Electronic Supplementary Material (ESI) for Chemical Communications.

Supplementary Information

Ruthenium(II)-Catalysed Electrooxidative [4+2] Annulation of Benzylic Alcohols with Internal Alkynes: Entry to Isocoumarins

Mu-Jia Luo, Ming Hu, Ren-Jie Song*, De-Liang He*, and Jin-Heng Li*

State Key Laboratory of Chemo/Biosensing and Chemometrics, Hunan University, Changsha 410082, China, Key Laboratory of Jiangxi Province for Persistent Pollutants Control and Resources Recycle, Nanchang Hangkong University, Nanchang 330063, China, and State Key Laboratory of Applied Organic Chemistry Lanzhou University, Lanzhou 730000, China

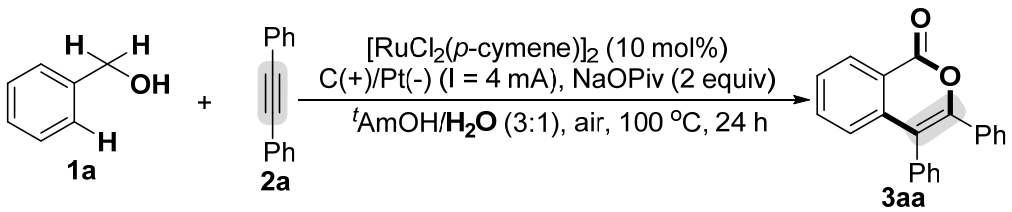
List of Contents

(A) Typical Experimental Procedure

(a) General

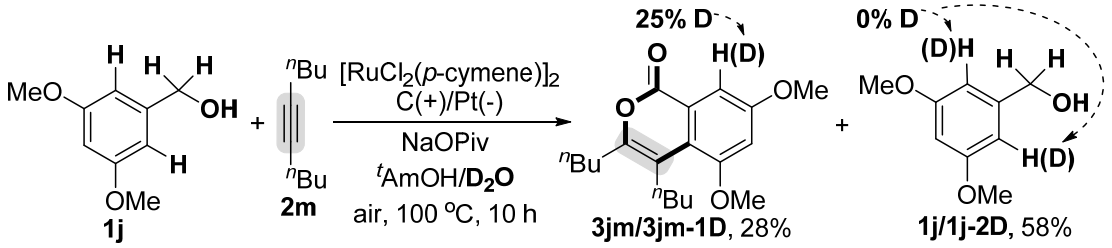
The ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ solvent on a NMR spectrometer using TMS as internal standard. HRMS was measured on an electrospray ionization (ESI) apparatus using time-of-flight (TOF) mass spectrometry. Melting points are uncorrected. The instrument for electrolysis is DC power source (PM3005B) (made in China). Cyclic voltammograms were obtained on a CHI 605E potentiostat, using Pt slice (1.0×1.0 cm^2) as working electrode, Pt slice (1.0×1.0 cm^2) and Ag/AgCl as counter and reference electrode. The anode electrode is graphite rod (Φ6mm×80mm) and cathode electrode is platinum electrodes (1.0×1.0 cm^2).

(b) General procedure for synthesis of compound 3.

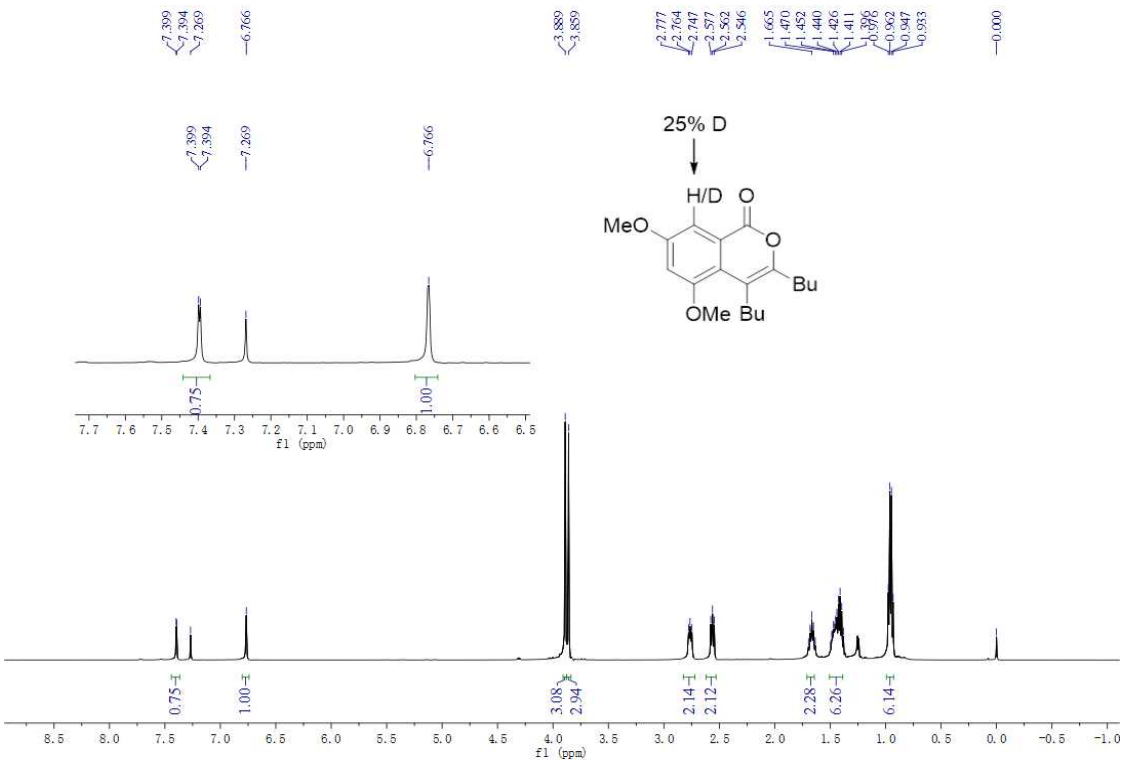


To an undivided three-necked bottle (10 mL) were added benzyl alcohol **1** (0.6 mmol), diphenylacetylene **2** (0.3 mmol), NaOPiv (2 equiv), [$RuCl_2$(*p*-cymene)] (10 mol%), and ^{t}AmOH/H_2O (3:1; 6 mL). The bottle was equipped with platinum electrodes (1.0×1.0 cm^2) as cathode and graphite rod electrode as anode under air. The reaction mixture was stirred and electrolyzed at a constant current of 4 mA at 100 °C for 24 h until complete consumption of starting material as monitored by TLC and/or GC-MS analysis. After the reaction was finished, the solution was extracted with EtOAc (3×10 mL). The combined organic layer was dried with Na_2SO_4, filtered and concentrated in vacuum. The resulting residue was purified by silica gel column chromatography (hexane/ethyl acetate) to afford the desired products **3**.

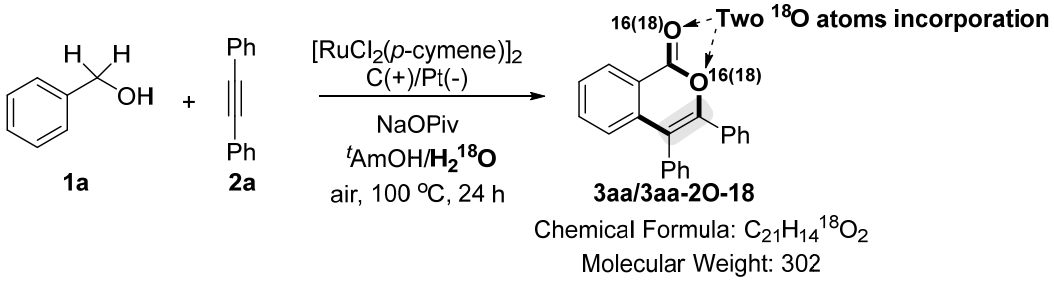
(c) H/D Exchange Experiment.



To an undivided three-necked bottle (10 mL) were added benzyl alcohol **1j** (0.6 mmol), Diphenylacetylene **2m** (0.3 mmol), NaOPiv (2 equiv), [$RuCl_2$(*p*-cymene)] (10 mol%), and ^{t}AmOH/H_2O (3:1; 6 mL). The bottle was equipped with platinum electrodes (1.0×1.0 cm^2) as cathode and graphite rod electrode as anode under air. The reaction mixture was stirred and electrolyzed at a constant current of 4 mA at 100 °C for 10 h until complete consumption of starting material as monitored by TLC and/or GC-MS analysis. After the reaction was finished, the solution was extracted with EtOAc (3×10 mL). The combined organic layer was dried with Na_2SO_4, filtered and concentrated in vacuum. The resulting residue was purified by silica gel column chromatography (hexane/ethyl acetate) to afford the desired products **3jm/3jm-1D**.



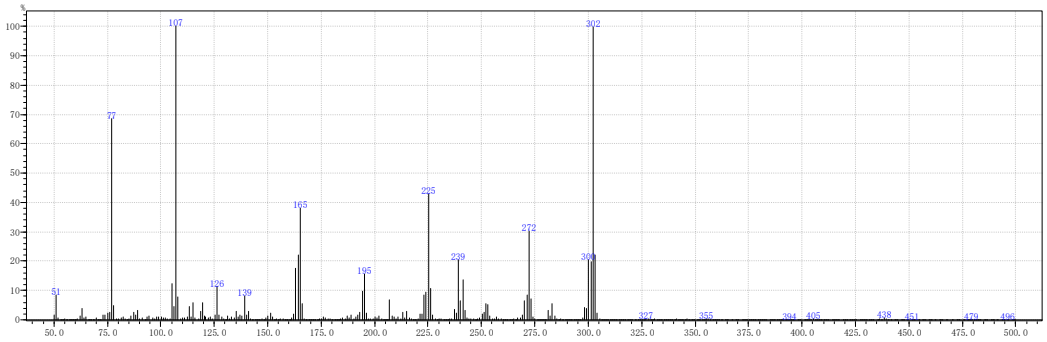
(d) $H_2{}^{18}O$ experiments

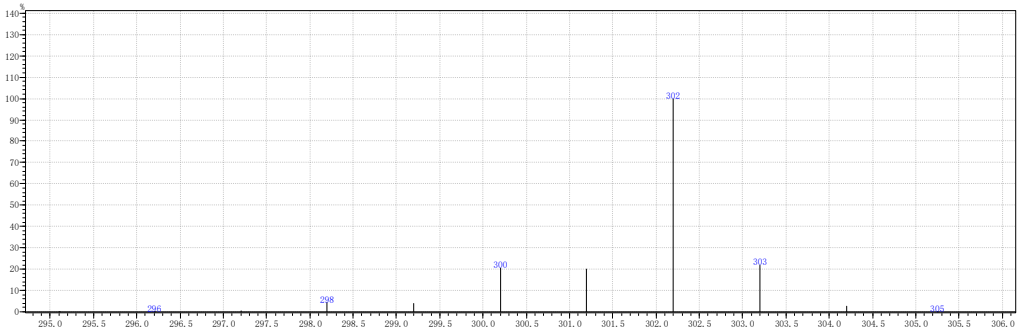


Chemical Formula: $C_{21}H_{14}{}^{18}O_2$

Molecular Weight: 302

GC-Mass dates:





[MS Spectrum]

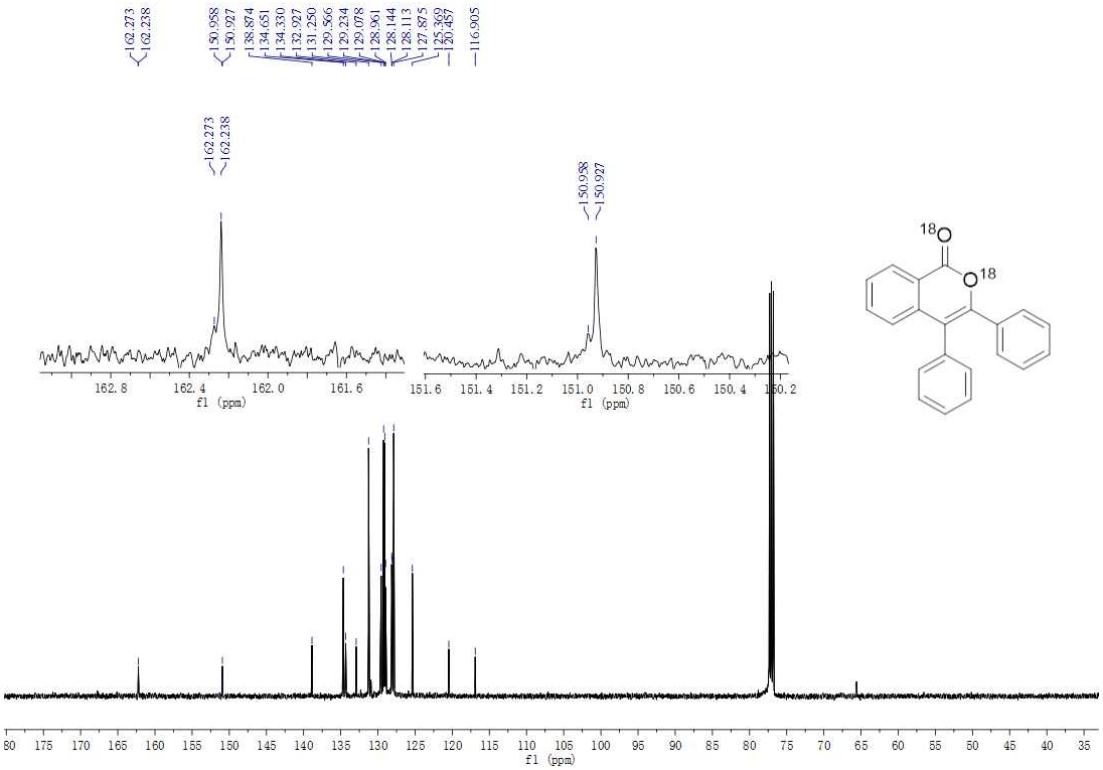
# of Peaks 414

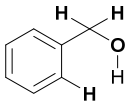
Raw Spectrum 19.015 (scan : 2604) Base Peak m/z 107.05 (Inten : 597,247)

Background No Background Spectrum

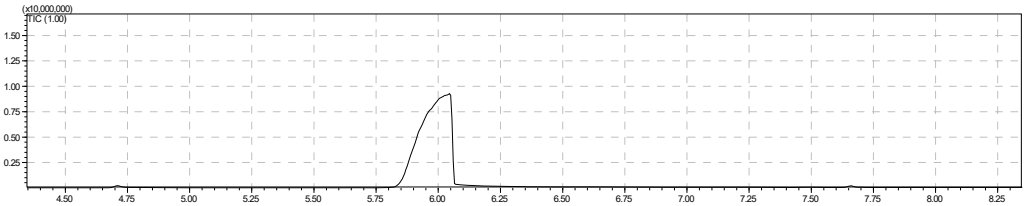
m/z	Absolute Intensity	Relative Intensity						
50.05	9620	1.61	113.05	27093	4.54	**302.20**	**595047**	**99.63**
51.05	51317	8.59	**298.20**	**25316**	**4.24**	303.20	131090	21.95
107.05	**597247**	**100.00**	299.20	23161	3.88	304.20	14358	2.40
108.05	47354	7.93	**300.20**	**121913**	**20.41**			
112.15	6553	1.10	301.20	119086	19.94			

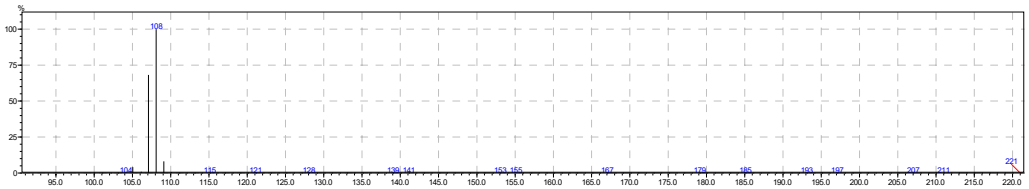
^{13}C NMR Data:





Chemical Formula: C_7H_8O
Molecular Weight: 108





[MS Spectrum]

# of Peaks 401

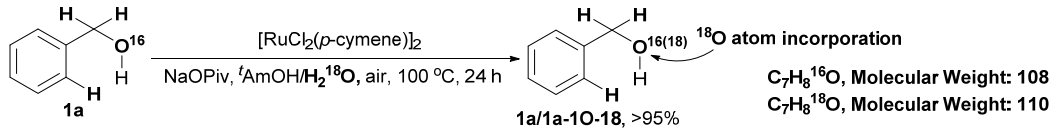
Raw Spectrum 6.035 (scan : 408)

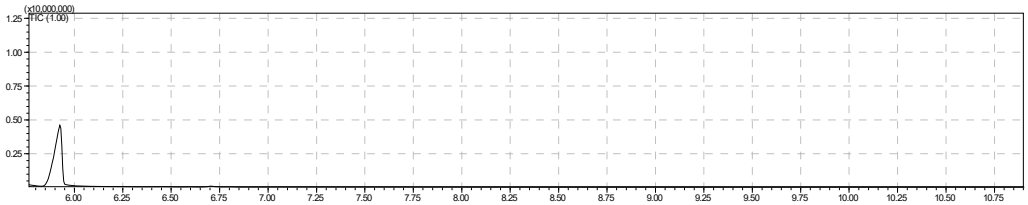
Background No Background Spectrum

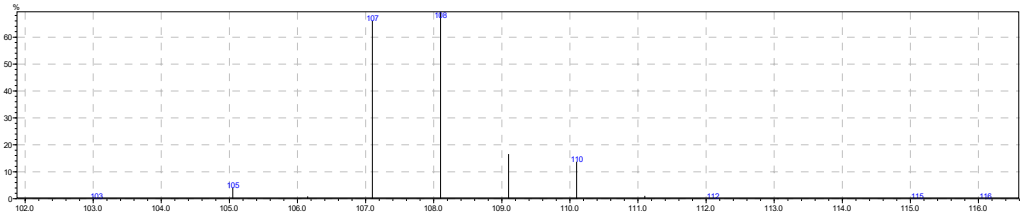
Base Peak m/z 108.10 (Inten : 5,009,356)

Event# 1

m/z	Absolute Intensity	Relative Intensity						
105.05	203808	4.07	**108.10**	**5009356**	**100.00**	111.10	1366	0.03
106.15	42201	0.84	109.10	403567	8.06	112.10	110	0.00
107.10	3414806	68.17	**110.10**	**25276**	**0.50**			







[MS Spectrum]

# of Peaks 399

Raw Spectrum 5.920 (scan : 385)

Background No Background Spectrum

Base Peak m/z 108.10 (Inten : 2,133,895)

Event# 1

m/z	Absolute Intensity	Relative Intensity						
105.05	86201	4.04	**108.10**	**2133895**	**100.00**	111.10	23399	1.10
106.15	18450	0.86	109.10	353748	16.58	112.10	937	0.04
107.10	1408300	66.00	**110.10**	**292305**	**13.70**			

(d) Cyclic Voltammogram Curves (Figure S1)

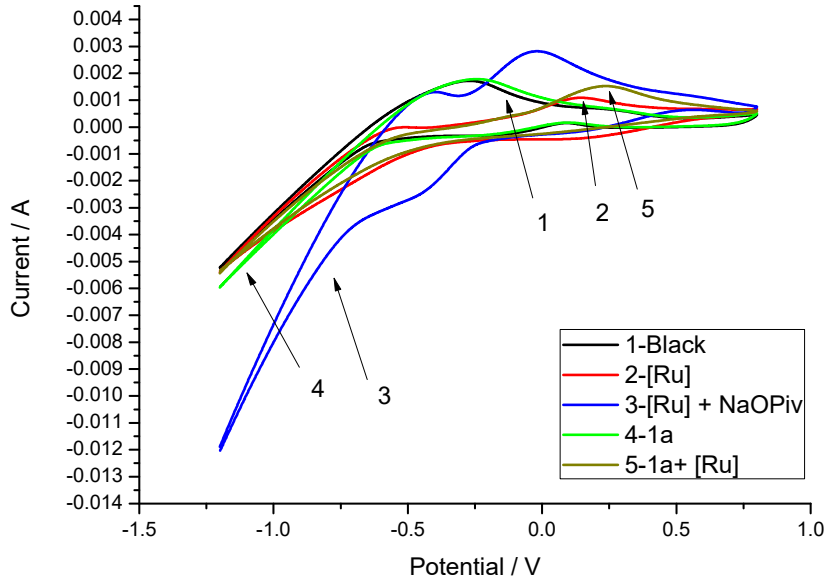
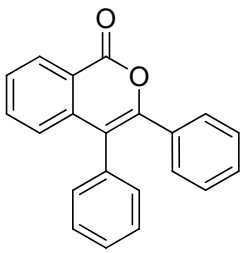


Figure S1. Cyclic voltammogram curves. Using Pt slice as working electrode, Pt slice and Ag/AgCl as counter and reference electrode at 100 mV/s scan rate.

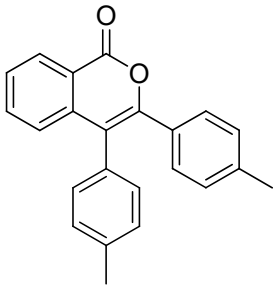
(B) Analytical data

3,4-Diphenyl-1*H*-isochromen-1-one (3aa)[1-2]:



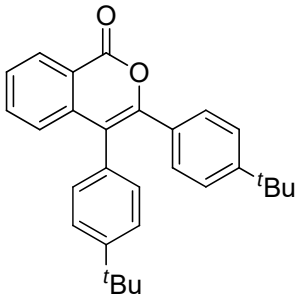
White solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.40-8.39 (m, 1H), 7.64 (t, *J* = 7.5 Hz, 1H), 7.52 (t, *J* = 7.5 Hz, 1H), 7.44-7.40 (m, 3H), 7.33 (d, *J* = 7.0 Hz, 2H), 7.27-7.25 (m, 2H), 7.23-7.18 (m, 4H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 162.3, 150.9, 138.9, 134.7, 134.3, 132.9, 131.3, 129.6, 129.2, 129.1, 129.0, 128.2, 128.1, 127.9, 125.4, 120.4, 116.9; LRMS (EI, 70 eV) *m/z* (%): 298 (M^+, 100), 270 (28), 221 (36), 165 (28), 105 (82).

3,4-Di-*p*-tolyl-1*H*-isochromen-1-one (3ab)[2]:



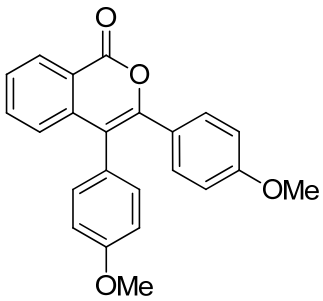
Light yellow solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.39 (d, *J* = 7.5 Hz, 1H), 7.61 (t, *J* = 7.5 Hz, 1H), 7.49 (t, *J* = 7.5 Hz, 1H), 7.25-7.22 (m, 4H), 7.19 (d, *J* = 8.0 Hz, 1H), 7.14 (d, *J* = 8.0 Hz, 2H), 7.01 (d, *J* = 8.0 Hz, 2H), 2.42 (s, 3H), 2.29 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 162.5, 151.0, 139.3, 139.0, 137.8, 134.6, 131.4, 131.0, 130.2, 129.8, 129.5, 129.1, 128.6, 127.8, 125.3, 120.3, 116.3, 21.4, 21.3; LRMS (EI, 70 eV) *m/z* (%): 326 (M^+, 100), 298 (48), 235 (23), 178 (17), 119 (50).

3,4-bis(4-(tert-butyl)phenyl)-1*H*-isochromen-1-one (3ac):



Light yellow solid, mp 165.0-165.8 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.39 (d, *J* = 8.0 Hz, 1H), 7.64-7.61 (m, 1H), 7.49 (t, *J* = 7.0 Hz, 1H), 7.45 (d, *J* = 8.5 Hz, 2H), 7.28-7.27 (m, 2H), 7.21-7.18 (m, 5H), 1.38 (s, 9H), 1.26 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 162.5, 152.0, 151.2, 150.7, 139.4, 134.5, 131.4, 130.8, 130.1, 129.4, 128.8, 127.8, 126.0, 125.5, 124.8, 120.3, 116.4, 34.7 (2C), 31.4, 31.1; LRMS (EI, 70 eV) *m/z* (%): 410 (M^+, 99), 354 (100), 297 (12), 221 (18), 161 (34); HRMS *m/z* (ESI) calcd for $C_{29}H_{31}O_2$ $[M+H]^+$ 411.2324, found 411.2341.

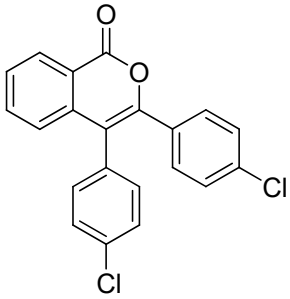
3,4-Bis(4-methoxyphenyl)-1*H*-isochromen-1-one (3ad)[3]:



Light yellow solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.37 (d, *J* = 8.0 Hz, 1H), 7.62 (t, *J* = 7.0 Hz, 1H), 7.48 (t, *J* = 7.0 Hz, 1H), 7.31-7.28 (m, 2H), 7.20 (d, *J* = 8.0 Hz, 1H), 7.18-7.15 (m, 2H), 6.98-6.95 (m, 2H), 6.74-6.71 (m, 2H), 3.86 (s, 3H), 3.77 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 162.5, 159.9, 159.3, 150.9, 139.5, 134.6, 132.4, 130.7, 129.5, 127.7, 126.6, 125.4,

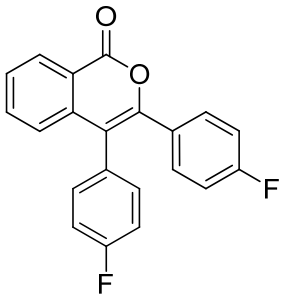
125.2, 120.2, 115.4, 114.6, 113.3, 76.8, 55.3, 55.2; LRMS (EI, 70 eV) *m/z* (%): 410 (M^+, 99), 354 (100), 297 (12), 221 (18), 161 (34).

3,4-Bis(4-chlorophenyl)-1*H*-isochromen-1-one (3ae)[2]:



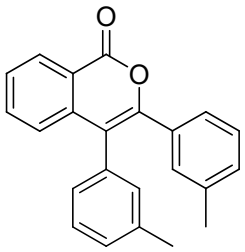
Light yellow solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.39 (t, *J* = 7.0 Hz, 1H), 7.68-7.63 (m, 1H), 7.56-7.53 (m, 1H), 7.42 (t, *J* = 6.5 Hz, 2H), 7.26-7.16 (m, 7H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 161.8, 150.1, 138.2, 135.3, 134.9, 134.5, 132.5, 131.1, 130.5, 129.8, 129.6, 128.5, 128.4, 125.1, 120.4, 116.1; LRMS (EI, 70 eV) *m/z* (%): 366 (M^+, 100), 338 (54), 303 (37), 239 (44), 139 (83).

