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One-pot synthesis of 1,5-diketones catalyzed by barium isopropoxide

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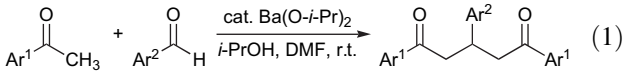
Abstract—A tandem cross-coupling reaction of aryl methyl ketones with aromatic aldehydes has been accomplished employing barium isopropoxide as a catalyst, in which barium enolates are generated and then three consecutive reactions (aldol reaction/β-elimination/conjugate addition) occur; this one-pot procedure is a convenient method to obtain symmetrical 1,5-diketones in good yields. In some cases, addition of *iso*-propanol is effective in improving the chemical yield.

1. Introduction

Michael addition of enolates to α,β-unsaturated carbonyl compounds is a beneficial method to obtain 1,5-dicarbonyl compounds,[1] which are further transformable into useful multifunctional molecules including 2-cyclohexen-1-one derivatives via a Robinson annelation-type process.[2] We have previously reported preliminary results on a barium hydride or barium alkoxide-promoted tandem cross-coupling reaction of ketones with aldehydes giving symmetrical 1,5-diketones in good yields.[3,4] We describe here further studies about the one-pot 1,5-diketone synthesis, which has been achieved by a catalytic amount of barium isopropoxide (Eq. 1).

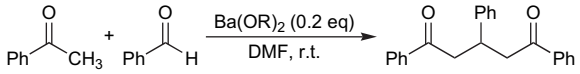


(1)

2. Results and discussion

Table 1 shows the catalytic ability of various barium alkoxides in the reaction of acetophenone with benzaldehyde. Among the barium alkoxides tested, barium isopropoxide has been found to be the most preferable in terms of reactivity and commercial availability (entries 2–4). For example, treatment of benzaldehyde with 2.5 equiv of acetophenone under the influence of 0.1 equiv of $Ba(O\text{-}i\text{-}Pr)_2$ in DMF at room temperature resulted in the formation of the desired 1,5-diketone in 58% yield (entry 4). There seems to be essentially no difference in reactivity between commercially available $Ba(O\text{-}i\text{-}Pr)_2$ and the in situ-prepared one (entries 2–4). Steric bulkiness of the barium alkoxide does not affect the chemical yield of the product and indeed, the highest yield (65%) was obtained when barium 1-adamantoxide was used as a catalyst (entry 7). In contrast, barium

Table 1. Catalytic activity of various barium alkoxides[a]



Entry	$Ba(OR)_2$[b]	Time (h)	Yield[c] (%)
1	$Ba(OMe)_2$	18	54
2	$Ba(O\text{-}i\text{-}Pr)_2$	22	60
3	$Ba(O\text{-}i\text{-}Pr)_2$[d]	0.3	57[e]
4	$Ba(O\text{-}i\text{-}Pr)_2$[f]	8	58[e]
5	$Ba(O\text{-L-menthyl})_2$	22	64
6	$Ba(O\text{-}t\text{-}Bu)_2$	23	52
7	$Ba(O\text{-1-adamantyl})_2$	22	65
8	$Ba(O\text{-2-naphthyl})_2$	22	<1

[a] Unless otherwise specified, the reaction was performed employing acetophenone (2.5 equiv), benzaldehyde (1 equiv), and barium alkoxide (0.2 equiv) in dry DMF at rt.
[b] Prepared from BaH_2 (0.2 equiv) and the corresponding alcohol (0.4 equiv) in dry DMF at rt.
[c] Isolated yield.
[d] Commercially available $Ba(O\text{-}i\text{-}Pr)_2$ of 1 equiv was used.
[e] The starting substrates and an unknown product except the product were obtained.
[f] Commercially available $Ba(O\text{-}i\text{-}Pr)_2$ of 0.1 equiv was used.

Keywords: Aldehydes; Barium isopropoxide; 1,5-Diketones; Enolates; Ketones; Michael addition; α,β-Unsaturated ketone.
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2-naphthoxide did not provide the targeted 1,5-diketone at all (entry 8).

