

## Note

### A convenient synthesis of $\gamma$ -methyl $\alpha$ -(alkylidene)- $\gamma$ -butyrolactones<sup>†</sup>

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$\gamma$ -Methyl- $\alpha$ -(alkylidene substituted)- $\gamma$ -butyrolactones have been synthesized in two steps. The anion of allyl phosphonate **1** is condensed with different carbonyl compounds to afford  $\alpha,\beta$ -unsaturated esters **2a-g** which are cyclised using 6*N* HCl to give title compounds.

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$\gamma$ -Substituted  $\alpha$ -(alkylidene)- $\gamma$ -butyrolactones continue to be target of new synthetic methodologies<sup>1</sup> due to the presence of this moiety in natural products<sup>2</sup> and the biological activity associated with these molecules. For example, these compounds show cytotoxic, antitumoral<sup>3,4</sup> antibacterial<sup>5</sup>, plant growth inhibition<sup>6</sup>, antifungal<sup>7</sup> and antiallergic activities.<sup>8</sup> In addition, these molecules have been used as synthetic precursors for the synthesis of cyclopentenones, butenolides and furans.<sup>1</sup>

Although there are various methods available for the synthesis of  $\gamma$ -substituted  $\alpha$ -(alkylidene substituted)- $\gamma$ -butyrolactones,<sup>1,9-18</sup> most of these involve use of organometallic reagents or multiple steps or harsh reaction conditions. Wittig reaction on aryl aldehydes for the synthesis of  $\alpha$ -benzylidene- $\gamma$ -methyl- $\gamma$ -butyrolactones is also reported.<sup>19</sup> However, this method is not reported for aliphatic aldehydes and fails in case of ketones. This prompts us to develop a simple

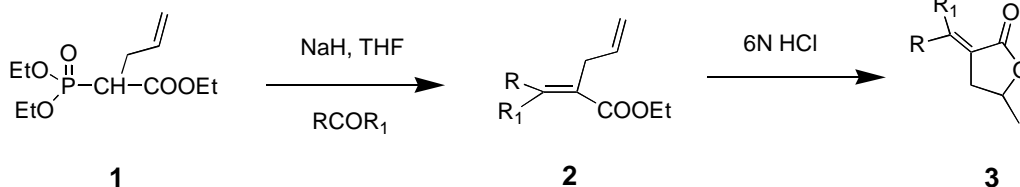
method utilizing Horner-Wordsworth-Emmons (HWE) reaction as a key reaction (**Scheme I**).

Initially, the anion of allyl phosphonate<sup>20</sup> generated at 0°C in THF was condensed with benzaldehyde to give exclusively (*E*)- $\alpha,\beta$ -unsaturated ester **2a** in excellent yield. 6*N* hydrochloric acid was chosen for lactonisation to avoid the problem of ring annulation to naphthalene. Thus,  $\alpha,\beta$ -unsaturated ester **2a** was refluxed with 6*N* HCl for 6hr to get the *E*-lactone **3a** in good yield. Similar sequence of reactions on other aryl aldehydes provided lactones **3b-d**. Compound **3d** is known to show antifungal activity<sup>7</sup>. This method was then extended for aliphatic aldehydes to get corresponding lactones **3e-f** (**Table I**). When ketonic compound acetone was reacted, we could obtain the corresponding lactone **3g**.

In conclusion, a convenient method has been developed using HWE reaction for the synthesis of  $\gamma$ -methyl  $\alpha$ -(alkylidene)- $\gamma$ -butyrolactones. The method becomes attractive because it can be also being applied to ketones. Further attempt to convert this method for the asymmetric synthesis of butenolides is in progress.

### Experimental Section

All melting points are uncorrected and measured by normal thiels tube method. Column chromatography was performed on silica gel 60-120 mesh size and TLC on silica gel G (13% CaSO<sub>4</sub> as binder). IR spectra were recorded on a Shimadzu FT-IR spectrophotometer (KBr pellet or neat sample). <sup>1</sup>H NMR and <sup>13</sup>CMR spectra (TMS, CDCl<sub>3</sub>) were recorded on a Bruker-300 MHz instrument. The multiplicities of carbon signals were obtained from DEPT experiments. Mass spectra were recorded on a QstarXL MS/MS of Applied Biosystems Mass, Canada.



