

# Synthesis of 2-(2-phenylethenyl) substituted 4,5-dihydrofurans by regioselective addition of 1,3-dicarbonyl compounds to dienes promoted by cerium(IV) ammonium nitrate

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## Abstract

Radical addition of 1,3-dicarbonyl compounds to conjugated dienes in the presence of cerium(IV) ammonium nitrate in THF produced 4,5-dihydrofurans in good to excellent yields. All radical additions occurred on the terminal double bond as regioselective. Two different dihydrofurans were obtained from the reaction of 1-phenyl-1,3-butanedione with 1-phenyl-1,3-butadiene and 3-methyl-1-phenyl-1,3-butadiene. All compounds were characterised by IR,  $^1\text{H}$ ,  $^{13}\text{C}$ -NMR and HRMS spectra.

**Keywords:** Cerium(IV) ammonium nitrate, oxidative addition, dihydrofuran, conjugated diene.

## Introduction

It is well known that  $\text{Mn}(\text{OAc})_3$ <sup>1-14</sup> and  $(\text{NH}_4)_2\text{Ce}(\text{NO}_2)_6$  (CAN)<sup>15-24</sup> are widely used as radical oxidants in the synthesis of poly functional organic compounds forming C-C bond between basic compounds. These radical oxidants enable formation of dihydrofurans obtained from reaction of 1,3-dicarbonyls, 3-oxopropanenitriles,  $\beta$ -ketoesters and their derivatives with alkenes easily prepared by basic methods.

Our research group has focused the synthesis of dihydrofuran derivatives by the radical addition of various activated methyl ketones to unsaturated units such as alkenes, alkynes, dienes, and acrylamides. Recently, we have prepared 2,3-dihydro-4H-furo[3,2-c]chromen-4-ones and 2,3-dihydronaphtho[2,3-b]furan-4,9-diones by the cyclization of 4-hydroxycoumarin and 2-hydroxy-1,4-naphthoquinone, respectively.<sup>25,26</sup> Also, we carried out the reactions of fluorinated-1,3-dicarbonyl compounds with dienes<sup>27</sup> and conjugated alkenes,<sup>28-31</sup> resulting in fluoroacetylated and fluoroalkylated 4,5-dihydrofurans. Moreover, we reported reactions of 1,3-dicarbonyl compounds with alkynes<sup>32</sup> and various substituted alkenes.<sup>33-36</sup> Very recently, we have prepared 4,5-dihydrofuran-carbonitriles by the treatment of 3-oxopropanenitriles with

alkenes,<sup>37-39</sup> unsaturated amides<sup>40, 41</sup> and esters<sup>41</sup> using  $\text{Mn}(\text{OAc})_3$ . In these reactions,  $\text{Mn}(\text{OAc})_3$  was frequently used as radical oxidant in the presence of HOAc. However, recently we performed an optimization study on the radical addition of 3-oxopropanenitriles to alkenes using CAN in ethereal solvents, resulting CAN / THF system formed dihydrofurans in high yields on mild condition.<sup>42</sup> In the present study, we applied the previous method to reactions of 1,3-dicarbonyl compounds with conjugated dienes promoted CAN / THF and obtained 2-(2-phenylethenyl) substituted 4,5-dihydrofurans as regioselectively in excellent yields.

## Results and Discussion

Conjugated dienes 1-phenyl-1,3-butadiene **2a**<sup>43</sup> and 3-methyl-1-phenyl-1,3-butadiene **2b**<sup>44</sup> were synthesized from the reaction of methyl-triphenylphosphonium bromide and suitable carbonyl compounds in the presence of NaH/THF. 1,1-Diphenyl-1,3-butadiene **2c**<sup>45</sup> was prepared from water elimination of alcohol obtained from Grignard reaction of benzophenone and allylmagnesium bromide.

Radical addition of dimedone **1a** and 1,3-cyclohexanedione **1b** to 1-phenyl-1,3-butadiene **2a** gave 2-(2-phenylethenyl) dihydrofuran **3a** (85%) and **3b** (80%) in excellent yields, respectively (Table 1). Also, treatment of 5-phenyl-1,3-cyclohexanedione **1c** with **2a** produced dihydrofuran **3c** in 78 % yield as diastereomeric mixture (determined by <sup>1</sup>H NMR spectrum, dr = 50:50) of. Moreover, the reaction of 2,4-pentanedione **1d** and ethyl 3-oxobutanoate **1e** with **2a** occur **3d** (65%) and **3e** (63%) in good yields, respectively. However, two different cyclic products **3f** and **3g** were obtained from the reaction of 1-phenyl-1,3-butanedione **1f** and **2a** since **1f** have two different enol forms. These products **3f** and **3g** were differentiated by the chemical shift of the carbonyl carbons in their <sup>13</sup>C NMR spectra which show 195 ppm for **3f** and 193.4 ppm for **3g**. Also, The <sup>1</sup>H NMR spectrum of compound **3g** show that protons H4 resonate with methyl group on the C-2 carbon (<sup>5</sup>J 1.6 Hz) as long range coupling, but same coupling is not observed in the spectrum of **3f**.

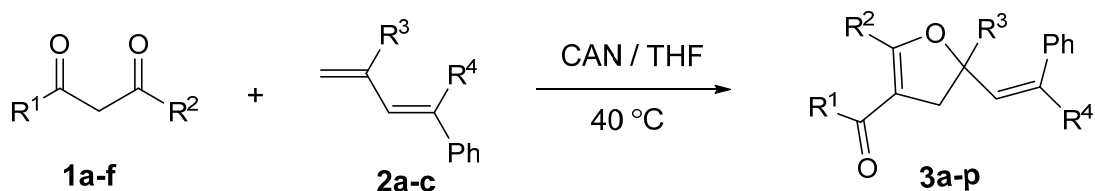
Treatments of **1a** and **1d** with 1,1-diphenyl-1,3-butadiene **2c** gave dihydrofuran **3h** (91%) (obtained in 78% yield by  $\text{Mn}(\text{OAc})_3$ )<sup>14</sup> and **3i** (84%) in excellent yields, respectively (Entries, 7 and 8). Upon comparing the addition reactions of both compounds **2a** and **2c**, it is observed that diene **2c** produced dihydrofuran in higher yield. This occurrence can be explained with the stability of intermediate radical formed in 1,3-dicarbonyl. Since **2c** has two phenyl groups, radical group of it is more stable compared to that of **2a**'s.

