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# CuCl-Catalyzed cycloisomerization reaction of 1,2-allenyl carboxylic acids. A cost-effective synthesis of β-unsubstituted butenolides

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**Abstract**—Fourteen examples of 2,3-butadienoic acids were synthesized according to the known procedures and their high-yielding CuCl-catalyzed cycloisomerization reaction to afford butenolides were described. 

Butenolides are a class of compounds of current interest due to their occurrence in natural products[1] and the potential broad range of biological activities of natural and unnatural products.[2] During the course of our study on transition metal-mediated cyclization of functionalized allenes,[3] we have developed the Pd(0)/$Ag^+$-cocatalyzed coupling-cyclization reaction of organic halides with 1,2-allenyl carboxylic acids to afford β-substituted butenolides.[4] Furthermore, among our efforts devoted to the development of new methodology for the synthesis of β-halobutenolides,[5] a $CuX_2$-mediated cyclization reaction of 1,2-allenyl carboxylic acids has been established.[6] Based on the observation that the in situ-generated CuCl mediated the formation of the by-products, i.e. β-unsubstituted butenolides,[6] we have developed the CuCl-*catalyzed* cycloisomerization reaction of 1,2-allenyl carboxylic acids, which provides an alternative cost-effective route for the synthesis of β-unsubstituted butenolides.

The CuCl-mediated cyclization of 2-methyl-4-phenyl-2,3-butadienoic acid was chosen as a starting point to optimize the reaction conditions and the results are summarized in Table 1.

From Table 1, it is obvious that this cycloisomerization reaction in aqueous acetone can be catalytic in CuCl (compare entry 1 with entries 2 and 3, Table 1). The corresponding catalytic reaction in acetone also afforded **2a** in a higher yield (entry 4, Table 1); however, the reaction did not occur in $CH_2Cl_2$ (entry 10, Table 1). The reaction also occurred in aqueous MeCN or DMF to afford **2a** in reasonable yields (entries 5–8, Table 1); the best reaction conditions were found to be using 4 mol% CuCl in $CH_3OH$ under reflux (entry 11, Table 1).

Compared to $AgNO_3$,[7] CuCl is relatively cheap,[8] thus, the current high-yielding CuCl-catalyzed cyclization reaction of 1,2-allenyl carboxylic acids merits close attention and some of the typical results are summarized in Table 2.

From Table 2, it should be pointed out that the yields for the reactions range from good to excellent; the yields starting from 4-aryl substituted 2,3-dienoic acids are usually higher than those starting from 4-alkyl substituted 2,3-dienoic acids (compare entries 1–6, 11, 14 with 7–10, 12, 13, Table 2). The reaction is fairly general, i.e. $R^1$ can be aryl or alkyl while $R^3$ can be H, alkyl, benzyl, or allyl. With an allyl group as the $R^3$ substituent, further elaboration of the C=C bond in the **2d**-type products would provide efficient access to other butenolide derivatives (entries 4, 13, and 14, Table 2). With fully substituted 2,3-allenoic acid **1k**, the reaction also afforded **2k** in 93% yield (entry 11, Table 2).

In conclusion, we have developed an efficient methodology for the synthesis of β-unsubstituted butenolides from 2,3-allenoic acids. Compared to known methodologies the current catalyst has the advantage of being relatively cheap (compared to $AgNO_3$), and more abundant,[9] low molecular weight (compared to $AgNO_3$), and high-yielding (at least comparable to $AgNO_3$[7] and much higher than that of the $H^+$-catalyzed reaction[10]), which makes this methodology more attractive. Further studies on the synthetic application to optically active butenolides and interesting natural and unnatural products are being carried out in our laboratory.

*Keywords*: butenolides; 1,2-allenyl carboxylic acids; CuCl-catalyzed cycloisomerization; cyclization.

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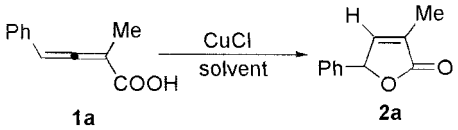
**Table 1.** CuCl-mediated cyclization of 2-methyl-4-phenyl-2,3-butadienoic acid




