electron-rich substitution group (**L3, L6, L7**), and impaired from electron-withdrawing or too bulky ones (**L8**~**L10**). Control experiments indicated that both [Ir(COD)Cl]$_2$ and **L7** are indispensable in this reaction (entries 17 and 18). Almost no reactivity was observed in classic Rh(I) catalysts and only 32% yield of **4-1** was produced with 50 mol% of Pd/C (entries 14-16).

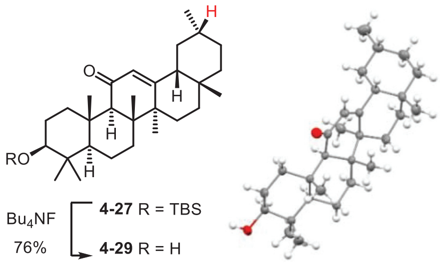
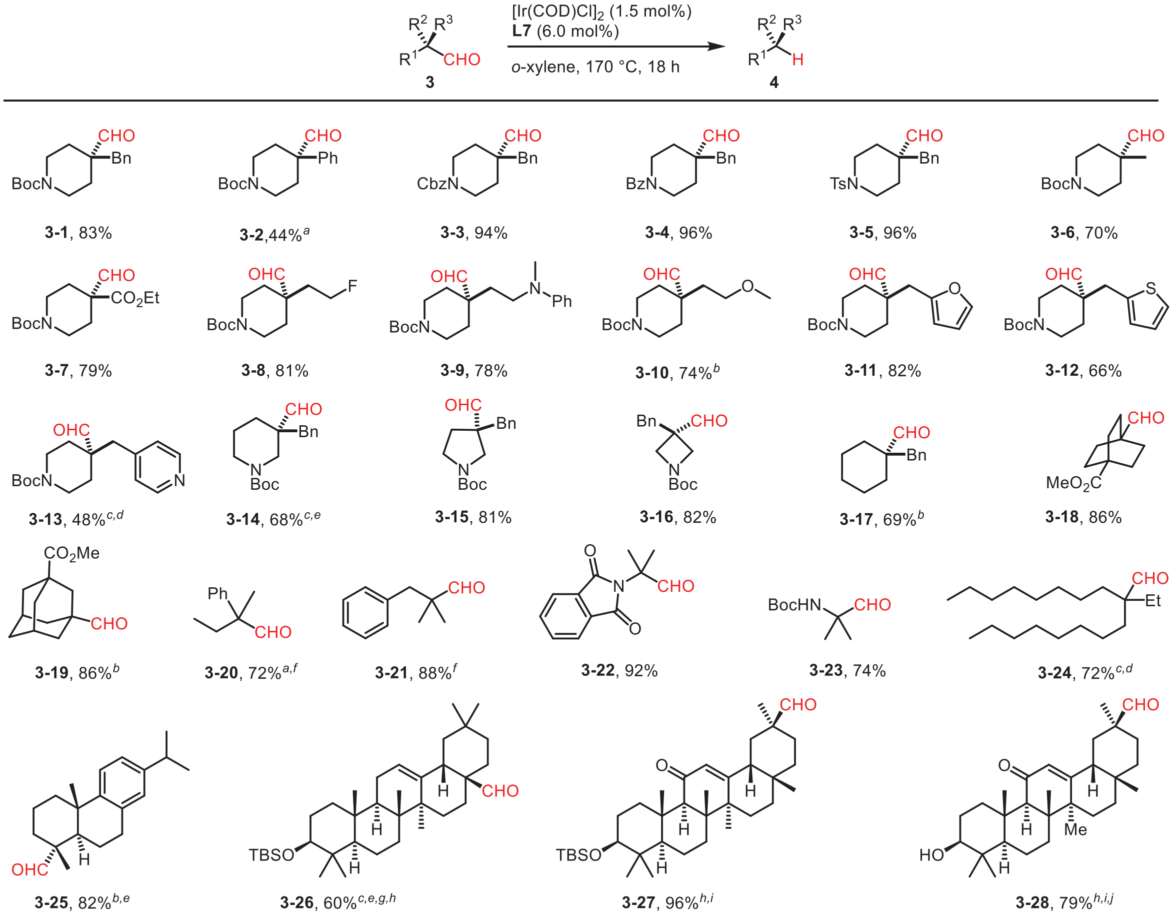
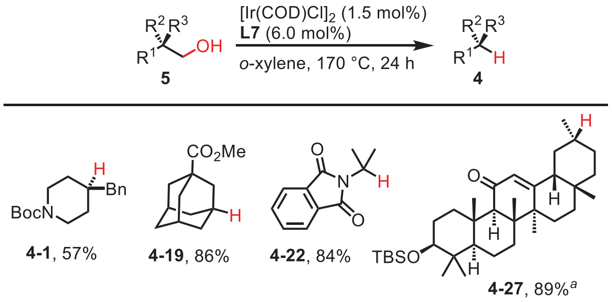


Fig. 2. Deprotection of **4-27** and X-ray crystallographic of **4-29**.

