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Synthesis of 3-methyl-2-cyclohexenones catalyzed by mercury(II) salts and their microwave assisted $BiCl_3$ catalyzed aldol condensations

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ABSTRACT

$HgSO_4$ catalyzed hydrative cyclization of 1,6-heptadiynes is present. This reaction proceeded smoothly under the mild condition for differently 4-sustituted 1,6-diynic substrates giving corresponding 3-methyl-2-cyclohexenones with high to excellent yield. The microwave assisted aldol condensation of cyclohexenones under the catalysis of $BiCl_3$ afforded 3-styryl-cyclohexenones with high regio- and stereo-selectivity.

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1. Introduction

The cyclohexenone derivatives are the starting materials and intermediates in many important natural products and synthetic drug syntheses [1]. Many synthetic methods for the construction of the conjugate cyclohexenone ring have been reported [2]. We have recently developed hydrative cyclization of 1,6-diynes to produce 3,5-substituted conjugate cyclohexenone ring systems using Au(I) or Pt(II) as a catalyst [3]. On the other hand, the aldol condensation is a very classic method for the formation of carbon–carbon bond. This reaction is important for the synthesis of natural products and other potentially biologically relevant substances. Control of the mixed aldol condensation between two different carbonyl compounds which present several possible sites for enolization is a challenging problem for synthetic chemists [4]. For α,β-unsaturated ketones, however, not only γ-position but also α'-position are capable of being deprotonated to form alkylation products. In the literature, Yamamoto and co-workers reported the directed aldol condensation of α, β-unsaturated cyclohexenones with aldehydes to generate γ-alkylation products by LDA, but this type condensation by Lewis acid has not been reported previously [5]. Herein, we wish to report the hydrative cyclization of 1,6-diynes in the presence of $HgSO_4$ and the subsequent of $BiCl_3$ catalyzed aldol condensation of the corresponding cyclohexenone derivatives with aldehydes.

2. Results and discussion

2.1. Mercury(II) salts catalyzed hydrative cyclization of 1,6-heptadiynes

Our initial studies focused on testing the feasibilities for the hydrative cyclization of 4,4-di(isopropanoloxycarbonyl)-1,6-heptadiyne (**1a**), catalyzed by mercury(II) salts (Table 1). The expected compound **2a** was isolated with 71% yield (entry 1) at 70 °C for 3 h from reaction of **1a** with H_2O (1000 mol%) using $HgSO_4$ (20 mol%) as the catalyst and trifluoromethanesulfonate (30 mol%) as the co-catalyst. To decrease the amount of catalyst ($HgSO_4$) to 5 mol% was ineffective to this reaction (entry 2), but yield of **2a** was reduced to 57% when the amount of $HgSO_4$ was reduced to 1 mol%. We test the effectives of the amount of H_2O, and found that a decrease in the amount of H_2O to 150 mol% should increase formation of **2a** (yield 81%, entry 4). However, formation of **2a** was decreased to 44% or 23% yield when the solvent had been changed to EtOH or reduced the amount of additive of trifluoromethanesulfonic acid to 10 mol%

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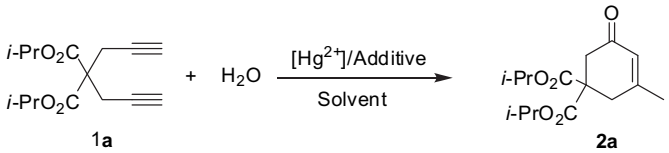
Table 1
Hg(II) salt catalized hydrative cyclization of **1a**.



