Iodine(III)-mediated α-acetoxylation of aromatic ketones

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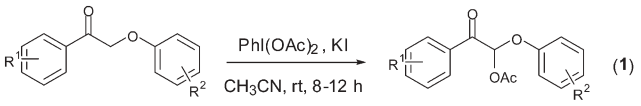
In the presence of iodobenzene diacetate with potassium iodide, aromatic α-phenoxy ketones underwent α-acetoxylation to afford corresponding α-acetoxy ketones smoothly at room temperature in moderate to good yields.

Keywords: α-acetoxyaromatic ketones, iodobenzene diacetate, potassium iodide

α-Acetoxy aromatic ketones are versatile intermediates in the synthesis of many biologically-active compounds.[1,2] The conventional synthetic methods for these compounds involve the metal-induced α-acetoxylation of ketones such as $Mn(OAc)_3$,[3–5] $CF_3SO_3Tl(I)$[6] and $Pb(OAc)_4$.[7–8] However, these methods require the use of hazardous chemicals. Hypervalent iodine compounds have been used extensively in organic synthesis as a result of their benign environmental character, ready availability and versatility.[9–24] An alternative approach may involve iodine(III)-mediated α-acetoxylation reaction for α-acetoxy ketones.[21–24] For example, (1) the treatment of *p*-substituted acetophenones with iodobenzene diacetate in acetic acid / acetic anhydride in the presence of sulfuric acid,[21–22] (2) nosyloxylation of aromatic ketones and subsequent solvolysis using acetic acid in the presence of silver carbonate,[23] and (3) tosyloxylation of ketones and subsequent solvolysis reaction in DMA and addition of water.[24] In these cases, the method is limited to substrate specificity, low yields or a cumbersome workup procedure. Recently an iodoxybenzene-catalysed α-acetoxylation reaction has also been reported,[25–27] the yield is still undesirab-le. Thus, the development of a new iodine (III)-mediated route to construct α-acetoxy aromatic ketones efficiently and selectively is still significant. Herein we wish to report a mild and general protocol for acetoxylation of aromatic ketones by the combination of $PhI(OAc)_2$ with KI [Equation (1)].

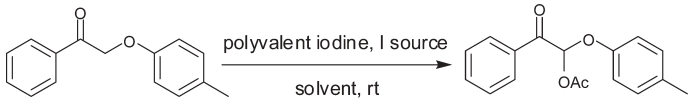


(1)

1-Phenyl-2-(p-tolyloxy)ethanone (**1a**) was chosen as the starting substrate to identify the optimal reaction conditions, and the results are summarised in Table 1. Initially, a series of polyvalent iodine compounds combined with an iodine source were investigated in CH_3CN at room temperature in air. In the absence of any iodine source, the reaction of ketone **1a** with $PhI(OAc)_2$ did not occur (entry 1). Identical results were also obtained using either I_2 or NIS (entries 2 and 3). Gratifyingly, the reaction of ketone **1a** with $PhI(OAc)_2$ and KI in CH_3CN gave the corresponding product 1-acetoxy-2-phenyl-1-(p-tolyloxy)ethan-2-one (**2a**) in 83 % yield for 8 h (entry 4). Changing the iodine source for NaI or Bu_4NI did not improve the results (entries 5 and 6). Subsequently, other solvents, including THF, DCE, were investigated but were less effective (entries 7 and 8). Finally, two other hypervalent iodine were also investigated. With iodomesitylene diacetate instead of $PhI(OAc)_2$, the reaction was less effective but still gave a good yield but no reaction was observed when the $PhI(OAc)_2$ was replaced by $PhI(O_2CCF_3)_2$ (entries 9 and 10).