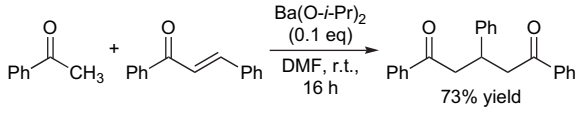
Thus, the barium alkoxide catalyst was fixed for barium isopropoxide and we carried out the tandem cross-coupling reaction between numerous aryl methyl ketones and aromatic aldehydes. The results are summarized in Table 2. Not only simple aromatic aldehydes but also benzaldehyde derivatives, which have a potentially labile functional group such as nitro or fluoro group, also furnished the corresponding 1,5-diketones in moderate yields (entries 6 and 7). Noteworthy is the fact that 2-pyridyl group, by which some chelate effect was anticipated, did not affect the reaction course and catalytic activity (entry 9).

Next, we attempted addition of *i*-PrOH so as to regenerate barium isopropoxide smoothly. As a consequence, increase in the amount of *i*-PrOH improved the chemical yield of the desired product (Table 3) and the best result was attained when 20 equiv of *i*-PrOH was added (entry 4). However, further improvement in the yield was not achieved in the presence of 50 equiv of the alcohol (entry 5).

Under the optimized reaction conditions, we reexamined the coupling reaction using diverse combinations of aryl methyl ketone and aromatic aldehyde. Selected examples are shown in Table 4. As a result, addition of *i*-PrOH was effective for obtaining higher yields in some cases (compare entries 2 and 7 in Table 4 with entries 2 and 7 in Table 2, respectively). However, reactions using other combinations of aryl methyl ketones and aromatic aldehydes were deactivated under the influence of the alcohol (compare entries 3–6 and 10–12 in Table 4 with entries 3–6 and 9–11 in Table 2, respectively).

In the present 1,5-diketone synthesis, aldol reaction of an aryl methyl ketone with an aromatic aldehyde, β-elimination of the resulting aldol adduct leading to an α,β-unsaturated ketone, and Michael addition of a barium enolate of the aryl methyl ketone to the enone are considered to occur sequentially (Fig. 1).[5]

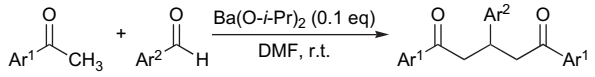
To clarify the reaction pathway, we attempted a catalytic Michael addition of acetophenone to chalcone in the presence of 0.1 equiv of $Ba(O\text{-}i\text{-}Pr)_2$ in DMF at room temperature for 16 h and found that the anticipated 1,5-diketone formed in 73% yield (Eq. 2). This result is a positive proof to support the aforementioned reaction course.



(2)

Furthermore, synthesis of an unsymmetrical 1,5-diketone was also achieved by this method. For example, when an

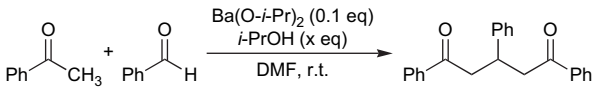
Table 2. Barium isopropoxide-catalyzed tandem coupling reaction of ketones with aldehydes[a]



Entry	Ar^1	Ar^2	Time (h)	Yield[b] (%)
1[c]	Ph	Ph	8	58
2	4-$MeOC_6H_4$	Ph	16	40
3	1-Naphthyl	Ph	16	71
4	Ph	4-$H_3CC_6H_4$	20	72
5	Ph	4-$MeOC_6H_4$	16	57
6	Ph	4-$O_2NC_6H_4$	16	48
7	Ph	4-FC_6H_4	6	56
8	Ph	2-Naphthyl	5	47
9	Ph	2-Pyridyl	5	44
10	Ph	3-Pyridyl	16	74
11	Ph	4-Pyridyl	16	71

[a] Unless otherwise specified, the reaction was performed employing ketone (3 equiv), aldehyde (1 equiv), and barium isopropoxide (0.1 equiv) in dry DMF at rt.
[b] Isolated yield.
[c] Acetophenone of 2.5 equiv was used.