1a **2a**

Entry	Cat. (mol%)	H_2O (mol%)	Additive (mol%)	Solvent	Temp. (°C)	Time (h)	Conv. (%)	Yield of **2** (%)[b]
1	$HgSO_4$ (20)	1000	CF_3SO_3H (30)	MeOH	70	3	>99	71
2	$HgSO_4$ (5)	1000	CF_3SO_3H (30)	MeOH	70	3	>99	71
3	$HgSO_4$ (1)	1000	CF_3SO_3H (30)	MeOH	70	3	>99	57
4	*$HgSO_4$ (5)*	*150*	*CF_3SO_3H (30)*	*MeOH*	*70*	*3*	*>99*	*81*
5	$HgSO_4$ (5)	150	CF_3SO_3H (30)	EtOH	70	3	>99	44
6	$HgSO_4$ (5)	150	CF_3SO_3H (10)	MeOH	70	3	>99	23
7	$Hg(OAc)_2$ (20)	1000	CF_3SO_3H (30)	MeOH	70	3	>99	68
8	$Hg(OAc)_2$ (5)	1000	CF_3SO_3H (30)	MeOH	70	3	>99	69
9	$Hg(OAc)_2$ (5)	150	CF_3SO_3H (30)	MeOH	70	3	>99	71
10	$Hg(OAc)_2$ (5)	150	CF_3SO_3H (30)	EtOH	70	3	>99	50
11	$HgCl_2$ (20)	1000	CF_3SO_3H (30)	MeOH	70	3	>99	70
12	$HgCl_2$ (5)	150	CF_3SO_3H (30)	MeOH	70	3	>99	17
13	$HgSO_4$ (5)	150	CF_3SO_3H (30)	MeOH	100	3	>99	46
14	$HgSO_4$ (5)	150	CF_3SO_3H (30)	MeOH	rt	10	~5	trace

[a]These reaction were carried out at 70 °C in MeOH (2 mL) for 3 h.
[b] Isolated yield.

(entries 5 and 6). Non obvious changes of yield of **2a** were observed when mercury(II) acetate was used as catalyst to the same reaction compared with mercury(II) sulfate, even though the amount reduced to 5 mol% (entry 7 and 8), **2a** was generated with 71% yield when the amount of H_2O was reduced to 150 mol% (entry 9), changed solvent to ethanol induced to decrease formation of **2a** (entry 10). When mercury(II) dichloride (20 mol%) was used for this reaction, **2a** was isolated with 70% yield (entry 11). We also test the same reaction by adding H_2O with 150 mol%, **2a** was obtained with 17% yield (entry 12). According to the results, we fixed the conditions (entry 4) for investigating the scope of mercury(II) sulfonate catalyzed hydrative cyclization of 1,6-heptadiynes.

Having reaction conditions being established, various 1,6-diynes were subjected to the reaction with water in order to investigate the reaction scope and several represent active results are summarized in Table 2. 5,5-Dimethoxycarbonyl and 5,5-diethoxycarbonyl substituted 3-methyl-2-hexenones (**2b** and **2c**) were obtained with high yield (Table 2, entries 2 and 3). Various 4-substituted 1,6-diynes were investigated, and the hydrative cyclization proceeded in mostly good efficiencies. The 5,5-di(alkoxycarbonyl)-substituted cyclohex-2-enones (**2d** and **2e**) were isolated with high yields (entries 4 and 5). The cyclic products with different substituent group pairs, such as diphenylphosphoryl and ethoxycarbonyl (**2f**), or phenyl and methoxycarbonyl (**2h**), or with single ethoxycarbonyl substituented (**2i**) were obtained with excellent yield respectively (entries 6, 8 and 9). From reaction of **1g**, compound **2h** was also obtained with 11% yield which was formed from **2g** via esterification of nitrile group (entry 7). Only **2b** was isolated with 45% yield from the reaction of **1j**.

2.2. Microwave assisted $BiCl_3$ catalyzed aldol condensation of cyclohexenones with aldehydes

Microwave dielectric heating causes an extremely rapid and uniform energy transfer to the reactants of chemical reactions. This will minimize by-product formation and increase product yields. Next we studied an improved, microwave assisted aldol condensation of methyl 3-mehyl-5-oxo-1-phenylcyclohex-3-ene-1-carboxylate (**2h**) with 2,3-dimethoxy (**3a**) (Table 3). Our initial experiments were conducted using $Hg(OTf)_2$, $Bi(NO_3)_3 \cdot 5H_2O$, $Fe(OTf)_2$, PPh_3AuCl_3, PPh_3AuNO_3, CuCN, or HgCl as the catalyst; however, the condensation product **4a** was formed in very low yield (entries 1–7, Table 3). When we used the $BiCl_3$ (10 mol%) as the catalyst, the yield of **4a** improved significantly (entries 8–13, Table 3). The best result was obtained by conducting the reaction in 150 °C for 1 h affording **4a** in 91% yield with perfect *oxo*-regioselectivity and high *E*-selectivity (entry 8, Table 3). It is remarkable to note that the reaction proceeded extremely well in the absence of any solvent (entries 14–16). To further assess the scope of this process, we have examined the aldol condensation of several cyclohexenones with aldehydes under the optimized reaction conditions in entry 8 of Table 3. The cyclohexenone with substituted group pairs, such as dimethoxycarbonyl **2b** is well tolerated, the corresponding adducts being formed in 50% (entry 3, Table 4). Various aldehydes were then examined, not only benzaldehyde but also electron-donating groups on benzene ring of benzaldehydes gave good isolated yields of the corresponding adducts (entries 2–5). In all cases, adducts were obtained with high selectivity.

