Synthesis of Hexahydrofuro[2,3-*b*]furan and Hexahydrofuro[2,3-*b*]pyran Derivatives Starting from Baylis-Hillman Adducts *via* the Ueno-Stork Reaction

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Fused polycyclic acetals are embodied in a wide range of natural products. Among bicyclic acetals, furofuran and furopyran derivatives are of special interest since both aliphatic and benzoannelated compounds of biological and pharmaceutical activity are known.[1-4] Especially, the hexahydrofurofuran unit is present in many biologically active natural products.[1d,1e] Some representative examples are communiol D, lupulin A, and asteltoxin as shown in Figure 1.[1d,1e]

During the investigation of radical cyclization of the suitably modified Baylis-Hillman adducts,[5] we reasoned that we could synthesize a variety of furofuran and furopyran derivatives by following the Scheme 1. We reasoned that synthesis of bromoacetals from cinnamyl alcohols and 2,3-dihydrofuran or 3,4-dihydro-2*H*-pyran[4,6] and the following radical cyclization (Ueno-Stork reaction)[3] would give the desired furofuran or furopyran derivatives.[2] The use of cinnamyl alcohol derivative like **1a** as the starting material would afford the furofuran or furopyran derivatives having the ester functionality at the 3-position, which could be functionalized for further transformations.

The reaction of the cinnamyl alcohol **1a**,[7] which was prepared from the Baylis-Hillman adduct of benzaldehyde and methyl acrylate, and 3,4-dihydro-2*H*-pyran in the presence of NBS (*N*-bromosuccinimide) in acetonitrile at room temperature gave the desired bromoacetal **2a** in moderate yield (74%). As reported, **2a** was obtained as a *trans* isomer *via* the ring opening reaction of the intermediate bromonium ion.[3,4,6] With bromoacetal **2a** in our hand we tried radical cyclization (Ueno-Stork reaction) under the typical condition, *n*-Bu_3SnH/AIBN in refluxing benzene. As expected, we could obtain the diastereomeric

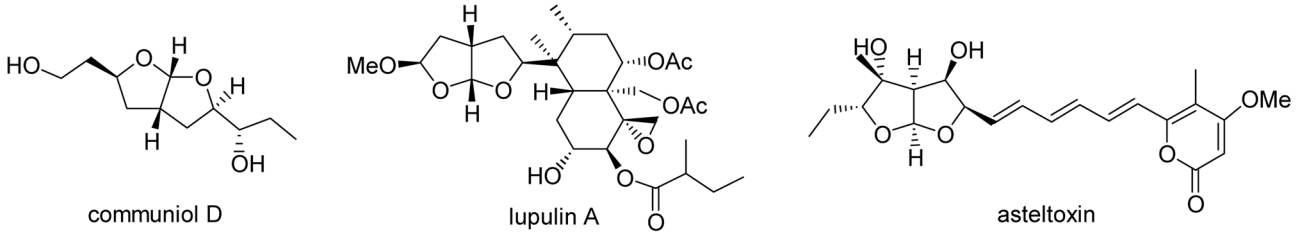
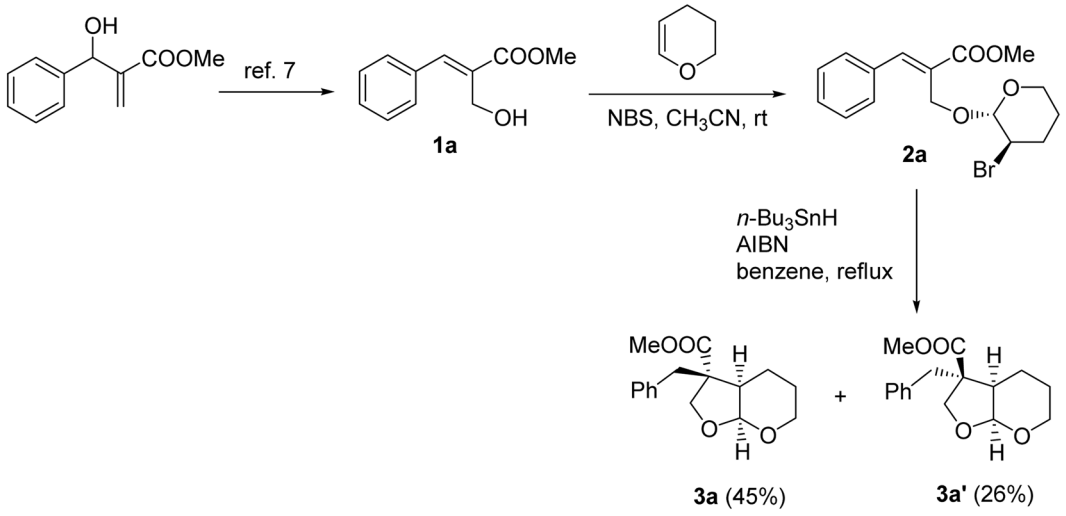
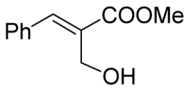
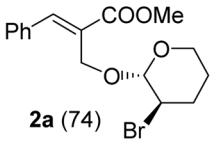
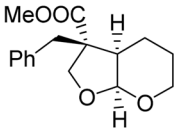
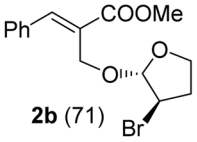
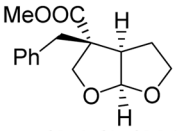
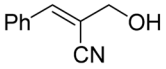
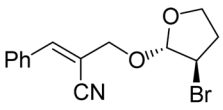
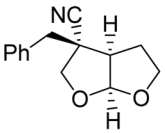
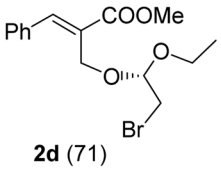
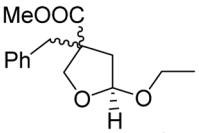
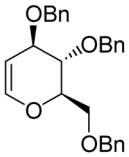
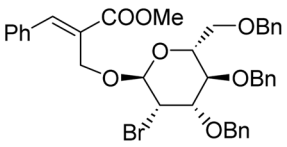
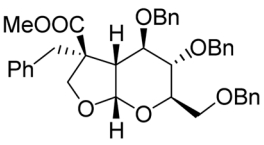