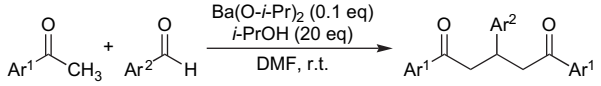
Table 3. Additive effect of *i*-PrOH on the chemical yield of 1,5-diketone[a]



Entry	*x* (equiv)	Yield[b] (%)
1	0	66
2	5	68
3	10	70
4	20	76
5	50	64

[a] The reaction was performed using acetophenone (3 equiv), benzaldehyde (1 equiv), barium isopropoxide (0.1 equiv), and *i*-PrOH (0–50 equiv) in dry DMF at rt for 18–24 h.
[b] Isolated yield.

Table 4. Barium isopropoxide-catalyzed tandem coupling reaction between ketones and aldehydes in the presence of *i*-PrOH[a]



Entry	Ar^1	Ar^2	Time (h)	Yield[b] (%)
1	Ph	Ph	7	76
2	4-$MeOC_6H_4$	Ph	16	51
3	1-Naphthyl	Ph	16	56
4	Ph	4-$H_3CC_6H_4$	20	66
5	Ph	4-$MeOC_6H_4$	24	51
6	Ph	4-$O_2NC_6H_4$	16	38
7	Ph	4-FC_6H_4	6	76
8	Ph	1-Naphthyl	16	86
9	Ph	2-Naphthyl	5	48
10	Ph	2-Pyridyl	16	37
11	Ph	3-Pyridyl	18	47
12	Ph	4-Pyridyl	18	47

[a] The reaction was performed using ketone (3 equiv), aldehyde (1 equiv), barium isopropoxide (0.1 equiv), and *i*-PrOH (20 equiv) in dry DMF at rt for 5–24 h.
[b] Isolated yield.

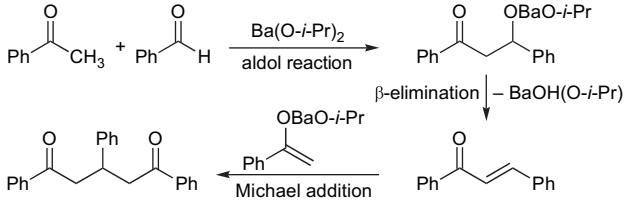
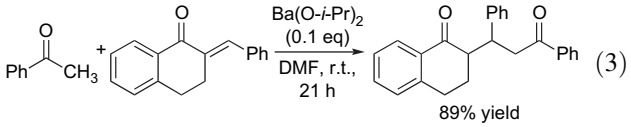


Figure 1. Plausible reaction pathway.

α-tetralone-derived α,β-unsaturated ketone was used as a Michael acceptor, the targeted adduct was obtained in 89% yield (Eq. 3).



(3)

A suggested catalytic cycle of the present tandem coupling reaction between acetophenone and benzaldehyde is indicated in Figure 2. First, $Ba(O\text{-}i\text{-}Pr)_2$ reacts with acetophenone to generate barium enolate **1**. Subsequently, the enolate **1** adds to benzaldehyde to give the barium alkoxide of aldol adduct **2**, which is then allowed to be transformed into α,β-unsaturated ketone **3** by β-elimination of BaOH(O-*i*-Pr) from the alkoxide **2**. Thus generated enone **3** is able to undergo Michael addition by another barium enolate of acetophenone **4** (X=O-*i*-Pr or OH) leading to the barium enolate of Michael adduct **5**. Finally, protonation of **5** by *i*-PrOH or H_2O furnishes the targeted 1,5-diketone **6** and generates BaX_2 (X=O-*i*-Pr or OH). In this step, addition of an excess amount of *i*-PrOH accelerates the protonation. In the second cycle and thereafter, the barium base BaX_2 is recycled and behaves as a catalyst.

3. Conclusion

We have demonstrated a novel tandem cross-coupling reaction between aryl methyl ketones and aromatic aldehydes catalyzed by barium isopropoxide, in which aldol reaction, β-elimination, and Michael addition occur sequentially. The main features of the present method are: (1) the procedure is operationally simple using readily available chemicals and can provide various symmetrical 1,5-diketones in moderate to high yields of up to 86%; (2) employment of *i*-PrOH as an additive is effective in improving the chemical yield in some cases; (3) unsymmetrical 1,5-diketones are also able to be prepared by the present method; (4) barium enolates can be generated directly from the corresponding ketones with the aforementioned barium alkoxide. Studies on related reactions using the barium reagent are now in progress.