It was reported in the literature that radical reaction of dimedone **1a** with 1-phenyl-3-methyl-1,3-butadiene **2b** produced **3j** by using  $\text{PbI}(\text{OAc})_2$  (69%)<sup>46</sup> and CAN / MeOH (40%)<sup>47</sup> as radical oxidants. But in this work, dihydrofuran **3j** was obtained in 92 % yield using CAN/THF system. Also, 2-(2-phenylethenyl) substituted dihydrofuran **3k** (88%) and diastereomeric mixture (dr = 50:50) of **3l** (82%) were obtained in excellent yields (entries 4 and 5). Similarly, while it was reported that synthesis of compounds **3m** and **3n** through CAN/MeOH in 45% and 40% yields,

respectively,<sup>47</sup> we obtained these compounds in very good yields (75% and 71%, respectively) by using THF as solvent.

The reaction of **1f** with **2b** gave two different cyclic products **3o** and **3p** in moderate yields. These compounds were differentiated by the chemical shift of carbonyl groups in their <sup>13</sup>C NMR spectra as mentioned above.

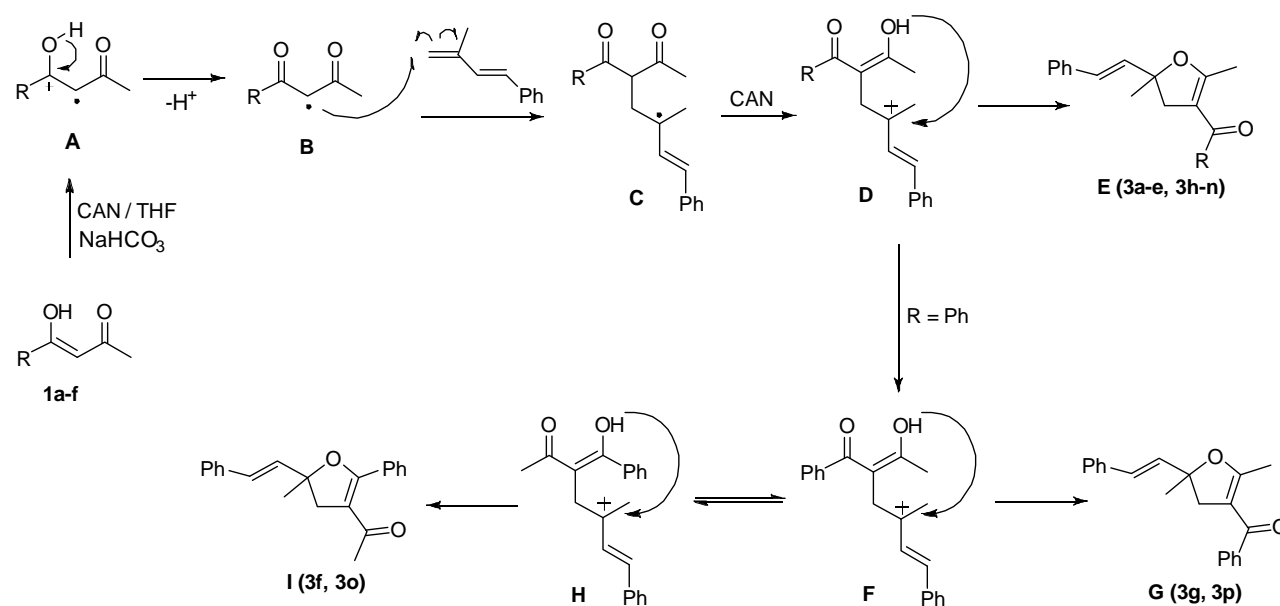
**Table 1.** Radical addition of 1,3-dicarbonyl compounds (**1a-f**) to conjugated dienes (**2a-c**)



Entry	R <sup>1</sup>	R <sup>2</sup>	R <sup>3</sup>	R <sup>4</sup>	Products and yields <sup>a</sup> (%)	
1	-CH <sub>2</sub> C(CH <sub>3</sub> ) <sub>2</sub> CH <sub>2</sub> -	<b>1a</b>	H	H	<b>2a</b> <b>3a</b> (85)	
2	-CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> -	<b>1b</b>	H	H	<b>2a</b> <b>3b</b> (80)	
3	-CH <sub>2</sub> CHPhCH <sub>2</sub> -	<b>1c</b>	H	H	<b>2a</b> <b>3c</b> (78)	
4	-CH <sub>3</sub>	-CH <sub>3</sub>	<b>1d</b>	H	H	<b>2a</b> <b>3d</b> (65)
5	-OCH <sub>2</sub> CH <sub>3</sub>	-CH <sub>3</sub>	<b>1e</b>	H	H	<b>2a</b> <b>3e</b> (63)
6	-CH <sub>3</sub>	-Ph	<b>1f</b>	H	H	<b>2a</b> <b>3f</b> (27) <sup>b</sup> <b>3g</b> (39) <sup>c</sup>
7	-CH <sub>2</sub> C(CH <sub>3</sub> ) <sub>2</sub> CH <sub>2</sub> -	<b>1a</b>	H	Ph	<b>2c</b> <b>3h</b> (91)	
8	CH <sub>3</sub>	CH <sub>3</sub>	<b>1d</b>	H	Ph	<b>2c</b> <b>3i</b> (84)
9	-CH <sub>2</sub> C(CH <sub>3</sub> ) <sub>2</sub> CH <sub>2</sub> -	<b>1a</b>	CH <sub>3</sub>	H	<b>2b</b> <b>3j</b> (92)	
10	-CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> -	<b>1b</b>	CH <sub>3</sub>	H	<b>2b</b> <b>3k</b> (88)	
11	-CH <sub>2</sub> CHPhCH <sub>2</sub> -	<b>1c</b>	CH <sub>3</sub>	H	<b>2b</b> <b>3l</b> (82)	
12	-CH <sub>3</sub>	-CH <sub>3</sub>	<b>1d</b>	CH <sub>3</sub>	H	<b>2b</b> <b>3m</b> (75)
13	-OCH <sub>2</sub> CH <sub>3</sub>	-CH <sub>3</sub>	<b>1e</b>	CH <sub>3</sub>	H	<b>2b</b> <b>3n</b> (71)
14	-CH <sub>3</sub>	-Ph	<b>1f</b>	CH <sub>3</sub>	H	<b>2b</b> <b>3o</b> (40) <sup>b</sup> <b>3p</b> (38) <sup>c</sup>

