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Supporting Information

Synthesis of 3(2H)-Furanone Derivatives: p-TsOH/Halotrimethylsilane

Promoted Cycloketonization of γ-Hydroxyl Ynones

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General Remarks

Column chromatography was carried out on silica gel (200-300 mesh). ^1H NMR spectra were recorded on 400, 500 or 600 MHz in CDCl3 or d_6acetone and chemical shifts (ppm) were recorded with tetramethylsilane (TMS) as the internal reference standard. ¹³C NMR spectra were recorded on 100, 126 or 151 MHz in CDCl₃ or d_6 -acetone, and ¹⁹F spectra were recorded on 376 or 471 MHz in CDCl₃ or d₆-acetone (CFCl₃ as outside standard and low field is positive). Multiplicities are given as: s (singlet), d (doublet), t (triplet), dd (doublet of doublets), or m (multiplet). IR spectra were recorded on a FT-IR spectrometer and only major peaks are reported in cm⁻¹. HR-MS was obtained using a Q-TOF instrument equipped with ESI source. X-Ray single crystal diffraction data were collected on a Bruker APEX-II CCD diffractometer equipped with liquid nitrogen cryogenic device. The crystal was grown in a mixed solvent of petroleum ether and CH₂Cl₂ with an approximate ratio of 5:1. The copies of NMR spectra of all compounds are provided in the Supporting Information. Room temperature is 23–25 °C. THF was distilled immediately before use from Na/benzophenone. 1,2-DCE was distilled from CaH₂ and stored in a dryer before use. Other commercially available reagents and solvents were used without further purification.

Computational Study

Computational methods

All calculations were carried out with the Gaussian 09 D.01programs.¹ Ground state geometry were fully optimized by using density functional theory (DFT)² and the M06-2X³ method with the 6-31G** basic set all atoms. Frequency calculations have been performed to verify the optimized structures as local minima. The Mulliken charge analysis results are presented below.







intermediate III



Figure 2 Charge analysis of two potential activation scenarios for intermediate III

Two potential activation scenarios for intermediate III, namely Brønsted acid (III-H) and TMSCI (III-TMS), were considered. Through DFT calculations, it was determined that TMSCI (III-TMS) exhibits a lower energy gap of 18.21 kcal/mol between the highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO) of intermediate III compared to Brønsted acid (III-H). Therefore, TMSCI is preferred over Brønsted acid in activating intermediate III.

Simultaneously, the charge analysis of the intermediate **III** activated through the two different methods is also taken into consideration. Evidently, in the case of intermediate **III** activated by TMSCI, the carbonyl carbon participating in the reaction exhibits a reduced electron cloud density, indicating heightened electrophilic activity.

These computational results are in agreement with experimental

observations.

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General Procedure for the Preparation of Starting Materials

For the synthesis of **1aa**:

Ethynylmagnesium bromide (129.2 g/mol, 0.5 mol/L in THF, 24 mL, 1.2 equiv) was added dropwise into a stirred solution of benzophenone **A** (182.2 g/mol, 10 mmol, 1.82 g) in THF (35 mL) under argon. The mixture was allowed to stir at 65 °C in an oil bath for 4 h. After the completion of the reaction determined by TLC, the reaction mixture was quenched by addition of an aqueous saturated solution of NH₄Cl (35 mL) and extracted with ethyl acetate (2×50 mL). The combined organic layers were washed with brine, dried over Na₂SO₄, and concentrated under reduced pressure. The resulting material 1,1-diphenylprop-2-yn-1-ol **B** (208.3 g/mol, 94%, 1.96 g, 9.4 mmol) was directly used for the next step without further purification.



n-BuLi (64.1 g/mol, 2.5 mol/L in hexane, 14 mL) was added dropwise to a solution of diisopropylamine (101.2 g/mol, 3.54 g, 35 mmol) in anhydrous THF (10 mL) at -78 °C under an argon atmosphere. The mixture was allowed to stir for 1 h prior to subsequent slow addition of 1,1diphenylprop-2-yn-1-ol **B** (208.3 g/mol, 2.08 g, 10 mmol). The mixture was allowed to stir at -78 °C for another 1 h. Benzaldehyde (106.1 g/mol, 1.59 g, 15 mmol, 1.5 equiv) dissolved in THF (5 mL) was added to the reaction and stirred under the same conditions for 1 h. The mixture was then warmed to room temperature and stirred for 10 h. After the completion of the reaction determined by TLC, the reaction mixture was quenched by an aqueous saturated solution of NH₄Cl (30 mL) and extracted with ethyl acetate (2×50 mL). The organic layers were combined to be washed with brine and dried over MgSO₄ for 30 min. Then the solution would be concentrated under reduced pressure and further purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 4:1) to give 1,1,4-triphenylbut-2-yne-1,4-diol **C** (314.4 g/mol, 72%, 2.26 g).

 MnO_2 (86.9 g/mol, 6.53 g, 75 mmol, 15 equiv) was added to a solution of 1,1,4-triphenylbut-2-yne-1,4-diol (**C**, 314.4 g/mol, 5 mmol, 1.57 g) in CH_2Cl_2 (15 mL) at room temperature. The resulting mixture was stirred overnight. Then the solid was filtered, and the solvents were removed under reduced pressure. The residue was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 10:1) to give 4hydroxy-1,4,4-triphenylbut-2-yn-1-one **1aa** (312.4 g/mol, 99%, 1.55 g).

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General Procedure for the Synthesis of Products

For the synthesis of 2aa:



TMSCI (108.6 g/mol, 54.3mg, 2.5 equiv) was added to an oven-dried tube charged with of 4-hydroxy-1,4,4-triphenylbut-2-yn-1-one (**1aa**, 312.4 g/mol, 62.5 mg, 0.2 mmol) and *p*-TsOH·H₂O (**2a**, 190.21 g/mol, 0.4 mg, 1 mol %) in anhydrous 1,2-DCE (2 mL). The resulting mixture was allowed to stir at 65 °C in an oil bath for 12 h, and then extracted with ethyl ether (2×15 mL), washed with a saturated aqueous solution of saturated brine, dried over Na₂SO₄, and evaporated under reduced pressure. The residue was further purified by chromatography on silica gel (petroleum ether/ethyl acetate, 20:1) to afford the product 2,2,5-triphenylfuran-3(2*H*)-one (**2aa**, 313.4 g/mol, 59.5 mg) in 95% yield.

