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Convenient Synthesis of Volatile *Streptomyces* Lactones

Chandan P. Amonkar,[a] Santosh G. Tilve,*[a] P. S. Parameswaran[b]

[a] Department of Chemistry, Goa University, Taleigao Plateau, Goa 403 206, India
Fax +91(832)245184; E-mail: santoshtilve@yahoo.com

[b] Chemical Oceanography Division, National Institute of Oceanography (CSIR), Dona-Paula-Goa 403 004, India

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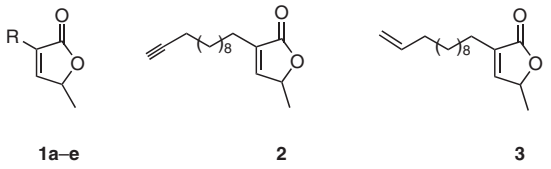
Dedicated to Prof. Ian Blair on the occasion of his 60th birthday

Abstract: A convenient three-step synthetic approach towards 3-alkyl-5-methyl-2[5*H*]furanones is described. The steps involved in the synthesis are domino primary alcohol oxidation–Wittig reaction, acid-catalysed lactonisation and isomerisation. This synthetic approach has been exploited to synthesise four *Streptomyces* lactones.

Key words: domino reaction, Wittig reaction, isomerisation, lactone, oxidation

Butenolide (3-alkyl-5-methyl-2[5*H*]furanone), a five-membered unsaturated lactone, is widely encountered in many natural products.[1] Butenolides are of interest[2] due to their broad range of biological activities e.g. butenolide **1a** is a component of mushroom flavour,[3] **1b** has fungicidal activity,[4] **1c**–**g** are metabolites from *Streptomyces griseus*,[1b,5] while **2** and **3** isolated from leaves of *Hortonia* exhibited mosquito larvicidal activities (Figure 1).[6] 3-Substituted-5-methyl-2[5*H*]furanone is believed to be one of the essential subunits responsible for the cytotoxicity of acetogenins.[2g]



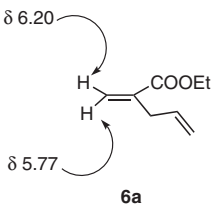
1a–e **2** **3**

1a R = CH_3
b R = CH_2CH_3
c R = $CH_2CH_2CH_2CH_3$
d R = $CH_2CH_2CH_2CH_2CH_2CH_3$
e R = $CH_2CH_2CH(CH_3)_2$
f R = $CH_2CH_2CH_2CH(CH_3)_2$
g R = $CH_2CH_2CH_2CH_2CH(CH_3)_2$

Figure 1

Domino reactions have attracted considerable attention[7] as they result in the reduction in the amount of by-products, solvents, eluents, time and energy used. In continuation of our research work dealing with the use of domino primary alcohol oxidation–Wittig reactions,[8] we were interested in testing functionalised Wittig reagent **5** for its own and its products' stability towards the domino reaction conditions (Scheme 1). Thus, butanol was subjected to the domino primary alcohol oxidation–Wittig reaction, after three hours the alcohol was found to be consumed (TLC). On usual work-up, only one product was obtained. As the product could be *E* or *Z*, it was necessary to know the geometry of the product. As the product was a trisubstituted olefin it was necessary to compare the ^{1}H NMR chemical shift values with *syn* and *anti* protons attached at the β-carbon of the unsaturated system. To do this, stable phosphorane **5** was condensed with formaldehyde to give **6a** (Figure 2). The protons at the β-carbon appeared at 5.77 and 6.20 ppm. The signal at 5.77 ppm was due to the *anti* proton and the signal at 6.20 ppm was due to the *syn* proton with respect to carboethoxy group and thus **6c** was confirmed to be the *E*-isomer. Prolonging the reaction time (> 4 h) resulted in a decrease in yield showing just how sensitive the product was to reaction conditions. The *E*-ester **6c** was then subjected to acid-catalysed lactonisation followed by isomerisation using $RhCl_3 \cdot 3H_2O$ as depicted (Scheme 1), to yield racemic 3-butyl-5-methyl-2[5*H*]furanone (**1c**), a naturally occurring butenolide. Optically active compound **1c** is a precursor[9] to (+)-blastmycinone and (–)-3-*epi*-blastmycinone. Similarly, hexanol and 3-methyl butanol were subjected to the above protocol to obtain (±)-butenolides **1d**,**e**. For volatile aldehydes direct condensation of phosphorane **5** with 37% formalin and 20% aqueous acetaldehyde was necessary to obtain **6a**,**b**. In our hands, pure butenolide **1a** was not obtained by the isomerisation of **7a**. Incidentally all the (±)-γ-methyl-α-alkylidene-γ-lactones (**7b**–**e**) have exclusive *E*-geometry.[10]