<sup>a</sup>Yields of isolated products based on the dienes. <sup>b</sup> For the compounds **3f** and **3o**, R<sup>1</sup> = CH<sub>3</sub>, R<sup>2</sup> = Ph; <sup>c</sup> For the compounds **3g** and **3p**, R<sup>1</sup> = Ph, R<sup>2</sup> = CH<sub>3</sub>.

The mechanism proposed for the radical addition of conjugated dienes **2a-c** with 1,3-dicarbonyls **1a-f** is presented in Scheme 1. According to this mechanism, while  $Ce^{+4}$  is reduced to  $Ce^{+3}$  a radical cation **B** is formed.<sup>48</sup> Then, addition of the radical to the terminal double bond of the diene forms an allylic radical intermediate **C**. Radical **C** is oxidized to the carbocation **D** by CAN, followed by cyclization of **D** to give 5-(2-phenylvinyl)-4,5-dihydrofuran **E**. Intermediates **F** and **H** which is the another enol form of **F** were obtained from the radical addition of 1-phenyl-1,3-butanedione **1f** to diene **2b**. Intramolecular cyclization of these intermediates gave dihydrofurans **G** and **I**, respectively. All radical additions to the dienes occurred on the terminal double bond of the dienes as regioselectively, other adduct products were not observed.



**Scheme 1.** Mechanism proposed for the formation of dihydrofurans.

## Conclusions

Cerium(IV) ammonium nitrate promoted radical addition of 1,3-dicarbonyl compounds to 1,3-butadiene derivatives was investigated, resulting in formation of various 2-phenylvinyl-4,5-dihydrofurans. Previous methodology that was optimized using alkenes was applied here to radical addition of 1,3-dicarbonyls to dienes. Upon doing a literature review and comparing the results, it is deduced that, in the radical addition of dienes to 1,3-dicarbonyls promoted CAN/THF system can be used in a highly effective way.

## Experimental Section

**General.** Melting points were determined on an electrothermal capillary melting point apparatus. IR spectra (ATR) were obtained with a Bruker Tensor-27 400-4000  $\text{cm}^{-1}$  range with 2  $\text{cm}^{-1}$  resolution.  $^1\text{H}$  NMR,  $^{13}\text{C}$  NMR, spectra were recorded on a Bruker Avance DPX-400 MHz and Varian Oxford NMR300 High performance Digital FT-NMR spectrophotometers. High Resolution Mass Time-of-Flight (TOF) was measured on an Agilent 1200/6210 LC/MS spectrophotometer. The mass spectra were measured on a Waters-2695-Alliance-Micromass-ZQ instrument in  $m/z$  (rel.%). Elemental analyses were performed on a VarioEL III CHNS instrument.  $[\text{Mn}(\text{OAc})_3] \cdot 2\text{H}_2\text{O}$  was prepared by electrochemically method according to the literature <sup>4</sup>. Thin layer chromatography (TLC) was performed on Merck aluminum-packed silica gel plates. Purification of the products was performed by column chromatography on silica gel (Merck silica gel 60, 40-63 mm).

**General procedure for the synthesis of 4,5-dihydrofurans (3a-p).** To a soln. of 1,3-dicarbonyls (1 mmol) and diene (1.5 mmol) in THF (10 mL) under  $\text{N}_2$  in an oil bath, a mixture of CAN (2.5 mmol) and  $\text{NaHCO}_3$  (2.5 mmol) was added at 30°C. Then, the temp. was slowly increased to 40°. The reaction was completed when the orange colour of CAN had disappeared (10 min) or when the diene spot on TLC had completely vanished.  $\text{H}_2\text{O}$  was added to the soln., and the mixture was extracted with  $\text{CHCl}_3$  (3x20 mL). The combined organic phase was dried ( $\text{Na}_2\text{SO}_4$ ) and concentrated and the crude product purified by column chromatography on silica gel (230 – 400 mesh) or preparative TLC (20x20 cm plates, 2 mm thickness, n-hexane/EtOAc (5 :1)).

**6,6-Dimethyl-2-[(E)-2-phenylvinyl]-3,5,6,7-tetrahydro-1-benzofuran-4(2H)-one (3a).** Light yellow oil, yield 85%, 228 mg, IR (ATR,  $\text{cm}^{-1}$ ): 3058, 2961, 1695 (C=O), 1625 (C=C), 1020, 756, 696.  $^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ ),  $\delta_{\text{H}}$  1.09 (6H, s, 2x $\text{CH}_3$ ), 2.22 (2H, s, H5), 2.30 (2H, s, H7), 2.70 (1H, dd,  $J$  14.4 and 7.6 Hz, H3), 3.07 (1H, dd,  $J$  14.4 and 10.4 Hz, H3), 5.37 (1H, dt,  $J$  10.4 and 7.6 Hz, H2), 6.22 (1H, dd,  $J$  16.0 and 7.6 Hz,  $\text{H}_{\text{olef}}$ ), 6.62 (1H, d,  $J$  16.0 Hz,  $\text{H}_{\text{olef}}$ ), 7.25 (1H, t,  $J$  7.2 Hz), 7.30 (2H, t,  $J$  7.2 Hz), 7.36 (2H, d,  $J$  7.6 Hz).  $^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ ),  $\delta_{\text{C}}$  28.9 ( $\text{CH}_3$ ), 28.95 ( $\text{CH}_3$ ), 32.3, 34.3, 38.0, 51.2 (C3), 86.4 (C2), 111.7 (C3a), 127.0, 127.4 (2xCH), 128.5 (2xCH), 128.9, 133.2, 136.0, 176.2 (C7a), 194.5 (C=O).  $m/z$  ( $\text{ESI}^+$ ) = 269 ( $\text{MH}^+$ , 100%). HRMS ( $\text{ESI}^+$ ):  $m/z$  ( $\text{M}+\text{H}^+$ )  $\text{C}_{18}\text{H}_{20}\text{O}_2$ : 269.15361 found: 269.15461. Anal. Calcd for  $\text{C}_{18}\text{H}_{20}\text{O}_2$  (268.35): C, 80.56; H, 7.51%. Found: C, 80.42; H, 7.73%.

