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Supporting Information

Lewis acid catalyzed [2 + 2] cycloaddition of ynamides and

propargyl silyl ethers: synthesis of alkylidenecyclobutenones and

their reactivity in ring-opening and ring expansion

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General experimental procedures

General: ¹H NMR (400 MHz) and ¹³C NMR (100 MHz) spectra were recorded on a Bruker Avance (400 MHz) spectrometer, using CDCl₃ as the solvent and TMS as internal standard; chemical shifts were quoted in parts per million and *J* values were given in hertz. High resolution mass spectrometry (HRMS) was performed on a Waters Micromass GCT instrument. Melting points were uncorrected. All solvents were dried according to standard procedures. Ynamides 2^1 and propargylic silyl ether

- $\mathbf{1}^2$ were prepared by reported methods.
- 1. X. Zhang, Y. Zhang, J. Huang, R.-P. Hsung, K. C. M. Kurtz, J. Oppenheimer, M. E.
- Petersen, I. K. Sagamanova, L. Shen and M. R. Tracey, J. Org. Chem., 2006, 71, 4170.
- 2. T. Ishikawa, S. Manabe, T. Aikawa, T. Kudo and S. Saito, Org. Lett., 2004, 6, 2361.

Typical procedure for the synthesis of alkylidenecyclobutenones 3



4-(diphenylmethylene)-2,3-diphenylcyclobut-2-enone 3a

A vial was charged with propargyl silyl ether **1a** (106.9 mg, 0.3 mmol) and ynamide **2a** (56.2 mg, 0.3 mmol) and evacuated under high vacuum and backfilled with N₂. CH₂Cl₂ (3 mL) and BF₃·OEt₂ (8.5 mg, 0.06 mmol) were next added and the solution was stirred at rt. Upon reaction completion (7 h, TLC, eluent: hexane-EtOAc, 15:1), the mixture was filtered over a plug of silica gel (washed with 50 mL EtOAc), and the filtrate was concentrated. The residue was purified by flash chromatography on silica gel (eluent: hexane/ethyl acetate = 15:1) to afford **3a** (75.0 mg, 65%) as a yellow solid, mp 181-183 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.75-7.77 (m, 2H), 7.21-7.41 (m, 9H), 7.02-7.13 (m, 5H), 6.93-6.97 (m, 2H), 6.89 (d, *J* = 7.2 Hz, 2H); ¹³C NMR (125 MHz, CDCl₃): δ 187.6, 176.0, 154.4, 147.3, 138.6, 138.1, 131.9, 130.64, 130.60, 130.56, 129.8, 129.6, 129.4, 128.7, 128.0, 127.9, 127.7, 127.5, 127.42, 127.37, 127.1. HRMS (ESI-TOF) m/z: [M + H]⁺ Calcd for C₂₉H₂₁O, 385.1587; found 385.1595.



4-(diphenylmethylene)-2-(4-fluorophenyl)-3-phenylcyclobut-2-enone 3b

The mobile phase for flash chromatography: hexane/ethyl acetate = 15:1. Yellow solid (142.8 mg, 71%), mp 180-182 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.74-7.78 (m, 2H), 7.31-7.39 (m, 5H), 7.20-7.21 (m, 1H), 6.92-7.10 (m, 9H), 6.87 (d, *J* = 7.2 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 187.4, 175.5 (d, *J* = 2.1 Hz), 163.3 (d, *J* = 250.2 Hz), 153.2, 147.2, 138.6, 138.1, 131.8, 130.8, 130.6, 130.5, 129.5, 129.4, 128.1, 127.7, 127.5, 127.4, 127.0, 126.1 (d, *J* = 3.4 Hz), 116.0 (d, *J* = 21.7 Hz). HRMS (ESI-TOF) m/z: [M + H]⁺ Calcd for C₂₉H₂₀FO, 403.1493; found 403.1491.



2-(4-chlorophenyl)-4-(diphenylmethylene)-3-phenylcyclobut-2-enone 3c

The mobile phase for flash chromatography: hexane/ethyl acetate = 15:1. Yellow solid (152.6 mg, 73%), mp 202-204 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.67-7.69 (m, 2H), 7.21-7.39 (m, 8H), 6.92-7.12 (m, 7H), 6.87 (d, *J* = 7.6 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 187.2, 176.3, 152.9, 147.1, 138.5, 138.0, 135.5, 131.7, 131.3, 130.6, 129.6, 129.0, 128.6, 128.2, 128.14, 128.07, 127.7, 127.6, 127.4, 127.0. HRMS (ESI-TOF) m/z: [M + H]⁺ Calcd for C₂₉H₂₀ClO, 419.1197; found 419.1187.