3,4-Bis(4-fluorophenyl)-1*H*-isochromen-1-one (3af)[2]:



Yellow solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.40 (dd, *J* = 7.0, 1.0 Hz, 1H), 7.68-7.65 (m, 1H), 7.56-7.53 (m, 1H), 7.3-7.29 (m, 2H), 7.25-7.21 (m, 2H), 7.18-7.12 (m, 3H), 6.94-6.90 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 163.8, 163.56, 162.0, 161.8, 161.6, 150.3, 138.6, 134.8, 133.0, 132.9, 131.3, 131.2, 130.1, 130.0, 129.7, 128.9 (2C), 128.3, 125.1, 120.4, 116.5, 116.3, 115.83, 115.3, 115.1; ^{19}F NMR (471 MHz, $CDCl_3$) δ -110.77, -112.85; LRMS (EI, 70 eV) *m/z* (%): 334 (M^+, 100), 306 (70), 277 (22), 183 (31), 123 (64).

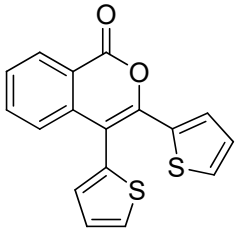
3,4-Di-*m*-tolyl-1*H*-isochromen-1-one (3ag)[2]:



Yellow solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.40 (dd, *J* = 8.0, 1.0 Hz, 1H), 7.65-7.62 (m, 1H), 7.53-7.50 (m, 1H), 7.32-7.29 (m, 2H), 7.21 (t, *J* = 8.0 Hz, 2H), 7.08 (s, 1H), 7.08-7.03 (m, 4H), 2.35 (s, 3H), 2.24 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 162.5, 150.9, 139.1, 138.7, 137.5, 134.6, 134.3, 132.8, 131.7, 129.7 (2C), 129.5, 128.9, 128.8,

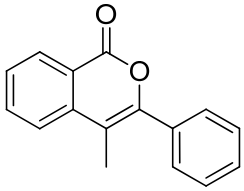
128.3, 128.0, 127.6, 126.4, 125.5, 120.4, 116.9, 21.5, 21.4; LRMS (EI, 70 eV) *m/z* (%): 326 (M^+, 99), 298 (39), 235 (34), 178 (25), 119 (100).

3,4-Di(thiophen-2-yl)-1*H*-isochromen-1-one (3ah):



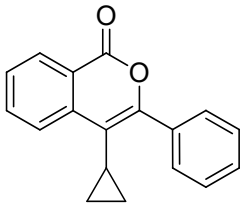
Yellow solid, mp 183.6-184.4 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.34 (d, *J* = 7.5 Hz, 1H), 7.67-7.64 (m, 2H), 7.49 (t, *J* = 7.5 Hz, 1H), 7.44-7.42 (m, 1H), 7.34-7.33 (m, 1H), 7.29-7.27 (m, 1H), 7.15 (d, *J* = 8.0 Hz, 1H), 7.13 (d, *J* = 2.5 Hz, 1H), 6.99 (t, *J* = 4.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 161.2, 148.2, 139.7, 135.0, 134.6, 133.8, 130.8, 130.0, 129.5, 129.5, 129.0, 128.3, 128.0, 127.0, 125.2, 119.7, 107.2; LRMS (EI, 70 eV) *m/z* (%): 310 (M^+, 83), 282 (100), 253 (58), 221 (28), 111 (33); HRMS *m/z* (ESI) calcd for $C_{17}H_{11}O_2S_2[M+H]^+$ 311.0200, found 311.0213.

4-Methyl-3-phenyl-1*H*-isochromen-1-one (3ai)[4]:



White solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.38 (d, *J* = 7.0 Hz, 1H), 7.82-7.79 (m, 1H), 7.64 (d, *J* = 8.0 Hz, 1H), 7.59 (d, *J* = 6.5 Hz, 2H), 7.55 (t, *J* = 8.0 Hz, 1H), 7.49-7.43 (m, 3H), 2.32 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 162.6, 151.2, 138.8, 134.8, 133.3, 129.8, 129.5, 129.4, 128.3, 128.0, 123.4, 120.8, 109.2, 13.61; LRMS (EI, 70 eV) *m/z* (%): 236 (M^+, 96), 208 (100), 178 (19), 105 (31), 77 (55).

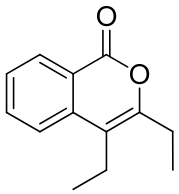
4-Cyclopropyl-3-phenyl-1*H*-isochromen-1-one (3aj)[3]:



Light yellow solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.33 (dd, *J* = 8.0, 1.0 Hz, 1H), 8.12 (d, *J* = 8.5 Hz, 1H), 7.82- 7.78 (m, 1H), 7.75-7.73 (m, 2H), 7.53 (t, *J* = 8.0 Hz, 1H), 7.48-7.42 (m, 3H), 1.94-1.89 (m, 1H), 0.96-0.93 (m, 2H), 0.21-0.17 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 162.5, 153.5, 139.7, 134.5, 133.2, 129.5 (2C), 129.4, 127.8 (2C), 124.6,

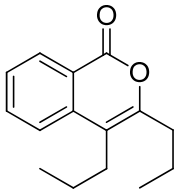
120.6, 114.6, 9.9, 9.1; LRMS (EI, 70 eV) *m/z* (%): 262 (M^+, 75), 233 (22), 217 (52), 185 (61), 105 (69).

3,4-diethyl-1H-isochromen-1-one (3ak)[2]:



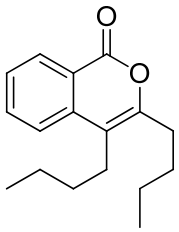
White solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.32 (dd, *J* = 8.0, 1.0 Hz, 1H), 7.76-7.72 (m, 1H), 7.55 (d, *J* = 8.0 Hz, 1H), 7.48-7.45 (m, 1H), 2.68-2.60 (m, 4H), 1.29 (t, *J* = 7.5 Hz, 3H), 1.21 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 163.0, 155.0, 137.8, 134.6, 129.9, 127.1, 122.5, 120.9, 113.1, 24.1, 19.3, 14.3, 12.6; LRMS (EI, 70 eV) *m/z* (%): 202 (M^+, 100), 187 (99), 259 (25), 131 (87), 115 (42).

3,4-Dipropyl-1*H*-isochromen-1-one (3al)[2]:



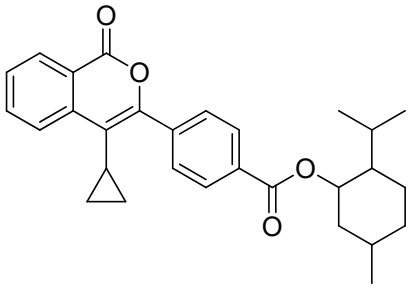
Colourless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.32 (d, *J* = 8.0 Hz, 1H), 7.75-7.72 (m, 1H), 7.53 (d, *J* = 8.0 Hz, 1H), 7.46 (t, *J* = 7.5 Hz, 1H), 2.62-2.56 (m, 4H), 1.77-1.73 (m, 2H), 1.62-1.57 (m, 2H), 1.04 (t, *J* = 7.5 Hz, 3H), 1.00 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 163.0, 154.2, 138.0, 134.6, 129.9, 127.1, 122.7, 120.8, 112.3, 32.7, 28.2, 22.9, 21.2, 14.2, 13.9; LRMS (EI, 70 eV) *m/z* (%): 230 (M^+, 52), 201 (100), 173 (12), 145 (18), 131 (64).

3,4-Dibutyl-1*H*-isochromen-1-one (3am)[4]:



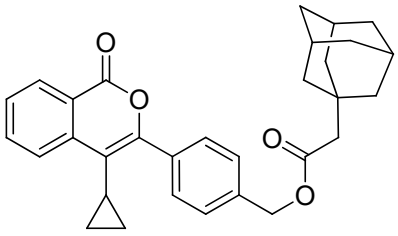
Colourless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.32 (dd, *J* = 8.0, 1.0 Hz, 1H), 7.75-7.72 (m, 1H), 7.53 (d, *J* = 8.5 Hz, 1H), 7.46 (t, *J* = 8.0 Hz, 1H), 2.62-2.57 (m, 4H), 1.73-1.68 (m, 2H), 1.56-1.51 (m, 2H), 1.48-1.44 (m, 2H), 1.44-1.39 (m, 2H), 1.00-0.94 (m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 163.0, 154.3, 138.0, 134.6, 129.9, 127.1, 122.6, 120.8, 112.3, 31.9, 30.6, 30.0, 26.0, 23.0, 22.5, 14.0, 13.9; LRMS (EI, 70 eV) *m/z* (%): 258 (M^+, 41), 215 (46), 173 (100), 145 (28), 131 (27).

2-Isopropyl-5-methylcyclohexyl-4-(4-cyclopropyl-1-oxo-1*H*-isochromen-3-yl)ben zoate (3ap):



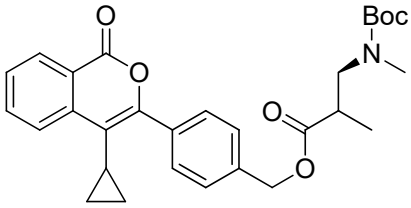
Light yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.34 (d, *J* = 7.5 Hz, 1H), 8.15-8.11 (m, 3H), 7.85-7.81 (m, 3H), 7.56 (t, *J* = 7.5 Hz, 1H), 5.00-4.95 (m, 1H), 2.17 (d, *J* = 11.5 Hz, 1H), 2.02-1.94 (m, 2H), 1.75 (d, *J* = 10.5 Hz, 2H), 1.62-1.58 (m, 2H), 1.19-1.11 (m, 2H), 1.00-0.94 (m, 9H), 0.82 (t, *J* = 7.0 Hz, 3H), 0.22-0.18 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.7, 162.1, 152.3, 139.4, 137.2, 134.7, 131.3, 129.6, 129.5, 129.0, 128.3, 124.7, 120.7, 115.9, 75.2, 47.3, 41.0, 34.3, 31.5, 26.5, 23.6, 22.1, 20.8, 16.6, 10.1 (2C), 9.1; LRMS (EI, 70 eV) *m/z* (%): 444 (M^+, 5), 307 (42), 289 (100), 261 (31), 138 (30); HRMS *m/z* (ESI) calcd for $C_{29}H_{33}O_4$ $[M+H]^+$ 445.2379, found 445.2388.

4-(4-cyclopropyl-1-oxo-1H-isochromen-3-yl)benzyl-2-((3r,5r,7r)-adamantan-1-yl) acetate (3aq):



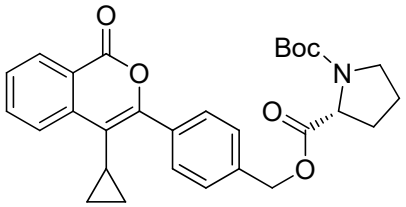
White solid, mp 131.0-131.3 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.33 (dd, *J* = 8.0, 1.0 Hz, 1H), 8.12 (d, *J* = 8.0 Hz, 1H), 7.82-7.79 (m, 1H), 7.75 (d, *J* = 8.0 Hz, 2H), 7.53 (t, *J* = 7.5 Hz, 1H), 7.44 (d, *J* = 8.5 Hz, 2H), 5.17 (s, 2H), 2.16 (s, 2H), 1.97 (s, 3H), 1.94-1.89 (m, 1H), 1.71-1.66 (m, 3H), 1.63-1.61 (m, 9H), 0.98-0.94 (m, 2H), 0.21-0.18 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.6, 162.4, 153.0, 139.7, 137.5, 134.6, 132.9, 129.7, 129.5, 127.9, 127.6, 124.6, 120.6, 114.8, 65.3, 48.9, 42.4, 36.7, 33.0, 28.6, 10.0, 9.0; LRMS (EI, 70 eV) *m/z* (%): 468 (M^+, 6), 450 (5), 292 (16), 274 (100), 246 (31); HRMS *m/z* (ESI) calcd for $C_{31}H_{33}O_4$ $[M+H]^+$ 469.2379, found 469.2394.

4-(4-cyclopropyl-1-oxo-1*H*-isochromen-3-yl)benzyl-3-((tert-butoxycarbonyl)(methyl)amino)-2-methylpropanoate (3ar):



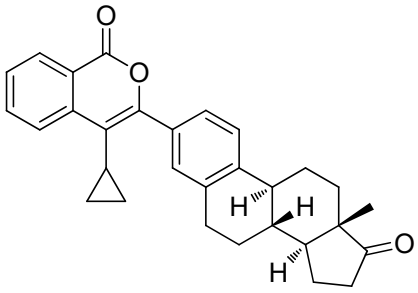
Light yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.33 (d, *J* = 7.5 Hz, 1H), 8.12 (d, *J* = 8.0 Hz, 1H), 7.81 (t, *J* = 7.5 Hz, 1H), 7.75 (s, 2H), 7.53 (t, *J* = 7.5 Hz, 1H), 7.42 (d, *J* = 7.0 Hz, 2H), 5.23 (s, 2H), 4.93-4.90 (m, 0.5H), 4.57-4.52 (m, 0.5H), 2.89-2.81 (m, 3H), 1.95-1.90 (m, 1H), 1.47-1.42 (m, 12H), 0.98-0.94 (m, 2H), 0.21-0.18 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 172.2, 162.3, 156.0, 155.3, 152.9, 139.6, 137.0, 134.6, 133.0, 129.7, 129.5, 127.9, 127.3, 127.2, 124.6, 120.6, 114.8, 80.4, 80.1, 66.2, 55.2, 53.7, 31.1, 30.6, 28.4, 15.3, 14.8, 10.0, 9.0; HRMS *m/z* (ESI) calcd for $C_{29}H_{34}NO_6$ $[M+H]^+$ 492.2386, found 492.2368.

(R)-1-Tert-butyl 2-(4-(4-cyclopropyl-1-oxo-1*H*-isochromen-3-yl)benzyl) pyrrolidine-1,2-dicarboxylate (3as):



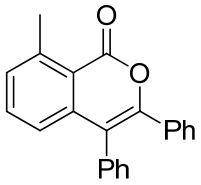
Light yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.34 (d, *J* = 8.0 Hz, 1H), 8.13 (d, *J* = 8.0 Hz, 1H), 7.81 (t, *J* = 7.5 Hz, 1H), 7.77-7.73 (m, 2H), 7.54 (t, *J* = 7.5 Hz, 1H), 7.43 (d, *J* = 8.5 Hz, 2H), 5.34-5.16 (m, 2H), 4.44-4.30 (m, 1H), 3.61-3.38 (m, 2H), 2.23-2.22 (m, 1H), 2.05-1.85 (m, 4H), 1.48-1.46 (m, 4H), 1.38 (s, 5H), 0.98-0.95 (m, 2H), 0.21-0.18 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 173.1, 172.8, 162.3, 154.5, 153.8, 153.0, 152.9, 139.6, 137.2, 136.9, 134.6, 133.2, 133.0, 129.8, 129.7, 129.5, 128.0, 127.9, 127.5, 127.2, 124.6, 120.6, 114.9, 114.8, 66.1 (2C), 59.2, 58.9, 46.6, 46.4, 30.9, 20.0, 28.5, 28.3, 24.4, 23.7, 10.0, 9.0; HRMS *m/z* (ESI) calcd for $C_{29}H_{32}NO_6$ $[M+H]^+$ 490.2230, found 490.2238.

4-Cyclopropyl-3-((8R,9S,13S,14S)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6*H*-cyclopenta[a]phenanthren-3-yl)-1*H*-isochromen-1-one (3at):



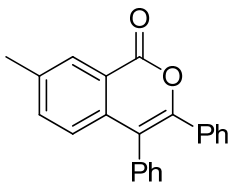
Light yellow solid, mp 225.2-226.1 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.33 (d, *J* = 7.5 Hz, 1H), 8.11 (d, *J* = 8.0 Hz, 1H), 7.80 (t, *J* = 8.0 Hz, 1H), 7.55-7.51 (m, 3H), 7.34 (d, *J* = 8.0 Hz, 1H), 2.99-2.97 (m, 2H), 2.56-2.46 (m, 2H), 2.40-2.35 (m, 1H), 2.21-2.05 (m, 3H), 2.02-1.99 (m, 1H), 1.95-1.90 (m, 1H), 1.75-1.66 (m, 2H), 1.60- 1.48 (m, 4H), 1.00-0.95 (m, 5H), 0.26-0.21 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 220.9, 162.6, 153.6, 141.2, 139.9, 136.1, 134.5, 130.6, 129.8, 129.5, 127.7, 127.1, 124.6, 124.5, 120.5, 114.2, 50.6, 48.0, 44.6, 38.0, 35.9, 31.6, 29.4, 26.5, 25.6, 21.6, 13.9, 10.2, 10.1, 9.2; HRMS *m/z* (ESI) calcd for $C_{30}H_{31}O_3$ $[M+H]^+$ 439.2273, found 439.2280.

8-Methyl-3,4-diphenyl-1*H*-isochromen-1-one (3ba)[1]:



Light yellow solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45 (t, *J* = 7.5 Hz, 1H), 7.42-7.38 (m, 3H), 7.32-7.29 (m, 2H), 7.25-7.23 (m, 2H), 7.22-7.16 (m, 3H), 7.00 (d, *J* = 8.0 Hz, 1H), 2.91 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 161.6, 150.6, 143.5, 140.5, 135.0, 133.74, 133.0, 131.4, 131.1, 129.1, 129.1, 128.8, 128.0, 127.8, 123.6, 118.9, 117.0, 23.60; LRMS (EI, 70 eV) *m/z* (%): 312 (M^+, 100), 284 (26), 235 (24), 178 (23), 105 (77).

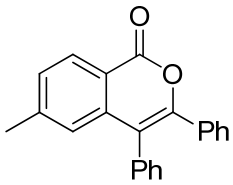
7-Methyl-3,4-diphenyl-1*H*-isochromen-1-one (3ca)[1,2]:



Light yellow solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.21 (s, 1H), 7.45-7.38 (m, 4H), 7.33-7.31 (m, 2H), 7.25-7.23 (m, 2H), 7.22-7.16 (m, 3H), 7.09 (d, *J* = 8.0 Hz, 1H), 2.47 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 162.5, 150.1, 138.5, 136.4, 135.9, 134.5, 133.0, 131.2,

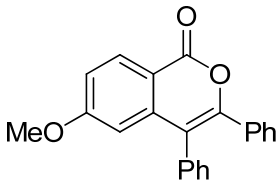
129.3, 129.2, 129.0, 128.8, 128.1, 127.9, 125.4, 120.3, 116.9, 21.28; LRMS (EI, 70 eV) *m/z* (%): 312 (M^+, 100), 284 (22), 235 (36), 178 (23), 105 (73).

6-Methyl-3,4-diphenyl-1*H*-isochromen-1-one (3da)[1,2]:



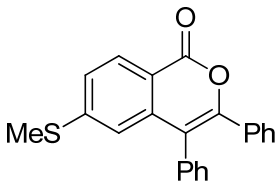
White solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.29 (d, *J* = 8.0 Hz, 1H), 7.44-7.40 (m, 3H), 7.35-7.30 (m, 3H), 7.26-7.23 (m, 2H), 7.23-7.21 (m, 1H), 7.20-7.16 (m, 2H), 6.97 (s, 1H), 2.37 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 162.4, 151.0, 145.8, 138.9, 134.4, 133.1, 131.3, 129.6, 129.5, 129.2, 129.1, 128.9, 128.1, 127.8, 125.3, 118.1, 116.9, 22.24; LRMS (EI, 70 eV) *m/z* (%): 312 (M^+, 100), 284 (25), 235 (34), 178 (19), 105 (57).

6-Methoxy-3,4-diphenyl-1*H*-isochromen-1-onee (3ea)[2]:



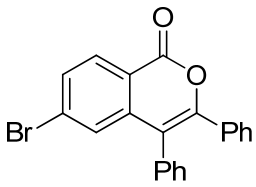
White solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.34 (d, *J* = 8.5 Hz, 1H), 7.42-7.38 (m, 3H), 7.33-7.31 (m, 2H), 7.26-7.24 (m, 2H), 7.23-7.21 (m, 1H), 7.20-7.16 (m, 2H), 7.06 (dd, *J* = 8.5, 2.0 Hz, 1H), 6.58 (d, *J* = 2.5 Hz, 1H), 3.75 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.7, 162.1, 151.5, 141.2, 134.4, 133.0, 131.9, 131.2, 129.3, 129.1, 129.0, 128.2, 127.8, 116.8, 115.7, 113.67, 108.5, 55.5; LRMS (EI, 70 eV) *m/z* (%): 328 (M^+, 100), 300 (21), 251 (36), 152 (20), 105 (60).

6-(Methylthio)-3,4-diphenyl-1*H*-isochromen-1-one (3fa)[4]:



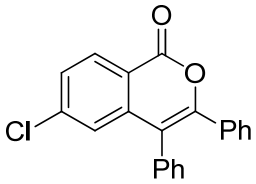
White solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.27 (d, *J* = 8.5 Hz, 1H), 7.44-7.40 (m, 3H), 7.34-7.31 (m, 3H), 7.26-7.22 (m, 3H), 7.20-7.17 (m, 2H), 6.92 (d, *J* = 1.5 Hz, 1H), 2.38 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 162.1, 151.7, 148.4, 139.1, 134.1, 132.9, 131.2, 129.7, 129.3, 129.1, 129.0, 128.2, 127.9, 124.9, 120.5, 116.7, 116.4, 14.6; LRMS (EI, 70 eV) *m/z* (%): 344 (M^+, 100), 297 (17), 269 (39), 239 (26), 105 (71).

6-Bromo-3,4-diphenyl-1*H*-isochromen-1-one (3ga)[1]:



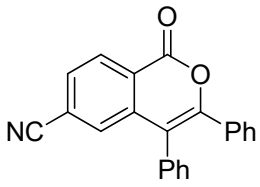
White solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.24 (d, *J* = 8.5 Hz, 1H), 7.64-7.62 (m, 1H), 7.44-7.41 (m, 3H), 7.33-7.30 (m, 3H), 7.25-7.23 (m, 3H), 7.21-7.18 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 161.6, 152.2, 140.4, 133.6, 132.6, 131.5, 131.2, 131.1, 130.5, 129.3, 129.3, 129.3, 129.1, 128.5, 128.1, 127.9, 119.1, 116.0; LRMS (EI, 70 eV) *m/z* (%): 376 (M^+, 45), 348 (8), 299 (15), 163 (36), 105 (100).

6-Chloro-3,4-diphenyl-1*H*-isochromen-1-one (3ha)[2]:



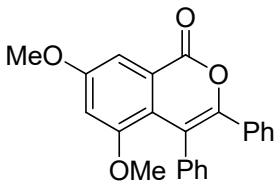
White solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.33 (d, *J* = 8.5 Hz, 1H), 7.47 (dd, *J* = 8.5, 2.0 Hz, 1H), 7.45-7.43 (m, 3H), 7.32 (d, *J* = 7.5 Hz, 2H), 7.25-7.24 (m, 3H), 7.20 (t, *J* = 7.5 Hz, 2H), 7.16 (d, *J* = 1.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ161.5, 152.2, 141.7, 140.4, 133.6, 132.6, 131.3, 131.1, 129.3, 129.30 (2C) , 128.6, 128.5, 127.0, 125.0, 118.7, 116.1; LRMS (EI, 70 eV) *m/z* (%): 332 (M^+, 98), 304 (24), 255 (31), 163 (31), 105 (100).

1-Oxo-3,4-diphenyl-1*H*-isochromene-6-carbonitrile (3ia):



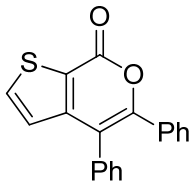
Light yellow solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.49 (d, *J* = 8.0 Hz, 1H), 7.74 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.50 (d, *J* = 1.0 Hz, 1H), 7.48-7.46 (m, 3H), 7.34-7.32 (m, 2H), 7.30-7.27 (m, 1H), 7.25-7.20 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 160.8, 152.8, 139.5, 132.9, 132.1, 131.909, 130.5, 130.2, 129.7 (2C), 129.6, 129.3, 128.9, 128.1, 123.0, 118.2, 117.7, 115.7; HRMS *m/z* (ESI) calcd for $C_{22}H_{14}NO_2$ $[M+H]^+$ 324.1025, found 324.1039.

5,7-Dimethoxy-3,4-diphenyl-1*H*-isochromen-1-one (3ja) [1]:



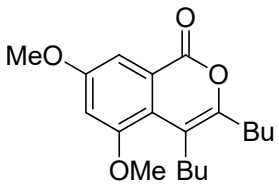
White solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.49 (d, *J* = 2.5 Hz, 1H), 7.25-7.20 (m, 5H), 7.19-7.11 (m, 5H), 6.69 (d, *J* = 2.5 Hz, 1H), 3.93 (s, 3H), 3.33 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 162.5, 160.3, 157.6, 149.0, 137.6, 133.6, 130.7, 129.5, 128.3, 127.6, 127.4, 126.7, 122.9, 122.1, 115.5, 106.9, 102.1, 55.9, 55.8; HRMS *m/z* (ESI) calcd for $C_{23}H_{19}O_4$ $[M+H]^+$ 359.1283, found 359.1296.

4,5-Diphenyl-7*H*-thieno[2,3-c]pyran-7-one (3ka)[1,2]:



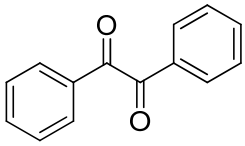
Light yellow solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.78 (d, *J* = 5.0 Hz, 1H), 7.41-7.38 (m, 3H), 7.36-7.35 (m, 2H), 7.29-7.26 (m, 3H), 7.21 (t, *J* = 7.5 Hz, 2H), 6.96 (d, *J* = 5.0 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 158.2, 153.4, 149.5, 136.3, 134.9, 132.4, 130.3, 129.3, 129.2, 129.1, 128.2, 128.0, 125.2, 122.8, 115.9; LRMS (EI, 70 eV) *m/z* (%): 304 (M^+, 100), 276 (18), 227 (43), 171 (18), 105 (48).

