A Tandem Intramolecular Michael Addition/Wittig Reaction for the Synthesis of Fused Cyclohexadiene Derivatives†

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A tandem intramolecular Michael addition/Wittig reaction has been developed for the synthesis of fused cyclohexadiene derivatives from phosphonium salt **3**. This annulation reaction affords the cyclohexadienes in moderate to excellent yields, depending on the substrates. A mechanism is proposed to account for the tandem process.

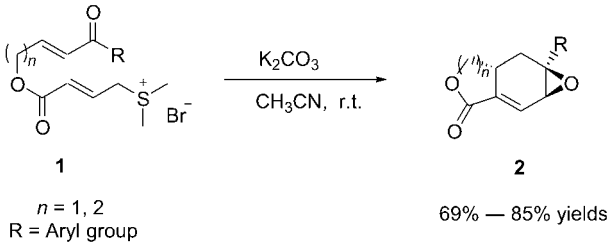
Keywords tandem intramolecular reaction, Michael addition, Wittig reaction, fused cyclohexadiene derivative, mechanism

Introduction

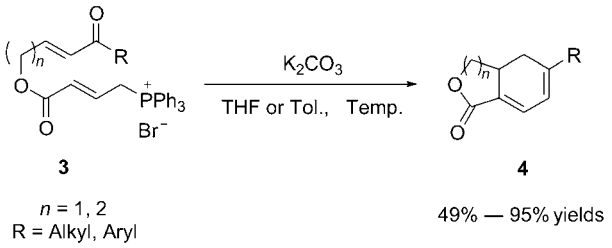
Ylide reactions have been developed as a very powerful method to construct carbon-carbon double bonds and carbon-heteroatom bonds since Wittig reaction was reported in 1950 s.[1] In the past decades, much attention has been paid to the construction of three-membered ring compounds, especially to epoxides,[2] cyclopropanes[2a,2c,2d,3] and aziridines,[2a,2c,2d,4] which occurs frequently in biologically active compounds as well as their utility as valuable synthetic intermediates. Recently, lots of effort has been concentrated on constructing cyclic compounds beyond three-membered rings via ylide routes,[5] such as dihydrofuran,[6] dihydropyrroles,[7] and isoxazoline *N*-oxide derivatives[8] *etc*. Besides, taking advantage of the tandem or cascade reactions via ylide to form multicylic rings compounds in one pot has also gained increasing attention.[9]

Allylic phosphorus ylides have been employed for the construction of cyclohexadiene derivatives via intermolecular ylide-initiated Michael addition/olefination reaction.[10] In our previous studies on ylide chemistry, we reported a tandem intramolecular Michael addition/ylide epoxidation reaction of allylic sulfur salts **1** for the synthesis of the highly functionalized fused cyclohexadiene epoxides **2** with three stereocenters (Scheme 1).[9c] Recently, we developed an intramolecular Michael addition/Wittig reaction for the synthesis of fused cyclohexadiene derivatives **4** when sulfur salts **1** are replaced with the corresponding phosphonium salts **3** (Scheme 2), which are very important subunits in a number of biologically active compounds as well as versatile intermediates in organic synthesis.[11] In this paper, we wish to report this reaction in details.

Scheme 1 Intramolecular Michael addition/ylide epoxidation for the synthesis of cyclohexadiene epoxides



Scheme 2 Intramolecular Michael addition/Wittig reaction for the synthesis of cyclohexadiene derivatives



Results and discussion

The synthesis of substrates **3** is shown in Scheme 3. Phosphonium salts **3** were obtained from the corresponding bromide compounds **5**, by treating with triphenylphosphine in toluene, followed by washing with ether, drying *in vacuo*, which were used directly without any further purification (Scheme 3).