2-(4-bromophenyl)-4-(diphenylmethylene)-3-phenylcyclobut-2-enone 3d

The mobile phase for flash chromatography: hexane/ethyl acetate = 15:1. Yellow solid (155.2 mg, 67%), mp 209-211 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.60-7.62 (m, 2H), 7.32-7.43 (m, 7H), 7.22-7.25 (m, 1H), 7.07-7.13 (m, 3H), 6.93-7.01 (m, 4H), 6.88 (d, J = 8.4 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 187.1, 176.4, 153.0, 147.1, 138.5,

138.0, 132.0, 131.7, 131.4, 130.5, 129.6, 128.8, 128.6, 128.1, 128.0, 127.7, 127.5, 127.4, 127.3, 127.0, 123.9. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for C₂₉H₂₀BrO, 463.0692; found 463.0676.



4-(diphenylmethylene)-3-(4-methoxyphenyl)-2-*p*-tolylcyclobut-2-enone 3e

The mobile phase for flash chromatography: hexane/ethyl acetate = 15:1. Yellow solid (126.4 mg, 59%), mp 145-147 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.68 (d, *J* = 7.6 Hz, 2H), 7.30-7.38 (m, 5H), 7.09-7.12 (m, 3H), 6.90-7.02 (m, 6H), 6.60 (d, *J* = 8.4 Hz, 2H), 3.77 (s, 3H), 2.33 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 187.8, 174.6, 160.6, 153.6, 147.5, 139.7, 138.9, 138.6, 130.8, 130.6, 129.8, 129.4, 129.3, 127.8, 127.6, 127.4, 127.3, 127.2, 124.1, 113.3, 55.4, 21.6. HRMS (ESI-TOF) m/z: [M + H]⁺ Calcd for C₃₁H₂₅O₂, 429.1849; found 429.1842.



4-(diphenylmethylene)-2,3-bis(4-methoxyphenyl)cyclobut-2-enone 3f

The mobile phase for flash chromatography: hexane/ethyl acetate = 15:1. Red solid (134.0 mg, 60%), mp 178-180 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.76 (d, *J* = 8.0 Hz, 2H), 7.27-7.37 (m, 5H), 7.07-7.09 (m, 1H), 6.88-7.00 (m, 6H), 6.82 (d, *J* = 8.8 Hz, 2H), 6.60 (d, *J* = 8.4 Hz, 2H), 3.75 (s, 3H), 3.74 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 187.9, 173.4, 160.6, 160.5, 153.4, 147.6, 139.0, 138.6, 130.8, 130.6, 129.3, 129.0, 127.7, 127.6, 127.4, 127.3, 124.3, 122.8, 114.2, 113.3, 55.4, 55.3. HRMS (ESI-TOF) m/z: [M + H]⁺ Calcd for C₃₁H₂₅O₃, 445.1798; found 445.1788.



4-(diphenylmethylene)-3-(4-methoxyphenyl)-2-o-tolylcyclobut-2-enone 3g

The mobile phase for flash chromatography: hexane/ethyl acetate = 15:1. Yellow solid (162.8 mg, 76%), mp 159-161 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.30-7.38 (m, 5H), 7.19-7.21 (m, 4H), 7.07-7.10 (m, 3H), 7.00-7.02 (m, 2H), 6.78-6.80 (m, 2H), 6.49-6.52 (m, 2H), 3.70 (s, 3H), 2.23 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 186.9, 176.4, 161.2, 156.8, 146.4, 139.3, 138.9, 136.9, 131.0, 130.8, 130.7, 130.5, 130.0, 129.3, 128.9, 128.8, 128.0, 127.75, 127.72, 127.6, 125.6, 123.5, 113.2, 55.3, 20.9. HRMS (ESI-TOF) m/z: [M + H]⁺ Calcd for C₃₁H₂₅O₂, 429.1849; found 429.1853.