3,4-Dibutyl-5,7-dimethoxy-1*H*-isochromen-1-one (3jm):



Colourless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 (d, *J* = 2.5 Hz, 1H), 6.76 (d, *J* = 2.5 Hz, 1H), 3.89 (s, 3H), 3.86 (s, 3H), 2.76 (t, *J* = 6.5 Hz, 2H), 2.56 (t, *J* = 7.5 Hz, 2H), 1.70-1.64 (m, 2H), 1.49-1.37 (m, 6H), 0.98-0.93 (m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ163.1, 159.2, 157.2, 151.3, 123.4, 122.5, 112.4, 106.3, 102.0, 55.8, 55.7, 33.4, 30.4, 30.3, 28.3, 23.1, 22.5, 14.0, 13.9; HRMS *m/z* (ESI) calcd for $C_{19}H_{27}O_4[M+H]^+$ 319.1909, found 319.1924.

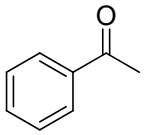
Benzil (4a):



Yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.99-7.97 (m, 4H), 7.68-7.65 (m, 2H), 7.53-7.50 (m, 4H); ^{13}C NMR (125 MHz,

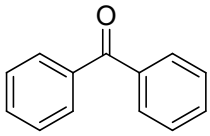
$CDCl_3$) δ 194.6, 135.0, 133.0, 130.0, 129.1.

Acetophenone (5l):



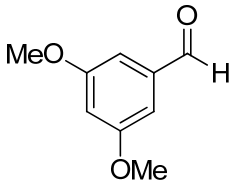
Light yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.02-7.93 (m, 2H), 7.60-7.54 (m, 1H), 7.50-7.43 (m, 2H), 2.61 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ198.2, 137.1, 133.1, 128.6, 128.3, 26.6.

Benzophenone (5m):



White solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.84-7.76 (m, 4H), 7.61-7.54 (m, 2H), 7.51-7.44 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 196.8, 137.6, 132.5, 130.1, 128.3.

3,5-Dimethoxybenzaldehyde (5j):



Yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.91 (s, 1H), 7.01 (d, *J* = 2.5 Hz, 2H), 6.71 (t, *J* = 2.0 Hz, 1H), 3.85 (s, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 192.0, 161.3, 138.4, 107.2, 107.1, 55.7.

(C) References

(1) Y. Qiu, C. Tian, L. Massignan, T. Rogge, L. Ackermann. *Angew. Chem., Int. Ed.* **2018**, *130*, 5920.

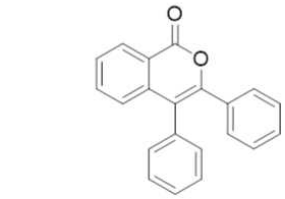
(2) H. Tan, H. Li, J. Wang, L. Wang. *Chem. Eur. J.* **2015**, *21*, 1904.

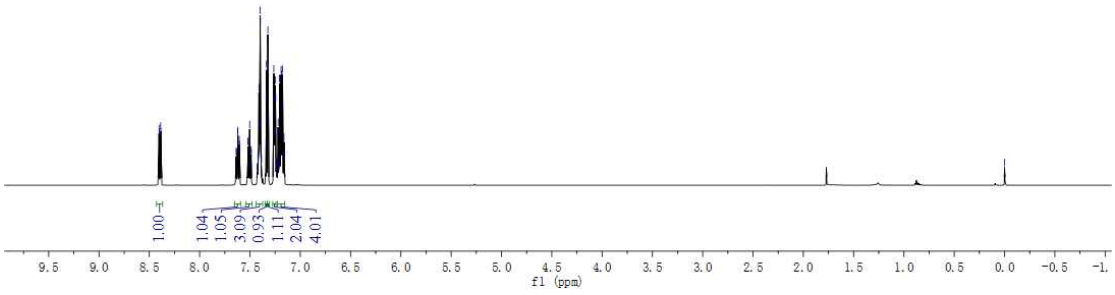
(3) E. Kudo, Y. Shibata, M. Yamazaki, K. Masutomi, Y. Miyauchi, M. Fukui, H. Sugiyama, H. Uekusa, T. Satoh, M. Miura, K. Tanaka. *Chem. Eur. J.* **2016**, *22*, 14190

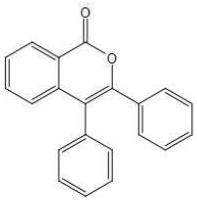
(4) J. Mo, L. Wang, X. Cui. *Org. Lett.* **2015**, *17*, 4960.

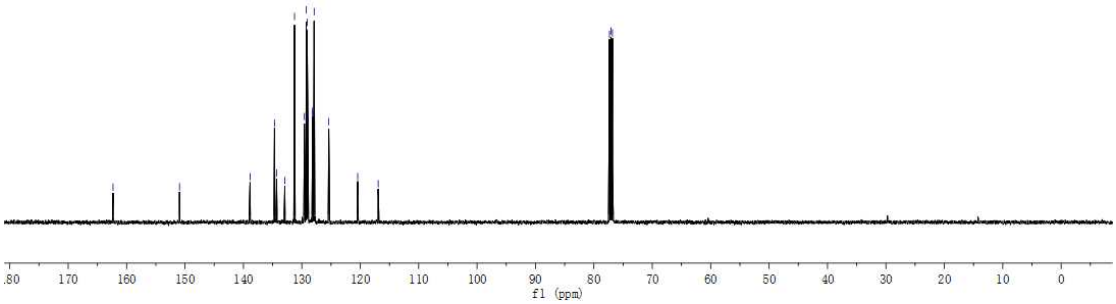
(D) Spectra

3,4-Diphenyl-1*H*-isochromen-1-one (3aa):

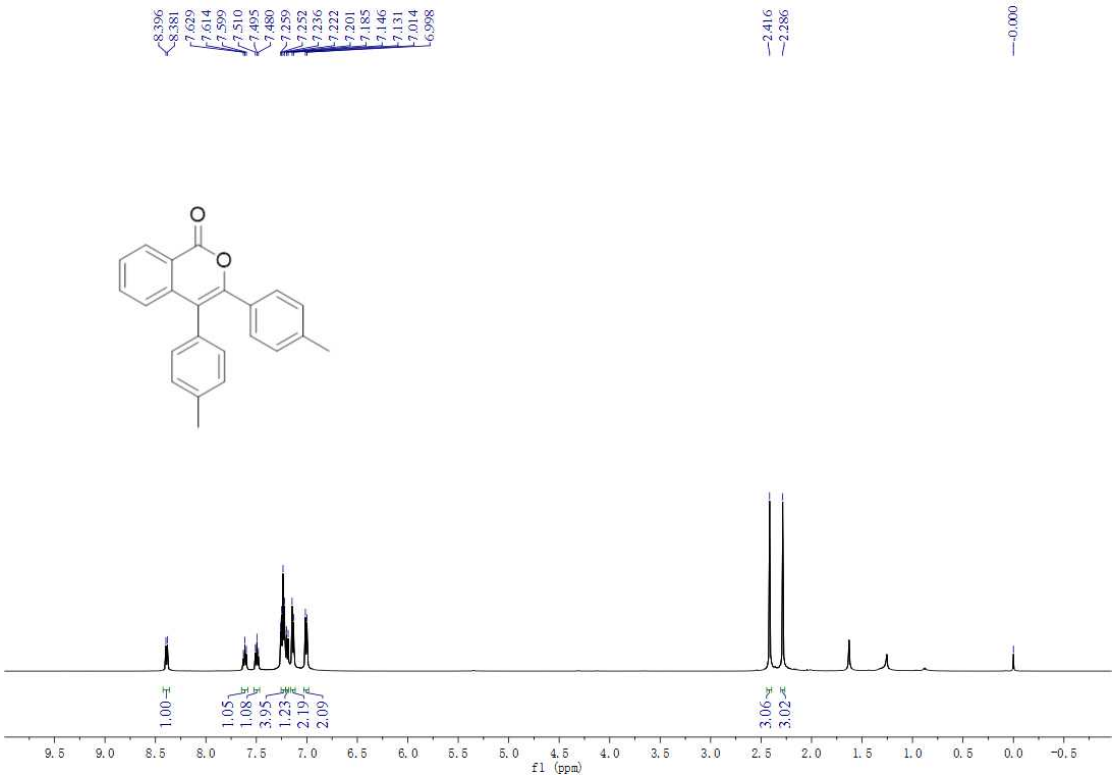


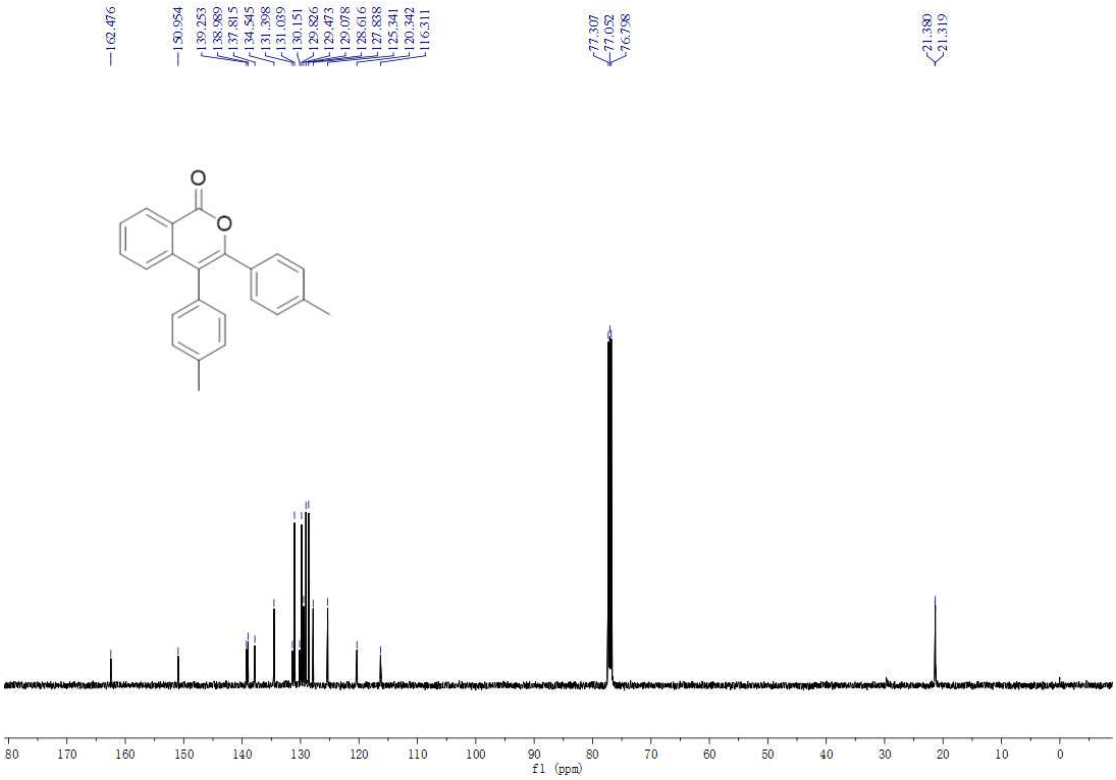




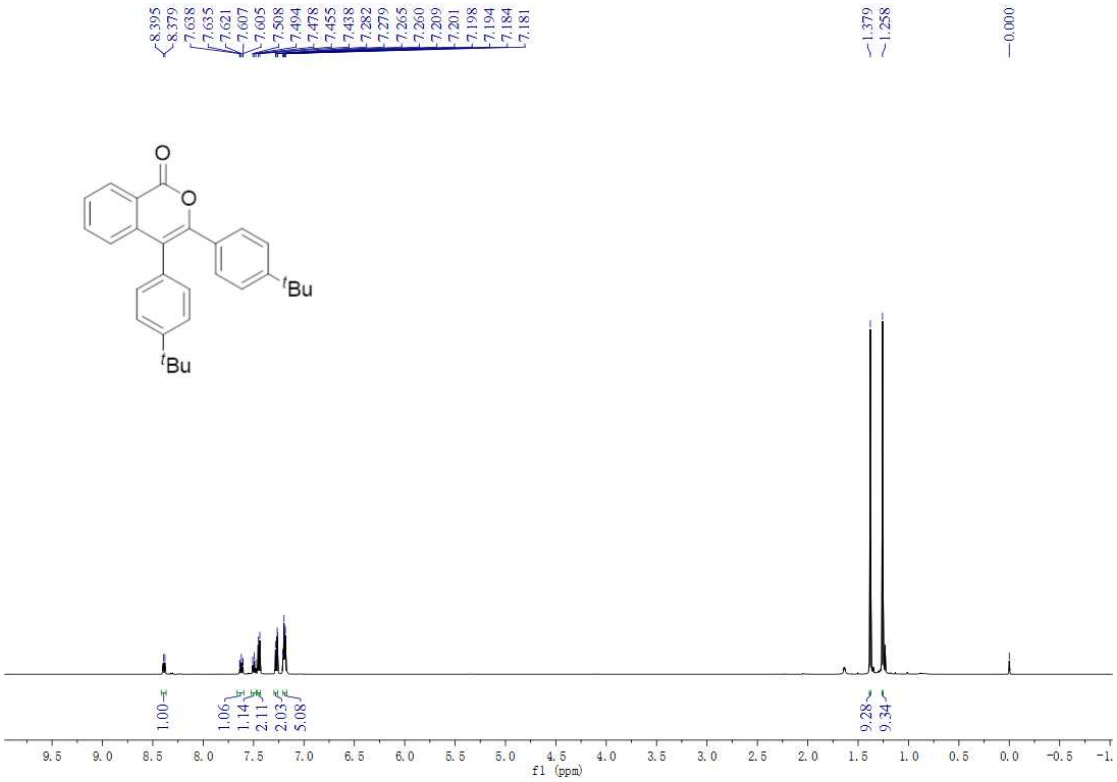


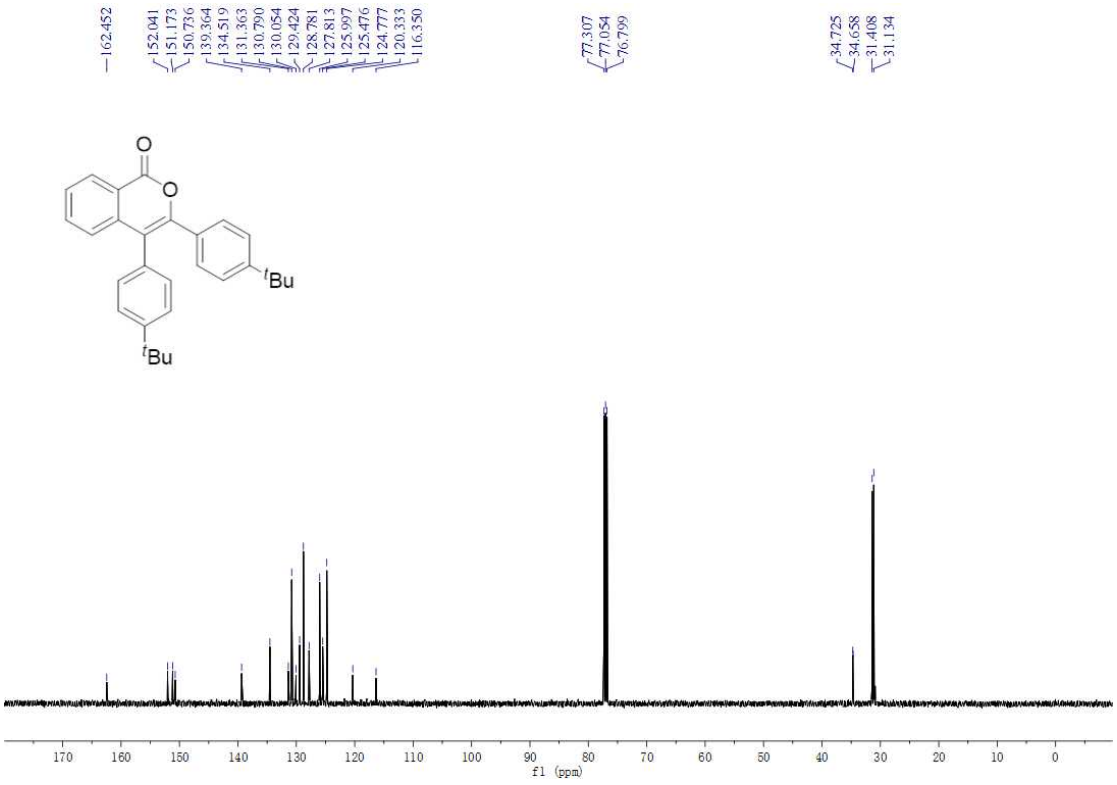
3,4-Di-*p*-tolyl-1*H*-isochromen-1-one (3ab):



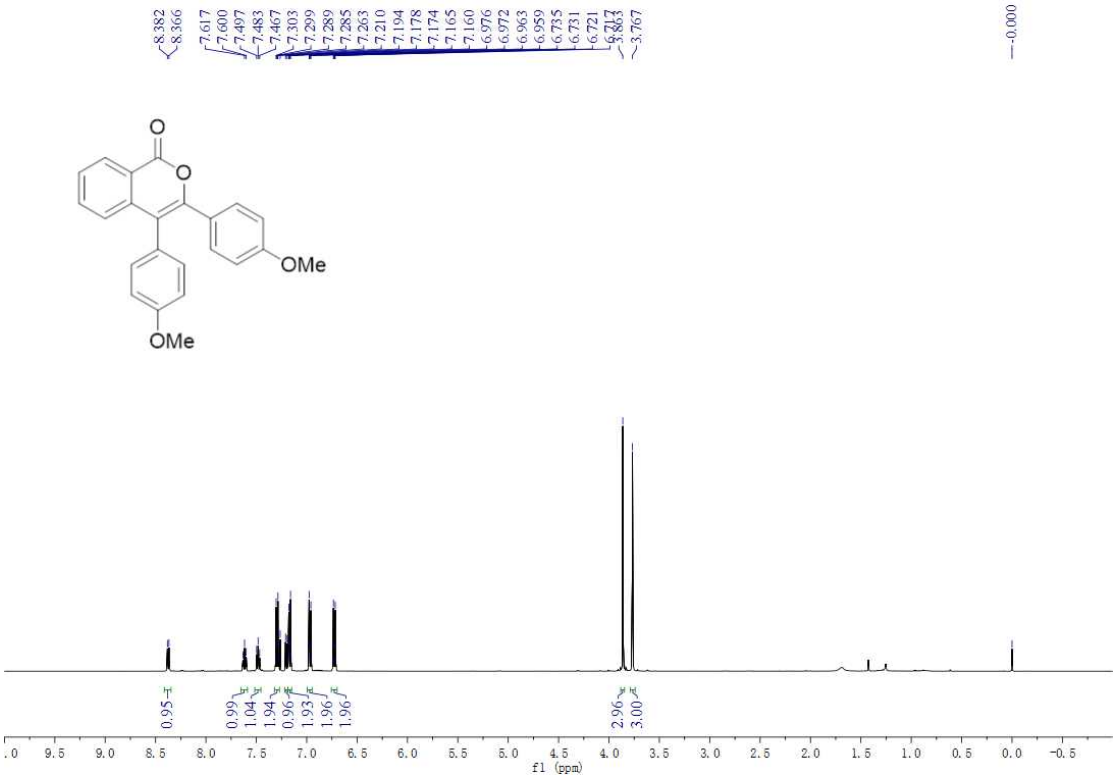


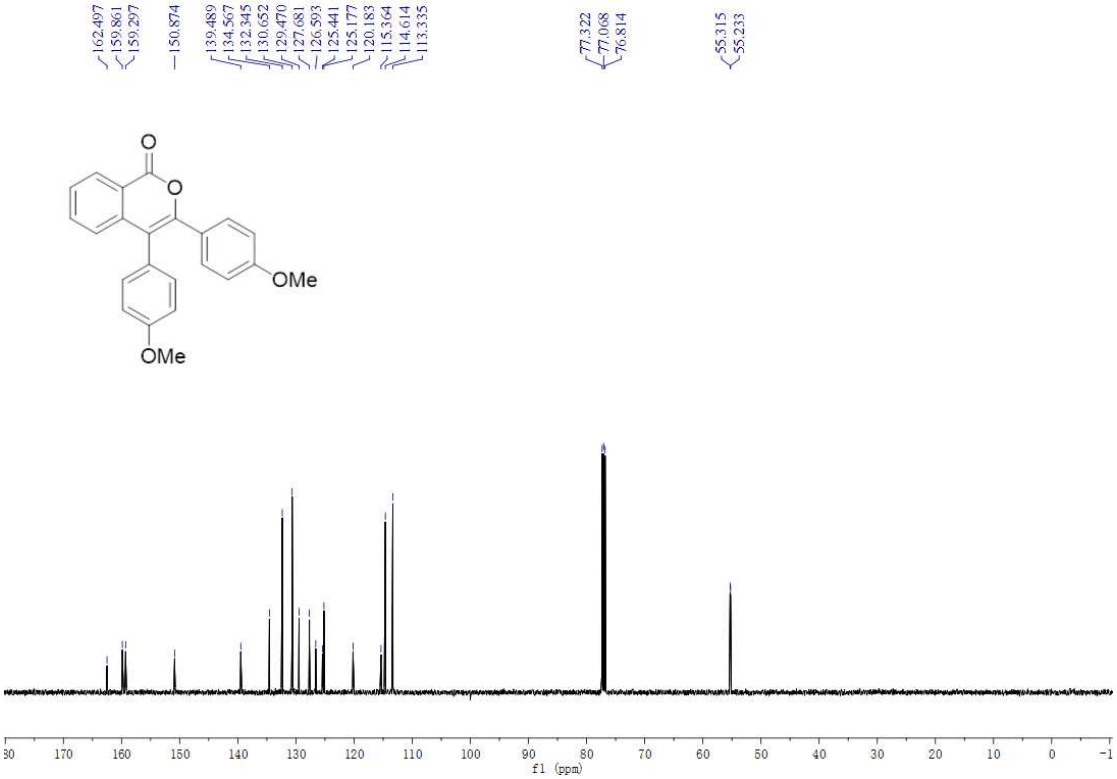
3,4-bis(4-(tert-butyl)phenyl)-1*H*-isochromen-1-one (3ac):



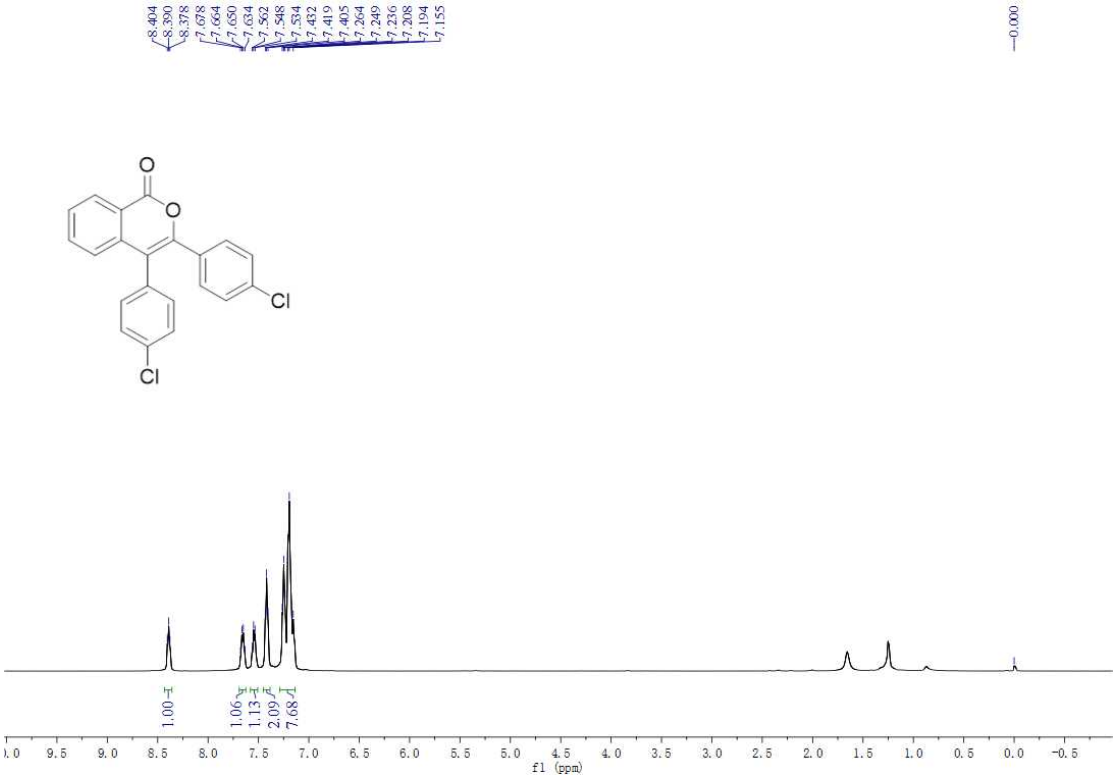


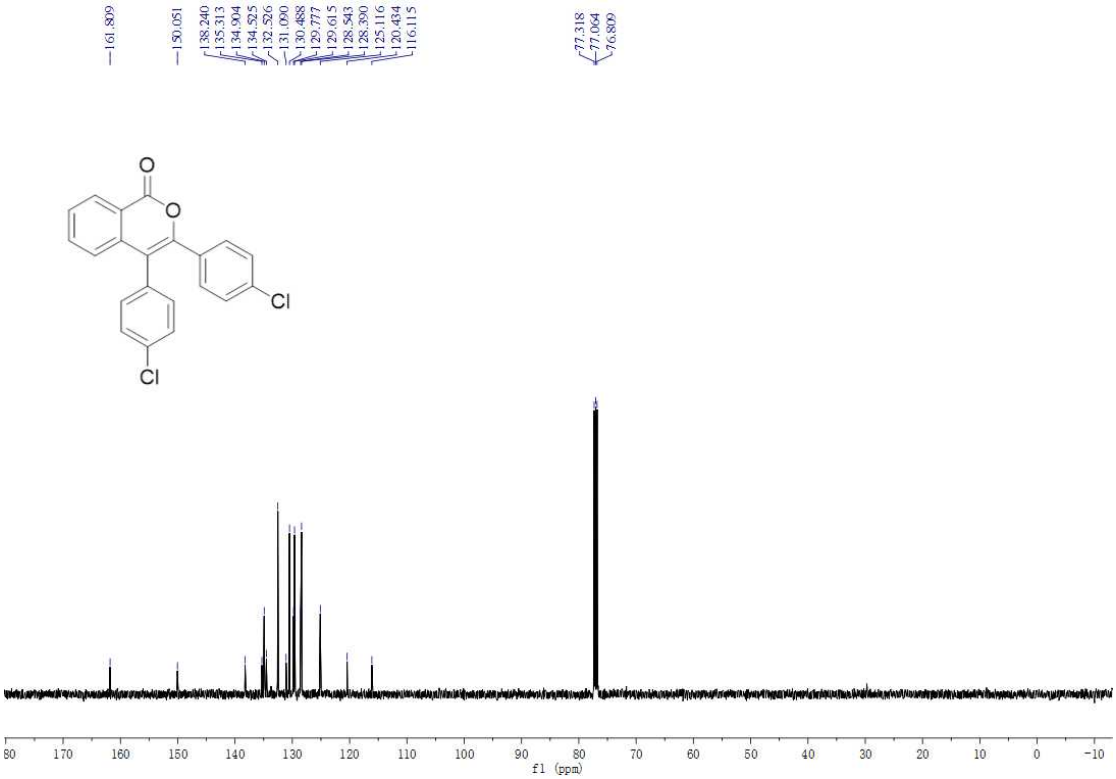
3,4-Bis(4-methoxyphenyl)-1*H*-isochromen-1-one (3ad):