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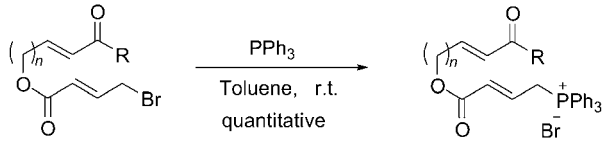
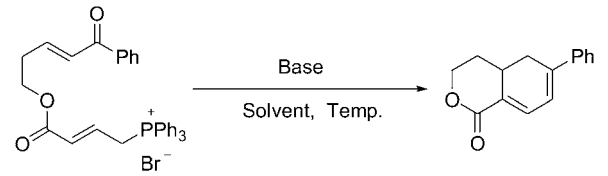
Scheme 3 Synthesis of the phosphonium salts **3**



5 **3**

With phosphonium salts **3** at hands, we firstly treated **3b** with K_2CO_3 in THF at 25 ℃ for 18 h. We were pleased to find that the desired product **4b** was isolated in 29% yield. To improve the yields, the reaction conditions were further optimized. As shown in Table 1, the reaction conditions influenced the yield greatly. The yield was only 29% at room temperature (Entry 1, Table 1), elevating the temperature increased the yield to 80% and 95% respectively (Entries 2 and 3, Table 1). The yield is also base-dependent. Compared with K_2CO_3, Na_2CO_3 was a good base for the reaction (Entry 4, Table 1). But both Cs_2CO_3 and *t*-BuOK decreased the yield obviously (Entries 5 and 6, Table 1). Although this tandem Michael addition/Wittig reaction could proceed in several solvents, THF was still the optimal (Entries 7—10, Table 1).

Table 1 Influence of reaction conditions on the intramolecular tandem reaction[a]



3b **4b**

Entry	Solvent	Base	*t*/℃	Yield[b]/%
1	THF	K_2CO_3	25	29
2	THF	K_2CO_3	50	80
3	THF	K_2CO_3	60	95
4	THF	Na_2CO_3	60	87
5	THF	Cs_2CO_3	60	57
6	THF	*t*-BuOK	60	41
7	CH_3CN	K_2CO_3	60	46
8	DCE	K_2CO_3	60	49
9	Dioxane	K_2CO_3	60	81
10	Toluene	K_2CO_3	60	51

[a] Salts (0.2 mmol), base (0.3 mmol), solvent 3 mL, 25—60 ℃, 18 h; [b] Isolated yield.

The generality of this reaction was investigated under the optimal reaction conditions. As shown in Table 2, this tandem reaction proves to be strongly influenced by the substrate structures and the fused cyclohexadienes were afforded in moderate to excellent yields. For example, the tandem reaction proceeded very well when *n* was 2 (Entries 1—8, Table 2). However, increasing *n* from 2 to 3, the annulation reaction did not work at all (Entry 9, Table 2), probably because of the unfavorable constrained transition state involving seven-membered ring system. Elevated temperature was necessary when *n* was 1, furnishing the desired fused cyclohexadiene **4a** in 55% yield (Entry 1, Table 2). The substituents of ketone moiety also affected the yield of this reaction. For instance, **3b** containing phenyl group (R=Ph) gave the cyclohexadiene **4b** in 95% yield (Entry 2, Table 2). Substrates bearing electron-withdrawing substituents on the phenyl group (R = C_6H_4-*p*-Cl, C_6H_4-*p*-Br) also worked very well, giving the desired products in 92% and 87% yields respectively (Entries 3 and 4, Table 2). However, an introduction of electron-donating substituent on the phenyl group (R=C_6H_4-*p*-Me, C_6H_4-*p*-OMe) decreased yields obviously (Entries 5 and 6, Table 2). We next tried the reaction of aliphatic substrate **3g** and found that the desired tandem process occurred smoothly, affording cyclohexadiene **4g** in 62% yield (Entry 7, Table 2). *α*,*β*-Unsaturated aldehyde **3h** was also suitable for this tandem process and 51% yield was achieved (Entry 8, Table 2).

The present tandem reaction could be accounted by the proposed mechanism as shown in Scheme 4. This reaction is initiated by intramolecular Michael addition of *α*,*β*-unsaturated ketone with ylide **3-A**, generated *in situ* from the corresponding phosphonium salt under basic conditions, to form intermediates **3-B**. Subsequent proton migration leads to phosphonium ylide **3-C**, followed by an intramolecular Wittig reaction affording the desired fused cyclohexadiene derivatives **4** via intermediate **3-INT**. In **3-INT**, both the oxygen anion and the phosphonium cation are located at *cis* position, favoring the *syn*-elimination of triphenyl phosphine oxide. A detailed mechanism waits for further investigation.