1a 2a

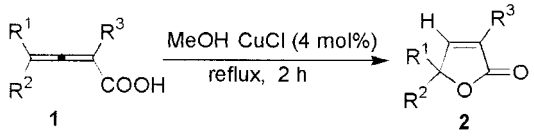
| Entry | Solvent | CuCl (mol%) | Temperature (°C) | Time (h) | Yield (%) |
|---|---|---|---|---|---|
| 1 | MeCOMe/$H_2O$ (3:2) | 200 | 65–70 | 4.5 | 53 |
| 2 | MeCOMe/$H_2O$ (3:2) | 12 | 65–70 | 1 | 81 |
| 3 | MeCOMe/$H_2O$ (3:1) | 22 | 65–70 | 1 | 65 |
| 4 | MeCOMe | 4 | 40 | 27 | 83 |
| 5 | MeCN/$H_2O$ (3:2) | 16 | 70–80 | 17.5 | 41 |
| 6 | DMF/$H_2O$ (3:2) | 11 | 70–80 | 0.5 | 64 |
| 7 | DMF/$H_2O$ (2:1) | 6 | 65–70 | 1 | 74 |
| 8 | DMF/$H_2O$ (5:1) | 7 | 65–70 | 1 | 75 |
| 9 | EtOH | 6 | 70 | 3.5 | 90 |
| 10 | $CH_2Cl_2$ | 8 | 55 | 5 | 0 |
| 11 | MeOH | 4 | Reflux | 2 | 95 |

## 1. Experimental

### 1.1. Starting materials

2,3-Undecadienoic acid (**1l**) was prepared according to a published procedure[11] via the reaction of $CO_2$ with the corresponding 1,2-allenic lithium salt, which in turn, was prepared from the treatment of the corresponding 1,2-allene with *n*-BuLi. The other allenoic acids (**1a**–**1k** and **1m**,**n**) were prepared according to the known method[12] by treatment of the acid chlorides with ethyl 2-(triphenylphoranylidene) propionate and subsequent hydrolysis of the 2,3-allenoic esters with 1.5 equiv. NaOH. CuCl (reagent grade) was purified according to the known procedure.[13]

**Table 2.** CuCl-catalyzed cyclization of 2,3-alkadienoic acids[a]




1 2

| Entry | 1 | | | Product 2 | Yield (%)[b] of 2 |
|---|---|---|---|---|---|
| | $R^1$ | $R^2$ | $R^3$ | | |
| 1 | Ph | H | $CH_3$ (**1a**) | **2a** | 95 |
| 2 | Ph | H | *n*-$C_3H_7$ (**1b**) | **2b** | 97 |
| 3 | Ph | H | $PhCH_2$ (**1c**) | **2c** | 90 |
| 4 | Ph | H | Allyl (**1d**) | **2d** | 96 |
| 5 | 1-naphthyl | H | $CH_3$ (**1e**) | **2e** | 92 |
| 6 | 1-naphthyl | H | *n*-$C_3H_7$ (**1f**) | **2f** | 96 |
| 7 | *n*-$C_3H_7$ | H | $CH_3$ (**1g**) | **2g** | 87 |
| 8 | *n*-$C_3H_7$ | H | $PhCH_2$ (**1h**) | **2h** | 85 |
| 9 | $CH_3$ | H | *n*-$C_3H_7$ (**1i**) | **2i** | 77 |
| 10 | *n*-$C_6H_{13}$ | H | $CH_3$ (**1j**) | **2j** | 85 |
| 11 | Ph | $C_2H_5$ | $CH_3$ (**1k**) | **2k** | 93 |
| 12 | *n*-$C_7H_{15}$ | H | H (**1l**) | **2l** | 81 |
| 13 | *n*-$C_3H_7$ | H | Allyl (**1m**) | **2m** | 91 |
| 14 | 1-naphthyl | H | Allyl (**1n**) | **2n** | 92 |

[a] The reactions were carried out using **1** (0.5 mmol), CuCl (4 mol%) in MeOH (5 mL).
[b] Isolated yield.

### 1.2. General procedure

Under $N_2$, a solution of **1** (0.5 mmol) and CuCl (0.02 mmol) in methanol (5 mL) was stirred under reflux for 2 h. After evaporation, the residues were purified via flash chromatography on silica gel with petroleum ether/ethyl acetate (10:1) as the eluent to afford the β-unsubstituted butenolides. All of the solid products were recrystallized from ethyl acetate and petroleum ether.

**1.2.1. 3-Methyl-5-phenyl-2(5H)-furanone (2a).**[14] Starting from **1a** (84 mg, 0.48 mmol) to afford 80 mg (95%) of **2a**; colorless oil; $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ 7.49–7.28 (m, 5H), 7.18 (bs, 1H), 5.92 (bs, 1H), 2.04 (s, 3H); EIMS *m*/*z* 175 ($M^+$+1, 49), 174 ($M^+$, 69), 105 (100); IR (neat) 1748, 1654, 1492 $cm^{-1}$.

**1.2.2. 5-Phenyl-3-propyl-2(5H)-furanone (2b).** Starting from **1b** (102 mg, 0.5 mmol) to afford 99 mg (97%) of **2b**; colorless oil; $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ 7.40–7.22 (m, 5H), 7.09 (d, *J*=1.6 Hz, 1H), 5.88 (d, *J*=1.6 Hz, 1H), 2.34 (t, *J*=5.2 Hz, 2H), 1.7–1.55 (m, 2H), 0.98 (t, *J*=7.3 Hz, 3H); EIMS *m*/*z* 203 ($M^+$+1, 30), 202 ($M^+$, 32), 105 (100); IR (neat) 1756, 1454 $cm^{-1}$. HRMS (EI) Calcd for $C_{13}H_{14}O_2$ 202.0994. Found 202.0978.