(*E*)-4-(diphenylmethylene)-3-(4-methoxyphenyl)-2-styrylcyclobut-2-enone 3h The mobile phase for flash chromatography: hexane/ethyl acetate = 15:1. Yellow solid (76.6 mg, 35%), mp 150-152 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.62 (d, *J* = 16.0 Hz, 1H), 7.39-7.41 (m, 2H), 7.15-7.31 (m, 8H), 7.06-7.12 (m, 1H), 6.85-7.02 (m, 7H), 6.58 (d, *J* = 8.4 Hz, 2H), 3.70 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 187.5, 173.0, 161.0, 152.5, 147.0, 139.2, 138.9, 138.0, 136.8, 130.9, 130.8, 130.3, 130.2, 128.84, 128.79, 127.9, 127.7, 127.5, 127.1, 123.9, 115.5, 113.4, 55.4. HRMS (ESI-TOF) m/z: [M + H]⁺ Calcd for C₃₂H₂₅O₂, 441.1849; found 441.1834.



4-(diphenylmethylene)-3-(4-methoxyphenyl)-2-phenylcyclobut-2-enone 3i

The mobile phase for flash chromatography: hexane/ethyl acetate = 15:1. Yellow solid (78.5 mg, 63%), mp 160-162 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.65 (d, *J* = 6.8 Hz, 2H), 7.13-7.26 (m, 8H), 6.96-6.98 (m, 1H), 6.77-6.89 (m, 6H), 6.47 (d, *J* = 8.4 Hz, 2H), 3.61 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 187.6, 175.6, 160.8, 153.5, 147.5, 138.9, 138.5, 130.8, 130.7, 130.5, 130.1, 129.4, 128.7, 127.9, 127.7, 127.54, 127.51, 127.4, 124.0, 113.4, 55.4. HRMS (ESI-TOF) m/z: [M + H]⁺ Calcd for C₃₀H₂₃NO₂, 415.1693; found 415.1699.



4-(diphenylmethylene)-2-phenyl-3-p-tolylcyclobut-2-enone 3j

The mobile phase for flash chromatography: hexane/ethyl acetate = 15:1. Yellow solid (129.5 mg, 65%), mp 184-186 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.76-7.78 (m, 2H), 7.27-7.39 (m, 8H), 7.07-7.08 (m, 1H), 6.88-6.98 (m, 8H), 2.30 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 187.7, 176.1, 154.0, 147.4, 139.9, 138.8, 138.4, 130.7, 130.6, 130.5, 129.9, 129.5, 128.9, 128.7, 128.6, 127.9, 127.6, 127.5, 127.4, 127.3, 21.6. HRMS (ESI-TOF) m/z: [M + H]⁺ Calcd for C₃₀H₂₃O, 399.1743; found 399.1748.



3-(4-chlorophenyl)-4-(diphenylmethylene)-2-phenylcyclobut-2-enone 3k

The mobile phase for flash chromatography: hexane/ethyl acetate = 15:1. Yellow solid (115.2 mg, 55%), mp 188-190 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.70-7.72 (m, 2H), 7.30-7.40 (m, 8H), 7.07-7.14 (m, 3H), 6.90-7.02 (m, 4H), 6.89 (d, *J* = 8.0 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 187.2, 174.3, 154.6, 147.1, 138.4, 138.1, 135.4, 130.9, 130.6, 130.5, 130.3, 129.8, 129.5, 128.8, 128.5, 128.3, 128.1, 127.74, 127.68, 127.5, 127.4. HRMS (ESI-TOF) m/z: [M + H]⁺ Calcd for C₂₉H₂₀ClO, 419.1197; found 419.1198.



4-(diphenylmethylene)-2-phenyl-3-(thiophen-2-yl)cyclobut-2-enone 31

The mobile phase for flash chromatography: hexane/ethyl acetate = 15:1. Yellow solid (56.4 mg, 48%), mp 172-174 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.88-7.90 (m, 2H), 7.31-7.39 (m, 9H), 7.21-7.23 (m, 1H), 7.12-7.16 (m, 2H), 7.07 (d, *J* = 7.6 Hz, 2H), 6.77-6.80 (m, 1H), 6.62 (d, *J* = 7.6 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 186.8, 166.8, 153.3, 146.6, 138.9, 138.8, 132.1, 131.6, 130.9, 130.8, 130.7, 130.4, 129.7, 129.5, 128.6, 128.1, 127.9, 127.8, 127.7, 127.6, 127.2. HRMS (ESI-TOF) m/z: [M + H]⁺ Calcd for C₂₇H₁₉OS, 391.1151; found 391.1146.