3. Conclusion

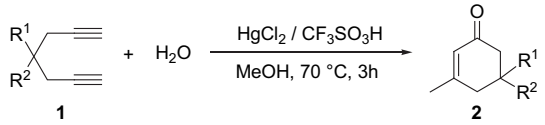
In conclusion, we have described a useful process of mercury(II) salts-catalyzed hydrative cyclization of 1,6-heptadiyne to provide 3-methyl-5-substituted-2-derivatives, advantages of the present method are the easily accessible starting materials, mild conditions, lack of coproducts and the fact that several types of functional groups were tolerated. We also have developed a directed microwave associated aldol condensation of the corresponding 3-methyl-5-substituted-2-hexenone derivatives with aromatic aldehydes catalyzed by $BiCl_3$ with perfect regioselectivity and high *E*-selectivity.

4. Experemental

4.1. General

Under otherwise noted, materials were obtained from commercial suppliers and used without further purification. Diynes were prepared by the procedures in the literature. Thin layer chromatography (TLC) was performed using silica gel 60 F254 and visualized using UV light. Column chromatography was performed with silica

Table 2
Mercury(II) sulfate catalyzed hydrative cyclization of 4-substituted 1,6-heptadiynes.[a]



1 **2**

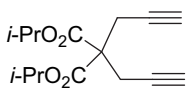
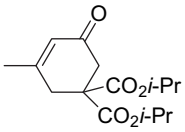
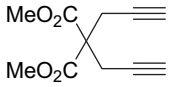
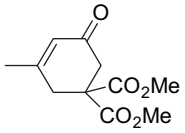
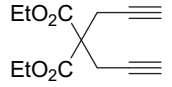
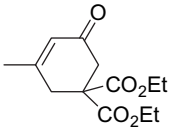
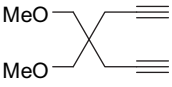
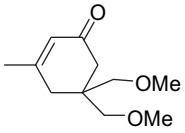
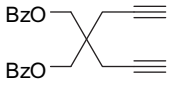
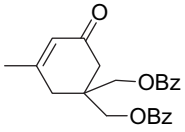
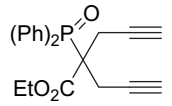
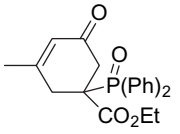
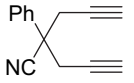
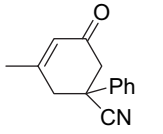
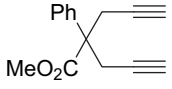
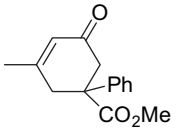
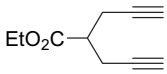
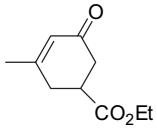
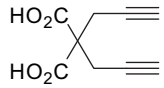
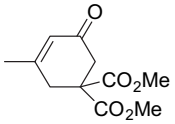
Entry	Diynes **1**	Product **2**	Yield (%)[b]
1	 **1a**	 **2a**	81
2	 **1b**	 **2b**	80
3	 **1c**	 **2c**	78
4	 **1d**	 **2d**	82
5	 **1e**	 **2e**	83
6	 **1f**	 **2f**	85
7	 **1g**	 **2g**	33[c]
8	 **1h**	 **2h**	91

Table 2 (*continued*)

Entry	Diynes **1**	Product **2**	Yield (%)[b]
9	 **1i**	 **2i**	90
10	 **1j**	 **2j**	45