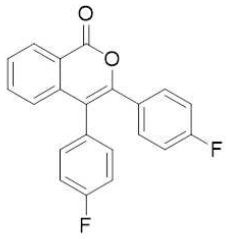


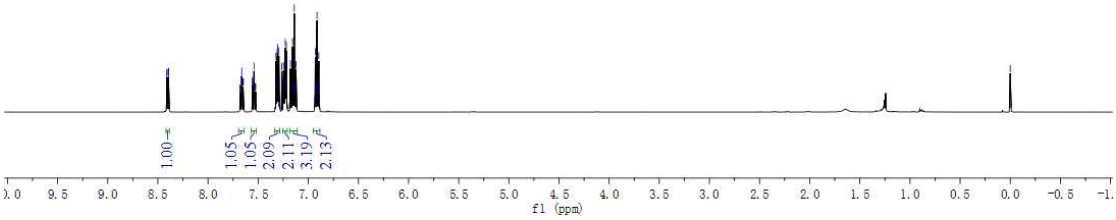
3,4-Bis(4-chlorophenyl)-1*H*-isochromen-1-one (3ae):

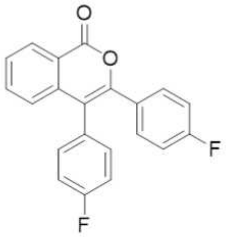


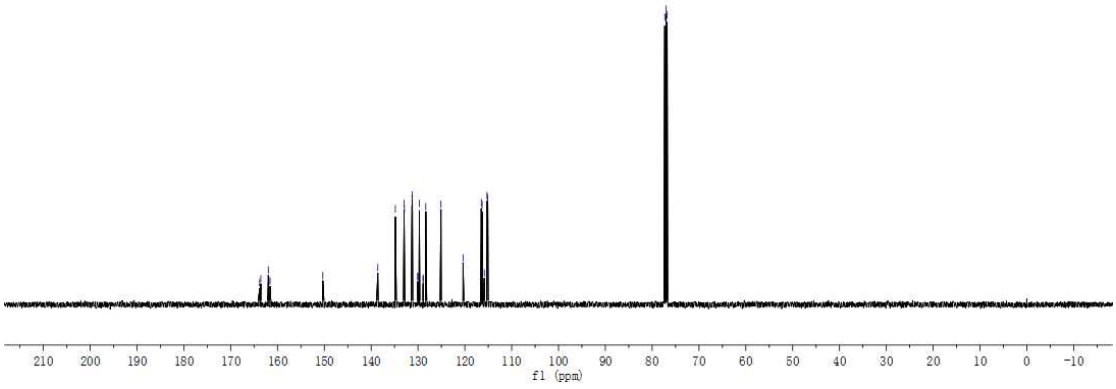


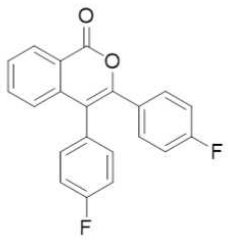
3,4-Bis(4-fluorophenyl)-1*H*-isochromen-1-one (3af):





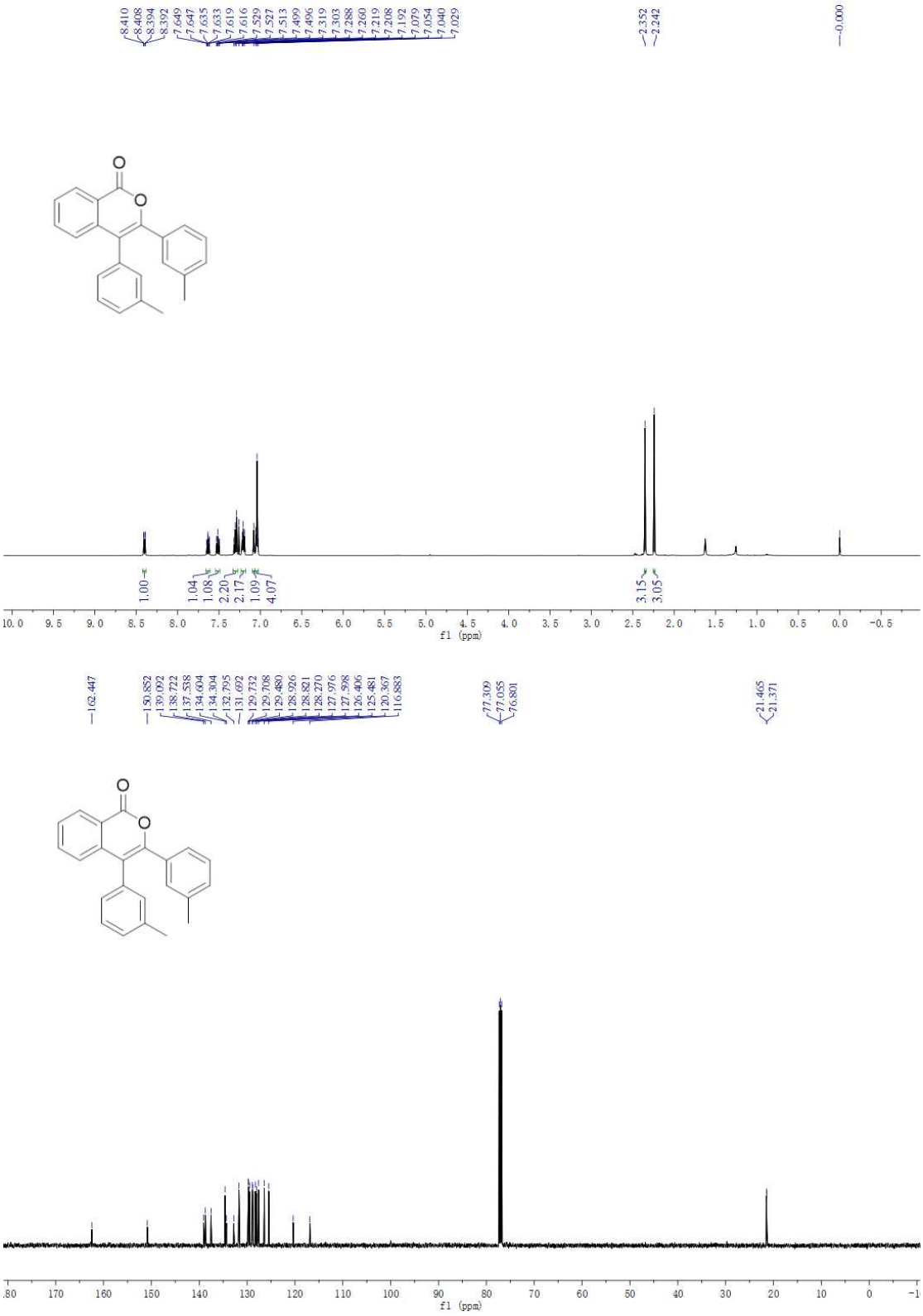




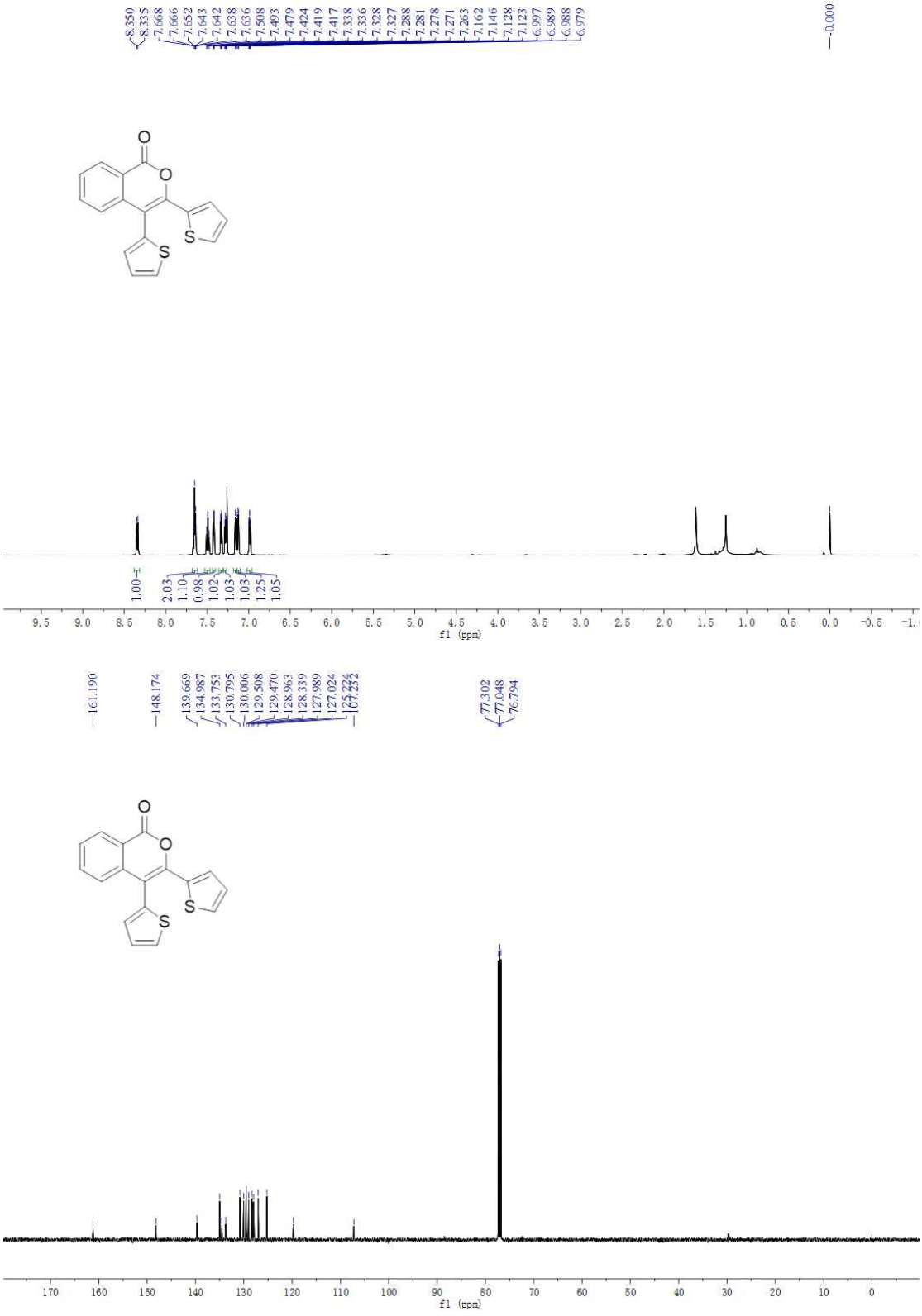

O
O
F
F

-110.766
-112.849
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-19
f1 (ppm)

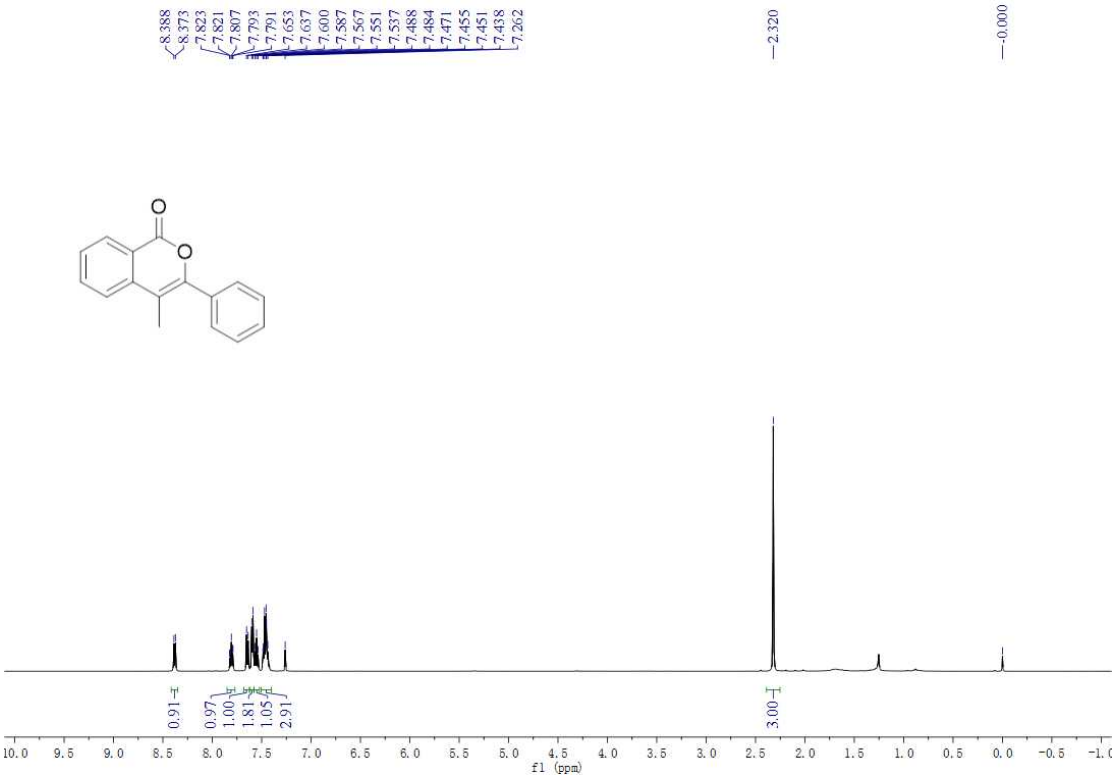
3,4-Di-*m*-tolyl-1*H*-isochromen-1-one (3ag):

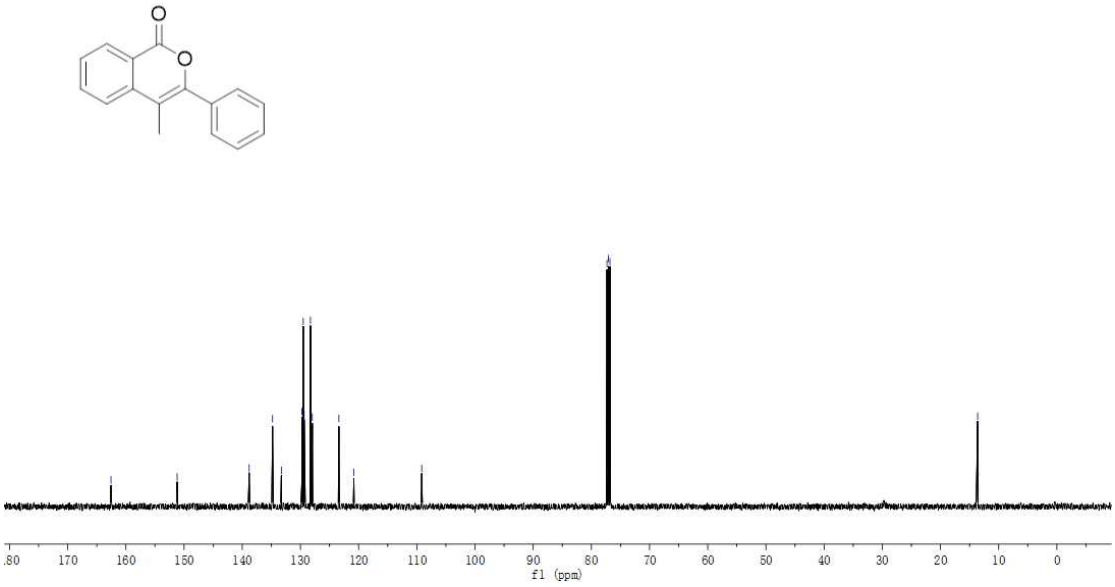


3,4-Di(thiophen-2-yl)-1*H*-isochromen-1-one (3ah):

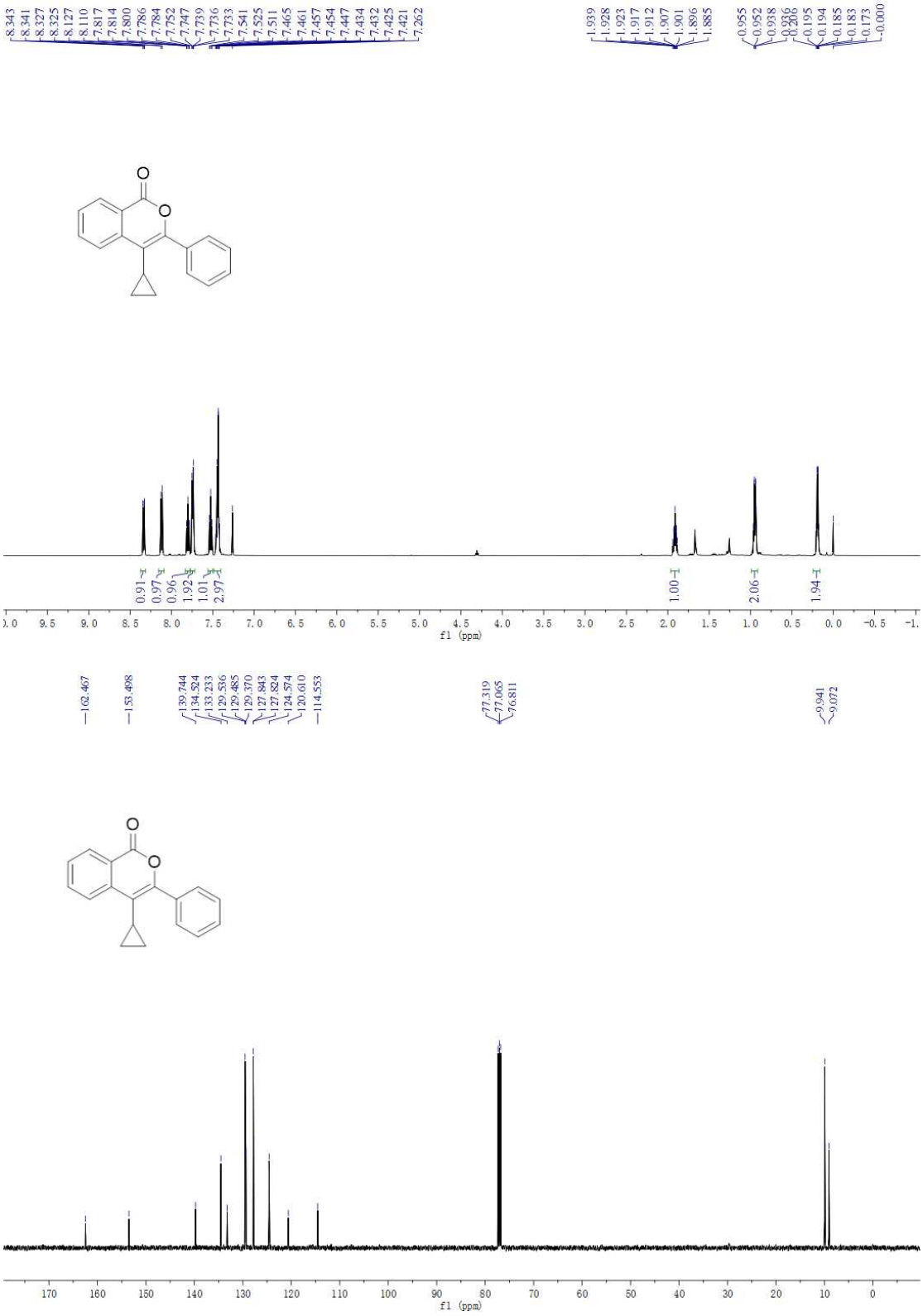


4-Methyl-3-phenyl-1*H*-isochromen-1-one (3ai):

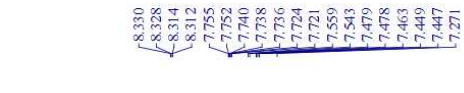


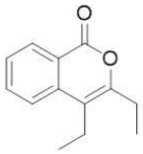


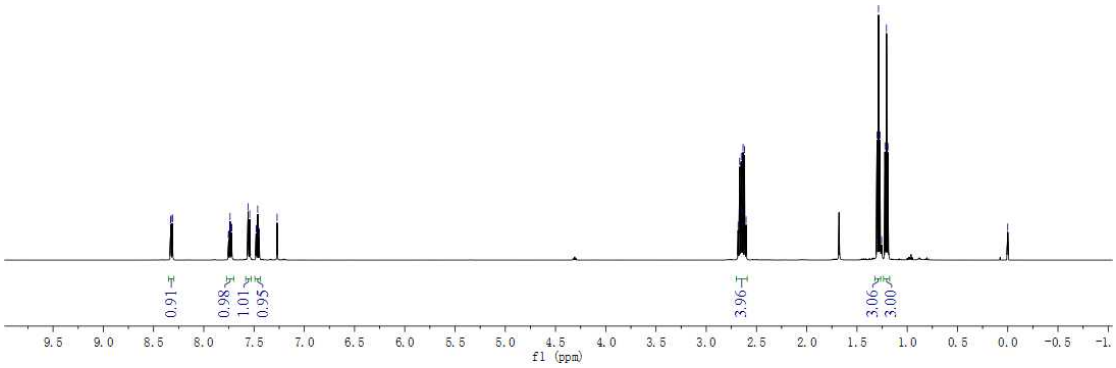
4-Cyclopropyl-3-phenyl-1*H*-isochromen-1-one (3aj):

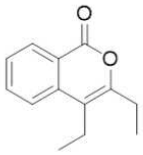


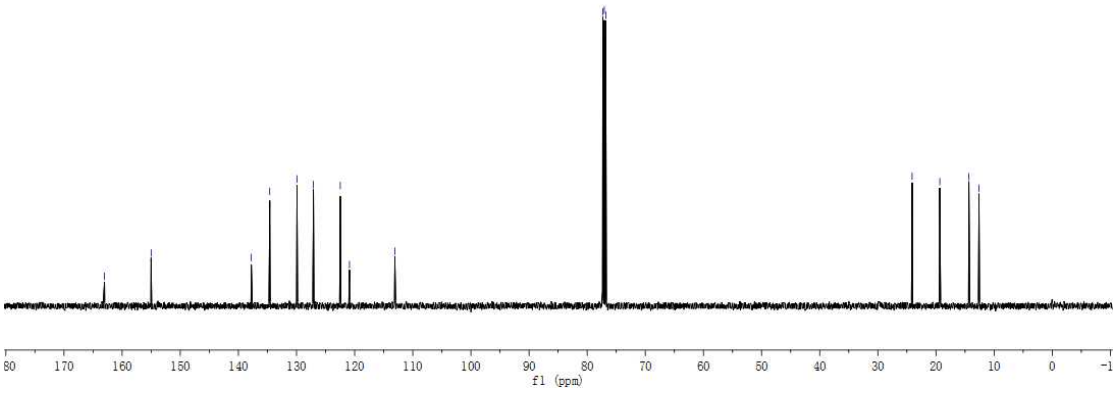
3,4-diethyl-1H-isochromen-1-one (3ak):