(*E*)-4-(diphenylmethylene)-2-phenyl-3-styrylcyclobut-2-enone 3m

The mobile phase for flash chromatography: hexane/ethyl acetate = 15:1. Yellow solid (99.1 mg, 48%), mp 140-142 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.89 (d, *J* = 8.0 Hz, 2H), 7.21-7.44 (m, 16H), 7.05-7.09 (m, 3H), 6.19 (d, *J* = 16.4 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 187.3, 170.8, 153.5, 146.8, 143.0, 139.9, 138.8, 135.4, 131.2, 130.7, 130.6, 130.4, 129.8, 129.4, 128.9, 128.8, 128.1, 128.0, 127.9, 127.8, 127.6, 117.0. HRMS (ESI-TOF) m/z: [M + H]⁺ Calcd for C₃₁H₂₃O, 411.1743; found 411.1747.



3-butyl-4-(diphenylmethylene)-2-phenylcyclobut-2-enone 3n

The mobile phase for flash chromatography: hexane/ethyl acetate = 15:1. Colorless oil (40.3 mg, 37%). ¹H NMR (400 MHz, CDCl₃): δ 7.76 (d, *J* = 7.2 Hz, 2H), 7.36-7.43 (m, 7H), 7.24-7.32 (m, 6H), 2.46 (t, *J* = 8.0 Hz, 2H), 1.19-1.22 (m, 2H), 1.05-1.08 (m, 2H), 0.69 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 187.8, 183.3, 155.1, 148.8, 139.3, 138.6, 130.4, 130.3, 130.0, 129.8, 129.1, 128.9, 128.1, 127.9, 127.7, 127.3, 29.0, 28.5, 22.9, 13.6. HRMS (ESI-TOF) m/z: [M + H]⁺ Calcd for C₂₇H₂₅O, 365.1900; found 365.1897.



4-(dip-tolylmethylene)-2,3-diphenylcyclobut-2-enone 30

The mobile phase for flash chromatography: hexane/ethyl acetate = 15:1. Yellow solid (127.9 mg, 62%), mp 162-164 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.74-7.76 (m, 2H), 7.02-7.29 (m, 12H), 6.74-6.76 (m, 4H), 2.37 (s, 3H), 2.22 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 188.0, 176.2, 153.5, 146.5, 137.9, 137.2, 136.0, 135.4, 132.1, 130.7, 130.5, 129.9, 129.4, 129.2, 128.6, 128.4, 127.9, 127.8, 127.4, 127.2, 21.4, 21.1. HRMS (ESI-TOF) m/z: [M + H]⁺ Calcd for C₃₁H₂₅O, 413.1900; found 413.1896.



4-(bis(4-methoxyphenyl)methylene)-2,3-diphenylcyclobut-2-enone 3p

The mobile phase for flash chromatography: hexane/ethyl acetate = 15:1. Red solid (151.1 mg, 68%), mp 171-173 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.74-7.76 (m, 2H), 7.21-7.35 (m, 6H), 7.11-7.15 (m, 2H), 7.04 (d, *J* = 7.6 Hz, 2H), 6.87 (d, *J* = 8.4 Hz, 2H), 6.79 (d, *J* = 8.4 Hz, 2H), 6.47 (d, *J* = 8.4 Hz, 2H), 3.81 (s, 3H), 3.67 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 188.3, 176.3, 159.6, 159.2, 152.7, 145.4, 132.2, 131.9, 131.9, 131.5, 130.8, 130.0, 129.3, 128.7, 127.9, 127.3, 127.2, 113.2, 112.8, 55.3. HRMS (ESI-TOF) m/z: [M + H]⁺ Calcd for C₃₁H₂₅O₃, 445.1798; found 445.1813.



