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# Ring-closing metathesis toward the synthesis of 2,5-dihydrofuran and 2,5-dihydropyrrole skeletons from Baylis–Hillman adducts

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**Abstract**—We have developed an efficient method for the synthesis of 2,5-dihydrofuran and 2,5-dihydropyrrole skeletons from the simply modified Baylis–Hillman adducts via RCM reaction.


Ring-closing metathesis (RCM) reaction is a powerful tool in modern chemistry due to its wide applicability in synthetic organic chemistry.[1] By using the RCM reaction tremendous cyclic compounds have been elegantly constructed including carbocyclic and heterocyclic ring.[1–4]

Crowe and Goldberg have reported the cross-metathesis (CM) between acrylonitrile and various terminal olefins.[2] Other π-conjugated olefins such as enones and enoic esters failed in cross-metathesis with less reactive old-fashioned catalyst. Recently, Grubbs and co-workers have reported the first successful results on the intermolecular cross-metathesis and intramolecular ring-closing metathesis with more reactive ruthenium catalyst for the α-functionalized olefin system bearing ester, aldehyde, benzoyl, or acetyl group.[3] More recently, ring-closing metathesis reaction of α-functionalized olefins have been studied extensively by many research groups.[4]

During the studies on the Baylis–Hillman and related chemistry[5] we envisioned that we could construct some useful ring systems by using the Baylis–Hillman adducts as the starting materials including 2,5-dihydropyrrole[6,8] and 2,5-dihydrofuran[7,8] as shown in Figure 1.

The Baylis–Hillman adducts have one olefinic double bond bearing an electron-withdrawing substituent such as an ester or nitrile functionality. As mentioned above Grubbs and co-workers have reported the CM reaction and RCM reaction by using such an olefinic double bond.[3] Thus, if we could introduce another double bond at the benzylic position of the Baylis–Hillman adducts[5e,9] we could prepare the desired RCM products (Scheme 1).

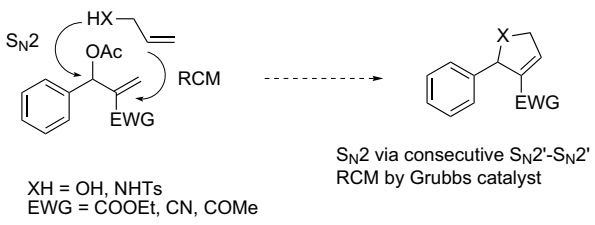
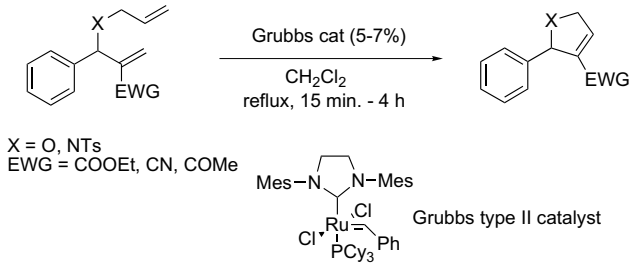



**Figure 1.**

To realize our idea we prepared *O*-allyl derivative **1a** of the Baylis–Hillman adduct via the well-known




**Scheme 1.**

*Keywords*: Ring-closing metathesis; 2,5-Dihydrofuran; 2,5-Dihydropyrrole; Baylis–Hillman adducts.
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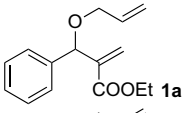
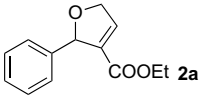
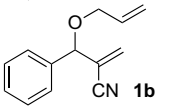
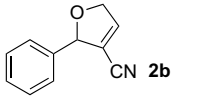
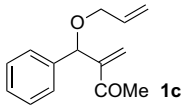
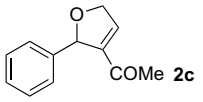
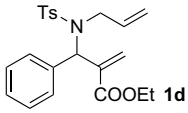
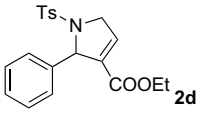
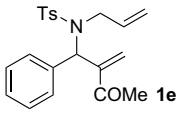
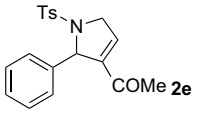
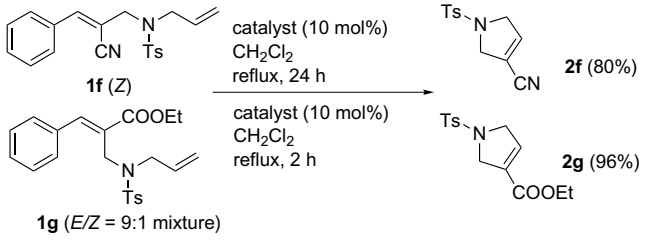
**Table 1.** RCM reaction of Baylis–Hillman adducts