**2-[(E)-2-Phenylvinyl]-3,5,6,7-tetrahydro-1-benzofuran-4(2H)-one (3b).** Yellow solid, yield 80%, 192 mg, mp 67-69 °C, IR (ATR,  $\text{cm}^{-1}$ ): 2948, 2866, 1623 (C=O), 1600 (C=C), 1227, 968, 756, 691.  $^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ ),  $\delta_{\text{H}}$  2.05 (2H, f,  $J$  6.4 Hz, H6), 2.36 (2H, t,  $J$  3.2 Hz, H5), 2.46 (2H, tt,  $J$  6.4 and  $^5J_{\text{H7-H3}} = 1.6$  Hz, H7), 2.7 (1H, ddd,  $J$  14.4, 8.0 and  $^5J_{\text{H3-H7}} = 1.6$  Hz, H3), 3.1 (1H, ddt,  $J$  14.4, 10.4 and  $^5J_{\text{H3-H7}} = 1.6$  Hz, H3), 5.30 (1H, ddd,  $J$  10.4 7.6 and 7.2 Hz, H2), 6.20 (1H, dd,  $J$  16.0 and 7.2 Hz,  $\text{H}_{\text{olef}}$ ), 6.60 (1H, d,  $J$  16.0 Hz,  $\text{H}_{\text{olef}}$ ), 7.20 (1H, tt,  $J$  7.6 and 2.4 Hz), 7.30 (2H, t,  $J$  7.6 Hz), 7.40 (2H, d,  $J$  8.4 Hz).  $^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ ),  $\delta_{\text{C}}$  21.9 ( $\text{CH}_2$ ),

24.2 (CH<sub>2</sub>), 32.5, 36.7 (C3), 86.2 (C2), 113.3 (C3a), 127.0, 127.4 (2xCH), 128.5 (2xCH), 128.9, 133.3, 136.0, 177.3 (C7a), 195.6 (C=O). *m/z* (ESI<sup>+</sup>) = 241 (MH<sup>+</sup>, 100%). HRMS (ESI<sup>+</sup>): *m/z* (M+H)<sup>+</sup> C<sub>16</sub>H<sub>16</sub>O<sub>2</sub>: 241.12231 found: 241.12334. Anal. Calcd for C<sub>16</sub>H<sub>16</sub>O<sub>2</sub> (240.29): C, 79.97; H, 6.71%. Found: C, 80.35; H, 6.52%.

**6-Phenyl-2-[(E)-2-phenylvinyl]-3,5,6,7-tetrahydro-1-benzofuran-4(2H)-one (3c).** Pale yellow solid, yield 78%, 246 mg, mp 64-66 °C, IR (ATR, cm<sup>-1</sup>): 3029, 2954, 2937, 1623 (C=O), 1600 (C=C), 1203, 748, 688. <sup>1</sup>H NMR (300MHz, CDCl<sub>3</sub>), δ<sub>H</sub> 2.56-2.70 (5H, m, H5, H6, H7), 3.07 (1H, m, H3), 3.38 (1H, m, H3), 5.35 (1H, m, H2), 6.20 (1H, dd, *J* 15.9 and 7.5 Hz, H<sub>olef.</sub>), 6.6 (1H, d, *J* 15.9 Hz, H<sub>olef.</sub>), 7.16-7.34 (10H, m). <sup>13</sup>C NMR (75MHz, CDCl<sub>3</sub>), δ<sub>C</sub> 31.8 (CH<sub>2</sub>), 32.2 (CH<sub>2</sub>), 40.5, 44.0 (C3), 86.6 (C2), 113.1 (C3a), 127.0, 127.05, 127.1 (2xCH), 127.4 (2xCH), 128.6 (2xCH), 128.9, 129.0, 133.3, 135.9, 142.8, 176.6 (C7a), 194.2 (C=O). *m/z* (ESI<sup>+</sup>) = 317 (MH<sup>+</sup>, 100%). HRMS (ESI<sup>+</sup>): *m/z* (M+H)<sup>+</sup> C<sub>22</sub>H<sub>20</sub>O<sub>2</sub>: 317.15361 found: 317.15315. Anal. Calcd for C<sub>22</sub>H<sub>20</sub>O<sub>2</sub> (316.39): C, 83.51; H, 6.37%. Found: C, 83.85; H, 6.30%.