These results led us to explore the scope of the α-acetoxylation reaction under these optimal conditions (Table 2). Firstly, a set of 1-aryl-2-phenoxyethanones **1b–e**, being electron-rich or electron-deficient substituents were treated with $PhI(OAc)_2$ and KI reacted smoothly to give products in moderate to good yields (entries 1–4). Gratifyingly, a good yield was still isolated from naphthyl substrate (entry 5). The screening results showed that several functional groups such as methoxy, iodo, cholo, allyl groups on the aryl ring of 2-phenoxy-1-phenylethanone moiety were perfectly tolerated (entries 6–10). A moderate electronic substrate effect was observed. For example, reaction of 1-phenyl-2-(p-tolyloxy)ethanone (**1a**) gave rise to the corresponding product (**2a**) in 83% yield, while reaction of 2-(4-chlorophenoxy)-1-phe-nylethanone (**1j**) reacted in 37% yield, respectively. In the presence of $PhI(OAc)_2$ and KI, **1l** and **1m** were also compatible with the reaction conditions and

Table 1 Screening optimal conditions[a]



1a **2a**

Entry	Polyvalent iodine /equiv.	I source /equiv.	Solvent	Time/h	Yield/%[b]
1	$PhI(OAc)_2$ (2)	___	CH_3CN	24	0
2	$PhI(OAc)_2$ (2)	I_2 (3)	CH_3CN	24	0
3	$PhI(OAc)_2$ (2)	NIS (3)	CH_3CN	24	0
4	$PhI(OAc)_2$ (2)	KI (3)	CH_3CN	8	83
5	$PhI(OAc)_2$ (2)	NaI (3)	CH_3CN	8	52
6	$PhI(OAc)_2$ (2)	Bu_4NI (3)	CH_3CN	24	80
7	$PhI(OAc)_2$ (2)	KI (3)	THF	8	65
8	$PhI(OAc)_2$ (2)	KI (3)	DCE	8	58
9	Iodomesitylene diacetate (2)	KI (3)	CH_3CN	16	77
10	$PhI(O_2CCF_3)_2$(2)	KI (3)	CH_3CN	24	0

[a] Reaction conditions: substrate **1a** (0.2 mmol), polyvalent iodine, I sourse and solvent (2 mL) at room temperature. [b] Isolated yield.

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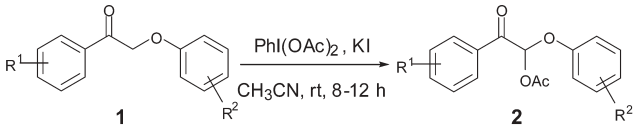
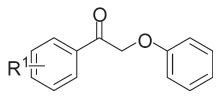
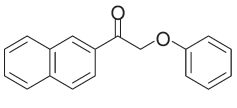
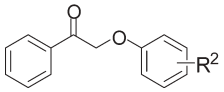
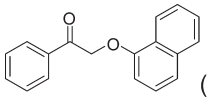
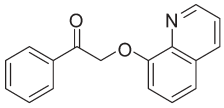
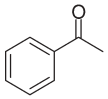
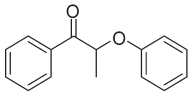
Table 2 $PhI(OAc)_2$-mediated α-acetoxylation of aromatic ketones[a]



1 **2**

Entry	Substrate **1**	Yeild/%[b]
		
1	R^1 = H (**1b**)	83(**2b**)
2	R^1 = 4-Me (**1c**)	91(**2c**)
3	R^1 = 4-MeO (**1d**)	81(**2d**)
4	R^1 = 4-F (**1e**)	62(**2e**)
5	 (**1f**)	77(**2f**)
		
6	R^2 = 2-MeO (**1g**)	82(**2g**)
7	R^2 = 3-MeO (**1h**)	81(**2h**)
8	R^2 = 2-I (**1i**)	75(**2i**)
9	R^2 = 4-Cl (**1j**)	37(**2j**)
10	R^2 = 2-Allyl (**1k**)	68(**2k**)
11	 (**1l**)	78(**2l**)
12	 (**1m**)	76(**2m**)
13	 (**1n**)	80(**2n**)
14	 (**1o**)	trace

[a] Reaction conditions: substrate **1** (0.2 mmol), $PhI(OAc)_2$ (2 equiv.) and KI (3 equiv.) in MeCN (2 mL) at room temperature for 8-12 h. [b] Isolated yield.

transformed into the desired product **2l** and **2m** in 78% and 76% yields, respectively. However, the reaction of substrate 2-phenoxy-1-phenylpropan-1-one (**1o**) was unsucceseful under the optimal conditions (entries 14).