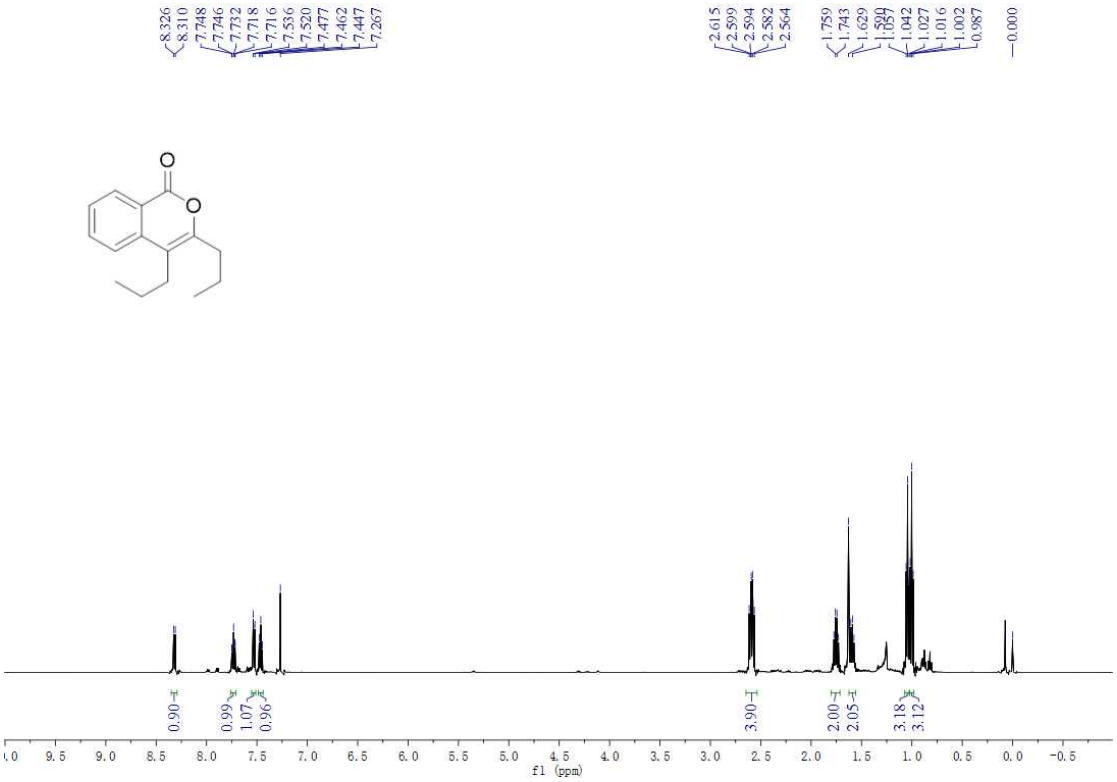


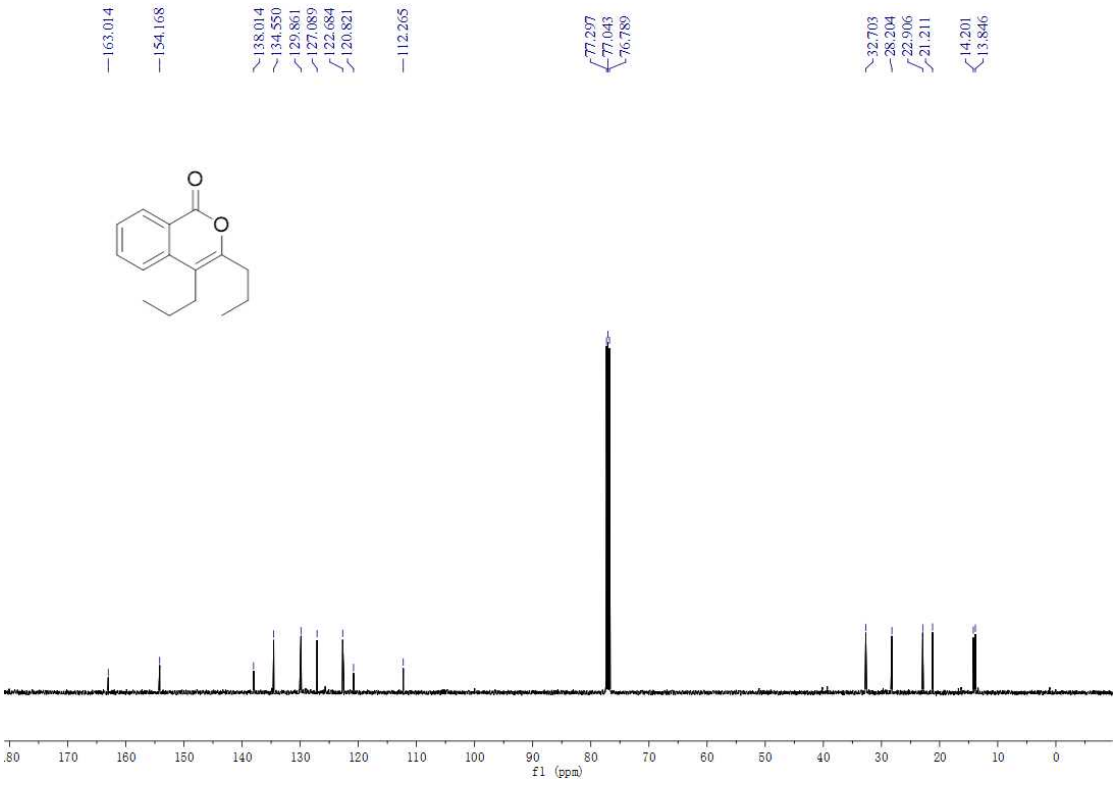




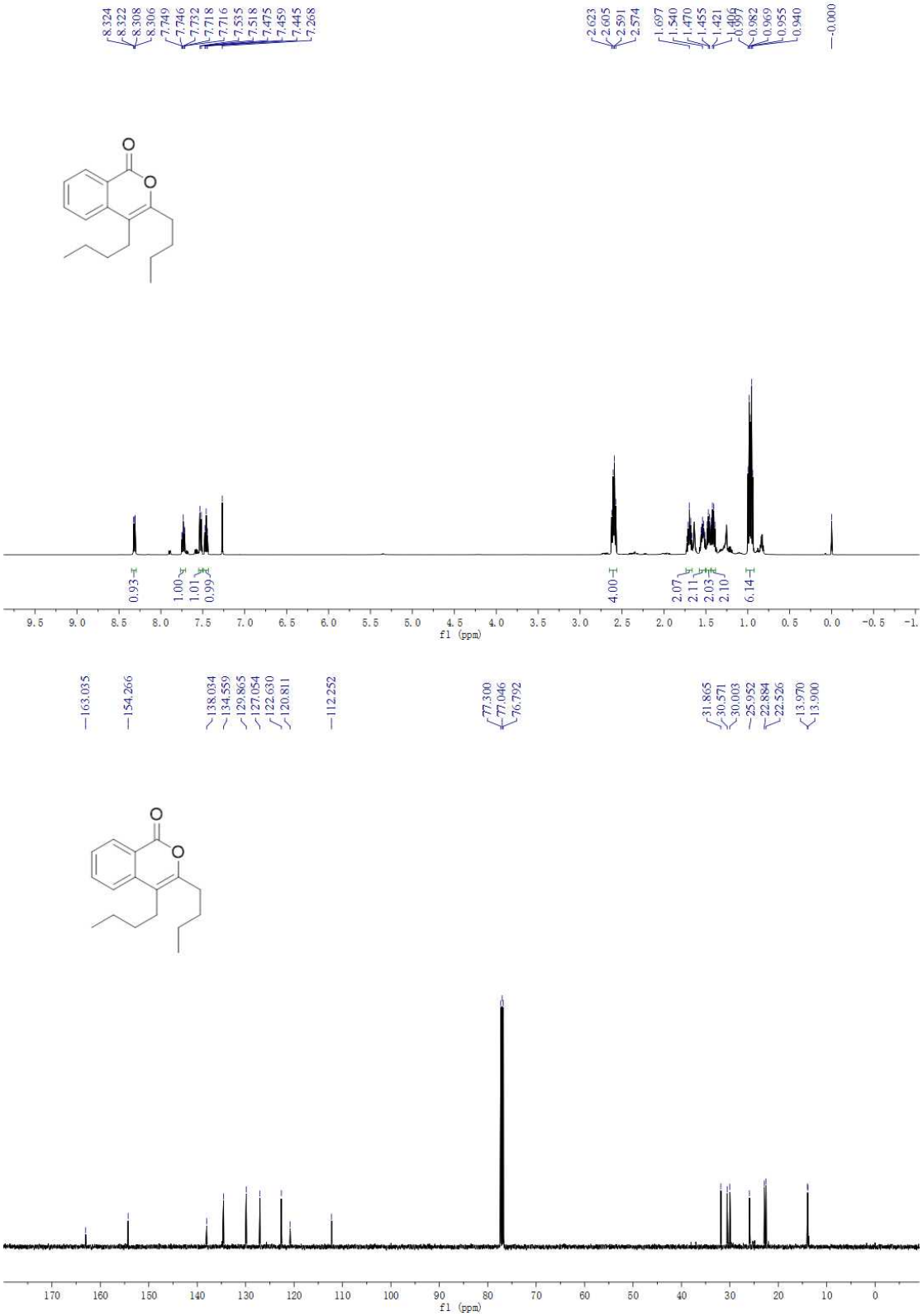


3,4-Dipropyl-1*H*-isochromen-1-one (3al):



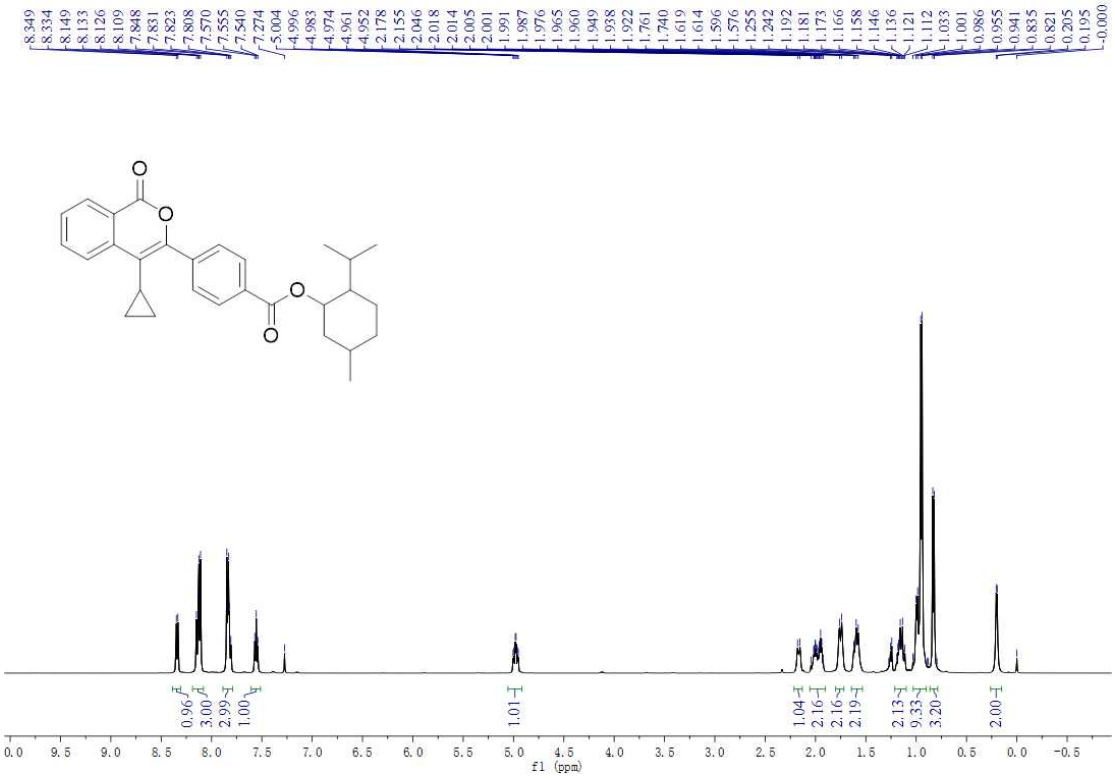


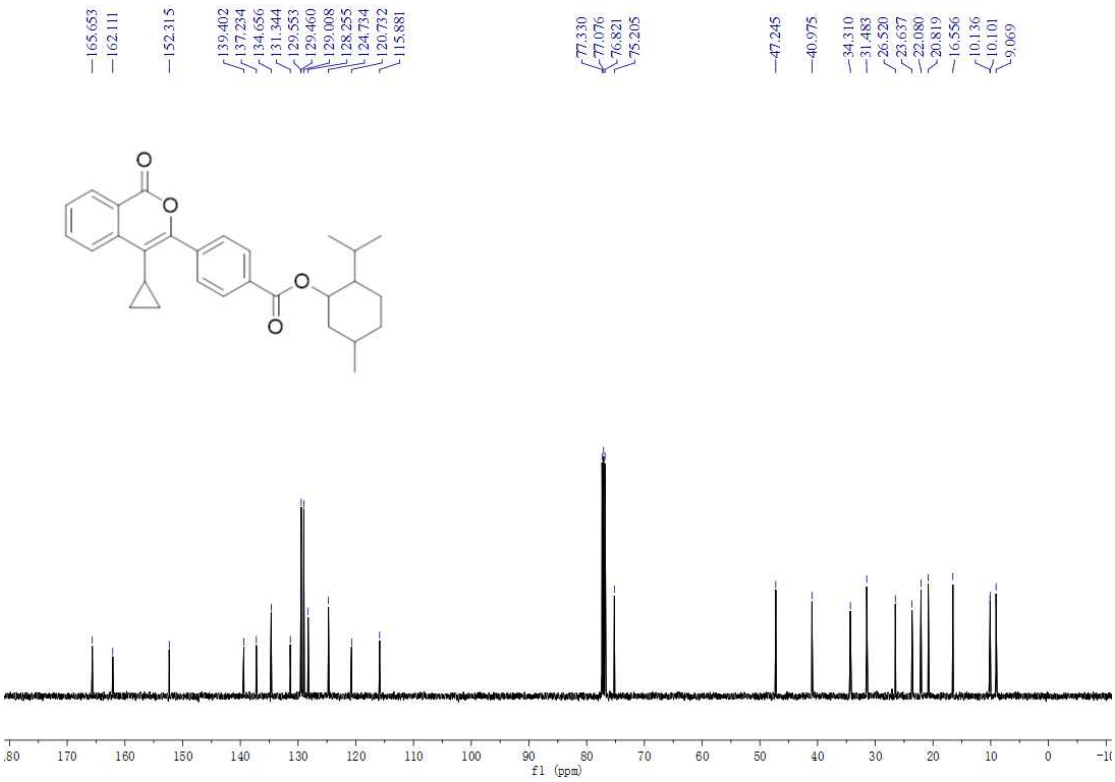
3,4-Dibutyl-1*H*-isochromen-1-one (3am):



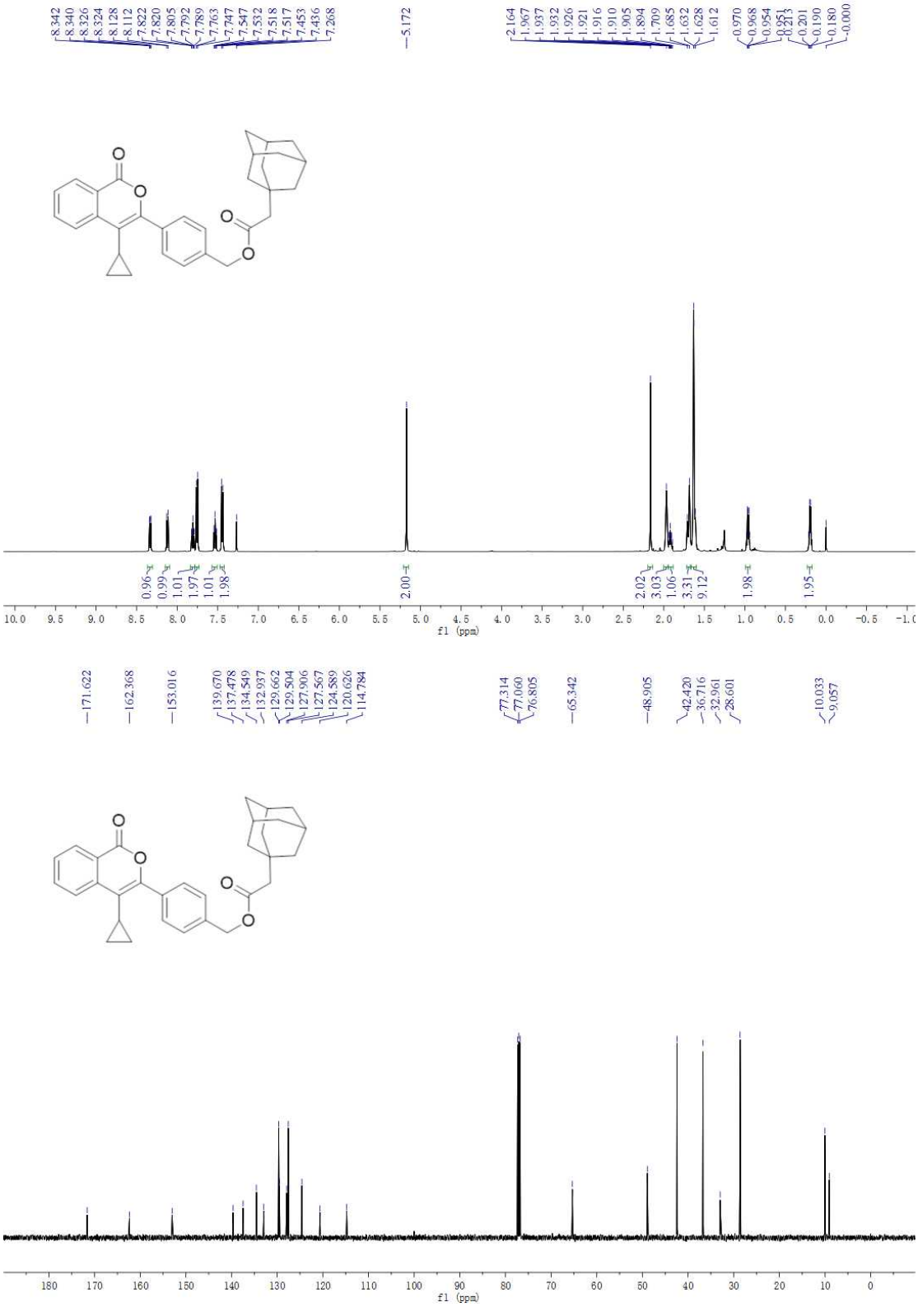
2-Isopropyl-5-methylcyclohexyl-4-(4-cyclopropyl-1-oxo-1*H*-isochromen-3-yl)ben

zoate (3ap):

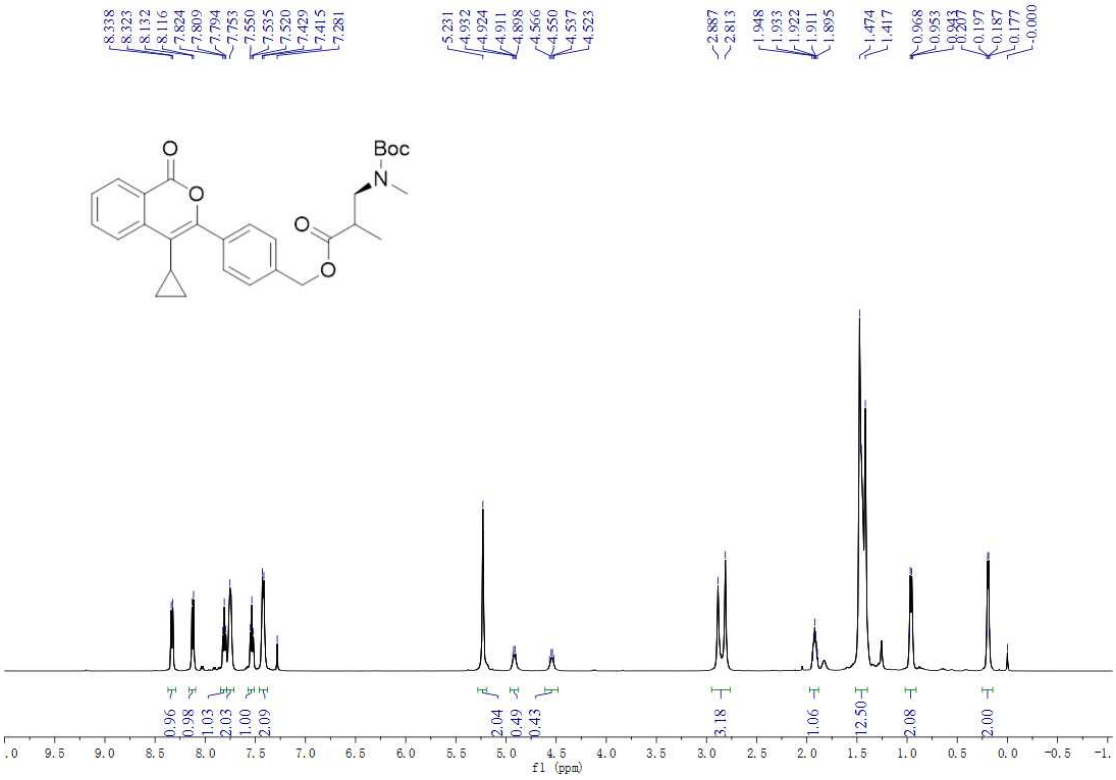


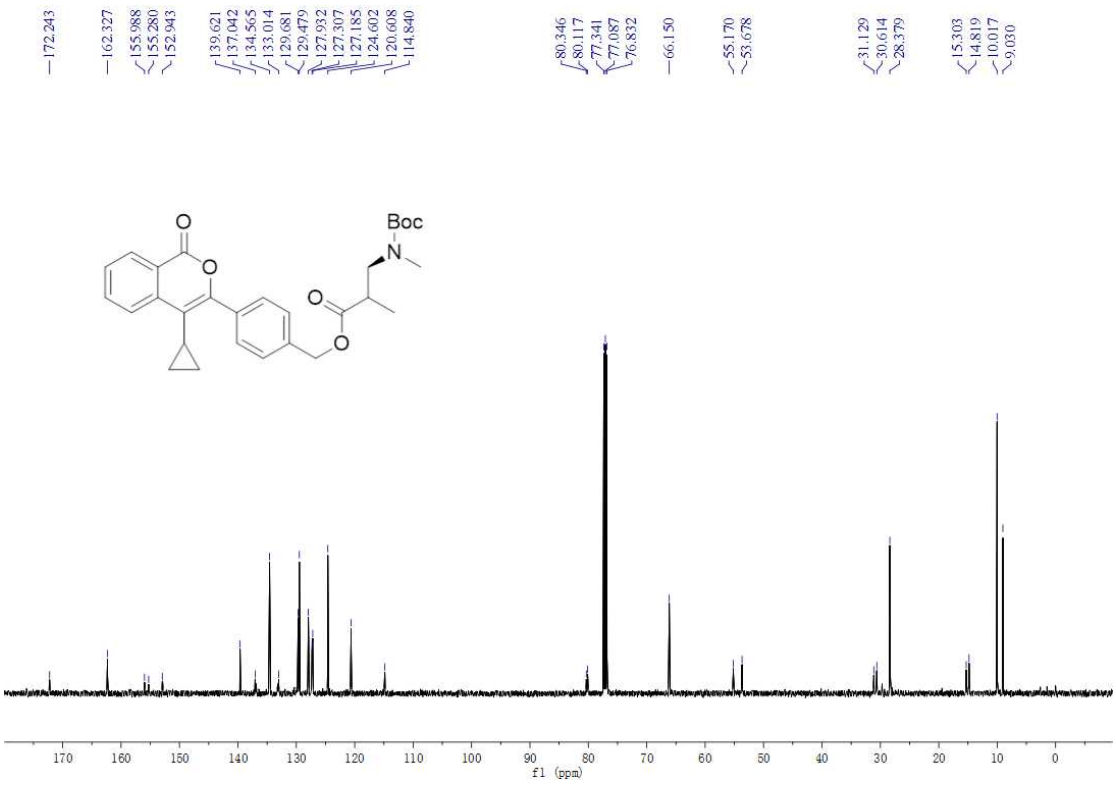


4-(4-cyclopropyl-1-oxo-1H-isochromen-3-yl)benzyl-2-((3r,5r,7r)-adamantan-1-yl) acetate (3aq):

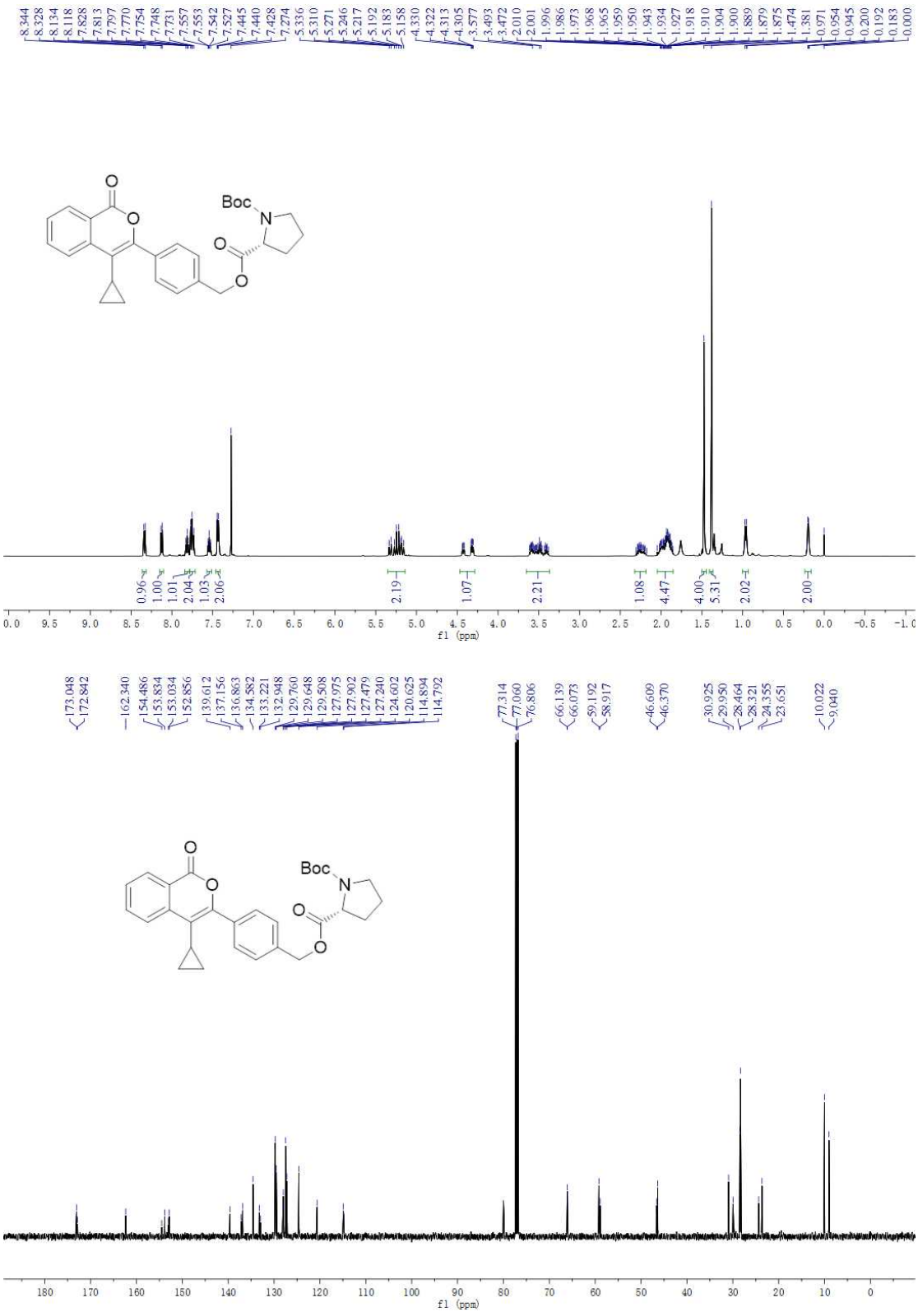


4-(4-cyclopropyl-1-oxo-1*H*-isochromen-3-yl)benzyl-3-((tert-butoxycarbonyl)(methyl)amino)-2-methylpropanoate (3ar):