**1-{2-Methyl-5-[(E)-2-phenylvinyl]-4,5-dihydrofuran-3-yl}ethanone (3d).** Yellow oil, yield 65%, 148 mg, IR (ATR, cm<sup>-1</sup>): 3027, 2925, 1698 (C=O), 1592 (C=C), 1221, 929, 749, 693. <sup>1</sup>H NMR (300MHz, CDCl<sub>3</sub>), δ<sub>H</sub> 2.21 (3H, s, CH<sub>3</sub>), 2.25 (3H, t, *J* 1.5 Hz, CH<sub>3</sub>), 2.81 (1H, ddq, *J* 14.1, 8.1 and 1.5 Hz, H4), 3.17 (1H, ddq, *J* 14.1, 10.5 and 1.5 Hz, H4), 5.20 (1H, ddd, *J* 10.2, 8.1 and 7.2 Hz, H5), 6.23 (1H, dd, *J* 15.9 and 7.5 Hz, H<sub>olef.</sub>), 6.60 (1H, d, *J* 15.9 Hz, H<sub>olef.</sub>), 7.25 (1H, t, *J* 8.4 Hz), 7.31 (2H, t, *J* 6.9 Hz), 7.4 (2H, d, *J* 8.4 Hz). <sup>13</sup>C NMR (75MHz, CDCl<sub>3</sub>), δ<sub>C</sub> 15.3 (CH<sub>3</sub>), 29.7 (CH<sub>3</sub>), 36.9 (C4), 83.0 (C5), 112.3 (C3), 126.9, 127.8 (2xCH), 128.5 (2xCH), 128.9, 132.8, 136.1, 167.8 (C2), 194.8 (C=O). *m/z* (ESI<sup>+</sup>) = 229 (MH<sup>+</sup>, 100%). HRMS (ESI<sup>+</sup>): *m/z* (M+H)<sup>+</sup> C<sub>15</sub>H<sub>16</sub>O<sub>2</sub>: 229.12231 found: 229.12284. Anal. Calcd for C<sub>15</sub>H<sub>16</sub>O<sub>2</sub> (228.28): C, 78.92; H, 7.06%. Found: C, 79.05; H, 7.18%.

**Ethyl 2-methyl-5-[(E)-2-phenylvinyl]-4,5-dihydrofuran-3-carboxylate(3e).** Pale yellow oil, yield 63%, 163 mg, IR (ATR, cm<sup>-1</sup>): 2962, 1692 (C=O), 1644 (C=C), 1078, 786, 692. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>), δ<sub>H</sub> 1.27 (3H, t, *J* 9.2 Hz, -OCH<sub>2</sub>CH<sub>3</sub>), 2.20 (3H, t, *J* 1.5 Hz, CH<sub>3</sub>), 2.75 (1H, ddq, *J* 14.1, 8.1 and 1.5 Hz, H4), 3.12 (1H, ddq, *J* 14.4, 10.2 and 1.5 Hz, H4), 4.2 (2H, q, *J* 7.2 Hz, -OCH<sub>2</sub>CH<sub>3</sub>), 5.20 (1H, ddd, *J* 10.5, 7.8 and 6.9 Hz, H5), 6.25 (1H, dd, *J* 15.9 and 7.2 Hz, H<sub>olef.</sub>), 6.60 (1H, d, *J* 15.9 Hz, H<sub>olef.</sub>), 7.25 (1H, t, *J* 6.6 Hz), 7.31 (2H, t, *J* 6.6 Hz), 7.4 (2H, d, *J* 6.9 Hz). <sup>13</sup>C NMR (75MHz, CDCl<sub>3</sub>), δ<sub>C</sub> 14.4 (CH<sub>3</sub>), 14.7 (CH<sub>3</sub>), 36.2 (C4), 59.7, 82.8 (C5), 102.0 (C3), 126.9, 128.1 (2xCH), 128.3, 128.8 (2xCH), 132.5, 136.3, 166.3 (C2), 167.8 (C=O). *m/z* (ESI<sup>+</sup>) = 259 (MH<sup>+</sup>, 100%). HRMS (ESI<sup>+</sup>): *m/z* (M+H)<sup>+</sup> C<sub>16</sub>H<sub>18</sub>O<sub>3</sub>: 259.13287 found: 259.13231. Anal. Calcd for C<sub>16</sub>H<sub>18</sub>O<sub>3</sub> (258.31): C, 74.39; H, 7.02%. Found: C, 74.55; H, 6.75%.

**1-{2-Phenyl-5-[(E)-2-phenylvinyl]-4,5-dihydrofuran-3-yl}ethanone (3f).** Yellow oil, yield 27%, 78 mg, IR (ATR, cm<sup>-1</sup>): 2925, 1717 (C=O), 1677 (C=C), 1595, 750, 693. <sup>1</sup>H NMR (300MHz, CDCl<sub>3</sub>), δ<sub>H</sub> 1.94 (3H, s, CH<sub>3</sub>), 3.0 (1H, dd, *J* 15.0 and 8.4 Hz, H4), 3.32 (1H, dd, *J* 15.0 and 10.2 Hz, H4), 5.30 (1H, ddd, *J* 10.0, 8.4 and 7.2 Hz, H5), 6.30 (1H, dd, *J* 15.9 and 7.2 Hz, H<sub>olef.</sub>), 6.65 (1H, d, *J* 15.9 Hz, H<sub>olef.</sub>), 7.22-7.30 (3H, m), 7.36-7.42 (5H, m), 7.30 (2H, dd, *J* 7.8 and 1.5 Hz). <sup>13</sup>C NMR (75MHz, CDCl<sub>3</sub>), δ<sub>C</sub> 29.1(CH<sub>3</sub>), 37.6 (C4), 83.2 (C5), 114.9 (C3), 127.0, 127.7, 128.0 (2xCH), 128.4 (2xCH), 128.6 (2xCH), 128.9 (2xCH), 129.5, 130.9, 133.1,

136.2, 168.7 (C2), 195.0 (C=O).  $m/z$  (ESI<sup>+</sup>) = 291 (MH<sup>+</sup>, 100%). HRMS (ESI<sup>+</sup>):  $m/z$  (M+H)<sup>+</sup> C<sub>20</sub>H<sub>19</sub>O<sub>2</sub>: 291.13796 found: 291.14053. Anal. Calcd for C<sub>20</sub>H<sub>18</sub>O<sub>2</sub> (290.35): C, 82.73; H, 6.25%. Found: C, 82.60; H, 6.47%.