Experimental section

All reactions were carried out under N_2 unless otherwise noted. All solvents were purified according to standard methods prior to use. ^{1}H NMR and ^{13}C NMR spectra were recorded in chloroform-d_3 on a VARIAN Mercury 300 M. All IR (Perkin-Elmer 983 or BioRad FTS-185), MSLR and MSHR (HP5989A and Premier CAB088) data were obtained by the analytical center of Shanghai Institute of Organic Chemistry.

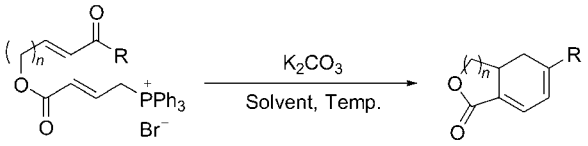
General procedure for synthesis of phosphonium salts 3 (3a as an example)

A mixture of crotonate-derived bromides **5a** (0.618 g, 2.0 mmol) and 1.1 equiv. of triphenylphosphine (0.576 g, 2.2 mmol) in toluene (2 mL) was stirred for 24 h. The white phosphonium salt was collected, washed with ether, and dried under reduced pressure to give 1.2 g of the crude phosphonium salts **3a** in quantitative yield, which was used directly without further purification.

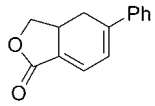
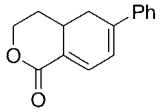
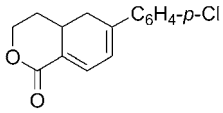
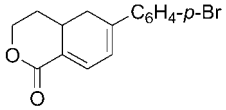
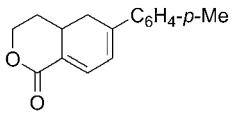
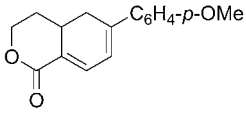
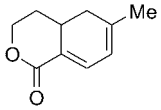
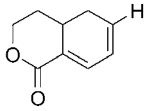
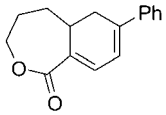
General procedure of the tandem intramolecular reaction (3a as an example)

To a stirred mixture of phosphonium salt **3a** (0.114

Table 2 Scope of the tandem intramolecular Michael addition/Wittig reaction[a]

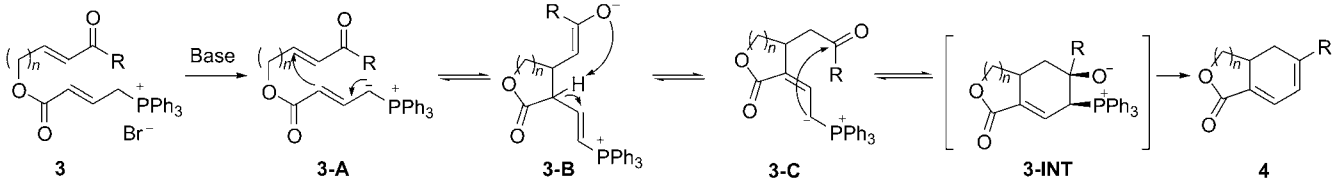


3 **4**

Entry	Substrate	*n*	R	Product	Yield[c]/%
1[b]	**3a**	1	Ph		55
2	**3b**	2	Ph		95
3	**3c**	2	*p*-ClC_6H_4		92
4	**3d**	2	*p*-BrC_6H_4		87
5	**3e**	2	*p*-MeC_6H_4		79
6[b]	**3f**	2	*p*-$MeOC_6H_4$		49
7	**3g**	2	Me		62
8	**3h**	2	H		51
9[b]	**3i**	3	Ph		trace

[a] Salts (0.2 mmol), K_2CO_3 (0.3 mmol) as base, THF as solvent, 60 ℃, 18 h; [b] Toluene as solvent, 100 ℃; [c] Isolated yield.

Scheme 4 A proposed mechanism of the tandem reaction



3 **3-A** **3-B** **3-C** **3-INT** **4**

g, 0.2 mmol) in dry THF (2.5 mL) was added K_2CO_3 (0.042 g, 0.3 mmol) at 60 ℃ for 18 h. After the reaction was complete, the reaction mixture was passed through a glass funnel with a thin layer of silica gel, and

eluted with ethyl acetate. The filtrate was concentrated and the residue was purified by a flash column chromatography to afford the fused cyclohexadiene **4a** as a white solid. Yield 0.024 g (55%).