Scheme I

<sup>†</sup> Dedicated to Dr. J. K. Kirtany

**Table I** — Yield for preparation of **2** and **3** and reaction time for **3**

Compd*	R	R <sub>1</sub>	<b>2</b> (% yield)	<b>3</b> (% yield)/m.p. °C	Reaction time (for <b>3</b> in hr)
<b>a</b>	C <sub>6</sub> H <sub>5</sub>	H	95	70	6
<b>b</b>	<i>o</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	H	90	60/(102-03)	6
<b>c</b>	<i>m</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	H	90	60/(125)	6
<b>d</b>	<i>o</i> -ClC <sub>6</sub> H <sub>4</sub>	H	85	58	6
<b>e</b>	CH <sub>3</sub> CH <sub>2</sub> CH <sub>2</sub>	H	70	60	9
<b>f</b>	(CH <sub>3</sub> ) <sub>2</sub> CH	H	65	65	9
<b>g</b>	CH <sub>3</sub>	CH <sub>3</sub>	40	60	6

\* All new compounds gave satisfactory elemental analysis for C and H.

**Table II** — <sup>1</sup>H NMR and <sup>13</sup>C NMR data for **2a-g**

Compd	<sup>1</sup> H NMR (δ, ppm)	<sup>13</sup> C NMR (δ, ppm)
<b>2a</b> <sup>19c</sup>	1.32 (t, 3H, <i>J</i> =7.1 Hz, -CH <sub>3</sub> ), 3.32 (d, 2H, <i>J</i> =5.5 Hz, -CH <sub>2</sub> -CH=), 4.24 (q, 2H, <i>J</i> =7.1 Hz, -OCH <sub>2</sub> -CH <sub>3</sub> ), 4.93-5.12 (m, 2H, =CH <sub>2</sub> ), 5.88-6.05 (m, 1H, -CH=CH <sub>2</sub> ), 7.19-7.50 (m, 5H, Ar-H), 7.78 (s, 1H, CH=C)	14.19 (CH <sub>3</sub> ), 31.56 (CH <sub>2</sub> ), 60.71 (OCH <sub>2</sub> ), 115.55 (=CH <sub>2</sub> ), 128.33, 128.46, 129.16, 130.48, 135.44, 135.61 and 140.04 (Ar, =CH and =C<), 167.83 (CO).
<b>2b</b> <sup>19a</sup>	1.35 (t, 3H, <i>J</i> =7.2 Hz, -CH <sub>3</sub> ), 3.05 (d, 2H, <i>J</i> =5.31 Hz, -CH <sub>2</sub> -CH=), 4.26 (q, 2H, <i>J</i> =7.2 Hz, -OCH <sub>2</sub> -CH <sub>3</sub> ), 4.82-5.07 (m, 2H, =CH <sub>2</sub> ), 5.84-5.96 (m, 1H, -CH=CH <sub>2</sub> ), 7.2-7.7 (m, 3H, Ar-H), 7.96 (s, 1H, CH=C), 8.15 (d, 1H, <i>J</i> =8.1 Hz, Ar-H)	14.07 (CH <sub>3</sub> ), 31.80 (CH <sub>2</sub> ), 60.96 (OCH <sub>2</sub> ), 115.72 (=CH <sub>2</sub> ), 124.32, 129.10, 130.77, 131.48, 132.17, 133.24, 135.40, 136.68 and 147.51 (Ar, =CH and =C<) 166.82 (CO)
<b>2c</b> <sup>19c</sup>	1.36 (t, 3H, <i>J</i> =7.1 Hz, -CH <sub>3</sub> ), 3.2 (d, 2H, <i>J</i> =5.4 Hz, -CH <sub>2</sub> -CH=), 4.29 (q, 2H, <i>J</i> =7.1 Hz, -OCH <sub>2</sub> -CH <sub>3</sub> ), 5.12-5.19 (m, 2H, =CH <sub>2</sub> ), 5.94-6.06 (m, 1H, -CH=CH <sub>2</sub> ), 7.50 (dd, 1H, <i>J</i> =7.8, 7.8 Hz, Ar-H), 7.69 (dd, 1H, <i>J</i> =7.2 Hz, 0.6 Hz, Ar-H), 7.79 (s, 1H, CH=C), 8.17 (dd, 1H, <i>J</i> =7.2 Hz and 0.9 Hz, Ar-H), 8.29 (s, 1H, Ar-H)	14.17 (CH <sub>3</sub> ), 31.68 (CH <sub>2</sub> ), 61.20 (OCH <sub>2</sub> ), 116.26 (=CH <sub>2</sub> ), 123.08, 123.86, 129.40, 133.44, 133.44, 134.80, 134.91, 137.12 and 148.37 (Ar, =CH, =C<), 167.20 (CO)