4. Experimental

4.1. General methods

Column chromatography was conducted with 63–230 mesh silica gel. Infrared (IR) spectra were recorded on a FTIR spectrometer. ^1H NMR spectra were recorded on a 400 or 500 MHz spectrometer. Chemical shifts of ^1H NMR spectra were reported relative to tetramethylsilane (δ 0) or chloroform (δ 7.26). Splitting patterns are indicated as s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br, broad. ^{13}C NMR spectra were recorded on a 100 or 125 MHz spectrometer. Chemical shifts of ^{13}C NMR spectra were reported relative to $CDCl_3$ (δ 77.0). Mass spectra were recorded on a JEOL JMS-AX500 mass spectrometer using fast atom bombardment (FAB) ionization.

All experiments were carried out in a two-necked flask under an atmosphere of standard grade argon gas (oxygen <10 ppm). (*E*)-2-Benzylidene-3,4-dihydronaphthalen-1(2*H*)-one shown in Eq. 3 was prepared by treatment of 3,4-dihydronaphthalen-1(2*H*)-one with 1.2 equiv of benzaldehyde in the presence of 0.1 equiv of $Ba(O\text{-}i\text{-}Pr)_2$ in dry DMF at room temperature for 12 h. Barium alkoxides (entries 1, 2, and 5–8 in Table 1) were generated in situ from BaH_2 and 2 equiv of the corresponding alcohol in dry DMF at room temperature. Other chemicals were used as purchased.

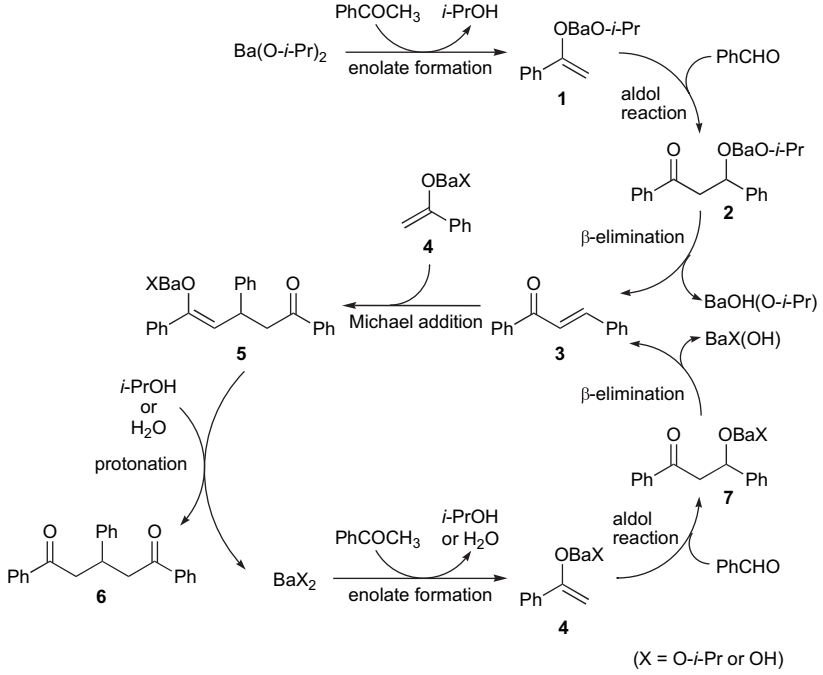


Figure 2. Suggested catalytic mechanism for the tandem coupling reaction catalyzed by $Ba(O\text{-}i\text{-}Pr)_2$.

4.2. Typical experimental procedure for tandem cross-coupling reaction of ketones with aldehydes catalyzed by barium isopropoxide