In conclusion, we have demonstrated that functionalised Wittig reagent **5** can also be used for the domino primary alcohol oxidation–Wittig reaction. Using this protocol, four volatile *Streptomyces* lactones were synthesized as racemates in just three steps in a convenient manner. Synthesis of two of them (**1d** and **1e**) is reported for the first time.



6a

Figure 2

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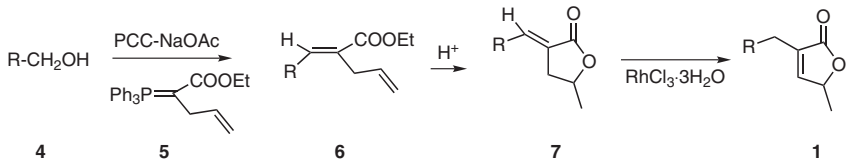

Scheme 1

Table 1 Yields for the Three-Step Synthesis

Compound	R	Yield (%)		
		6	**7**	**1**
a	H	50[a]	87	–
b	CH_3	50[a]	85	92
c[b]	$CH_2CH_2CH_3$	57	92	77
d	$CH_2(CH_2)_3CH_3$	60	89	93
e	$CH_2CH(CH_3)_2$	65	90	94

[a] Yield based on phosphorane consumed.
[b] Yield: 49%, obtained using IBX.[11]

Column chromatography was performed on silica gel (60–120 mesh) and TLC on silica gel (13% $CaSO_4$ as binder). IR spectra were recorded on Shimadzu FT-IR spectrophotometer (KBr pellet or neat sample). ^{1}H NMR and ^{13}C NMR were recorded on a Bruker-300 MHz instrument. The multiplicities of carbon signals were obtained from DEPT experiments. Low resolution mass spectra were recorded on triple quadrapole MS/MS instrument (Applied Biosystem Inc.) and high resolution mass spectra (HRMS) were recorded on a MicroMass ES-QTOF Mass spectrometer.

Phosphorane 5
A mixture of carboethoxymethylenetriphenylphosphorane (10 g, 2.87 mmol) and allyl bromide (4.16 g, 3.44 mmol) was refluxed in $CHCl_3$ (25 mL) for 5 h. The solvent was evaporated under vacuum. H_2O (100 mL) was added and the reaction mixture was washed with benzene (2 × 20 mL). Benzene (50 mL) and phenolphthalein (2 drops) were added to the aqueous layer and 2 N NaOH was added dropwise with vigorous shaking till a pink colour persisted. The benzene layer was separated, washed with H_2O (20 mL), brine (10 mL), dried over Na_2SO_4 and concentrated to get a thick syrupy liquid which on scratching after addition of anhyd hexane (15 mL) resulted in a solid product. The solid, on recrystallisation (benzene–hexane), yielded phosphorane **5** (7.8 g, 70%); mp 122 °C.[12]

Esters 6a,b; General Procedure
To a solution of phosphorane **5** (1 mmol) in MeOH (10 mL) was added an aq solution of aldehyde (2 mL, 5 mmol) and the reaction mixture was refluxed for 2 h. After the completion of reaction, hexanes (20 mL) was added and the reaction mixture was shaken vigorously. The upper layer was separated, evaporated and the resulting crude product was purified by silica gel column chromatography (hexanes) to afford a pleasant-smelling volatile liquid.

Ethyl-2-methylidenepent-4-enoate (6a)
Yield: 50%.