Characterization data of all new synthesized compounds 4a—4h

5-Phenyl-3a,4-dihydroisobenzofuran-1(3*H*)-one (4a) White solid, 55% yield. ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.50 (d, J=6.6 Hz, 2H), 7.42—7.34 (m, 3H), 7.11—7.08 (m, 1H), 6.64—6.61 (m, 1H), 4.77 (t, J=8.7 Hz, 1H), 4.01 (t, J=8.7 Hz, 2H), 3.36—3.21 (m, 1H), 2.98 (dd, J=16.2, 8.7 Hz, 1H), 2.59—2.46 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 169.4, 142.7, 139.2, 130.2, 128.7, 128.6, 125.6, 124.7, 121.4, 72.7, 33.8, 30.5; IR (KBr) v: 1752 (s), 1662 (w), 1550 (m), 1225 (m), 1038 (m), 734 (m) cm^{-1}; MS (EI) m/z (%): 212 (M^+, 100); HRMS (EI) calcd for $C_{14}H_{12}O_2$ (M^+) 212.0837, found 212.0833.

6-Phenyl-3,4,4a,5-tetrahydroisochromen-1-one (4b) White solid, 95% yield. ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.51—7.48 (m, 2H), 7.41—7.32 (m, 4H), 6.56—6.53 (m, 1H), 4.51—4.47 (m, 1H), 4.29 (t, J=11.4 Hz, 1H), 2.96—2.76 (m, 2H), 2.51—2.39 (m, 1H), 2.12—2.07 (m, 1H), 1.98—1.85 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 165.3, 144.9, 138.9, 136.8, 128.6, 128.5, 125.3, 123.8, 120.7, 67.6, 32.8, 32.7, 29.1; IR (KBr) v: 1687 (s), 1543 (m), 1401 (w), 1269 (m), 766 (m) cm^{-1}; MS (EI) m/z (%): 226 (M^+, 97.92), 154 (100); HRMS (EI) calcd for $C_{15}H_{14}O_2$ (M^+) 226.0994, found 226.0991.

6-(4-Chlorophenyl)-3,4,4a,5-tetrahydroisochromen-1-one (4c) White solid, 92% yield. ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.45—7.32 (m, 5H), 6.55—6.51 (m, 1H), 4.53—4.47 (m, 1H), 4.30 (dt, J=11.4, 2.4 Hz, 1H), 2.97—2.88 (m, 1H), 2.79—2.71 (m, 1H), 2.50—2.38 (m, 1H), 2.14—2.08 (m, 1H), 1.99—1.84 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 165.1, 143.4, 137.4, 136.5, 134.3, 128.7, 126.6, 124.2, 121.0, 67.6, 32.74, 32.71, 29.1; IR (KBr) v: 1684 (s), 1542 (m), 1490 (w), 1268 (w), 815 (m) cm^{-1}; MS (EI) m/z (%): 260 (M^+, 100); HRMS (EI) calcd for $C_{15}H_{13}O_2Cl$ (M^+) 260.0604, found 260.0607.

6-(4-Bromophenyl)-3,4,4a,5-tetrahydroisochromen-1-one (4d) White solid, 87% yield. ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.51—7.48 (m, 2H), 7.41—7.34 (m, 3H), 6.55—6.53 (m, 1H), 4.53—4.47 (m, 1H), 4.30 (t, J=11.4 Hz, 1H), 2.98—2.87 (m, 1H), 2.79—2.70 (m, 1H), 2.51—2.39 (m, 1H), 2.14—2.08 (m, 1H), 1.98—1.85 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 165.1, 143.5, 137.9, 136.5, 131.7, 126.9, 124.3, 122.6, 121.1, 67.6, 32.7, 32.6, 29.1; IR (KBr) v: 1683 (s), 1542 (m), 1488 (w), 1269 (w), 812 (m) cm^{-1}; MS (EI) m/z (%): 304 (M^+, 62.56), 152 (100); HRMS (EI) calcd for $C_{15}H_{13}O_2Br$ (M^+) 304.0099, found 304.0103.