X-ray Single Crystal Diffraction Data



Data completeness= 0.951	Theta(max)= 77.300
R(reflections)= 0.0488(3236)	wR2(reflections)= 0.1331(3640)
S = 1.066	Npar= 244





5aa CCDC: 2265196 The ellipsoid contour percent probability level is 30% in the caption of the thermal ellipsoid plot.

Bond precision:	C-C = 0.0021 A	Wavele	ngth=1.54184
Cell:	a=11.4368(3)	b=6.67839(14)	c=22.2255(6)
Temperature:	alpha=90 304 K	beta=91.647(2)	gamma=90
Volume Space group Hall group Moiety formula Sum formula Mr Dx,g cm-3 Z Mu (mm-1) F000 F000' h,k,lmax	Calculated 1696.87(7) P 21/n -P 2yn C22 H16 O2 C22 H16 O2 312.35 1.223 4 0.612 656.0 657.91 14,8,27	Rej 169 P 1 -P 2 C22 312 1.2 4 0.6 650 14,	20orted 96.87(8) 21/n 1 2yn 2 H16 O2 2 H16 O2 2.35 23 12 5.0 8,27
Nref	3545	33!	53
Tmin,Tmax	0.912,0.935	0.4	30,1.000
Tmin'	0.912		

Correction method= # Reported T Limits: Tmin=0.430 Tmax=1.000 AbsCorr = MULTI-SCAN

Data completeness= 0.946	Theta(max)= 76.059
R(reflections)= 0.0422(2776)	wR2(reflections)= 0.1196(3353)
S = 1.067	Npar= 217

Characterization Data

Characterization Data of 2aa-2ar



2,2,5-triphenylfuran-3(2H)-one (2aa): yellow solid; melting point 175–177 °C; 59.3 mg; 95%; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.97 (d, *J* = 7.6 Hz, 2H), 7.61–7.51 (m, 7H), 7.37–7.32 (m, 6H), 6.10 (s, 1H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 202.1, 184.1, 138.4, 133.0, 129.0, 128.7, 128.4, 128.4, 127.3, 126.6, 99.8, 92.9. IR (neat, cm⁻¹): 3085, 1695, 1603, 1351, 1173, 1054, 983, 881, 765, 694; HRMS (ESI) m/z: [M+H]⁺ Calcd for C₂₂H₁₇O₂ 313.1223; Found 313.1228 (1.6 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



2,2-diphenyl-5-(p-tolyl)furan-3(2H)-one (2ab): yellow solid; melting point 149–151 °C; 58.8 mg; 90%; ¹H NMR (600 MHz, CDCl₃) δ ppm 7.84 (d, *J* = 8.4 Hz, 2H), 7.55–7.54 (m, 4H), 7.35–7.32 (m, 4H), 7.31–7.29 (m, 4H), 6.04 (s, 1H), 2.42 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 201.9, 184.3, 143.9, 138.5, 129.6, 128.4, 128.3, 127.2, 126.6, 125.9, 99.1, 92.8, 21.7. IR (neat, cm⁻¹): 3065, 2917, 1695, 1565, 1349, 1173, 1056, 983, 822, 697; HRMS (ESI) m/z: [M+H]⁺ Calcd for C₂₃H₁₉O₂ 327.1380; Found 327.1381 (0.3 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



5-(4-ethylphenyl)-2,2-diphenylfuran-3(2H)-one (2ac): yellow solid; melting point 126–128 °C; 52.4 mg; 77%; ¹H NMR (600 MHz, CDCl₃) δ ppm 7.88 (d, *J* = 5.2 Hz, 2H), 7.55–7.54 (m, 4H), 7.35–7.29 (m, 8H), 6.05 (s, 1H), 2.72 (dd, *J* = 10.0 Hz 5.2 Hz, 2H), 1.26 (t, *J* = 5.2 Hz, 3H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 202.0, 184.3, 150.1, 138.5, 128.5, 128.4, 128.3, 127.4, 126.6, 126.2, 99.1, 92.8, 29.0, 15.2. IR (neat, cm⁻¹): 3066, 2971, 1692, 1607, 1347, 1173, 1054, S10 909, 843, 698; HRMS (ESI) m/z: $[M+H]^+$ Calcd for C₂₄H₂₁O₂ 341.1536; Found 341.1531 (1.5 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



5-(4-(tert-butyl)phenyl)-2,2-diphenylfuran-3(2H)-one (2ad): yellow solid; melting point 181–183 °C; 70.0 mg; 95%; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.91 (d, *J* = 7.2 Hz, 2H), 7.55–7.53 (m, 6H), 7.35–7.33 (m, 6H), 6.06 (s, 1H), 1.36 (s, 9H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 202.0, 184.2, 156.9, 138.5, 128.4, 128.3, 127.2, 126.6, 126.0, 125.9, 99.2, 92.8, 35.2, 31.0. IR (neat, cm⁻¹): 3090, 2960, 1694, 1609, 1558, 1344, 1172, 1111, 987, 697; HRMS (ESI) m/z: [M+H]⁺ Calcd for C₂₆H₂₅O₂ 369.1849; Found 369.1841 (2.2 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



5-(4-fluorophenyl)-2,2-diphenylfuran-3(2H)-one (2ae): white solid; melting point 190–191 °C; 43.6 mg; 66%; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.98 (dd, *J* = 7.6 Hz 5.6 Hz, 2H), 7.52 (d, *J* = 8.0 Hz, 4H), 7.39–7.32 (m, 6H), 7.26–7.20 (m, 2H), 6.05 (s, 1H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 201.9, 182.9, 165.5 (d, *J* = 254 Hz, 1C), 138.3, 129.7 (d, *J* = 9 Hz, 1C), 128.5, 126.6, 125.1 (d, *J* = 3 Hz, 1C), 116.4, 116.3, 99.6 (d, *J* = 1 Hz, 1C), 93.2. IR (neat, cm⁻¹): 3057, 1652, 1598, 1324, 1069, 1055, 968, 757, 668; HRMS (ESI) m/z: [M+H]⁺ Calcd for C₂₂H₁₆FO₂ 331.1129; Found 331.1126 (0.9 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