[a] All the reactions were performed with **1** (0.5 mmol), H_2O (0.75 mmol), and catalyst ($HgSO_4$, 5 mol%) refluxed at 70 °C in MeOH (2 mL).
[b] Isolated yields after column chromatography.
[c] Compound **2h** was obtained with 11% yield as by-product.

gel (mesh 300–400). ^1H NMR and ^{13}C NMR spectra were recorded on a Bruker Avance 400 MHz or 500 MHz spectrometer in $CDCl_3$ with Me_4Si as an internal standard. Data are reported as follows: chemical shifts in ppm (δ), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, br = broad and m = multiplet), coupling constant (Hz) and integration. Infrared spectra (IR) were obtained on a 370 FT-IR spectrometer; absorptions are reported in cm^{-1}. Mass spectra (MS) and high resolution mass spectra (HRMS) were obtained at the Zhejiang University of Technology Mass Spectrometry Facility.

4.2. General procedure for the hydrative cyclization of diynes

To a reactor containing diyne (0.5 mmol), methanol (2 mL), and H_2O (150 mol%) under nitrogen $HgSO_4$ (5 mol%) and TfOH (30 mo% l) were added. The resulting yellow solution was then sealed and

Table 3
Lewis acid catalyzed aldol condensation of **2h** with **3a**.[a]

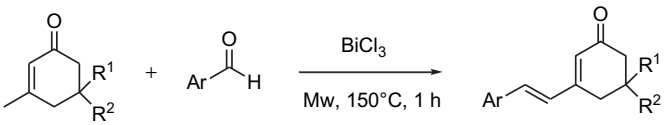
2h **3a** **4a**

.

Entry	Cat. (mol%)	Solvent	Temp. (°C)	Time (h)	Yield of **4a** (%)[b]
1	$Hg(SO_3CF_3)_2$	–	150	1	14
2	$Bi(NO_3)_3{\cdot}5H_2O$	–	150	1	trace
3	PPh_3AuCl_3	–	150	1	trace
4	PPh_3AuNO_3	–	150	1	trace
5	CuCN	–	150	1	trace
6	$Fe(SO_3CF_3)_2$	–	150	1	11
7	HgCl	–	150	1	trace
8	$BiCl_3$	–	150	1	91
9	$BiCl_3$	–	150	2	82
10	$BiCl_3$	–	150	3	32
11	$BiCl_3$	–	200	1	55
12	$BiCl_3$	–	100	1	27
13	$BiCl_3$	–	100	3	33
14	$BiCl_3$	THF	150	3	trace
15	$BiCl_3$	DME	150	3	trace
16	$BiCl_3$	MeCN	150	3	trace

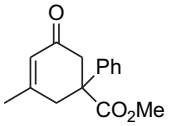
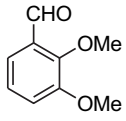
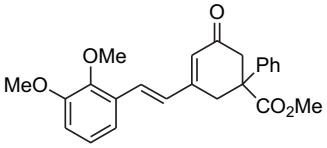
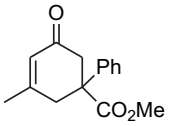
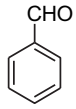
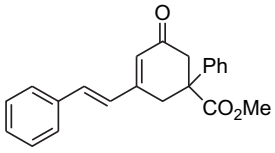
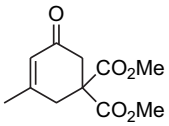
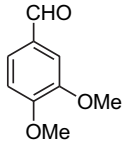
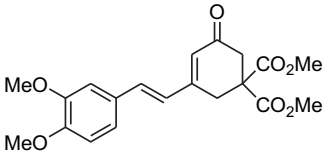
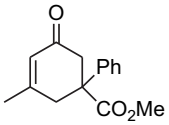
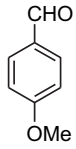
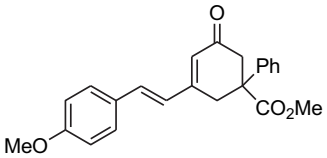
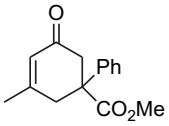
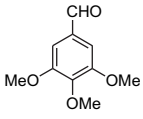
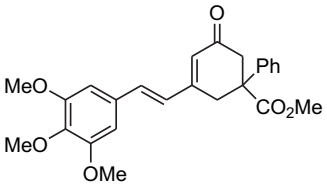
[a] All the reactions were performed with **2h** (0.14 mmol), **3a** (0.56 mmol), and catalyst (10 mol%).
[b] Isolated yields after column chromatography.