4-(bis(4-chlorophenyl)methylene)-2,3-diphenylcyclobut-2-enone 3q

The mobile phase for flash chromatography: hexane/ethyl acetate = 15:1. Yellow solid (117.9 mg, 52%), mp 190-192 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.74-7.76 (m, 2H),

7.29-7.32 (m, 8H), 7.16-7.20 (m, 2H), 7.03 (d, J = 7.2 Hz, 2H), 6.92 (d, J = 8.4 Hz, 2H), 6.79 (d, J = 8.4 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 186.9, 175.5, 155.4, 148.5, 136.6, 136.2, 134.1, 133.7, 131.73, 131.67, 130.0, 129.7, 129.5, 128.8, 128.2, 128.0, 127.8, 127.6, 127.5, 127.0. HRMS (ESI-TOF) m/z: [M + H]⁺ Calcd for C₂₉H₁₉Cl₂O, 453.0807; found 453.0804.

Typical procedure for the synthesis of alkylidenecyclobutenols 4



4-(diphenylmethylene)-1-methyl-2,3-diphenylcyclobut-2-enol 4a

A vial was charged with **3a** (115.4 mg, 0.3 mmol) and evacuated under high vacuum and backfilled with N₂. THF (3 mL) was next added to dissolve **3a**. Then MeMgBr (0.45 mmol, 0.45 mL, 1 M in THF) was added and the solution was stirred at rt for 10 min. The resulting mixture was then quenched by saturated aqueous solution of NH₄Cl (20 mL). After extraction with diethyl ether (30 mL), the organic layer was washed with brine, dried over anhydrous Na₂SO₄, filtered and concentrated. The residue was purified by flash chromatography on silica gel (eluent: hexane/ethyl acetate = 9:1) to afford **4a** (95.8 mg, 80%) as a white solid, mp 140-142 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.48-7.57 (m, 4H), 7.20-7.32 (m, 6H), 6.94-7.08 (m, 6H), 6.84-6.86 (m, 4H), 2.42 (s, 1H), 1.69 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 151.9, 149.9, 144.7, 140.9, 139.6, 133.8, 131.9, 130.4, 129.9, 128.6, 128.5, 128.1, 127.9, 127.8, 127.4, 127.3, 127.1, 126.9, 126.3, 81.5, 23.0. HRMS (ESI-TOF) m/z: [M + Na]⁺ Calcd for C₃₀H₂₄NaO, 423.1719; found 423.1717.



1-butyl-4-(diphenylmethylene)-2,3-diphenylcyclobut-2-enol 4b

The mobile phase for flash chromatography: hexane/ethyl acetate = 9:1. White solid (99.6 mg, 75%), mp 169-171 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.43-7.53 (m, 4H), 7.22-7.32 (m, 6H), 6.90-7.15 (m, 6H), 6.85-6.87 (m, 4H), 2.38 (s, 1H), 1.99-2.05 (m, 1H), 1.75-1.82 (m, 1H), 1.23-1.43 (m, 4H), 0.76 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 150.1, 145.9, 144.9, 141.1, 139.6, 133.7, 132.3, 130.2, 129.6, 128.5, 128.4, 128.0, 127.9, 127.8, 127.3, 127.2, 127.1, 126.9, 126.3, 84.8, 34.6, 27.2, 22.8, 13.9. HRMS (ESI-TOF) m/z: [M + Na]⁺ Calcd for C₃₃H₃₀NaO, 465.2189; found 465.2181.



1-benzyl-4-(diphenylmethylene)-2,3-diphenylcyclobut-2-enol 4c

The mobile phase for flash chromatography: hexane/ethyl acetate = 9:1. Colorless oil (109.0 mg, 76%). ¹H NMR (400 MHz, CDCl₃): δ 7.52-7.66 (m, 4H), 7.10-7.38 (m, 9H), 6.84-6.96 (m, 6H), 6.71-6.75 (m, 2H), 6.50 (d, *J* = 7.2 Hz, 2H), 6.42 (d, *J* = 7.2 Hz, 2H), 3.46 (d, *J* = 13.2 Hz, 1H), 3.15 (d, *J* = 13.2 Hz, 1H), 2.83 (s, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 149.1, 147.1, 144.5, 141.1, 139.4, 137.2, 133.8, 132.6, 130.5, 130.3, 129.9, 128.6, 128.5, 128.4, 128.2, 127.6, 127.5, 127.33, 127.29, 127.0, 126.89, 126.87, 126.4, 126.2, 85.0, 47.8. HRMS (ESI-TOF) m/z: [M + Na]⁺ Calcd for C₃₆H₂₈NaO, 499.2032; found 499.2028.