A working mechanism as outlined in Scheme 1 is proposed on the basis of the present results and the previously reported mechanisms.[15,23,28,29] Initially, an iodine(III) intermediate **A** is generated by the interaction of $PhI(OAc)_2$ with KI, and the KOAc acts as a base to deprotonate ketones **1**. The resulting enolate anion of substrate **B** reacts with the iodine(III) intermediate **A** via a ligand exchange reaction to yield an intermediate **C**. Finally, intermediate **C** undergoes the reductive elimination of PhI to the afford product **2**.

In summary, we have developed a mild and general protocol for the acetoxylation of aromatic α-phenoxy ketones. In the presence of $PhI(OAc)_2$ and KI, a wide variety of α-acetoxy α-phenoxy aromatic ketones obtained smoothly at room temperature in air in moderate to good yields. Work to apply the reaction in organic synthesis and develop more novel acetoxylation processes is currently underway.

Experimental

NMR spectroscopy was performed on a Bruker-500 spectrometer operating at 500 MHz (^{1}H NMR) and 125 MHz (^{13}C NMR). TMS (tetramethylsilane) was used an internal standard and $CDCl_3$ was used as the solvent. Micro analyses were performed on a Perkin-Elmer-2400 CHNS analyser in Changsha. Mass spectrometric analysis was performed on Bruker APEX IV FTMS mass spectrometer analysis. Melting points were determined on a digital melting-point apparatus and were not corrected. Synthesis of substrates (**1a–m**,**1o**) were accomplished via general routes from 2-bromo-1-arylethanone and arylphenol. [30] Substrate (**1n**) was purchased from Aldrich and used without further purification.

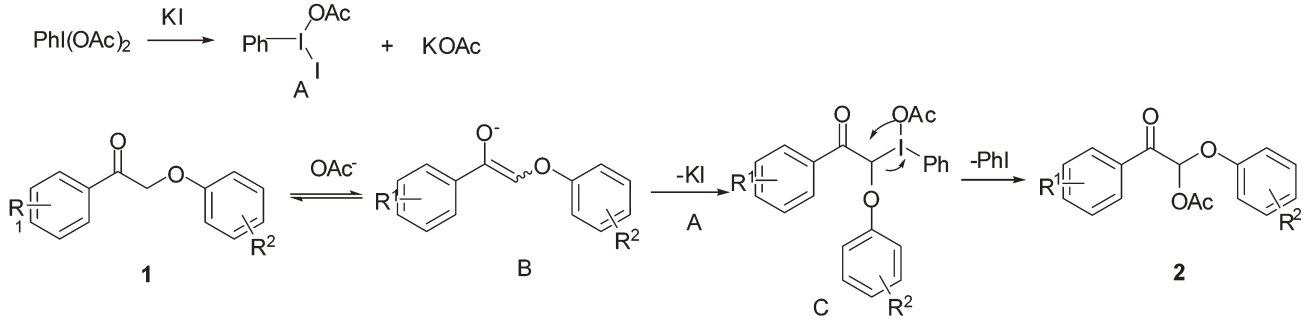
*Synthesis of α-acetoxy Aromatic Ketone (**2**); general procedure*

The aromatic ketone **1** (0.2 mmol), $PhI(OAc)_2$ (0.4 mmol), KI (0.6 mmol) in CH_3CN (2 mL) was stirred at room tempertature in air for 8–12 h. and the reaction was monitored by TLC and GC-MS. The reaction mixture was quenched with saturated $Na_2S_2O_3$, and extracted by ethyl ether (25 mL x 3). The organic layer was dried over anhydrous Na_2SO_4 and evaporated under vacuum. The residue was purified by flash column chromatography on silica gel (hexane/ethyl acetate) to afford the desired product **2**.