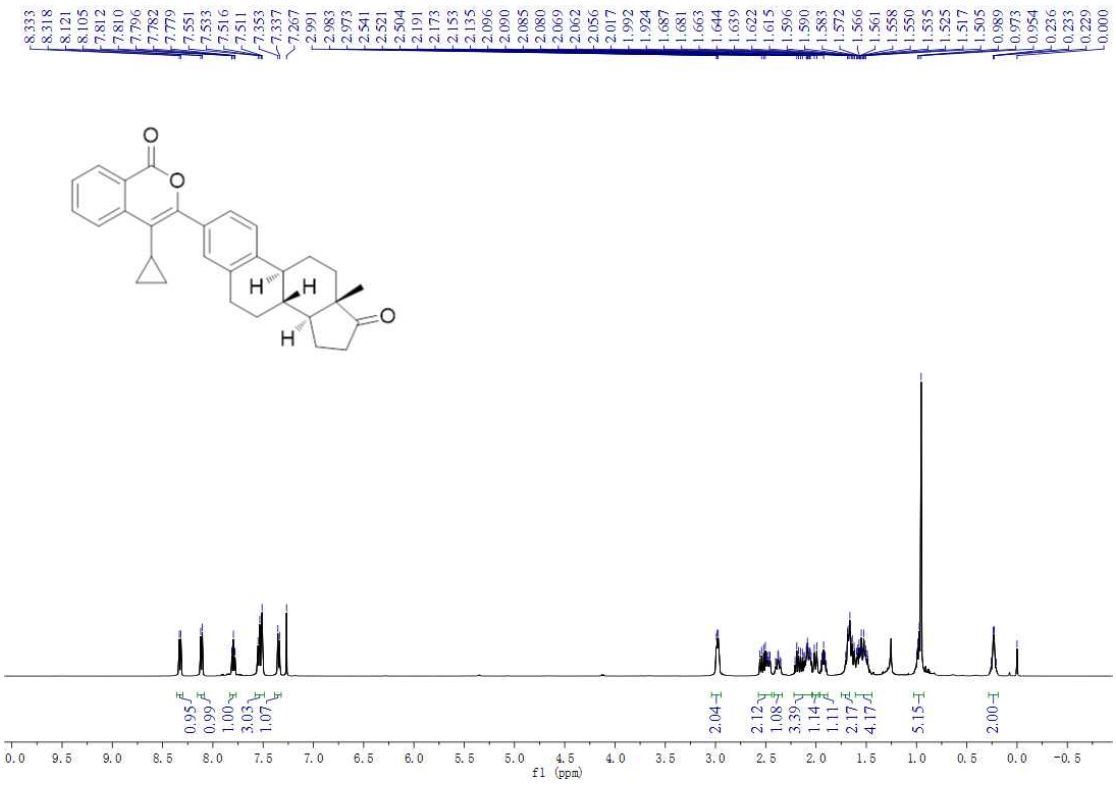


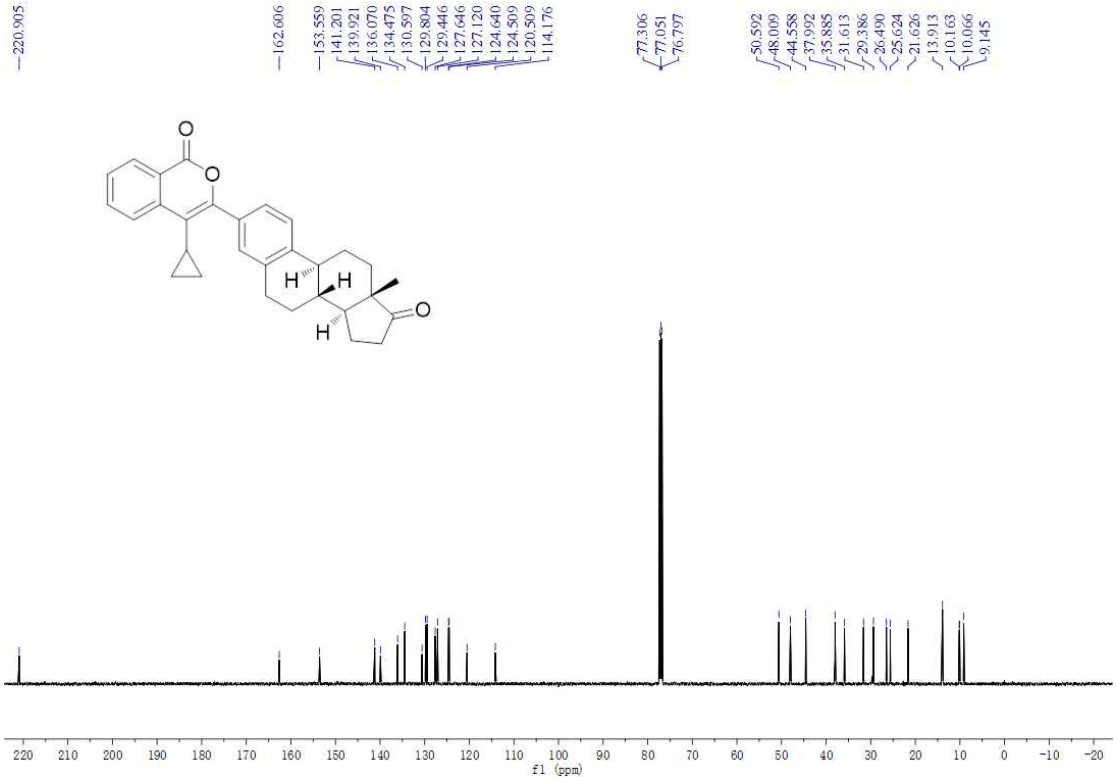


(R)-1-Tert-butyl 2-(4-(4-cyclopropyl-1-oxo-1*H*-isochromen-3-yl)benzyl) pyrrolidine-1,2-dicarboxylate (3as):

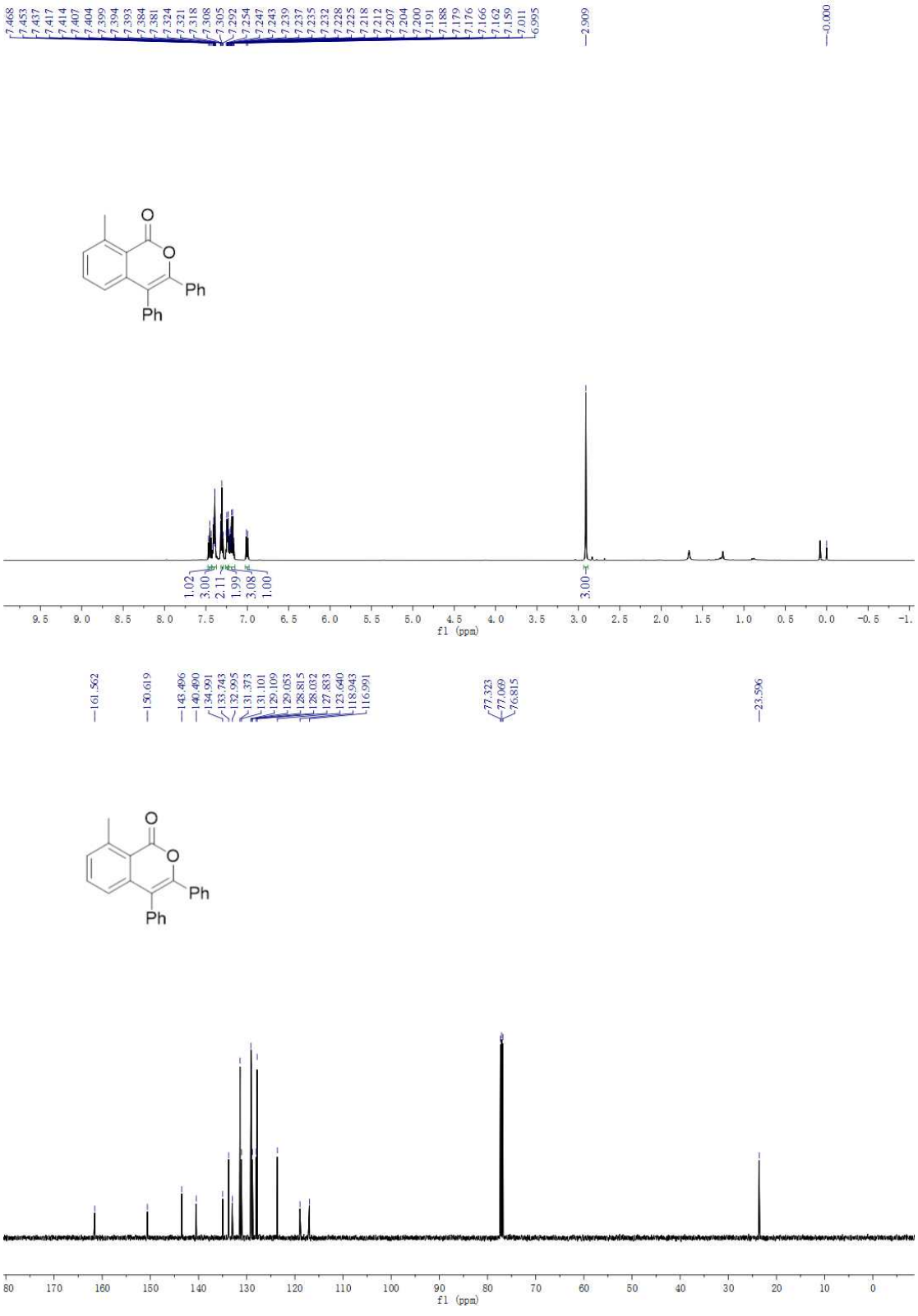


4-Cyclopropyl-3-((8R,9S,13S,14S)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6*H*-cyclopenta[a]phenanthren-3-yl)-1*H*-isochromen-1-one (3at):

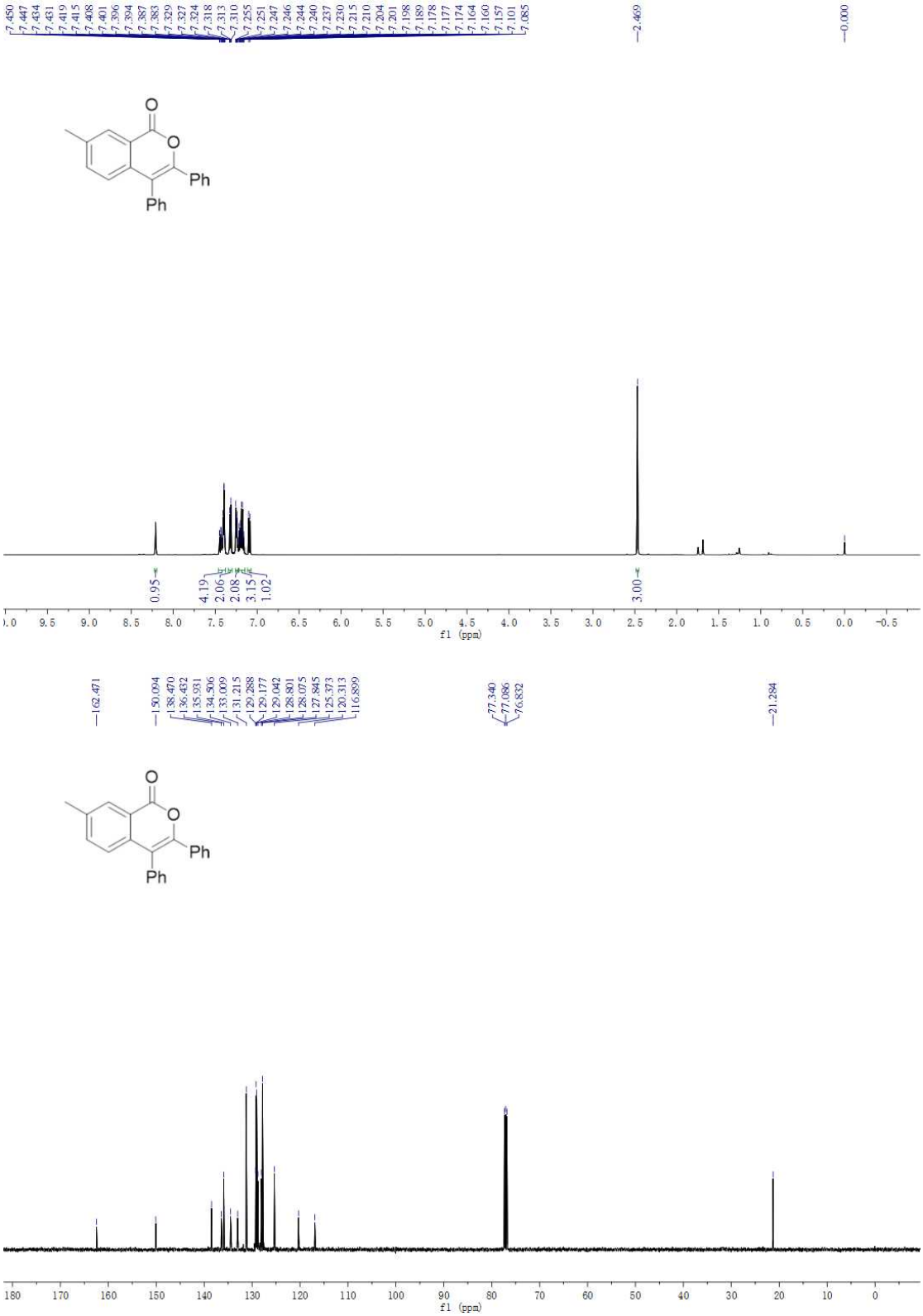




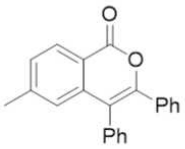
8-Methyl-3,4-diphenyl-1*H*-isochromen-1-one (3ba):

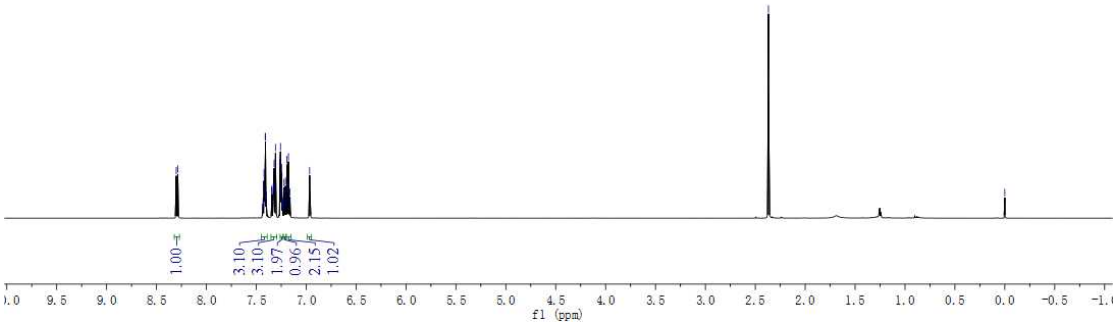


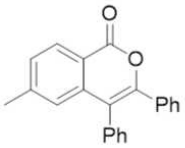
7-Methyl-3,4-diphenyl-1*H*-isochromen-1-one (3ca):

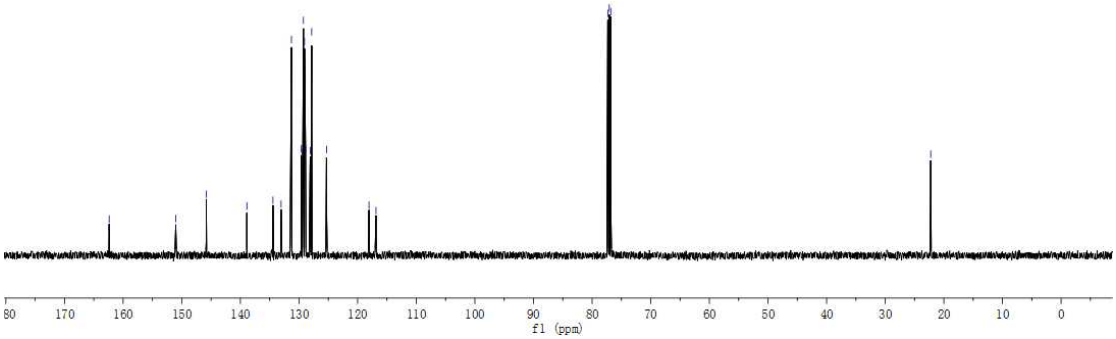


6-Methyl-3,4-diphenyl-1*H*-isochromen-1-one (3da):

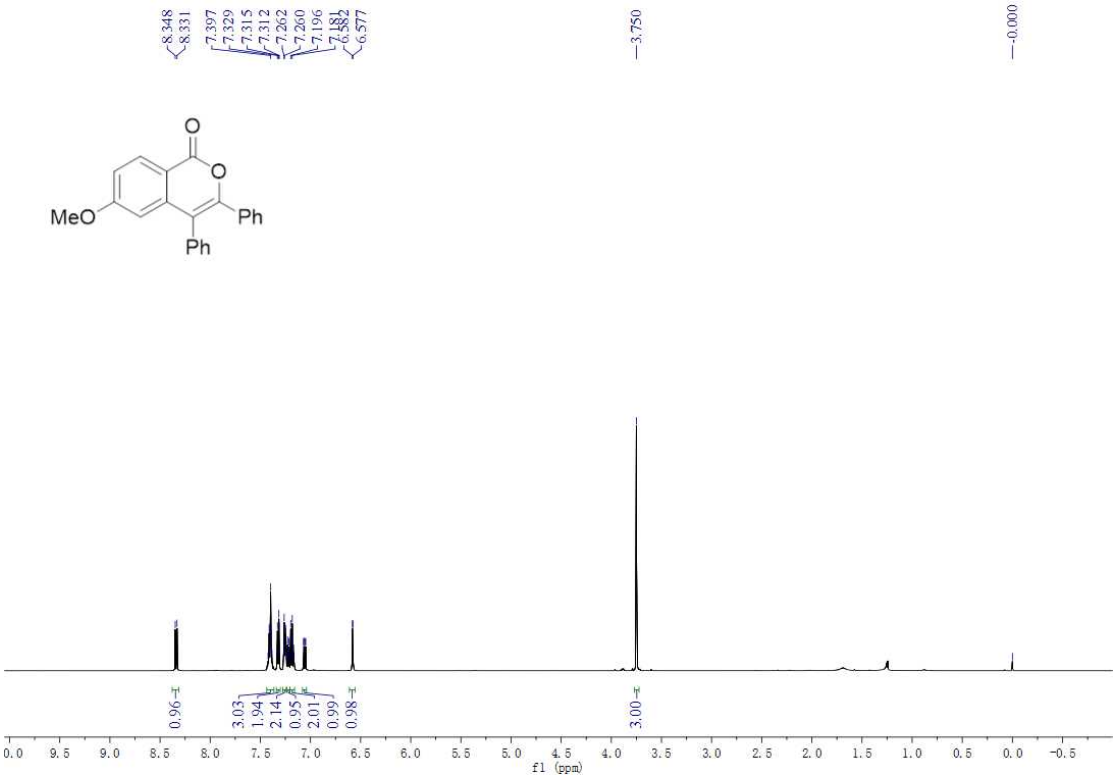


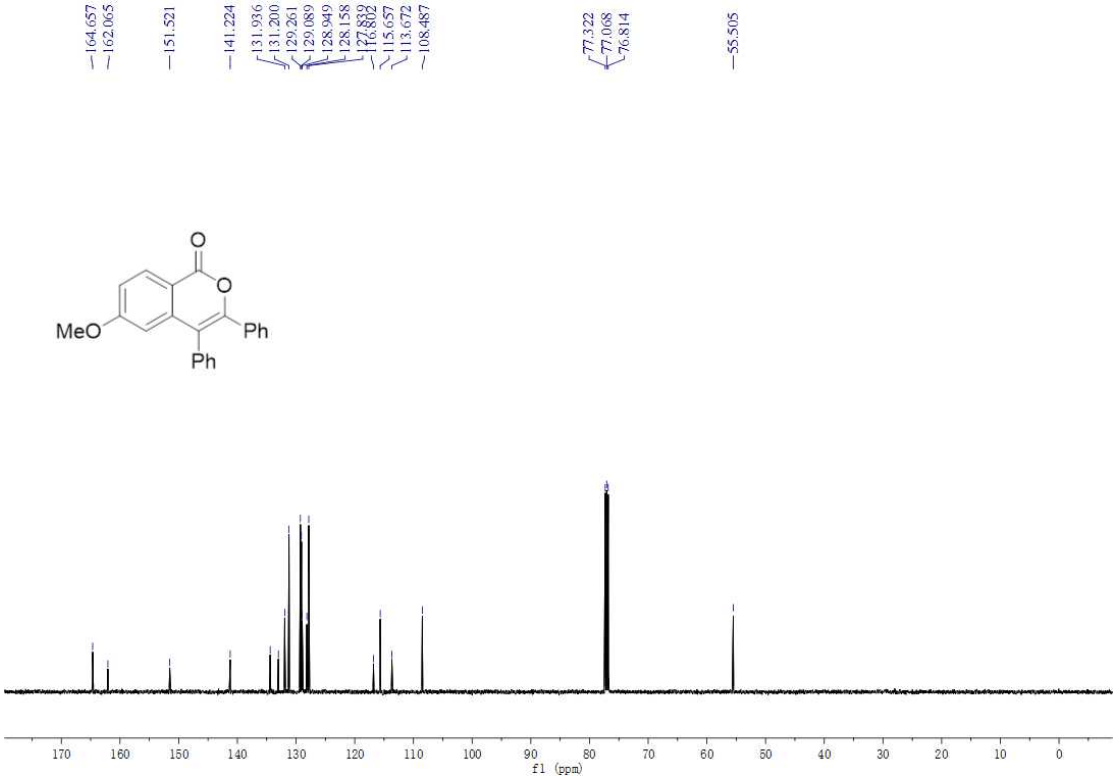




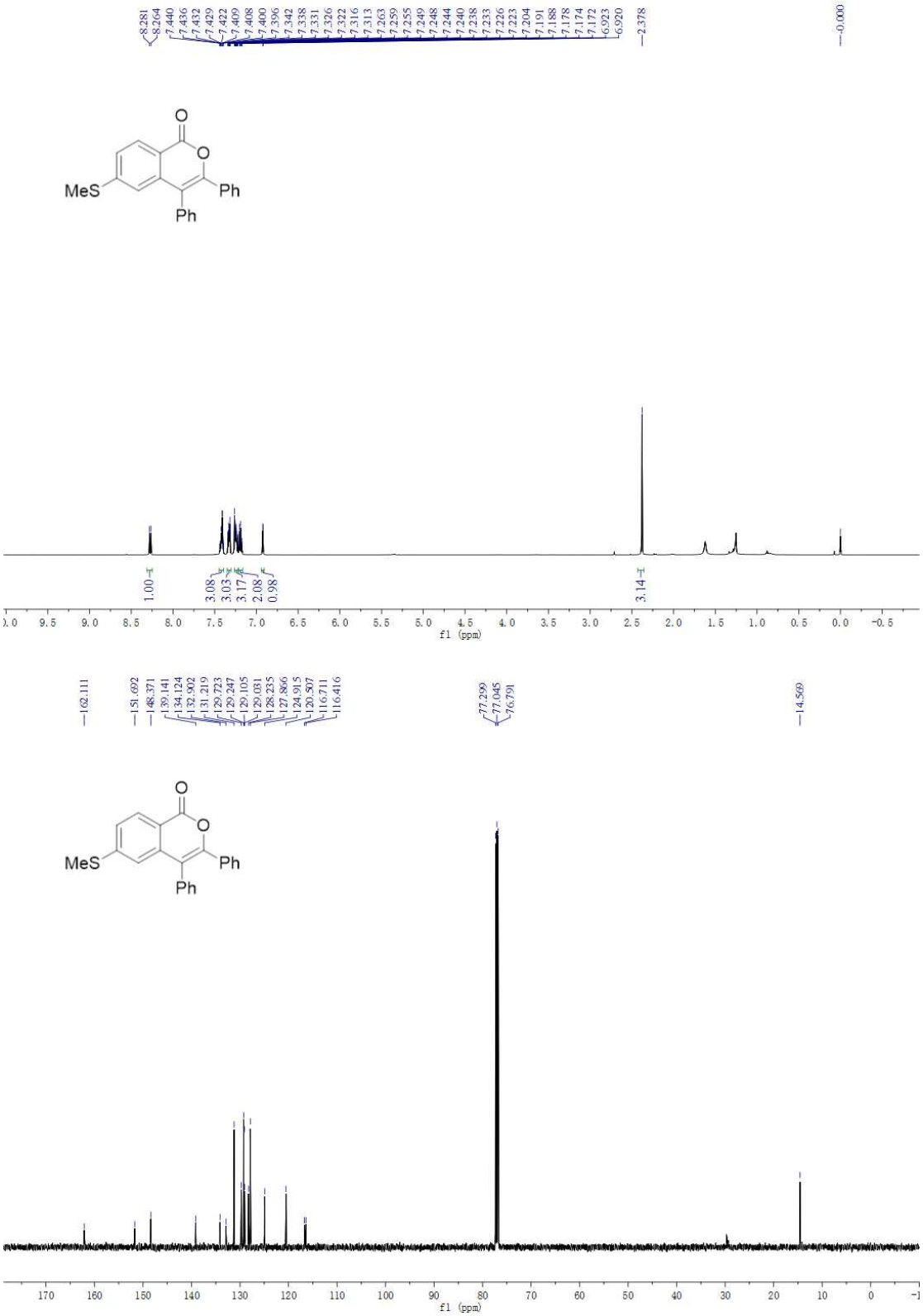


6-Methoxy-3,4-diphenyl-1*H*-isochromen-1-onee (3ea):