**{2-Methyl-5-[(E)-2-phenylvinyl]-4,5-dihydrofuran-3-yl}(phenyl)methanone (3g).** Yellow oil, yield 39%, 113 mg, IR (ATR, cm<sup>-1</sup>): 3059, 1717 (C=O), 1700 (C=C), 1600 (C=C), 1597, 1219, 749, 692. <sup>1</sup>H NMR (300MHz, CDCl<sub>3</sub>), δ<sub>H</sub> 1.94 (3H, t, <sup>5</sup>J 1.5Hz, CH<sub>3</sub>), 3.02 (1H, ddq, *J* 14.7, 8.4 and <sup>5</sup>J 1.5Hz, H4), 3.34 (1H, ddq, *J* 14.7, 10.2 and <sup>5</sup>J 1.5Hz, H4), 5.30 (1H, ddd, *J* 10.0, 8.4 and 7.8 Hz, H5), 6.34 (1H, dd, *J* 15.6 and 7.2 Hz, H<sub>olef.</sub>), 6.68 (1H, d, *J* 15.6 Hz, H<sub>olef.</sub>), 7.28-7.36 (3H, m), 7.41-7.50 (5H, m), 7.60-7.63 (2H, m). <sup>13</sup>C NMR (75MHz, CDCl<sub>3</sub>), δ<sub>C</sub> 15.8 (CH<sub>3</sub>), 37.6 (C4), 83.2 (C5), 112.7 (C3), 127.0, 127.7, 128.0 (2xCH), 128.4 (2xCH), 128.5 (2xCH), 128.9 (2xCH), 131.3, 130.0, 136.2, 141.1, 168.9 (C2), 193.4 (C=O).  $m/z$  (ESI<sup>+</sup>) = 291 (MH<sup>+</sup>, 100%). HRMS (ESI<sup>+</sup>):  $m/z$  (M+H)<sup>+</sup> C<sub>20</sub>H<sub>19</sub>O<sub>2</sub>: 291.13796 found: 291.14061. Anal. Calcd for C<sub>20</sub>H<sub>18</sub>O<sub>2</sub> (290.35): C, 82.73; H, 6.25%. Found: C, 82.87; H, 6.15%.

**2-(2,2-Diphenylvinyl)-6,6-dimethyl-3,5,6,7-tetrahydro-1-benzofuran-4(2H)-one (3h).**<sup>14</sup> Pale yellow solid, yield 91%, 313 mg, mp 113-115 °C, IR (ATR, cm<sup>-1</sup>): 2916, 1690 (C=O), 1686 (C=C), 1073, 742, 693. <sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>), δ<sub>H</sub> 1.08 (3H, s, CH<sub>3</sub>), 1.12 (3H, s, CH<sub>3</sub>), 2.23 (2H, s, H5), 2.95 (2H, t, *J* 2.0 Hz, H7), 2.73 (1H, dd, *J* 14.4 and 7.6 Hz, H3), 3.01 (1H, dd, *J* 14.4 and 10.4 Hz, H3), 5.27 (1H, td, *J* 10.0 and 7.6 Hz, H2), 6.14 (1H, d, *J* 9.2 Hz), 7.21 (2H, dd, *J* 8.4 and 2.0 Hz), 7.23-7.30 (5H, m), 7.36-7.41 (3H, m). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>), δ<sub>C</sub> 29.0 (2xCH<sub>3</sub>), 33.2, 34.3, 38.2, 51.2 (C3), 83.8 (C2), 111.8 (C3a), 126.5, 128.0 (2xCH), 128.2 (2xCH), 128.4 (2xCH), 128.5 (2xCH), 128.6, 130.1, 138.8, 141.3, 146.3, 176.4 (C7a), 195.0 (C=O).  $m/z$  (ESI<sup>+</sup>) 345 (MH<sup>+</sup>, %100). HRMS (ESI<sup>+</sup>):  $m/z$  (M+H)<sup>+</sup> C<sub>24</sub>H<sub>24</sub>O<sub>2</sub>: 345.18491 found: 345.18699.

**1-[5-(2,2-Diphenylvinyl)-2-methyl-4,5-dihydrofuran-3-yl]ethanone (3i).** Yellow oil, yield 84%, 255 mg, IR (ATR, cm<sup>-1</sup>): 3048, 2944, 1628 (C=O), 1602 (C=C), 1249, 1180, 968, 728, 692. <sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>), δ<sub>H</sub> 2.19 (3H, s, CH<sub>3</sub>), 2.22 (3H, s, CH<sub>3</sub>), 2.86 (1H, ddq, *J* 14.0, 8.0 and 1.6 Hz, H4), 3.09 (1H, ddq, *J* 14.4, 10.0 and 1.6 Hz, H4), 5.10 (1H, ddd, *J* 10.4, 9.2 and 8.0 Hz, H5), 6.16 (1H, d, *J* 9.2 Hz), 7.20 (2H, dd, *J* 8.4 and 2.0 Hz), 7.25-7.29 (5H, m), 7.35-7.40 (3H, m). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>), δ<sub>C</sub> 15.4 (CH<sub>3</sub>), 29.7 (CH<sub>3</sub>), 37.8 (C4), 80.3 (C5), 112.4 (C3), 126.9, 128.0 (2xCH), 128.1 (2xCH), 128.3 (2xCH), 128.5 (2xCH), 128.6, 130.1, 138.9, 141.4, 145.8, 167.8 (C2), 194.6 (C=O).  $m/z$  (ESI<sup>+</sup>) 305 (MH<sup>+</sup>, %100). HRMS (ESI<sup>+</sup>):  $m/z$  (M+H)<sup>+</sup> C<sub>21</sub>H<sub>20</sub>O<sub>2</sub>: 305.15361 found: 305.15616. Anal. Calcd for C<sub>21</sub>H<sub>20</sub>O<sub>2</sub> (304.38): C, 82.86; H, 6.62%. Found: C, 83.14; H, 6.55%.