Typical procedure for the synthesis of 4,5-dien-2-ones 5



1,2,3,5,5-pentaphenylpenta-3,4-dien-1-one 5a

A vial was charged with **3a** (115.4 mg, 0.3 mmol) and evacuated under high vacuum and backfilled with N₂. THF (3 mL) was next added to dissolve **3a**. Then PhMgBr (0.45 mmol, 0.45 mL, 1 M in THF) was added and the solution was stirred at rt for 10 min. The resulting mixture was then quenched by saturated aqueous solution of NH₄Cl (20 mL). After extraction with diethyl ether (30 mL), the organic layer was washed with brine, dried over anhydrous Na₂SO₄, filtered and concentrated. The residue was purified by flash chromatography on silica gel (eluent: hexane/ethyl acetate = 9:1) to afford **5a** (108.1 mg, 78%) as a colorless oil. ¹H NMR (400 MHz, CDCl₃): δ 7.96 (d, *J* = 7.6 Hz, 2H), 7.40-7.45 (m, 3H), 7.15-7.33 (m, 18H), 7.03-7.05 (m, 2H), 6.00 (s, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 209.3, 196.5, 137.1, 136.4, 136.1, 135.5, 135.3, 132.9, 129.6, 128.8, 128.7, 128.6, 128.3, 128.2, 127.9, 127.53, 127.46, 127.37, 127.27, 126.2, 115.3, 109.2, 55.8. HRMS (ESI-TOF) m/z: [M + H]⁺ Calcd for C₃₅H₂₇O, 463.2056; found 463.2047.



1-(3-methoxyphenyl)-2,3,5,5-tetraphenylpenta-3,4-dien-1-one 5b

The mobile phase for flash chromatography: hexane/ethyl acetate = 9:1. Colorless oil (103.3 mg, 70%). ¹H NMR (400 MHz, CDCl₃): δ 7.49 (d, *J* = 7.6 Hz, 1H), 7.34-7.36 (m, 3H), 7.07-7.24 (m, 17H), 6.93-6.96 (m, 2H), 6.83-6.86 (m, 1H), 5.92 (s, 1H), 3.57 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 209.5, 196.4, 159.8, 137.7, 137.2, 136.1, 135.5, 135.3, 129.6, 128.9, 128.8, 128.7, 128.4, 128.3, 127.9, 127.64, 127.56, 127.47, 127.38, 126.2, 121.3, 119.6, 115.4, 113.1, 109.3, 55.9, 55.3. HRMS (ESI-TOF) m/z: [M + Na]⁺ Calcd for C₃₆H₂₈NaO₂, 515.1982; found 515.1987.

Procedure for the synthesis of 3,4,6,6-tetraphenylhexa-4,5-dien-2-one 5c



A vial was charged with 4a (120.1 mg, 0.3 mmol) and evacuated under high vacuum and backfilled with N₂. Toluene (3 mL) was next added to dissolve 4a. Then LDA (0.9 mmol, 0.45 mL, 2 M in THF) was added and the solution was stirred at rt for 20 min. The resulting mixture was then quenched by saturated aqueous solution of NH₄Cl (20 mL). After extraction with diethyl ether (30 mL), the organic layer was washed with brine, dried over anhydrous Na₂SO₄, filtered and concentrated. The residue was purified by flash chromatography on silica gel (eluent: hexane/ethyl acetate = 9:1) to afford **5c** (102.1 mg, 85%) as a colorless oil. ¹H NMR (400 MHz, CDCl₃): δ 7.25-7.42 (m, 18H), 7.04-7.07 (m, 2H), 5.12 (s, 1H), 2.14 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 208.1, 205.4, 136.7, 136.0, 135.8, 135.3, 129.3, 128.8, 128.7, 128.6, 128.56, 128.54, 128.4, 128.1, 127.7, 127.6, 127.4, 126.2, 115.1, 108.1, 61.3, 29.4. HRMS (ESI-TOF) m/z: [M + Na]⁺ Calcd for C₃₀H₂₄NaO, 423.1719; found 423.1742. **Procedure for the synthesis of 4-(diphenylmethylene)-2,3-diphenylcyclohex-2-**