IR (neat):1727 (C=O), 1640 (C=C) cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.31 (t, *J* = 7.2 Hz, 3 H, OCH_2CH_3), 3.07 (br d, *J* = 6.6 Hz, 2 H, $CH_2CH{=}CH_2$), 4.22 (q, *J* = 7.1 Hz, 2 H, OCH_2CH_3), 5.11 (m, 2 H, $CH_2CH{=}CH_2$), 5.57 (br s, 1 H, HC*H*=C), 5.84 (m, 1 H, $CH_2CH{=}CH_2$), 6.20 (br s, 1 H, *H*CH=C).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.10 (CH_3), 35.82 (CH), 60.60 (OCH_2), 116.65 ($CH{=}CH_2$), 125.08 (=CH_2), 135.10 (=CH), 139.18 (C), 166.86 (C=O).

MS: *m*/*z* (%) = 141 (28, M^+ + 1), 113 (100), 112 (12), 95 (48), 67(36).

(*E*)-Ethyl-2-ethylidenepent-4-enoate (6b)
Yield: 50%.

IR (neat):1722 (C=O), 1660 (C=C), 1643 (C=C) cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.28 (t, *J* = 7.2 Hz, 3 H, OCH_2CH_3), 1.79 (d, *J* = 6.9 Hz, 3 H, CH_3), 3.08 (d, *J* = 6.0 Hz, 2 H, $CH_2CH{=}CH_2$), 4.19 (q, *J* = 7.2 Hz, 2 H, OCH_2CH_3), 5.00 (m, 2 H, $CH_2CH{=}CH_2$), 5.81 (m, 1 H, $CH_2CH{=}CH_2$), 6.95 (q, *J* = 6.9 Hz, 1 H, =C*H*CH_3).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.12 (CH_3), 14.21 (CH_3), 30.45 (CH_2), 60.36 (OCH_2), 114.91 ($CH{=}CH_2$), 130.96 (C), 135.19 (*C*H=CH_2), 138.28 (=CH), 167.45 (C=O).

MS: *m*/*z* (%) = 155 (22, M^+ + 1), 127 (100), 109 (54), 99 (48), 81 (39), 79 (11).

Esters 6c–e; General Procedure
To a magnetically stirred suspension of PCC (1.5 mmol) and NaOAc (1.5 mmol) in anhyd CH_2Cl_2 (10 mL), alcohol **4** (1 mmol) in anhyd CH_2Cl_2 (5 mL) was added followed by phosphorane **5** (1 mmol) in one portion. After 3 h, Et_2O (5 mL) was added and the supernatant solution was decanted from the black granular solid. The combined organic layers were filtered through a short pad of celite. The residue obtained after evaporation of the solvent was further purified by column chromatography using hexanes as the eluent to afford a pleasant-smelling liquid.

(*E*)-Ethyl-2-(prop-2-enyl)hex-2-enoate (6c)
Yield: 57%.

IR (neat): 1716 (C=O), 1654 (C=C), 1643 (C=C) cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.95 (t, *J* = 7.5 Hz, 3 H, CH_2CH_3), 1.29 (t, *J* = 7.2 Hz, 3 H, OCH_2CH_3), 1.49 (q, *J* = 7.5 Hz, 2 H, CH_2CH_3), 2.17 (q, *J* = 7.5 Hz, 2 H, $CH_2CH_2CH_3$), 3.07 (d, *J* = 6.0 Hz, 2 H, $CH_2CH{=}CH_2$), 4.19 (q, *J* = 7.2 Hz, 2 H, OCH_2CH_3), 4.99 (m, 2 H, $CH_2CH{=}CH_2$), 5.80 (m, 1 H, $CH_2CH{=}CH_2$), 6.84 (t, *J* = 7.5 Hz, 1 H, =CH).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 13.80 (CH_3), 14.15 (CH_3), 21.89 (CH_2), 30.46 (CH_2), 30.75 (CH_2), 60.30 (CH_2), 114.00 ($CH{=}CH_2$), 130.02 (C), 135.55 (*C*H=CH_2), 143.45 (CH), 167.52 (C=O).

MS: *m*/*z* (%) = 183 (6, M^+ + 1), 155 (3), 137 (73), 109 (100), 67 (43).

(*E*)-Ethyl-2-(prop-2-enyl)oct-2-enoate (6d)
Yield: 60%.

