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Synthesis of 1,3-diaryl butanones from acetophenones via a tandem reaction

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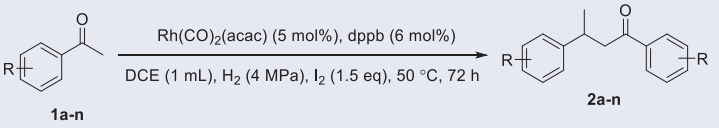
ABSTRACT

A tandem reaction for the simple construction of 1,3-diaryl butanones from acetophenones was developed. Anhydrous HI was generated *in situ* by the promotion of the [Rh]- complex with molecular hydrogen and iodine. The acetophenones undergo aldol reactions by anhydrous HI firstly, and then hydrogenation reactions by the same [Rh]-complex to generate the corresponding 1,3-diaryl butanones in one pot.

GRAPHICAL ABSTRACT



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KEYWORDS
Acetophenones; aldol reaction; anhydrous HI; molecular hydrogen; molecular iodine

Introduction

Arylated ketones are important motifs found in bioactive natural products and are important synthetic intermediates of many industrial products like pharmaceuticals, cosmetics, or agrochemicals.[1–7] In this article, a kind of particular arylated ketones *viz* 1,3-diaryl butanones has been paid attention. These ketones are useful as important precursors for the preparation of NK2 and NK3 receptor antagonists for the treatment of asthma,[8] GABAB receptor agonist (*R*)-baclofen hydrochloride,[9,10] matrix metalloprotease inhibitors for tumor cell invasion,[11] anti-inflammatory agents,[12] inhibitors of stomach acid secretion,[13] and as a solvent for inks and lacquers.[14] Generally, 1,3-diaryl butanones were reported to be prepared by methods like the selective coupling of alcohols using both heterogeneous[15–17] and homogeneous catalyst,[18,19] Friedel-Crafts-type reactions,[20] Wittig olefination,[21] Heck arylation.[22,23] Most of these reaction procedures suffer from one or other limitations like the use of elevated temperature, generation of large amounts of by-products, use of expensive substrates. Thus,

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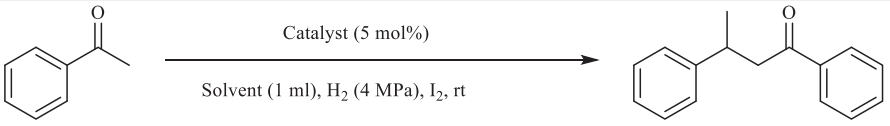
there is still a need for the development of new methodologies for the synthesis of 1,3-diaryl butanones. Among them, the aldol reaction was also attracted as a powerful method for the synthesis of 1,3-diaryl butanones.[24–26] Much effort has been devoted to developing more convenient and efficient strategies for these kinds of reactions. Recently, Akazome's group has reported a novel solvent-free reaction of acetophenone derivatives for the generation of α-alkylated compounds by using anhydrous HI gas. They prepared safe laboratory scale anhydrous HI gas before the reaction.[27] In their methodology, HI acts as both acid and as a reducing agent. Commercially available HI is only 55–57 wt% aqueous solution. So, many researchers have used anhydrous HI in different reactions by preparing with various methods, such as the combination of tetrahydronaphthalene and I_2,[28] RSH and I_2,[29–31] ROH and I_2,[32–34] RCOOH and I_2,[35,36] BI_3:N,N-diethylaniline complex and acetic acid.[37] Although direct use of HI in some reaction has been introduced,[38,39] but still are limited. Very recently, our group developed the rhodium catalyzed direct preparation of anhydrous HI from molecular hydrogen and iodine; and successfully utilized in the formation of iodoalkanes from alkenes, aldehydes, alcohols, and ethers,[40] and alkynes.[41] Inspired with this we describe herein the tandem reaction for the construction of 1,3-diaryl butanones, which is initiated by the *in situ* generation of HI, aldol condensation and then hydrogenation. The anhydrous HI was generated *in situ* by the $Rh(CO)_2(acac)$/dppb promoted reaction of molecular iodine and hydrogen. The *in situ* generated HI resulted in the condensation of acetophenones to generate enone intermediates. The [Rh]-complex then catalyzed hydrogenation of the enones to give the 1,3-diaryl butanones as final product via a tandem reaction.