A vial was charged with **3a** (115.4 mg, 0.3 mmol) and evacuated under high vacuum and backfilled with N₂. THF (3 mL) was next added to dissolve **3a**. Then C₂H₃MgCl (0.45 mmol, 0.45 mL, 1 M in THF) was added and the solution was stirred at rt for 10 min. The resulting mixture was then quenched by saturated aqueous solution of NH₄Cl (20 mL). After extraction with diethyl ether (30 mL), the organic layer was washed with brine, dried over anhydrous Na₂SO₄, filtered and concentrated. The residue was purified by flash chromatography on silica gel (eluent: hexane/ethyl acetate = 9:1) to afford **6** (69.3 mg, 56%) as a colorless solid, mp 176-178 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.07-7.33 (m, 8H), 6.84-6.95 (m, 5H), 6.64-6.75 (m, 7H), 3.11 (t, *J* = 6.4 Hz, 2H), 2.73 (t, *J* = 6.4 Hz, 2H),; ¹³C NMR (100 MHz, CDCl₃): δ 197.6, 157.2, 147.5, 142.7, 142.5, 139.8, 137.9, 137.1, 135.1, 130.9, 130.7, 130.2, 128.2, 127.8, 127.4, 127.3, 127.0, 126.7, 126.6, 126.5, 39.7, 34.6. HRMS (ESI-TOF) m/z: [M + Na]⁺ Calcd for C₃₁H₂₄NaO, 435.1719; found 435.1752. Typical procedure for the synthesis of imines 7



(Z)-3-((isobutylimino)(phenyl)methyl)-1,4,4-triphenylbut-3-en-2-one 7a

A vial was charged with **3a** (115.4 mg, 0.3 mmol) and evacuated under high vacuum and backfilled with N₂. 1,4-Dioxane (3 mL) was next added to dissolve **3a**. Then *i*-BuNH₂ (32.8 mg, 0.45 mmol) was added and the solution was stirred at 80 °C for 24 h. The resulting mixture was then concentrated. The residue was purified by flash chromatography on silica gel (eluent: hexane/ethyl acetate = 9:1) to afford **7a** (101.6 mg, 74%) as a colorless solid, mp 135-137 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.82 (d, *J* = 7.6 Hz, 2H), 7.08-7.44 (m, 16H), 6.76-6.78 (m, 2H), 3.05 (s, 2H), 3.31 (dd, *J*₁ = 6.8 Hz, *J*₂ = 13.6 Hz, 1H), 2.89 (dd, *J*₁ = 6.8 Hz, *J*₂ = 13.6 Hz, 1H), 1.85-1.89 (m, 1H), 0.92 (d, *J* = 6.8 Hz, 3H), 0.88 (d, *J* = 6.8 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 201.5, 163.4, 150.9, 140.5, 140.2, 139.5, 137.6, 133.9, 130.3, 129.8, 129.4, 129.3, 129.1, 128.9, 128.4, 128.16, 128.15, 128.0, 126.8, 61.6, 49.0, 29.9, 21.0, 20.9. HRMS (ESI-TOF) m/z: [M + H]⁺ Calcd for C₃₃H₃₂NO, 458.2478; found 458.2471.



(Z)-1,4,4-triphenyl-3-(phenyl(propylimino)methyl)but-3-en-2-one 7b

The mobile phase for flash chromatography: hexane/ethyl acetate = 9:1. Colorless oil (110.4 mg, 83%). ¹H NMR (400 MHz, CDCl₃): δ 7.81-7.83 (m, 2H), 7.08-7.40 (m, 16H), 6.78-6.79 (m, 2H), 3.47-3.51 (m, 3H), 3.09-3.12 (m, 1H), 1.46-1.62 (m, 2H), 0.90 (t, *J* = 8.0 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 201.4, 163.4, 150.8, 140.5, 140.2, 139.3, 137.5, 133.8, 130.2, 129.85, 129.79, 129.4, 129.2, 129.1, 128.9, 128.4, 128.1, 128.0, 126.8, 55.9, 49.0, 23.9, 12.3. HRMS (ESI-TOF) m/z: [M + H]⁺ Calcd for C₃₂H₃₀NO, 444.2322; found 444.2358.