<b>2d</b>	1.35 (t, 3H, <i>J</i> =7.1 Hz, -CH <sub>3</sub> ), 3.15 (d, 2H, <i>J</i> =5.4 Hz, -CH <sub>2</sub> -CH=), 4.3 (q, 2H, <i>J</i> =7.1 Hz, -OCH <sub>2</sub> -CH <sub>3</sub> ), 5.02-5.11 (m, 2H, =CH <sub>2</sub> ), 5.89-6.02 (m, 1H, -CH=CH <sub>2</sub> ), 7.25-7.29 (m, 2H, Ar-H), 7.36-7.40 (m, 2H, Ar-H), 7.85 (s, 1H, CH=C)	14.17 (CH <sub>3</sub> ), 31.82 (CH <sub>2</sub> ), 60.97 (OCH <sub>2</sub> ), 115.77 (=CH <sub>2</sub> ), 125.35, 128.80, 130.25, 130.67, 131.05, 132.47, 134.13, 134.92 and 136.36 (Ar, =CH, =C<), 167.44 (CO)
<b>2e</b>	0.93 (t, 3H, <i>J</i> =7.2 Hz, -CH <sub>3</sub> ), 1.28 (t, 3H, <i>J</i> =7.2 Hz, -OCH <sub>2</sub> -CH <sub>3</sub> ), 1.44-1.53 (m, 2H, -CH <sub>2</sub> -), 2.16 (m, 2H, =CH-CH <sub>2</sub> -CH <sub>2</sub> ), 3.06 (d, 2H, <i>J</i> =5.7 Hz, -CH <sub>2</sub> -CH=), 4.18 (q, 2H, <i>J</i> =7.2 Hz, -OCH <sub>2</sub> -CH <sub>3</sub> ), 4.95-5.03 (m, 2H, =CH <sub>2</sub> ), 5.74-5.87 (m, 1H, -CH=CH <sub>2</sub> ), 6.84 (t, 1H, <i>J</i> =7.34 Hz, CH=C)	13.8 (CH <sub>3</sub> ), 14.15 (CH <sub>3</sub> ), 21.89 (CH <sub>2</sub> ), 30.46 (CH <sub>2</sub> ), 30.75 (CH <sub>2</sub> ), 60.30 (OCH <sub>2</sub> ), 114.84 (=CH <sub>2</sub> ), 130.02 and 135.55 (=CH), 143.45 (=C<), 167.52 (CO)
<b>2f</b>	1.03 (d, 6H, <i>J</i> =2.4 Hz, -CH(CH <sub>3</sub> ) <sub>2</sub> ), 1.29 (t, 3H, <i>J</i> =7.2 Hz, -CH <sub>3</sub> ), 2.64-2.67 (m, 1H, CH(CH <sub>3</sub> ) <sub>2</sub> ), 3.07 (d, 2H, <i>J</i> =5.3 Hz, -CH <sub>2</sub> -CH=), 4.19 (q, 2H, <i>J</i> =7.2 Hz, -OCH <sub>2</sub> -CH <sub>3</sub> ), 4.96-5.04 (m, 2H, =CH <sub>2</sub> ), 5.79-5.88 (m, 1H, -CH=CH <sub>2</sub> ), 6.62-6.66 (d, 1H, <i>J</i> =9.6 Hz, CH=C)	14.12 (CH <sub>3</sub> ), 22.01 (2 × CH <sub>3</sub> ), 27.78 (CH <sub>2</sub> ), 30.75 (CH <sub>2</sub> ), 60.30 (OCH <sub>2</sub> ), 114.78 (=CH <sub>2</sub> ), 127.60 and 135.96 (=CH), 149.86 (=C<), 167.72 (CO)
<b>2g</b> <sup>#</sup>	1.21 (t, 3H, <i>J</i> =7.1 Hz, -CH <sub>3</sub> ), 1.74 (s, 3H, CH <sub>3</sub> ), 2.99 (d, 2H, <i>J</i> =6.0 Hz, -CH <sub>2</sub> -CH=), 4.15 (q, 2H, <i>J</i> =7.1 Hz, -OCH <sub>2</sub> -CH <sub>3</sub> ), 4.89-4.97 (m, 2H, =CH <sub>2</sub> ), 5.60-5.80 (m, 1H, -CH=CH <sub>2</sub> )	14.27 (CH <sub>3</sub> ), 22.08 (CH <sub>3</sub> ), 23.02 (CH <sub>3</sub> ), 34.03 (CH <sub>2</sub> ), 66.02 (OCH <sub>2</sub> ), 114.96 (=CH <sub>2</sub> ), 125.30 (=CH), 135.51 and 144.14 (=C<), 169.99 (CO)