Table 4
The condensation reaction of 3-methyl-2-cyclohexenones with aromatic benzaldehyde[a]:



2 **3** **4**

.

Entry	Cyclohexenone	Aldehyde	Product	Yield[b] (*Z*/*E*)[c]
1	 **2h**	 **3a**	 **4a**	91 (100:6)
2	 **2h**	 **3b**	 **4b**	66 (100:5)
3	 **2b**	 **3c**	 **4c**	50 (100:3)
4	 **2h**	 **3d**	 **4d**	51 (100:0)
5	 **2h**	 **3e**	 **4e**	37 (100:0)

[a] All the reactions were performed with **2** (0.14 mmol), **3** (0.56 mmol), and catalyst (10 mol%).
[b] Isolated yields after column chromatography.
[c] Determined by ^{1}H NMR analysis.

stirred at 70 °C for 3–10 h until the starting diyne was consumed, as judged by TLC. The mixture was quenched with a saturated solution of $NaHCO_3$ and then extracted with ethyl acetate (20 mL × 3). The organic layer was washed with brine, dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by column chromatography (silica gel) (Eluent: hexane/ethyl acetate) to yield the corresponding products in an analytically pure form.

4.3. General procedure for the aldol condensation of 3-methyl-2-cyclohexenone with aldehydes

To a reactor containing 3-methyl-2-cyclohexenone (0.14 mmol) and aldehyde (0.56 mmol) $BiCl_3$ (5 mol%) was added. The resulting yellow solution was then sealed and reacted at 150 °C for 1–3 h under the MW condition. The mixture was quenched with a saturated solution of $NaHCO_3$ and then extracted with ethyl acetate (20 mL × 3). The organic layer was washed with brine, dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by column chromatography (silica gel) (Eluent: hexane/ethyl acetate) to yield the corresponding products.

*4.3.1. (E)-Methyl 3-(2,3-dimethoxystyryl)-5-oxo-1-phenylcyclohex-3-enecarboxylate (**4a**) (E/Z = 100:6)*

Mp: 139–142 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.95 (d, J = 13 Hz, 1H), 3.07 (d, J = 13 Hz, 1H), 3.30 (d, J = 13 Hz, 1H), 3.58 (d, J = 13 Hz, 1H), 3.64 (s, 3H), 3.87 (s, 3H), 3.88 (s, 3H), 6.09 (s, 1H), 6.91

(*t*, *J* = 21 Hz, 2H), 7.05 (*t*, *J* = 13 Hz, 1H), 7.18 (d, *J* = 6 Hz, 1H), 7.30 (*t*, *J* = 13 Hz, 1H), 7.37 (*t*, *J* = 12 Hz, 2H), 7.41 (d, *J* = 6 Hz, 2H), 7.50 (d, *J* = 13 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.1, 174.0, 154.9, 153.0, 147.7, 130.3, 129.9, 129.8, 129.0, 127.8, 127.7, 125.7, 124.3, 118.2, 113.0, 61.3, 55.8, 52.9, 51.7, 46.0, 34.4. HRMS (EI) for $C_{24}H_{24}O_5$: calcd. 392.1624. Found 392.1617.

4.3.2. (E)-Methyl 5-oxo-1-phenyl-3-styrylcyclohex-3-enecarboxylate (E/Z = 100:5)

^{1}H NMR (400 MHz, $CDCl_3$): major: δ 2.95 (d, *J* = 13 Hz, 1H), 3.04 (d, *J* = 13 Hz, 1H), 3.30 (d, *J* = 13 Hz, 1H), 3.55 (d, *J* = 13 Hz, 1H), 3.64 (s, 3H), 3.83 (s, 3H), 6.09 (s, 1H), 6.92 (d, *J* = 13 Hz, 1H), 7.16 (d, *J* = 13 Hz, 1H), 7.30 (m, 2H), 7.37 (*t*, *J* = 12 Hz, 4H), 7.41 (d, *J* = 6 Hz, 2H), 7.51 (d, *J* = 6 Hz, 2H); minor: δ 6.22 (d, *J* = 9.6 Hz, 1H), 6.83 (d, *J* = 9.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.8, 174.0, 154.3, 140.0, 135.9, 135.7, 129.3, 128.9, 128.6, 127.8, 127.7, 127.4, 125.7, 52.8, 51.6, 45.9, 34.5. HRMS (EI) for $C_{22}H_{20}O_3$: calcd. 332.1412. Found 332.1413.