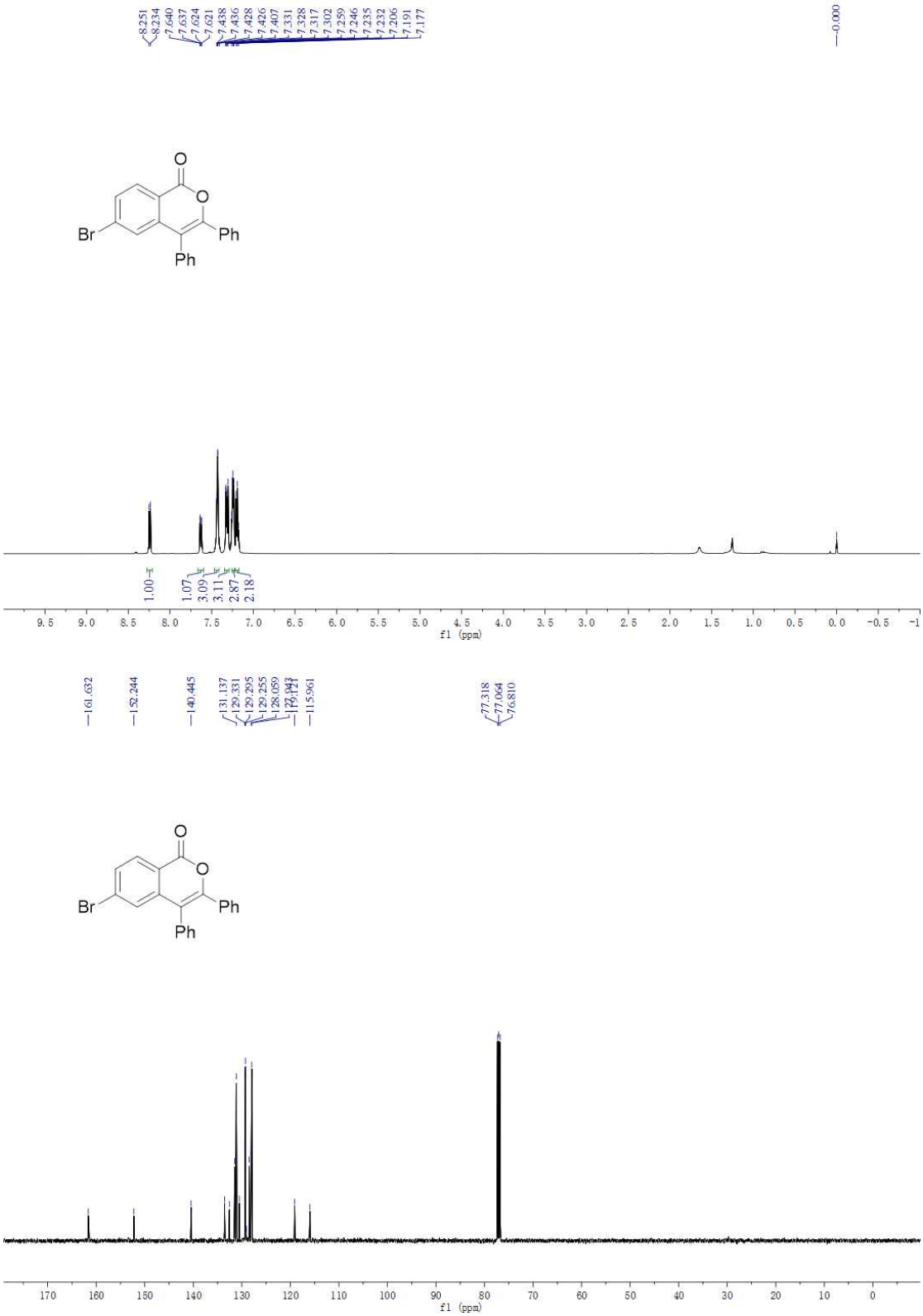




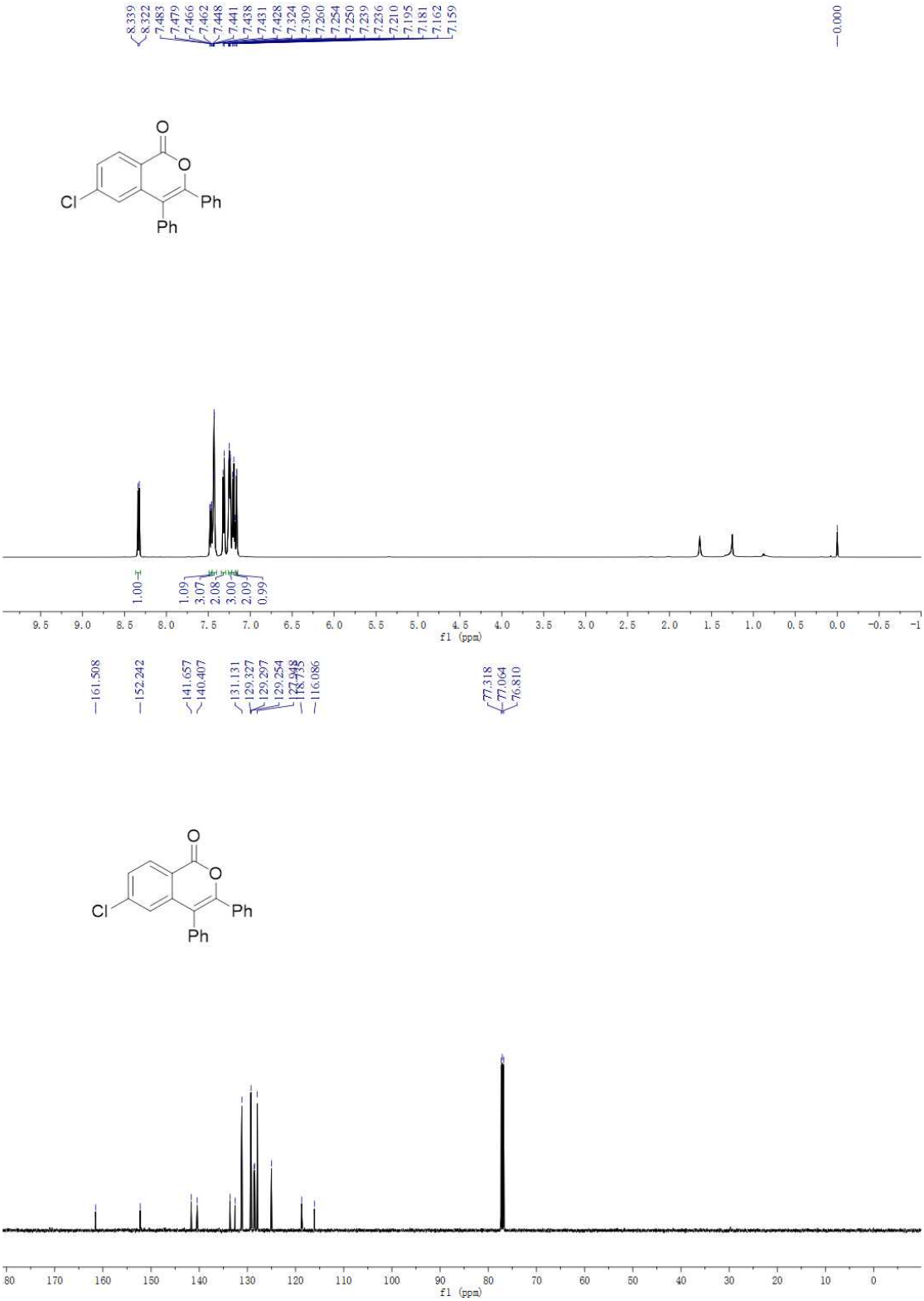
6-(Methylthio)-3,4-diphenyl-1*H*-isochromen-1-one (3fa):



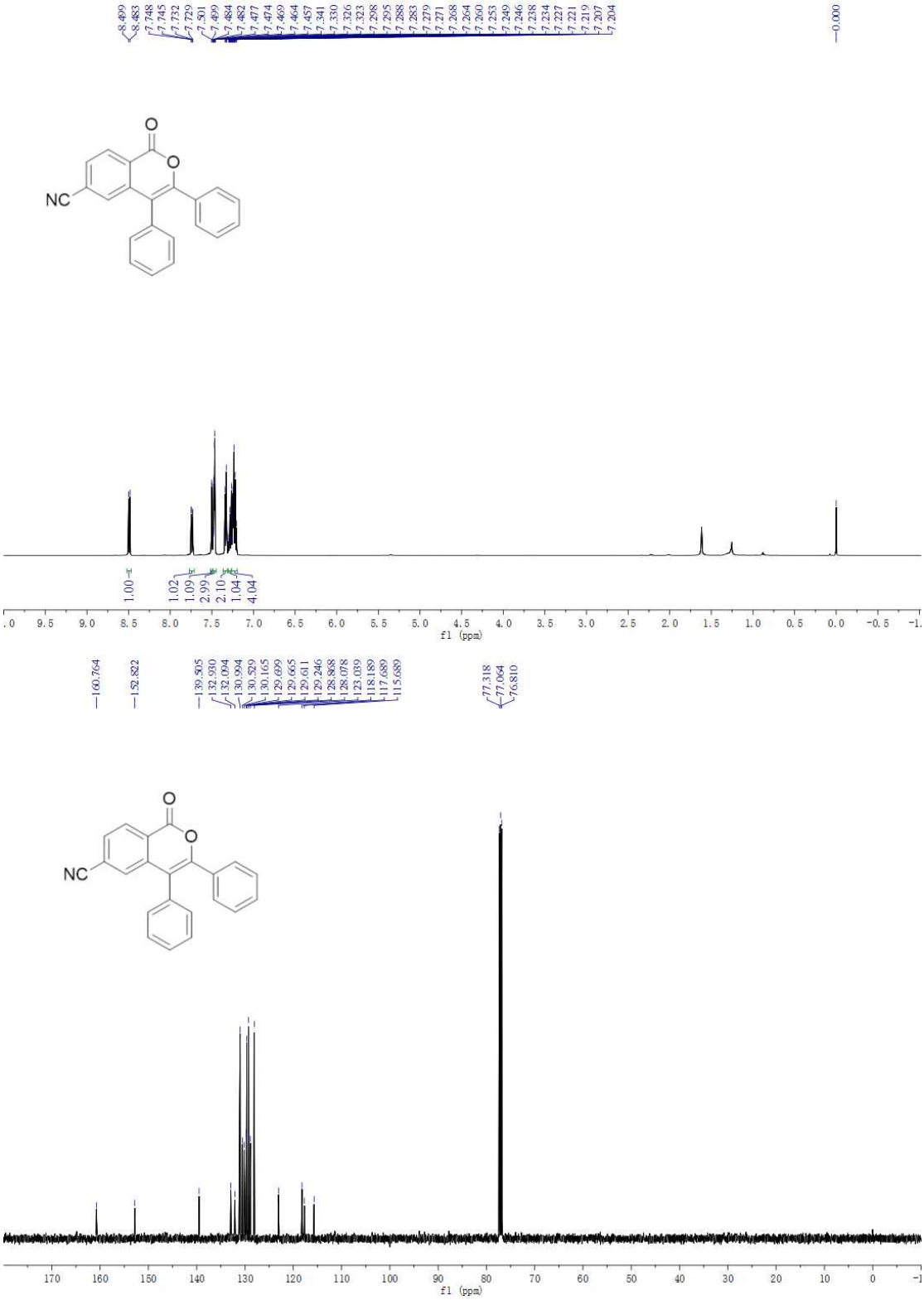
6-Bromo-3,4-diphenyl-1*H*-isochromen-1-one (3ga):



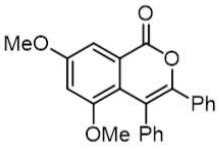
6-Chloro-3,4-diphenyl-1*H*-isochromen-1-one (3ha):

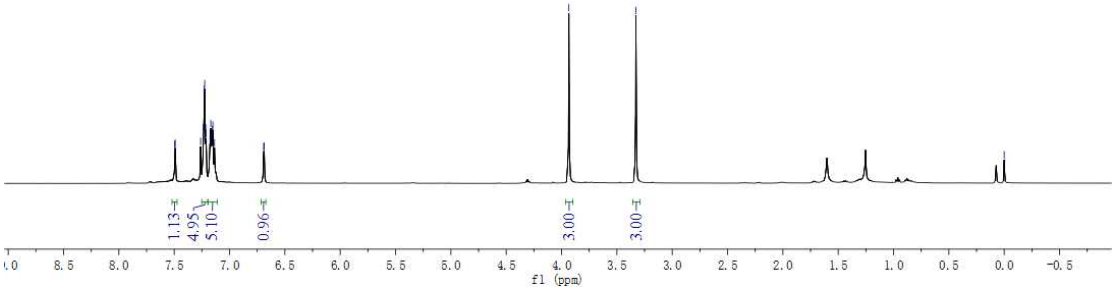


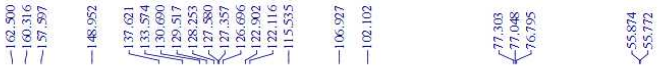
1-Oxo-3,4-diphenyl-1*H*-isochromene-6-carbonitrile (3ia):

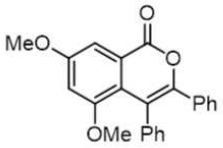


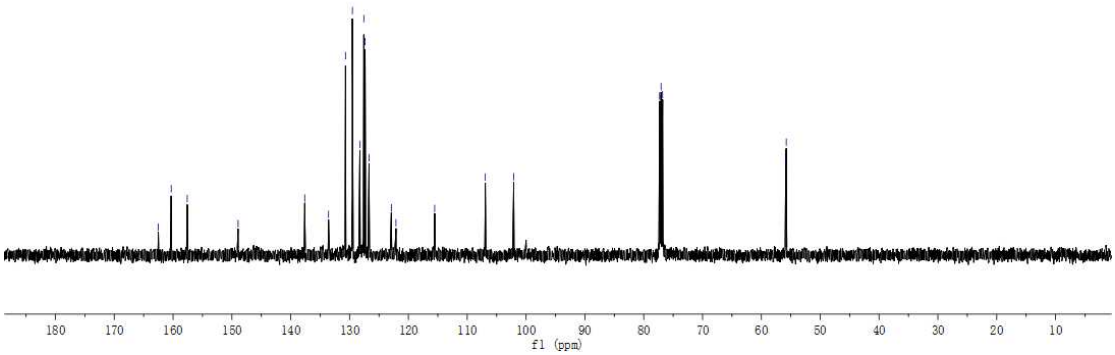
5,7-Dimethoxy-3,4-diphenyl-1*H*-isochromen-1-one (3ja):



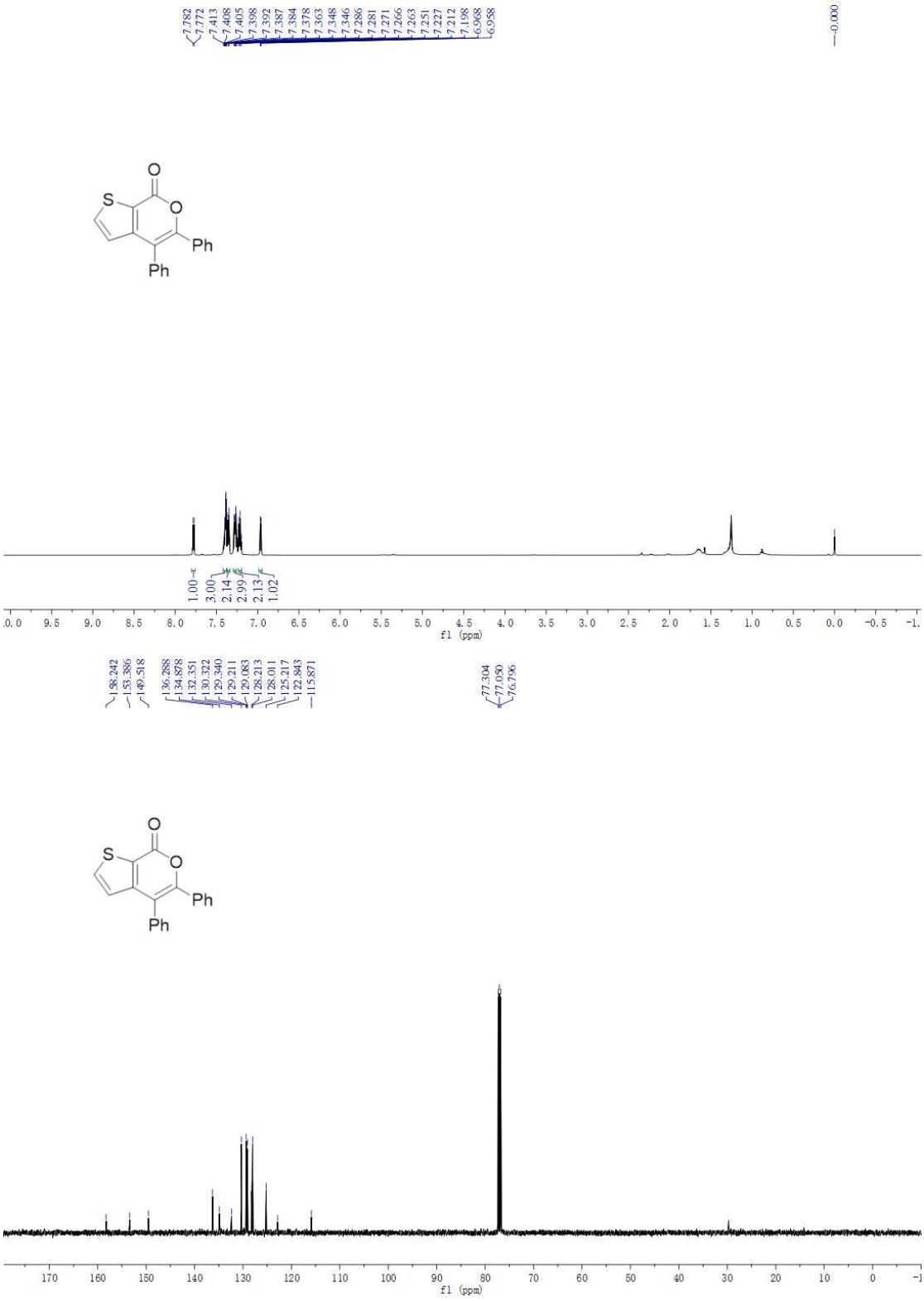




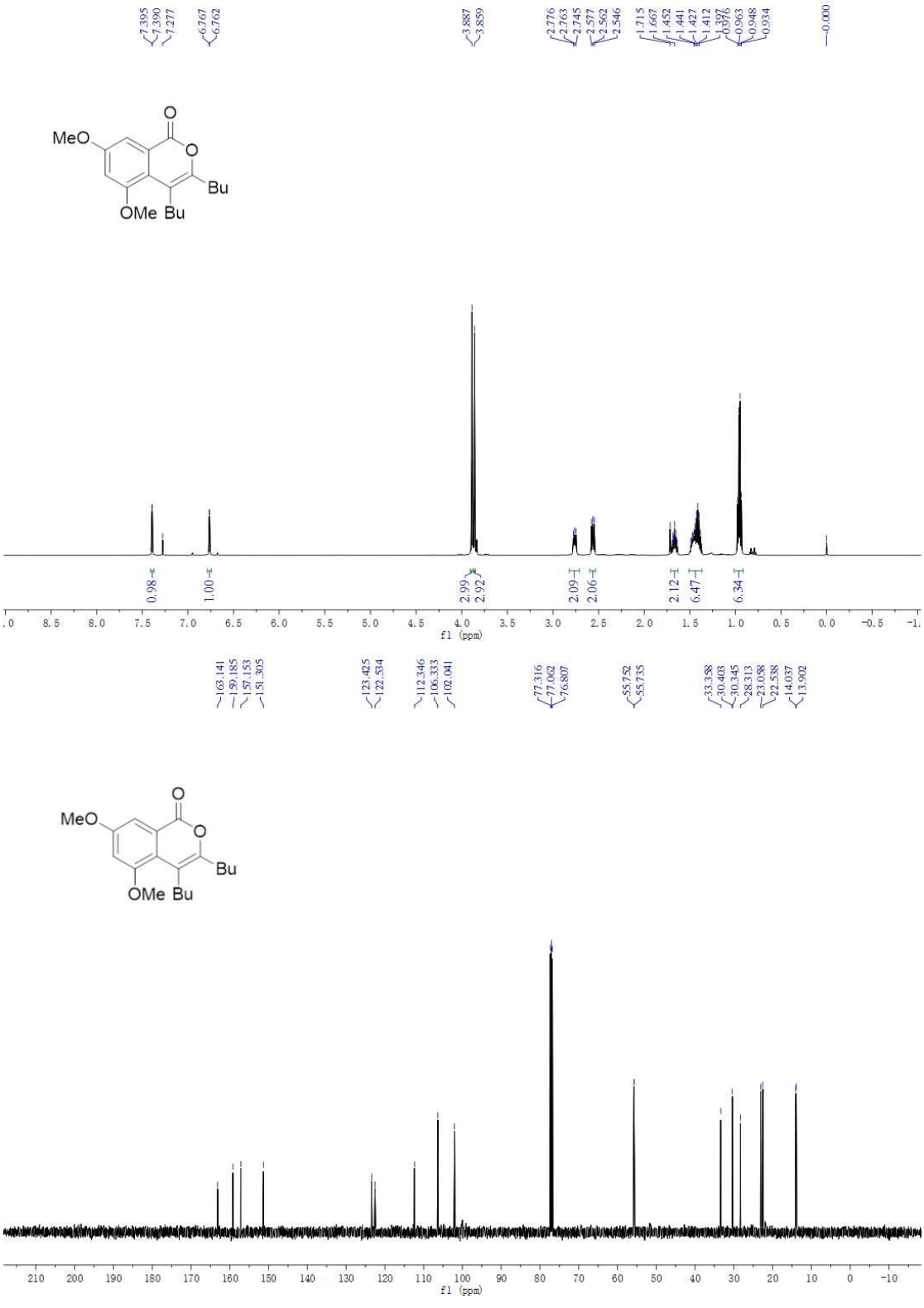




4,5-Diphenyl-7*H*-thieno[2,3-c]pyran-7-one (3ka):



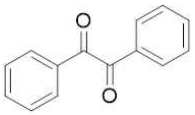
3,4-Dibutyl-5,7-dimethoxy-1*H*-isochromen-1-one (3jm):

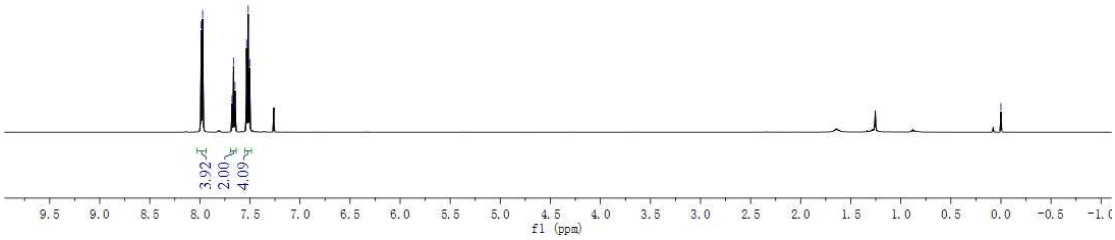


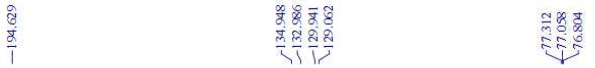
Benzil (4a):

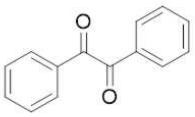

7.988
7.986
7.976
7.972
7.969
7.681
7.679
7.676
7.664
7.651
7.649
7.647
7.533
7.530
7.517
7.506
7.502

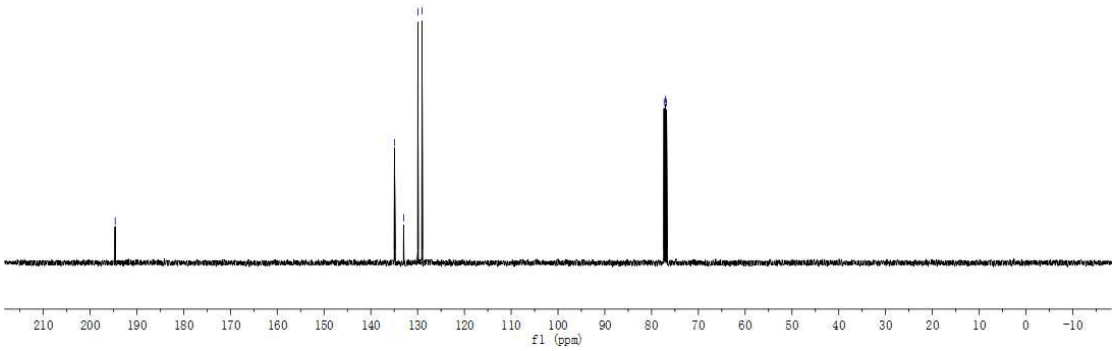
0.000



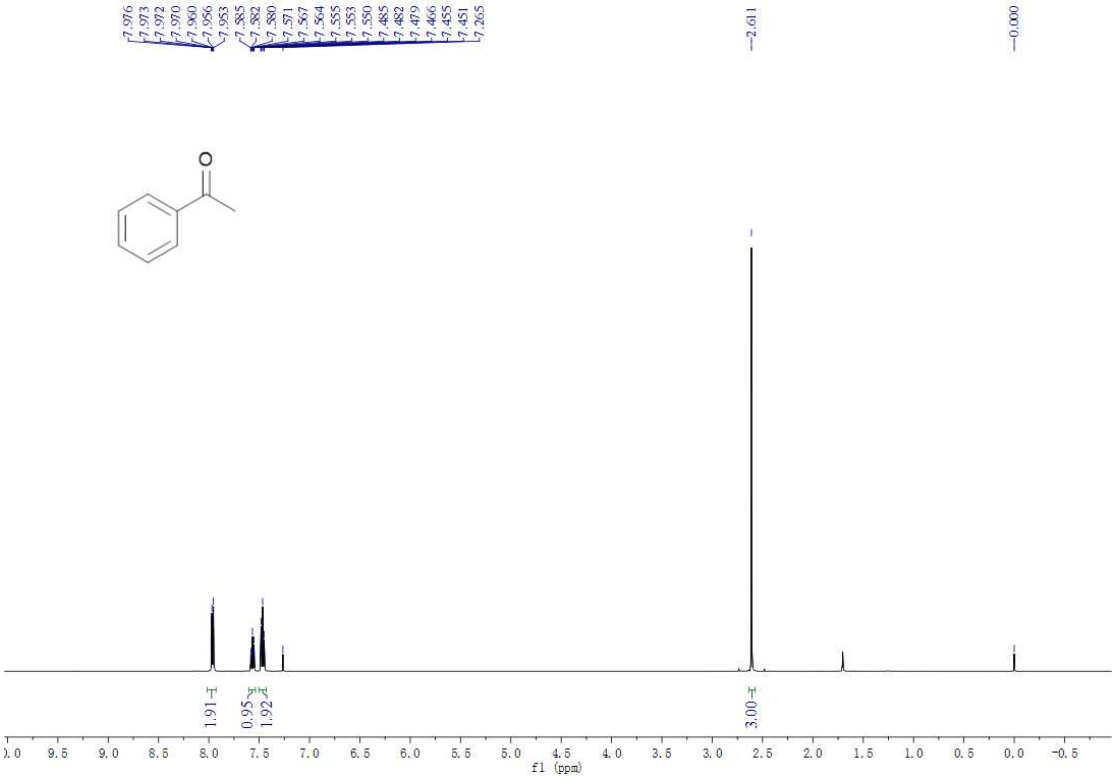

3.92
2.00
4.09
f1 (ppm)