# MS-ESI(CH<sub>3</sub>OH + H<sub>2</sub>O + CH<sub>3</sub>COONa): *m/z*(amu) 169.1026 (M+1); Calculated 169.2352 (M+1).

**Table III** —  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR data for **3a-g**

Compd	$^1\text{H}$ NMR ( $\delta$ , ppm)	$^{13}\text{C}$ NMR ( $\delta$ , ppm)
<b>3a</b> <sup>18</sup>	1.47 (d, 3H, $J=6.3$ Hz, CH-CH <sub>3</sub> ), 2.81(ddd, 1H, $J= 17.4, 5.5$ and $3.0$ Hz, -HCH-), 3.39(ddd, 1H, $J=17.4, 7.7, 2.7$ Hz -HCH-), 4.79-4.84 (m, 1H, -CH-), 7.34-7.51 (m, 5H, Ar-H), 7.56 (t, 1H, $J=2.8$ Hz, CH=)	22.27 (CH <sub>3</sub> ), 35.20 (CH <sub>2</sub> ), 73.98 (CH), 124.90, 128.82, 129.68, 129.85, 134.64 and 136.42 (Ar, =CH, =C<), 171.95 (CO)
<b>3b</b> <sup>19b</sup>	1.47 (d, 3H, $J=6.3$ Hz, CH-CH <sub>3</sub> ), 2.69 (ddd, 1H, $J= 17.2, 7.3$ and $2.6$ Hz, -HCH-), 3.19 (ddd, 1H, $J= 17.2, 6.0$ and $3.1$ Hz, -HCH-), 4.64-4.80 (m, 1H, -CH-), 7.45-7.72 (m, 3H, Ar-H), 7.85 (t, 1H, $J= 2.7$ Hz, CH=), 8.12 (d, 1H, $J= 8.1$ Hz, Ar-H)	21.92 (CH <sub>3</sub> ), 34.53(CH <sub>2</sub> ), 74.23 (CH), 125.05, 129.56, 129.84, 130.35, 133.29, 134.95 and 148.40 (Ar, =CH, =C<), 170.33 (CO)
<b>3c</b> <sup>19c</sup>	1.52 (d, 3H, $J=5.5$ Hz, CH-CH <sub>3</sub> ), 2.89 (ddd, 1H, $J= 17.7, 8.5$ and $3.1$ Hz, -HCH-), 3.44 (ddd, 1H, $J=17.7, 10.4, 2.7$ Hz -HCH-); 4.78-4.87(m, 1H, -CH-), 7.61(t, 1H, $J=2.9$ Hz, CH=), 7.65(d, 1H, $J=8.10$ Hz, Ar-H), 7.81(d, 1H, $J=7.8$ Hz, Ar-H), 8.25 (d, 1H, $J= 8.1$ Hz, Ar-H), 8.34 (s, 1H, Ar-H)	22.30 (CH <sub>3</sub> ), 35.13 (CH <sub>2</sub> ), 74.24 (CH), 123.54, 124.06, 128.46, 130.00, 133.55, 135.77, 136.29, and 148.60 (Ar and =CH), 171.03 (CO)
<b>3d</b>	1.48 (d, 3H, $J=6.3$ Hz, CH-CH <sub>3</sub> ), 2.75 (ddd, 1H, $J= 17.4, 8.7$ and $3.0$ Hz, -HCH-), 3.30 (ddd, 1H, $J=17.4, 7.2, 2.7$ Hz -HCH-), 4.69-4.80 (m, 1H, -CH-), 7.30-7.34 (m, 2H, Ar-H), 7.44-7.49 (m, 2H, Ar-H), 7.90 (t, 1H, $J=2.7$ Hz, CH=)	22.11 (CH <sub>3</sub> ), 35.09 (CH <sub>2</sub> ), 74.18 (CH), 126.77, 127.72, 129.19, 130.20, 130.55, 132.54, 132.89 and 135.50 (Ar and =CH), 171.30 (CO).
<b>3e</b>	0.95 (t, 3H, $J=7.35$ Hz, -CH <sub>3</sub> ), 1.4 (d, 3H, $J=6.3$ Hz, CH-CH <sub>3</sub> ), 1.45-1.59 (m, 2H, -CH <sub>2</sub> -), 2.11-2.21 (m, 2H, -CH <sub>2</sub> -CH=), 2.43 (ddd, 1H, $J= 16.8, 7.8$ and $3.0$ Hz, -HCH-), 3.04 (ddd, 1H, $J=16.8, 6.8, 2.7$ Hz -HCH-), 4.64-4.75 (m, 1H, -CH-), 6.67-6.76 (m, 1H, CH=)	13.52 (CH <sub>3</sub> ), 21.24 (CH <sub>3</sub> ), 21.89 (CH <sub>3</sub> ), 31.90 (CH <sub>2</sub> ), 32.69 (CH <sub>2</sub> ), 73.89 (CH), 129.66(=CH), 140.36 (=C<), 170.85 (CO)
<b>3f</b>	1.07 (d, 6H, $J=6.6$ Hz, CH(CH <sub>3</sub> ) <sub>2</sub> ), 1.42 (d, 3H, $J=6.3$ Hz, CH-CH <sub>3</sub> ), 2.37-2.53 (m, 2H, -HCH- and CH(CH <sub>3</sub> ) <sub>2</sub> ), 3.02 (ddd, 1H, $J=16.8, 7.8, 2.7$ Hz -HCH-), 4.60-4.73 (m, 1H, -CH-), 6.55 (m, 1H, CH=)	21.36 (CH <sub>3</sub> ), 22.10 (CH <sub>3</sub> ), 29.67 (CH <sub>2</sub> ), 32.50(CH <sub>2</sub> ), 73.88 (CH), 124.25 (=CH), 146.75 (=C<), 171.20 (CO)
<b>3g</b> <sup>18#</sup>	1.38 (d, 3H, $J=6.3$ Hz, CH-CH <sub>3</sub> ), 1.86 (s, 3H, CH <sub>3</sub> ), 2.26 (s, 3H, CH <sub>3</sub> ), 2.43 (ddd, 1H, $J= 15.9, 7.8$ and $3.9$ Hz, -HCH-), 3.00 (ddd, 1H, $J= 15.9, 7.8$ and $3.9$ Hz, -HCH-), 4.51-4.63 (m, 1H, -CH-)	19.79 (CH <sub>3</sub> ), 22.27 (CH <sub>3</sub> ), 22.42 (CH <sub>3</sub> ), 35.42 (CH <sub>2</sub> ), 72.36 (CH), 119.72 and 149.88 (=C<), 170.19 (CO)