**1.2.3. 3-Benzyl-5-phenyl-2(5H)-furanone (2c).** Starting from **1c** (125 mg, 0.5 mmol) to afford 113 mg (90%) of **2c**; colorless platelet crystals mp 65–66°C; $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ 7.40–7.18 (m, 10H), 6.91 (d, *J*=1.3 Hz, 1H), 5.87 (d, *J*=1.3 Hz, 1H), 3.66 (s, 2H); EIMS *m*/*z* 250 ($M^+$, 10), 205 (100); IR (KBr) 1740, 1644, 1492, 1452 $cm^{-1}$. Anal. Calcd for $C_{17}H_{14}O_2$: C 81.58, H 5.64. Found: C 81.79, H 5.90.

**1.2.4. 3-Allyl-5-phenyl-2(5H)-furanone (2d).** Starting from **1d** (106 mg, 0.53 mmol) to afford 102 mg (96%) of **2d**; colorless oil; $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ 7.45–7.22 (m, 5H), 7.15–7.11 (m, 1H), 5.82–6.00 (m, 2H), 5.26–5.13 (m, 2H), 3.11 (d, *J*=5.4 Hz, 2H); EIMS *m*/*z* 221 ($M^+$+1, 70), 220 ($M^+$, 58), 105 (100); IR (neat) 1758, 1636, 1492, 1452, 1428 $cm^{-1}$. HRMS (EI) Calcd for $C_{13}H_{12}O_2$ 200.0834. Found 200.0833.

**1.2.5. 3-Methyl-5-naphthyl-2(5H)-furanone (2e).** Starting from **1e** (112 mg, 0.5 mmol) to afford 103 mg (92%) of **2e**; colorless needle crystals, mp 91–92°C; $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ 8.06 (d, $J$=8.0 Hz, 1H), 7.96–7.82 (m, 2H), 7.68–7.50 (m, 2H), 7.50–7.38 (m, 3H), 6.65 (s, 1H), 2.02 (s, 3H); EIMS $m/z$ 224 ($M^+$, 100); IR (KBr) 1744, 1650, 1598, 1506, 1438 cm$^{-1}$. Anal. Calcd for $C_{15}H_{12}O_2$: C 80.34, H 5.39. Found: C 80.23, H 5.45.

**1.2.6. 5-Naphthyl-3-propyl-2(5H)-furanone (2f).** Starting from **1f** (127 mg, 0.5 mmol) to afford 122 mg (96%) of **2f**; colorless crystal mp 84–85°C; $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ 8.08 (d, $J$=7.8 Hz, 1H), 7.9–7.8 (m, 2H), 7.6–7.5 (m, 2H), 7.45–7.3 (m, 3H), 6.66 (d, $J$=1.6 Hz, 1H), 2.36 (t, $J$=7.1 Hz, 2H), 1.7–1.56 (m, 2H), 0.99 (t, $J$=7.4 Hz, 3H); EIMS $m/z$ 252 ($M^+$, 5), 177 (100); IR (KBr) 1744, 1650, 1596, 1540, 1453, 1436 cm$^{-1}$. Anal. Calcd for $C_{17}H_{16}O_2$: C 80.93, H 6.39. Found: C 80.54, H 6.18.

**1.2.7. 3-Methyl-5-propyl-2(5H)-furanone (2g).**[15] Starting from **1g** (84 mg, 0.6 mmol) to afford 73 mg (87%) of **2g**; colorless oil; $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ 7.04 (bs, 1H), 4.92–4.86 (m, 1H), 1.92 (s, 3H), 1.75–1.4 (m, 4H), 0.97 (t, $J$=7.5 Hz, 3H); EIMS $m/z$ 141 ($M^+$+1, 100); IR (neat) 1748, 1654, 1464 cm$^{-1}$.

**1.2.8. 3-Benzyl-5-propyl-2(5H)-furanone (2h).** Starting from **1h** (110 mg, 0.51 mmol) to afford 93 mg (85%) of **2h**; colorless oil; $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ 7.36–7.19 (m, 5H), 6.79–6.82 (m, 1H), 4.95–4.85 (m, 1H), 3.57 (s, 2H), 1.71–1.51 (m, 2H), 1.5–1.35 (m, 2H), 0.93 (t, $J$=2.9 Hz, 3H); EIMS $m/z$ 217 ($M^+$+1, 33), 216 ($M^+$, 22), 129 (100); IR (neat) 1748, 1600, 1492, 1452 cm$^{-1}$. HRMS (EI) Calcd for $C_{14}H_{16}O_2$ 216.1146. Found 216.1183.

**1.2.9. 5-Methyl-3-propyl-2(5H)-furanone (2i).**[16] Starting from **1i** (81 mg, 0.58 mmol) to afford 63 mg (77%) of **2i**; colorless oil; $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ 6.99 (d, $J$=1.2 Hz, 1H), 4.99 (dq, $J$=1.2 and 6.9 Hz, 1H), 2.23 (t, $J$=7.3 Hz, 2H), 1.65–1.51 (m, 2H), 1.39 (d, $J$=6.9 Hz, 3H), 0.94 (t, $J$=7.5 Hz, 3H); EIMS $m/z$ 141 ($M^+$+1, 17), 140 ($M^+$, 48), 97 (100); IR (neat) 1746, 1650, 1454 cm$^{-1}$.

**1.2.10. 5-Hexyl-3-methyl-2(5H)-furanone (2j).**[17] Starting from **1j** (100 mg, 0.55 mmol) to afford 85 mg (85%) of **2j**; colorless oil; $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ 7.04 (bs, 1H), 4.95–4.85 (m, 1H), 1.92 (s, 3H), 1.76–1.58 (m, 2H), 1.50–1.24 (m, 8H), 0.88 (t, $J$=3.0 Hz, 3H); EIMS $m/z$ 183 ($M^+$+1, 3), 182 ($M^+$, 3), 43 (100); IR (neat) 1752, 1464 cm$^{-1}$.

**1.2.11. 5-Ethyl-3-methyl-5-phenyl-2(5H)-furanone (2k).**[10c] Starting from **1k** (106 mg, 0.52 mmol) to afford 99 mg (93%) of **2k** (eluent: petroleum ether/ethyl acetate=20:1); colorless oil; $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ 7.61–7.22 (m, 6H), 2.21–2.06 (m, 1H), 2.04–1.89 (m, 4H), 0.86 (t, $J$=5.6 Hz, 3H); EIMS $m/z$ 202 ($M^+$, 3), 173 (100); IR (neat) 1758, 1660, 1494, 1448 cm$^{-1}$.

**1.2.12. 5-Heptyl-2(5H)-furanone (2l).**[18] Starting from **1l** (98 mg, 0.54 mmol) to afford 79 mg (81%) of **2l** (eluent: petroleum ether/ethyl acetate=20:1); colorless oil; $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ 7.46 (d, $J$=5.6 Hz, 1H), 6.10 (d, $J$=5.6 Hz, 1H), 5.04 (t, $J$=6.4 Hz, 1H), 1.82–1.59 (m, 2H), 1.5–1.19 (m, 10H), 0.88 (t, $J$=4.0 Hz, 3H); EIMS $m/z$ 183 ($M^+$+1, 4), 182 ($M^+$, 7), 111 (100); IR (neat) 1747, 1599, 1465 cm$^{-1}$.

**1.2.13. 3-Propenyl-5-propyl-2(5H)-furanone (2m).** Starting from **1m** (86 mg, 0.52 mmol) to afford 78 mg (91%) of **2m** (eluent: petroleum ether/ethyl acetate=20:1); colorless oil; $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ 7.02 (s, 1H), 5.93–5.79 (m, 1H), 5.2–5.19 (m, 2H), 4.91 (m, 2H), 3.0 (d, $J$=5.8 Hz, 2H), 1.75–1.38 (m, 4H), 0.94 (t, $J$=7.3 Hz, 3H); EIMS $m/z$ 167 ($M^+$+1, 3), 166 ($M^+$, 7), 95 (100); IR (neat) 1747, 1636, 1465, 1430 cm$^{-1}$. HRMS (EI) Calcd for $C_{10}H_{14}O_2$ 166.0994. Found 166.0957.

**1.2.14. 5-Naphthyl-3-propenyl-2(5H)-furanone (2n).** Starting from **1n** (125 mg, 0.5 mmol) to afford 115 mg (92% yield) of **2n** (eluent: petroleum ether/ethyl acetate=20:1); colorless platelet crystals, mp 71–72°C; $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ 8.05 (d, $J$=8.3 Hz, 1H), 7.98–7.76 (m, 2H), 7.6–7.5 (m, 2H), 7.48–7.3 (m, 3H), 6.67 (s, 1H), 6.0–5.8 (m, 1H), 5.25–5.10 (m, 2H), 3.12 (d, $J$=6.7 Hz, 2H); EIMS $m/z$ 250 ($M^+$, 100); IR (neat) 1758, 1635, 1597, 1507 cm$^{-1}$, HRMS (EI) Calcd for $C_{17}H_{14}O_2$ 250.0994. Found 250.1029.

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