4.2.1. Synthesis of 1,3,5-triphenylpentane-1,5-dione (entry 1 in Table 4, cf. entries 1–7 in Table 1, entry 1 in Table 2, entries 1–5 in Table 3, and Eq. 2).[6] An oven-dried, 30 mL two-necked round-bottomed flask equipped with a Teflon®-coated magnetic stirring bar was flushed with argon. Barium isopropoxide (26 mg, 0.10 mmol) was put into the apparatus and dry DMF (5 mL) and 2-propanol (1.5 mL, 20 mmol) were added, and the mixture was stirred for 10 min at room temperature. To the resulting solution were added acetophenone (0.35 mL, 3.0 mmol) and benzaldehyde (0.10 mL, 1.0 mmol). After being stirred for 7 h at room temperature, the mixture was treated with a saturated NH_4Cl aqueous solution (10 mL) at this temperature and the aqueous layer was extracted with ether (20 mL). The combined organic extracts were dried over anhydrous Na_2SO_4 and concentrated in vacuo after filtration. The residual crude product was purified by column chromatography on silica gel to give the 1,5-diketone (0.25 g, 76% yield) as colorless crystals. Spectral data (IR, ^{1}H NMR, ^{13}C NMR, and MS) and melting point of the product have been reported in the literature.[6]

4.2.2. 1,5-Bis(4-methoxyphenyl)-3-phenylpentane-1,5-dione (entry 2 in Table 4, cf. entry 2 in Table 2).[7] IR (KBr) 2938, 2880, 2839, 1677, 1598, 1510, 1455, 1421 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ=3.26 (dd, 2H, J=16.4, 7.2 Hz, CH_2), 3.42 (dd, 2H, J=16.4, 7.0 Hz, CH_2), 3.79 (s, 6H, $2CH_3$), 4.04 (m, 1H, CH), 6.97 (d, 4H, J=8.9 Hz, aromatic), 7.14 (t, 1H, J=6.8 Hz, aromatic), 7.26 (m, 4H, aromatic), 7.92 (d, 4H, J=8.9 Hz, aromatic); ^{13}C NMR (125 MHz, $CDCl_3$) δ=37.6, 44.7, 55.4, 113.7, 126.6, 127.4, 128.6, 130.0, 130.4, 144.0, 163.4, 197.2; MS (FAB) *m/e*: 389 (M+H). These spectral data have not been reported in the literature.[7]

4.2.3. 1,5-Di(naphthalen-1-yl)-3-phenylpentane-1,5-dione (entry 3 in Table 4, cf. entry 3 in Table 2). IR ($CDCl_3$) 3055, 2956, 1676, 1506 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ=3.39 (dd, 2H, J=16.4, 7.6 Hz, CH_2), 3.56 (dd, 2H, J=16.5, 6.8 Hz, CH_2), 4.13 (m, 1H, CH), 7.11–7.22 (m, 5H, aromatic), 7.37 (t, 2H, J=7.4 Hz, aromatic), 7.41–7.47 (m, 4H, aromatic), 7.74 (dd, 2H, J=7.3, 1.2 Hz, aromatic), 7.75–7.80 (m, 2H, aromatic), 7.88 (d, 2H, J=8.3 Hz, aromatic), 8.29–8.34 (m, 2H, aromatic); ^{13}C NMR (125 MHz, $CDCl_3$) δ=38.1, 48.2, 124.2, 125.6, 126.3, 126.7, 127.3, 127.6, 127.6, 128.2, 128.5, 129.9, 132.4, 133.8, 135.9, 143.2, 202.8; MS (FAB) *m/e*: 429 (M+H).

4.2.4. 1,5-Diphenyl-3-*p*-tolylpentane-1,5-dione (entry 4 in Table 4, cf. entry 4 in Table 2).[6b] ^{13}C NMR (125 MHz, $CDCl_3$) δ=21.0, 36.8, 45.0, 125.1, 127.2, 128.1, 128.5, 129.2, 133.0, 136.9, 140.7, 198.6. Other spectral data (IR, ^{1}H NMR, and MS) of the product have been reported in the literature.[6b]

4.2.5. 3-(4-Methoxyphenyl)-1,5-diphenylpentane-1,5-dione (entry 5 in Table 4, cf. entry 5 in Table 2).[6b,8] Spectral data (IR, ^{1}H NMR, ^{13}C NMR, and MS) and melting point of the product have been reported in the literature.[6b,8]