# MS-ESI(CH<sub>3</sub>OH + H<sub>2</sub>O + CH<sub>3</sub>COONa):  $m/z$ (amu)141.0883 (M+1); Calculated 141.1816 (M+1).

**General procedure for preparation of ethyl ( $\alpha$ -allyl)- $\alpha,\beta$ -unsaturated esters **2a-g**.** To a suspension of NaH (60% in mineral oil) (0.225g, 9.4 mmole) in dry THF (10 mL) was added dropwise with cooling allylphosphonate **1** (2.48g, 9.4 mmole). The mixture was stirred at 0°C, till evolution of H<sub>2</sub> ceased. The

corresponding carbonyl compound (9.4 mmole) in THF was added. After 30 min, aq. NH<sub>4</sub>Cl (10 mL) was added. The reaction mixture was then extracted with ether (3  $\times$  20 mL). The combined extracts were washed with brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and evaporated to dryness. Column chromatography of the

residue over silica gel using pet. ether as an eluent furnished the products **2a-g** as viscous oils.

**General procedure for preparation of  $\gamma$ -methyl  $\alpha$ -(alkylidene substituted)- $\gamma$ -butyrolactones **3a-f**.**

To ethyl ( $\alpha$ -allyl)- $\alpha,\beta$ -unsaturated esters **2a-g** (4.6 mmole) was added 6*N* HCl (10 mL). The reaction mixture was then refluxed for the time as mentioned in **Table I**. It was then cooled and extracted with CHCl<sub>3</sub> (2  $\times$  20 mL). The combined extracts were washed with brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and evaporated to dryness. Column chromatography of the residue over silica gel using pet. ether - ethyl acetate (95:5) as an eluent, furnished  $\gamma$ -methyl  $\alpha$ -(alkylidene substituted)- $\gamma$ -butyrolactones **3a, d-g** as viscous oils and **3b-c** as solids.

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