6-*p*-Tolyl-3,4,4a,5-tetrahydroisochromen-1-one (4e) White solid, 79% yield. ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.45—7.39 (m, 3H), 7.20—7.17 (m, 2H), 6.55—6.52 (m, 1H), 4.52—4.47 (m, 1H), 4.30 (t, J=11.4 Hz, 1H), 2.95—2.75 (m, 2H), 2.50—2.42 (m, 1H), 2.37 (s, 3H), 2.14—2.07 (m, 1H), 1.99—1.85 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 165.4, 144.9, 138.8, 137.1, 136.1, 129.3, 125.3, 123.4, 119.9, 67.6, 32.87, 32.84, 29.2, 21.1; IR (KBr) v: 1688 (s), 1543 (m), 1268 (m), 810 (m), 747 (w) cm^{-1}; MS (EI) m/z (%): 240 (M^+, 100); HRMS (EI) calcd for $C_{16}H_{16}O_2$ (M^+) 240.1150, found 240.1145.

6-(4-Methoxyphenyl)-3,4,4a,5-tetrahydroisochromen-1-one (4f) White solid, 49% yield. ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.49—7.42 (m, 3H), 6.93—6.89 (m, 2H), 6.51—6.48 (m, 1H), 4.52—4.48 (m, 1H), 4.30 (t, J=11.7 Hz, 1H), 3.84 (s, 3H), 2.97—2.78 (m, 2H), 2.48—2.35 (m, 1H), 2.14—2.07 (m, 1H), 1.99—1.86 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 165.5, 160.0, 144.6, 137.3, 131.3, 126.8, 122.8, 119.0, 113.9, 67.6, 55.3, 32.89, 32.84, 29.3; IR (KBr) v: 1685 (s), 1544 (m), 1270 (w), 814 (w), 748 (w) cm^{-1}; MS (EI) m/z (%): 256 (M^+, 100); HRMS (EI) calcd for $C_{16}H_{16}O_3$ (M^+) 256.1099, found 256.1098.

6-Methyl-3,4,4a,5-tetrahydroisochromen-1-one (4g) White solid, 62% yield. ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.26—7.23 (m, 1H), 5.95—5.92 (brs, 1H), 4.48—4.42 (m, 1H), 4.25 (dt, J=11.7, 2.1 Hz, 1H), 2.85—2.75 (m, 1H), 2.23—1.97 (m, 3H), 1.92 (s, 3H), 1.89—1.75 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 165.6, 146.3, 137.2, 122.0, 120.4, 67.6, 35.6, 32.5, 29.2, 23.6; IR (KBr) v: 1719 (s), 1401 (w), 1258 (m), 1097 (w), 1031 (w) cm^{-1}; MS (EI) m/z (%): 164 (M^+, 38.75), 91 (100); HRMS (EI) calcd for $C_{10}H_{12}O_2$ (M^+) 164.0837, found 164.0836.

3,4,4a,5-Tetrahydroisochromen-1-one (4h) White solid, 51% yield. ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.29—7.26 (m, 1H), 6.28—6.15 (m, 2H), 4.50—4.43 (m, 1H), 4.27 (dt, J=11.7, 2.1 Hz, 1H), 2.85—2.74 (m, 1H), 2.41—2.30 (m, 1H), 2.06—1.95 (m, 2H), 1.89—1.77 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 165.4, 135.9, 134.4, 125.1, 67.6, 32.1, 29.6, 29.1; IR (KBr) v: 1715 (s), 1612 (m), 1247 (m), 1094 (m), 758 (m) cm^{-1}; MS (EI) m/z (%): 150 (M^+, 26.55), 91 (100); HRMS (EI) calcd for $C_9H_{10}O_2$ (M^+) 150.0681, found 150.0679.

Conclusion

In summary, a tandem intramolecular Michael addition/Wittig reaction has been developed for the synthesis of fused cyclohexadiene derivatives, which were important subunits in a number of biologically active compounds, as well as versatile intermediates in organic synthesis. The readily accessible starting material, mild reaction conditions and reasonable yields made the present reaction potentially useful in organic synthesis. Further studies on the mechanism are under investigation in our laboratory.

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