5-(4-bromophenyl)-2,2-diphenylfuran-3(2H)-one (2af): white solid; melting point

167–168 °C; 52.4 mg; 67%; ¹H NMR (600 MHz, CDCl₃) δ ppm 7.80 (d, *J* = 5.6 Hz, 2H), 7.65 (d, *J* = 5.6 Hz, 2H), 7.52–7.51 (m, 4H), 7.36–7.31 (m, 6H), 6.08 (s, 1H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 201.8, 182.8, 138.2, 132.3, 128.6, 128.5, 128.5, 127.8, 127.6, 126.6, 100.1, 93.1. IR (neat, cm⁻¹): 3061, 1699, 1602, 1342, 1173, 1072, 986, 885, 764, 696; HRMS (ESI) m/z: [M+H]⁺ Calcd for C₂₂H₁₆BrO₂ 391.0328; Found 391.0322 (1.5 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



2,2-diphenyl-5-(m-tolyl)furan-3(2H)-one (2ag): yellow solid; melting point 172–173 °C; 43.1 mg; 66%; ¹H NMR (600 MHz, CDCl₃) δ ppm 7.76–7.74 (m, 2H), 7.55–7.53 (m, 4H), 7.41–7.38 (m, 2H), 7.36–7.30 (m, 6H), 6.07 (s, 1H), 2.43 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 202.1, 184.4, 138.8, 138.4, 133.8, 128.9, 128.7, 128.4, 128.4, 127.6, 126.7, 124.6, 99.7, 92.9, 21.4. IR (neat, cm⁻¹): 3061, 1698, 1605, 1572, 1348, 1171, 1060, 989, 785, 698; HRMS (ESI) m/z: [M+H]⁺ Calcd for C₂₃H₁₉O₂ 327.1380; Found 327.1380 (0.0 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



5-(3-chlorophenyl)-2,2-diphenylfuran-3(2H)-one (2ah): white solid; melting point 154–155 °C; 50.6 mg; 73%; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.94 (d, *J* = 1.2 Hz, 1H), 7.81 (dd, *J* = 7.6 Hz 0.8 Hz, 1H), 7.55–7.51 (m, 5H), 7.45 (t, *J* = 8.0 Hz, 1H), 7.38–7.31 (m, 6H), 6.10 (s, 1H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 201.9, 182.4, 138.1, 135.3, 132.8, 130.5, 130.3, 128.5, 128.5, 127.0, 126.6, 125.5, 100.6, 93.2. IR (neat, cm⁻¹): 3093, 1698, 1606, 1563, 1349, 1174, 1058, 986, 765, 698; HRMS (ESI) m/z: [M+H]⁺ Calcd for C₂₃H₁₉O₂ 347.0833; Found 347.0832 (0.3 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



2,2-diphenyl-5-(o-tolyl)furan-3(2H)-one (2ai): white solid; melting point 134–135 °C; 56.8 mg; 87%; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.92 (d, *J* = 7.6 Hz, 1H), 7.56–7.54 (m, 4H), 7.45 (t, *J* = 7.6 Hz, 1H), 7.38–7.31 (m, 8H), 6.00 (s, 1H), 2.54 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 202.4, 185.0, 138.4, 138.1, 132.1, 131.9, 128.7, 128.6, 128.4, 128.3, 126.6, 126.3, 103.9, 92.0, 22.0. IR (neat, cm⁻¹): 3142, 1693, 1602, 1333, 1176, 1034, 984, 882, 770, 697; HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₂₃H₁₈NaO₂ 349.1199; Found 349.1193 (1.7 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



5-(2-chlorophenyl)-2,2-diphenylfuran-3(2H)-one (2aj): white solid; melting point 147–148 °C; 48.6 mg; 70%; ¹H NMR (400 MHz, CDCl₃) δ ppm 8.10–8.08 (m, 1H), 7.55–7.54 (m, 5H), 7.48–7.40 (m, 2H), 7.38–7.32 (m, 6H), 6.52 (s, 1H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 202.8, 180.6, 138.2, 134.2, 132.9, 131.3, 129.5, 128.5, 128.4, 127.8, 127.1, 126.6, 105.5, 91.5. IR (neat, cm⁻¹): 3063, 1701, 1597, 1332, 1170, 1035, 985, 887, 763, 698; HRMS (ESI) m/z: [M+H]⁺ Calcd for C₂₂H₁₆ClO₂ 347.0833; Found 347.0830 (0.9 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



5-(2-azidophenyl)-2,2-diphenylfuran-3(2H)-one (2ak): yellow solid; melting point 177–178 °C; 44.5 mg; 63%; ¹H NMR (600 MHz, CDCl₃) δ ppm 8.16 (dd, *J* = 7.8 Hz 1.2 Hz, 1H), 7.57–7.55 (m, 1H), 7.54–7.52 (m, 4H), 7.35–7.26 (m, 8H), 6.56 (s, 1H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 203.0, 179.1, 140.1, 138.4, 133.4, 128.7, 128.4, 128.3, 126.6, 124.9, 120.2, 119.2, 105.0, 91.0. IR (neat, cm⁻¹): 3062, 2927, 1697, 1602, 1341, 1270, 1169, 1039, 761, 697; HRMS (ESI) m/z: [M+H]⁺ Calcd for C₂₂H₁₆N₃O₂ 354.1237; Found 354.1237 (0.0 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



5-(3,4-dimethylphenyl)-2,2-diphenylfuran-3(2H)-one (2al): white solid; melting point 153–154 °C; 56.5 mg; 83%; ¹H NMR (600 MHz, CDCl₃) δ ppm 7.72 (s, 1H), 7.69 (d, *J* = 7.2 Hz, 1H), 7.55–7.53 (m, 4H), 7.36–7.30 (m, 6H), 7.26 (d, *J* = 8.4 Hz, 1H), 6.03 (s, 1H), 2.34 (s, 6H, two singlet peaks should be observed, however, due to the limitation of the resolution of the NMR machine, a singlet peak was identified); ¹³C NMR (151 MHz, CDCl₃) δ ppm 202.0, 184.6, 142.7, 138.5, 137.4, 130.2, 128.4, 128.3, 128.1, 126.7, 126.3, 125.0, 99.0, 92.8, 20.1, 19.8. IR (neat, cm⁻¹): 3061, 1696, 1594, 1495, 1448, 1342, 1170, 1063, 936, 698; HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₂₄H₂₀NaO₂ 363.1356; Found 363.1347 (2.5 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