IR (neat): 1722 (C=O), 1653 (C=C), 1643 (C=C) cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.82 (t, *J* = 7.5 Hz, 3 H, CH_2CH_3), 1.11–1.35 (m, 9 H, OCH_2CH_3 and 3 × CH_2), 2.11 (m, 2 H, =CCH_2), 3.00 (d, *J* = 6 Hz, 2 H, $CH_2CH{=}CH_2$), 4.12 (q, *J* = 7.2 Hz, 2 H,

OCH_2CH_3), 4.91 (m, 2 H, $CH_2CH=CH_2$), 5.74 (m, 1 H, $CH_2CH=CH_2$), 6.77 (t, *J* = 7.5 Hz, 1 H, =CH).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 13.97 (CH_3), 14.28 (CH_3), 22.49 (CH_2), 28.40 (CH_2), 28.54 (CH_2), 30.84 (CH_2), 31.62 (CH_2), 60.44 (OCH_2), 114.96 ($CH=CH_2$), 129.86 (C), 135.66 ($CH=CH_2$), 143.92 (=CH), 167.69 (C=O).

HRMS: *m*/*z* calcd for $C_{13}H_{23}O_2$ (M + H^+): 211.1698; found: 211.1696.

(*E*)-Ethyl-5-methyl-2-(prop-2-enyl)hex-2-enoate (6e)
Yield: 65%.

IR (neat): 1720 (C=O), 1650 (C=C) cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.90 [d, *J* = 6.6 Hz, 6 H, $CH(CH_3)_2$], 1.22 (t, *J* = 7.2 Hz, 3 H, OCH_2CH_3), 1.69 [m, 1 H, $CH(CH_3)_2$], 2.01 (m, 2 H, CH_2), 2.99 (d, *J* = 6.0 Hz, 2 H, $CH_2CH=CH_2$), 4.11 (q, *J* = 7.2 Hz, 2 H, OCH_2CH_3), 4.91 (m, 2 H, $CH_2CH=CH_2$), 5.73 (m, 1 H, $CH_2CH=CH_2$), 6.79 (t, *J* = 7.2 Hz, 1 H, CH_2CH=C).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.23 (CH_3), 22.32 (CH_3), 22.66 (CH_3), 29.08 (CH), 31.58 (CH_2), 37.58 (CH_2), 60.38 (OCH_2), 114.98 ($CH=CH_2$), 130.48 (C), 135.56 ($CH=CH_2$), 148.13 (=CH), 167.58 (C=O).

HRMS: *m*/*z* calcd for $C_{12}H_{20}O_2$ + Na (M + Na^+): 219.1361; found: 219.1361.

7a–e; General Procedure
To a flask containing ice-cold ester **6** (1 mmol) was added ice-cold concd H_2SO_4 (2 mL) and the reaction mixture was stirred in an ice bath for 1 h. After the completion of reaction sufficient crushed ice (10 mL) was added to the reaction mixture and the reaction mixture was extracted with Et_2O (3 × 5 mL). The combined organic extracts were dried over anhyd Na_2SO_4 and the crude product was purified by silica gel column chromatography (EtOAc–hexanes, 1:9) to yield liquid lactone.

5-Methyl-3-(methylidene)dihydrofuran-2(5*H*)-one (7a)
Yield: 87%.

IR (neat): 1765 (C=O), 1665 (C=C) cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.43 (d, *J* = 6.3 Hz, 3 H, $CHCH_3$), 2.56 (ddt, *J* = 16.8, 5.7, 2.7 Hz, 1 H, HC*H*CH), 3.10 (ddt, *J* = 16.8, 7.2, 2.7 Hz, 1 H, *H*CHCH), 4.66 (m, 1 H, $CHCH_3$), 5.63 (t, *J* = 2.7 Hz, 1 H, HC*H*=C), 6.23 (t, *J* = 2.7 Hz, 1 H, *H*CH=C).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 21.88 (CH_3), 35.10 (CH_2), 73.82 (CH), 121.91(CH_2), 134.80 (C), 176.20 (C=O).

MS: *m*/*z* (%) = 113 (8, M^+ + 1), 95 (17), 67 (100), 65 (20), 43 (13).

5-Methyl-3-[(*E*)-ethylidene]dihydrofuran-2(5*H*)-one (7b)
Yield: 85%.