2-Oxo-2-phenyl-1-(p-tolyloxy)ethyl acetate (**2a**): Yellow oil; ^{1}H NMR (500 MHz, CDCl3) δ: 8.05 (d, J = 7.0 Hz, 2H), 7.62–7.59 (m, 1H), 7.47 (t, *J* = 7.5 Hz, 2H), 7.13 (d, *J* = 2.5 Hz, 1H), 7.11 (d, *J* = 3.5 Hz, 2H), 6.97 (d, *J* = 8.5 Hz, 2H), 2.31 (s, 3H), 2.19 (s, 3H); ^{13}C NMR (125 MHz, CDCl3) δ: 189.4, 169.7, 153.5, 134.0, 133.2, 130.3, 129.4, 128.7, 116.6, 93.8, 20.8, 20.6; IR (KBr, cm^{-1}): 1752, 1701; MS (EI): 284 (M^+), 241, 105, 43. Anal. Calcd for $C_{17}H_{16}O_4$: C, 71.82; H, 5.67. Found: C, 71.80; H, 5.64%.

2-Oxo-1-phenoxy-2-phenylethyl acetate (**2b**): Yellow solid, m.p. 50.1–51.9 °C (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.05 (d, *J* = 7.5 Hz, 2H), 7.61 (t, *J* = 7.5 Hz, 1H), 7.48 (t, *J* = 8.0 Hz, 2H), 7.35–7.32 (m, 2H), 7.16 (s, 1H), 7.13–7.07 (m, 3H), 2.20 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 189.3, 169.6, 155.6, 134.1, 133.2, 129.8, 129.4, 128.7, 123.7, 116.6, 93.4, 20.8; IR (KBr, cm^{-1}): 1752, 1701; MS (EI): 270 (M^+), 227, 105, 43. Anal. Calcd for $C_{16}H_{16}O_4$: C, 71.10; H, 5.22. Found: C, 71.05; H, 5.19%.

2-Oxo-1-phenoxy-2-p-tolylethyl acetate (**2c**): Yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.95 (d, *J* = 8.5 Hz, 2H), 7.35–7.32 (m, 2H), 7.27 (d, *J* = 8.0 Hz, 2H), 7.15 (s, 1H), 7.12–7.06 (m, 3H), 2.41 (s, 3H),



Scheme 1 A working mechanism.

2.20 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 188.9, 169.7, 155.6, 145.2, 130.7, 129.8, 129.5, 129.4, 123.6, 116.6, 93.3, 21.8, 20.8; IR (KBr, cm^{-1}): 1752, 1700; MS (EI): 284 (M^+), 241, 119, 43. Anal. Calcd for $C_{17}H_{16}O_4$: C, 71.82; H, 5.67. Found: C, 71.76; H, 5.60%.

2-(4-Methoxyphenyl)-2-oxo-1-phenoxyethyl acetate (**2d**): Yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.04 (d, *J* = 9.0 Hz, 2H), 7.34–7.31 (m, 2H), 7.13 (s, 1H), 7.11–7.06 (m, 3H), 6.94 (d, *J* =8.5 Hz, 2H), 3.85 (s, 3H), 2.19 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 187.7, 169.6, 164.2, 155.6, 131.8, 129.8, 126.0, 123.5, 116.5, 113.9, 93.4, 55.4, 20.8; IR (KBr, cm^{-1}): 1752, 1700; MS (EI): 300 (M^+), 257, 135, 43. Anal. Calcd for $C_{17}H_{16}O_5$: C, 67.99; H, 5.37. Found: C, 67.87; H, 5.28%.

2-(4-Fluorophenyl)-2-oxo-1-phenoxyethyl acetate (**2e**): Yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.12–8.09 (m, 2H), 7.36–7.33 (m, 2H), 7.17–7.14 (m, 3H), 7.12 (s, 1H), 7.11–7.05 (m, 2H), 2.21 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 188.0, 169.6, 155.4, 138.3, 132.3, 132.3, 129.9, 129.5, 123.8, 116.6, 93.6, 20.8; IR (KBr, cm^{-1}): 1752, 1700; MS (EI): 288 (M^+), 245, 123, 43. Anal. Calcd for $C_{16}H_{13}FO_4$: C, 66.66; H, 4.55. Found: C, 66.58; H, 4.47%.