5-(3,4-dichlorophenyl)-2,2-diphenylfuran-3(2H)-one (2am): white solid; melting point 132–133 °C; 54.1 mg; 71%; ¹H NMR (400 MHz, CDCl₃) δ ppm 8.04 (s, 1H), 7.75 (d, J = 7.6 Hz, 1H), 7.60 (d, J = 8.0 Hz, 1H), 7.51–7.49 (m, 4H), 7.36–7.35 (m, 6H), 6.09 (s, 1H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 201.7, 181.4, 138.0, 137.3, 133.8, 131.1, 128.7, 128.6, 128.6, 128.5, 126.6, 126.3, 100.8, 93.4. IR (neat, cm⁻¹): 3063, 2926, 1701, 1606, 1336, 1171, 1060, 986, 760, 697; HRMS (ESI) m/z: [M+H]⁺ Calcd for C₂₂H₁₅Cl₂O₂ 381.0444; Found 381.0440 (1.0 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).

5,5-diphenyl-[2,2'-bifuran]-4(5H)-one (2an): white solid; melting point 171–173 °C; 32.0 mg; 53%; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.68 (s, 1H), 7.51–7.49 (m, 4H), 7.37–7.32 (m, 6H), 7.25 (d, J = 3.0 Hz, 1H), 6.62 (t, J = 2.0 Hz, 1H), 5.97 (s, 1H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 201.2, 174.3, 147.1, 144.9, 138.3, 128.4, 128.4, 126.7, 115.4, 112.6, 98.7, 92.7. IR (neat, cm⁻¹): 3059, 1698, 1623, 1351, 1169, 1068, 984, 844, 763, 697; HRMS (ESI) m/z: [M+H]⁺ Calcd for C₂₀H₁₅O₃ 303.1016; Found 303.1011 (1.6 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



2,2-diphenyl-5-(thiophen-2-yl)furan-3(2H)-one (2ao): yellow solid; melting point 177–179 °C; 45.8 mg; 72%; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.79 (d, *J* = 2.4 Hz, 1H), 7.67–7.66 (m, 1H), 7.54–7.52 (m, 4H), 7.37–7.31 (m, 6H), 7.20 (dd, *J* = 4.8 Hz 4.0 Hz, 1H), 5.93 (s, 1H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 201.1, 178.4, 138.3, 132.2, 131.8, 130.9, 128.6, 128.4, 128.4, 126.7, 98.7, 93.1. IR (neat, cm⁻¹): 3092, 1694, 1583, 1377, 1168, 1025, 982, 835, 763, 698; HRMS (ESI) m/z: [M+H]⁺ Calcd for C₂₀H₁₅O₂S 319.0787; Found 319.0782 (1.6 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



5-(benzo[d][1,3]dioxol-5-yl)-2,2-diphenylfuran-3(2H)-one (2ap): yellow solid; melting point 214–216 °C; 44.9 mg; 63%; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.56–7.51 (m, 5H), 7.38–7.33 (m, 7H), 6.92 (d, *J* = 8.4 Hz, 1H), 6.06 (s, 2H), 5.95 (s, 1H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 201.6, 183.5, 151.7, 148.3, 138.5, 128.4, 128.3, 126.6, 123.1, 122.7, 108.8, 107.0, 102.0, 98.6, 92.9. IR (neat, cm⁻¹): 3061, 2919, 1690, 1574, 1448, 1318, 1036, 923, 759, 694; HRMS (ESI) m/z: [M+H]⁺ Calcd for C₂₃H₁₇O₄ 357.1121; Found 357.1122 (0.3 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



5-(naphthalen-2-yl)-2,2-diphenylfuran-3(2H)-one (**2aq**): yellow solid; melting point 160–162 °C; 60.9 mg; 84%; ¹H NMR (400 MHz, CDCl₃) δ ppm 8.56 (s, 1H), 7.99–7.88 (m, 4H), 7.63–7.58 (m, 6H), 7.39–7.32 (m, 6H), 6.21 (s, 1H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 202.1, 184.0, 138.5, 135.4, 132.7, 129.3, 128.8, 128.5, 128.5, 128.4, 127.9, 127.8, 127.2, 126.7, 125.9, 123.4, 100.2, 93.0. IR (neat, cm⁻¹): 3060, 1696, 1604, 1582, 1369, 1172, 1053, 988, 758, 699; HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₂₆H₁₈NaO₂ 385.1199; Found 385.1192 (1.8 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



5-pentyl-2,2-diphenylfuran-3(2H)-one (2ar): yellow oil; 17.3 mg; 27%; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.46–7.44 (m, 4H), 7.36–7.29 (m, 6H), 5.49 (m, 1H), 2.63 (t, J = 7.6 Hz, 2H), 1.78–1.71 (m, 2H), 1.41–1.36 (m, 2H), 1.31–1.30 (m, 4H), 0.88 (t, J = 6.8 Hz, 3H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 202.6, 192.9, 138.4, 128.4, 128.3, 126.5, 102.4, 92.5, 31.4, 30.9, 28.8, 26.1, 22.4, 14.0. IR (neat, cm⁻¹): 3062, 2929, 1704, 1601, 1449, 1369, 1170, 762, 697, 602; HRMS (ESI) m/z: [M+H]⁺ Calcd for C₂₂H₂₅O₂ 321.1849; Found 321.1843 (1.9 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).