Figure 1



Scheme 1

Table 1. Synthesis of furopyran and furofuran derivatives

Entry	Alcohols	Enol ethers	Bromoacetals (%)	Products (%)[a]
1	 **1a**		 **2a** (74)	 **3a** (45), **3a'** (26)
2	**1a**		 **2b** (71)	 **3b** (43), **3b'** (41)
3	 **1b**		 **2c** (60)	 **3c** (75), **3c'** (-)[b]
4	**1a**		 **2d** (71)	 **3d** + **3d'** (86)[c]
5	**1a**		 **2e** (67)	 **3e** (74), **3e'** (20)

[a]Isolated yields and we showed the structures of the major isomers of **3a-c** and **3e**. [b]The other diastereomer was not isolated. [c]The two diastereomers were not separated.

mixtures of products **3a** and **3a'** in 45 and 26%, respectively. The two protons at the ring junction of **3a** and **3a'** must be *cis*-relationships based on the previous reports and the small coupling constants between the protons at the ring junction.[1-4] The coupling constants of **3a** and **3a'** between the two protons at the ring junction were 3.9 and 4.2 Hz, respectively. Thus, the relationships of **3a** and **3a'** must be diastereomers having different stereochemistry at the 3-position as shown in Table 1 (entry 1). Encouraged by the successful results we synthesized starting materials **2b-e** and examined the synthesis of a variety of fused ring systems. The results are summarized in Table 1.

For the reaction of **2b**, which was derived from the reaction of **1a** and 2,3-dihydrofuran (entry 2), we obtained **3b** and **3b'** in 43 and 41%, respectively. In this case, the stereoselectivity was almost lost. However, when we used **2c** as the starting material, we could isolate only **3c** in 75% yield to our surprise (entry 3).[8] We could not isolate the other stereoisomer **3c'**. From the NOE experiments of **3c** we confirmed the structure as shown in Figure 2. In the reaction

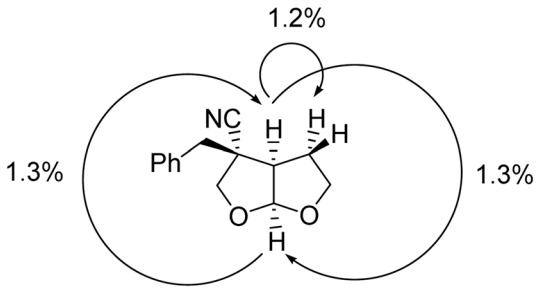


Figure 2

of **2d**, the two isomers **3d** and **3d'** have almost same R_f values and we could not separate them (entry 4). When we used **2e**, which was synthesized from **1a** and tri-*O*-benzyl-D-glucal, we isolated **3e** and **3e'** in 74 and 20%, respectively (entry 5).