IR (neat): 1756 (C=O), 1680 (C=C) cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.35 (d, *J* = 6.3 Hz, 3 H, $CHCH_3$), 1.84 (dt, *J* = 6.9, 2.1, 1.8 Hz, 3 H, $=CHCH_3$), 2.35 (ddt, *J* = 16.8, 5.2, 1.8 Hz, 1 H, HC*H* CH), 3.01 (ddt, *J* = 16.8, 7.8, 2.1 Hz, 1 H, *H*CHCH), 4.61 (ddq, *J* = 7.8, 6.3, 5.2 Hz, 1 H, $CHCH_3$), 6.78 (m, 1 H, =CH).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 15.55 (CH_3), 22.20 (CH_3), 32.65 (CH_2), 73.85 (CH), 127.55 (C), 135.38 (CH), 170.64 (C=O)

MS: *m*/*z* (%) = 127 (7, M^+ + 1), 109 (29), 81 (100), 79 (37), 77 (6), 43 (5).

5-Methyl-3-[(*E*)-butylidene]dihydrofuran-2(5*H*)-one (7c)
Yield: 92%.

IR (neat): 1753 (C=O), 1690 (C=C) cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.95 (t, *J* = 7.5 Hz, 3 H, CH_2CH_3), 1.42 (d, *J* = 6.3 Hz, 3 H, $CHCH_3$), 1.51 (m, 2 H, CH_2CH_3), 2.16 (m, 2 H, $=CHCH_2$), 2.44 (m, 1 H, HC*H*CH), 3.01 (m, 1 H, *H*CHCH), 4.66 (m, 1 H, CH), 6.73 (m, 1 H, =CH).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 13.78 (CH_3), 21.47 (CH_2), 22.26 (CH_3), 32.16 (CH_2), 32.91 (CH_2), 73.94 (CH), 126.67 (C), 140.55 (=CH), 170.72 (C=O).

MS: *m*/*z* (%) = 155 (6, M^+ + 1), 137 (50), 109 (100), 95 (26), 81 (28), 67 (96), 65 (10), 43 (8).

5-Methyl-3-[(*E*)-hexylidene]dihydrofuran-2(5*H*)-one (7d)
Yield: 89%.

IR (neat): 1753 (C=O), 1685 (C=C) cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.90 (t, *J* = 6.6 Hz, 3 H, CH_2CH_3), 1.25–1. 50 (m, 6 H, 3 × CH_2), 1.42 (d, *J* = 6.3 Hz, 3 H, CH_3), 2.18 (q, *J* = 7.2 Hz, 2 H, $=CHCH_2$), 2.40 (m, 1 H, HC*H*CH), 3.00 (br dd, *J* = 16.8, 7.8 Hz, 1 H, *H*CHCH), 4.65 (m, 1 H, CH), 6.72 (t, *J* = 7.5 Hz, 1 H, =CH).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 13.91 (CH_3), 22.25 (CH_3), 22.40 (CH_2), 27.80 (CH_2), 30.13 (CH_2), 31.43 (CH_2), 32.88 (CH_2), 73.94 (CH), 126.45 (C), 140.87 (=CH), 170.96 (C=O).

HRMS: *m*/*z* calcd for $C_{11}H_{19}O_2$ (M + H^+): 183.1385; found: 183.1376.

5-Methyl-3-[(*E*)-3-methylbutylidene]dihydrofuran-2(5*H*)-one (7e)
Yield: 90%.

IR (neat): 1752 (C=O), 1680 (C=C) cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.94 [d, *J* = 6.6 Hz, 6 H, $CH(CH_3)_2$], 1.41 (d, *J* = 6.0 Hz, 3 H, $CHCH_3$), 1.81 (m, 1 H, $CHCH_3$), 2.06 (t, *J* = 6.6 Hz, 2 H, $=CHCH_2$), 2.40 (m, 1 H, HC*H* CH), 3.00 (br dd, *J* = 16.8, 7.8 Hz, 1 H, *H*CHCH), 4.66 (m, 1 H, CH), 6.76 (t, *J* = 7.8 Hz, 1 H, =CH).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 22.25 (CH_3), 22.39 (CH_3), 28.09 (CH_3), 33.05 (CH_2), 39.28 (CH_2), 73.93 (CH), 127.17 (C), 139.68 (=CH), 170.86 (C=O).