Characterization Data of 2ba-2br



5-phenyl-2,2-di-p-tolylfuran-3(2H)-one (2ba): yellow oil; 46.3 mg; 68%; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.94 (d, *J* = 7.6 Hz, 2H), 7.55 (t, *J* = 7.2 Hz, 1H), 7.49 (t, *J* = 7.6 Hz, 2H), 7.42–7.40 (m, 4H), 7.16–7.14 (m, 4H), 6.07 (s, 1H), 2.32 (s, 6H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 202.5, 184.0, 138.2, 135.6, 132.8, 129.1, 128.9, 128.8, 127.2, 126.6, 99.7, 93.1, 21.1. IR (neat, cm⁻¹): 3029, 1698, 1606, 1352, 1169, 1054, 987, 886, 767, 689; HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₂₄H₂₀NaO₂ 363.1356; Found 363.1354 (0.6 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



2,2-bis(4-fluorophenyl)-5-phenylfuran-3(2H)-one (2bb): white solid; melting point 137–138 °C; 64.8 mg; 93%; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.96–7.94 (m, 2H), 7.61 (t, *J* = 7.6 Hz, 1H), 7.55–7.47 (m, 6H), 7.07–7.02 (m, 4H), 6.10 (s, 1H); ¹³C NMR (151 MHz, CDCl₃) δ ppm

201.8, 184.2, 162.7 (d, $J^1 = 249.2 \text{ Hz}$), 134.1 (d, $J^4 = 3.0 \text{ Hz}$), 133.2, 129.1, 128.5 (d, $J^3 = 9.1 \text{ Hz}$), 128.4, 127.3, 115.5 (d, $J^2 = 21.1 \text{ Hz}$), 99.7, 91.9; ¹⁹F NMR (376 MHz, CDCl₃) δ ppm -113.47--113.54 (m, 2F). IR (neat, cm⁻¹): 3070, 1698, 1605, 1352, 1185, 1054, 989, 887, 769, 688; HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₂₂H₁₄F₂NaO₂ 371.0854; Found 371.0847 (1.9 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



2,2-bis(4-chlorophenyl)-5-phenylfuran-3(2H)-one (2bc): yellow solid; melting point 141–142 °C; 63.3 mg; 83%; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.94 (d, *J* = 7.2 Hz, 2H), 7.61 (t, *J* = 7.6 Hz, 1H), 7.55–7.51 (m, 2H), 7.47–7.45 (m, 4H), 7.34–7.32 (m, 4H),6.10 (s, 1H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 201.1, 184.3, 136.5, 134.7, 133.3, 129.1, 128.7, 128.3, 127.9, 127.2, 99.7, 91.5. IR (neat, cm⁻¹): 3066, 1699, 1605, 1352, 1165, 1054, 988, 834, 769, 688; HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₂₂H₁₄Cl₂NaO₂ 403.0263; Found 403.0266 (0.7 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



2-(4-fluorophenyl)-2-(4-methoxyphenyl)-5-phenylfuran-3(2H)-one (2bd): yellow oil; 40.4 mg; 56%; ¹H NMR (600 MHz, CDCl₃) δ ppm 7.94 (d, *J* = 7.8 Hz, 2H), 7.59 (t, *J* = 8.4 Hz, 1H), 7.53–7.49 (m, 4H), 7.41 (d, *J* = 8.4 Hz, 2H), 7.03 (t, *J* = 9.0 Hz, 2H), 6.88 (d, *J* = 8.4 Hz, 2H), 6.09 (s, 1H), 3.78 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 202.3, 184.1, 162.7 (d, *J*¹ = 247.6 Hz), 159.7, 134.4 (d, *J*⁴ = 3.0 Hz), 133.0, 130.4, 129.0, 128.7, 128.5 (d, *J*³ = 7.6 Hz), 128.2, 127.3, 115.3 (d, *J*² = 21.1 Hz), 113.9, 99.7, 92.5, 55.3; ¹⁹F NMR (376 MHz, CDCl₃) δ ppm -118.70–118.75 (m, 1F). IR (neat, cm⁻¹): 3045, 1697, 1605, 1352, 1249, 1169, 887, 831, 768, 688; HRMS (ESI) m/z: [M+H]⁺ Calcd for C₂₃H₁₈FO₃ 361.1234; Found 361.1228 (1.7 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



5-phenyl-2,2-di-m-tolylfuran-3(2H)-one (2be): yellow oil; 32.7 mg; 48%; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.96 (d, *J* = 4.8 Hz, 2H), 7.60–7.57 (m, 1H), 7.52 (t, *J* = 5.2 Hz, 2H), 7.33–7.32 (m, 4H), 7.25–7.22 (m, 2H), 7.13 (d, *J* = 4.8 Hz, 2H), 6.08 (s, 1H), 2.33 (s, 6H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 202.3, 184.1, 138.4, 138.1, 132.9, 129.2, 129.0, 128.8, 128.3, 127.3, 127.2, 123.8, 99.8, 93.2, 21.6. IR (neat, cm⁻¹): 3058, 1698, 1606, 1351, 1288, 1152, 1056, 887, 767, 692; HRMS (ESI) m/z: [M+H]⁺ Calcd for C₂₄H₂₁O₂ 341.1536; Found 341.1530 (1.8 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



2,5-diphenyl-2-(p-tolyl)furan-3(2H)-one (2bf): colorless oil; 32.6 mg; 50%; ¹H NMR (600 MHz, CDCl₃) δ ppm 7.95 (d, *J* = 7.8 Hz, 2H), 7.58–7.49 (m, 5H), 7.41 (d, *J* = 8.4 Hz, 2H), 7.35–7.30 (m, 3H), 7.16 (d, *J* = 7.8 Hz, 2H), 6.08 (s, 1H), 2.32 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 202.3, 184.1, 138.5, 138.3, 135.5, 132.9, 129.1, 128.9, 128.8, 128.4, 128.3, 127.2, 126.7, 126.6, 99.8, 93.0, 21.1. IR (neat, cm⁻¹): 3060, 2919, 1697, 1606, 1352, 1171, 1054, 985, 768, 691; HRMS (ESI) m/z: [M+H]⁺ Calcd for C₂₃H₁₉O₂ 327.1380; Found 327.1375 (1.5 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



2-(4-methoxyphenyl)-2,5-diphenylfuran-3(2H)-one (2bg): yellow oil; 60.3 mg; 88%; ¹H NMR (600 MHz, CDCl₃) δ ppm 7.96–7.94 (m, 2H), 7.59–7.57 (m, 1H), 7.53–7.50 (m, 4H), 7.43 (d, *J* = 9.0 Hz, 2H), 7.36–7.30 (m, 3H), 6.88 (d, *J* = 9.0 Hz, 2H), 6.09 (s, 1H), 3.78 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 202.5, 184.1, 159.7, 138.5, 132.9, 130.6, 129.0, 128.8, 128.4, 128.3, 128.3, 127.3, 126.5, 113.8, 99.7, 93.0, 55.3. IR (neat, cm⁻¹): 3063, 2928, 1696, 1607, 1510, 1352, 1172, 885, 767, 691; HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₂₃H₁₈NaO₃ 365.1148; Found 365.1139 (2.5 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