In summary, we prepared some oxabicyclic compounds by using the Ueno-Stork radical cyclization reaction of bromoacetals, which were prepared starting from the Baylis-

Hillman adducts. Further studies on the synthetic applications of this protocol are currently underway.

Experimental Section

Typical synthetic procedure for the bromoacetal 2a. To a stirred solution of cinnamyl alcohol **1a** (192 mg, 1 mmol) and 3,4-dihydro-2*H*-pyran (168 mg, 2 mmol) in CH_3CN (2 mL) was added NBS (354 mg, 2 mmol) and stirred at room temperature for 4 h. After the usual aqueous extractive workup with ether and column chromatographic purification process (hexanes/ether, 9 : 2), desired bromoacetal **2a** was obtained as clear oil, 262 mg (74%). Other bromoacetals **2b-e** were synthesized similarly and the spectroscopic data are as follows.

Compound **2a**: 74%; oil; IR (neat) 2951, 1716, 1238, 1026 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz) δ 1.51-1.63 (m, 1H), 1.86-2.01 (m, 2H), 2.35-2.46 (m, 1H), 3.54-3.62 (m, 1H), 3.85 (s, 3H), 3.94-4.05 (m, 2H), 4.36 (d, J = 10.5 Hz, 1H), 4.66 (d, J = 10.5 Hz, 1H), 4.77 (d, J = 4.8 Hz, 1H), 7.37-7.57 (m, 5H), 7.94 (s, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 23.34, 30.29, 49.24, 52.12, 62.66, 62.77, 101.29, 128.22, 128.49, 129.32, 129.69, 134.46, 144.62, 167.79.

Compound **2b**: 51%; oil; IR (neat) 1716, 1238, 1022 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz) δ 2.20-2.29 (m, 1H), 2.57-2.75 (m, 1H), 3.85 (s, 3H), 4.09-4.30 (m, 3H), 4.32 (d, J = 10.2 Hz, 1H), 4.55 (d, J = 10.2 Hz, 1H), 5.38 (s, 1H), 7.37-7.50 (m, 5H), 7.90 (s, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 33.85, 49.96, 52.21, 61.65, 66.90, 108.63, 128.22, 128.53, 129.44, 129.67, 134.53, 144.48, 167.80.

Compound **2c**: 60%; oil; IR (neat) 2958, 2214, 1030 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz) δ 2.21-2.29 (m, 1H), 2.65-2.78 (m, 1H), 4.11-4.33 (m, 3H), 4.25 (d, J = 12.3 Hz, 1H), 4.40 (d, J = 12.3 Hz, 1H), 5.32 (s, 1H), 7.12 (s, 1H), 7.40-7.79 (m, 5H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 33.74, 49.61, 67.27, 68.23, 107.74, 107.83, 117.50, 128.91, 129.05, 130.80, 132.80, 145.37.

Compound **2d**: 70%; oil; IR (neat) 1716, 1238, 1115 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz) δ 1.24 (t, J = 7.2 Hz, 3H), 3.41-3.48 (m, 2H), 3.60-3.79 (m, 2H), 3.85 (s, 3H), 4.41 (d, J = 10.2 Hz, 1H), 4.51 (d, J = 10.2 Hz, 1H), 4.84 (t, J = 5.4 Hz, 1H), 7.37-7.60 (m, 5H), 7.96 (s, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 15.07, 31.70, 52.17, 61.20, 62.66, 101.80, 127.97, 128.56, 129.48, 129.81, 134.44, 145.04, 167.83.

Compound **2e**: 67%; oil; IR (neat) 1712, 1115 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz) δ 3.65-4.05 (m, 5H), 3.82 (s, 3H), 4.29 (d, J = 11.1 Hz, 1H), 4.38-4.40 (m, 1H), 4.46-4.73 (m, 6H), 4.86 (d, J = 10.8 Hz, 1H), 5.26 (d, J = 1.2 Hz, 1H), 7.14-7.44 (m, 20H), 7.92 (s, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 51.53, 52.23, 62.55, 68.72, 71.25, 72.58, 73.39, 74.50, 75.21, 100.40, 127.44, 127.60, 127.68, 127.78, 127.94, 127.97, 128.05, 128.25, 128.28, 128.40, 128.66, 129.45, 129.48, 134.46, 137.76, 138.36, 138.39, 144.80, 167.57.