HRMS: *m*/*z* calcd for $C_{10}H_{17}O_2$ (M + H^+): 169.1228; found: 169.1219.

1b–e; General Procedure
To a solution of lactone **7** (1 mmol) in degassed EtOH (10 mL) was added $RhCl_3·3H_2O$ (0.01 mmol) and the reaction mixture was refluxed for 15 h. The crude product was purified by silica gel column chromatography (EtOAc–hexanes, 1:9) to yield liquid butenolide.

3-Ethyl-5-methyl-2[5*H*]furanone (1b)
Yield: 92%.

IR (neat): 1750 (C=O), 1650 (C=C) cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.19 (t, *J* = 7.2 Hz, 3 H, CH_2CH_3), 1.34 (d, *J* = 6.6 Hz, 3 H, $CHCH_3$), 2.22 (q, *J* = 7.2 Hz, 2 H, CH_2CH_3), 4.94 (m, 1 H, CH), 6.93 (br s, 1 H, =CH).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 11.43 (CH_3), 18.24 (CH_3), 18.98 (CH_2), 77.23 (CH), 134.86 (C), 148.54 (=CH), 173.57 (C=O).

MS: *m*/*z* (%) = 127 (88, M^+ + 1), 109 (100), 99 (26), 69 (34).

3-Butyl-5-methyl-2[5*H*]furanone (1c)
Yield: 77%.

IR (neat): 1750 (C=O), 1650 (C=C) cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.87 (t, *J* = 7.5 Hz, 3 H, CH_2CH_3), 1.19–1.53 (m, 4 H, 2 × CH_2), 1.34 (d, *J* = 6.0 Hz, 3 H, $CHCH_3$), 2.22

(t, J = 7.5 Hz, 2 H, =CCH_2), 4.95 (q, J = 7.5 Hz, 1 H, CH), 6.90 (m, 1 H, =CH).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 13.68 (CH_3), 19.13 (CH_3), 22.18 (CH_2), 24.78 (CH_2), 29.43 (CH_2), 75.45 (CH), 134.24 (C), 148.73 (=CH), 173.60 (C=O).

MS: *m*/*z* (%) = 155 (32, M^+ + 1), 137 (47), 109 (100), 67 (20).

3-Hexyl-5-methyl-2[5*H*]furanone (1d)
Yield: 93%.

IR (neat): 1750 (C=O), 1650 (C=C) cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.88 (d, J = 6.3 Hz, 3 H, CH_2CH_3), 1.26–1.60 (m, 8 H, 4 × CH_2), 1.47 (d, J = 6.6 Hz, 3 H, $CHCH_3$), 2.27 (t, J = 7.5 Hz, 2 H, =CCH_2), 5.00 (m, 1 H, CH), 6.99 (br d, J = 0.9 Hz, 1 H, =CH).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.01 (CH_3), 19.12 (CH_3), 22.51 (CH_2), 22.16 (CH_2), 27.35 (CH_2), 28.82 (CH_2), 31.48 (CH_2), 73.87 (CH), 134.35 (C), 148.83 (=CH), 173.87 (C=O).

HRMS: *m*/*z* calcd for $C_{11}H_{19}O_2$ ($M + H^+$): 183.1385; found: 183.1393.

3-(3-Methyl-butyl)-5-methyl-2[5*H*]furanone (1e)
Yield: 94%.

IR (neat): 1750 (C=O), 1650 (C=C) cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.93 [d, J = 6.6 Hz, 6 H, $CH(CH_3)_2$], 1.18–1.65 (m, 3 H, CH_2, CH), 1.42 (d, J = 6.3 Hz, 3 H, $CHCH_3$), 2.28 (t, J = 7.8 Hz, 2 H, =CCH_2), 4.99 (m, 1 H, CH), 6.98 (d, J = 1.2 Hz, 1 H, =CH).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 19.22 (CH_3), 22.36 (CH_3), 23.12 (CH_3), 27.74 (CH_2), 29.69 (CH), 36.41 (CH_2), 77.02 (CH), 134.59 (C), 148.65 (=CH), 170.01 (C=O).

HRMS: *m*/*z* calcd for $C_{10}H_{17}O_2$ ($M + H^+$): 169.1228; found: 169.1232.

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