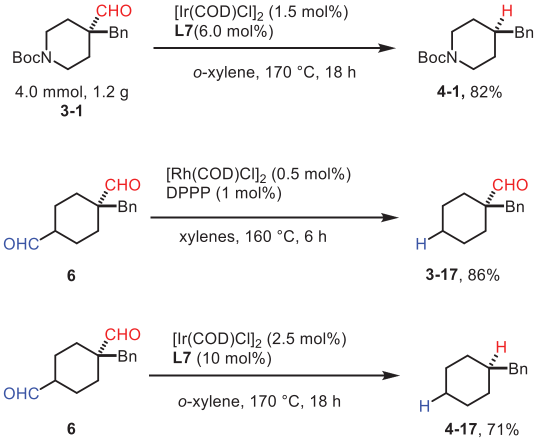
With the optimized reaction conditions developed, we next investigated the scope of α-quaternary aldehydes in the decarbonylation reactions (Scheme 2). All α-quaternary aldehydes (**3-1**~**3-28**) reacted smoothly to give the corresponding products (**4-1**~**4-28**) in moderate to high yields (44%–96%). Protecting groups such as Boc (**3-1**, **3-6**~**3-16**, **3-23**), Cbz (**3-3**), Bz (**3-4**), Ts (**3-5**), phthaloyl (**3-22**) and silyl ether (**3-26**, **3-27**) were survived in this reaction. This reaction is also compatible with many functional groups, like tertiary amine (**3-9**), ether (**3-10**), fluoride (**3-8**), ketone (**3-27**, **3-28**), alkene (**3-26**~**3-28**) and esters (**3-7**, **3-18**, **3-19**). α-Carbon in five-member (**3-15**), four-member (**3-16**) and rigid ring (**3-18, 3-19**) or open-chain (**3-24**) structures were suitable substrates. Heterocycles such as furan (**3-11**), thiophene (**3-12**), and pyridine (**3-13**) were also applicable to the reaction and produced the corresponding products in moderate to good yields. More encouragingly, this recipe can be applied in the modification of complex molecules. The α-quaternary aldehydes derivatives from dehydroabietic acid (**3-25**), oleanolic acid (**3-26**) and 18-β-glycyrrhetic acid (**3-27**, **3-28**) proceeded smoothly, affording the desired products in moderate to excellent yields with memory of chirality at α-position of aldehyde group, without any obvious racemization from NMR spectra [101,102]. If there is a phenyl group in α-position (**3-2**, **3-20**), decarbonylation-dehydrogenation products were also observed probably because of the stabilized benzyl Ir(III) intermediate that might undergo β-hydrogen elimination more easily. This catalytic system is also applicable to the decarbonylation of (hetero)aromatic and less bulky aliphatic substrates (Table S4 and Page S5 in Supporting information). Silyl group in **4-27** could be easily removed, and the absolute configuration of the generated methine was determined by X-ray crystallographic of **4-29** (Fig. 2, CCDC: 2160126). It should be noting that although



Scheme 2. Substrates scope in Ir(I) catalyzed decarbonylation of α-quaternary aldehydes. Conditions: **3** (1.0 mmol), [Ir(COD)Cl]$_2$ (0.015 mmol), **L7** (0.06 mmol), *o*-xylene (1.5 mL), 170 °C, 18 h. [a] Decarbonylation-dehydrogenation products were observed in this reaction, see Supporting information for details. [b] 24 h. [c] 36 h. [d] [Ir(COD)Cl]$_2$ (0.05 mmol), **L7** (0.2 mmol). [e] [Ir(COD)Cl]$_2$ (0.025 mmol), **L7** (0.1 mmol). [f] The yield was determined by ^{1}H NMR analysis of the crude reaction mixture with mesitylene as the internal standard. [g] *o*-Xylene (2.0 mL). [h] The reaction was conducted at 0.5 mmol scale. [i] *o*-Xylene (1.0 mL). [j] Completed dehydrogenation of the hydroxy group was observed in this reaction.



Scheme 3. Methine formation through Ir(I) catalyzed dehydroxymethyl of alcohols. Conditions: **5** (1.0 mmol), $[Ir(COD)Cl]_2$ (0.015 mmol), **L7** (0.06 mmol), *o*-xylene (1.5 mL), 170 °C, 24 h. [a] The reaction was conducted at 0.5 mmol scale in 1 mL of *o*-xylene.



Scheme 4. Gram-scale reaction and selective decarbonyaltion of α-tertiary and quaternary aldehydes.

high temperature (160–170 °C) is used in Rh(I) and Ir(I) system, the function group compatibility is excellent in both recipes.

When a free secondary hydroxyl group was presented in the substrate, ketone was formed as well as the expected decarbonylation product in **4-28**, demonstrating the highly efficient dehydrogenation process coexists in this reaction [90]. Inspired by Madsen's iridium-catalyzed dehydrogenative decarbonylation of primary alcohols to remove hydroxymethyl directly, producing aromatic, primary or secondary C–H bonds [103–105], we tested the similar strategy on several representative substrates with the Ir(I)/**L7** combination to see if more challenging tertiary C–H bond could be formed. To our delight, all these four substrates worked well in this condition, and hydroxymethyl was removed in good to excellent yields. The chirality can also be memorized totally in **4-27** in this reaction as in the direct decarbonylation of aldehyde substrate (Scheme 3).

This reaction can be easily scaled up to 1 g (1.21 g of **3-1**, 4.0 mmol) and similar yield was obtained, demonstrating the practicality of the method (Scheme 4). Selective decarbonylation of α-tertiary aldehyde in the presence of α-quaternary aldehyde is easy with Rh(I)/DPPP recipe. Both aldehydes would be removed under the catalysis of Ir(I)/**L7**, indicating the high efficiency of this catalytic system in decarbonyaltion reaction.

According to Madsen and coworkers's studies [65,104], a possible mechanism is proposed in Fig. 3. The initial decarbonylation of one aldehyde affords a diphosphine coordinated carbonyl metal, which is transformed into a M(III)–acyl complex *via* oxidative addition of the C(O)–H bond. The decarbonylation process is completed by a migratory extrusion of carbon monoxide and reductive elimination and dissociation one CO to regenerate the carbonyl metal.

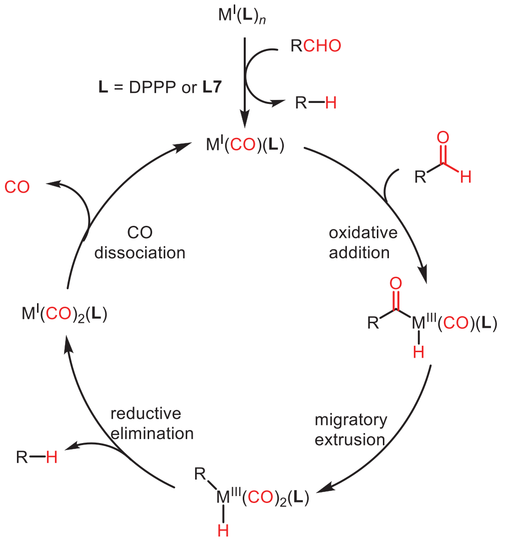


Fig. 3. Proposed mechanism.

In conclusion, we have described a BIPHEP type ligand (**L7**) enabled Ir(I) catalyzed decarbonylation of α-quaternary aldehydes with excellent functional groups compatibility that were rarely reported in previous catalytic systems. This Ir(I)/**L7** combination has been used in the dehydroxymethyl of alcohols to form methine through sequential dehydrogenation and decarbonylation. The α-chirality of substrates is memorized in both decarbonylations processes. We have also improved the known Rh(I)/DPPP recipe by optimizing the solvent and temperature, as well as the Rh(I)-DPPP ratio. This modified recipe could be applied to the efficient decarbonylation of α-secondary and α-tertiary aldehydes containing multifunctional groups including spiramycin. This Rh(I) catalytic system was also applied to the selective decarbonylation of α-tertiary aldehyde in the presence of a α-quaternary one. Both of this Ir(I) and Rh(I) systems could be potentially applied in complex molecular synthesis.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2022.108027.

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