Typical procedure for the radical cyclization of 2a to 3a and 3a'. A stirred mixture of bromoacetal **2a** (177 mg, 0.5 mmol), AIBN (16 mg, 0.1 mmol), and *n*-Bu_3SnH (160 mg, 0.55 mmol) in benzene (3 mL) was heated to reflux for 2 h. After the usual aqueous extractive workup with ether and column chromatographic purification process (hexanes/EtOAc, 9 : 1), desired products **3a** (62 mg, 45%) and **3a'** (36 mg, 26%) were obtained. Other compounds were synthesized similarly and the spectroscopic data are as follows. We could not separate **3d** and **3d'** in pure states. R_f values for **3** and **3'** were checked (hexanes/EtOAc, 6 : 4) and reported together.

Compound **3a**: 45%; oil; R_f = 0.53; IR (neat) 2951, 1732, 1153, 1022 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz) δ 1.59-1.81 (m, 3H), 1.95-2.03 (m, 1H), 2.41-2.48 (m, 1H), 2.97 (d, J = 13.5 Hz, 1H), 3.14 (d, J = 13.5 Hz, 1H), 3.59 (s, 3H), 3.62-3.68 (m, 1H), 3.78-3.87 (m, 1H), 4.05 (d, J = 8.7 Hz, 1H), 4.32 (d, J = 8.7 Hz, 1H), 5.20 (d, J = 3.9 Hz, 1H), 7.02-7.26 (m, 5H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 21.11, 23.11, 37.43, 41.01, 52.08, 57.41, 61.40, 71.62, 101.24, 126.89, 128.47, 128.97, 136.89, 174.82; ESIMS *m/z* 277.1 (M^++H).

Compound **3a'**: 26%; white solid, mp 130-132 °C; R_f = 0.56; IR (neat) 2947, 1728, 1200, 1026 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz) δ 1.49-1.59 (m, 3H), 1.79-1.87 (m, 1H), 2.11-2.18 (m, 1H), 2.87 (d, J = 13.5 Hz, 1H), 3.22 (d, J = 13.5 Hz, 1H), 3.58-3.77 (m, 2H), 3.65 (s, 3H), 3.91 (d, J = 9.3 Hz, 1H), 4.32 (d, J = 9.3 Hz, 1H), 5.52 (d, J = 4.2 Hz, 1H), 7.05-7.30 (m, 5H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 21.54, 22.46, 42.20, 42.93, 51.59, 58.11, 60.92, 69.11, 100.87, 127.05, 128.42, 129.62, 136.50, 172.55.

Compound **3b**: 43%; oil; R_f = 0.50; IR (neat) 2954, 1732, 1207, 1011 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz) δ 2.02-2.15 (m, 2H), 2.97 (d, J = 14.1 Hz, 1H), 3.08 (d, J = 14.1 Hz, 1H), 3.28-3.36 (m, 1H), 3.62 (s, 3H), 3.79 (d, J = 8.7 Hz, 1H), 3.86-3.94 (m, 1H), 4.01-4.09 (m, 1H), 4.30 (d, J = 8.7 Hz, 1H), 5.70 (d, J = 5.1 Hz, 1H), 7.03-7.30 (m, 5H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 26.18, 36.89, 47.97, 52.26, 59.02, 68.77, 74.14, 109.43, 126.96, 128.53, 128.80, 136.63, 174.81; ESIMS *m/z* 263.1 (M^++H).

Compound **3b'**: 41%; white solid, mp 95-98 °C; R_f = 0.53; IR (neat) 2954, 1732, 1088, 1011 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz) δ 1.57-1.70 (m, 1H), 2.05-2.19 (m, 1H), 2.83 (d, J = 13.4 Hz, 1H), 2.82-2.90 (m, 1H), 3.17 (d, J = 13.4 Hz, 1H), 3.68 (s, 3H), 3.81-4.00 (m, 4H), 5.82 (d, J = 4.8 Hz, 1H), 7.06-7.30 (m, 5H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 28.50, 42.28, 51.67, 59.70, 68.33, 69.44, 77.20, 108.57, 127.08, 128.44, 129.63, 136.56, 172.79.