2-([1,1'-biphenyl]-4-yl)-2,5-diphenylfuran-3(2H)-one (2bh): colorless oil; 56.7 mg; 73%; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.96 (d, J = 6.8 Hz, 2H), 7.63–7.48 (m, 11H), 7.42–7.32 (m, 6H), 6.11 (s, 1H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 202.0, 184.2, 141.3, 140.4, 138.3, 137.4, 133.0, 129.0, 128.7, 128.7, 128.5, 128.4, 127.4, 127.3, 127.2, 127.1, 127.1, 126.6, 99.8, 92.8. IR (neat, cm⁻¹): 3061, 1697, 1606, 1489, 1352, 1172, 1054, 986, 767, 654; HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₂₈H₂₀NaO₂ 411.1356; Found 411.1348 (1.9 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



2-(4-chlorophenyl)-2,5-diphenylfuran-3(2H)-one (2bi): yellow oil; 57.6 mg; 83%; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.95 (d, J = 7.2 Hz, 2H), 7.60 (t, J = 7.2 Hz, 1H), 7.55–7.48 (m, 6H), 7.35–7.31 (m, 5H), 6.10 (s, 1H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 201.6, 184.2, 138.0, 136.9, 134.5, 133.1, 129.0, 128.6, 128.6, 128.5, 128.5, 128.0, 127.2, 126.5, 99.7, 92.2. IR (neat, cm⁻¹): 3062, 1698, 1606, 1351, 1172, 1054, 987, 886, 769, 691; HRMS (ESI) m/z: [M+H]⁺ Calcd for C₂₂H₁₆ClO₂ 347.0833; Found 347.0833 (0.0 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



2,5-diphenyl-2-(m-tolyl)furan-3(2H)-one (2bj): yellow oil; 50.3 mg; 77%; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.96 (d, *J* = 7.2 Hz, 2H), 7.60–7.50 (m, 5H), 7.37–7.30 (m, 5H), 7.24 (t, *J* = 8.0 Hz, 1H), 7.13 (d, *J* = 7.6 Hz, 1H), 6.09 (s, 1H), 2.32 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 202.2,

184.1, 138.4, 138.3, 138.1, 132.9, 129.2, 129.0, 128.8, 128.4, 128.3, 128.3, 127.3, 127.2, 126.6, 123.8, 99.8, 93.0, 21.5. IR (neat, cm⁻¹): 3061, 1697, 1606, 1352, 1171, 1054,985, 885, 768, 691 HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{23}H_{19}O_2$ 327.1380; Found 327.1375 (1.5 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



2,5-diphenyl-2-(o-tolyl)furan-3(2H)-one (2bk): yellow solid; melting point 171–172 °C; 55.5 mg; 85%; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.94 (d, *J* = 7.6 Hz, 2H), 7.70–7.68 (m, 1H), 7.58 (t, *J* = 7.6 Hz, 1H), 7.53–7.50 (m, 2H), 7.31–7.26 (m, 6H), 7.24–7.20 (m, 2H), 6.09 (s, 1H), 2.23 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 202.0, 183.7, 139.1, 138.2, 135.1, 132.9, 132.5, 129.0, 128.9, 128.7, 128.5, 127.9, 127.2, 125.5, 125.3, 99.6, 94.6, 21.2. IR (neat, cm⁻¹): 3061, 1697, 1607, 1352, 1174, 1055, 983, 885, 764, 690; HRMS (ESI) m/z: [M+H]⁺ Calcd for C₂₃H₁₉O₂ 327.1380; Found 327.1383 (0.9 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



2-(2-fluorophenyl)-2,5-diphenylfuran-3(2H)-one (**2bl**): yellow solid; melting point 183–184 °C; 50.2 mg; 76%; ¹H NMR (600 MHz, CDCl₃) δ ppm 7.95 (d, *J* = 7.2 Hz, 2H), 7.58–7.55 (m, 3H), 7.50 (t, *J* = 5.2 Hz, 2H), 7.38–7.33 (m, 5H), 7.12–7.09 (m, 1H), 7.08–7.04 (m, 1H), 6.10 (s, 1H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 201.6, 183.9, 161.60 (d, *J*¹ = 252.2 Hz), 136.8, 132.9, 131.0 (d, *J*³ = 7.6 Hz), 130.1 (d, *J*³ = 3.0 Hz), 128.9, 128.7, 128.4, 128.4, 127.3, 125.7, 125.7 (d, *J*² = 12.1 Hz), 123.7 (d, *J*⁴ = 3.0 Hz), 116.5 (d, *J*² = 21.1 Hz), 99.5 (d, *J*³ = 1.5 Hz), 90.8; ¹⁹F NMR (376 MHz, CDCl₃) δ ppm -113.97–-114.03 (m, 1F). IR (neat, cm⁻¹): 3087, 1697, 1606, 1349, 1278, 1173, 1052, 990, 759, 684; HRMS (ESI) m/z: [M+H]⁺ Calcd for C₂₂H₁₆FO₂ 331.1129; Found 331.1129 (0.0 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



2-(3,4-dimethylphenyl)-2,5-diphenylfuran-3(2H)-one (2bm): yellow solid; melting point 167–168 °C; 45.6 mg; 67%; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.95 (d, *J* = 7.2 Hz, 2H), 7.55–7.48 (m, 5H), 7.34–7.24 (m, 5H), 7.11 (d, *J* = 2.9 Hz, 1H), 6.08 (s, 1H), 2.22 (s, 6H, two singlet peaks should be observed, however, due to the limitation of the resolution of the NMR machine, a singlet peak was identified); ¹³C NMR (151 MHz, CDCl₃) δ ppm 202.3, 184.0, 138.5, 137.0, 136.7, 135.9, 132.8, 129.6, 128.9, 128.8, 128.3, 128.2, 127.9, 127.2, 126.5, 124.3, 99.7, 93.1, 19.9, 19.4. IR (neat, cm⁻¹): 3061, 1697, 1606, 1351, 1184, 1054, 1026, 888, 767, 692; HRMS (ESI) m/z: [M+H]⁺ Calcd for C₂₄H₂₁O₂ 341.1536; Found 341.1531 (1.5 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