| Entry | B–H adducts **1** | Conditions | Products **2** | Yields (%) |
|---|---|---|---|---|
| 1 |  **1a** | Catalyst (5 mol%) $CH_2Cl_2$ reflux, 15 min |  **2a** | 99 |
| 2 |  **1b** | Catalyst (5 mol%) $CH_2Cl_2$ reflux, 15 min |  **2b** | 88 |
| 3 |  **1c** | Catalyst (5 mol%) $CH_2Cl_2$ reflux, 15 min |  **2c** | 98 |
| 4 |  **1d** | Catalyst (7 mol%) $CH_2Cl_2$ reflux, 4 h |  **2d** | 98 |
| 5 |  **1e** | Catalyst (5 mol%) $CH_2Cl_2$ reflux, 4 h |  **2e** | 99 |

consecutive $S_N2'$–$S_N2'$ reaction using DABCO.[5e,9] With this compound in hand we examined the RCM reaction by using the commercial second generation Grubbs catalyst (5 mol%) in dichloromethane (reflux, 15 min) and obtained the 2,5-dihydrofuran **2a** in quantitative yield (99%).[10]

Thus, we prepared similar analogs **1b–e** and prepared the RCM products **2b–e** in excellent yields. We wish to report herein the results. As shown in Scheme 1 and Table 1, 2,5-dihydrofuran derivatives **2b** and **2c** were synthesized irrespective of the EWG including ester, nitrile, and acetyl group. As a next trial, we prepared the nitrogen analog **1d** and **1e** and examined the RCM reaction. Fortunately we could obtain the corresponding 2,5-dihydropyrroles **2d** and **2e** in good yields.

It is interesting to note that the RCM reaction of primary analog **1f** afforded the corresponding *N*-tosyl-3-cyano-2,5-dihydropyrrole **2f** in 80% yield (Scheme 2). This compound was generated by the elimination of styrene moiety (isolated as *trans*-stilbene by CM in 79% yield) during the RCM reaction.[11] We thought at first that the configuration around the double bond in such a primary compound is very important for the successful RCM reaction. The starting material **1f** has *Z*-configuration around the double bond and the RCM reaction can occur via the metal carbene intermediate, generated at the *N*-allyl moiety initially. However, the starting material **1g**, which has the *E*-configuration, gave also the corresponding RCM product, *N*-tosyl-3-ethoxycarbonyl-2,5-dihydropyrrole **2g** without any problem irrespective of the double bond configuration.




**Scheme 2.**

In conclusion, we have developed an efficient method for the synthesis of 2,5-dihydrofuran and 2,5-dihydropyrrole backbone from the simply modified Baylis–Hillman adducts via RCM reaction.