Compound **3c**: 75%; white solid, mp 70-72 °C; R_f = 0.23; IR (neat) 2958, 2877, 2237, 1014 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz) δ 2.04-2.12 (m, 2H), 2.95 (d, J = 13.8 Hz, 1H), 3.05 (d, J = 13.8 Hz, 1H), 3.12-3.19 (m, 1H), 3.87 (d, J = 9.3 Hz, 1H), 3.90-3.98 (m, 1H), 4.01-4.09 (m, 2H), 5.93 (d, J = 4.8 Hz, 1H), 7.28-7.40 (m, 5H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 25.56, 36.72, 48.50, 51.41, 68.94, 74.09, 109.24, 122.64, 127.84, 128.90, 129.38, 134.68; ESIMS *m/z* 230.1 (M^++H).

Compound **3d** + **3d'** (as a mixture): 86%; oil; R_f = 0.72; IR (neat) 2951, 1736, 1207, 1115 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz) δ 1.16 (t, J = 7.2 Hz, 1.5H), 1.22 (t, J = 7.2 Hz, 1.5H), 1.92 (d, J = 13.8 Hz, 0.5H), 2.14 (dd, J = 13.8 and 5.4 Hz, 0.5H), 2.36 (d, J = 13.8 Hz, 0.5H), 2.64 (dd, J = 13.8 and 5.4

Hz, 0.5H), 2.96 (d, J = 13.5 Hz, 0.5H), 3.03 (d, J = 13.5 Hz, 0.5H), 3.05 (d, J = 13.5 Hz, 0.5H), 3.19 (d, J = 13.5 Hz, 0.5H), 3.36-3.52 (m, 1H), 3.65 (s, 1.5H), 3.67 (s, 1.5H), 3.70-3.89 (m, 2H), 4.16-4.24 (m, 1H), 5.14-5.18 (m, 1H), 7.05-7.30 (m, 5H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 15.12, 15.25, 41.16, 41.86 (overlapped), 43.07, 51.85, 52.11, 54.68, 55.89, 62.75, 63.41, 71.33, 73.95, 103.41, 104.30, 126.78, 126.90, 128.35 (overlapped), 129.22, 129.48, 137.12, 137.44, 174.38, 175.24; ESIMS m/z 265.1 (M^++H).

Compound **3e**: 74%; white solid, mp 105-108 °C; R_f = 0.50; IR (neat) 2920, 1732, 1092 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 2.83-2.89 (m, 2H), 3.45 (d, J = 13.5 Hz, 1H), 3.57 (s, 3H), 3.70-3.95 (m, 5H), 4.00 (d, J = 8.7 Hz, 1H), 4.35 (d, J = 8.7 Hz, 1H), 4.53 (d, J = 12.3 Hz, 1H), 4.64-4.77 (m, 4H), 5.12 (d, J = 11.4 Hz, 1H), 5.35 (d, J = 4.5 Hz, 1H), 6.97-7.01 (m, 2H), 7.15-7.35 (m, 18H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 38.26, 49.75, 52.21, 57.95, 68.40, 71.67, 71.73, 72.92, 73.61, 74.36, 77.97, 78.76, 101.17, 126.96, 127.31, 127.53, 127.67, 127.73, 127.81, 127.89, 128.37, 128.46, 129.01, 136.51, 137.84, 138.18, 174.05 (three carbons were overlapped); ESIMS m/z 609.3 (M^++H).

Compound **3e'**: 20%; oil; R_f = 0.48; IR (neat) 1732, 1095 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 3.20-3.23 (m, 1H), 3.30 (d, J = 13.5 Hz, 1H), 3.53 (s, 3H), 3.65-3.79 (m, 4H), 4.02-4.04 (m, 2H), 4.31 (s, 2H), 4.52-4.66 (m, 5H), 4.74 (d, J = 11.1 Hz, 1H), 5.29 (d, J = 4.5 Hz, 1H), 7.07-7.11 (m, 2H), 7.17-7.37 (m, 18H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 37.76, 52.12, 56.08, 69.69, 72.21, 73.37, 73.93, 74.57, 75.76, 79.27, 102.45, 126.47, 127.48, 127.61, 127.78, 127.95, 128.05, 128.22, 128.28, 128.37, 129.71, 137.91, 137.98, 138.06, 138.21, 175.24 (four carbons were overlapped).

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References and Notes

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8. We calculated the relative energies of **3c** and **3c'** by using MM2 and found that **3c** was more stable than **3c'** in about 3.0 kcal/mol. The difference in energy might result in the selective formation of **3c**. More precise energy calculations will be carried out in due course.