2-(Naphthalen-2-yl)-2-oxo-1-phenoxyethyl acetate (**2f**): Yellow solid, m.p. 79.1–81.1 °C (uncorrected);^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.53 (s, 1H), 7.98 (d, *J* = 7.5 Hz, 1H), 7.84–7.77 (m, 3H), 7.54–7.51 (m, 1H), 7.47–7.44 (m, 1H), 7.27 (d, *J* = 2.5 Hz, 2H), 7.20 (d, *J* = 2.5 Hz, 1H), 7.05–7.02 (m, 3H), 2.14 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 189.4, 169.8, 155.7, 136.0, 132.4, 131.8, 130.6, 130.0, 129.2, 128.7, 127.9, 127.0, 124.5, 123.8, 116.8, 93.7, 21.0; IR (KBr, cm^{-1}): 1753, 1702; MS (EI): 320 (M^+), 277, 155, 43. Anal. Calcd for $C_{20}H_{16}O_4$: C, 74.99; H, 5.03. Found: C, 74.90; H, 4.97%.

1-(2-Methoxyphenoxy)-2-oxo-2-phenylethyl acetate (**2g**): Yellow solid, m.p. 80.1–82.3 °C (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.22–8.20 (m, 2H), 7.62–7.58 (m, 1H), 7.50–7.47 (m, 2H), 7.14–7.09 (m, 3H), 6.93–6.88 (m, 2H), 3.80 (s, 3H), 2.16 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 189.9, 169.5, 150.9, 144.3, 133.8, 133.5, 129.5, 128.5, 125.3, 121.0, 120.8, 112.5, 94.1, 55.6, 20.8; IR (KBr, cm^{-1}): 1752, 1700; MS (EI): 300 (M^+), 257, 105, 43. Anal. Calcd for $C_{17}H_{16}O_5$: C, 67.99; H, 5.37. Found: C, 67.89; H, 5.30%.

1-(3-Methoxyphenoxy)-2-oxo-2-phenylethyl acetate (**2h**): Yellow solid, m.p. 79.1–81.6 °C (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.05–8.03 (m, 2H), 7.62–7.59 (m, 1H), 7.49–7.46 (m, 2H), 7.25–7.22 (m, 1H), 7.15 (s, 1H),6.68–6.65 (m, 2H), 6.63 (t, *J* =2.5 Hz, 1H), 3.780 (s, 3H), 2.20 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 189.2, 169.6, 160.9, 156.7, 134.1, 133.1, 130.3, 129.4, 128.7, 109.3, 108.1, 103.0, 93.2, 55.3, 20.8; IR (KBr, cm^{-1}): 1752, 1700; MS (EI): 300 (M^+), 257, 105, 43. Anal. Calcd for $C_{17}H_{16}O_4$: C, 67.99; H, 5.37. Found: C, 67.88; H, 5.29%.

1-(2-Iodophenoxy)-2-oxo-2-phenylethyl acetate (**2i**): Yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.16–8.14 (m, 2H), 7.80–7.78 (m, *J* = 9.0 Hz, 1H), 7.64–7.61 (m, 1H), 7.52–7.49 (m, 2H), 7.35–7.32 (m, 1H), 7.15 (s, 1H), 7.09 (d, *J* = 8.0 Hz, 2H), 6.87–6.84 (m, 1H), 2.21 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 189.2, 169.4, 155.1, 140.0, 134.2, 132.9, 129.8, 129.7, 128.7, 125.4, 115.6, 94.4, 87.4, 20.8; IR (KBr, cm^{-1}): 1752, 1700; MS (EI): 396 (M^+), 353, 269, 105. Anal. Calcd for $C_{16}H_{13}IO_4$: C, 48.51; H, 3.31. Found: C, 48.42; H, 3.23%.