## Acknowledgements

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10. Synthesis of allyl ether **1a**: To a stirred solution of the corresponding Baylis–Hillman acetate (500 mg, 2 mmol) in THF (3 mL) was added DABCO (452 mg, 4 mmol) and stirred for 10 min at rt. To the reaction mixture was added allyl alcohol (1.5 mL) and heated to 50–60 °C for 3 days. After usual workup process and column chromatographic purification process (hexane/ether, 30:1) we could obtain the desired compound **1a** in 71% isolated yield (350 mg). Other compounds **1b** and **1c** were synthesized by using the same experimental procedure. The spectroscopic data of prepared compounds are listed below.
**1a** (71%): $^1$H NMR ($CDCl_3$) $\delta$ 1.22 (t, $J = 7.2$ Hz, 3H), 3.96 (dt, $J = 5.4$ and 1.5 Hz, 2H), 4.09–4.20 (m, 2H), 5.13–5.29 (m, 3H), 5.85–5.98 (m, 2H), 6.31 (s, 1H), 7.23–7.37 (m, 5H); $^{13}$C NMR ($CDCl_3$) $\delta$ 14.31, 60.67, 69.85, 78.38, 116.90, 124.81, 127.65, 127.82, 128.26, 134.59, 139.68, 141.52, 165.84.
**1b** (50%): $^1$H NMR ($CDCl_3$) $\delta$ 4.00 (dt, $J = 5.7$ and 1.5 Hz, 2H), 4.93 (s, 1H), 5.20–5.34 (m, 2H), 5.85–5.98 (m, 1H), 6.00–6.01 (m, 2H), 7.32–7.42 (m, 5H); $^{13}$C NMR ($CDCl_3$) $\delta$ 69.74, 80.09, 116.86, 117.74, 125.12, 127.00, 128.75 (2C), 130.35, 133.64, 137.28.
**1c** (71%): $^1$H NMR ($CDCl_3$) $\delta$ 2.81 (s, 3H), 3.91–3.94 (m, 2H), 5.13–5.29 (m, 2H), 5.41 (s, 1H), 5.84–5.97 (m, 1H), 6.17 (s, 2H), 7.21–7.37 (m, 5H); $^{13}$C NMR ($CDCl_3$) $\delta$ 26.30, 69.79, 77.18, 116.85, 125.03, 127.36, 127.65, 128.25, 134.58, 140.09, 149.58, 198.40.
Synthesis of *N*-allyl derivative **1d**: The reaction of tosylamide and allyl bromide gave the *N*-allyltosylamide in 53% yield ($K_2CO_3$, acetone, reflux, 7 h). To a stirred solution of the corresponding Baylis–Hillman acetate (300 mg, 1.2 mmol) in aqueous THF (4 mL, THF/$H_2O$, 3:1) was added DABCO (163 mg, 1.45 mmol) and stirred for 10 min at rt. To the reaction mixture was added *N*-allyltosylamide (255 mg, 1.2 mmol) and heated to 50–60 °C for 3 days. After usual workup and column chromatographic purification process (hexane/ethyl acetate, 4:1) we could obtain 220 mg of crude product as the mixture of Baylis–Hillman alcohol and desired product **1d** (in a ratio of 2:5 by $^1$H NMR). The two compounds have very similar mobility on TLC and we cannot separate them easily. Thus, we converted the Baylis–Hillman alcohol into its acetate again ($Ac_2O$, DMAP, $CH_2Cl_2$, rt, 4 h) and separate the desired product **1d** in 31% isolated yield (149 mg). The spectroscopic data of prepared compound **1d** are listed below.
**1d** (31%): $^1$H NMR ($CDCl_3$) $\delta$ 1.08 (t, $J = 7.1$ Hz, 3H), 2.43 (s, 3H), 3.79–3.84 (m, 2H), 3.98–4.12 (m, 2H), 4.80 (s, 1H), 4.84 (d, $J = 7.3$ Hz, 1H), 5.20–5.26 (m, 1H), 5.70 (s, 1H), 6.11 (s, 1H), 6.43 (s, 1H), 7.00–7.28 (m, 7H), 7.69 (d, $J = 8.3$ Hz, 2H).
Synthesis of *N*-allyl derivative **1e**: To a stirred solution of the corresponding Baylis–Hillman acetate (400 mg, 1.835 mmol) in aqueous THF (4 mL, THF/$H_2O$, 3:1) was added DABCO (247 mg, 2.2 mmol) and stirred for 10 min at rt. To the reaction mixture was added tosylamide (314 mg, 1.835 mmol) and heated to 60–70 °C for 20 h. After usual workup and column chromatographic purification process (hexane/$CH_2Cl_2$/ether, 10:10:1) we could obtain 252 mg of the Baylis–Hillman adduct derived from *N*-tosylimine in 42% yield.[5e] To the reaction mixture of this compound (130 mg, 0.395 mmol) and allyl bromide (72 mg, 0.593 mmol) in DMF (2 mL) was added $K_2CO_3$ (82 mg, 0.593 mmol) and stirred at room temperature for 1 h. After usual workup and column chromatographic purification process (hexane/ether, 3:1) we could obtain the desired product **1e** in 103 mg (71%).
**1e**: $^1$H NMR ($CDCl_3$) $\delta$ 2.29 (s. 3H), 2.43 (s, 3H), 3.82 (d, $J = 6.9$ Hz, 2H), 4.81–4.84 (m, 1H), 4.87 (s, 1H), 5.22–5.35 (m, 1H), 6.00 (d, $J = 1.5$ Hz, 1H), 6.12 (s, 1H), 6.32 (d, $J = 1.5$ Hz, 1H), 6.93–6.97 (m, 2H), 7.18–7.28 (m, 5H), 7.67 (d, $J = 8.4$ Hz, 2H); $^{13}$C NMR ($CDCl_3$) $\delta$ 21.49, 26.31, 48.99, 60.99, 117.68, 127.50, 127.76, 127.88, 128.44, 128.61, 129.40, 134.39, 137.19, 137.79, 143.17, 147.44, 198.03.
Synthesis of *N*-allyl derivative **1f**: To a stirred solution of the corresponding Baylis–Hillman acetate (100 mg, 0.498 mmol) and *N*-allyltosylamide (158 mg, 0.747 mmol) in DMF (2 mL) was added $K_2CO_3$ (103 mg, 0.747 mmol) and stirred at room temperature for 1 h. After usual workup and column chromatographic purification process (hexane/ether, 4:1) we could obtain the desired product **1f** in 106 mg (60%). Starting material **1g** was prepared similarly in 40% yield.
**1f**: 60%; $^1$H NMR ($CDCl_3$) $\delta$ 2.41 (s, 3H), 3.93 (d, $J = 6.3$ Hz, 2H), 4.11 (s, 2H), 5.19–5.26 (m, 2H), 7.12 (s, 1H), 7.29 (d, $J = 8.1$ Hz, 2H), 7.41–7.44 (m, 3H), 7.67–7.71 (m, 2H), 7.74 (d, $J = 8.1$ Hz, 2H).
**1g**: 40%; $^1$H NMR ($CDCl_3$) $\delta$ 1.31 (t, $J = 7.2$ Hz, 3H), 2.40 (s, 3H), 3.74 (d, $J = 6.3$ Hz, 2H), 4.19 (q, $J = 7.2$ Hz, 2H), 4.24 (s, 2H), 4.90–4.97 (m, 2H), 5.45–5.56 (m, 1H), 7.21 (d, $J = 8.1$ Hz, 2H), 7.39–7.41 (m, 5H), 7.54 (d, $J = 8.1$ Hz, 2H), 7.80 (s, 1H).
Typical procedure for the synthesis of 2,5-dihydrofuran derivative **2a**: To a stirred solution of allyl ether **1a** (123 mg, 0.5 mmol) in dichloromethane (50 mL) was added

Grubbs catalyst (21 mg, 0.025 mmol, 5 mol %) and heated to reflux for 15 min. After removal of the solvent and column chromatographic purification process (hexane/ether, 2:1) we could obtain the 2,5-dihydrofuran derivative **2a**, 108 mg (99%). Other compounds were synthesized by using the same experimental procedure. The spectroscopic data of prepared compounds are listed below.

**2a** (99%): IR (KBr) 1720, 1261 $cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta$ 1.51 (t, $J = 7.2$ Hz, 3H), 4.00–4.17 (m, 2H), 4.86 (ddd, $J = 15.9$, 3.9, and 1.8 Hz, 1H), 5.01 (ddd, $J = 15.9$, 6.3, and 1.8 Hz, 1H), 5.90–5.94 (m, 1H), 7.00 (q, $J = 1.8$ Hz, 1H), 7.25–7.36 (m, 5H); $^{13}C$ NMR ($CDCl_3$) $\delta$ 13.90, 60.51, 75.26, 86.88, 127.10, 128.11, 128.24, 135.91, 138.25, 140.81, 162.23; HRMS calcd for $C_{13}H_{14}O_3$ 218.0943, found 218.0947.

**2b** (88%): IR (KBr) 2229 $cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta$ 4.91 (ddd, $J = 15.9$, 4.2, and 1.8 Hz, 1H), 5.03 (ddd, $J = 15.9$, 6.3, and 1.8 Hz, 1H), 5.82–5.87 (m, 1H), 6.88 (q, $J = 1.8$ Hz, 1H), 7.32–7.44 (m, 5H); $^{13}C$ NMR ($CDCl_3$) $\delta$ 76.00, 87.61, 113.15, 116.04, 126.34, 128.85, 129.01, 138.22, 142.75; HRMS calcd for $C_{11}H_9NO$ 171.0684, found 171.0688.

**2c** (98%): IR (KBr) 1763, 1685 $cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta$ 2.27 (s, 3H), 4.90 (ddd, $J = 16.5$, 3.3, and 2.1 Hz, 1H), 5.06 (ddd, $J = 16.5$, 6.0, and 2.1 Hz, 1H), 5.96–5.99 (m, 1H), 6.91 (q, $J = 2.1$ Hz, 1H), 7.26–7.35 (m, 5H); $^{13}C$ NMR ($CDCl_3$) $\delta$ 27.56, 75.26, 86.96, 127.00, 128.11, 128.35, 138.17, 140.80, 143.96, 193.43; HRMS calcd for $C_{12}H_{12}O_2$ 188.0837, found 188.0835.

**2d** (98%): IR (KBr) 1720, 1346, 1161 $cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta$ 1.09 (t, $J = 7.2$ Hz, 3H), 2.36 (s, 3H), 3.93–4.10 (m, 2H), 4.37 (ddd, $J = 16.8$, 5.7, and 2.1 Hz, 1H), 4.48–4.55 (m, 1H), 5.72–5.75 (m, 1H), 6.77 (q, $J = 2.1$ Hz, 1H), 7.13 (d, $J = 8.1$ Hz, 2H), 7.18-7.25 (m, 5H), 7.41 (d, $J = 8.1$ Hz, 2H); $^{13}C$ NMR ($CDCl_3$) $\delta$ 13.82, 21.42, 54.85, 60.76, 68.97, 127.06, 127.78, 127.93, 128.19, 129.41, 135.39, 135.58, 135.98, 129.43, 143.22, 161.76; HRMS calcd for $C_{20}H_{21}NO_4S$ 371.1191, found 371.1187.

**2e** (99%): white solid, mp 155–156 °C; IR (KBr) 1674, 1342, 1165 $cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta$ 2.16 (s, 3H), 2.36 (s, 3H), 4.43 (ddd, $J = 17.7$, 5.7, and 2.4 Hz, 1H), 4.51–4.59 (dt, $J = 17.7$ and 2.4 Hz, 1H), 5.76–5.79 (m, 1H), 6.66–6.68 (m, 1H), 7.13 (d, $J = 8.4$ Hz, 2H), 7.21 (s, 5H), 7.42 (d, $J = 8.4$ Hz, 2H); $^{13}C$ NMR ($CDCl_3$) $\delta$ 21.41, 26.99, 55.06, 68.78, 127.05, 127.61, 127.88, 128.26, 129.43, 135.08, 135.42, 139.47, 143.27, 143.91, 192.91; HRMS calcd for $C_{19}H_{19}NO_3$ S 341.1086, found 341.1081.

**2f** (80%): mp 161–162 °C; IR (KBr) 2233 $cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta$ 2.45 (s, 3H), 4.24–4.32 (m, 4H), 6.52–6.53 (m, 1H), 7.36 (d, $J = 8.4$ Hz, 2H), 7.71 (d, $J = 8.4$ Hz, 2H); $^{13}C$ NMR ($CDCl_3$) $\delta$ 21.51, 54.56, 55.19, 110.73, 112.81, 127.36, 130.10, 133.20, 141.83, 144.40; HRMS calcd for $C_{12}H_{12}N_2O_2S$ 248.0619, found 248.0623.

**2g** (96%): mp 103–104 °C; IR (KBr) 1712, 1346, 1161 $cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta$ 1.27 (t, $J = 7.2$ Hz, 3H), 2.43 (s, 3H), 4.18 (q, $J = 7.2$ Hz, 2H), 4.28 (s, 4H), 6.58 (s, 1H), 7.33 (d, $J = 8.4$ Hz, 2H), 7.73 (d, $J = 8.4$ Hz, 2H); $^{13}C$ NMR ($CDCl_3$) $\delta$ 14.07, 21.47, 53.47, 55.38, 60.92, 127.40, 129.87, 131.90, 133.73, 135.71, 143.79, 162.05; HRMS calcd for $C_{14}H_{17}NO_4S$ 295.0878, found 295.0871.

11. For the RCM reaction involving the styryl moiety, see: (a) Maishal, T. K.; Sarkar, A. *Synlett* **2002**, 1925; (b) Maity, B. C.; Swamy, V. M.; Sarkar, A. *Tetrahedron Lett.* **2001**, *42*, 4373.