2,5-diphenyl-2-(thiophen-2-yl)furan-3(2H)-one (2bn): red solid; melting point 160–161 °C; 26.1 mg; 41%; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.95 (d, *J* = 7.6 Hz, 2H), 7.61–7.57 (m, 3H), 7.54–7.50 (m, 2H), 7.38–7.32 (m, 4H), 7.23 (d, *J* = 3.2 Hz, 1H), 7.01–6.99 (m, 1H), 6.09 (s, 1H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 200.8, 184.1, 141.3, 137.8, 133.1, 129.0, 128.7, 128.6, 128.4, 127.3, 126.9, 126.7, 126.6, 125.8, 99.0, 90.5. IR (neat, cm⁻¹): 3064, 1701, 1604, 1588, 1491, 1349, 1157, 1051, 768, 694; HRMS (ESI) m/z: [M+H]⁺ Calcd for C₂₀H₁₅O₂S 319.0787; Found 319.0783 (1.3 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



2-phenyl-1-oxaspiro[4.5]dec-2-en-4-one (2bo): colorless oil; 38.8 mg; 85%; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.87–7.85 (m, 2H), 7.58–7.54 (m, 1H), 7.51–7.47 (m, 2H), 5.98 (s, 1H), 1.83–1.78 (m, 4H), 1.75–1.69 (m, 5H), 1.42–1.37 (m, 1H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 206.9, 183.4, 132.4, 129.3, 128.8, 127.1, 99.2, 90.7, 31.9, 24.5, 21.9. IR (neat, cm⁻¹): 3061, 1693,

1606, 1567, 1449, 1363, 1258, 886, 771, 690; HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{15}H_{17}O_2$ 229.1223; Found 229.1223 (0.0 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



2-phenyl-1,8-dioxaspiro[4.5]dec-2-en-4-one (2bp): white solid; melting point 147–148 °C; 41.9 mg; 91%; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.88–7.86 (m, 2H), 7.61–7.57 (m, 1H), 7.52 (t, J = 7.6 Hz, 2H), 6.03 (s, 1H), 4.09–4.05 (m, 2H), 3.91–3.85 (m, 2H), 2.17–2.10 (m, 2H), 1.62 (dd, J = 14 Hz 1.6 Hz, 2H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 204.8, 183.4, 132.8, 128.9, 128.9, 127.1, 99.5, 87.2, 63.8, 32.0. IR (neat, cm⁻¹): 3067, 1684, 1563, 1452, 1368, 1256, 1074, 1021, 886, 690; HRMS (ESI) m/z: [M+H]⁺ Calcd for C₁₄H₁₅O₃ 231.1016; Found 231.1012 (1.7 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



2,2-dimethyl-5-phenylfuran-3(2H)-one (2bq): yellow solid; melting point 70–72 °C; 35.8 mg; 95%; ¹H NMR (600 MHz, CDCl₃) δ ppm 7.84 (d, *J* = 7.8 Hz, 2H), 7.56 (d, *J* = 7.8 Hz, 1H), 7.49 (d, *J* = 7.8 Hz, 2H), 5.97 (s, 1H), 1.50 (s, 6H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 206.9, 183.4, 132.5, 129.2, 128.8, 127.1, 98.5, 88.9, 23.1. IR (neat, cm⁻¹): 3063, 1696, 1600, 1360, 1172, 1050, 954, 855, 774, 690; HRMS (ESI) m/z: [M+H]⁺ Calcd for C₁₂H₁₃O₂ 189.0910; Found 189.0910 (0.0 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).



2-methyl-2,5-diphenylfuran-3(2H)-one (2br): colorless oil; 45.6 mg; 91%; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.93 (d, *J* = 8.0 Hz, 2H), 7.60–7.50 (m, 5H), 7.38–7.28 (m, 3H), 6.00 (s, 1H), 1.86 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 204.2, 183.8, 138.4, 132.8, 128.9, 128.8, 128.5, 128.0,

127.1, 124.5, 98.7, 90.3, 24.4. IR (neat, cm⁻¹): 3062, 1697, 1602, 1567, 1355, 1124, 1049, 862, 772, 692; HRMS (ESI) m/z: $[M+H]^+$ Calcd for C₁₇H₁₅O₂ 251.1067; Found 251.1063 (1.6 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).

Characterization Data of 3ab



2,2-diphenyl-5-(p-tolyl)furan-3(2H)-thione (3ab): yellow solid; melting point 178–195 °C; 55.5 mg; 81%; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.87 (d, *J* = 8.0 Hz, 2H), 7.53–7.50 (m, 4H), 7.33–7.29 (m, 8H), 6.89 (s, 1H), 2.41 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 229.9, 182.4, 144.7, 139.5, 130.0, 128.4, 128.1, 127.9, 127.6, 125.1, 118.4, 105.8, 21.9. IR (neat, cm⁻¹): 1610, 1556, 1498, 1376, 1316, 1180, 1062, 809, 763, 701; HRMS (ESI) m/z: [M+H]⁺ Calcd for C₂₃H₁₉OS 343.1151; Found 343.1151 (0.0 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).

Characterization Data of 3ad



4-bromo-5-(4-(tert-butyl)phenyl)-2,2-diphenylfuran-3(2H)-one (3ad): white solid; melting point 194–195 °C; 82.3 mg; 92%; ¹H NMR (400 MHz, CDCl₃) δ ppm 8.32 (d, J = 8.4 Hz, 2H), 7.58 (d, J = 8.8 Hz, 2H), 7.55–7.53 (m, 4H), 7.37–7.30 (m, 6H), 1.37 (s, 9H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 196.5, 177.5, 157.1, 137.8, 128.6, 128.5, 128.5, 126.5, 125.8, 125.6, 93.9, 91.1, 35.3, 31.0. IR (neat, cm⁻¹): 1714, 1606, 1577, 1498, 1187, 1078, 910, 844, 765, 696; HRMS (ESI) m/z: [M+H]⁺ Calcd for C₂₆H₂₄BrO₂ 447.0954; Found 447.0957 (0.7 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).