Result and discussion

As initiation of screening for tandem reaction, acetophenone **1a** was chosen as the model substrate using Rh activated with carbon as a catalyst with molecular hydrogen and iodine (100 mol%) in DCE at room temperature (rt) for 72 h. The reaction afforded the desired product in 22% yield (see the supporting information Table 1). Other carbon activated catalysts of Pd, Ru, and Pt were also studied but provided poor yield or no reaction (see supplementary Table 1). Among the different catalytic systems studied, $Rh(CO)_2(acac)$/dppb was identified as the best catalyst for the tandem reaction of acetophenone in DCE at rt (entry 5, Table 1). Other Rh complexes with dppb were also studied, but the results were not appreciable (entries 1–4, Table 1). The complexes of $Rh(CO)_2(acac)$ with other ligands like PPh_3, dppp, dppm were also studied, but the yield of the reaction could not be improved (see supplementary Table 1). Solvents like DCM, $CHCl_3$, CH_3CN, and toluene were also tested for the reaction to enhance the reactivity and yield, but the results were poor (entries 6–9, Table 1). The temperature was also checked but increasing the temperature lowered the reactivity of the reaction (entry 10, Table 1). Therefore, rt was chosen as optimal temperature for the studies. Notably, increasing catalytic loading of I_2 to 1.5 equiv increased the yield of the reaction giving the desired product in 72% yield (entry 11, Table 1). Further increasing or lowering the loading of I_2 did not improve the reaction (see supplementary Table 1). Therefore, we optimized the I_2 loading at 1.5 equiv.

Table 1. Screening of catalyst and the reaction conditions for the transformation of 1,3-diphenyl butanone from acetophenone.[a]



1a 2a

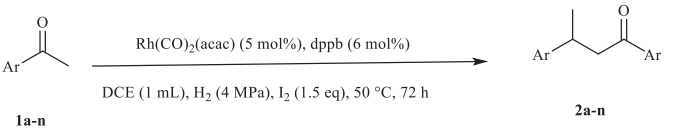
Entry	Catalyst (5 mol%)	Solvent	I_2 loading (equiv)	Yield (%)[b]
1	$[Rh(COD)Cl]_2$/dppb	DCE	1.0	11
2	$[Rh(NBD)Cl]_2$/dppb	DCE	1.0	13
3	$Rh(COD)_2SbF_6$/dppb	DCE	1.0	26
4	$Rh(COD)_2BF_4$/dppb	DCE	1.0	20
5	$Rh(CO)_2(acac)$/dppb	DCE	1.0	58
6	$Rh(CO)_2(acac)$/dppb	DCM	1.0	40
7	$Rh(CO)_2(acac)$/dppb	$CHCl_3$	1.0	18
8	$Rh(CO)_2(acac)$/dppb	MeCN	1.0	N.R.
9	$Rh(CO)_2(acac)$/dppb	Toluene	1.0	52
10[c]	$Rh(CO)_2(acac)$/dppb	DCE	1.0	40/50/24
11	**$Rh(CO)_2(acac)$/dppb**	**DCE**	**1.5**	**72**

[a]Reaction conditions: Metal (0.01 mmol) and ligand (0.012 mol) in solvent (1 mL) were stirred at rt for 30 min under Ar. Acetophenone **1a** (0.4 mmol) and I_2 were added. The reaction mixture was stirred in a hydrogen atmosphere at rt for 72 h). [b]Isolated yield. [c]Reaction at 40, 50 and 60 °C.
Bold indicates the optimal condition of the reaction

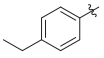
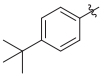
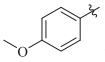
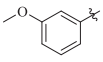
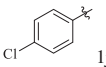
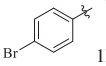
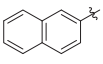
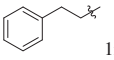
With the optimum conditions in hand, we explored the scope and limitation of the reaction of various acetophenone derivatives using the catalytic system of $Rh(CO)_2(acac)$/dppb with H_2 and I_2 (Table 2). We were delighted that our reaction conditions could provide tandem products across the various acetophenones. However, for the substituted aryl moiety in the ketones, the yields were low at rt. So, the reaction temperature was elevated to 50 °C to get higher yields. The alkyl substituents in the aryl ring of the ketones also gave the desired product in moderate yields. These might be due to increased electron density at the carbonyl carbon which will lower the nucleophilicity. Among the methyl substituted aryl moiety substrates, the *para*-substituted gave the highest yield 69% while the *ortho*-substituted gave only trace, these might be due to the steric-effect of the substituent (entries 2–4, Table 2). The bulkier alkyl substituents like ethyl and *t*-butyl in the *para*-position also gave the desired products in moderate yields (entries 5–6, Table 2). Notably, upon substitution of the stronger electron-donating group such as methoxy either at the *para*- or *meta*- position, no desired products were observed (entries 7 and 8, Table 2). Higher yields were expected from the halogen substituents like fluoro, chloro and bromo in the *para*-position of the phenyl ring due to the increased nucleophilicity at the carbonyl carbon but the yields were only moderates (entries 9–11, Table 2). The relatively low yield of the naphthyl methyl ketone 14% (entry 12, Table 2) might be attributed to the sterically hindered effects from the coordination of the catalyst with the substrate. Alkyl ketone and heterocyclic ketone were also studied but could not get the desired products (entries 13 and 14, Table 2).

We also tried with different acetophenones having different electronic properties like *para*-fluoro acetophenone and *para*-methyl acetophenone in the same reaction to furnish the cross-aldol hydrogenated product. But, mixtures of cross-aldol and

Table 2. Scope for the transformation of 1,3-diaryl butanones from acetophenones.[a]

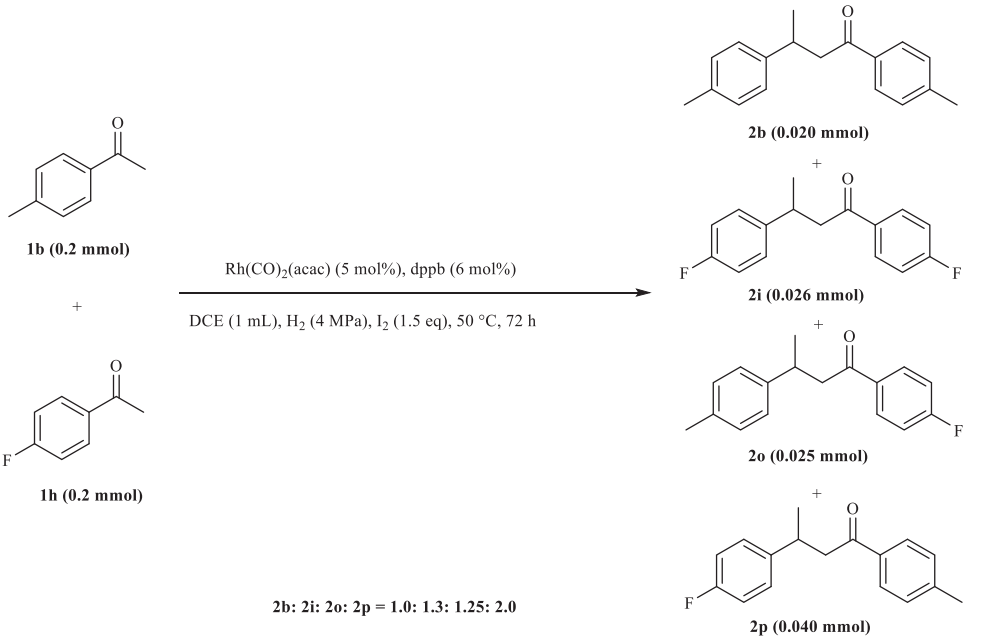


1a-n **2a-n**

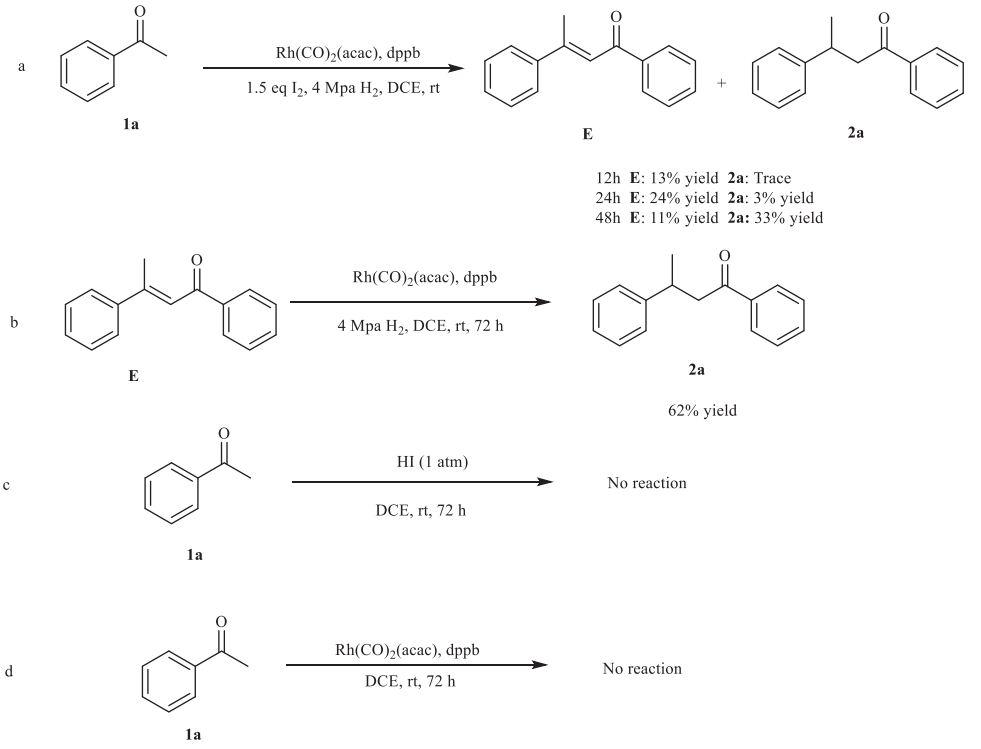
Entry	Ar	Yield (%)[b]
1[c]	 1a	72
2	 1b	69
3	 1c	52
4	 1d	Trace
5	 1e	55
6	 1f	52
7	 1g	NR
8	 lh	NR
9	 1i	44
10	 1j	49
11	 1k	56
12	 1l	14
13	 1m	Complex mixture
14	 1n	Complex mixture

[a]Reaction conditions: $Rh(CO)_2(acac)$ (0.01 mmol) and dppb (0.012 mol) in DCE (1 mL) were stirred at rt for 30 min under Ar. Ketones (**1a–1n**) (0.4 mmol) and iodine (0.3 mmol) were added. The reaction mixt-ure was stirred in a hydrogen atmosphere at 50 °C for 72 h. [b]Isolated yield. [c]Reaction at rt.

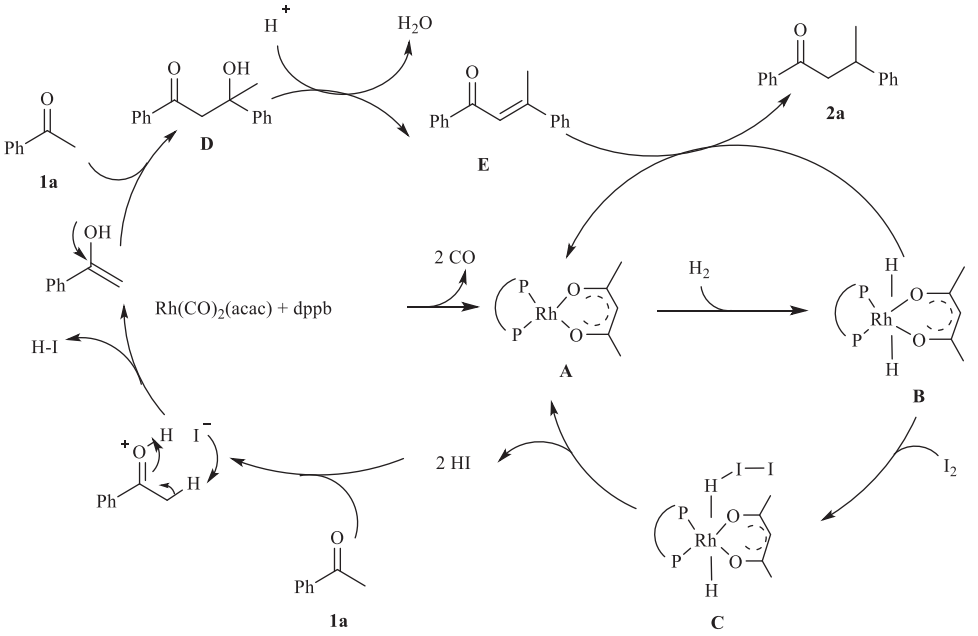
self-condensed aldol products were obtained (Scheme 1). In all of the reactions, unreacted starting materials were the main reason for the moderate or low yields. Only a few impurities were observed and confirmed from GC-MS. Since the products have a chiral carbon, we tried the asymmetric version of the reaction to get the enantiomeric products by using chiral ligands. We tried with (*R*)-DIOP but unfortunately, no enantiomeric product was obtained (see supporting information). Other chiral ligands were also tested and still under investigation.



Scheme 1. Cross-aldol hydrogenation.



Scheme 2. Control experiments.



Scheme 3. Plausible mechanism of the reaction.

To better understand the reaction pathway, sets of control experiments were carried out as shown in Scheme 2. First, the condensation was carried out at three different reaction times under the same optimal conditions using acetophenone **1a** as a model substrate (Scheme 2a). After 12 h it gave the condensation product **E** in 13% yield with a trace amount of hydrogenated product **2a**. And as the reaction continued for 24 h the yield of **E** increased to 24% and that of **2a** to 3%. While continuing the reaction for 48 h the yield of **E** decreased and that of **2a** increased. Thus, confirmed the formation of **E** followed by the hydrogenation to give the desired hydrogenated product **2a**. Next, the reduction of **E** was carried out using the same catalytic conditions without I_2 for 72 h, it gave the hydrogenated product **2a** in 62% yield (Scheme 2b). For the stronger support of the reaction pathway we further carried out the condensation reaction of **1a** in the absence of [Rh]-complex (Scheme 2c). Firstly, the reaction was carried out by passing HI gas (1 atm) released from the reaction of NaI and phosphoric acid to the solution of acetophenone **1a** in DCE. But it cannot bring the condensation of acetophenone to the desired product; it might be due to the low concentration of HI in DCE which cannot promote the condensation of the acetophenone to give the aldol product. Similar result was reported in the supporting information of Brown's work where, they used acetophenone **1a** (1 equiv) and phosphoric acid (2 equiv) and refluxed for 3 days to get 9% yield of enone **E**.[42] This explains enone intermediate **E** cannot be generated in the solution with low concentration of HI. Next, the tandem reaction for preparation of 1,4-butanones was performed using acetophenone **1a** in [Rh]-complex without H_2 and I_2 (Scheme 2d). To our anticipation, no product was formed confirming the initial formation of intermediate **E** by the *in situ* generated HI followed by hydrogenation with [Rh]-complex to give the hydrogenated aldol product.

Based on control reactions a plausible mechanism for the transformation of the 1,3-diaryl butanone (**2a**) was developed as shown in Scheme 3. The coordination of rhodium precursor with dppb generates the complex **A**, which then added H_2 followed by I_2 to give **C** with the generation of HI. This HI generated *in situ* activated the acetophenone to enol and proceeded the aldol reaction by reacting another molecule of acetophenone to give **D** which on dehydration to give α,β-unsaturated ketone **E**. The rhodium complex **B** reduced **E** to furnished the product (**2a**) with the regeneration of rhodium compound **A**. Based on the proposed mechanism, the final product was obtained by the hydrogenation of the aldol product catalyzed by rhodium complex, unlike the earlier reported mechanism.[27]

Conclusion

In summary, we developed a tandem reaction for the preparation of 1,3-diaryl butanones using simple and readily available starting material acetophenones. The methodology also showed the *in situ* generations of anhydrous HI from the $Rh(CO)_2(acac)$/dppb promoted reaction of molecular iodine and hydrogen in the presence of the substrate. The HI thus generated initiated the condensation of acetophenones. Thus, this methodology opens up the applications of this conveniently generated anhydrous HI in other reactions.

Experimental

Materials

The reactions and manipulations were performed under an atmosphere of argon by using standard Schlenk techniques and glovebox (Mikrouna, Supper1220/750). Anhydrous toluene was distilled from sodium benzophenone ketyl prior to use. Anhydrous CH_3CN (acetonitrile), DCE (dichloroethane), DCM (dichloromethane) were distilled from calcium hydride. Acetophenones were purchased from Sigma Aldrich and used without further purification. ^{1}H NMR and 13 C NMR spectra were recorded on Bruker-Avance 400 MHz spectrometer. $CDCl_3$ was used as solvent. Chemical shifts (δ) were reported in ppm with tetramethylsilane as internal standard and J values were given in Hz, abbreviation were used to denoted signals multiplicities (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplate). Melting points were measured on X-4 melting point apparatus and uncorrected. High resolution mass spectra (HRMS) were performed on a VGAutospec-3000 spectrometer. Column chromatography was performed with silica gel using petroleum ether and ethyl acetate as eluents.

General procedure for the synthesis of 2a

$Rh(CO)_2(acac)$ (0.01 mmol), dppb (0.012 mol) and 1 mL of DCE were added to a Schlenk tube under an argon atmosphere in a glovebox. The mixture was stirred at room temperature for 30 min. Then acetophenone **1a** (0.4 mmol) and iodine (0.3 mmol) were added. The reaction mixture was stirred in a hydrogen atmosphere at rt for 72 h. After vacuum evaporation of the solvent, the residue was purified by silica gel

column chromatography to provide the desired product **2a**. The compounds **2b–2n** were also synthesized by using this procedure.

Compound **2a** was obtained as colorless oil, 72% yield; ^{1}H NMR (400 MHz, Chloroform-d) δ 7.93–7.91 (m, 2H), 7.54 (t, J = 7.3 Hz, 1H), 7.44 (t, J = 7.8 Hz, 2H), 7.31–7.25 (m, 4H), 7.21–7.16 (m, 1H), 3.54–3.46 (m, 1H), 3.30 (dd, J = 16.4, 5.7 Hz, 1H), 3.18 (dd, J = 16.5, 8.3 Hz, 1H), 1.34 (d, J = 6.9 Hz, 3H). 13 C NMR (101 MHz, Chloroform-d) δ 199.2, 146.6, 137.2, 133.1, 128.6, 128.6, 128.2, 126.9, 126.4, 47.1, 35.6, 22.0. HRMS(EI$^+$): *m/z* calcd for $C_{16}H_{16}O$ $[M]^+$: 224.1213. Found 224.1210.

Experimental details, ^{1}H, 13 C NMR and HRMS spectra have been provided in supporting information.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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