(Z)-3-((benzylimino)(phenyl)methyl)-1,4,4-triphenylbut-3-en-2-one 7c

The mobile phase for flash chromatography: hexane/ethyl acetate = 9:1. Colorless oil (100.3 mg, 68%). ¹H NMR (400 MHz, CDCl₃): δ 7.89 (d, *J* = 6.4 Hz, 2H), 7.12-7.45 (m, 21H), 6.76-6.77 (m, 2H), 4.78 (d, *J* =16.0 Hz, 1H), 4.25 (d, *J* =16.0 Hz, 1H), 3.47-3.51 (m, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 201.4, 164.3, 151.5, 140.4, 140.1, 140.0, 139.1, 137.1, 133.7, 130.3, 130.2, 129.8, 129.6, 129.3, 129.0, 128.4, 128.32, 128.28, 128.27, 128.21, 128.0, 126.9, 126.6, 57.6, 49.1. HRMS (ESI-TOF) m/z: [M + H]⁺ Calcd for C₃₆H₃₀NO, 492.2322; found 492.2336.

Procedure for the synthesis of 5-benzhydryl-3,4-diphenylfuran-2(5H)-one 8



A solution of **3a** (115.4 mg, 0.3 mmol) and 1,4-diaza-bicyclo[2.2.2]octane (33.7 mg, 0.3 mmol) in wet THF (5 mL) was irradiated using high pressure Hg lamp at rt for 3 d. The residue after removal of solvent was subjected to column chromatography on silica gel (hexane–EtOAc = 9:1) to give **8** (61.6 mg, 51%) as a colorless solid, mp 206-208 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.47 (d, *J* = 7.6 Hz, 2H), 7.09-7.36 (m, 14H), 6.82-6.91 (m, 4H), 6.11 (s, 1H), 4.21 (s, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 172.3, 158.8, 141.3, 136.6, 131.1, 130.4, 129.94, 129.88, 129.1, 129.0, 128.7, 128.63, 128.58, 128.52, 128.0, 127.4, 127.0, 82.3, 52.9. HRMS (ESI-TOF) m/z: [M + Na]⁺ Calcd for C₂₉H₂₂NaO₂, 425.1512; found 425.1519.



NMR spectra of new compounds





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-CJ-461 CPD CDC13														190
XLW- C13(- 8



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-6. 0E+08	-5. 5E+08	-5. 0E+08	-4. 5E+08	-4. 0E+08	-3. 5E+08	-3. 0E+08	-2. 5E +08	-2. 0E+08	-1. 5E+08	- -1. 0E+08	-5. 0E+07	-0. 0E+00	5. 0E+07	
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- 5. 5E+08	-5. 0E+08	- -4. 5E+08	- -4. 0E+08	- 3. 5E +08	-3. 0E+08	- -2.5E+08	- -2. 0E+08	- -1.5E+08	- -1. 0E+08	-5. 0E+07	-0. 0E+00	5. 0E+07	
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¹⁸O-Labeling Experiments

¹⁸O-labelled **1a** was prepared from dichlorodiphenylmethane and $H_2^{18}O$ (90% ¹⁸O).^[1]



[1] (a) J. M. Risley and R. L. Van Etten, J. Am. Chem. Soc., 1980, 102, 4609; (b) H. Zheng, M. Lejkowski and D. G. Hall, Chem. Sci., 2011, 2, 1305.

Mass Analysis of 1a:

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Mass Analysis of **1a** (¹⁸O labelled): 丰度







Mass Analysis of 3a:



Mass Analysis of **3a** (¹⁸O labelled):

丰度



D-Labeling Experiments

D-Labelled 8 was prepared according to the procedure for the synthesis of 5-benzhydryl-3,4-diphenylfuran-2(5H)-one **8** except that 0.1 mL D₂O (99% D) was added.

¹H NMR of **D-Labelled 8:**



¹H NMR of 8:



Crystal Structure of 3a.

This crystal was deposited in the Cambridge Crystallographic Data Centre and assigned as CCDC 1446541. Further information can be found in the CIF file.



Crystal Structure of 7a.

This crystal was deposited in the Cambridge Crystallographic Data Centre and assigned as CCDC 1446542. Further information can be found in the CIF file.



Crystal Structure of 8.

This crystal was deposited in the Cambridge Crystallographic Data Centre and assigned as CCDC 1446543. Further information can be found in the CIF file.