*4.3.3. (E)-Dimethyl 3-(3,4-dimethoxystyryl)-5-oxocyclohex-3-ene-1,1-dicarboxylate (**4c**) (E/Z = 100:3)*

Mp: 139–142 °C; ^{1}H NMR (400 MHz, $CDCl_3$) major: δ 2.96 (s, 2H), 3.23 (s, 2H), 3.76 (s, 6H), 3.92 (s, 3H), 3.93 (s, 3H), 6.05 (s, 1H), 6.72 (d, *J* = 13 Hz, 1H), 6.86 (d, *J* = 7.0 Hz, 1H), 7.08 (m, 3H); minor: 5.68 (d, *J* = Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.9, 174.1, 154.8, 150.3, 149.2, 140.0, 136.0, 128.9, 127.8, 125.7, 121.6, 111.0, 109.1, 55.9, 51.6, 45.8,34.4. HRMS (EI) for $C_{20}H_{22}O_7$: calcd. 374.1366. Found 374.1356.

*4.3.4. (E)-Methyl 3-(4-methoxystyryl)-5-oxo-1-phenylcyclohex-3-enecarboxylate (**4d**)*

Mp: 91–95 °C; ^{1}H NMR (400 MHz, $CDCl_3$) major: δ 2.95 (d, *J* = 13 Hz, 1H), 3.02 (d, *J* = 13 Hz, 1H), 3.29 (d, *J* = 13 Hz, 1H), 3.56 (d, *J* = 13 Hz, 1H), 3.65 (s, 3H), 3.85 (s, 3H), 6.07 (s, 1H), 6.76 (d, *J* = 13 Hz, 1H), 6.91 (d, *J* = 7 Hz, 2H), 7.12 (d, *J* = 13 Hz, 1H), 7.30 (*t*, *J* = 12 Hz, 1H), 7.37 (*t*, *J* = 12 Hz, 2H), 7.42 (d, *J* = 6 Hz, 2H), 7.47 (d, *J* = 7 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) major: δ 196.8, 174.0, 160.7, 154.8, 140.1, 135.7, 128.9, 128.5, 127.7, 126.8, 126.8, 126.4, 125.7, 114.4, 55.3, 52.8, 51.6, 45.9, 34.5; HRMS (EI) for $C_{23}H_{22}O_4$: calcd. 362.1518. Found 362.1511.

*4.3.5. (E)-Methyl 5-oxo-1-phenyl-3-(3,4,5-trimethoxystyryl) cyclohex-3-enecarboxylate (**4e**)*

Mp: 105–109 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.96 (d, *J* = 13 Hz, 1H), 3.04 (d, *J* = 13 Hz, 1H), 3.30 (d, *J* = 13 Hz, 1H), 3.57 (d, *J* = 14 Hz, 1H), 3.64 (s, 3H), 3.87 (s, 3H), 3.90 (s, 6H), 6.10 (s, 1H), 6.74 (s, 2H), 6.82 (d, *J* = 13 Hz, 1H), 7.10 (d, *J* = 13 Hz, 1H), 7.30 (*t*, *J* = 11 Hz, 1H), 7.38 (*t*, *J* = 12 Hz, 2H), 7.42 (d, *J* = 6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.9, 174.0, 154.3, 153.4, 139.9, 136.0, 131.3, 128.9, 127.9, 127.8, 127.5, 125.7, 125.5, 104.5, 61.0, 56.1, 52.9, 51.6, 45.8, 34.5. HRMS (EI) for $C_{23}H_{30}O_6$: calcd. 402.2042. Found 402.2047.

Appendix A. Supplementary information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.jorganchem.2010.08.037.

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