4.2.6. 3-(4-Nitrophenyl)-1,5-diphenylpentane-1,5-dione (entry 6 in Table 4, cf. entry 6 in Table 2).[9] IR ($CDCl_3$) 3064, 2902, 1682, 1597, 1516, 1344 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ=3.38 (dd, 2H, J=17.1, 7.3 Hz, CH_2), 3.52 (dd, 2H, J=17.1, 6.3 Hz, CH_2), 4.18 (m, 1H, CH), 7.36–7.44 (m, 6H, aromatic), 7.49 (t, 2H, J=7.4 Hz, aromatic), 7.86 (d, 4H, J=7.1 Hz, aromatic), 8.12 (d, 2H, J=8.5 Hz, aromatic); ^{13}C NMR (125 MHz, $CDCl_3$) δ=36.7, 44.2, 123.8, 128.0, 128.5, 128.7, 133.4, 136.5, 151.7, 197.5; MS (FAB) *m/e*: 374 (M+H). These spectral data have not been reported in the literature.[9]

4.2.7. 3-(4-Fluorophenyl)-1,5-diphenylpentane-1,5-dione (entry 7 in Table 4, cf. entry 7 in Table 2). IR (neat) 3060, 2970, 1682, 1598, 1580, 1510, 1448 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ=3.30 (dd, 2H, J=16.7, 7.2 Hz, CH_2), 3.47 (dd, 2H, J=16.7, 6.5 Hz, CH_2), 4.08 (m, 1H, CH), 6.91 (t, 2H, J=8.8 Hz, aromatic), 7.24 (m, 2H, aromatic), 7.39 (t, 4H, J=7.6 Hz, aromatic), 7.49 (t, 2H, J=7.4 Hz, aromatic), 7.92 (d, 4H, J=7.2 Hz, aromatic); ^{13}C NMR (125 MHz, $CDCl_3$) δ=36.4, 44.9, 115.2, 115.4, 128.0, 128.6, 133.1, 136.8, 139.4, 198.3; MS (FAB) *m/e*: 347 (M+H).

4.2.8. 3-(Naphthalen-1-yl)-1,5-diphenylpentane-1,5-dione (entry 8 in Table 4). IR ($CDCl_3$) 3059, 2893, 1680, 1597, 1448 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ=3.42 (dd, 2H, J=17.1, 6.6 Hz, CH_2), 3.52 (dd, 2H, J=17.1, 7.1 Hz, CH_2), 4.96 (m, 1H, CH), 7.15–7.39 (m, 10H, aromatic), 7.66 (m, 1H, aromatic), 7.71 (m, 1H, aromatic), 7.93 (m, 4H, aromatic), 8.08 (m, 1H, aromatic); ^{13}C NMR (125 MHz, $CDCl_3$) δ=37.2, 44.9, 125.5, 125.9, 126.0, 127.6, 127.7, 128.1, 128.3, 128.6, 132.4, 133.1, 133.5, 136.9, 141.3, 198.4; MS (FAB) *m/e*: 379 (M+H).

4.2.9. 3-(Naphthalen-2-yl)-1,5-diphenylpentane-1,5-dione (entry 9 in Table 4, cf. entry 8 in Table 2). IR (KBr) 3055, 2963, 2898, 1677, 1596, 1579, 1508, 1445, 1409 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ=3.43 (dd, 2H, J=16.9, 7.2 Hz, CH_2), 3.52 (dd, 2H, J=16.9, 7.0 Hz, CH_2), 4.25 (m, 1H, CH), 7.36–7.53 (m, 9H, aromatic), 7.69–7.78 (m, 4H, aromatic), 7.93 (d, 4H, J=7.0 Hz, aromatic); ^{13}C NMR (125 MHz, $CDCl_3$) δ=44.4, 123.1, 125.3, 125.6, 126.3, 127.2, 128.1, 128.5, 129.0, 131.4, 133.0, 134.1, 136.9, 140.0, 198.6; MS (FAB) *m/e*: 379 (M+H).