1-(4-Chlorophenoxy)-2-oxo-2-phenylethyl acetate (**2j**): Yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.95 (d, *J* = 7.5 Hz, 2H), 7.54 (t, *J* = 7.5 Hz, 1H), 7.42-7.40 (m, 2H), 7.28–7.20 (m, 2H), 7.02 (s, 1H), 6.95–6.92 (m, 2H), 2.14 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 189.1, 169.6, 154.1, 134.2, 133.1, 129.8, 129.4, 128.9, 128.8, 118.2, 93.4, 20.8; IR (KBr, cm^{-1}): 1752, 1701; IR (KBr, cm^{-1}): 1753, 1702; MS (EI): 304 (M^+), 261, 105, 43. Anal. Calcd for $C_{16}H_{13}ClO_4$: C, 63.06; H, 4.30. Found: C, 62.98; H, 4.23%.

1-(2-Allylphenoxy)-2-oxo-2-phenylethyl acetate (**2k**): Yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.05 (d, *J* = 7.5 Hz, 2H), 7.62–7.59 (m, 1H), 7.47 (t, *J* =8.0 Hz, 2H), 7.25–7.19 (m, 2H), 7.14 (s, 1H), 7.09–7.05 (m, 2H), 5.86–5.79 (m, 1H), 4.95–4.90 (m, 2H), 3.31 (d, *J* = 6.5 Hz, 2H), 2.19 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 189.4, 169.6, 153.4, 136.2, 134.0, 133.2, 130.6, 130.0, 129.4, 128.6, 123.5, 115.9, 113.8, 93.7, 34.1, 20.8; IR (KBr, cm^{-1}): 1752, 1700; MS (EI): 310 (M^+), 267, 105, 43. Anal. Calcd for $C_{19}H_{18}O_4$: C, 73.53; H, 5.85. Found: C, 73.43; H, 5.77%.

1-(Naphthalen-1-yloxy)-2-oxo-2-phenylethyl acetate (**2l**): Yellow solid, m.p. 72.2–74.6 °C (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.02–8.00 (m, 3H), 7.72 (d, *J* = 8.0 Hz, 1H), 7.52–7.49 (m, 2H), 7.41–7.31 (m, 5H), 7.27 (s, 1H), 7.05 (d, *J* = 8.0 Hz, 1H), 2.12 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 189.3, 169.7, 151.5, 134.6, 134.1, 133.2, 129.4, 128.7, 127.5, 126.7, 125.9, 125.7, 125.5, 123.3, 121.8, 108.1, 93.5, 20.8; IR (KBr, cm^{-1}): 1754, 1702; MS (EI): 320 (M^+), 277, 105. Anal. Calcd for $C_{20}H_{16}O_4$: C, 74.99; H, 5.03. Found: C, 74.89; H, 4.95%.

2-Oxo-2-phenyl-1-(quinolin-8-yloxy)ethyl acetate (**2m**): Yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.79–8.78 (m, 1H), 8.35 (d, *J* = 8.5 Hz, 2H), 8.16–8.14 (m, 1H), 7.71 (s, 1H), 7.60–7.57 (m, 2H), 7.50–7.39 (m, 5H), 2.15 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 169.6, 151.3, 149.2, 140.4, 136.2, 133.8, 133.7, 129.7, 129.6, 128.6, 126.5, 123.6, 121.6, 117.5, 94.0, 20.8; IR (KBr, cm^{-1}): 1758, 1709; MS (EI): 321 (M^+), 278, 105, 43. Anal. Calcd for $C_{19}H_{15}NO_4$: C, 71.02; H, 4.71; N, 4.36. Found: C, 70.92; H, 4.63; N, 4.32%.

2-Oxo-2-phenylethyl acetate (**2n**) [26]: White solid; M.p. 48–50 °C (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.95 (d, *J* = 7.0 Hz, 2H), 7.60 (t, *J* = 4.5 Hz, 1H), 7.47 (t, *J* = 8.0Hz, 2H), 5.36 (s, 2H), 2.24 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 190.2, 169.5, 134.2, 133.9, 128.7, 125.7, 65.8, 20.8; IR (KBr, cm^{-1}): 1751, 1701; MS (EI): 178(M^+), 105.

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