**2,6,6-Trimethyl-2-[(E)-2-phenylvinyl]-3,5,6,7-tetrahydro-1-benzofuran-4(2H)-one (3j).**<sup>44, 45</sup> Colourless oil, yield 92%, 259 mg, <sup>1</sup>H NMR (300MHz, CDCl<sub>3</sub>), δ<sub>H</sub> 1.07 (3H, s, CH<sub>3</sub>), 1.08 (3H, s, CH<sub>3</sub>), 1.57 (3H, s, CH<sub>3</sub>), 2.21 (2H, s, H5), 2.29 (2H, s, H7), 2.72 (1H, d, *J* 14.4 Hz, H3), 2.90 (1H, d, *J* 14.4 Hz, H3), 6.27 (1H, d, *J* 15.9 Hz, H<sub>olef.</sub>), 6.53 (1H, d, *J* 15.9 Hz, H<sub>olef.</sub>), 7.20 (1H, t, *J* 6.9 Hz), 7.28 (2H, d, *J* 7.5 Hz), 7.35 (2H, d, *J* 6.9 Hz). HRMS (ESI<sup>+</sup>):  $m/z$  (M+H)<sup>+</sup> C<sub>19</sub>H<sub>22</sub>O<sub>2</sub>: 283.16926 found: 283.17136.

**2-Methyl-2-[(E)-2-phenylvinyl]-3,5,6,7-tetrahydro-1-benzofuran-4(2H)-one (3k).** Oil, yield 88%, 224 mg, IR (ATR,  $\text{cm}^{-1}$ ): 3026, 2943, 2866, 1620 (C=O), 1595 (C=C), 1244, 998, 749, 692.  $^1\text{H}$  NMR (300MHz,  $\text{CDCl}_3$ ),  $\delta_{\text{H}}$  1.58 (3H, s,  $\text{CH}_3$ ), 2.02 (2H, f,  $J$  6.6 Hz, H6), 2.34 (1H, t,  $J$  6.9 Hz, H5), 2.44 (2H, td,  $J$  6.9 and 1.5 Hz, H7), 2.71 (1H, td,  $J$  14.4 and 1.8 Hz, H3), 2.90 (1H, td,  $J$  14.4 and 1.8 Hz, H3), 6.29 (1H, d,  $J$  15.9 Hz,  $\text{H}_{\text{olef}}$ ), 6.55 (1H, d,  $J$  15.9 Hz,  $\text{H}_{\text{olef}}$ ), 7.24 (1H, tt,  $J$  6.6 and 1.5 Hz), 7.29 (2H, t,  $J$  6.9 Hz), 7.35 (2H, dd,  $J$  6.9 and 1.8 Hz).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ),  $\delta_{\text{C}}$  21.9 ( $\text{CH}_3$ ), 24.4, 27.1, 36.6, 38.6 (C3), 91.5 (C3a), 112.4 (C2), 126.9, 128.2 (2xCH), 128.7 (2xCH), 128.8, 132.1, 136.3, 176.4 (C7a), 196.0 (C=O).  $m/z$  ( $\text{ESI}^+$ ) 255 ( $\text{MH}^+$ , %100). HRMS ( $\text{ESI}^+$ ):  $m/z$  ( $\text{M}+\text{H}^+$ )  $\text{C}_{17}\text{H}_{18}\text{O}_2$ : 255.13795 found: 255.13822. Anal. Calcd for  $\text{C}_{17}\text{H}_{18}\text{O}_2$  (254.32): C, 80.28; H, 7.13%. Found: C, 80.36; H, 7.43%.

**2-Methyl-6-phenyl-2-[(E)-2-phenylvinyl]-3,5,6,7-tetrahydro-1-benzofuran-4(2H)-one (3l).** Colourless solid, yield 82%, 271 mg, mp 158-160  $^{\circ}\text{C}$ , IR (ATR,  $\text{cm}^{-1}$ ): 2977, 1649 (C=O), 1623 (C=C), 1218, 1028, 750, 690.  $^1\text{H}$  NMR (300MHz,  $\text{CDCl}_3$ ),  $\delta_{\text{H}}$  1.65 (3H, s,  $\text{CH}_3$ ), 2.65 (2H, dd,  $J$  9.0 and 2.4 Hz, H5), 2.68-2.73 (2H, m, H7), 2.80 (1H, ddd,  $J$  14.4, 4.2 and 1.5 Hz, H3), 2.98 (1H, ddd,  $J$  14.4, 4.2 and 1.5 Hz, H3), 3.46 (1H, m, H6), 6.32 (1H, d,  $J$  15.9 Hz,  $\text{H}_{\text{olef}}$ ), 6.55 (1H, d,  $J$  15.9 Hz,  $\text{H}_{\text{olef}}$ ), 7.25-7.40 (10H, m).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ),  $\delta_{\text{C}}$  27.1 ( $\text{CH}_3$ ), 31.9, 38.5, 40.6, 44.0 (C2), 92.2 (C3a), 112.6, 126.9, 127.0 (2xCH), 127.3 (2xCH), 128.3(2xCH), 128.9(2xCH), 129.0, 132.0, 136.2, 142.9, 175.5 (C7a), 194.5 (C=O).  $m/z$  ( $\text{ESI}^+$ ) 331 ( $\text{MH}^+$ , %100). HRMS ( $\text{ESI}^+$ ):  $m/z$  ( $\text{M}+\text{H}^+$ )  $\text{C}_{23}\text{H}_{22}\text{O}_2$ : 331.16926 found: 331.16895. Anal. Calcd for  $\text{C}_{23}\text{H}_{22}\text{O}_2$  (330.41): C, 83.60; H, 6.71%. Found: C, 83.91; H, 6.88%.

**1-{2,5-Dimethyl-5-[(E)-2-phenylvinyl]-4,5-dihydrofuran-3-yl}ethanone (3m).**<sup>45</sup> Yellow oil, yield 75%, 181 mg,  $^1\text{H}$  NMR (300MHz,  $\text{CDCl}_3$ ),  $\delta_{\text{H}}$  1.57 (3H, s,  $\text{CH}_3$ ), 2.27 (3H, t,  $J$  1.5 Hz,  $\text{CH}_3$ ), 2.86 (1H, dq,  $J$  14.1 and 1.5 Hz, H3), 3.02 (1H, dq,  $J$  14.1 and 1.5 Hz, H3), 6.30 (1H, d,  $J$  16.2 Hz,  $\text{H}_{\text{olef}}$ ), 6.55 (1H, d,  $J$  16.2 Hz,  $\text{H}_{\text{olef}}$ ), 7.27 (1H, tt,  $J$  6.9 and 1.5 Hz), 7.31(2H, t,  $J$  6.9 Hz), 7.38 (2H, dd,  $J$  6.9 and 1.8 Hz). HRMS ( $\text{ESI}^+$ ):  $m/z$  ( $\text{M}+\text{H}^+$ )  $\text{C}_{16}\text{H}_{18}\text{O}_2$ : 243.13796 found: 243.13702.