Characterization Data of 4ab



5-methyl-2,2-diphenyl-5-(p-tolyl)dihydrofuran-3(2H)-one (4ab): yellow oil; 34.9 mg; 51%; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.72 (d, *J* = 7.6 Hz, 2H), 7.42–7.39 (m, 4H), 7.35 (t, *J* = 7.6 Hz, 2H), 7.28 (d, *J* = 7.2 Hz, 1H), 7.25–7.19 (m, 3H), 7.16 (d, *J* = 8.0 Hz, 2H), 3.08 (d, *J* = 17.6 Hz, 1H), 2.93 (d, *J* = 17.6 Hz, 1H), 2.34 (s, 3H), 1.53 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 212.3, 143.6, 142.4, 142.0, 136.8, 129.0, 128.2, 128.1, 127.5, 127.4, 126.2, 126.2, 124.7, 86.7, 80.9, 49.7, 31.6, 21.0. IR (neat, cm⁻¹): 1753, 1491, 1447, 1159, 1046, 1026, 818, 754, 699, 608; HRMS (ESI) m/z: [M+H]⁺ Calcd for C₂₄H₂₃O₂ 343.1693; Found 343.1692 (0.3 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1).

Characterization Data of 5aa



1,4,4-triphenylbut-3-ene-1,2-dione (5aa): yellow solid; melting point 97–98 °C; 39.4 mg; 63%; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.72 (d, *J* = 7.2 Hz, 2H), 7.54 (t, *J* = 7.6 Hz, 1H), 7.44–7.34 (m, 7H), 7.19–7.16 (m, 1H), 7.11–7.05 (m, 4H), 6.93 (s, 1H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 194.1, 191.9, 160.1, 139.7, 137.8, 133.8, 132.7, 130.5, 130.4, 129.7, 129.2, 128.8, 128.5, 128.2, 128.0, 123.4. IR (neat, cm⁻¹): 2920, 2850, 1675, 1639, 1444, 1172, 959, 745, 697; HRMS (ESI) m/z: [M+H]⁺ Calcd for C₂₂H₁₇O₂ 313.1223; Found 313.1223 (0.0 ppm). This product was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 50:1).

Characterization Data of 1ab-OMe



4-methoxy-4,4-diphenyl-1-(p-tolyl)but-2-yn-1-one (1ab-OMe): yellow oil; ¹H NMR (400 MHz, CDCl₃) δ ppm 8.02 (d, *J* = 8.0 Hz, 2H), 7.59–7.57 (m, 4H), 7.37–7.33 (m, 4H), 7.31–7.26 (m, 4H), 3.44 (s, 3H), 2.41 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ ppm 177.0, 145.5, 141.9, 134.4, 129.6, 129.4, 128.4, 128.1, 126.7, 92.1, 86.9, 81.2, 53.1, 21.8. IR (neat, cm⁻¹): 2929, 2218, 1645, 1620, 1448, 1264, 1175, 1072, 734, 698; HRMS (ESI) m/z: [M+H]⁺ Calcd for C₂₄H₂₁O₂ 341.1536; Found 341.1537 (0.3 ppm). This product was purified by flash column chromatography (silica

gel, petroleum ether/ethyl acetate, 30:1).

¹H, ¹³C, and ¹⁹F NMR Spectra



1aa





1ab







1ac







1ad

¹H NMR spectrum was recorded on 400 MHz in CDCl₃.



S29



1ae









¹H NMR spectrum was recorded on 400 MHz in CDCl₃.

1af



S32



1ag







ОНО

¹H NMR spectrum was recorded on 400 MHz in CDCl₃.

1ai


O CI OH

¹H NMR spectrum was recorded on 400 MHz in CDCl₃.

1aj



S36

O N₃OH

1ak

¹H NMR spectrum was recorded on 600 MHz in d₆-acetone.



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ppm

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210 200 190 180 170 160 150 140 130 120 110 100 90 80 70

ppm







1be



OH OH

¹H NMR spectrum was recorded on 400 MHz in CDCl₃.

1bf



OH OMe

1bg

¹H NMR spectrum was recorded on 400 MHz in CDCl₃.



$^{13}\mbox{C}\mbox{H}\mbox{ NMR}$ spectrum was recorded on 151 MHz in $\mbox{CDCI}_3.$

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¹H NMR spectrum was recorded on 400 MHz in CDCl₃.







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O OH OH

¹H NMR spectrum was recorded on 400 MHz in CDCl₃.

1bj



O OH OH

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220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10

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1bo



220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



1bp







1br







1ab-OMe







2aa





2ab

¹H NMR spectrum was recorded on 600 MHz in CDCl₃.





2ac

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2ad




¹H NMR spectrum was recorded on 400 MHz in CDCl₃.

2ae









2af





2ag

¹H NMR spectrum was recorded on 600 MHz in CDCl₃.





2ah

¹H NMR spectrum was recorded on 400 MHz in CDCl₃.





2ai





¹H NMR spectrum was recorded on 400 MHz in CDCl₃.

2aj





2ak

¹H NMR spectrum was recorded on 600 MHz in CDCl₃.





¹H NMR spectrum was recorded on 600 MHz in CDCl₃.

2al





⁰ 2am ¹H NMR spectrum was recorded on 400 MHz in CDCl₃.





2an

¹H NMR spectrum was recorded on 400 MHz in CDCl₃.





2ao

¹H NMR spectrum was recorded on 400 MHz in CDCl₃.





2ap





2aq





2ar





2ba





2bb









2bc



Contraction of the second seco

2bd

¹H NMR spectrum was recorded on 600 MHz in CDCl₃.



S91







2be

¹H NMR spectrum was recorded on 400 MHz in CDCl₃.



2bf



OMe

2bg





¹H NMR spectrum was recorded on 400 MHz in CDCl₃.

2bh





2bi





¹H NMR spectrum was recorded on 400 MHz in CDCl₃.

2bj





2bk





¹H NMR spectrum was recorded on 600 MHz in CDCl₃.

2bl







2bm





2bn





⁰ 2bo ¹H NMR spectrum was recorded on 400 MHz in CDCl₃.





¹H NMR spectrum was recorded on 400 MHz in CDCl₃.

2bp









¹H NMR spectrum was recorded on 400 MHz in CDCl₃.

2br




3ab

¹H NMR spectrum was recorded on 400 MHz in CDCl₃.





3ad

¹H NMR spectrum was recorded on 400 MHz in CDCl₃.





4ab

¹H NMR spectrum was recorded on 400 MHz in CDCl₃.

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5aa

¹H NMR spectrum was recorded on 400 MHz in CDCl₃.