4.2.10. 1,5-Diphenyl-3-(pyridin-2-yl)pentane-1,5-dione (entry 10 in Table 4, cf. entry 9 in Table 2).[10] IR ($CDCl_3$) 3062, 2895, 1680, 1593, 1446 cm^{-1}; MS (FAB) *m/e*: 330 (M+H). Other spectral data (^{1}H NMR and ^{13}C NMR) and melting point of the product have been reported in the literature.[10]

4.2.11. 1,5-Diphenyl-3-(pyridin-3-yl)pentane-1,5-dione (entry 11 in Table 4, cf. entry 10 in Table 2). IR ($CDCl_3$) 3059, 2900, 1680, 1595, 1579, 1448 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ=3.40 (dd, 2H, J=17.1, 7.4 Hz, CH_2), 3.56 (dd, 2H, J=17.1, 6.7 Hz, CH_2), 4.13 (m, 1H, CH), 7.21 (dd, 1H, J=7.9, 4.9 Hz, aromatic), 7.45 (t, 4H, J=7.9 Hz, aromatic), 7.56 (t, 2H, J=7.4 Hz, aromatic), 7.67 (dt, 1H, J=8.0, 2.1 Hz, aromatic), 7.94 (dd, 4H, J=8.5, 1.5 Hz, aromatic), 8.44 (dd, 1H, J=4.8, 1.5 Hz, aromatic), 8.58 (d, 1H, J=1.9 Hz, aromatic); ^{13}C NMR (125 MHz, $CDCl_3$) δ=34.5, 44.2, 123.3, 128.0, 128.6, 133.2, 135.3,

136.6, 139.2, 148.0, 149.2, 197.8; MS (FAB) *m/e*: 330 (M+H).

4.2.12. 1,5-Diphenyl-3-(pyridin-4-yl)pentane-1,5-dione (entry 12 in Table 4, cf. entry 11 in Table 2).[11] ^{13}C NMR (125 MHz, $CDCl_3$) δ=36.1, 43.7, 122.9, 128.0, 128.6, 133.3, 136.5, 149.9, 152.9, 197.6; MS (FAB) *m/e*: 330 (M+H). Other spectral data (IR and ^{1}H NMR) and melting point of the product have been reported in the literature.[11]

4.3. Typical experimental procedure for Michael addition of acetophenone to α,β-unsaturated ketones catalyzed by barium isopropoxide

4.3.1. Synthesis of 3,4-dihydro-2-(3-oxo-1,3-diphenylpropyl)naphthalen-1(2*H*)-one (Eq. 3).[12] An oven-dried, 30 mL two-necked round-bottomed flask equipped with a Teflon®-coated magnetic stirring bar was flushed with argon. Barium isopropoxide (26 mg, 0.10 mmol) was put into the apparatus and dry DMF (3 mL) was added, and the mixture was stirred for 10 min at room temperature. To the resulting solution were added acetophenone (0.14 mL, 1.2 mmol) and (*E*)-2-benzylidene-3,4-dihydronaphthalen-1(2*H*)-one (235 mg, 1.0 mmol). After being stirred for 21 h at room temperature, the mixture was treated with a saturated NH_4Cl aqueous solution (10 mL) at this temperature and the aqueous layer was extracted with ether (20 mL). The combined organic extracts were dried over anhydrous Na_2SO_4 and concentrated in vacuo after filtration. The residual crude product was purified by column chromatography on silica gel to give the unsymmetrical 1,5-diketone (316 mg, 89% yield). Spectral data of the product (a 1:1 mixture of diastereomers): IR (neat) 3026, 2952, 2816, 1676, 1597, 1493, 1450 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.68 (m, 0.5H, one proton of CH_2), 2.02 (m, 1H, one proton of CH_2), 2.15 (m, 0.5H, one proton of CH_2), 2.72–3.10 (m, 3H, CH and CH_2), 3.29–3.64 (m, 2H, CH_2), 3.96 (m, 0.5H, CH), 4.22 (m, 0.5H, CH), 7.08–7.29 (m, 7H, aromatic), 7.31–7.49 (m, 4H, aromatic), 7.76–8.0 (m, 3H, aromatic); ^{13}C NMR (125 MHz, $CDCl_3$) δ=25.6, 26.2, 26.8, 29.4, 39.3, 39.8, 41.0, 43.5, 51.8, 53.2, 126.5, 126.6, 126.7, 127.4, 127.6, 128.0, 128.1, 128.2, 128.3, 128.4, 128.4, 128.5, 128.6, 128.6, 128.7, 132.8, 132.9, 133.0, 133.3, 133.4, 137.0, 142.0, 142.2, 143.5, 144.8, 198.4, 198.9, 199.0, 199.8; MS (FAB) *m/e*: 355 (M+H). These spectral data have not been reported in the literature.[12]

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References and notes

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