**Ethyl 2,5-dimethyl-5-[(E)-2-phenylvinyl]-4,5-dihydrofuran-3-carboxylate (3n).**<sup>45</sup> Pale yellow oil, yield 71%, 193 mg,  $^1\text{H}$  NMR (300MHz,  $\text{CDCl}_3$ ),  $\delta_{\text{H}}$  1.27 (3H, t,  $J$  7.2 Hz,  $-\text{OCH}_2\text{CH}_3$ ), 1.55 (3H, s,  $\text{CH}_3$ ), 2.24 (3H, t,  $J$  1.5 Hz,  $\text{CH}_3$ ), 2.80 (1H, dq,  $J$  14.4 and 1.5 Hz, H3), 2.97 (1H, dq,  $J$  14.4 and 1.5 Hz, H3), 4.16 (2H, q,  $J$  7.2 Hz,  $-\text{OCH}_2\text{CH}_3$ ), 6.29 (1H, d,  $J$  16.2 Hz,  $\text{H}_{\text{olef}}$ ), 6.54 (1H, d,  $J$  16.2 Hz,  $\text{H}_{\text{olef}}$ ), 7.24 (1H, tt,  $J$  7.2 and 1.2 Hz), 7.30 (2H, t,  $J$  6.9 Hz), 7.37 (2H, dd,  $J$  6.9 and 1.2 Hz). HRMS ( $\text{ESI}^+$ ):  $m/z$  ( $\text{M}+\text{H}^+$ )  $\text{C}_{17}\text{H}_{20}\text{O}_3$ : 273.14852 found: 273.14841.

**1-{5-Methyl-2-phenyl-5-[(E)-2-phenylvinyl]-4,5-dihydrofuran-3-yl}ethanone (3o).** Yellow oil, yield 40%, 122 mg, IR (ATR,  $\text{cm}^{-1}$ ): 3018, 1716 (C=O), 1600 (C=C), 1587 (C=C), 1241, 749, 693.  $^1\text{H}$  NMR (300MHz,  $\text{CDCl}_3$ ),  $\delta_{\text{H}}$  1.59 (3H, s,  $\text{CH}_3$ ), 1.88 (3H, s,  $\text{CH}_3$ ), 2.96 (1H, d,  $J$  14.4 Hz, H3), 3.20 (1H, d,  $J$  14.4 Hz, H3), 6.30 (1H, d,  $J$  16.2 Hz,  $\text{H}_{\text{olef}}$ ), 6.53 (1H, d,  $J$  16.2 Hz,  $\text{H}_{\text{olef}}$ ), 7.25 (2H, t,  $J$  6.9 Hz), 7.31-7.39 (5H, m), 7.56 (3H, m).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ),  $\delta_{\text{C}}$  15.9 ( $\text{CH}_3$ ), 27.0 ( $\text{CH}_3$ ), 43.6 (C4), 87.6 (C5), 114.6 (C3), 126.8, 127.9, 127.9 (2xCH), 128.1 (2xCH), 128.4 (2xCH), 128.8 (2xCH), 129.4, 131.1, 132.6, 136.5, 141.2, 168.0 (C2), 195.1



(C=O).  $m/z$  (ESI<sup>+</sup>) 305 (MH<sup>+</sup>, %100). HRMS (ESI<sup>+</sup>):  $m/z$  (M+H)<sup>+</sup> C<sub>21</sub>H<sub>20</sub>O<sub>2</sub>: 305.15361 found: 305.15495. Anal. Calcd for C<sub>21</sub>H<sub>20</sub>O<sub>2</sub> (304.38): C, 82.86; H, 6.62%. Found: C, 83.06; H, 6.84%.

**{2,5-Dimethyl-5-[(E)-2-phenylvinyl]-4,5-dihydrofuran-3-yl}(phenyl)methanone (3p)**. Yellow oil, yield 38%, 116 mg, IR (ATR, cm<sup>-1</sup>): 3027, 2974, 2927, 1650 (C=O), 1593 (C=C), 1239, 746, 693. <sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>), δ<sub>H</sub> 1.62 (3H, s, CH<sub>3</sub>), 1.89 (3H, t, *J* 1.6 Hz, CH<sub>3</sub>), 3.0 (1H, dq, *J* 14.4 and 1.6 Hz, H3), 3.20 (1H, dq, *J* 14.4 and 1.6 Hz, H3), 6.35 (1H, d, *J* 16.0 Hz, H<sub>olef.</sub>), 6.60 (1H, d, *J* 16.0 Hz, H<sub>olef.</sub>), 7.32 (2H, t, *J* 7.6 Hz), 7.39-7.42 (5H, m), 7.56 (3H, m). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>), δ<sub>C</sub> 15.7 (CH<sub>3</sub>), 27.1 (CH<sub>3</sub>), 43.4 (C4), 87.4 (C5), 112.0 (C3), 126.6, 127.7, 127.9 (2xCH), 128.1 (2xCH), 128.3(2xCH), 128.6 (2xCH), 132.1, 132.4, 136.2, 141.0, 167.7 (C2), 193.2 (C=O).  $m/z$  (ESI<sup>+</sup>) 305 (MH<sup>+</sup>, %100). HRMS (ESI<sup>+</sup>):  $m/z$  (M+H)<sup>+</sup> C<sub>21</sub>H<sub>20</sub>O<sub>2</sub>: 305.15361 found: 305.15534. Anal. Calcd for C<sub>21</sub>H<sub>20</sub>O<sub>2</sub> (304.38): C, 82.86; H, 6.62%. Found: C, 83.12; H, 6.41%.

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