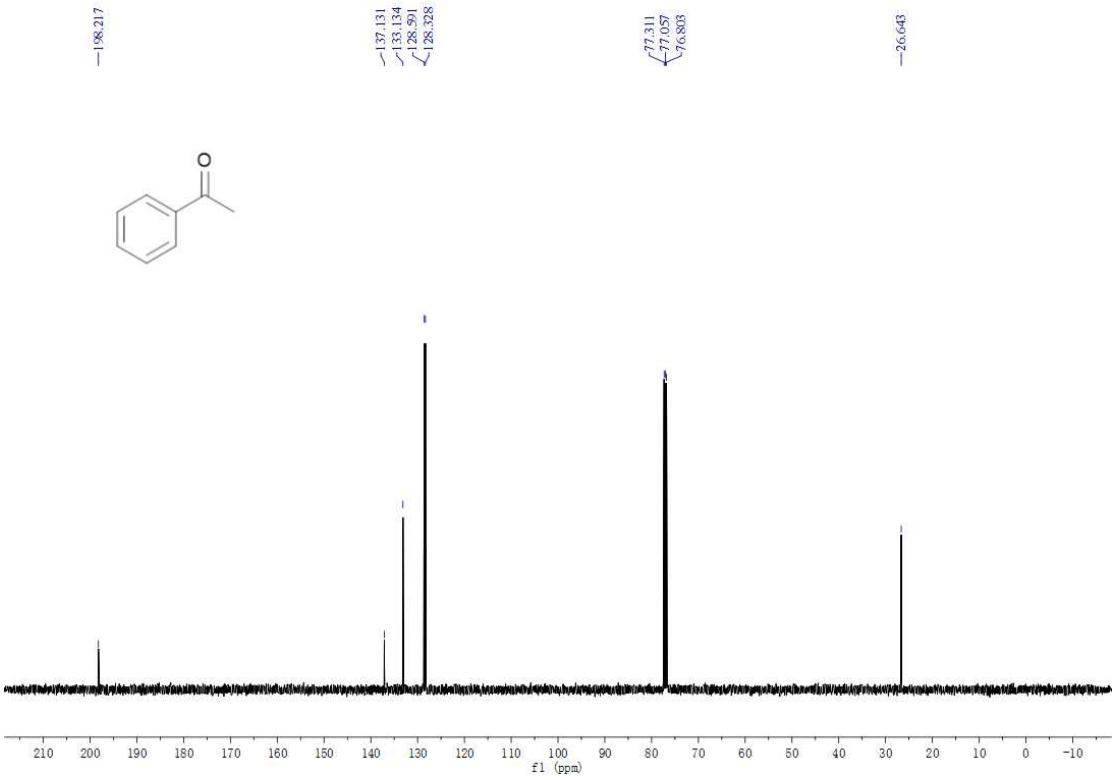

194.629
134.948
132.986
129.941
129.062
77.312
77.058
76.804



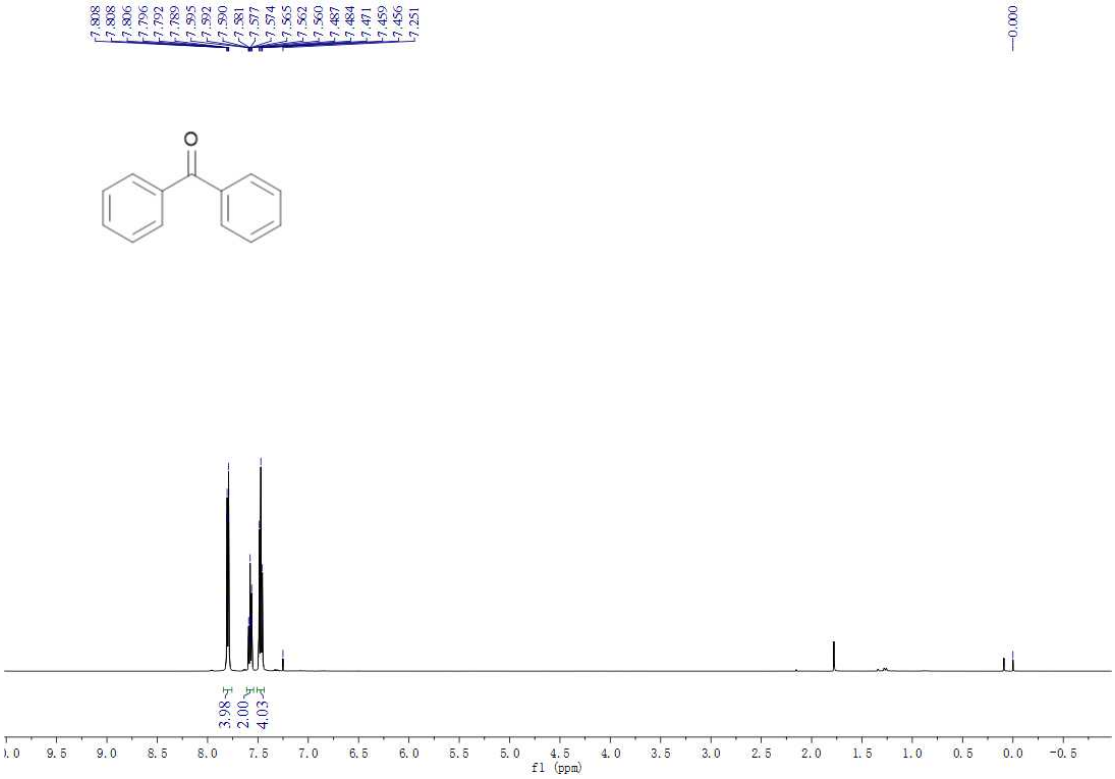

f1 (ppm)

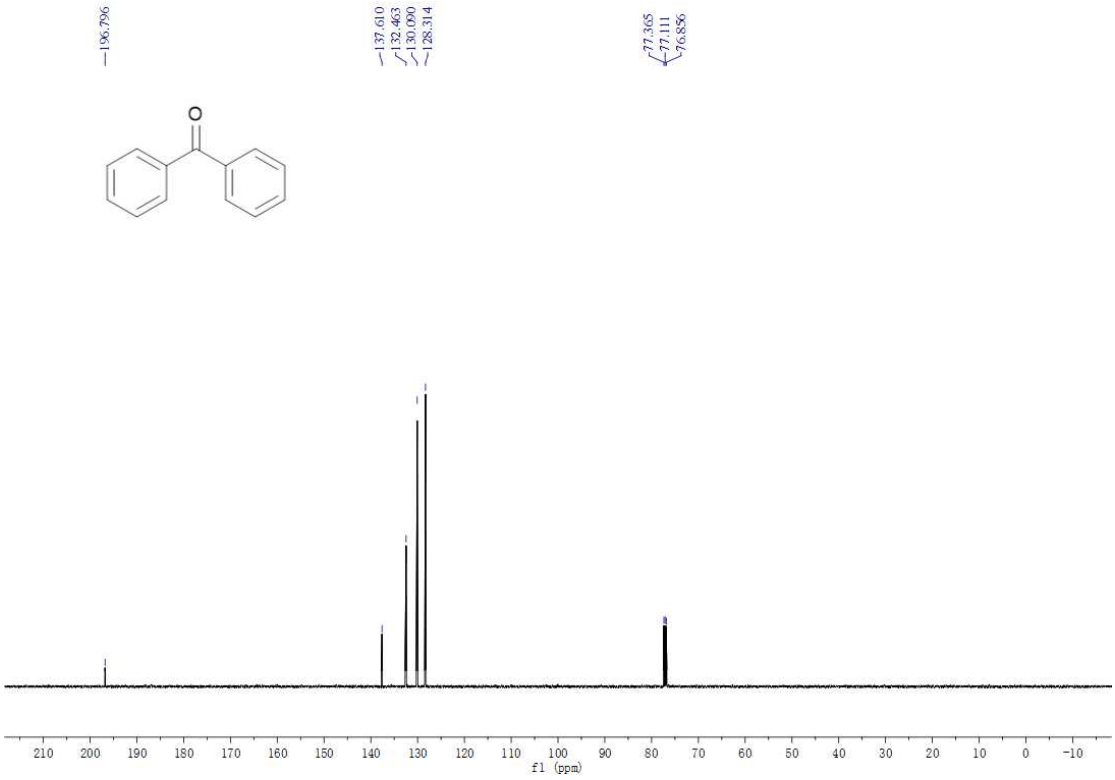
Acetophenone (5l):



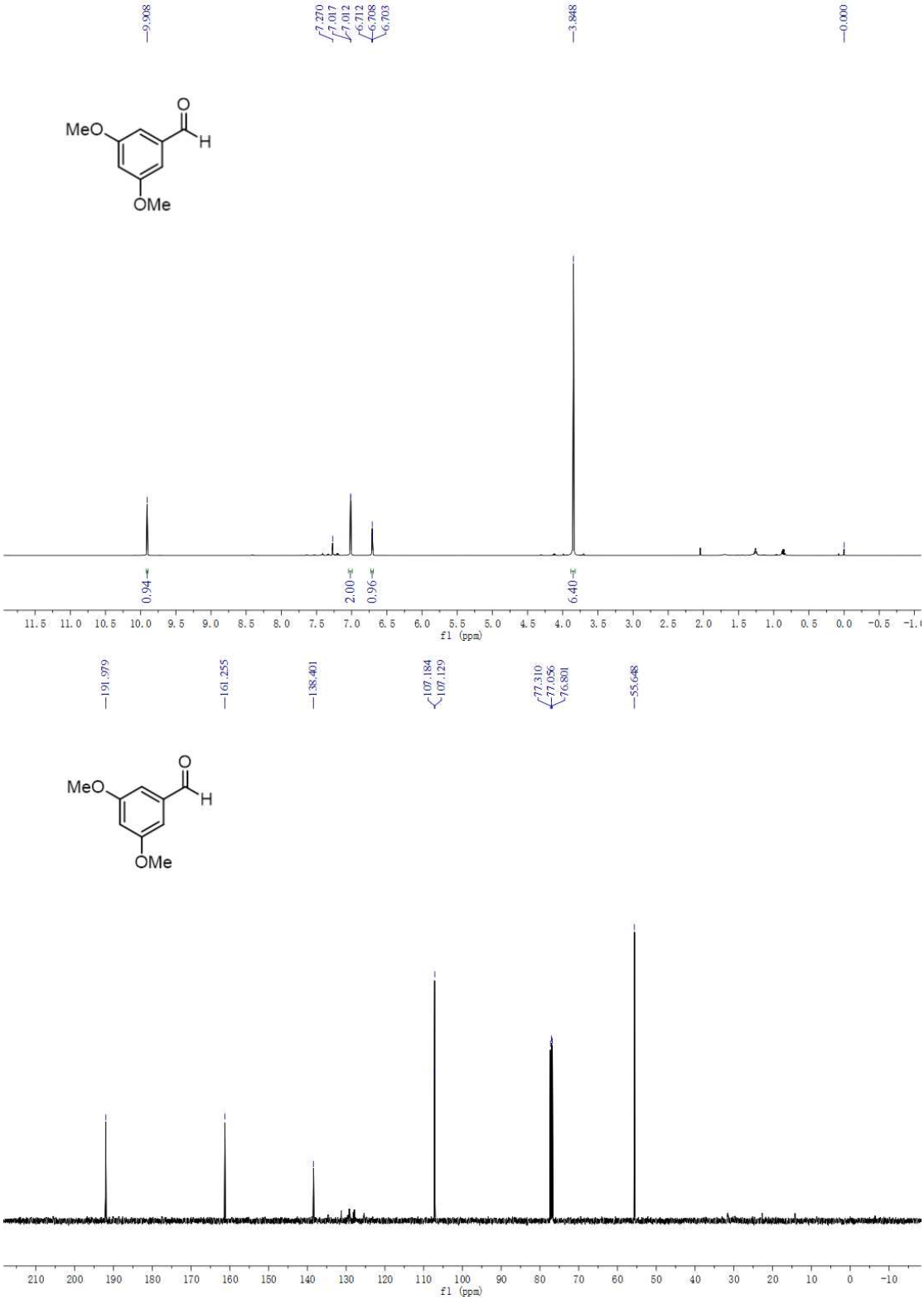


Benzophenone (5m):





3,5-Dimethoxybenzaldehyde (5j):



(E) The X-ray single-crystal diffraction analysis of 3aj (CCDC 1874275):

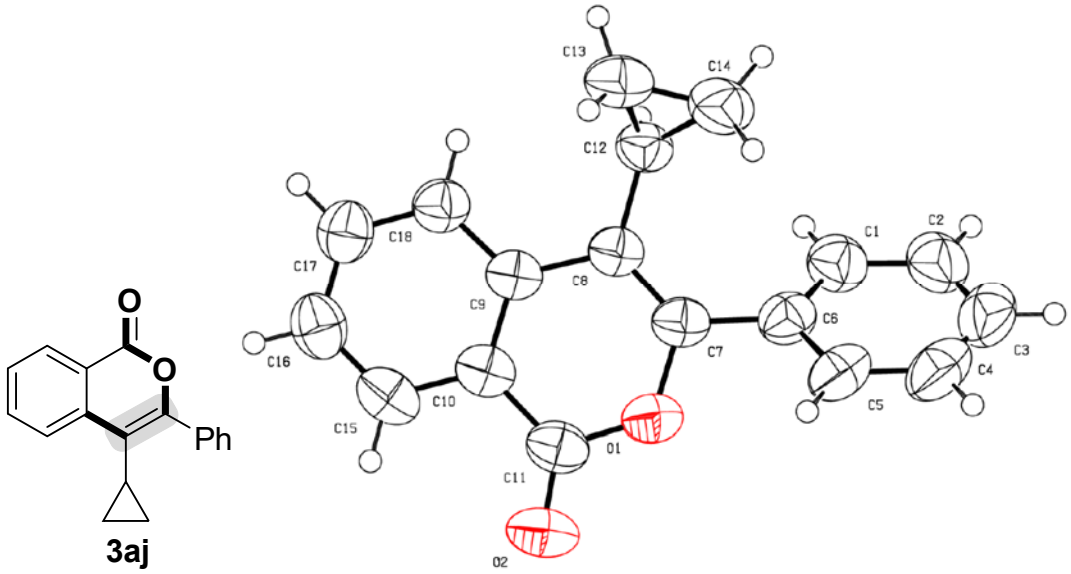


Table S1. Crystal data and structure refinement for A.

Identification code	a
Empirical formula	C18 H14 O2
Formula weight	262.29
Temperature	296(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, C2/c
Unit cell dimensions	a = 22.242(4) A alpha = 90 deg.
	b = 10.3242(19) A beta = 125.704(2) deg.
	c = 14.505(3) A gamma = 90 deg.
Volume	2704.8(8) A^3
Z, Calculated density	8, 1.288 Mg/m^3
Absorption coefficient	0.083 mm^-1
F(000)	1104

Crystal size	0.21 x 0.18 x 0.16 mm
Theta range for data collection	2.27 to 25.49 deg.
Limiting indices	-26<=h<=26, -12<=k<=12, -17<=l<=17
Reflections collected / unique	10142 / 2517 [R(int) = 0.0233]
Completeness to theta = 25.49	100.0 %
Absorption correction	None
Max. and min. transmission	0.9868 and 0.9828
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2517 / 0 / 181
Goodness-of-fit on F^2	1.063
Final R indices [I>2sigma(I)]	R1 = 0.0419, wR2 = 0.1018
R indices (all data)	R1 = 0.0544, wR2 = 0.1144
Largest diff. peak and hole	0.167 and -0.289 e.A^-3

Table S2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for A.

U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
C(1)	2830(1)	5562(2)	3209(2)	72(1)
C(2)	3597(1)	5534(2)	3946(2)	87(1)
C(3)	3942(1)	4897(2)	4963(2)	96(1)
C(4)	3533(1)	4272(2)	5255(2)	88(1)
C(5)	2774(1)	4268(2)	4522(1)	72(1)
C(6)	2408(1)	4926(1)	3491(1)	62(1)
C(7)	1593(1)	4913(1)	2724(1)	56(1)
C(8)	1116(1)	5893(1)	2171(1)	52(1)
C(9)	328(1)	5612(1)	1451(1)	52(1)
C(10)	82(1)	4342(1)	1367(1)	56(1)
C(11)	614(1)	3304(1)	1972(1)	62(1)
C(12)	1360(1)	7265(1)	2268(1)	58(1)
C(13)	1153(1)	8260(2)	2789(2)	76(1)
C(14)	1934(1)	7866(2)	3378(2)	85(1)
C(15)	-671(1)	4046(2)	699(1)	66(1)

C(16)	-1184(1)	5000(2)	112(1)	72(1)
C(17)	-953(1)	6255(2)	168(1)	70(1)
C(18)	-213(1)	6564(2)	820(1)	61(1)
O(1)	1347(1)	3647(1)	2638(1)	65(1)
O(2)	478(1)	2172(1)	1948(1)	85(1)

Table S3. Bond lengths [A] and angles [deg] for A.

C(1)-C(6)	1.387(2)
C(1)-C(2)	1.388(2)
C(1)-H(1)	0.9300
C(2)-C(3)	1.369(3)
C(2)-H(2)	0.9300
C(3)-C(4)	1.370(3)
C(3)-H(3)	0.9300
C(4)-C(5)	1.373(3)
C(4)-H(4)	0.9300
C(5)-C(6)	1.394(2)
C(5)-H(5)	0.9300
C(6)-C(7)	1.474(2)
C(7)-C(8)	1.342(2)
C(7)-O(1)	1.3940(17)
C(8)-C(9)	1.452(2)
C(8)-C(12)	1.4945(19)
C(9)-C(10)	1.3990(19)
C(9)-C(18)	1.402(2)
C(10)-C(15)	1.395(2)

C(10)-C(11)	1.452(2)
C(11)-O(2)	1.2022(18)
C(11)-O(1)	1.3705(18)
C(12)-C(14)	1.485(2)
C(12)-C(13)	1.498(2)
C(12)-H(12)	0.9800
C(13)-C(14)	1.476(3)
C(13)-H(13A)	0.9700
C(13)-H(13B)	0.9700
C(14)-H(14A)	0.9700
C(14)-H(14B)	0.9700
C(15)-C(16)	1.365(2)
C(15)-H(15)	0.9300
C(16)-C(17)	1.380(2)
C(16)-H(16)	0.9300
C(17)-C(18)	1.374(2)
C(17)-H(17)	0.9300
C(18)-H(18)	0.9300
C(6)-C(1)-C(2)	120.11(17)
C(6)-C(1)-H(1)	119.9
C(2)-C(1)-H(1)	119.9
C(3)-C(2)-C(1)	120.3(2)

C(3)-C(2)-H(2)	119.9
C(1)-C(2)-H(2)	119.9
C(2)-C(3)-C(4)	120.20(19)
C(2)-C(3)-H(3)	119.9
C(4)-C(3)-H(3)	119.9
C(3)-C(4)-C(5)	120.09(19)
C(3)-C(4)-H(4)	120.0
C(5)-C(4)-H(4)	120.0
C(4)-C(5)-C(6)	120.83(18)
C(4)-C(5)-H(5)	119.6
C(6)-C(5)-H(5)	119.6
C(1)-C(6)-C(5)	118.46(15)
C(1)-C(6)-C(7)	121.84(14)
C(5)-C(6)-C(7)	119.69(14)
C(8)-C(7)-O(1)	121.41(13)
C(8)-C(7)-C(6)	129.81(13)
O(1)-C(7)-C(6)	108.78(12)
C(7)-C(8)-C(9)	118.78(12)
C(7)-C(8)-C(12)	122.75(13)
C(9)-C(8)-C(12)	118.47(12)
C(10)-C(9)-C(18)	117.21(14)
C(10)-C(9)-C(8)	119.73(13)

C(18)-C(9)-C(8)	123.06(13)
C(15)-C(10)-C(9)	121.05(14)
C(15)-C(10)-C(11)	118.94(13)
C(9)-C(10)-C(11)	120.00(14)
O(2)-C(11)-O(1)	116.62(15)
O(2)-C(11)-C(10)	126.62(15)
O(1)-C(11)-C(10)	116.76(12)
C(14)-C(12)-C(8)	122.22(13)
C(14)-C(12)-C(13)	59.30(11)
C(8)-C(12)-C(13)	119.70(13)
C(14)-C(12)-H(12)	114.8
C(8)-C(12)-H(12)	114.8
C(13)-C(12)-H(12)	114.8
C(14)-C(13)-C(12)	59.92(11)
C(14)-C(13)-H(13A)	117.8
C(12)-C(13)-H(13A)	117.8
C(14)-C(13)-H(13B)	117.8
C(12)-C(13)-H(13B)	117.8
H(13A)-C(13)-H(13B)	114.9
C(13)-C(14)-C(12)	60.78(11)
C(13)-C(14)-H(14A)	117.7
C(12)-C(14)-H(14A)	117.7

C(13)-C(14)-H(14B)	117.7
C(12)-C(14)-H(14B)	117.7
H(14A)-C(14)-H(14B)	114.8
C(16)-C(15)-C(10)	120.24(15)
C(16)-C(15)-H(15)	119.9
C(10)-C(15)-H(15)	119.9
C(15)-C(16)-C(17)	119.61(15)
C(15)-C(16)-H(16)	120.2
C(17)-C(16)-H(16)	120.2
C(18)-C(17)-C(16)	120.92(16)
C(18)-C(17)-H(17)	119.5
C(16)-C(17)-H(17)	119.5
C(17)-C(18)-C(9)	120.94(15)
C(17)-C(18)-H(18)	119.5
C(9)-C(18)-H(18)	119.5
C(11)-O(1)-C(7)	123.27(11)

Symmetry transformations used to generate equivalent atoms:

Table S4. Anisotropic displacement parameters (A^2 x 10^3) for A.

The anisotropic displacement factor exponent takes the form:

-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
C(1)	73(1)	68(1)	73(1)	-7(1)	41(1)	-4(1)
C(2)	76(1)	72(1)	112(2)	-25(1)	54(1)	-9(1)
C(3)	70(1)	77(1)	101(2)	-25(1)	28(1)	15(1)
C(4)	92(1)	73(1)	71(1)	-2(1)	31(1)	31(1)
C(5)	82(1)	63(1)	64(1)	6(1)	38(1)	21(1)
C(6)	69(1)	55(1)	57(1)	-2(1)	36(1)	6(1)
C(7)	70(1)	51(1)	50(1)	1(1)	37(1)	-2(1)
C(8)	63(1)	51(1)	44(1)	0(1)	33(1)	-3(1)
C(9)	65(1)	52(1)	44(1)	-2(1)	36(1)	-3(1)
C(10)	73(1)	56(1)	48(1)	-3(1)	40(1)	-7(1)
C(11)	80(1)	54(1)	61(1)	-3(1)	46(1)	-8(1)
C(12)	66(1)	50(1)	54(1)	2(1)	34(1)	-5(1)
C(13)	98(1)	55(1)	78(1)	-8(1)	53(1)	-5(1)
C(14)	90(1)	64(1)	69(1)	-9(1)	28(1)	-12(1)
C(15)	79(1)	69(1)	59(1)	-11(1)	45(1)	-21(1)
C(16)	64(1)	90(1)	62(1)	-3(1)	37(1)	-9(1)

C(17)	64(1)	79(1)	62(1)	3(1)	36(1)	4(1)
C(18)	67(1)	59(1)	57(1)	1(1)	36(1)	0(1)
O(1)	78(1)	49(1)	68(1)	6(1)	43(1)	2(1)
O(2)	108(1)	51(1)	102(1)	2(1)	63(1)	-11(1)

Table S5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for A.

	x	y	z	U(eq)
H(1)	2598	6008	2525	87
H(2)	3878	5951	3749	104
H(3)	4457	4888	5456	115
H(4)	3769	3851	5951	106
H(5)	2500	3820	4717	87
H(12)	1355	7576	1625	69
H(13A)	1012	9114	2446	91
H(13B)	892	7967	3102	91
H(14A)	2151	7335	4053	102
H(14B)	2270	8481	3396	102
H(15)	-825	3196	653	80
H(16)	-1687	4804	-324	86
H(17)	-1303	6903	-240	83
H(18)	-69	7416	843	73

Table S6. Torsion angles [deg] for A.

C(6)-C(1)-C(2)-C(3)	-1.0(2)
C(1)-C(2)-C(3)-C(4)	0.5(3)
C(2)-C(3)-C(4)-C(5)	0.9(3)
C(3)-C(4)-C(5)-C(6)	-2.0(2)
C(2)-C(1)-C(6)-C(5)	0.0(2)
C(2)-C(1)-C(6)-C(7)	-178.93(14)
C(4)-C(5)-C(6)-C(1)	1.5(2)
C(4)-C(5)-C(6)-C(7)	-179.57(14)
C(1)-C(6)-C(7)-C(8)	-48.2(2)
C(5)-C(6)-C(7)-C(8)	132.94(16)
C(1)-C(6)-C(7)-O(1)	132.61(14)
C(5)-C(6)-C(7)-O(1)	-46.30(17)
O(1)-C(7)-C(8)-C(9)	-1.35(19)
C(6)-C(7)-C(8)-C(9)	179.50(13)
O(1)-C(7)-C(8)-C(12)	178.44(12)
C(6)-C(7)-C(8)-C(12)	-0.7(2)
C(7)-C(8)-C(9)-C(10)	1.48(18)
C(12)-C(8)-C(9)-C(10)	-178.32(12)
C(7)-C(8)-C(9)-C(18)	-178.58(13)
C(12)-C(8)-C(9)-C(18)	1.61(19)

C(18)-C(9)-C(10)-C(15)	-1.14(19)
C(8)-C(9)-C(10)-C(15)	178.80(12)
C(18)-C(9)-C(10)-C(11)	178.20(12)
C(8)-C(9)-C(10)-C(11)	-1.86(19)
C(15)-C(10)-C(11)-O(2)	1.6(2)
C(9)-C(10)-C(11)-O(2)	-177.79(14)
C(15)-C(10)-C(11)-O(1)	-178.57(12)
C(9)-C(10)-C(11)-O(1)	2.07(19)
C(7)-C(8)-C(12)-C(14)	-45.2(2)
C(9)-C(8)-C(12)-C(14)	134.55(16)
C(7)-C(8)-C(12)-C(13)	-115.66(16)
C(9)-C(8)-C(12)-C(13)	64.14(18)
C(8)-C(12)-C(13)-C(14)	112.04(16)
C(8)-C(12)-C(14)-C(13)	-107.87(16)
C(9)-C(10)-C(15)-C(16)	0.0(2)
C(11)-C(10)-C(15)-C(16)	-179.39(13)
C(10)-C(15)-C(16)-C(17)	1.1(2)
C(15)-C(16)-C(17)-C(18)	-0.9(2)
C(16)-C(17)-C(18)-C(9)	-0.3(2)
C(10)-C(9)-C(18)-C(17)	1.3(2)
C(8)-C(9)-C(18)-C(17)	-178.62(13)
O(2)-C(11)-O(1)-C(7)	177.89(13)

C(10)-C(11)-O(1)-C(7)	-1.99(18)
C(8)-C(7)-O(1)-C(11)	1.7(2)
C(6)-C(7)-O(1)-C(11)	-178.98(11)

Symmetry transformations used to generate equivalent atoms: