Supporting Information

Visible-light photocatalytic bicyclization of *β*-alkynyl propenones for accessing diastereoenriched *syn*-fluoren-9-ones

Zheng-Jia Shen,[a] Hao-Nan Shi,[a] Wen-Juan Hao,*,[a] Shu-Jiang Tu,*,[a] and Bo Jiang*,[a]

[a]School of Chemistry & Materials Science, Jiangsu Key Laboratory of Green Synthetic Chemistry for Functional Materials, Jiangsu Normal University, Xuzhou 221116, P. R. China; email: wjhao@jsnu.edu.cn (W.J.H.), laotu@jsnu.edu.cn (S.J.T.), jiangchem@jsnu.edu.cn (B.J.)

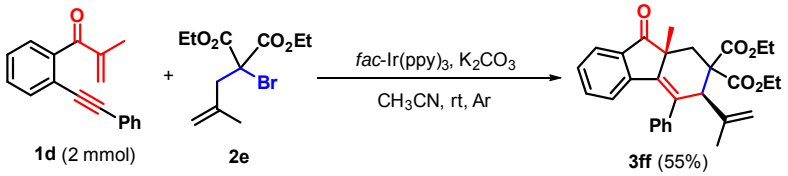
Context

General Information

^{1}H NMR (^{13}C NMR) spectra were measured on a Bruker DPX 400 MHz spectrometer in $CDCl_3$ (DMSO-d_6) with chemical shift (δ) given in ppm relative to TMS as internal standard [(s = singlet, d = doublet, t = triplet, brs = broad singlet, m = multiplet), coupling constant (Hz)]. HRMS (APCI) was determined by using microTOF-QII HRMS/MS instrument (BRUKER). X-Ray crystallographic analysis was performed with a Siemens SMART CCD and a Siemens P4 diffractometer.

To expand potential application of this method, an amplification reaction was performed under the standard conditions. We were delighted to find that *syn*-product **3ff** was obtained in a 55% yield on a 2 mmol scale (Scheme S1).

Scheme S1. Amplification Reaction for the Synthesis of 3ff



Synthetic Details: Under the argon conditions, photocatalyst *fac*-Ir(ppy)$_3$ (0.02 mol, 13 mg), K_2CO_3 (2.0 mmol, 276 mg) were added into a dry 50-mL-Schlenk tube. Then, β-alkynyl propanone **1d** (2.0 mmol, 492 mg) and diethyl α-isobutenyl-α-bromomalonate **2e** (4.0 mmol, 1172 mg) were added into the above reaction system. Next, CH_3CN (24.0 mL) as injected slowly into as injected into the above reaction mixture. The solution was stirred at room temperature with the irradiation of blue LEDs (12 W) for 28.0 hour. After completion of the reaction (TLC monitored), solvent was removed under vacuum to give the crude product, which was purified by flash column chromatography (pentane/ ethyl acetate = 45:1) to afford the desired compound (504 mg).

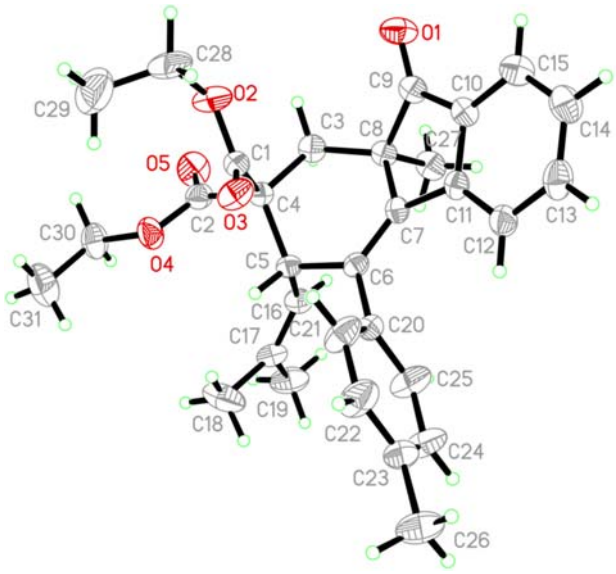
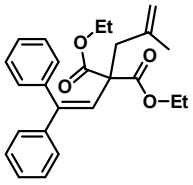
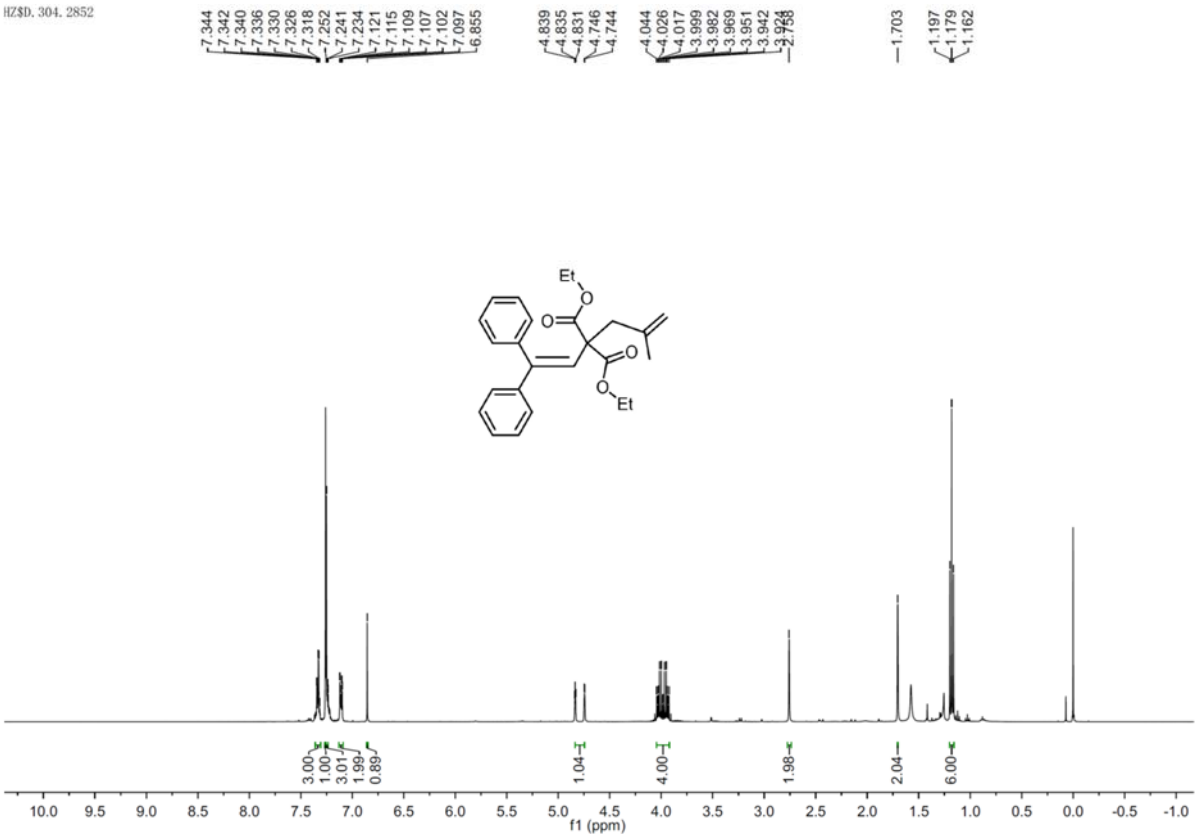


Figure 1. The ORTEP Drawing of **3a**

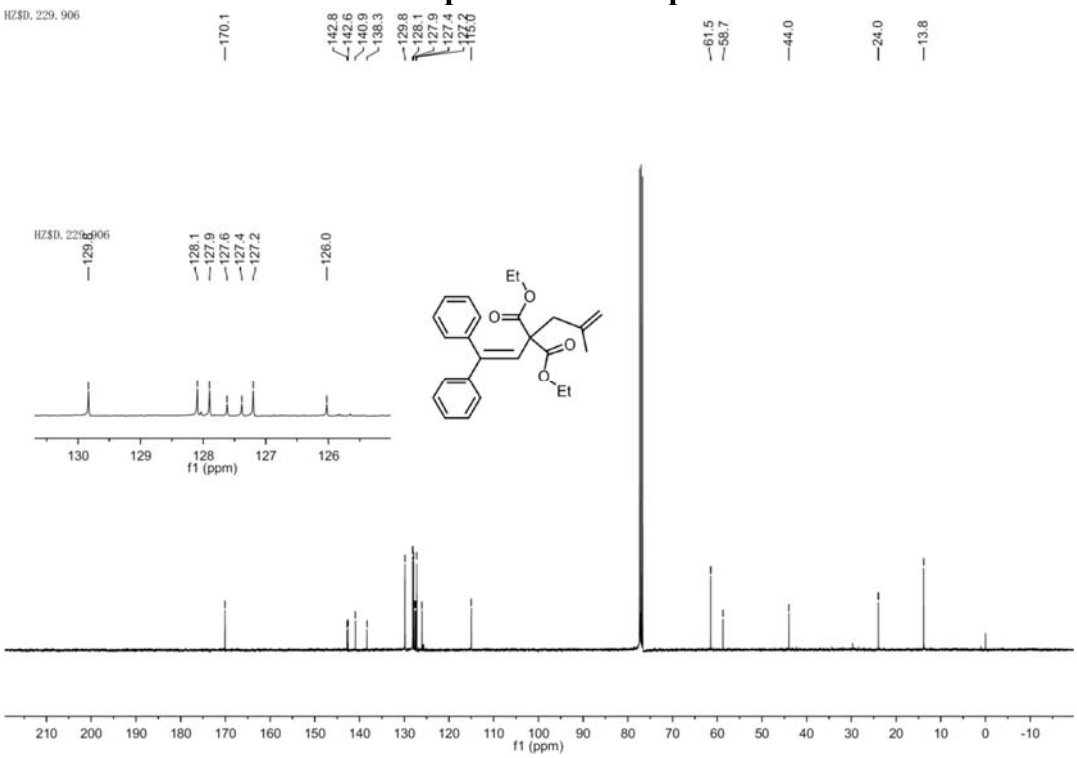
Diethyl 2-(2,2-diphenylvinyl)-2-(2-methylallyl)malonate (**4**)



Oil, 35% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36–7.31 (m, 3H), 7.26 (s, 1H), 7.25–7.23 (m, 3H), 7.13–7.09 (m, 2H), 6.85 (s, 1H), 4.84–4.75 (m, 1H), 4.05–3.90 (m, 4H), 2.76 (s, 2H), 1.70 (s, 2H), 1.20–1.16 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.1, 142.8, 142.6, 140.9, 138.3, 129.8, 128.1, 127.9, 127.6, 127.4, 127.2, 126.0, 115.0, 61.5, 58.7, 44.0, 24.0, 13.9. IR (KBr, ν, cm^{-1}) 2980, 1735, 1598, 1444, 1367, 1095, 863, 702. HRMS (ESI) m/z calcd for $C_{25}H_{28}FO_4Na$, 415.1885 $[M+Na]^+$; found 415.1888.



^{1}H NMR Spectrum of Compound 4



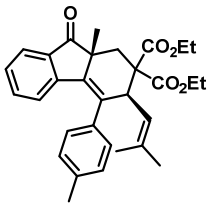
^{13}C NMR Spectrum of Compound 4

General Procedure for the Synthesis of Products 3

Example for the synthesis of **3a**

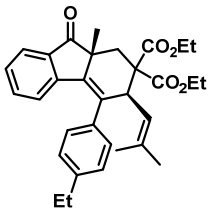
Under the argon conditions, *fac*-Ir(ppy)$_3$ (0.004 mmol, 2.6 mg) and K_2CO_3 (0.4 mmol, 55.2 mg) were added into a dry 25-mL-Schlenk tube. Then, *β*-alkynyl propanone **1a** (0.4 mmol, 104 mg) and α-isopentenyl-α-bromomalonate **2a** (0.8 mmol, 234 mg) were added into the above reaction system. Next, CH_3CN (6.0 mL) was injected into the above reaction mixture. The solution was stirred at room temperature with the irradiation of blue LEDs (12 W) for 24.0 hour. After completion of the reaction (TLC monitored), solvent was removed under vacuum to give the crude product, which was purified by flash column chromatography (pentane/ ethyl acetate = 45:1) to afford the desired compound **3a**.

Diethyl 9a-methyl-3-(2-methylprop-1-en-1-yl)-9-oxo-4-(p-tolyl)-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3a**)



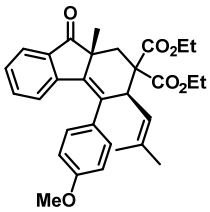
White solid, 118 mg, 61% yield, mp 135-137 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70-7.67 (m, 1H), 7.26-7.14 (m, 6H), 6.72 (d, *J* = 7.6 Hz, 1H), 5.24 (d, *J* = 10.8 Hz, 1H), 4.23-4.19 (m, 1H), 4.14-4.08 (m, 2H), 3.94-3.88 (m, 2H), 3.35-3.31 (m, 1H), 2.42 (d, *J* = 14.0 Hz, 4H), 1.69 (d, *J* = 0.8 Hz, 3H), 1.50 (s, 3H), 1.33 (d, *J* = 0.8 Hz, 3H), 1.20-1.16 (m, 3H), 0.93-0.89 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.1, 170.6, 169.4, 146.9, 137.7(1), 137.7(2), 137.4, 136.3, 136.0, 134.0, 133.8, 128.0, 124.0, 124.0, 118.4, 61.7, 61.5, 60.0, 49.8, 48.8, 31.8, 30.6, 26.3, 21.4, 18.1, 14.0, 13.6. IR (KBr, ν, cm^{-1}) 2979, 1734, 1576, 1473, 1300, 1092, 852, 458. HRMS (APCI-TOF) m/z calcd for $C_{31}H_{34}O_5Na$, 509.2298 $[M+Na]^+$; found 509.2275.

Diethyl 4-(4-ethylphenyl)-9a-methyl-3-(2-methylprop-1-en-1-yl)-9-oxo-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarb oxylate (*rac*-**3b**)



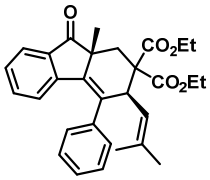
White solid, 124 mg, 62% yield, mp 133-135 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 7.2 Hz, 1H), 7.38 (s, 1H), 7.25 (s, 1H), 7.21 (d, *J* = 3.6 Hz, 3H), 7.18 (d, *J* = 7.2 Hz, 1H), 6.72 (d, *J* = 7.6 Hz, 1H), 5.24 (d, *J* = 10.4 Hz, 1H), 4.21 (d, *J* = 9.6 Hz, 1H), 4.12 (d, *J* = 7.2 Hz, 2H), 3.93-3.91(m, 2H), 3.35-3.30 (m, 1H), 2.74-2.68 (m, 2H), 2.42 (d, *J* = 15.2 Hz, 1H), 1.70 (s, 3H), 1.51 (s, 3H), 1.36 (s, 3H), 1.30-1.27 (m, 3H), 1.19 (d, *J* = 6.8 Hz, 3H), 0.93-0.90 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.1, 170.6, 169.4, 146.9, 143.8, 137.9, 137.8, 136.3, 136.0, 134.0, 133.8, 128.0, 124.1, 124.0, 118.4, 61.7, 61.5, 60.0, 49.8, 48.9, 31.8, 31.4, 30.6, 28.7, 26.3, 18.1, 15.5, 14.0, 13.6. IR (KBr, ν, cm^{-1}) 2967, 1731, 1575, 1471, 1300, 1094, 854, 466. HRMS (APCI-TOF) m/z calcd for $C_{32}H_{36}O_5Na$, 523.2360 $[M+Na]^+$; found 523.2371.

Diethyl 4-(4-methoxyphenyl)-9a-methyl-3-(2-methylprop-1-en-1-yl)-9-oxo-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-3c)



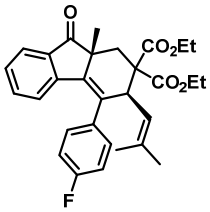
White solid, 136 mg, 68% yield, mp 110-112 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 6.8 Hz, 1H), 7.38 (s, 1H), 7.24-7.18 (m, 3H), 6.94 (d, *J* = 6.8 Hz, 2H), 6.75 (d, *J* = 7.2 Hz, 1H), 5.23 (d, *J* = 9.6 Hz, 1H), 4.20 (d, *J* = 10.8 Hz, 1H), 4.14-4.09 (m, 2H), 3.93-3.89 (m, 2H), 3.87 (s, 3H), 3.33 (d, *J* = 15.2 Hz, 1H), 2.43 (d, *J* = 15.2 Hz, 1H), 1.69 (s, 3H), 1.50 (s, 3H), 1.34 (s, 3H), 1.21-1.17 (m, 3H), 0.93-0.8 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.1(1), 206.1(2), 170.7, 169.4, 159.2, 146.9, 137.6, 136.3, 136.1, 134.0, 133.8, 133.0, 128.0, 124.0, 123.9, 118.4, 61.7, 61.5, 60.0, 55.3, 49.8, 49.0, 31.8, 30.6, 26.3, 18.1, 14.0, 13.6. IR (KBr, ν, cm^{-1}) 2981, 1731, 1540, 1465, 1295, 1089, 853, 466. HRMS (APCI-TOF) m/z calcd for $C_{31}H_{34}O_6$ Na, 525.2253 $[M+Na]^+$; found 525.2240.

Diethyl 9a-methyl-3-(2-methylprop-1-en-1-yl)-9-oxo-4-phenyl-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3d**)



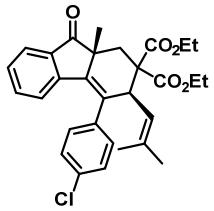
White solid, 117 mg, 62% yield, mp 112-113 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 7.6 Hz, 1H), 7.38 (s, 4H), 7.26 (s, 1H), 7.22 (d, *J* = 7.6 Hz, 1H), 7.19-7.15 (m, 1H), 6.64 (d, *J* = 7.6 Hz, 1H), 5.25 (d, *J* = 10.4 Hz, 1H), 4.22 (d, *J* = 10.8 Hz, 1H), 4.14-4.11 (m, 2H), 3.96-3.89 (m, 2H), 3.34 (d, *J* = 15.2 Hz, 1H), 2.44 (d, *J* = 15.2 Hz, 1H), 1.70 (s, 3H), 1.51 (s, 3H), 1.31 (s, 3H), 1.20-1.64 (m, 3H), 0.95-0.91 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.9, 170.6, 169.3, 146.8, 140.8, 137.5, 136.4, 136.2, 134.0, 133.9, 128.1, 127.8, 124.0, 124.0, 118.3, 61.8, 61.5, 60.0, 49.8, 48.7, 31.8, 30.5, 26.3, 18.0, 14.0, 13.6. IR (KBr, ν, cm^{-1}) 2982, 1730, 1576, 1471, 1293, 1080, 850, 457. HRMS (APCI-TOF) m/z calcd for $C_{30}H_{32}O_5$ Na, 495.2147 $[M+Na]^+$; found 495.2169.

Diethyl 4-(4-fluorophenyl)-9a-methyl-3-(2-methylprop-1-en-1-yl)-9-oxo-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicar boxylate (*rac*-**3e**)



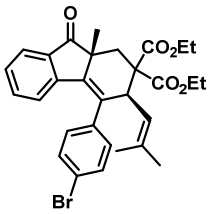
White solid, 113 mg, 58% yield, mp 123-125 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 7.2 Hz, 1H), 7.45 (s, 1H), 7.24-7.19 (m, 3H), 7.11 (d, *J* = 7.2 Hz, 2H), 6.63 (d, *J* = 7.6 Hz, 1H), 5.22 (d, *J* = 10.4 Hz, 1H), 4.16 (d, *J* = 11.2 Hz, 1H), 4.14-4.08 (m, 2H), 3.95-3.89 (m, 2H), 3.35-3.31(m, 1H), 2.45 (d, *J* = 15.6 Hz, 1H), 1.70 (s, 3H), 1.50 (s, 3H), 1.33 (s, 3H), 1.20-1.16 (m, 3H), 0.95-0.93 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.6, 170.7, 169.2, 163.7($^1J_{CF}$ = 245.1 Hz), 161.2, 146.6, 136.8($^1J_{CF}$ = 3.3 Hz), 136.7, 136.6, 136.6, 134.0($^1J_{CF}$ = 10.8 Hz), 133.9, 128.3, 124.1($^1J_{CF}$ = 19.9 Hz), 123.9, 118.1, 61.8, 61.6, 60.0, 49.8, 48.7, 31.8, 30.5, 26.3, 18.1, 14.0, 13.6. IR (KBr, ν, cm^{-1}) 2979, 1731, 1507, 1469, 1296, 1080, 855, 455. HRMS (APCI-TOF) m/z calcd for $C_{30}H_{31}FO_5$Na, 513.2053 $[M+Na]^+$; found 513.2095.

Diethyl 4-(4-chlorophenyl)-9a-methyl-3-(2-methylprop-1-en-1-yl)-9-oxo-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3f**)



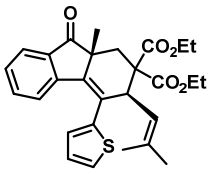
White solid, 121 mg, 60% yield, mp 90-92 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (d, *J* = 7.2 Hz, 1H), 7.38 (d, *J* = 7.6 Hz, 3H), 7.28 (s, 1H), 7.25-7.20 (m, 2H), 6.66 (d, *J* = 7.6 Hz, 1H), 5.22 (d, *J* = 10.4 Hz, 1H), 4.16-4.08 (m, 3H), 3.95 -3.90 (m, 2H), 3.36-3.30 (m, 1H), 2.45 (d, *J* = 15.2 Hz, 1H), 1.70 (s, 3H), 1.50 (s, 3H), 1.34 (s, 3H), 1.20-1.66 (m, 3H), 0.96-0.92 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.5, 170.7, 169.2, 146.4, 139.4, 136.7, 136.6, 136.3, 134.1, 134.0, 133.7, 128.4, 124.1, 124.0, 118.1, 61.8, 61.6, 60.0, 49.9, 48.5, 31.8, 30.5, 26.3, 18.1, 14.0, 13.6. IR (KBr, ν, cm^{-1}) 2978, 1732, 1574, 1465, 1293, 1094, 856, 418. HRMS (APCI-TOF) m/z calcd for $C_{30}H_{31}ClO_5Na$, 529.1758 $[M+Na]^+$; found 529.1780.

Diethyl 4-(4-bromophenyl)-9a-methyl-3-(2-methylprop-1-en-1-yl)-9-oxo-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3g**)



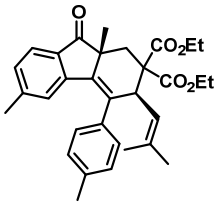
White solid, 134 mg, 61% yield, mp 108-109 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (d, *J* = 6.8 Hz, 1H), 7.54 (d, *J* = 8.4 Hz, 2H), 7.34 (s, 2H), 7.25-7.11 (m, 2H), 6.66 (d, *J* = 7.2 Hz, 1H), 5.22 (d, *J* = 10.8 Hz, 1H), 4.15-4.08 (m, 3H), 3.95- 3.90 (m, 2H), 3.32 (d, *J* = 15.2 Hz, 1H), 2.44 (d, *J* = 15.6 Hz, 1H), 1.70 (s, 3H), 1.49 (s, 3H), 1.35 (s, 3H), 1.20-1.64 (m, 3H), 0.96-0.92 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.5, 170.7, 169.2, 146.4, 139.8, 136.8, 136.6, 136.2, 134.1, 134.0, 128.4, 124.1, 124.0, 121.9, 118.1, 61.8, 61.6, 60.0, 49.9, 48.5, 31.8, 30.5, 26.3, 18.1, 14.0, 13.6. IR (KBr, ν, cm^{-1}) 2982, 1726, 1601, 1509, 1299, 1102, 860, 436. HRMS (APCI-TOF) m/z calcd for $C_{30}H_{31}O_5Na$, 573.1253 $[M+Na]^+$; found 573.1251.

Diethyl 9a-methyl-3-(2-methylprop-1-en-1-yl)-9-oxo-4-(thiophen-2-yl)-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3h**)



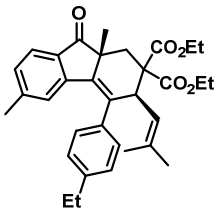
White solid, 112 mg, 59% yield, mp 110-112 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73-7.69 (m, 1H), 7.40 (d, *J* = 5.2 Hz, 1H), 7.30-7.27 (m, 2H), 7.09-7.06 (m, 1H), 7.04-6.97 (m, 2H), 5.23 (d, *J* = 10.4 Hz, 1H), 4.21 (d, *J* = 9.6 Hz, 1H), 4.14 -4.08 (m, 2H), 3.97-3.91 (m, 2H), 3.34-3.30 (m, 1H), 2.44 (d, *J* = 15.6 Hz, 1H), 1.72 (s, 3H), 1.50 (s, 3H), 1.43 (s, 3H), 1.21-1.17 (m, 3H), 0.96-0.93 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.4, 170.5, 169.2, 146.4, 141.6, 139.3, 137.0, 134.2, 133.9, 130.6, 128.6, 127.4, 126.9, 126.5, 124.1, 124.0, 118.2, 61.8, 61.5, 60.0, 50.0, 49.6, 31.6, 30.6, 26.4, 18.1, 14.0, 13.7. IR (KBr, ν, cm^{-1}) 2980, 1734, 1576, 1446, 1365, 1093, 851, 451. HRMS (APCI-TOF) m/z calcd for $C_{28}H_{30}O_5SNa$, 501.1712 $[M+Na]^+$; found 501.1765.

Diethyl 6,9a-dimethyl-3-(2-methylprop-1-en-1-yl)-9-oxo-4-(p-tolyl)-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3i**)



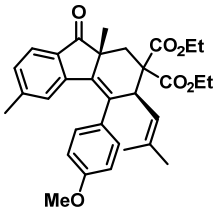
White solid, 122 mg, 63% yield, mp 104-106 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (d, *J* = 7.6 Hz, 1H), 7.27 (s, 1H), 7.19 (d, *J* = 6.8 Hz, 3H), 7.04 (d, *J* = 8.0 Hz, 1H), 6.50 (s, 1H), 5.23 (d, *J* = 10.8 Hz, 1H), 4.23-4.18 (m, 1H), 4.23-4.18 (m, 2H), 3.94-3.87 (m, 2H), 3.33-3.29 (m, 1H), 2.43-2.38 (m, 4H), 2.10 (s, 3H), 1.69 (s, 3H), 1.49 (s, 3H), 1.33 (s, 3H), 1.1-1.16 (m, 3H), 0.94-0.90 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.7, 170.6, 169.4, 147.3, 144.8, 137.8, 137.4, 137.3, 136.2, 136.1, 131.6, 129.2, 124.6, 123.8, 118.5, 61.7, 61.4, 60.0, 49.9, 48.8, 31.9, 30.6, 26.3, 22.2, 21.4, 18.1, 14.0, 13.6. IR (KBr, ν, cm^{-1}) 2982, 1726, 1540, 1445, 1299, 1022, 860, 436. HRMS (APCI-TOF) m/z calcd for $C_{32}H_{36}O_5Na$, 523.2460 $[M+Na]^+$; found 523.2476.

Diethyl 4-(4-ethylphenyl)-6,9a-dimethyl-3-(2-methylprop-1-en-1-yl)-9-oxo-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3j**)



White solid, 119 mg, 58% yield, mp 96-98 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (d, *J* = 7.6 Hz, 1H), 7.28 (s, 1H), 7.22 (d, *J* = 7.2 Hz, 3H), 7.04 (d, *J* = 7.6 Hz, 1H), 6.42 (s, 1H), 5.24 (d, *J* = 10.4 Hz, 1H), 4.22 (d, *J* = 9.6 Hz, 1H), 4.14-4.09 (m, 2H), 3.94-3.88 (m, 2H), 3.33-3.29 (m, 1H), 2.74-2.68 (m, 2H), 2.41 (d, *J* = 15.2 Hz, 1H), 2.09 (s, 3H), 1.70 (s, 3H), 1.49 (s, 3H), 1.34 (s, 3H), 1.29-1.26 (m, 3H), 1.20-1.17 (m, 3H), 0.95-0.91 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.7, 170.6, 169.4, 147.3, 144.7, 143.9, 138.0, 137.5, 136.2, 136.1, 131.6, 129.1, 124.6, 123.8, 118.5, 61.7, 61.4, 60.0, 49.9, 48.7, 31.9, 30.6, 28.8, 26.3, 22.2, 18.1, 15.9, 14.0, 13.6. IR (KBr, ν, cm^{-1}) 2970, 1728, 1559, 1455, 1313, 1095, 858, 454. HRMS (APCI-TOF) m/z calcd for $C_{33}H_{38}O_5Na$, 537.2617 $[M+Na]^+$; found 537.2644.

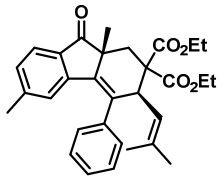
Diethyl 4-(4-methoxyphenyl)-6,9a-dimethyl-3-(2-methylprop-1-en-1-yl)-9-oxo-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3k**)



White solid, 142 mg, 69% yield, mp 95-97 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (d, *J* = 8.0 Hz, 1H), 7.40 (s, 1H), 7.24 -7.13 (m, 1H), 7.05 (d, *J* = 7.6 Hz, 1H), 6.94 (d, *J* = 6.4 Hz, 2H), 6.55 (s, 1H), 5.23 (d, *J* = 10.8 Hz, 1H), 4.21-4.09 (m, 1H), 4.14-4.09 (m, 2H), 3.91 (d, *J* = 6.8 Hz, 2H), 3.87 (s, 3H), 3.33-3.29 (m, 1H), 2.41 (d, *J* = 15.2 Hz, 1H), 2.13 (s, 3H), 1.69 (s, 3H), 1.49 (s, 3H), 1.34 (s, 3H), 1.20-1.67 (m, 3H), 0.94-0.91 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.7, 170.7, 169.4, 159.2, 147.3, 144.8, 137.2, 136.2, 136.1, 133.1, 131.6, 129.2, 124.4, 123.8, 118.4, 61.7, 61.5, 60.0, 55.3, 49.9, 49.0, 32.0, 30.6, 26.3, 22.3, 18.1, 14.0, 13.6. IR (KBr, ν, cm^{-1}) 2968, 1723, 1570, 1476, 1330,

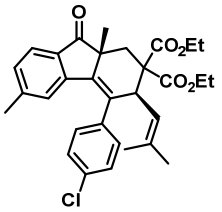
1047, 835, 439. HRMS (APCI-TOF) m/z calcd for $C_{32}H_{36}O_6Na$, 539.2410 $[M+Na]^+$; found 539.2449.

Diethyl 6,9a-dimethyl-3-(2-methylprop-1-en-1-yl)-9-oxo-4-phenyl-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3l**)



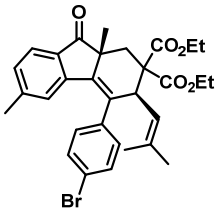
White solid, 114 mg, 59% yield, mp 100-102 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (d, *J* = 7.6 Hz, 1H), 7.38 (s, 5H), 7.05 (d, *J* = 8.0 Hz, 1H), 6.39 (s, 1H), 5.25 (d, *J* = 10.8 Hz, 1H), 4.23-4.19 (m, 1H), 4.14-4.09 (m, 2H), 3.96-3.90 (m, 2H), 3.34-3.30 (m, 1H), 2.43 (d, *J* = 15.2 Hz, 1H), 2.09 (s, 3H), 1.70 (s, 3H), 1.50 (s, 3H), 1.32 (s, 3H), 1.20-1.16 (m, 3H), 0.96-0.93 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.5, 170.7, 169.4, 147.1, 144.8, 140.9, 137.3, 136.3, 136.2, 131.6, 129.3, 127.7, 124.6, 123.8, 118.4, 61.7, 61.5, 60.0, 49.9, 48.7, 32.0, 30.6, 26.3, 22.2, 18.1, 14.0, 13.6. IR (KBr, ν, cm^{-1}) 2965, 1730, 1576, 1491, 1299, 1078, 857, 438. HRMS (APCI-TOF) m/z calcd for $C_{31}H_{34}O_5Na$, 509.2304 $[M+Na]^+$; found 509.2350.

***Diethyl 4-(4-chlorophenyl)-6,9a-dimethyl-3-(2-methylprop-1-en-1-yl)-9-oxo-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylat*e** (*rac*-**3m**)



White solid, 112 mg, 54% yield, mp 86-88 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60-7.52 (m, 3H), 7.34 (s, 2H), 7.08 (d, *J* = 7.6 Hz, 1H), 6.45 (s, 1H), 5.22 (d, *J* = 10.8 Hz, 1H), 4.15-4.09 (m, 3H), 3.95-3.90(m, 2H), 3.31 (d, *J* = 15.6 Hz, 1H), 2.43 (d, *J* = 15.6 Hz, 1H), 2.15 (s, 3H), 1.71 (s, 3H), 1.55 (s, 3H), 1.35 (s, 3H), 1.20-1.17 (m, 3H), 0.97-0.94 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.1, 170.7, 169.2, 146.8, 145.0, 139.9, 136.7, 135.9, 131.7, 129.6, 124.4, 124.0, 121.8, 118.2, 61.8, 61.5, 60.0, 50.0, 48.5, 31.9, 30.5, 26.3, 22.3, 18.1, 14.0, 13.7. IR (KBr, ν, cm^{-1}) 2981, 1727, 1601, 1485, 1299, 1071, 858, 436. HRMS (APCI-TOF) m/z calcd for $C_{31}H_{33}ClO$ Na, 543.1914 $[M+Na]^+$; found 543.1915.

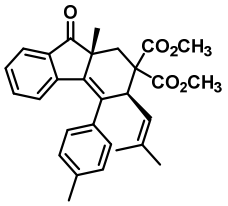
Diethyl 4-(4-bromophenyl)-6,9a-dimethyl-3-(2-methylprop-1-en-1-yl)-9-oxo-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3n**)



White solid, 128 mg, 57% yield, mp 109-111 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, *J* = 8.0 Hz, 1H), 7.39 (d, *J* = 7.6 Hz, 2H), 7.26 (s, 1H), 7.26-7.25 (m, 1H), 7.08 (d, *J* = 7.6 Hz, 1H), 6.44 (s, 1H), 5.22 (d, *J* = 10.8 Hz, 1H), 4.16-4.09 (m, 3H), 3.96-3.90 (m, 2H), 3.33-3.29 (m, 1H), 2.43 (d, *J* = 15.2 Hz, 1H), 2.14 (s, 3H), 1.71 (s, 3H), 1.49 (s, 3H), 1.35 (s, 3H), 1.20-1.17 (m, 3H), 0.97-0.94 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.1, 170.7, 169.2, 146.8, 145.0, 139.5, 136.7, 136.6, 136.0, 133.6, 131.7, 129.5, 124.4, 124.0, 118.2, 61.8, 61.5, 60.0, 50.0, 48.5, 31.9, 30.5, 26.3, 22.3, 18.1, 14.0, 13.7. IR (KBr, ν, cm^{-1}) 2982, 1726, 1601, 1445, 1299, 1043, 859, 436. HRMS (APCI-TOF) m/z

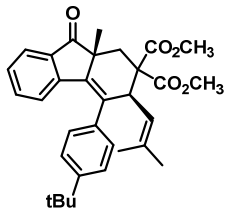
calcd for $C_{31}H_{33}BrO_5Na$, 587.1409 $[M+Na]^+$; found 587.1401.

Dimethyl 9a-methyl-3-(2-methylprop-1-en-1-yl)-9-oxo-4-(p-tolyl)-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3o**)



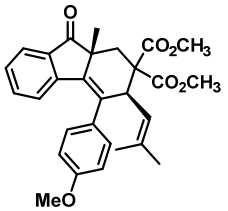
White solid, 119 mg, 65% yield, mp 131-133 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (d, *J* = 7.2 Hz, 1H), 7.24-7.17 (m, 6H), 6.72 (d, *J* = 7.6 Hz, 1H), 5.22 (d, *J* = 10.4 Hz, 1H), 4.20 (d, *J* = 10.8 Hz, 1H), 3.65 (s, 3H), 3.43 (s, 3H), 3.43-3.39 (m, 1H), 2.43 (s, 1H), 2.40 (s, 3H), 1.71 (s, 3H), 1.50 (s, 3H), 1.37 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.9, 171.0, 169.7, 146.8, 137.6, 137.5, 137.4, 136.7, 136.2, 133.9, 133.7, 128.1, 124.1, 124.0, 118.4, 60.0, 52.6, 49.9, 49.0, 31.6, 30.7, 26.4, 21.4, 18.0. IR (KBr, ν, cm^{-1}) 2948, 1734, 1598, 1466, 1332, 1046, 887, 465. HRMS (APCI-TOF) m/z calcd for $C_{29}H_{30}O_5Na$, 481.1991 $[M+Na]^+$; found 481.2001.

Dimethyl 4-(4-(tert-butyl)phenyl)-9a-methyl-3-(2-methylprop-1-en-1-yl)-9-oxo-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3p**)



White solid, 120 mg, 60% yield, mp 188-190 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (d, *J* = 7.2 Hz, 1H), 7.39 (d, *J* = 8.0 Hz, 2H), 7.23-7.18 (m, 4H), 6.71 (d, *J* = 7.6 Hz, 1H), 5.23 (d, *J* = 10.4 Hz, 1H), 4.21 (d, *J* = 10.8 Hz, 1H), 3.65 (s, 3H), 3.43 (s, 3H), 3.32 (d, *J* = 15.2 Hz, 1H), 2.41 (d, *J* = 15.2 Hz, 1H), 1.71 (s, 3H), 1.50 (s, 3H), 1.36 (s, 12H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.9, 171.0, 169.7, 150.8, 146.8, 137.5, 137.4, 136.7, 136.3, 133.9, 133.7, 128.0, 124.1, 124.0, 118.5, 60.0, 52.5, 50.0, 48.9, 34.6, 31.5, 31.4, 30.7, 26.4, 17.9. IR (KBr, ν, cm^{-1}) 2963, 1732, 1594, 1464, 1295, 1078, 859, 458.
HRMS (APCI-TOF) m/z calcd for $C_{32}H_{36}O_5Na$, 523.2460 $[M+Na]^+$; found 523.2507.

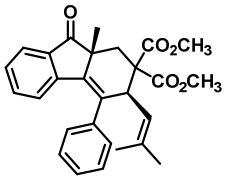
Dimethyl 4-(4-methoxyphenyl)-9a-methyl-3-(2-methylprop-1-en-1-yl)-9-oxo-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3q**)



White solid, 127 mg, 67% yield, mp 167-169 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (d, *J* = 6.8 Hz, 1H), 7.27 (s, 1H), 7.26-7.21 (m, 2H), 7.21-7.17 (m, 1H), 6.93 (d, *J* = 8.4 Hz, 2H), 6.75 (d, *J* = 7.6 Hz, 1H), 5.22 (d, *J* = 10.4 Hz, 1H), 4.21-4.17 (m, 1H), 3.87 (s, 3H), 3.65 (s, 3H), 3.43 (s, 3H), 3.43-3.29 (m, 1H), 2.42 (d, *J* = 15.2 Hz, 1H), 1.70 (s, 3H), 1.50 (s, 3H), 1.38 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.9, 171.1, 169.7, 159.3, 146.9, 137.2, 136.7, 136.3, 134.0, 133.7, 132.9, 128.1, 124.1, 124.0, 118.3, 60.0, 55.3, 52.6, 52.6, 49.9, 49.1, 31.6, 30.7, 26.4, 18.0. IR (KBr, ν, cm^{-1}) 2949, 1731, 1604, 1472, 1289, 1081, 854, 471. HRMS (APCI-TOF) m/z calcd for $C_{29}H_{30}O_6Na$, 497.1940 $[M+Na]^+$;

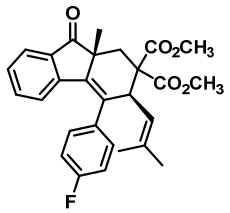
found 497.1998.

Dimethyl 9a-methyl-3-(2-methylprop-1-en-1-yl)-9-oxo-4-phenyl-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3r**)



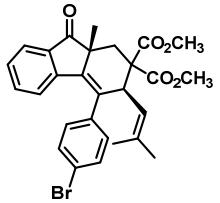
White solid, 101 mg, 57% yield, mp 129-131 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (d, *J* = 7.2 Hz, 1H), 7.39 (d, *J* = 6.8 Hz, 4H), 7.27 (s, 1H), 7.24-7.18 (m, 2H), 6.64 (d, *J* = 7.6 Hz, 1H), 5.23 (d, *J* = 10.8 Hz, 1H), 4.21 (d, *J* = 10.8 Hz, 1H), 3.65 (s, 3H), 3.45 (s, 3H), 3.35-3.30 (m, 1H), 2.43 (d, *J* = 15.6 Hz, 1H), 1.71 (s, 3H), 1.51 (s, 3H), 1.35 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.7, 171.0, 169.7, 146.7, 140.7, 137.2, 136.8, 136.4, 134.0, 133.8, 128.2, 127.8, 124.1, 124.1, 118.3, 59.9, 52.6, 52.6, 49.9, 48.9, 31.6, 30.6, 26.4, 17.9. IR (KBr, ν, cm^{-1}) 2952, 1735, 1576, 1466, 1300, 1081, 851, 455. HRMS (APCI-TOF) m/z calcd for $C_{28}H_{28}O_5Na$, 467.1834 $[M+Na]^+$; found 467.1845.

Dimethyl 4-(4-fluorophenyl)-9a-methyl-3-(2-methylprop-1-en-1-yl)-9-oxo-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dic arboxylate (*rac*-**3s**)



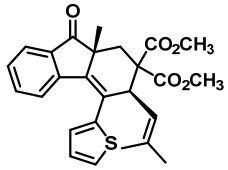
White solid, 116 mg, 63% yield, mp 138-140 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71 (d, *J* = 7.2 Hz, 1H), 7.38 (s, 2H), 7.24-7.19 (m, 2H), 7.12-7.08 (m, 2H), 6.63 (d, *J* = 7.6 Hz, 1H), 5.21 (d, *J* = 10.4 Hz, 1H), 4.15 (d, *J* = 10.8 Hz, 1H), 3.65 (s, 3H), 3.45 (s, 3H), 3.31 (d, *J* = 15.2 Hz, 1H), 2.44 (d, *J* = 15.2 Hz, 1H), 1.71 (s, 3H), 1.49 (s, 3H), 1.36 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.5, 171.1, 169.6, 163.7($^1J_{CF}$ = 245.2 Hz), 161.2, 146.5, 137.0, 136.7($^1J_{CF}$ =5.6 Hz), 136.7($^1J_{CF}$ = 3.3 Hz), 136.6, 136.2, 134.0, 133.8, 128.3, 124.2($^1J_{CF}$ = 20.8 Hz), 124.0, 118.1, 59.9, 52.6, 49.9, 48.9, 31.6, 30.5, 26.3, 17.9. IR (KBr, ν, cm^{-1}) 2954, 1731, 1601, 1467, 1299, 1080, 859, 468. HRMS (APCI-TOF) m/z calcd for $C_{28}H_{27}FO_5Na$, 485.1740 $[M+Na]^+$; found 485.1731.

Dimethyl 4-(4-bromophenyl)-9a-methyl-3-(2-methylprop-1-en-1-yl)-9-oxo-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dic arboxylate (*rac*-**3t**)



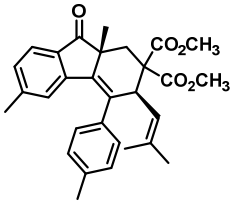
White solid, 123 mg, 59% yield, mp 156-158 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71 (d, *J* = 7.6 Hz, 1H), 7.53 (d, *J* = 8.4 Hz, 2H), 7.28 (d, *J* = 7.2 Hz, 1H), 7.23 (d, *J* = 7.2 Hz, 3H), 6.67 (d, *J* = 7.2 Hz, 1H), 5.20 (d, *J* = 10.8 Hz, 1H), 4.13 (d, *J* = 10.0 Hz, 1H), 3.65 (s, 3H), 3.45 (s, 3H), 3.33-3.28 (m, 1H), 2.44 (d, *J* = 15.6 Hz, 1H), 1.72 (s, 3H), 1.49 (s, 3H), 1.38 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.3, 171.0, 169.5, 146.3, 139.7, 137.2, 136.8, 135.9, 134.1, 133.9, 128.4, 124.2, 124.0, 122.0, 118.1, 59.9, 52.6, 52.6, 50.0, 48.6, 31.6, 30.5, 26.4, 18.0. IR (KBr, ν, cm^{-1}) 2956, 1727, 1600, 1488, 1323, 1074, 843, 436. HRMS (APCI-TOF) m/z calcd for $C_{28}H_{27}BrO_5Na$, 545.0940 $[M+Na]^+$; found 545.0965.

Dimethyl 9a-methyl-3-(2-methylprop-1-en-1-yl)-9-oxo-4-(thiophen-2-yl)-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3u**)



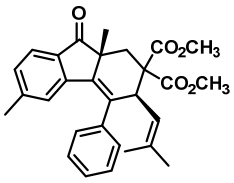
White solid, 102 mg, 57% yield, mp 152-154 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73-7.71 (m, 1H), 7.39 (d, *J* = 4.8 Hz, 1H), 7.31-7.27 (m, 2H), 7.08-7.06 (m, 1H), 7.01-6.98 (m, 2H), 5.22 (d, *J* = 10.8 Hz, 1H), 4.23-4.20 (m, 1H), 3.65 (s, 3H), 3.47 (s, 3H), 3.33-3.29 (m, 1H), 2.42 (d, *J* = 15.2 Hz, 1H), 1.73 (s, 3H), 1.49 (s, 3H), 1.46 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.2, 170.9, 169.5, 146.3, 141.5, 139.5, 137.4, 134.2, 133.9, 130.3, 128.7, 127.3, 126.9, 126.5, 124.1, 124.0, 118.1, 59.9, 52.7, 52.6, 50.2, 49.7, 31.4, 30.7, 26.4, 17.9. IR (KBr, ν, cm^{-1}) 2958, 1732, 1576, 1470, 1297, 1081, 852, 436. HRMS (APCI-TOF) m/z calcd for $C_{26}H_{26}O_5SNa$, 473.1366 $[M+Na]^+$; found 473.1380.

Dimethyl 6,9a-dimethyl-3-(2-methylprop-1-en-1-yl)-9-oxo-4-(p-tolyl)-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3v**)



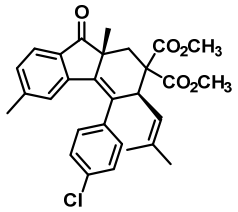
White solid, 124 mg, 66% yield, mp 167-169 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, *J* = 7.6 Hz, 1H), 7.19 (d, *J* = 6.0 Hz, 4H), 7.05 (d, *J* = 7.6 Hz, 1H), 6.50 (s, 1H), 5.22 (d, *J* = 10.8 Hz, 1H), 4.20 (d, *J* = 10.8 Hz, 1H), 3.65 (s, 3H), 3.44 (s, 3H), 3.29 (d, *J* = 15.2 Hz, 1H), 2.41 (s, 4H), 2.11 (s, 3H), 1.70 (s, 3H), 1.48 (s, 3H), 1.37 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.5, 171.0, 169.7, 147.2, 144.8, 137.7, 137.4, 137.1, 136.6, 136.3, 131.5, 129.2, 124.6, 123.9, 118.5, 60.0, 52.5, 50.0, 49.0, 31.7, 30.7, 26.4, 22.2, 21.4, 18.0. IR (KBr, ν, cm^{-1}) 2952, 1725, 1600, 1509, 1314, 1079, 851, 448. HRMS (APCI-TOF) m/z calcd for $C_{30}H_{32}O_5Na$, 495.2147 $[M+Na]^+$; found 495.2168.

Dimethyl 6,9a-dimethyl-3-(2-methylprop-1-en-1-yl)-9-oxo-4-phenyl-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3w**)



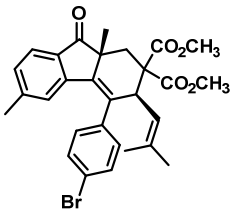
White solid, 106 mg, 58% yield, mp 143-145 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (d, *J* = 7.6 Hz, 1H), 7.37 (s, 5H), 7.05 (d, *J* = 8.0 Hz, 1H), 6.39 (s, 1H), 5.23 (d, *J* = 10.4 Hz, 1H), 4.21 (d, *J* = 10.8 Hz, 1H), 3.64 (d, *J* = 0.8 Hz, 3H), 3.45 (d, *J* = 0.8 Hz, 3H), 3.30 (d, *J* = 15.2 Hz, 1H), 2.41 (d, *J* = 15.2 Hz, 1H), 2.08 (s, 3H), 1.71 (s, 3H), 1.49 (s, 3H), 1.35 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.3, 171.0, 169.7, 147.1, 144.8, 140.8, 136.9, 136.7, 136.4, 131.5, 129.3, 127.8, 124.6, 123.9, 118.3, 60.0, 52.6, 52.5, 50.0, 48.8, 31.7, 30.6, 26.4, 22.2, 17.9. IR (KBr, ν, cm^{-1}) 2952, 1724, 1602, 1491, 1302, 1071, 834, 441. HRMS (APCI-TOF) m/z calcd for $C_{29}H_{30}O_5Na$, 481.1991 $[M+Na]^+$; found 481.2033.

Dimethyl 4-(4-chlorophenyl)-6,9a-dimethyl-3-(2-methylprop-1-en-1-yl)-9-oxo-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3x**)



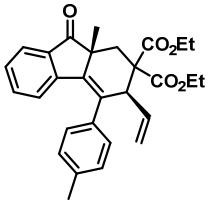
White solid, 116 mg, 59% yield, mp 145-147 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, *J* = 7.6 Hz, 1H), 7.37 (d, *J* = 8.0 Hz, 3H), 7.22 (s, 1H), 7.07 (d, *J* = 7.6 Hz, 1H), 6.44 (s, 1H), 5.19 (d, *J* = 10.8 Hz, 1H), 4.12 (d, *J* = 10.4 Hz, 1H), 3.64 (s, 3H), 3.44 (s, 3H), 3.29 (d, *J* = 15.2 Hz, 1H), 2.41 (d, *J* = 15.2 Hz, 1H), 2.13 (s, 3H), 1.71 (s, 3H), 1.47 (s, 3H), 1.37 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.9, 171.1, 169.5, 146.7, 145.0, 139.4, 137.0, 136.8, 135.6, 133.7, 131.6, 129.6, 124.5, 124.0, 118.2, 59.9, 52.6, 52.6, 50.1, 48.7, 31.7, 30.5, 26.3, 22.3, 17.9. IR (KBr, ν, cm^{-1}) 2956, 1735, 1603, 1487, 1299, 1014, 858, 479. HRMS (APCI-TOF) m/z calcd for $C_{29}H_{29}ClO_5Na$, 515.1601 $[M+Na]^+$; found 515.1625.

Dimethyl 4-(4-bromophenyl)-6,9a-dimethyl-3-(2-methylprop-1-en-1-yl)-9-oxo-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3y**)



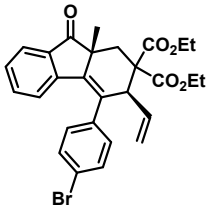
White solid, 120 mg, 56% yield, mp 153-155 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, *J* = 8.0 Hz, 1H), 7.53 (d, *J* = 7.6 Hz, 2H), 7.37 (s, 1H), 7.08 (d, *J* = 8.0 Hz, 2H), 6.44 (s, 1H), 5.19 (d, *J* = 10.8 Hz, 1H), 4.12 (d, *J* = 10.8 Hz, 1H), 3.64 (s, 3H), 3.44 (s, 3H), 3.29 (d, *J* = 15.2 Hz, 1H), 2.41 (d, *J* = 15.2 Hz, 1H), 2.14 (s, 3H), 1.71 (s, 3H), 1.47 (s, 3H), 1.38 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.9, 171.1, 169.5, 146.7, 145.0, 139.8, 137.1, 136.8, 135.6, 131.6, 129.6, 124.5, 124.0, 121.9, 118.2, 59.9, 52.6, 52.6, 50.1, 48.6, 31.7, 30.5, 26.4, 22.3, 18.0. IR (KBr, ν, cm^{-1}) 2956, 1727, 1600, 1488, 1328, 1074, 843, 436. HRMS (APCI-TOF) m/z calcd for $C_{29}H_{29}BrO_5Na$, 559.1096 $[M+Na]^+$; found 559.1068.

Diethyl 9a-methyl-9-oxo-4-(p-tolyl)-3-vinyl-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3aa**)



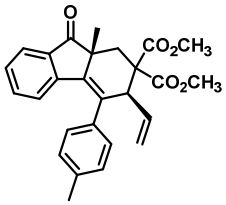
White solid, 108 mg, 59% yield, mp 116-118 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71 (d, *J* = 7.6 Hz, 1H), 7.28 (s, 2H), 7.26-7.18 (m, 4H), 6.83 (d, *J* = 8.0 Hz, 1H), 6.00-5.92 (m, 1H), 5.24-5.19 (m, 2H), 4.21-4.15 (m, 2H), 4.02 (d, *J* = 7.6 Hz, 1H), 3.91-3.86 (m, 2H), 3.36-3.32 (m, 1H), 2.41 (s, 3H), 2.22 (d, *J* = 15.2 Hz, 1H), 1.45 (s, 3H), 1.29-1.22 (m, 3H), 0.89-0.86 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.8, 170.2, 169.2, 146.5, 138.1, 137.8, 137.5, 135.8, 134.0(1), 134.0(2), 133.4, 128.4, 124.1, 124.0, 119.4, 61.9, 61.7, 60.4, 54.0, 50.2, 30.9, 30.9, 21.4, 14.1, 13.5. IR (KBr, ν, cm^{-1}) 2982, 1717, 1596, 1470, 1365, 1075, 860, 467. HRMS (APCI-TOF) m/z calcd for $C_{29}H_{30}O_5Na$, 481.1991 $[M+Na]^+$; found 481.1999.

Diethyl 4-(4-bromophenyl)-9a-methyl-9-oxo-3-vinyl-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3bb**)



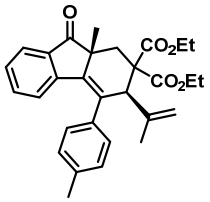
White solid, 116 mg, 56% yield, mp 106-108 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (d, *J* = 7.6 Hz, 1H), 7.55 (d, *J* = 8.0 Hz, 2H), 7.29 (d, *J* = 7.2 Hz, 3H), 7.24 (s, 1H), 6.77 (d, *J* = 7.6 Hz, 1H), 5.98-5.89 (m, 1H), 5.22 (d, *J* = 10.4 Hz, 2H), 4.21-4.16 (m, 2H), 3.95-3.87 (m, 3H), 3.36-3.32 (m, 1H), 2.25 (d, *J* = 15.2 Hz, 1H), 1.45 (s, 3H), 1.23 (d, *J* = 7.2 Hz, 3H), 0.92-0.89 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.2, 170.2, 169.0, 146.0, 139.5, 138.6, 134.4, 134.2, 134.1, 133.1, 128.7, 124.2, 124.1, 122.2, 119.6, 62.0, 61.8, 60.4, 53.8, 50.3, 31.0, 30.8, 14.1, 13.6. IR (KBr, ν, cm^{-1}) 2983, 1731, 1596, 1470, 1300, 1074, 844, 466. HRMS (APCI-TOF) m/z calcd for $C_{28}H_{27}BrO_5Na$, 545.0940 $[M+Na]^+$; found 545.0946.

Dimethyl 9a-methyl-9-oxo-4-(p-tolyl)-3-vinyl-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3cc**)



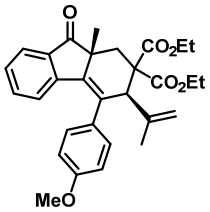
White solid, 94 mg, 55% yield, mp 116-118 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (d, *J* = 7.6 Hz, 1H), 7.28 (d, *J* = 7.2 Hz, 1H), 7.25-7.20 (m, 5H), 6.83 (d, *J* = 7.6 Hz, 1H), 5.98-5.90 (m, 1H), 5.24-5.20 (m, 2H), 4.02 (d, *J* = 7.2 Hz, 1H), 3.72 (s, 3H), 3.40 (s, 3H), 3.35-3.30 (m, 1H), 2.41 (s, 3H), 2.24 (d, *J* = 14.8 Hz, 1H), 1.45 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.6, 170.6, 169.6, 146.4, 138.2, 137.9, 137.4, 135.4, 134.0, 133.9, 133.4, 128.4, 124.2, 124.1, 119.6, 60.4, 54.1, 52.8, 52.6, 50.3, 31.0, 30.8, 21.4. IR (KBr, ν, cm^{-1}) 2974, 1737, 1601, 1444, 1301, 1071, 849, 465. HRMS (APCI-TOF) m/z calcd for $C_{27}H_{26}O_5Na$, 453.1678 $[M+Na]^+$; found 453.1678.

Diethyl 9a-methyl-9-oxo-3-(prop-1-en-2-yl)-4-(p-tolyl)-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3dd**)



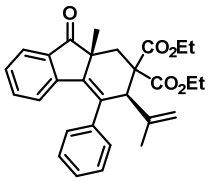
White solid, 122 mg, 65% yield, mp 112-114 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (d, *J* = 7.2 Hz, 1H), 7.37 (s, 2H), 7.23-7.17 (m, 4H), 6.78 (d, *J* = 7.6 Hz, 1H), 5.05 (s, 2H), 4.25-4.09 (m, 2H), 4.06 (s, 1H), 3.95-3.80 (m, 2H), 3.34-3.30 (m, 1H), 2.38 (d, *J* = 17.6 Hz, 4H), 1.72 (s, 3H), 1.45 (s, 3H), 1.27-1.23 (m, 3H), 0.97-0.94 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.4, 170.4, 169.8, 146.8, 140.8, 137.9, 137.8, 137.5, 137.3, 134.1, 134.0, 128.2, 123.9, 123.8, 118.1, 61.9, 61.7, 59.7, 56.9, 50.2, 30.8, 30.4, 23.5, 21.4, 14.0, 13.6. IR (KBr, ν, cm^{-1}) 2989, 1734, 1597, 1466, 1295, 1097, 859, 467. HRMS (APCI-TOF) m/z calcd for $C_{30}H_{32}O_5Na$, 495.2147 $[M+Na]^+$; found 495.2195.

Diethyl 4-(4-methoxyphenyl)-9a-methyl-9-oxo-3-(prop-1-en-2-yl)-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3ee**)



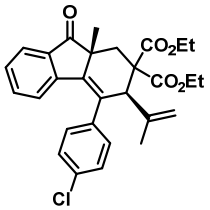
White solid, 124 mg, 64% yield, mp 142-144 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69-7.65 (m, 1H), 7.52-7.27 (m, 2H), 7.25-7.17 (m, 2H), 6.95 (d, *J* = 8.8 Hz, 2H), 6.79 (d, *J* = 7.2 Hz, 1H), 5.05 (d, *J* = 4.8 Hz, 2H), 4.25-4.21 (m, 1H), 4.13-4.09 (m, 1H), 4.04 (s, 1H), 3.90 (d, *J* = 7.2 Hz, 1H), 3.85 (s, 3H), 3.82 (d, *J* = 7.2 Hz, 1H), 3.33-3.29 (m, 1H), 2.37 (d, *J* = 15.2 Hz, 1H), 1.72 (s, 3H), 1.44 (s, 3H), 1.27-1.23 (m, 3H), 0.96-0.93 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.4, 170.4, 169.8, 159.4, 146.9, 140.9, 137.6, 137.1, 134.1, 134.0, 133.1, 128.2, 123.9, 123.8, 118.0, 61.9, 61.7, 59.7, 57.0, 55.3, 50.2, 30.8, 30.4, 23.55, 14.0, 13.6. IR (KBr, ν, cm^{-1}) 2975, 1731, 1607, 1464, 1301, 1082, 848, 467. HRMS (APCI-TOF) m/z calcd for $C_{30}H_{32}O_6Na$, 511.2097 $[M+Na]^+$; found511.2103.

Diethyl 9a-methyl-9-oxo-4-phenyl-3-(prop-1-en-2-yl)-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3ff**)



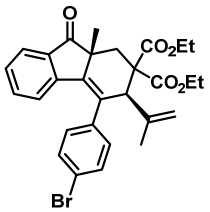
White solid, 113 mg, 62% yield, mp 118-120 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (d, *J* = 7.2 Hz, 1H), 7.40 (d, *J* = 6.8 Hz, 5H), 7.25-7.17 (m, 1H), 7.17 (d, *J* = 7.6 Hz, 1H), 6.68 (d, *J* = 7.6 Hz, 1H), 5.07 (s, 2H), 4.28-4.19 (m, 1H), 4.15-4.08 (m, 1H), 4.06 (s, 1H), 3.93-3.85 (m, 2H), 3.35-3.30 (m, 1H), 2.40 (d, *J* = 15.2 Hz, 1H), 1.74 (s, 3H), 1.46 (s, 3H), 1.27-1.23 (m, 3H), 1.00-0.96 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.2, 170.4, 169.7, 146.7, 141.0, 140.7, 137.7, 137.1, 134.1, 134.0, 128.2, 128.0, 123.9, 123.9, 118.2, 61.9, 61.7, 59.7, 56.8, 50.2, 30.7, 30.4, 23.5, 14.0, 13.6. IR (KBr, ν, cm^{-1}) 2991, 1730, 1600, 1466, 1288, 1084, 861, 465. HRMS (APCI-TOF) m/z calcd for $C_{29}H_{30}O_5Na$, 481.1991 $[M+Na]^+$; found 481.1992.

Diethyl 4-(4-chlorophenyl)-9a-methyl-9-oxo-3-(prop-1-en-2-yl)-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3gg**)



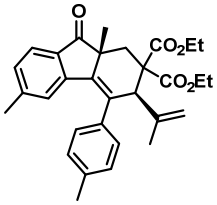
White solid, 118 mg, 60% yield, mp 124-126 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, *J* = 7.6 Hz, 1H), 7.53-7.41 (m, 1H), 7.39 (d, *J* = 8.0 Hz, 2H), 7.27 (s, 1H), 7.25-7.23 (m, 2H), 6.71 (d, *J* = 7.6 Hz, 1H), 5.06 (d, *J* = 18.0 Hz, 2H), 4.26- 4.22 (m, 2H), 3.98-3.82 (m, 3H), 3.34-3.29 (m, 1H), 2.40 (d, *J* = 15.2 Hz, 1H), 1.74 (s, 3H), 1.45 (s, 3H), 1.27-1.23 (m, 3H), 1.01-0.98 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.8, 170.5, 169.6, 146.4, 140.6, 139.5, 138.1, 135.7, 134.2, 134.1, 133.9, 128.5, 124.0, 123.9, 118.4, 62.0, 61.8, 59.6, 56.7, 50.3, 30.6, 30.4, 23.4, 14.0, 13.6. IR (KBr, ν, cm^{-1}) 2964, 1733, 1602, 1489, 1295, 1087, 846, 474. HRMS (APCI-TOF) m/z calcd for $C_{29}H_{29}ClO_5Na$, 515.1601 $[M+Na]^+$; found 515.1614.

Diethyl 4-(4-bromophenyl)-9a-methyl-9-oxo-3-(prop-1-en-2-yl)-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3hh**)



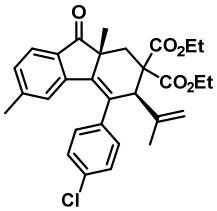
White solid, 130 mg, 61% yield, mp 152-154 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 6.8 Hz, 1H), 7.54 (d, *J* = 8.8 Hz, 2H), 7.28 (s, 3H), 7.24 (s, 1H), 6.71 (d, *J* = 7.6 Hz, 1H), 5.06 (d, *J* = 19.6 Hz, 2H), 4.23 (d, *J* = 7.2 Hz, 1H), 4.12 (d, *J* = 7.2 Hz, 1H), 3.96-3.82 (m, 3H), 3.34-3.29 (m, 1H), 2.42 (s, 1H), 1.74 (s, 3H), 1.45 (s, 3H), 1.27-1.24 (m, 3H), 1.02- 0.98 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.8, 170.5, 169.6, 146.4, 140.6, 140.0, 138.1, 135.7, 134.2, 134.1, 128.5, 124.0, 123.9, 122.1, 118.4, 62.0, 61.8, 59.6, 56.6, 50.3, 30.6, 30.4, 23.4, 14.0, 13.6. IR (KBr, ν, cm^{-1}) 2981, 1732, 1601, 1468, 1295, 1083, 844, 465. HRMS (APCI-TOF) m/z calcd for $C_{29}H_{29}BrO_5Na$, 559.1096 $[M+Na]^+$; found 559.1102.

Diethyl 6,9a-dimethyl-9-oxo-3-(prop-1-en-2-yl)-4-(p-tolyl)-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3ii**)



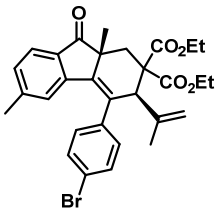
White solid, 124 mg, 64% yield, mp 140-142 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 (d, *J* = 7.6 Hz, 1H), 7.37 (s, 2H), 7.22 (d, *J* = 8.0 Hz, 2H), 7.05 (d, *J* = 7.6 Hz, 1H), 6.55 (s, 1H), 5.06 (s, 2H), 4.25-4.21 (m, 2H), 4.05 (s, 1H), 3.95-3.80 (m, 2H), 3.32-3.28 (m, 1H), 2.42-2.34 (m, 4H), 2.11 (s, 3H), 1.73 (s, 3H), 1.44 (s, 3H), 1.27-1.23 (m, 3H), 0.99-0.96 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.0, 170.4, 169.8, 147.2, 144.8, 140.9, 138.0, 137.7, 137.6, 137.0, 131.9, 129.3, 124.5, 123.7, 118.1, 61.9, 61.7, 59.7, 56.9, 50.3, 30.8, 30.6, 23.5, 22.2, 21.4, 14.0, 13.6. IR (KBr, ν, cm^{-1}) 2992, 1732, 1601, 1509, 1284, 1060, 836, 441. HRMS (APCI-TOF) m/z calcd for: $C_{31}H_{34}O_5Na$, 509.2304 $[M+Na]^+$; found 509.2305.

Diethyl 4-(4-chlorophenyl)-6,9a-dimethyl-9-oxo-3-(prop-1-en-2-yl)-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3jj**)



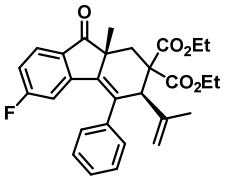
White solid, 121 mg, 60% yield, mp 160-162 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (d, *J* = 8.0 Hz, 1H), 7.47-7.38 (m, 4H), 7.08 (d, *J* = 8.0 Hz, 1H), 6.49 (s, 1H), 5.06 (d, *J* = 18.0 Hz, 2H), 4.26-4.08 (m, 2H), 3.97-3.83 (m, 3H), 3.32-3.28 (m, 1H), 2.37 (d, *J* = 15.2 Hz, 1H), 2.14 (s, 3H), 1.73 (s, 3H), 1.43 (s, 3H), 1.27-1.23 (m, 3H), 1.02-0.99 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.4, 170.5, 169.6, 146.8, 145.0, 140.6, 139.6, 138.2, 135.4, 133.8, 132.0, 129.6, 124.44, 123.9, 118.4, 61.9, 61.7, 59.6, 56.7, 50.4, 30.6, 30.5, 23.4, 22.3, 14.0, 13.6. IR (KBr, ν, cm^{-1}) 2993, 1732, 1601, 1489, 1284, 1089, 839, 462. HRMS (APCI-TOF) m/z calcd for $C_{30}H_{31}ClO_5Na$, 529.1758 $[M+Na]^+$; found 529.1730.

Diethyl 4-(4-bromophenyl)-6,9a-dimethyl-9-oxo-3-(prop-1-en-2-yl)-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3kk**)



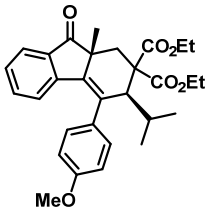
White solid, 123 mg, 56% yield, mp 147-149 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58-7.53 (m, 3H), 7.39 (d, *J* = 8.0 Hz, 2H), 7.08 (d, *J* = 7.2 Hz, 1H), 6.49 (s, 1H), 5.05 (d, *J* = 18.8 Hz, 2H), 4.22 (d, *J* = 7.2 Hz, 1H), 4.11 (d, *J* = 7.2 Hz, 1H), 3.96-3.91 (m, 2H), 3.85 (d, *J* = 7.2 Hz, 1H), 3.31-3.27 (m, 1H), 2.37 (d, *J* = 15.2 Hz, 1H), 2.15 (s, 3H), 1.73 (s, 3H), 1.43 (s, 3H), 1.26-1.23 (m, 3H), 1.02-0.98 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.3, 170.5, 169.6, 146.8, 145.0, 140.6, 140.1, 138.2, 135.3, 132.0, 129.6, 124.4, 123.9, 122.0, 118.4, 61.9, 61.7, 59.6, 56.6, 50.5, 30.6, 30.5, 23.4, 22.3, 14.0, 13.6. IR (KBr, ν, cm^{-1}) 2992, 1732, 1601, 1507, 1254, 1088, 839, 441. HRMS (APCI-TOF) m/z calcd for $C_{30}H_{31}BrO_5Na$, 573.1253 $[M+Na]^+$; found 573.1230.

Diethyl 6-fluoro-9a-methyl-9-oxo-4-phenyl-3-(prop-1-en-2-yl)-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3ll**)



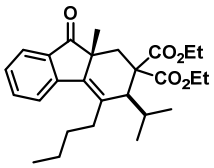
White solid, 103 mg, 54% yield, mp 153-154 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 – 7.65 (m, 1H), 7.58 – 7.32 (m, 5H), 6.95 – 6.90 (m, 1H), 6.32 – 6.25 (m, 1H), 5.09 – 5.03 (m, 2H), 4.28 – 4.20 (m, 1H), 4.15 – 4.07 (m, 2H), 3.98 – 3.85 (m, 2H), 3.34 – 3.28 (m, 1H), 2.41 (d, *J* = 15.2 Hz, 1H), 1.73 (s, 3H), 1.46 (s, 3H), 1.28 – 1.24 (m, 3H), 1.03 – 0.98 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.4, 170.4, 169.6, 167.6($^1J_{CF}$ = 252.4 Hz), 165.0, 149.4, 149.2, 140.5, 140.3, 138.8, 136.9($^2J_{CF}$ = 2.9 Hz), 136.8, 130.5, 130.4, 128.4, 126.1($^3J_{CF}$ =10.4 Hz), 126.0, 118.3, 116.1($^4J_{CF}$ = 23.9 Hz), 115.9, 111.0, 110.7, 61.9, 61.7, 59.5, 56.7, 50.5, 30.8, 30.4, 23.5, 14.0, 13.6. IR (KBr, ν, cm^{-1}) 2979, 1731, 1490, 1442, 1306, 1081, 854, 458. HRMS (ESI) m/z calcd for $C_{29}H_{29}FO_5Na$, 499.1897 $[M+Na]^+$; found 499.1898.

Diethyl 3-isopropyl-4-(4-methoxyphenyl)-9a-methyl-9-oxo-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3mm**)

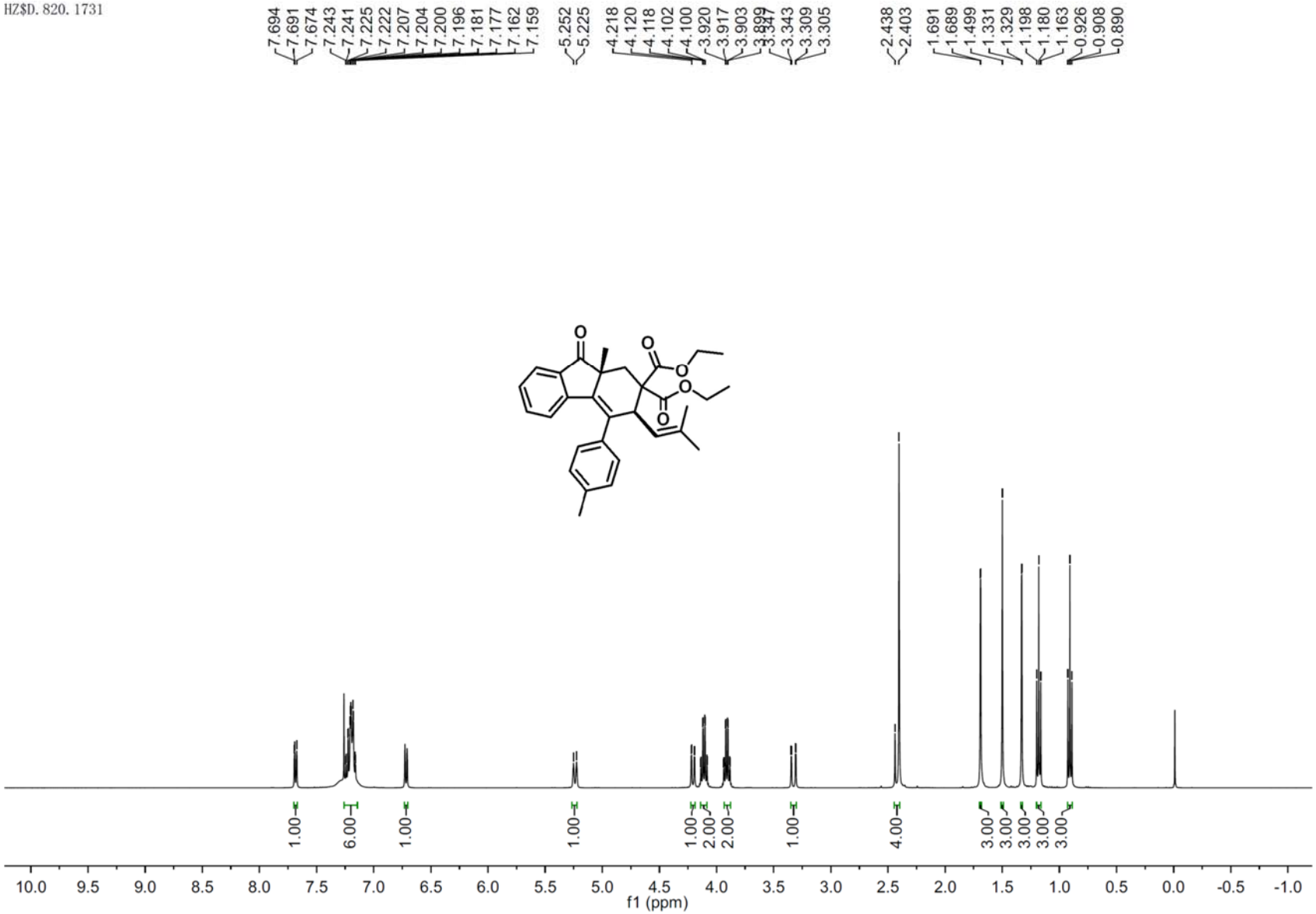


White solid, 115 mg, 59% yield, mp 115-117 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70-7.55 (m, 3H), 7.25-7.16 (m, 2H), 7.01-0.96 (m, 3H), 4.26-4.13 (m, 2H), 3.88 (s, 3H), 3.81-3.77 (m, 1H), 3.66-3.62 (m, 1H), 3.41-3.32 (m, 2H), 2.38 (d, *J* = 14.8 Hz, 1H), 2.08 (d, *J* = 6.8 Hz, 1H), 1.48 (s, 3H), 1.29-1.26 (m, 3H), 0.92 (d, *J* = 6.8 Hz, 3H), 0.84 (d, *J* = 6.4 Hz, 3H), 0.80-0.76 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.4, 170.6, 170.1, 159.5, 146.7, 140.3, 137.5, 134.6, 134.2, 133.8, 128.2, 123.9, 123.5, 61.8, 61.7, 60.9, 56.5, 55.3, 49.5, 31.1, 30.4, 29.4, 24.3, 23.0, 14.0, 13.4. IR (KBr, ν, cm^{-1}) 2972, 1731, 1606, 1465, 1299, 1098, 849, 459. HRMS (APCI-TOF) m/z calcd for $C_{30}H_{34}O_6Na$, 513.2253 $[M+Na]^+$; found 513.2238.

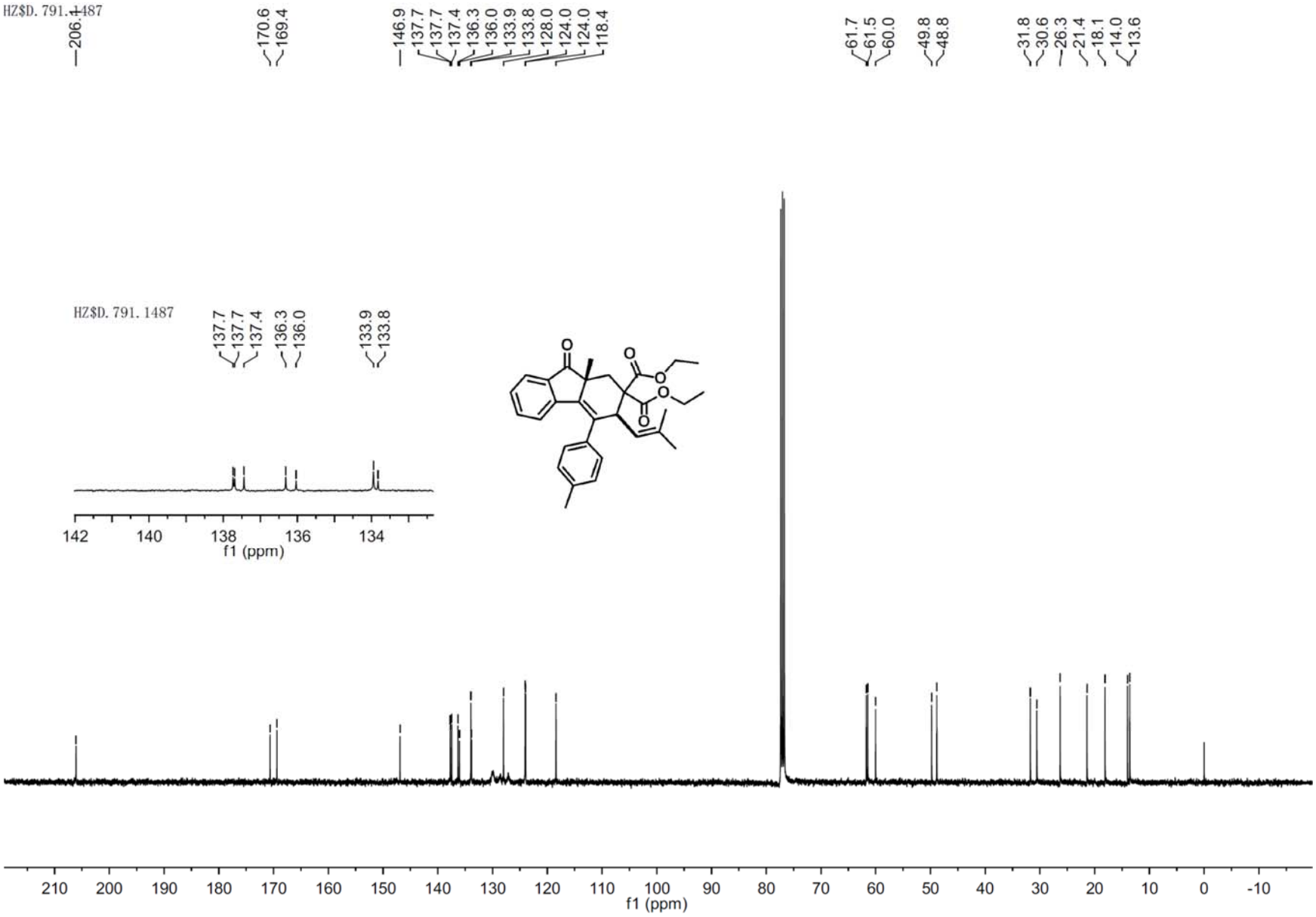
Diethyl 4-butyl-3-isopropyl-9a-methyl-9-oxo-1,3,9,9a-tetrahydro-2H-fluorene-2,2-dicarboxylate (*rac*-**3nn**)



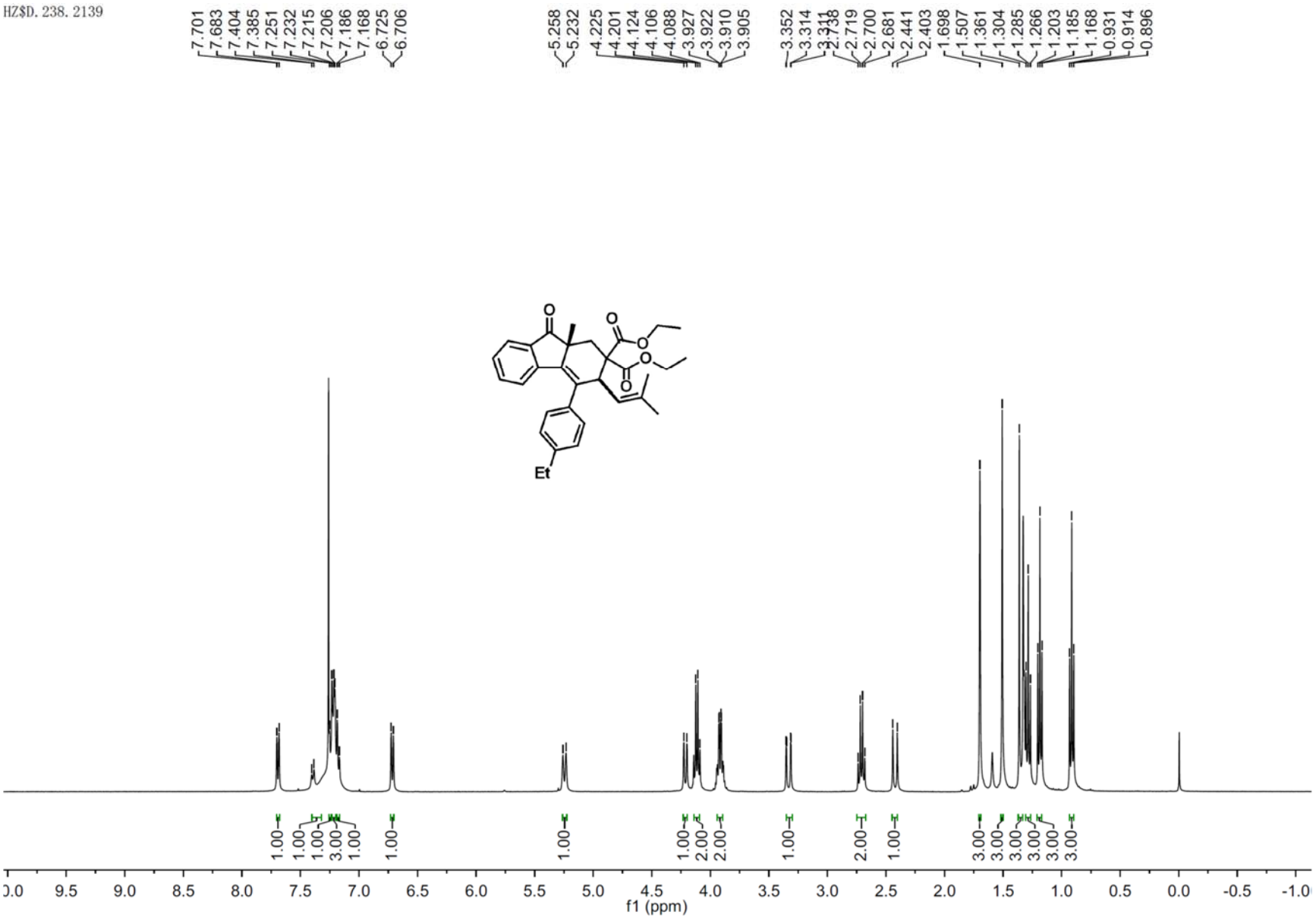
Yellow oil, 91 mg, 52% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 7.6 Hz, 1H), 7.63 – 7.54 (m, 2H), 7.34 – 7.30 (m, 1H), 4.25 – 4.10 (m, 2H), 3.78 – 3.68 (m, 2H), 3.29 – 3.18 (m, 1H), 3.01 – 2.95 (m, 1H), 2.76 – 2.64 (m, 1H), 2.28 – 2.17 (m, 2H), 2.06 – 1.97 (m, 1H), 1.59 – 1.47 (m, 1H), 1.43 – 1.33 (m, 3H), 1.30 – 1.24 (m, 6H), 1.07 (d, *J* = 6.8 Hz, 3H), 1.00 (d, *J* = 6.8 Hz, 3H), 0.93 (d, *J* = 7.2 Hz, 3H), 0.88 – 0.84 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.0, 170.2, 170.1, 147.3, 139.5, 135.4, 134.3, 134.1, 127.5, 125.4, 124.3, 61.6, 60.8, 54.3, 50.4, 36.7, 30.3, 30.1, 29.1, 28.0, 23.8, 23.5, 22.7, 13.9(1), 13.9(2), 13.5. IR (KBr, ν, cm^{-1}) 2960, 1732, 1601, 1466, 1366, 1197, 1094, 769. HRMS (ESI) m/z calcd for $C_{27}H_{36}FO_5Na$, 463.2460 $[M+Na]^+$; found 463.2465.



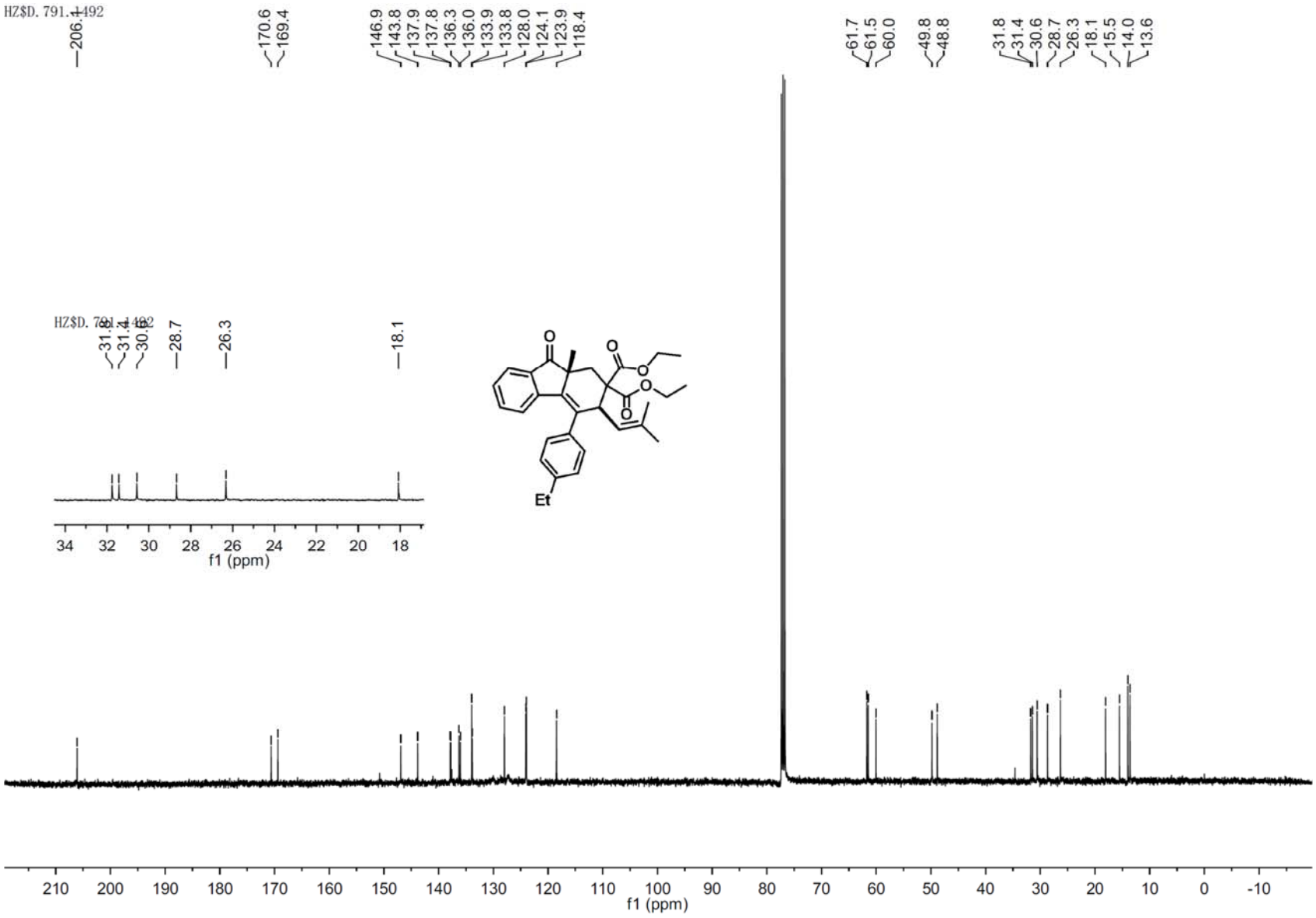
^{1}H NMR Spectrum of Compound 3a



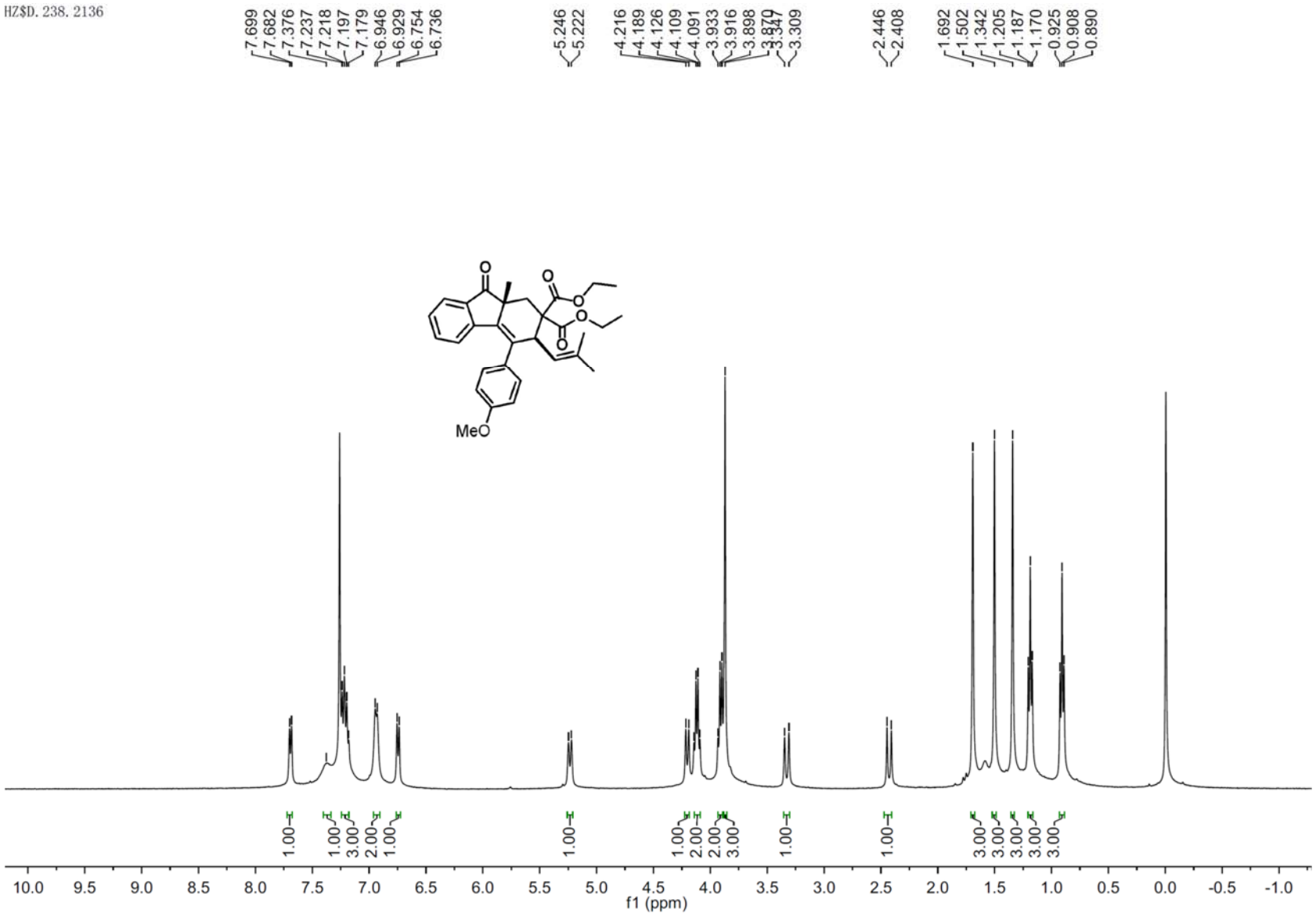
^{13}C NMR Spectrum of Compound 3a



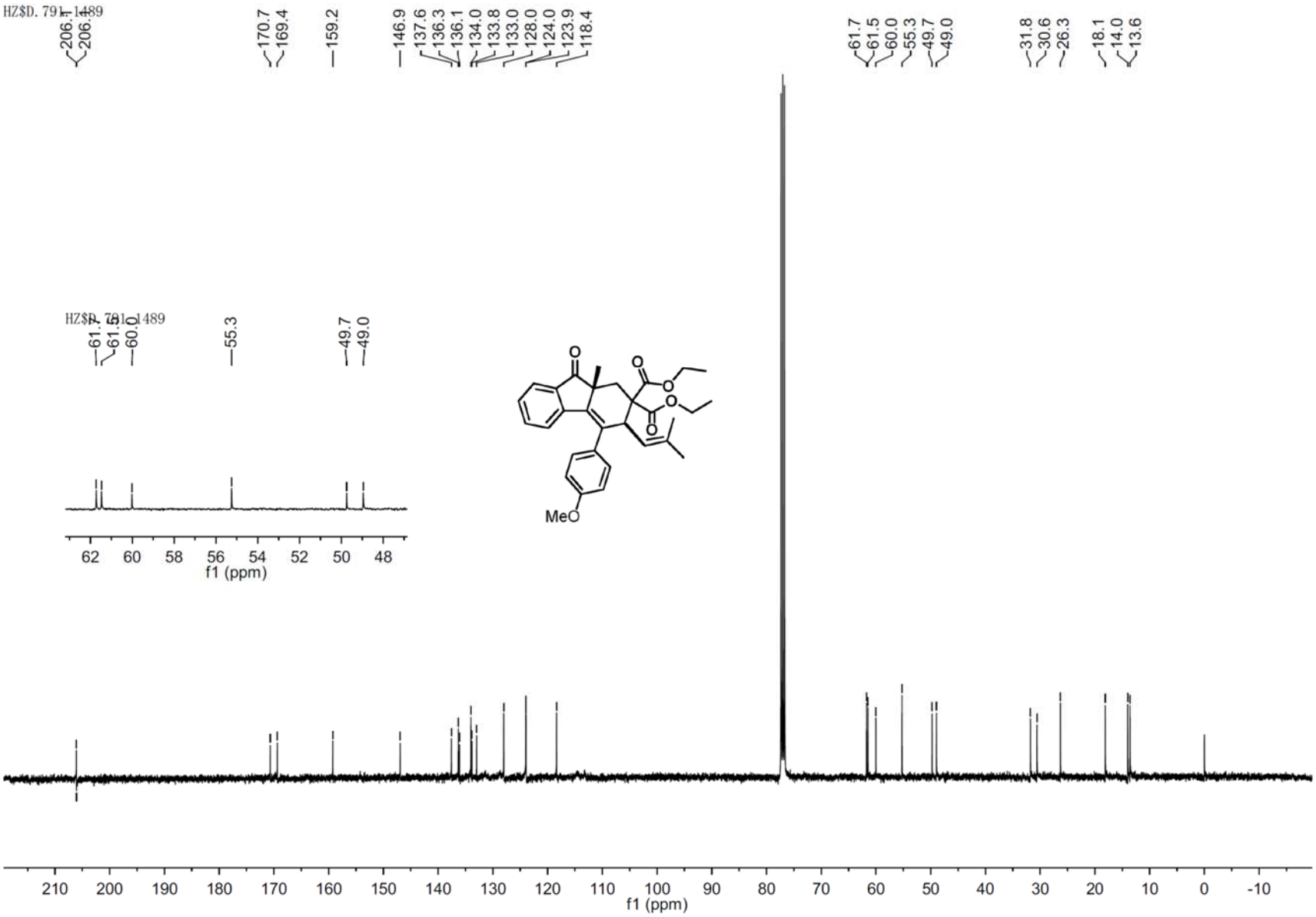
^{1}H NMR Spectrum of Compound 3b



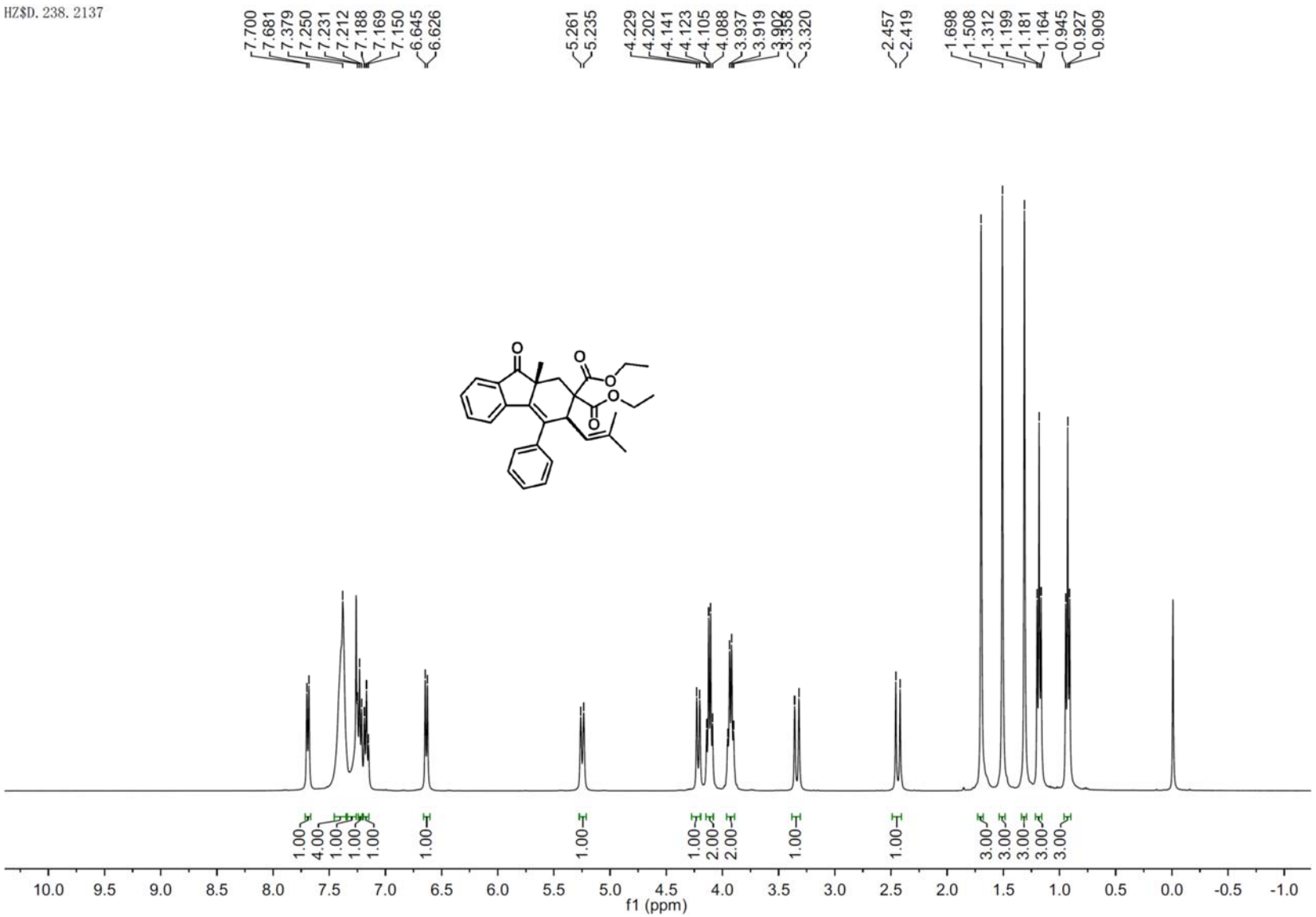
^{13}C NMR Spectrum of Compound 3b



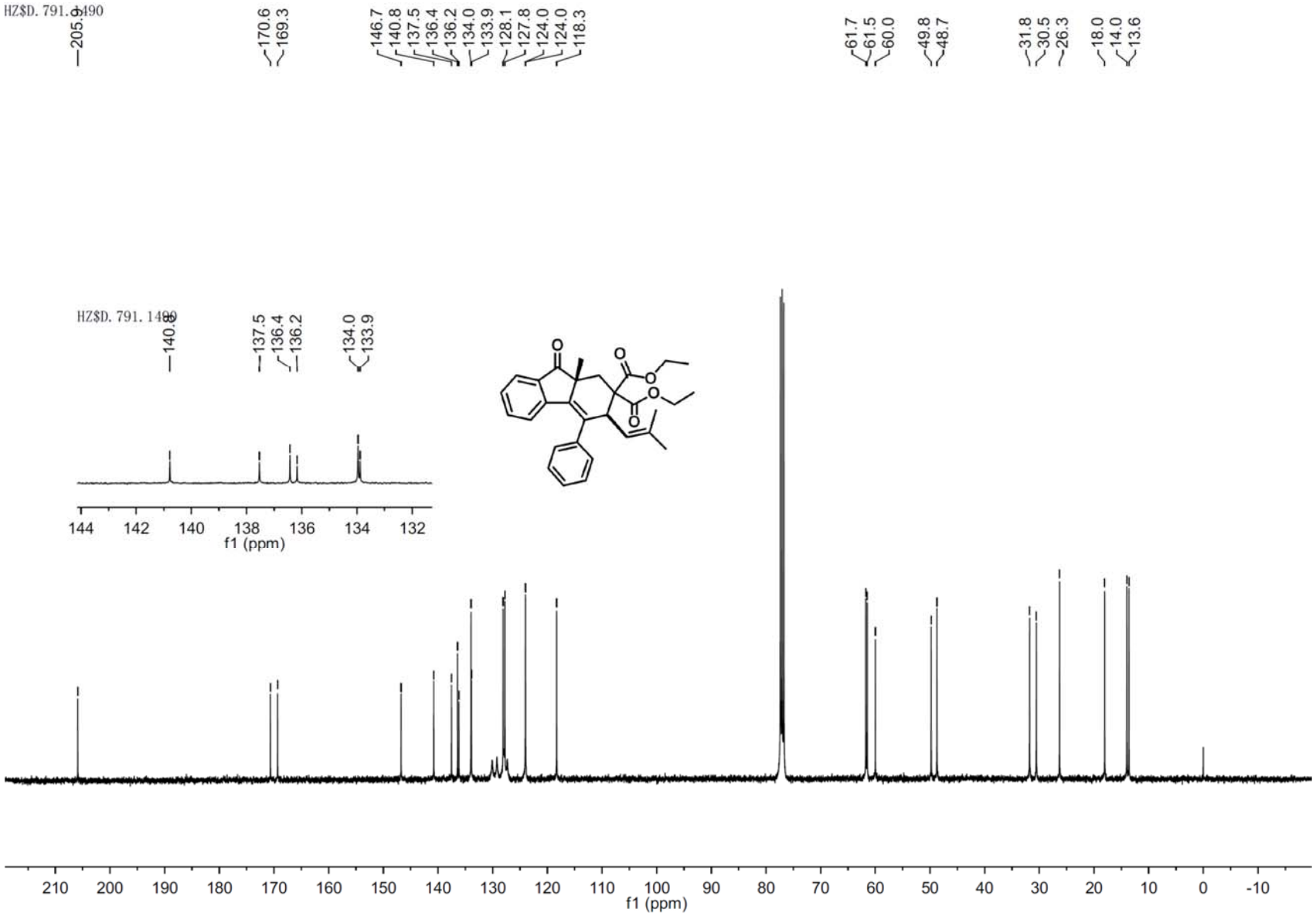
^{1}H NMR Spectrum of Compound 3c



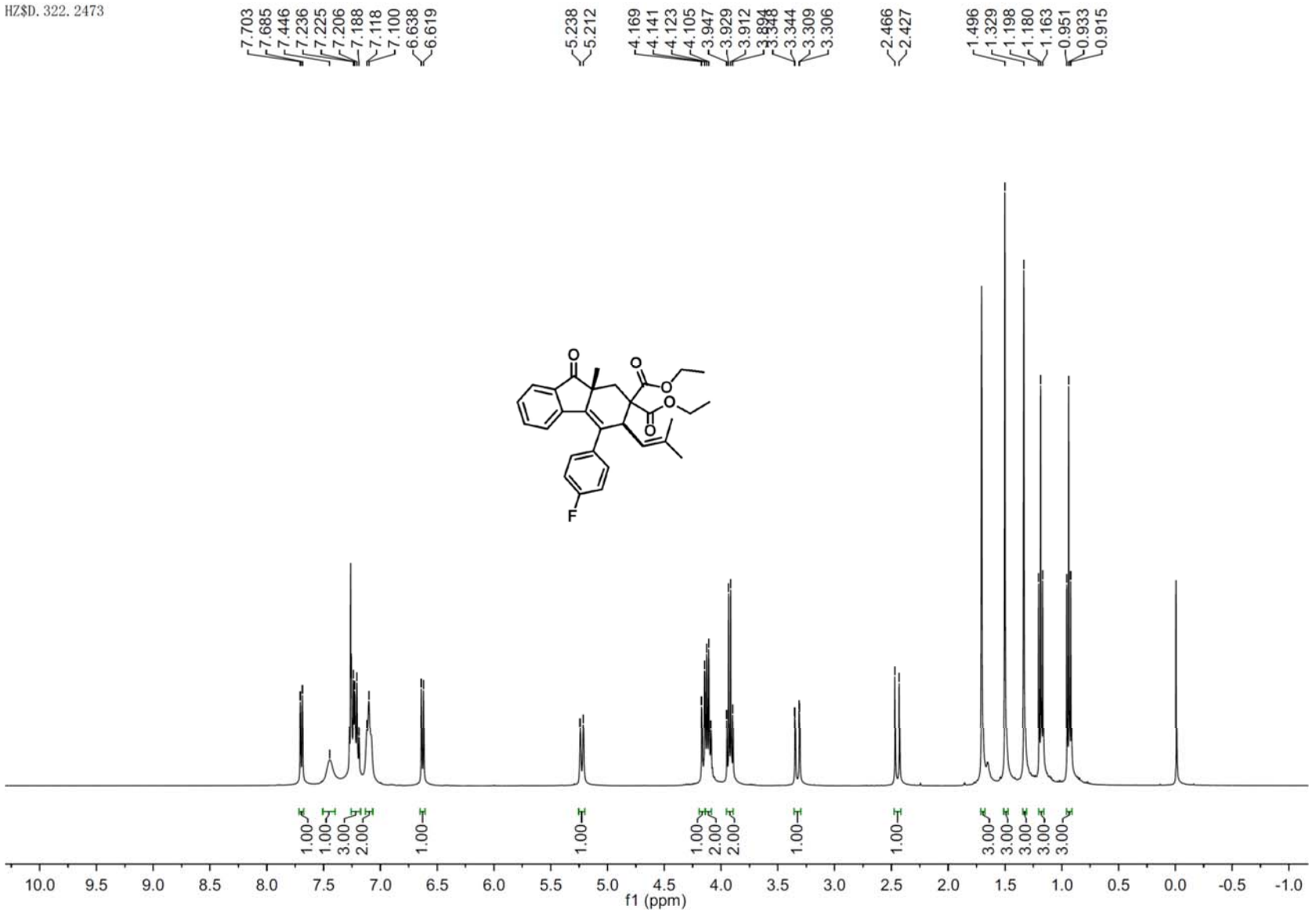
^{13}C NMR Spectrum of Compound 3c



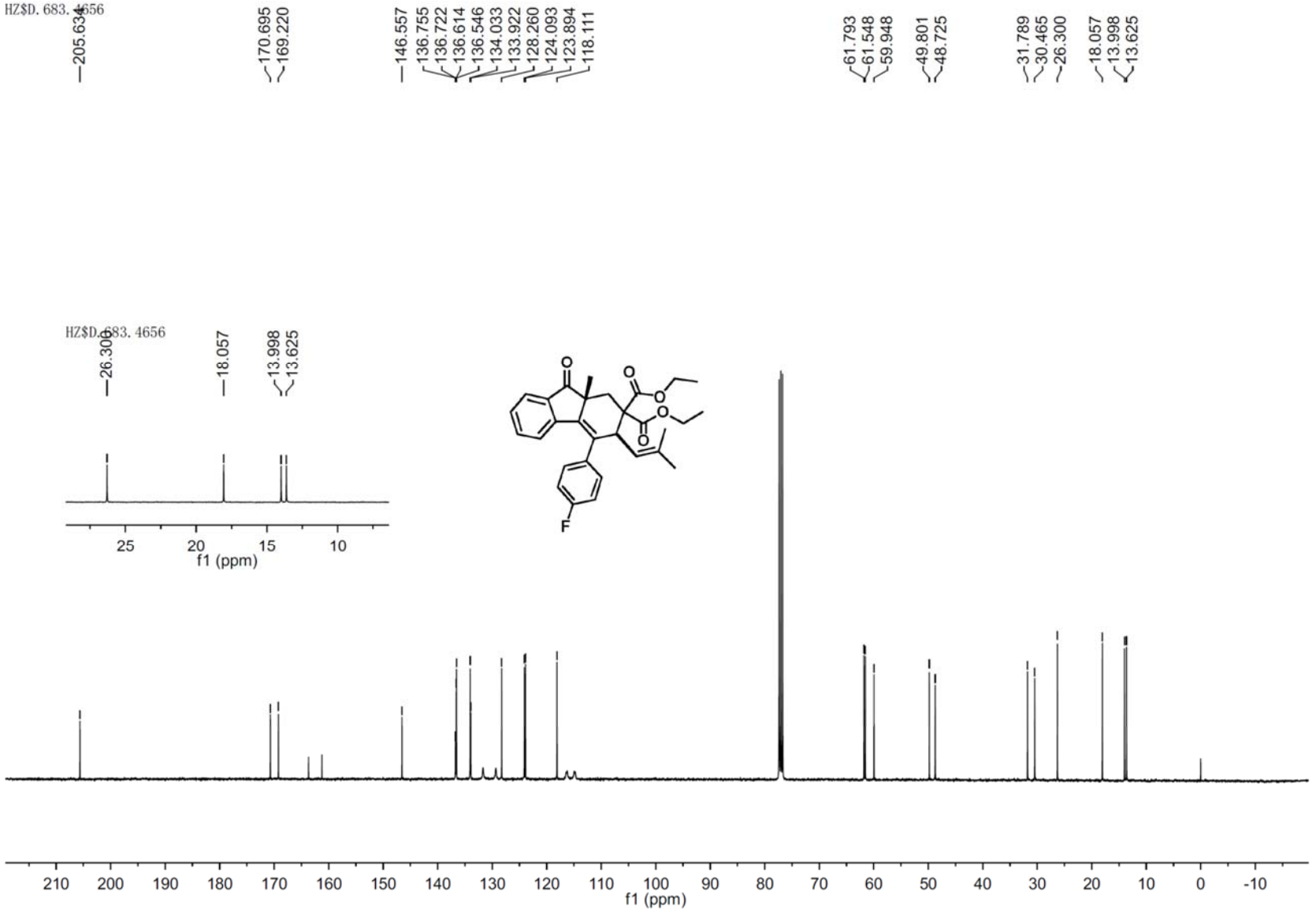
^{1}H NMR Spectrum of Compound 3d



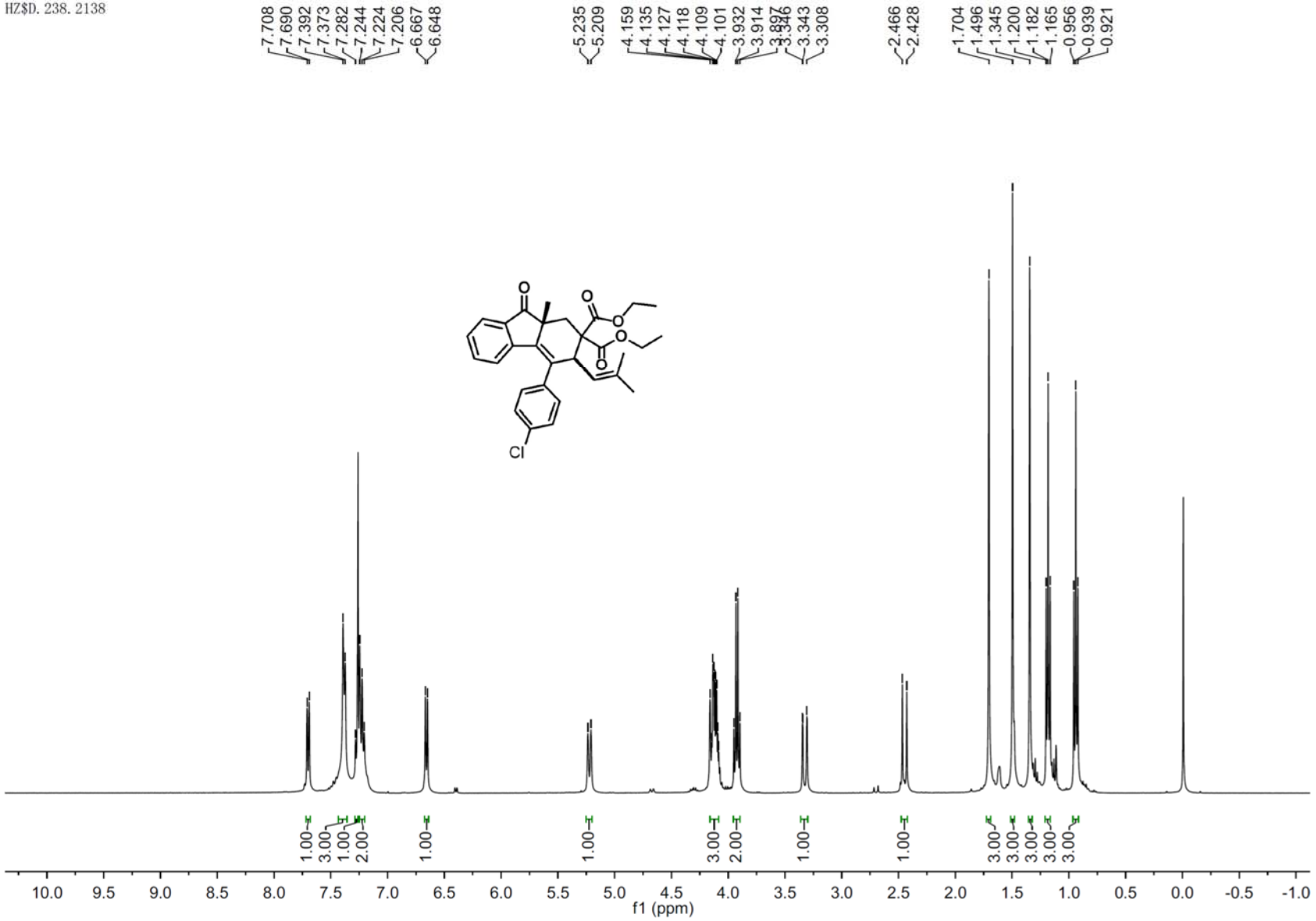
^{13}C NMR Spectrum of Compound 3d



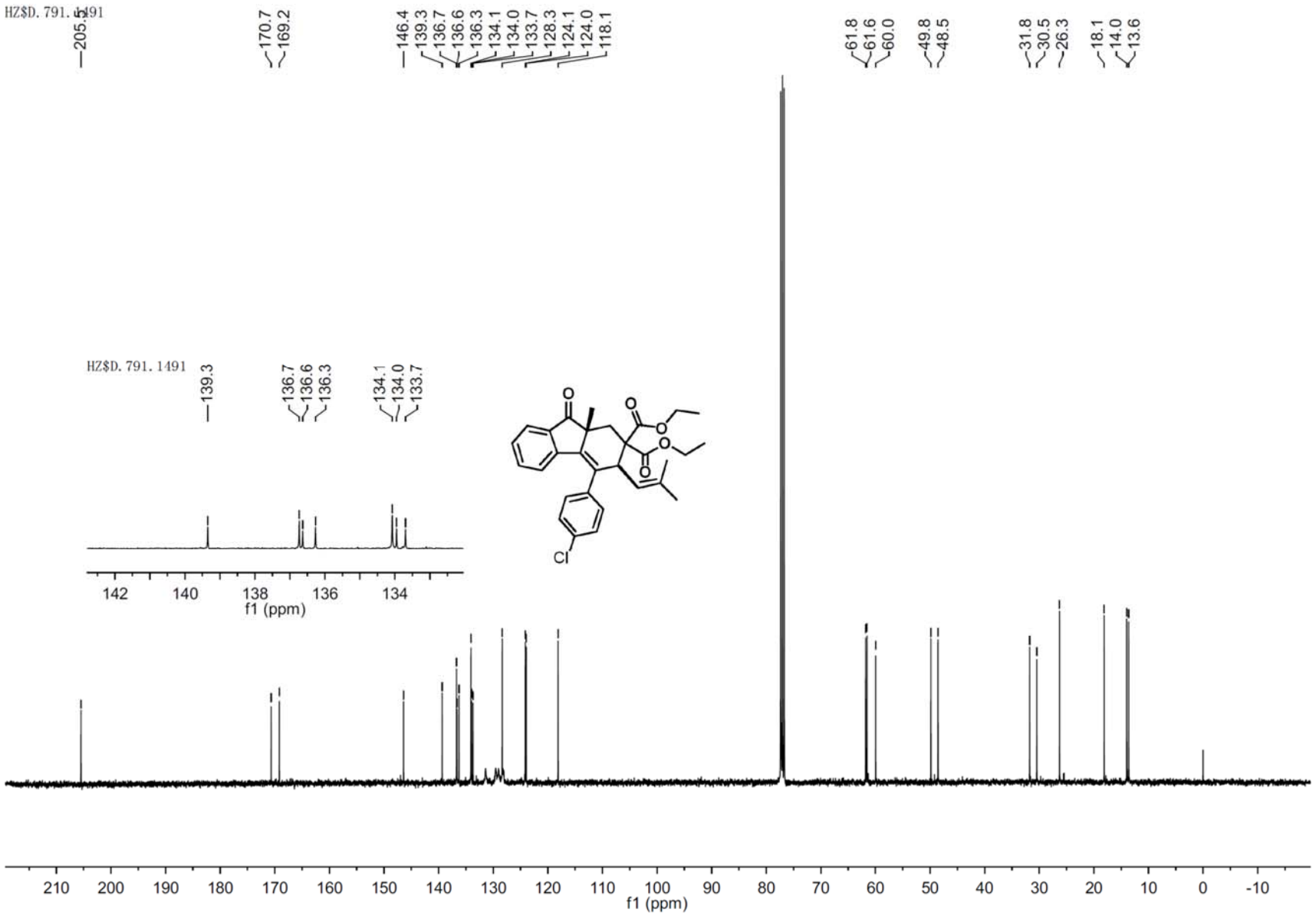
^{1}H NMR Spectrum of Compound 3e



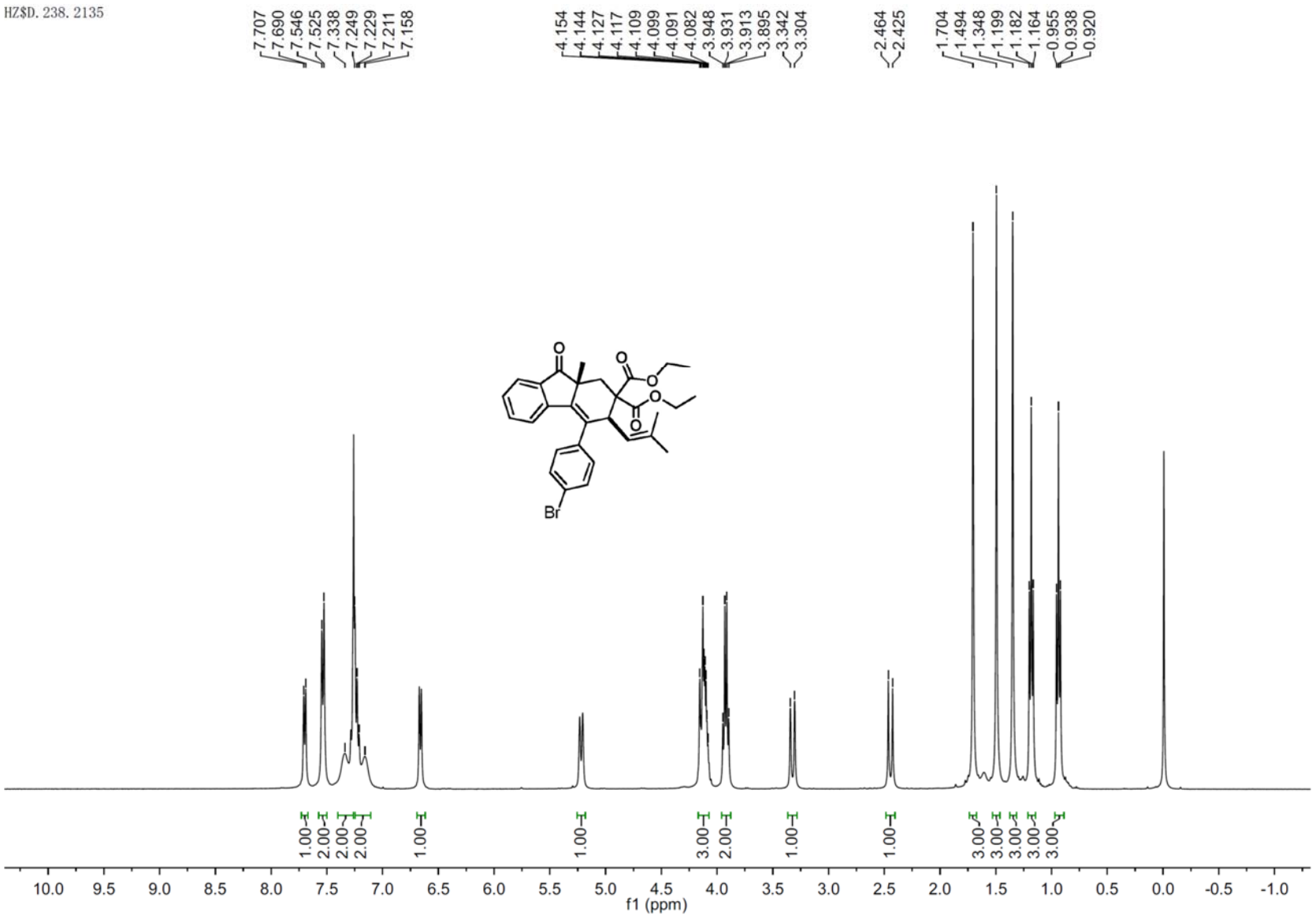
^{13}C NMR Spectrum of Compound 3e



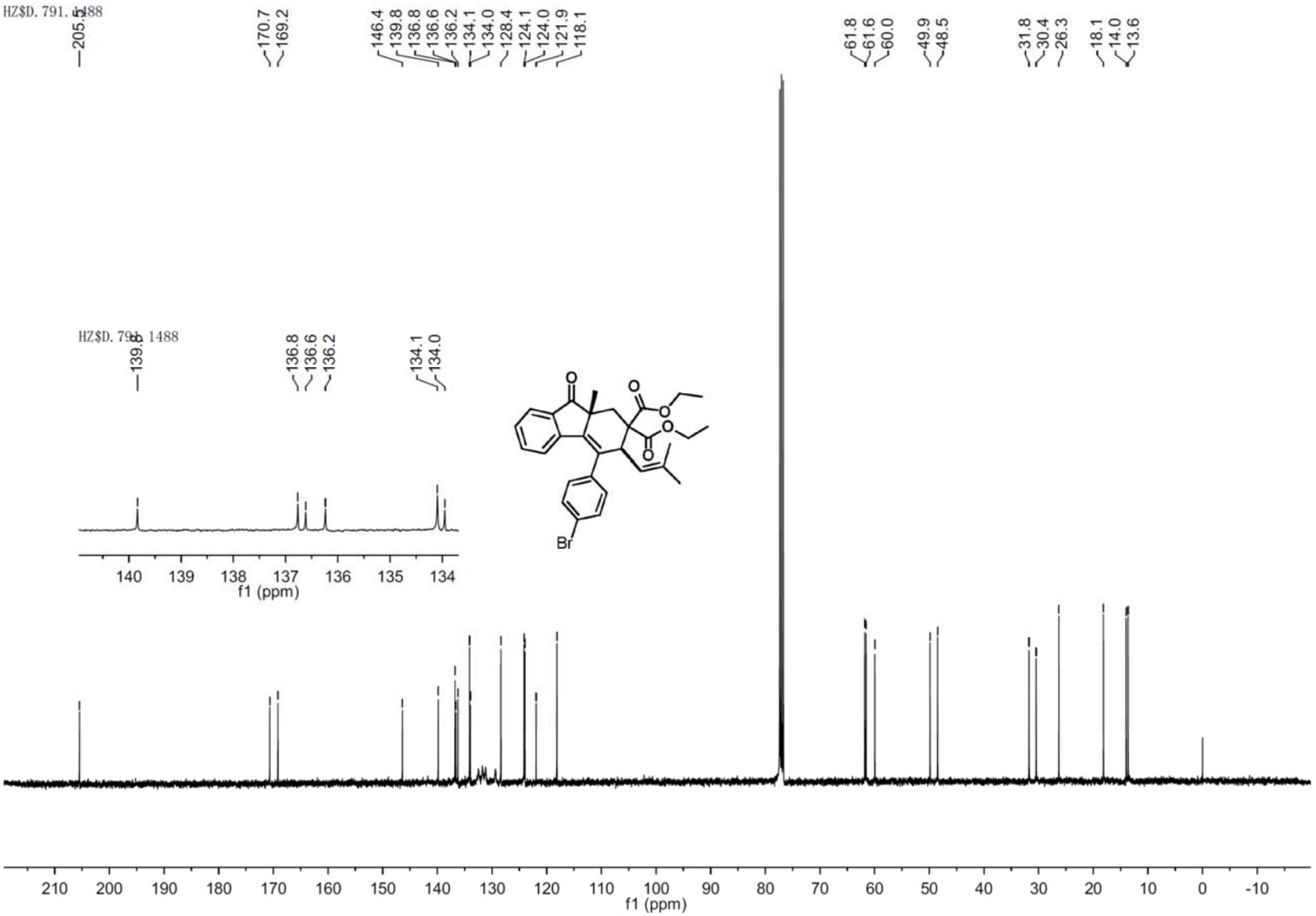
^{1}H NMR Spectrum of Compound 3f



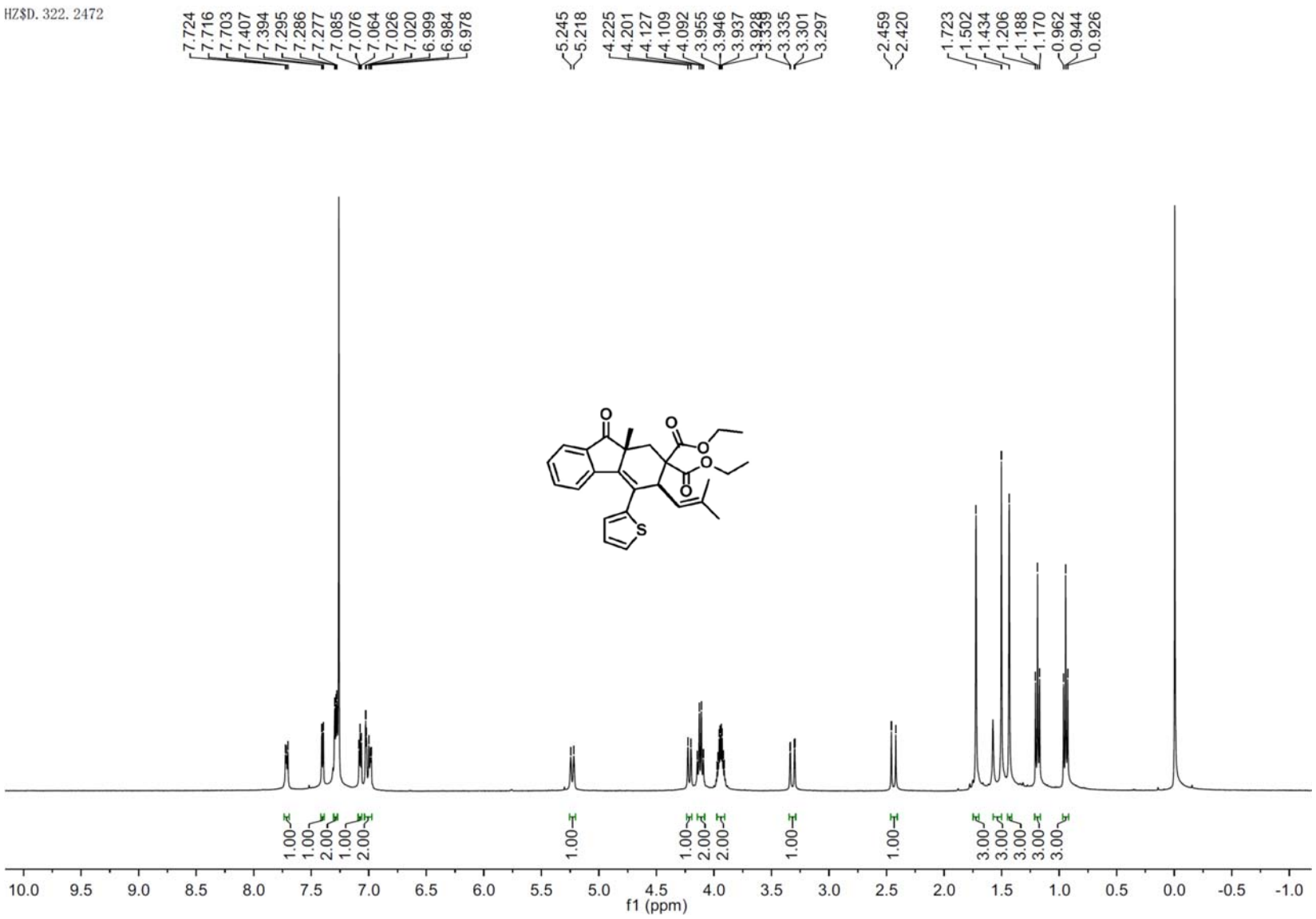
^{13}C NMR Spectrum of Compound 3f



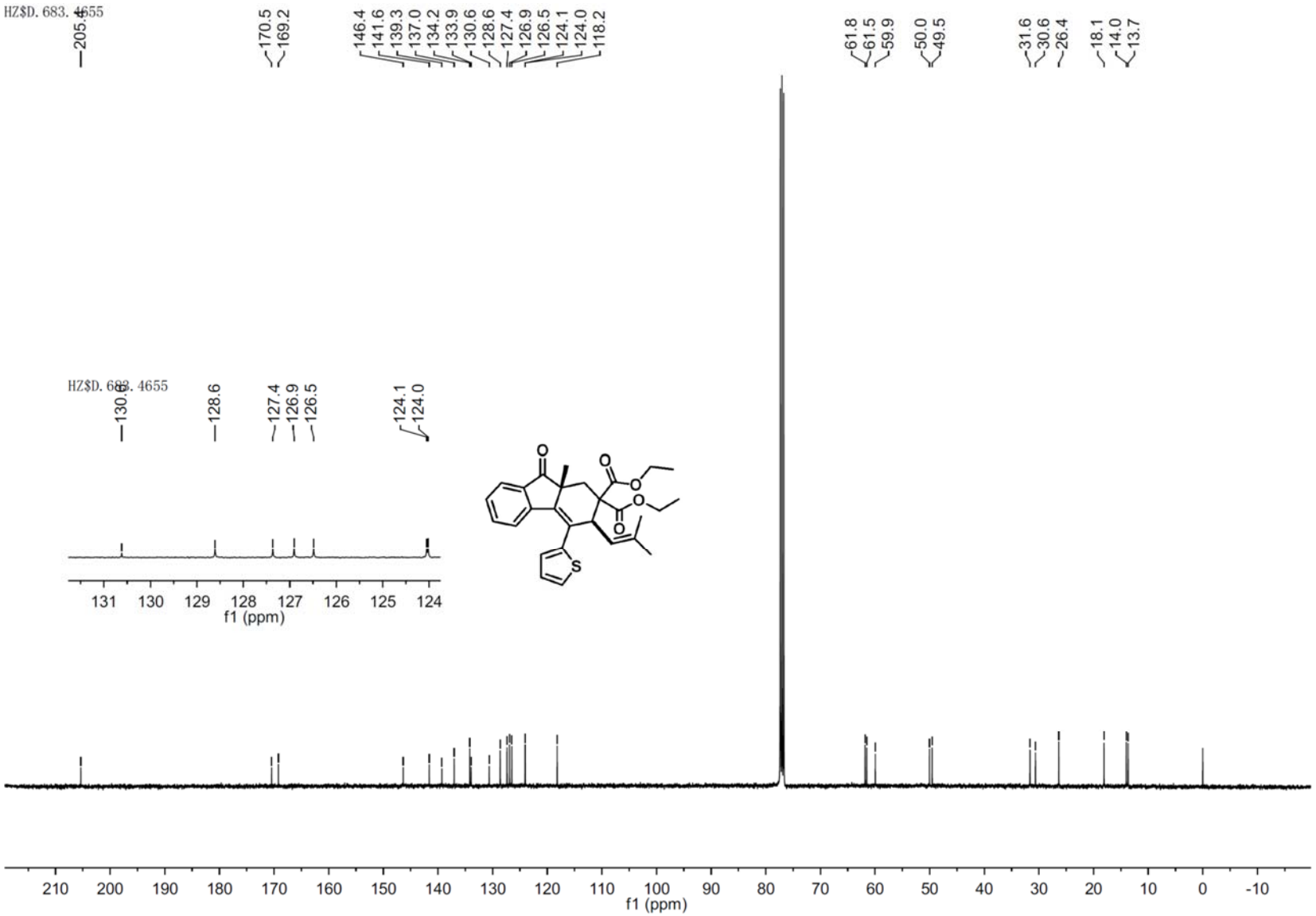
^{1}H NMR Spectrum of Compound 3g



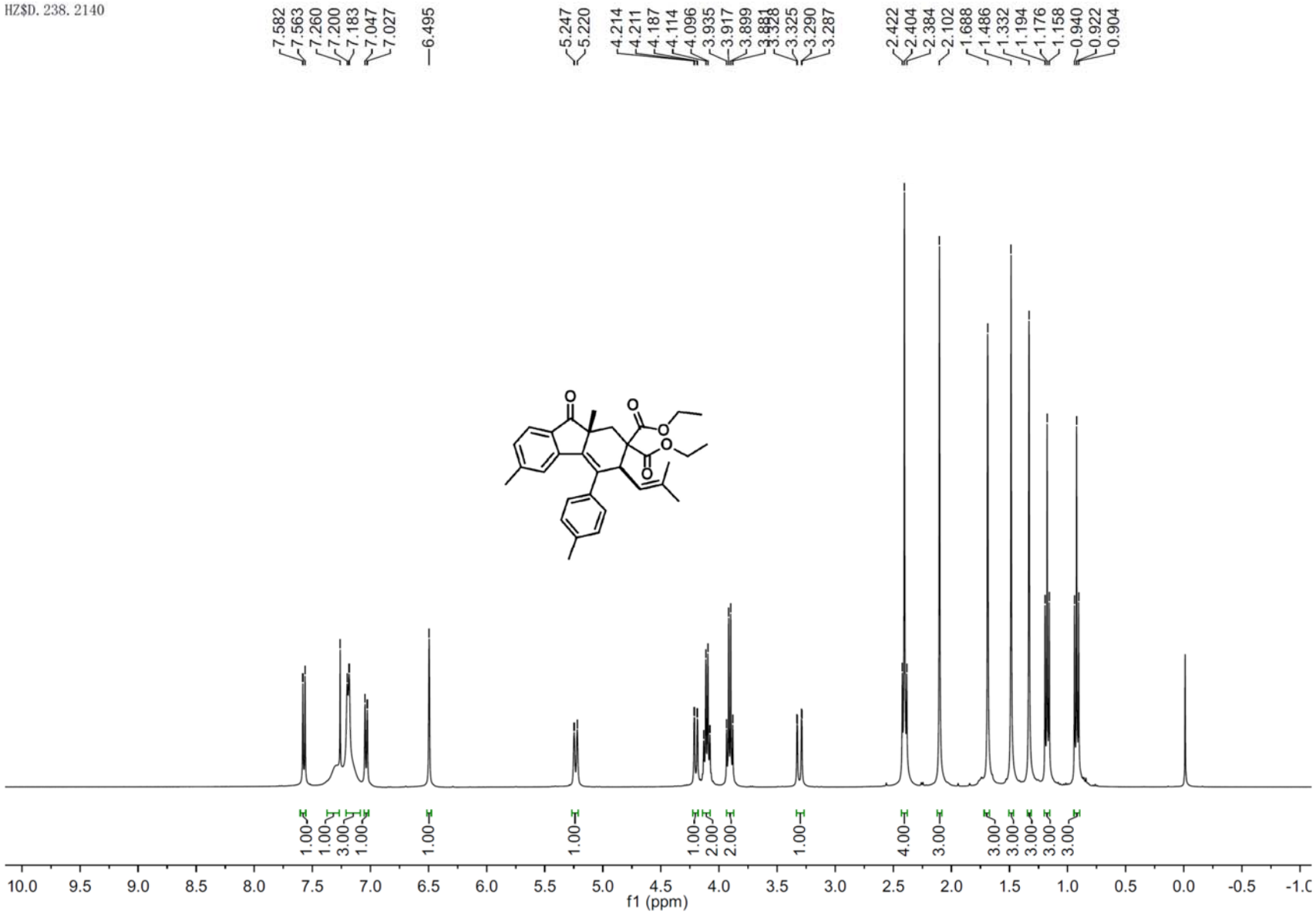
^{13}C NMR Spectrum of Compound 3g



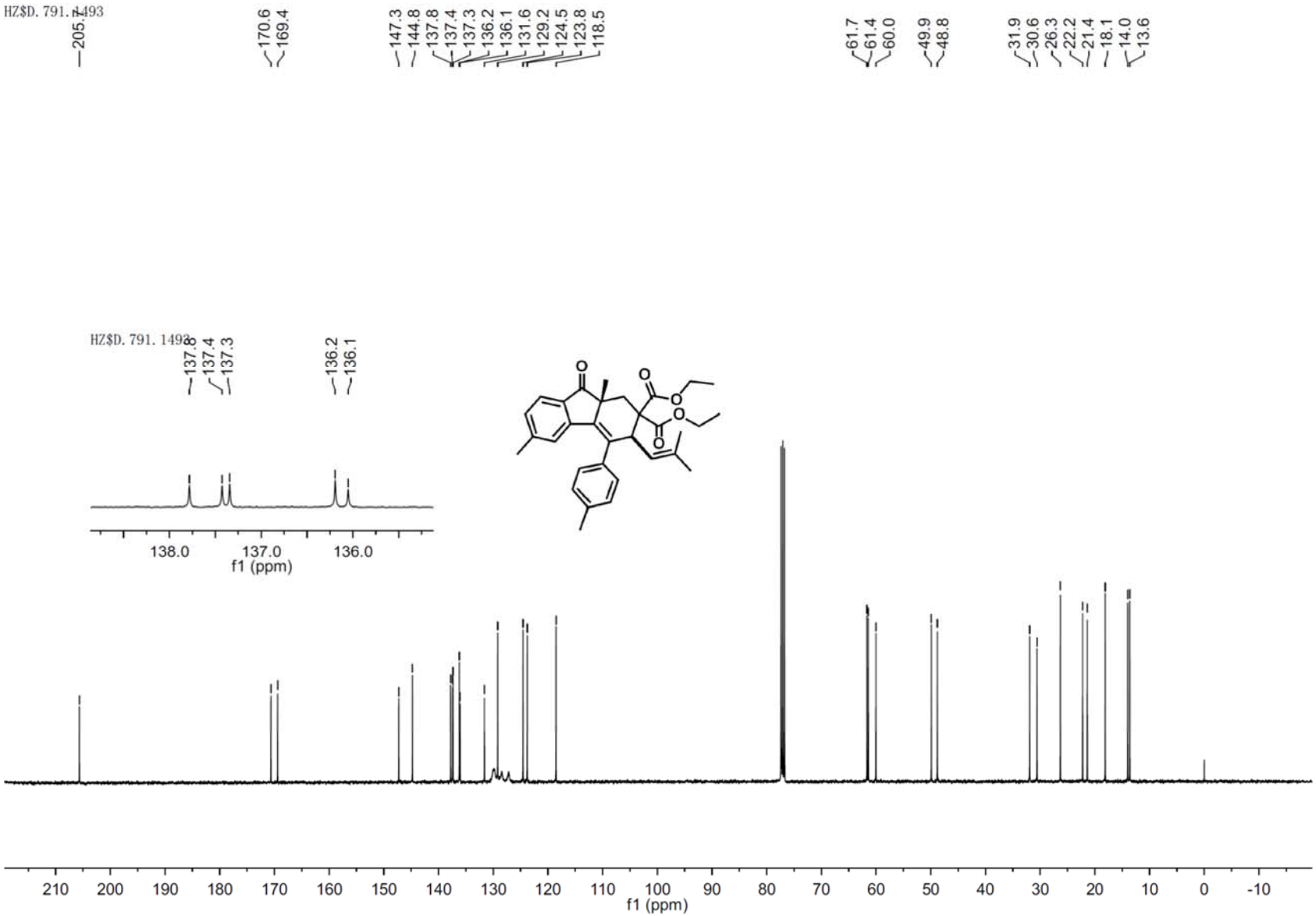
^{1}H NMR Spectrum of Compound 3h



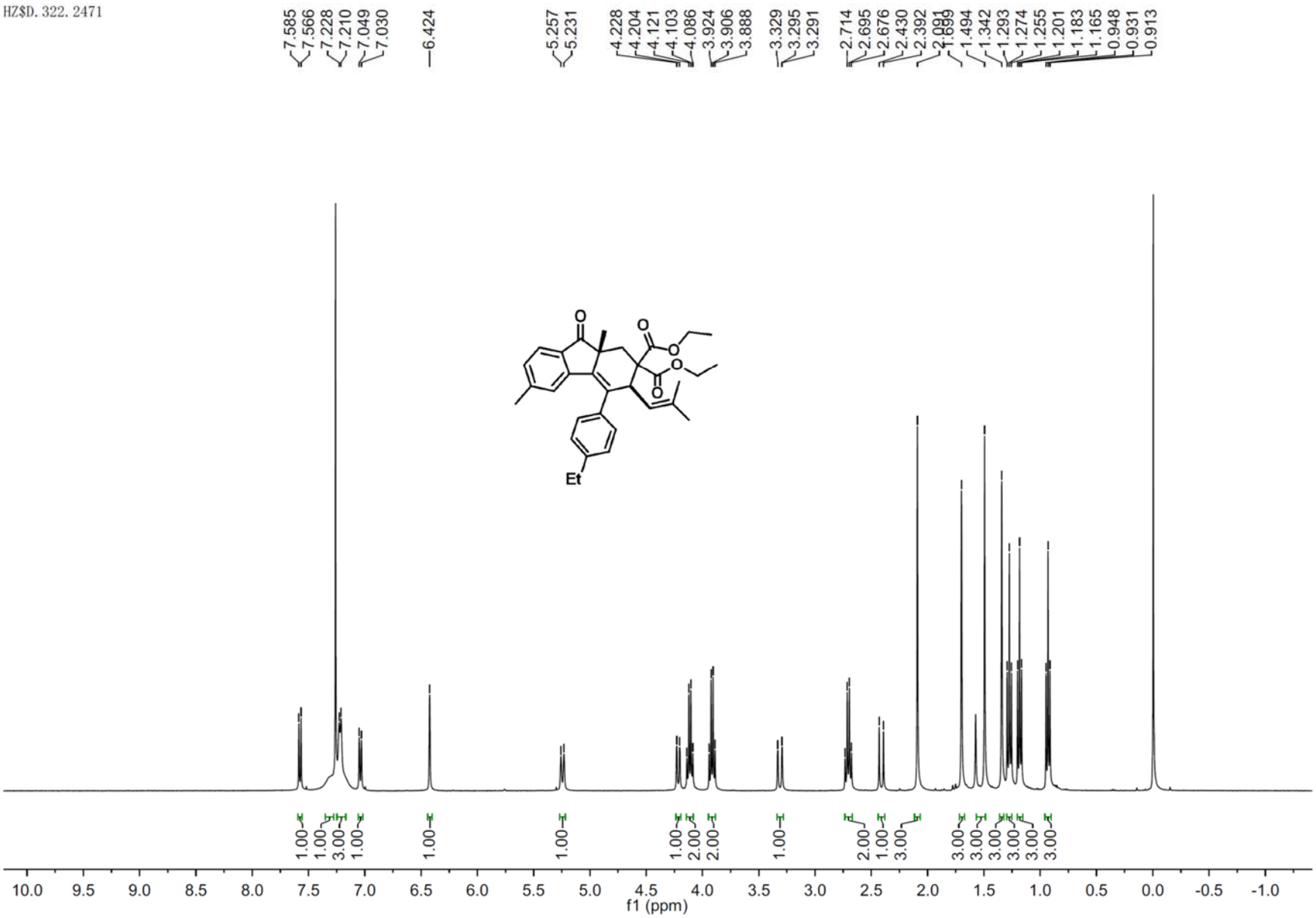
^{13}C NMR Spectrum of Compound 3h



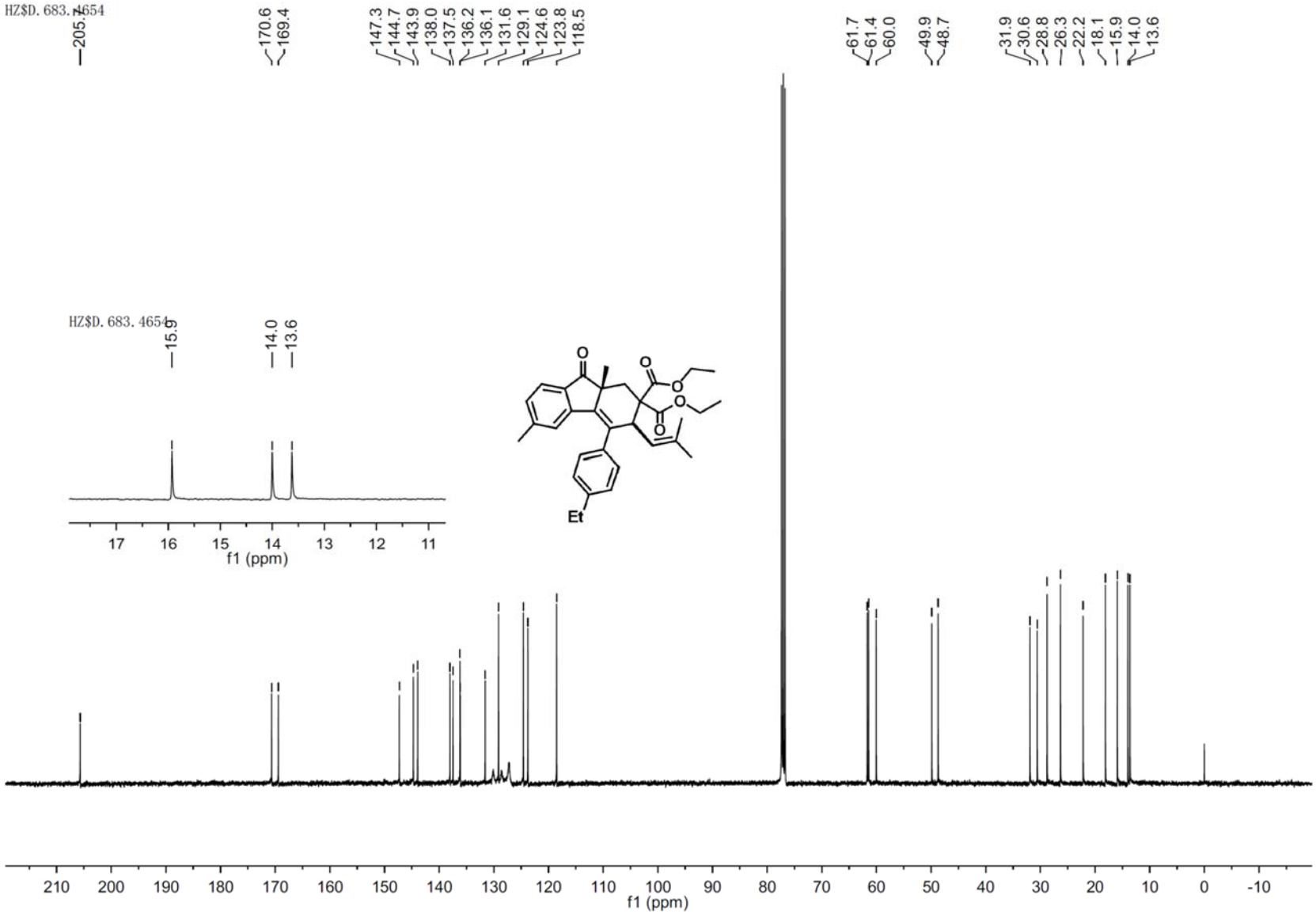
^{1}H NMR Spectrum of Compound 3i



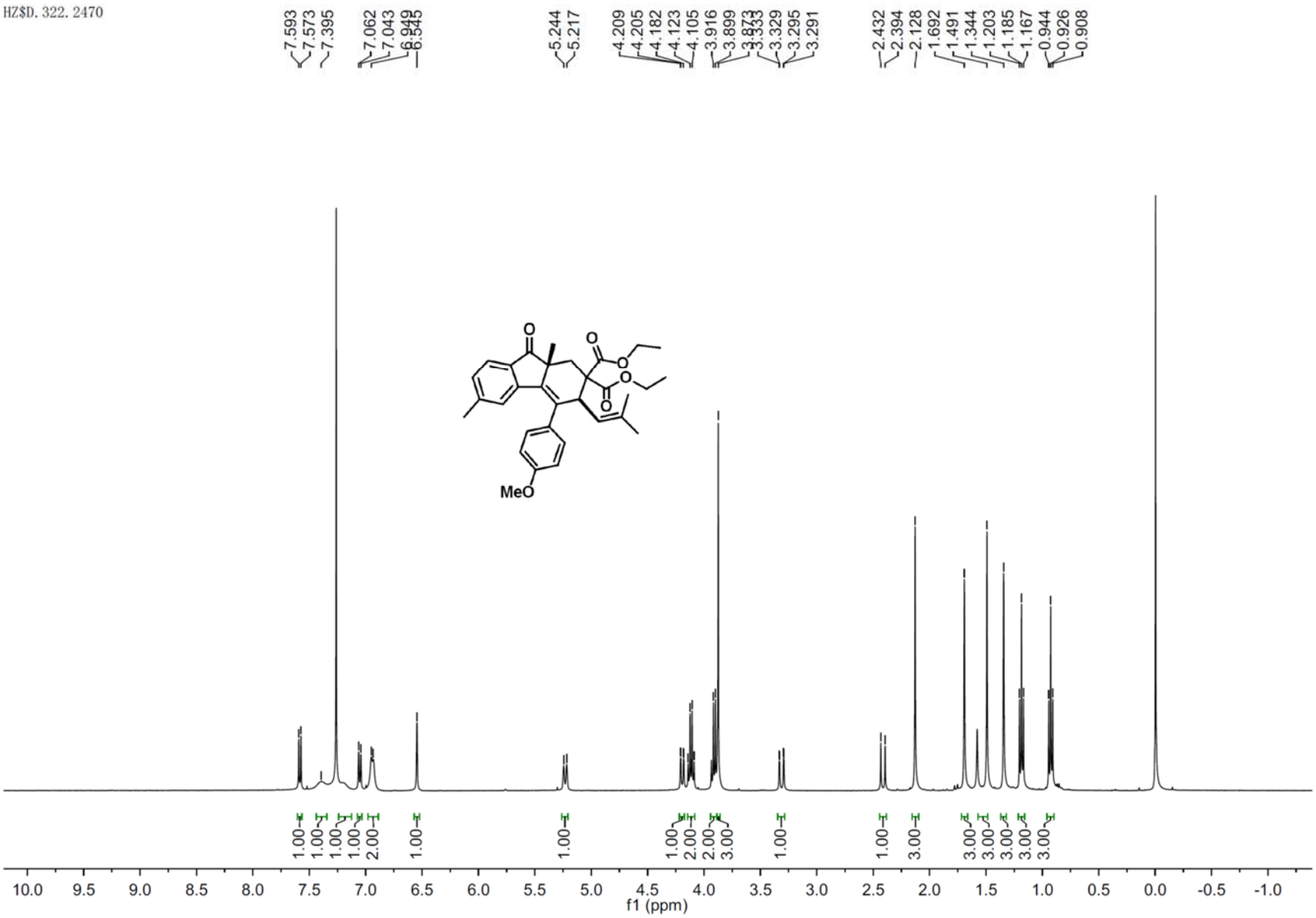
^{13}C NMR Spectrum of Compound 3i



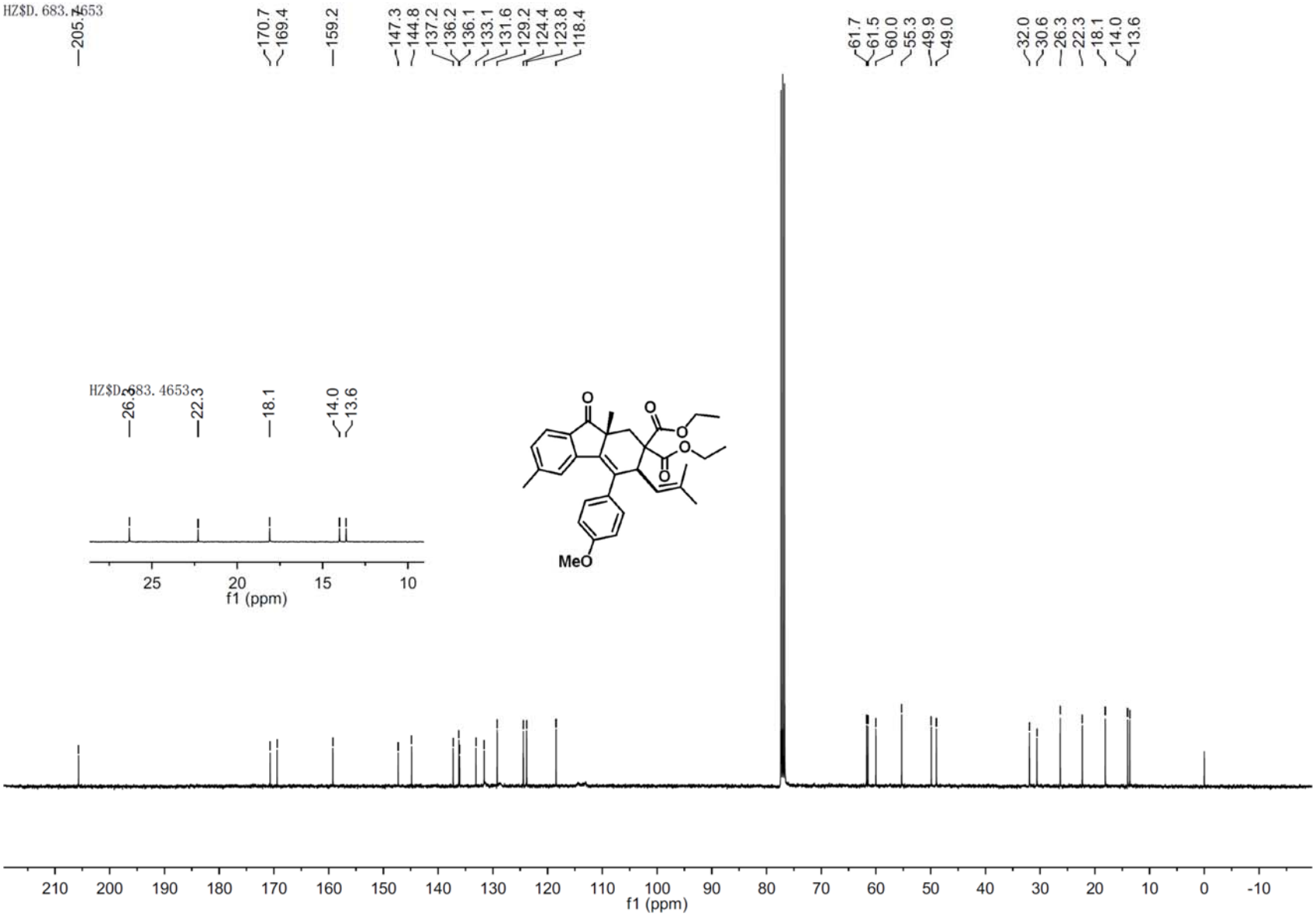
^{1}H NMR Spectrum of Compound 3j



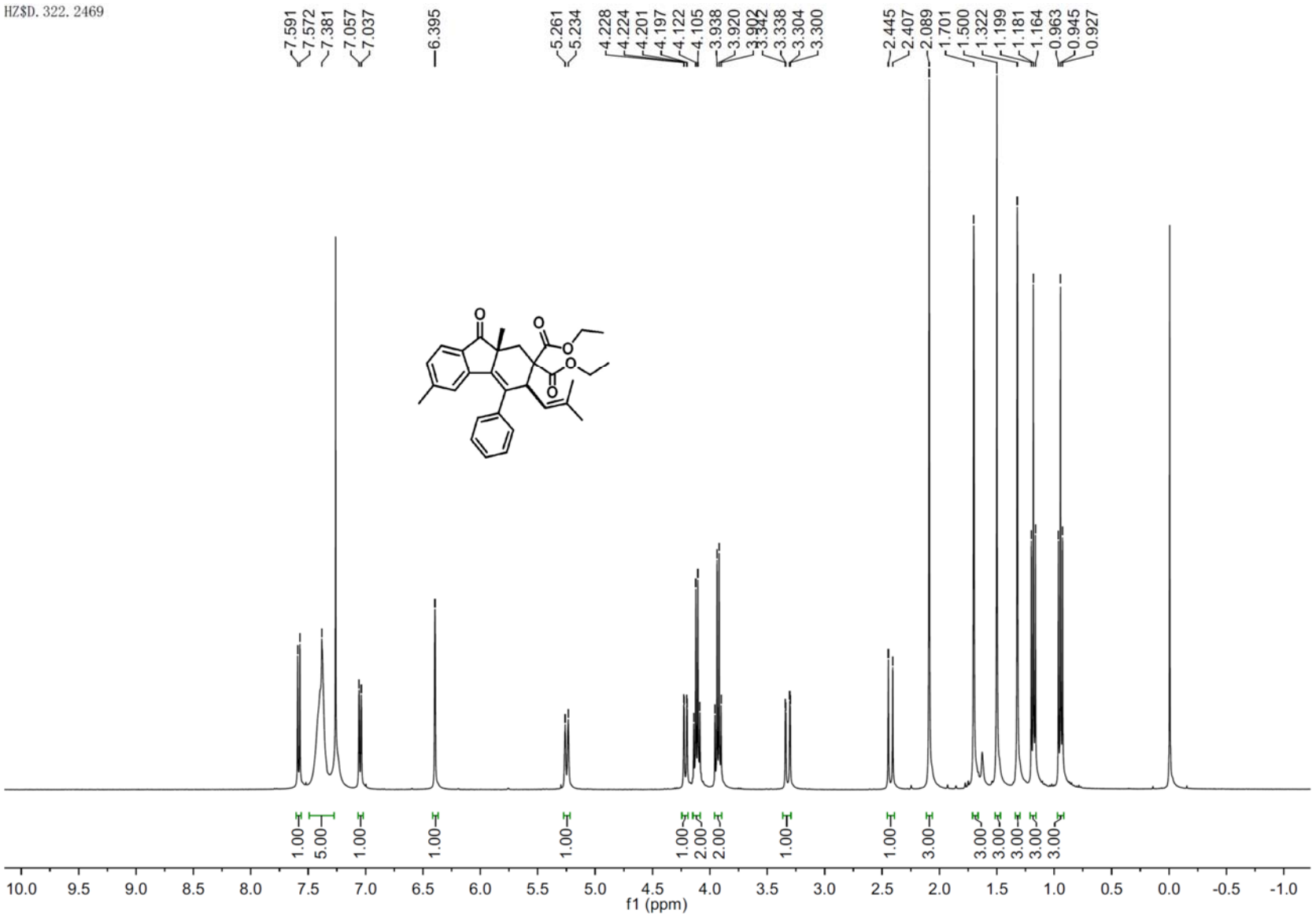
^{13}C NMR Spectrum of Compound 3j



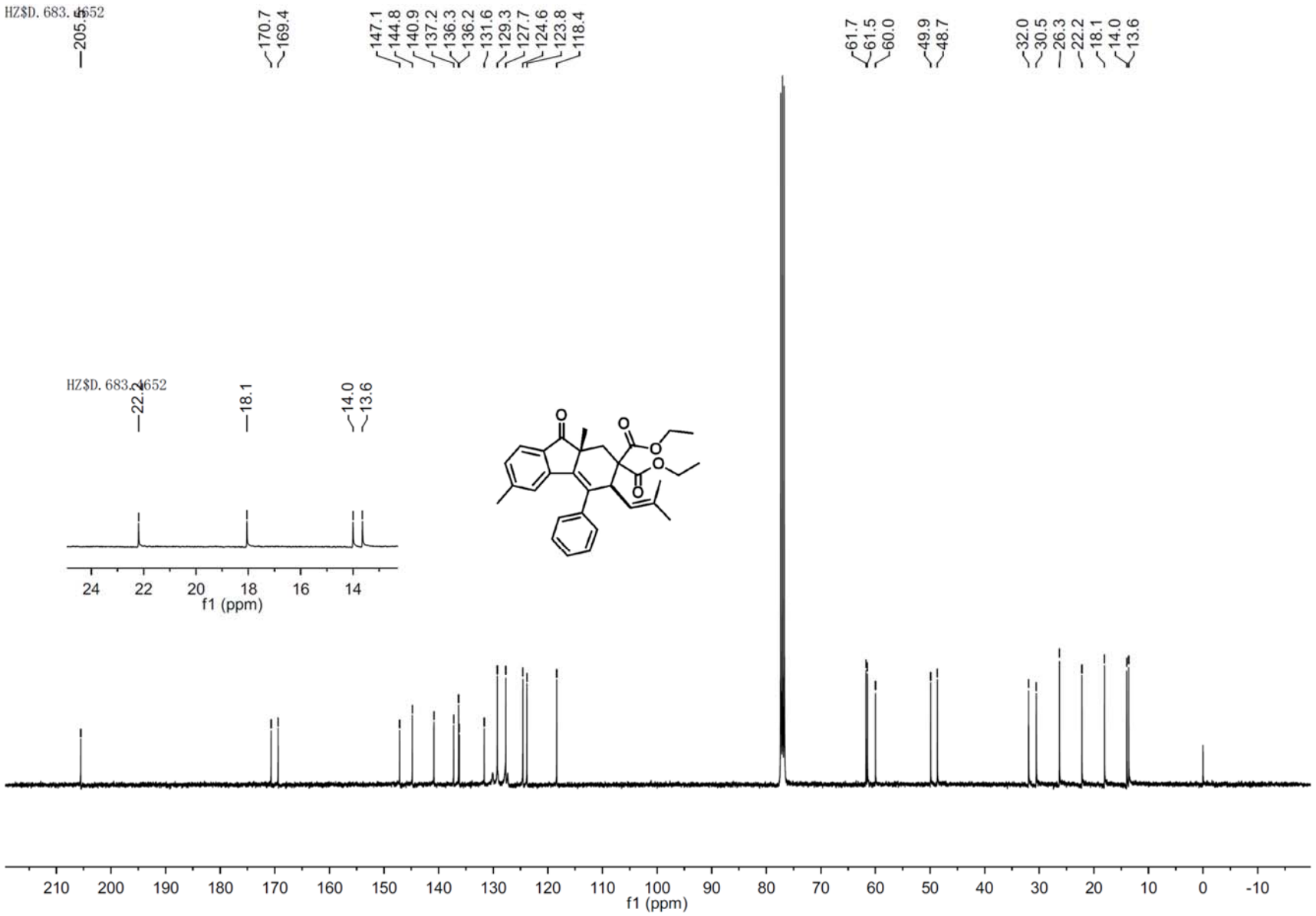
^{1}H NMR Spectrum of Compound 3k



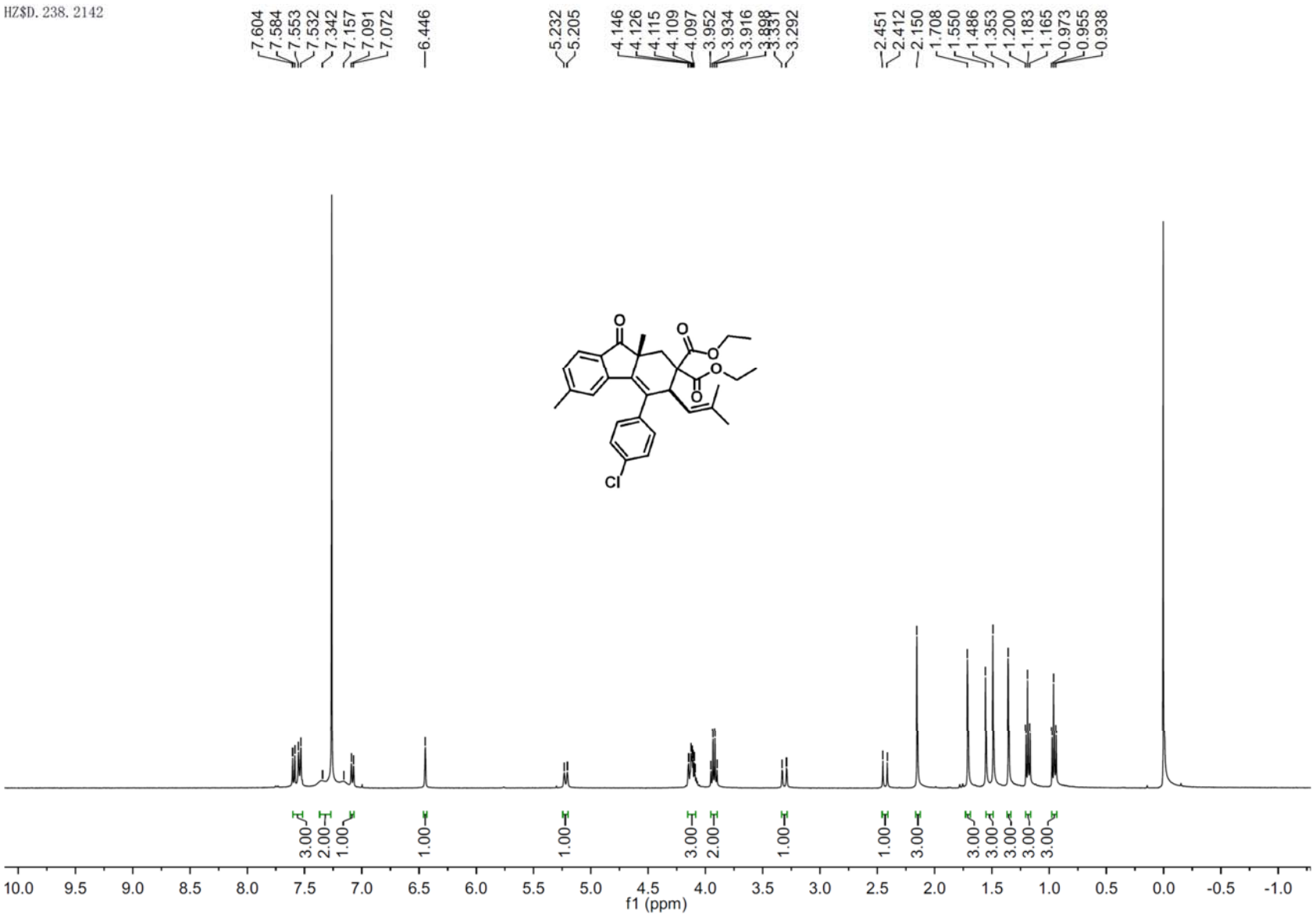
^{13}C NMR Spectrum of Compound 3k



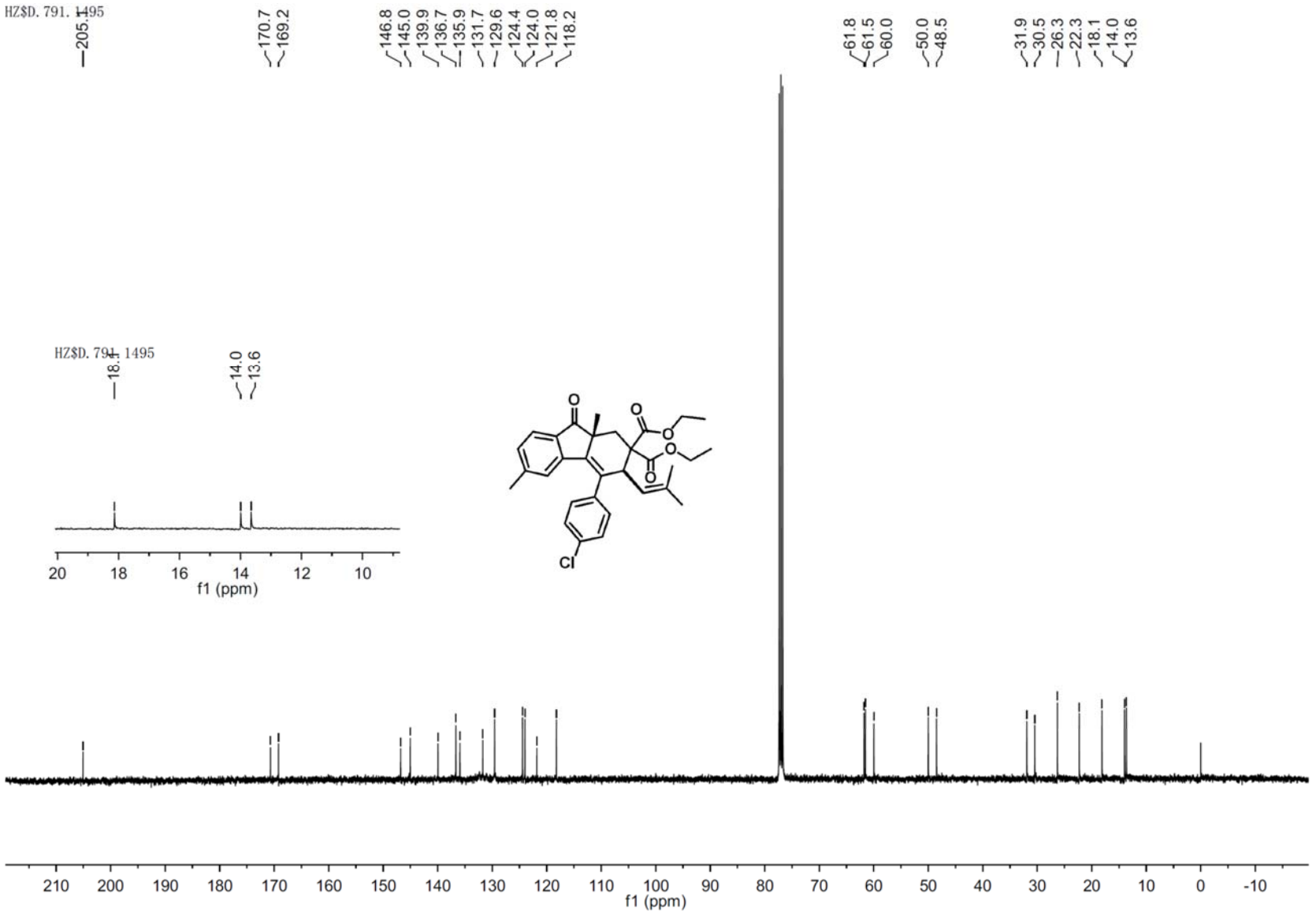
^{1}H NMR Spectrum of Compound 3l



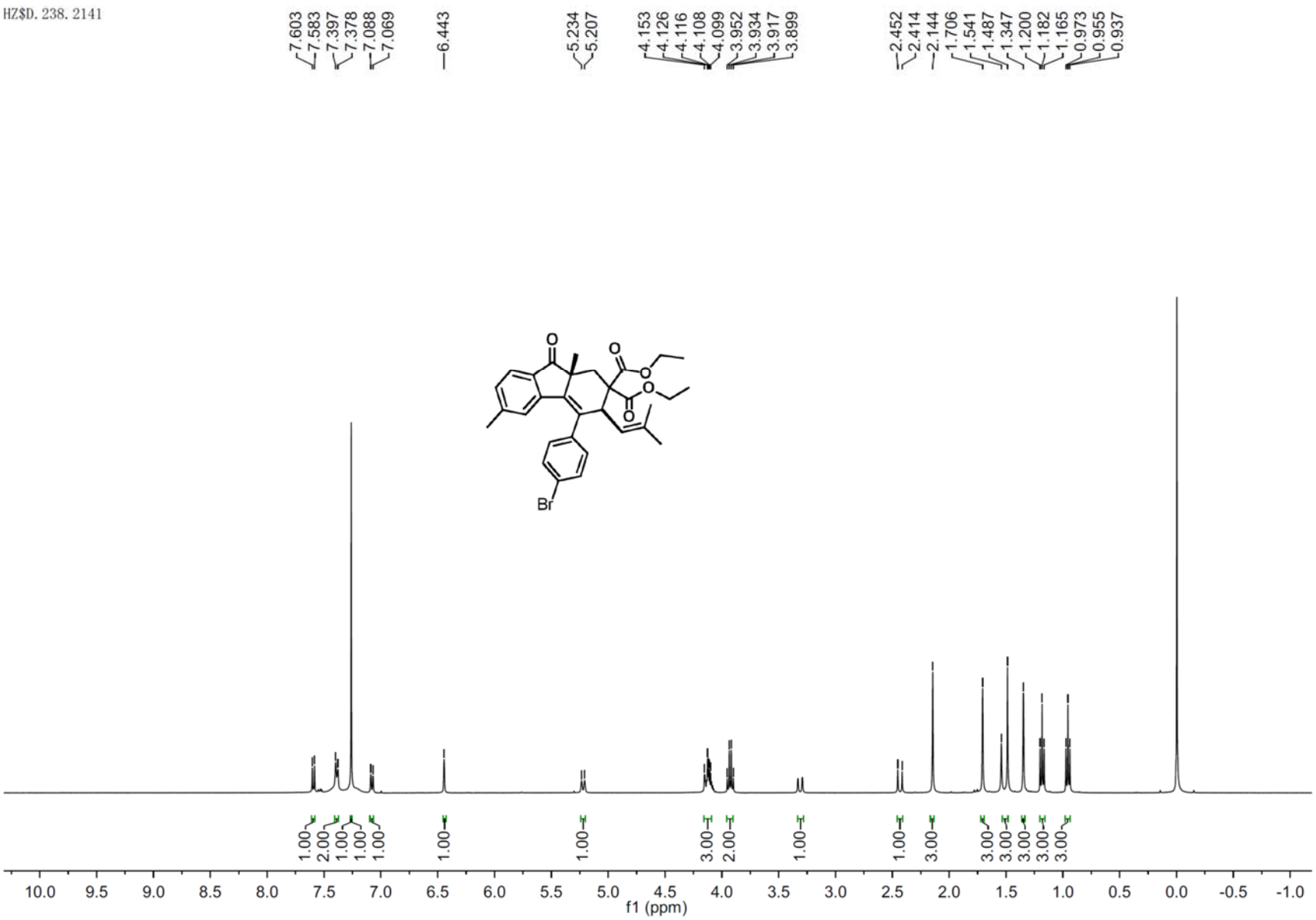
^{13}C NMR Spectrum of Compound 3l



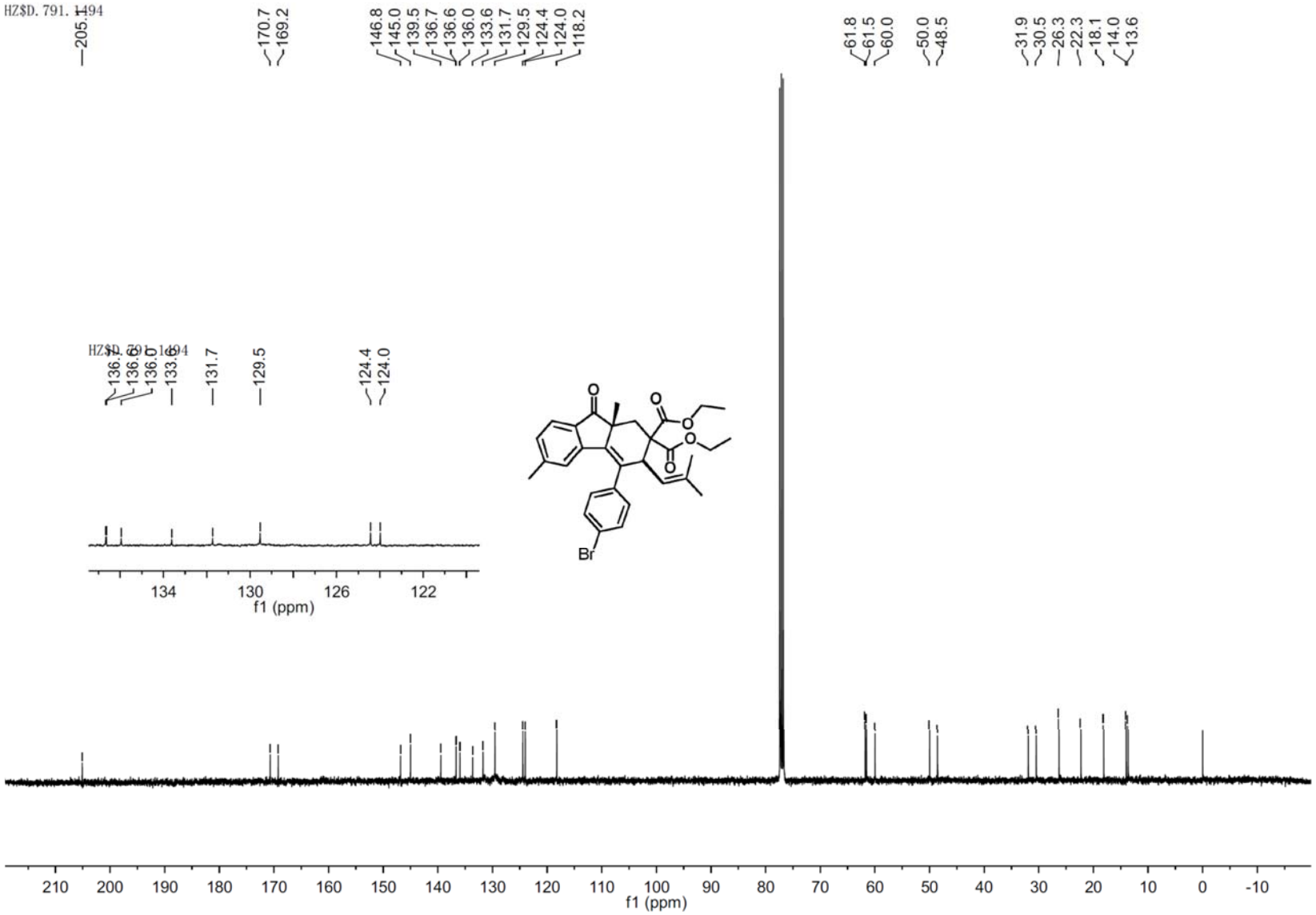
^{1}H NMR Spectrum of Compound 3m



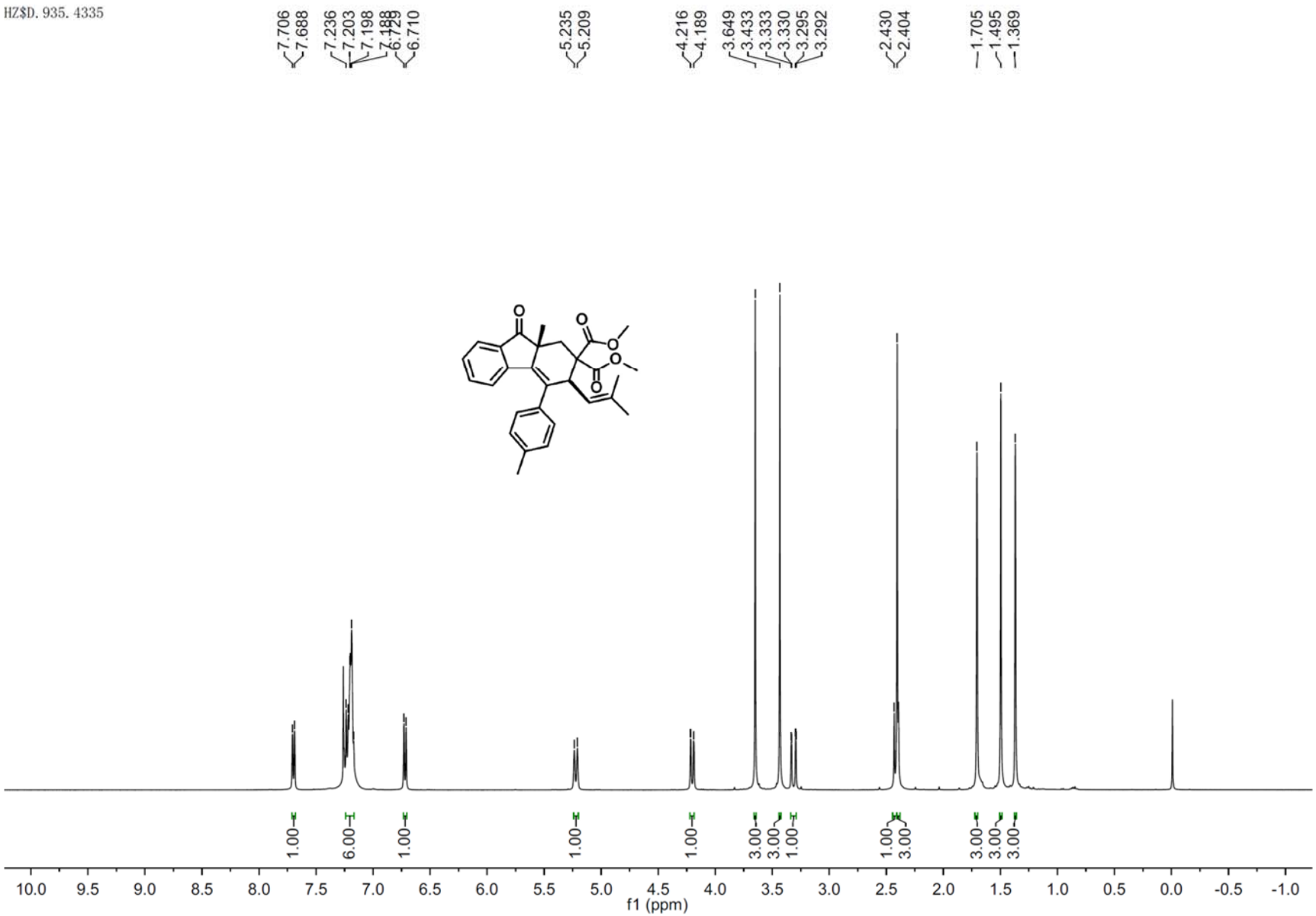
^{13}C NMR Spectrum of Compound 3m



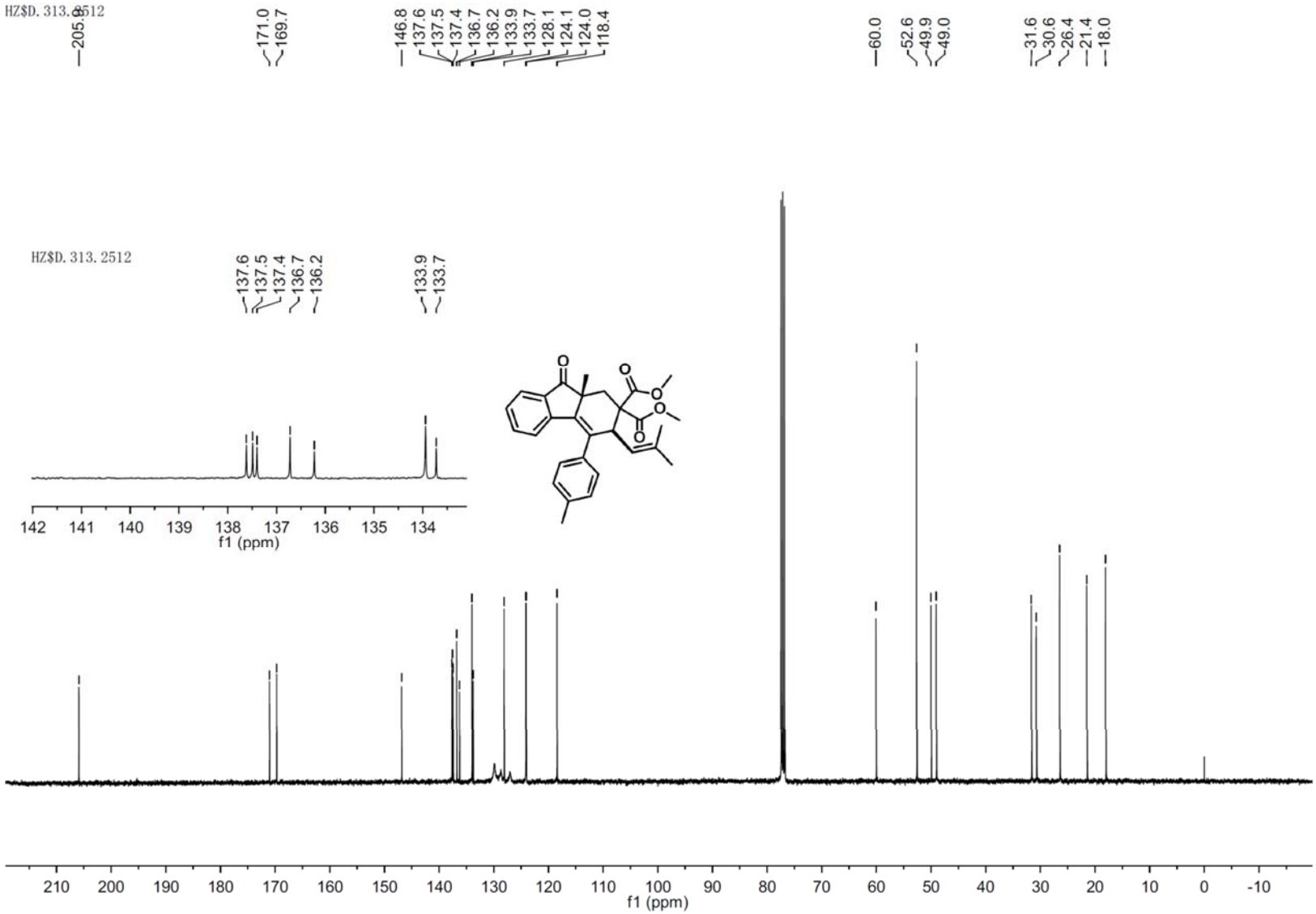
^{1}H NMR Spectrum of Compound 3n



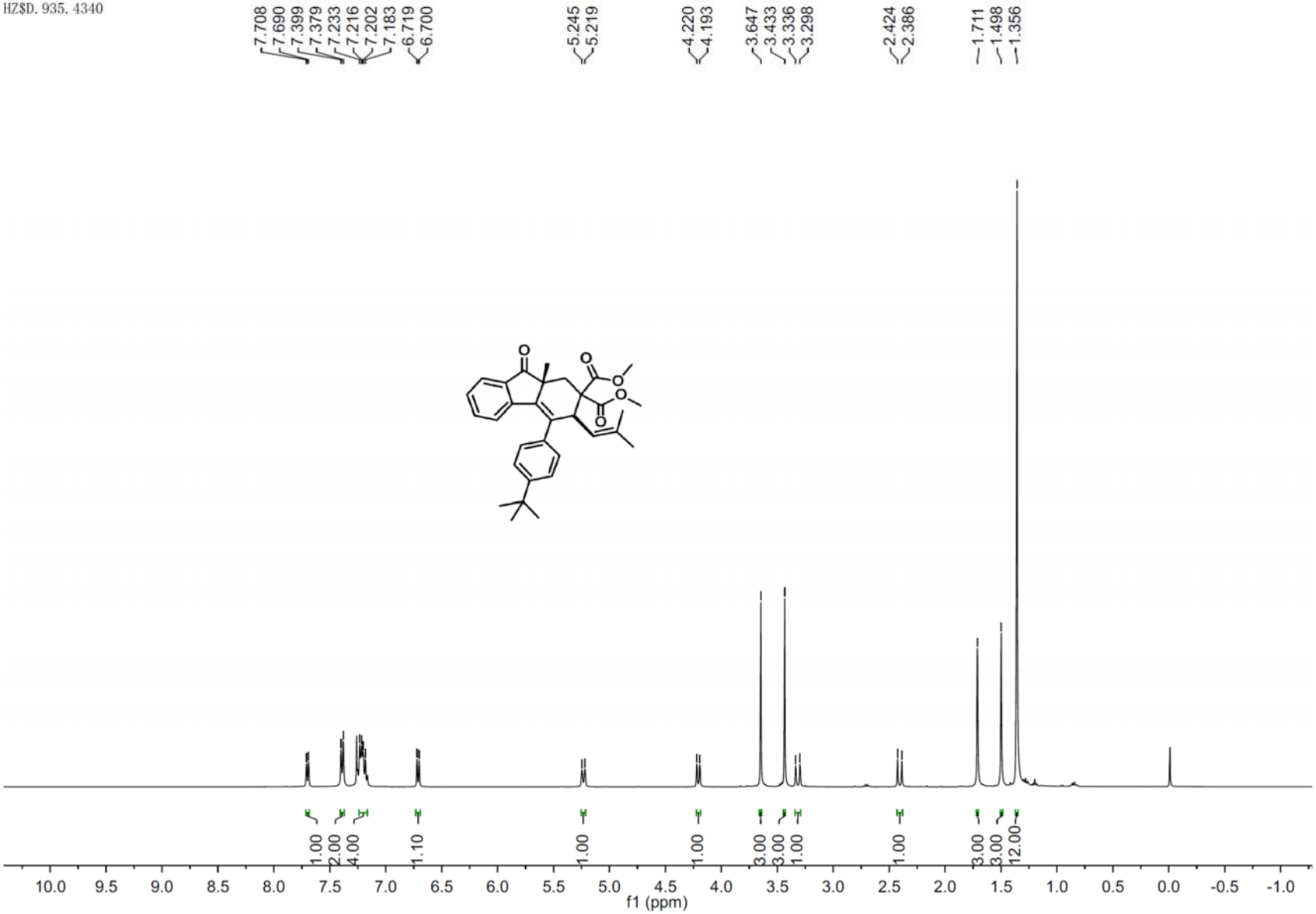
^{13}C NMR Spectrum of Compound 3n



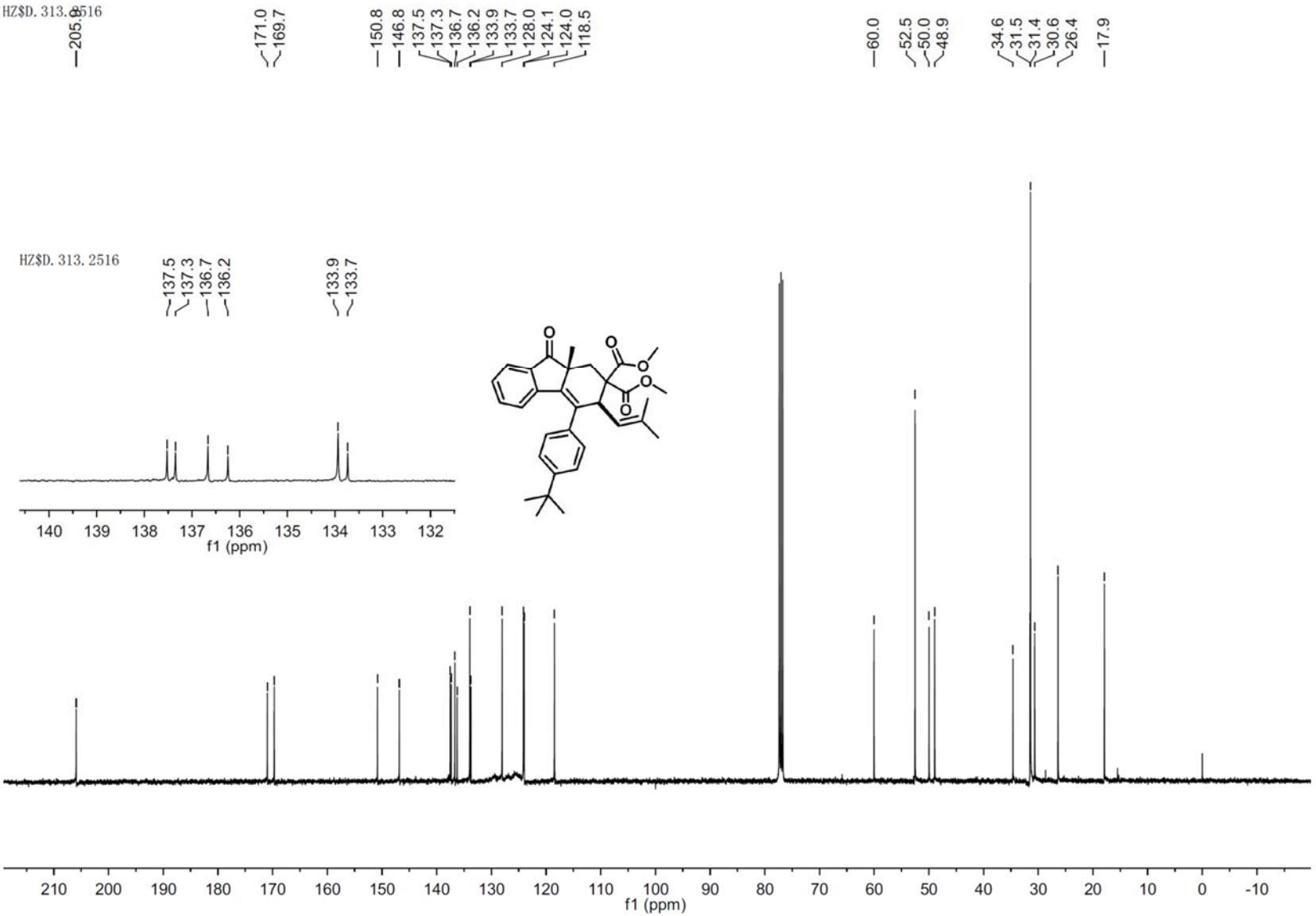
^{1}H NMR Spectrum of Compound 3o



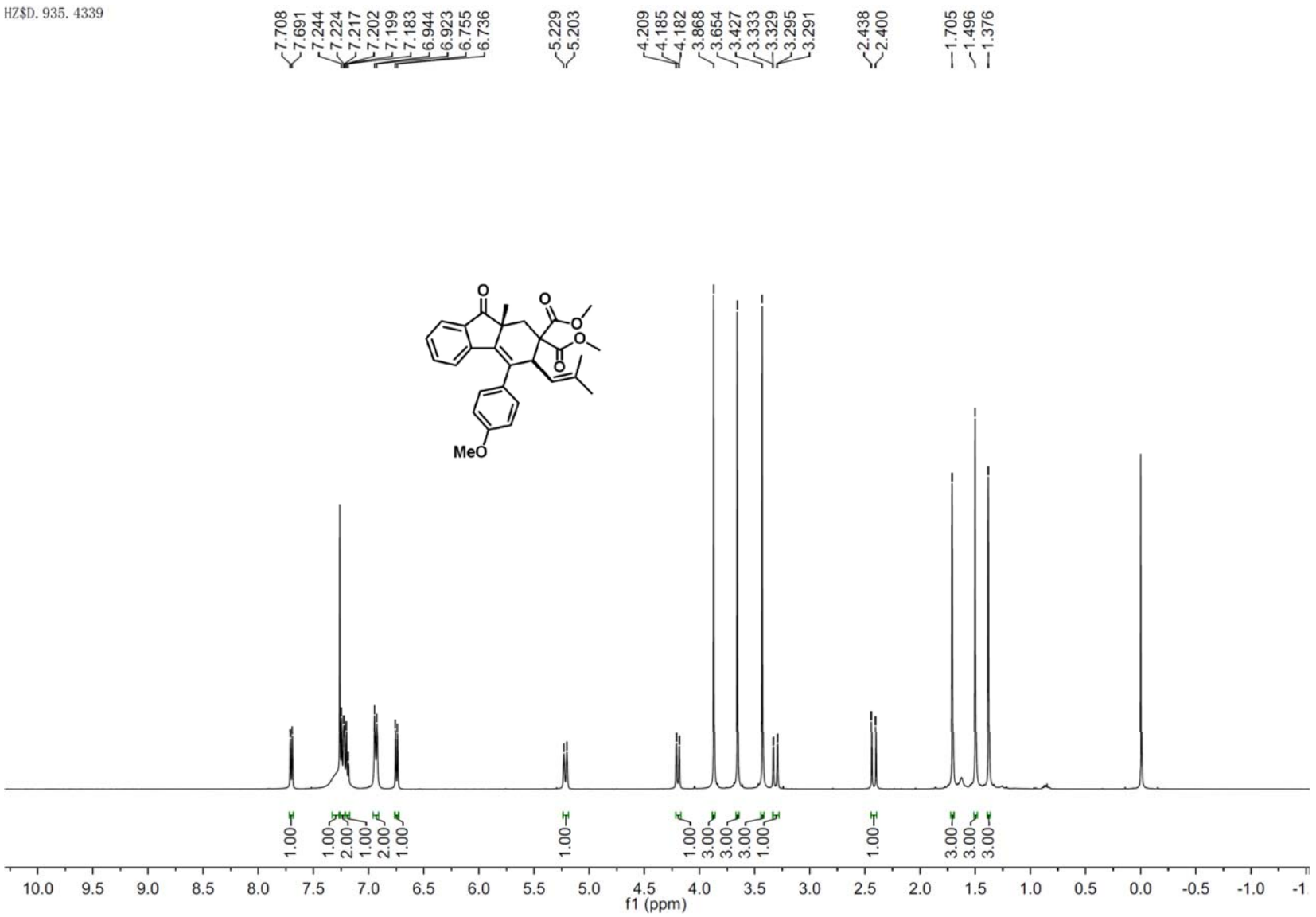
^{13}C NMR Spectrum of Compound 3o



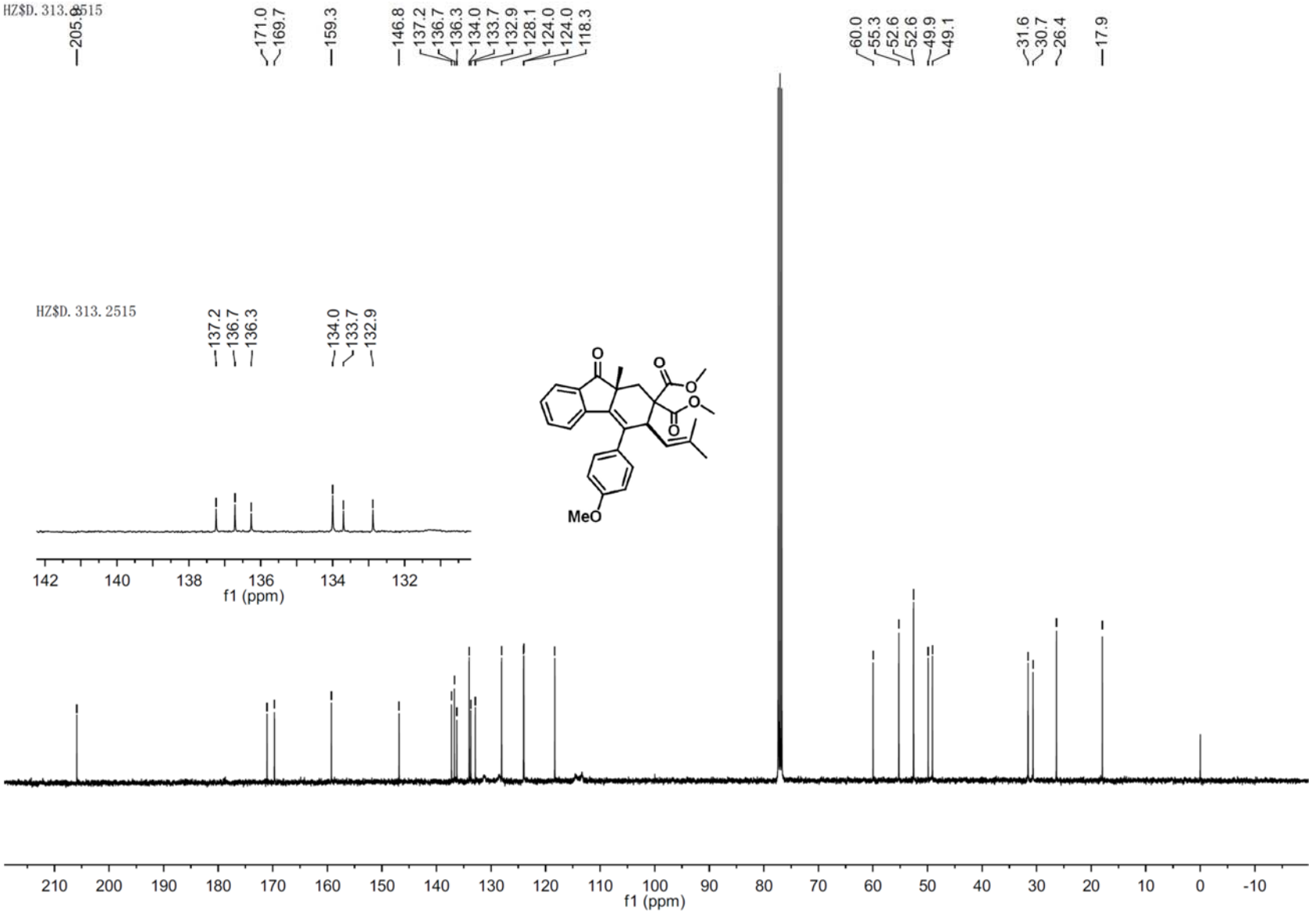
^{1}H NMR Spectrum of Compound 3p



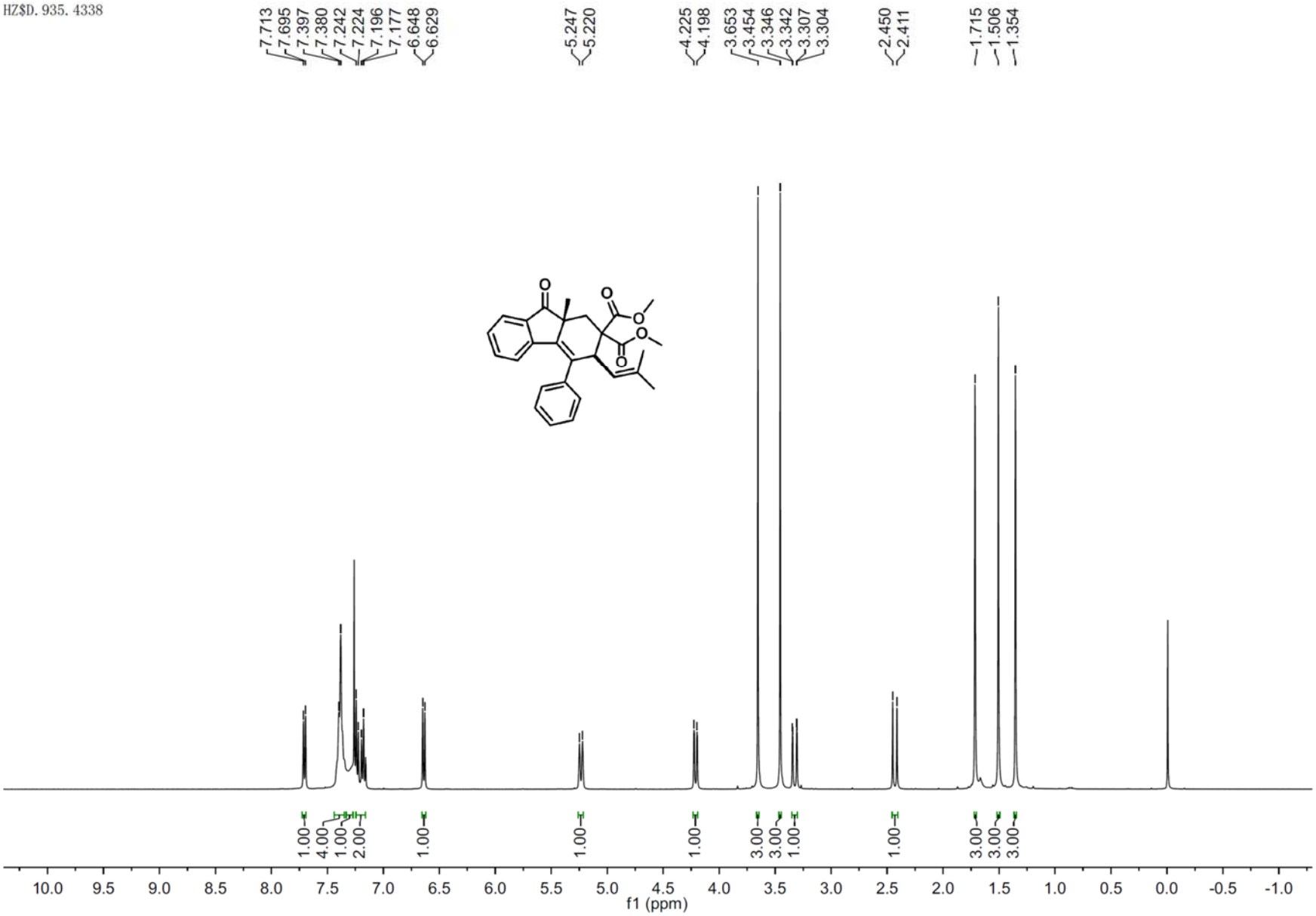
^{13}C NMR Spectrum of Compound 3p



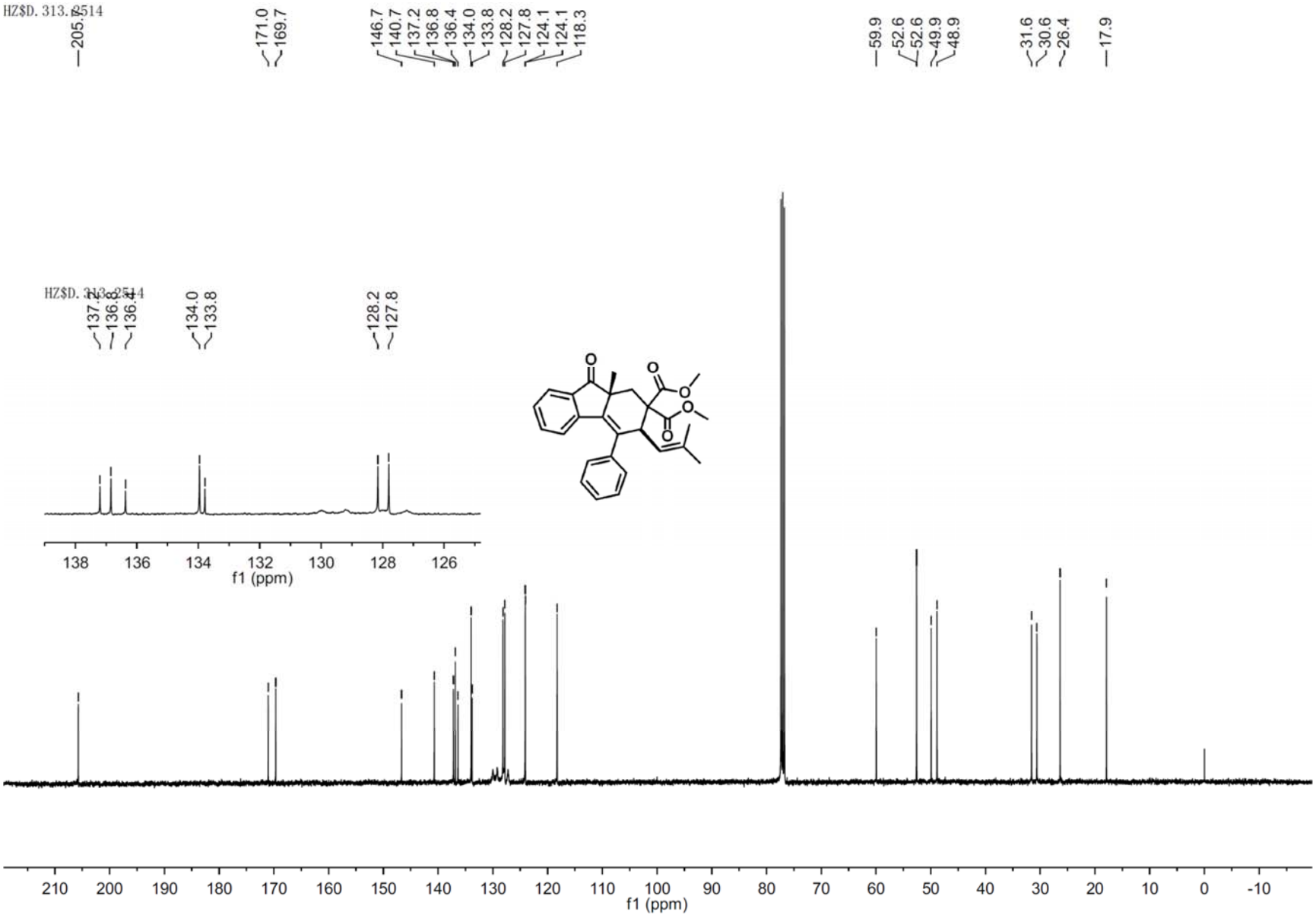
^{1}H NMR Spectrum of Compound 3q



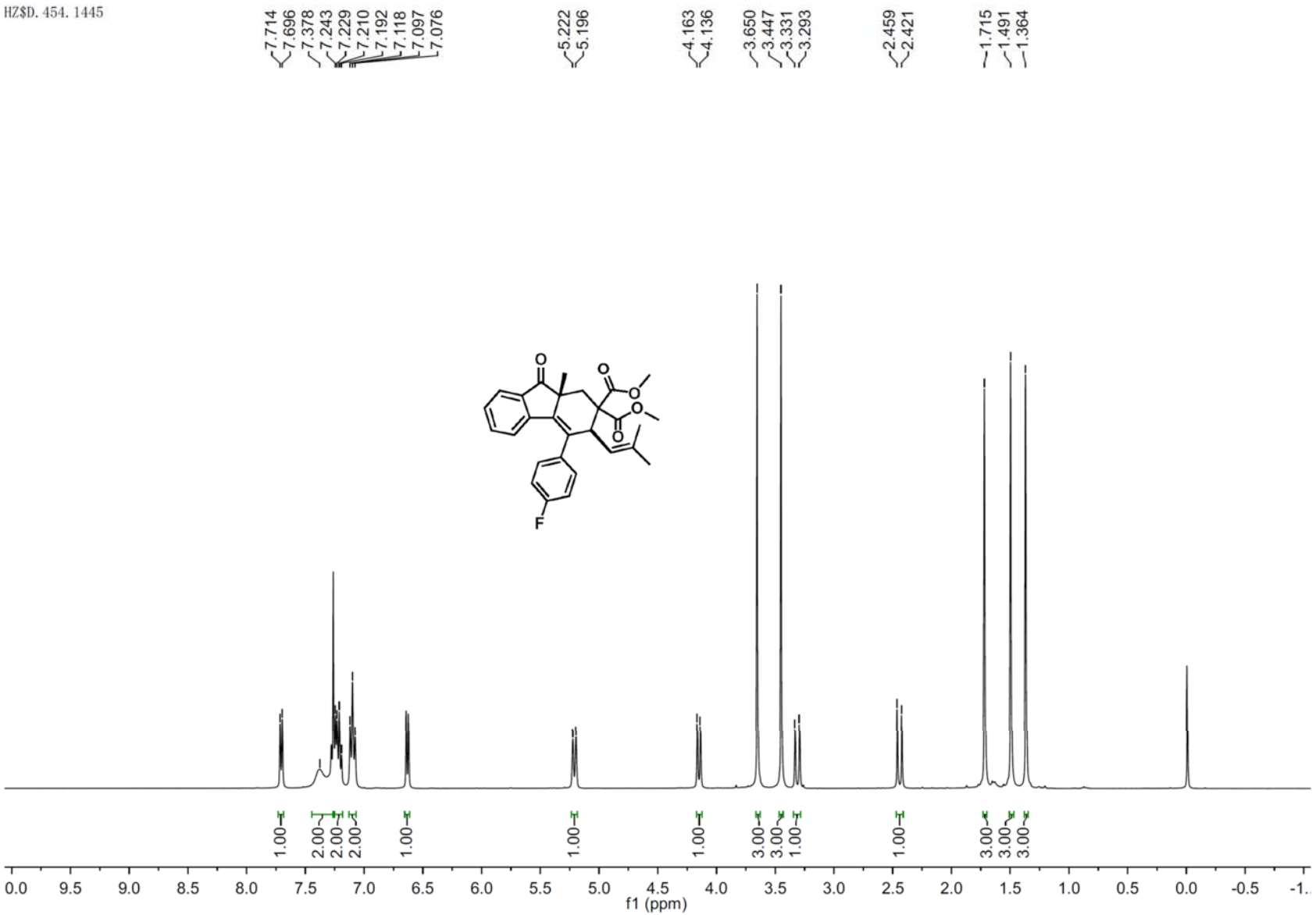
^{13}C NMR Spectrum of Compound 3q



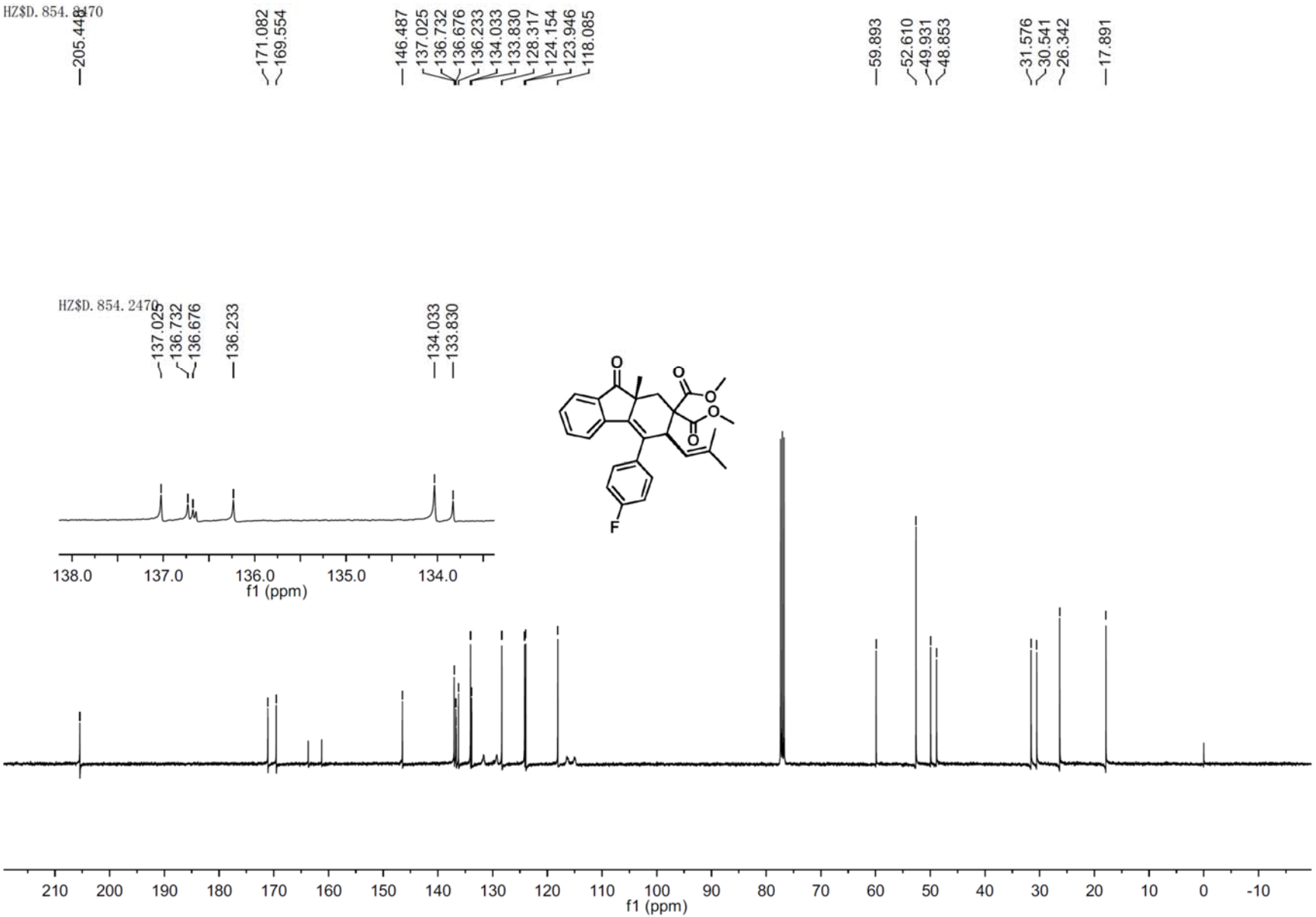
^{1}H NMR Spectrum of Compound 3r



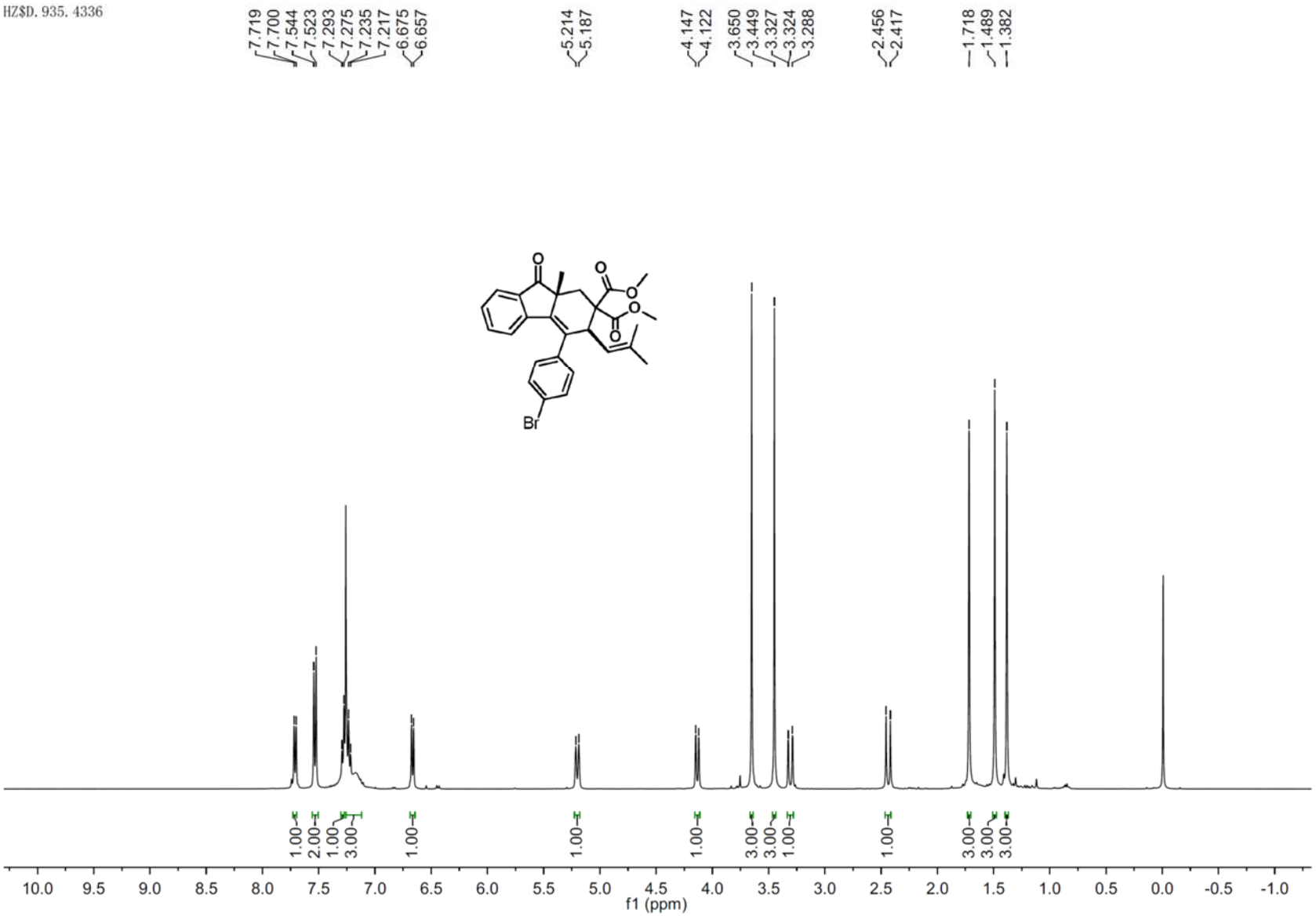
^{13}C NMR Spectrum of Compound 3r



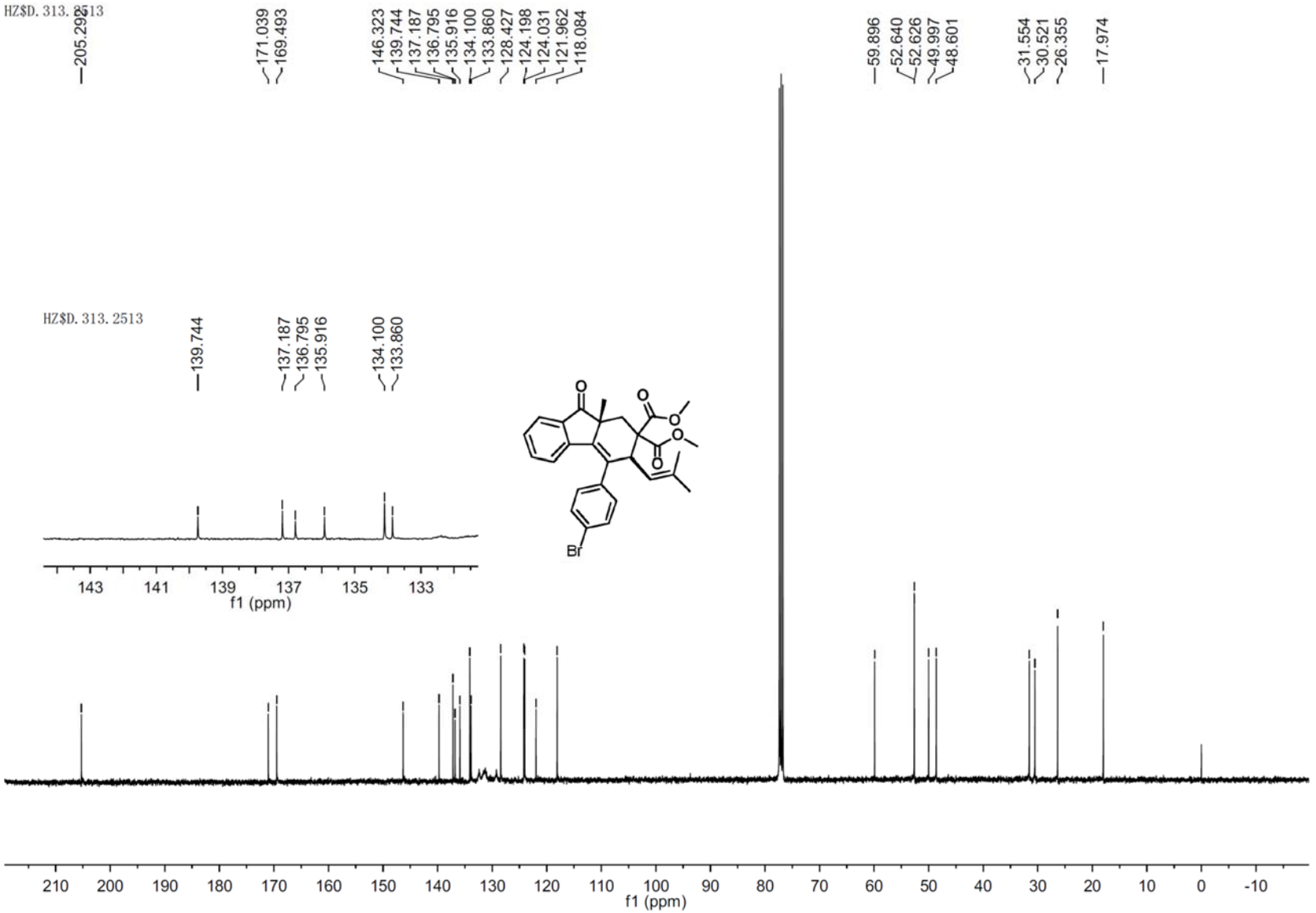
^{1}H NMR Spectrum of Compound 3s



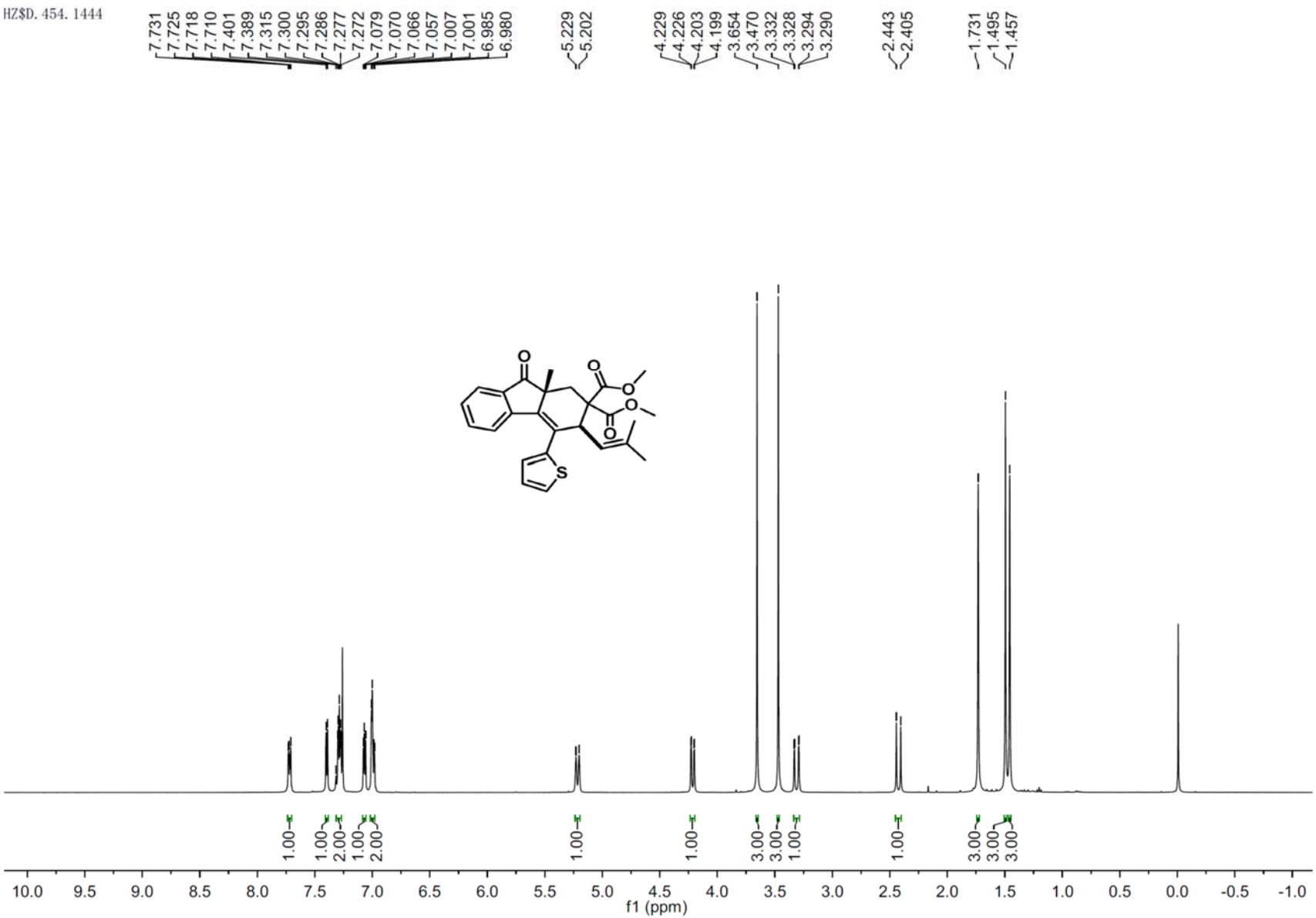
^{13}C NMR Spectrum of Compound 3s



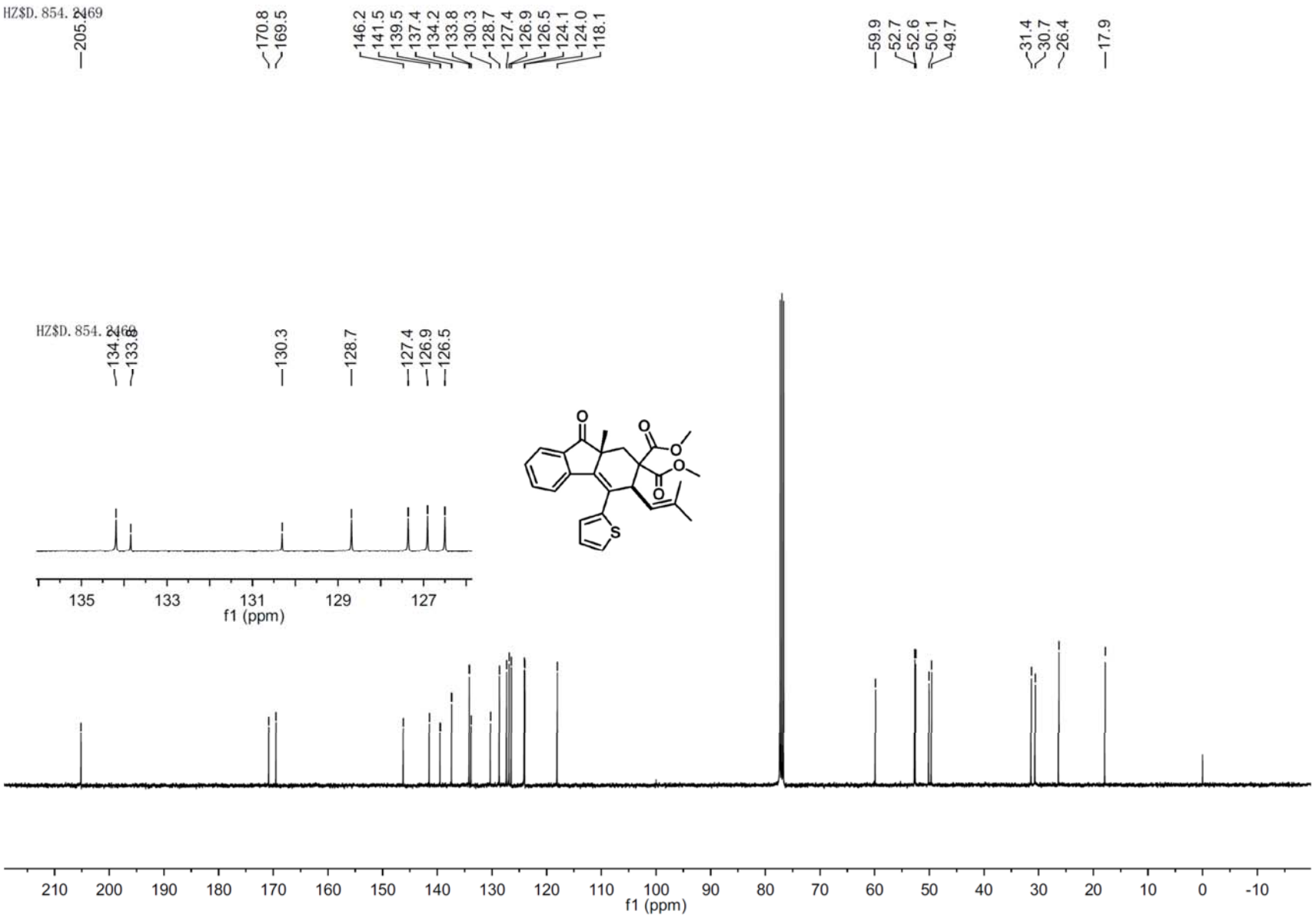
^{1}H NMR Spectrum of Compound 3t



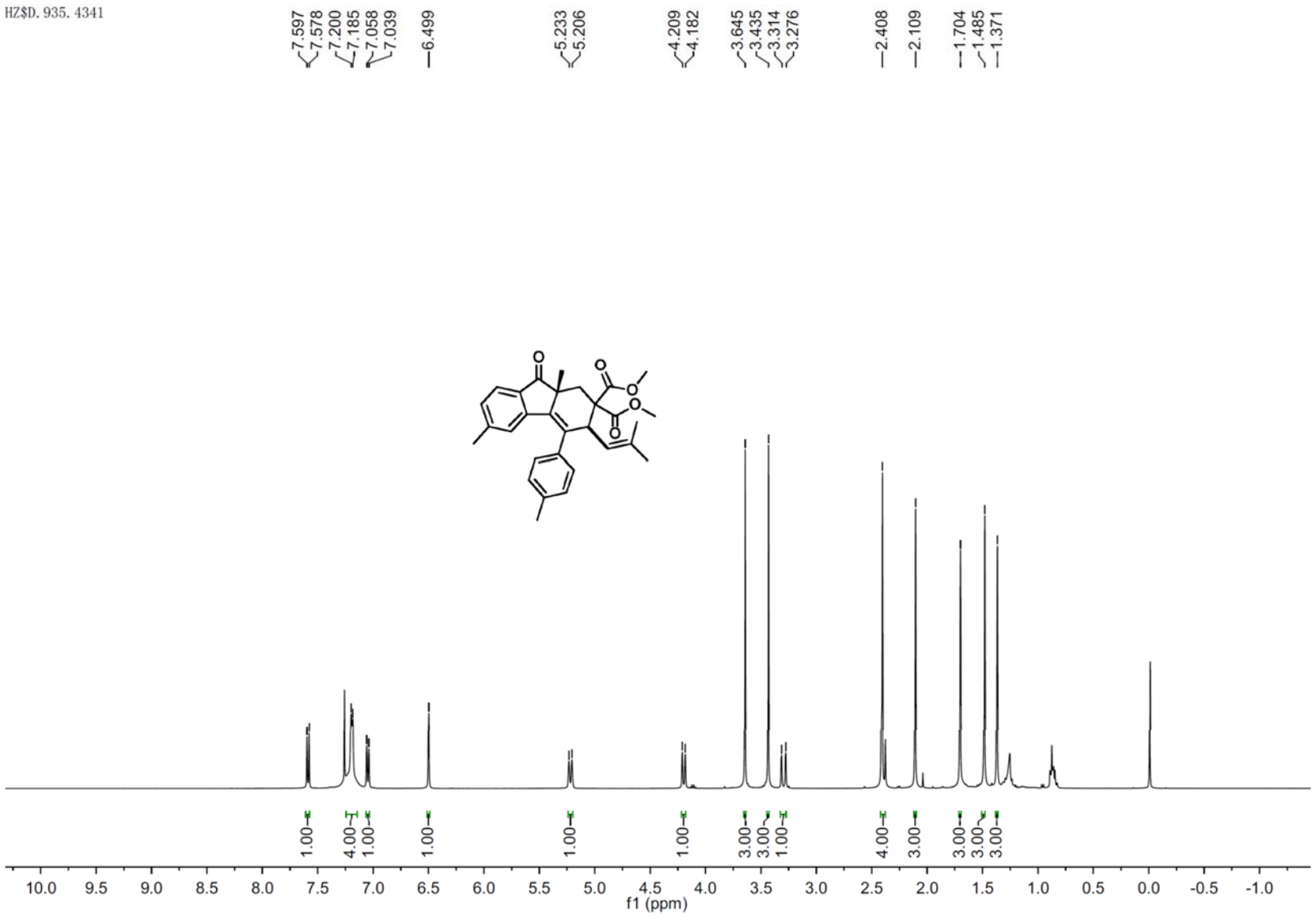
^{13}C NMR Spectrum of Compound 3t



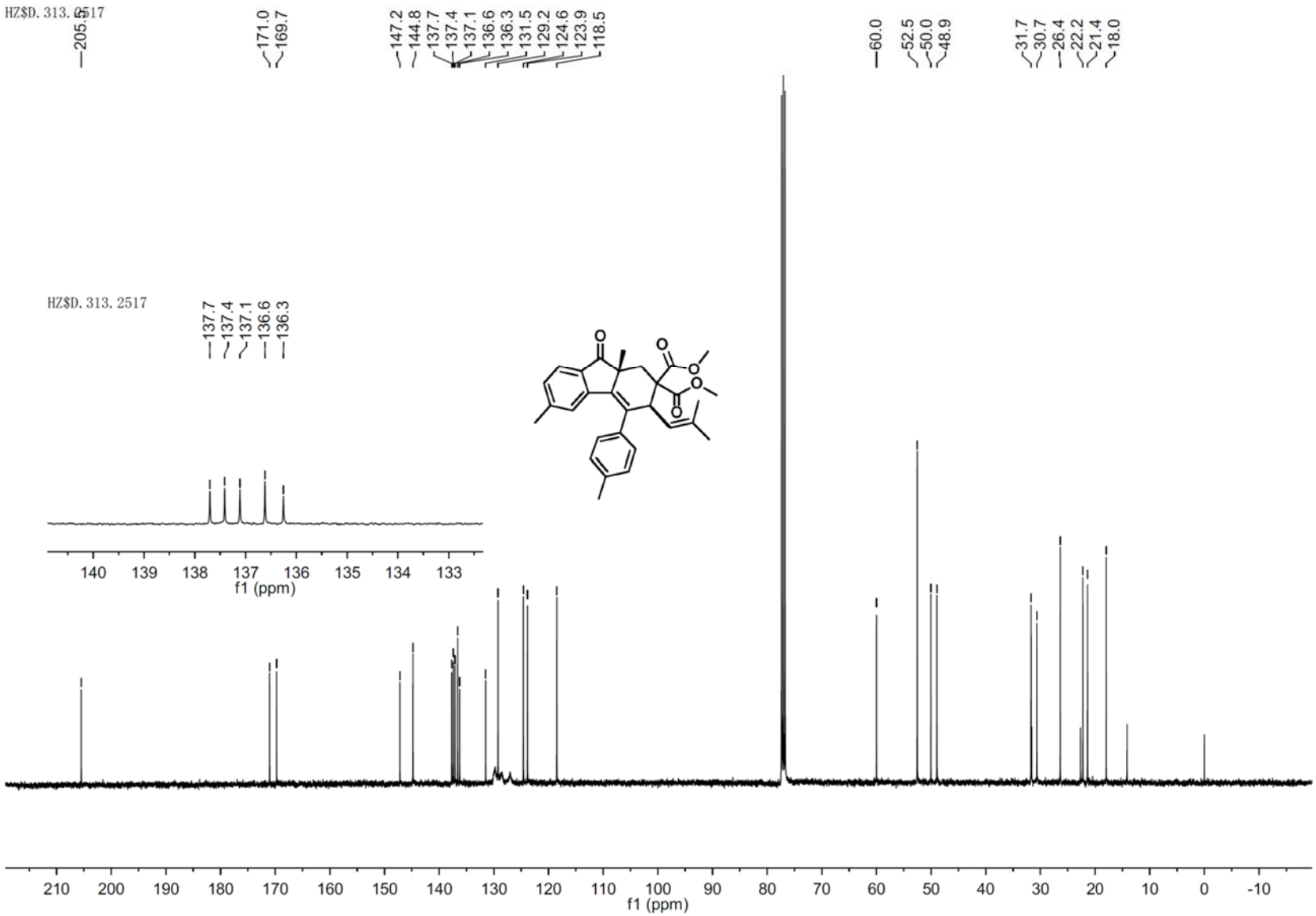
^{1}H NMR Spectrum of Compound 3u



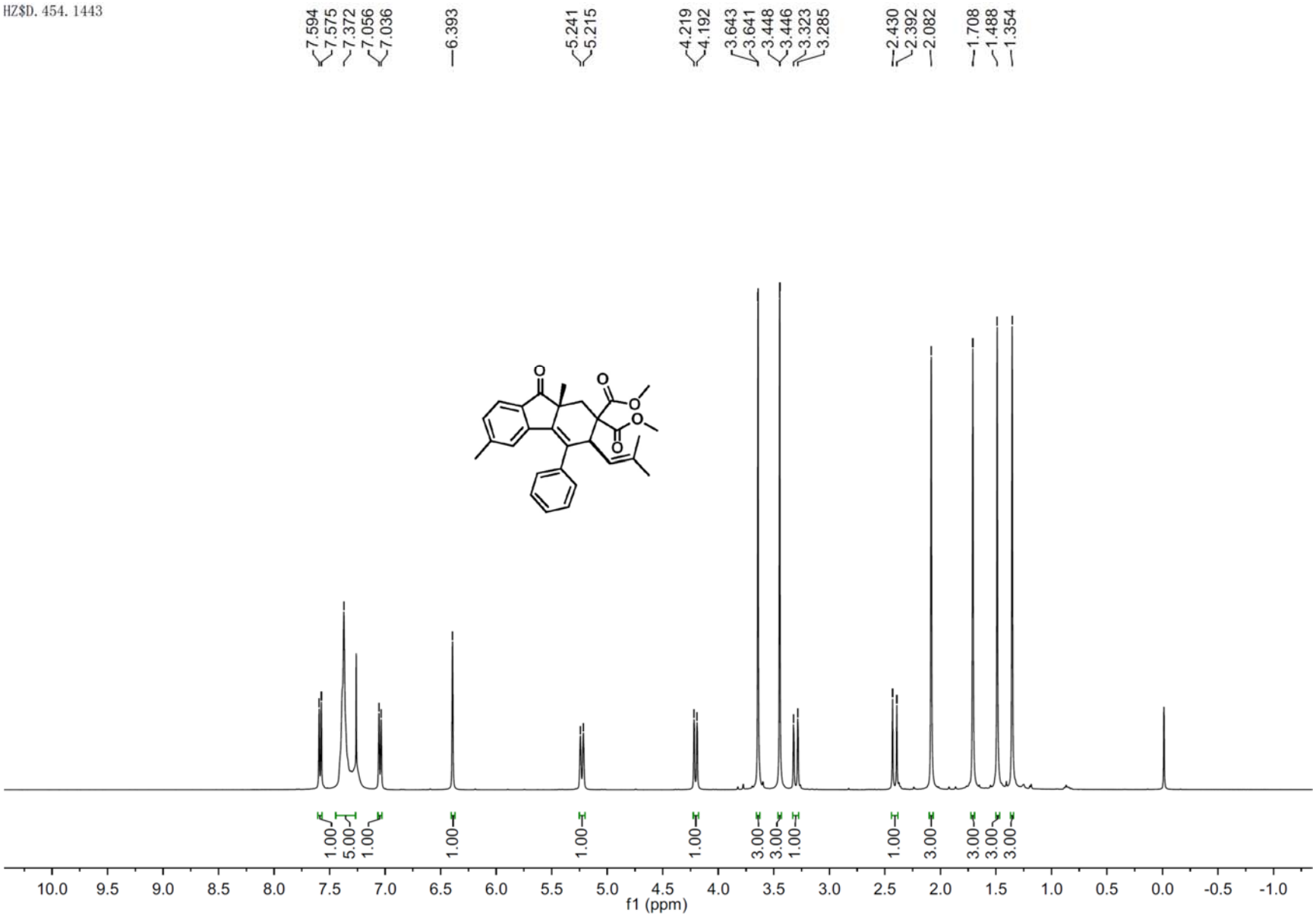
^{13}C NMR Spectrum of Compound 3u



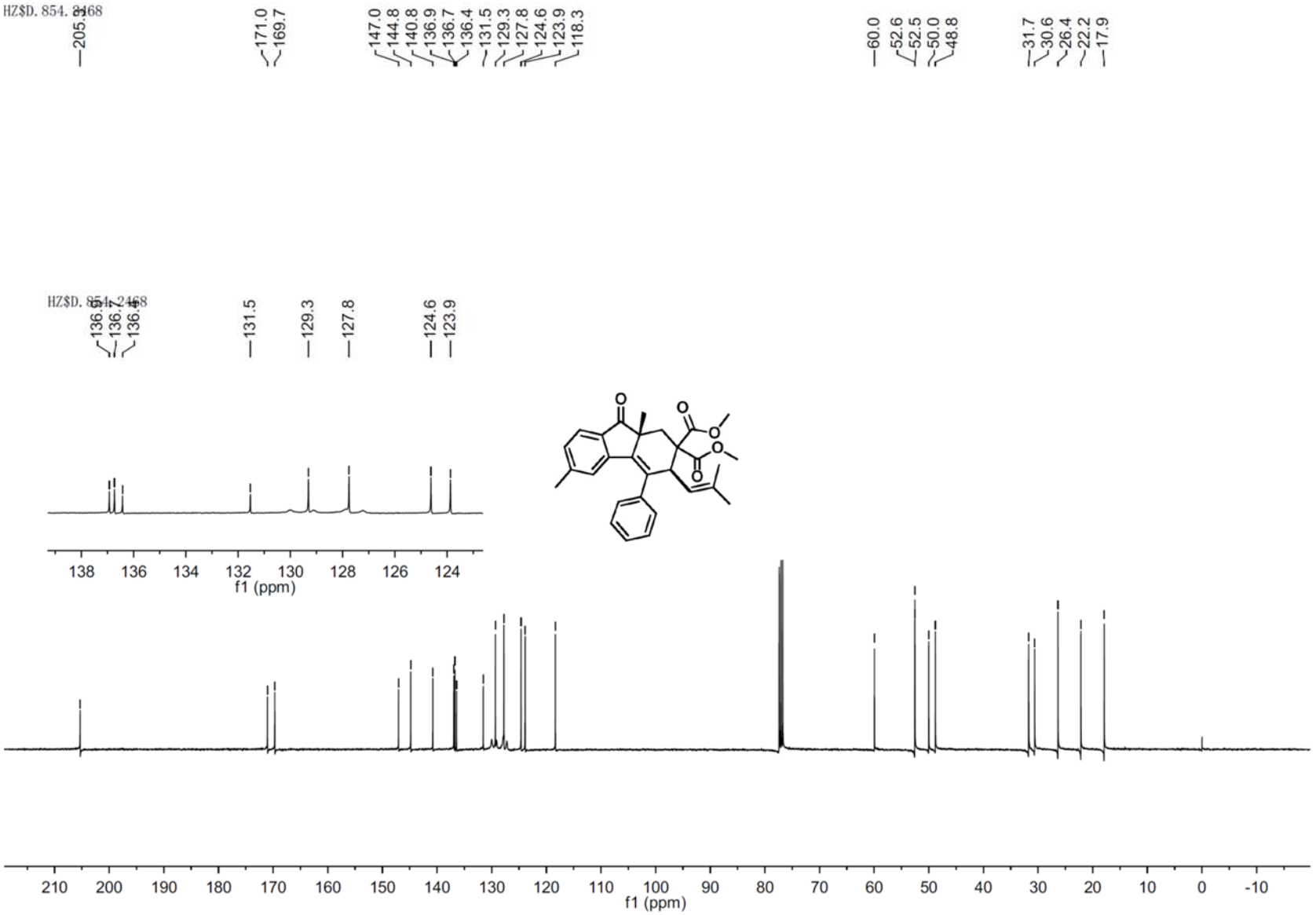
^{1}H NMR Spectrum of Compound 3v



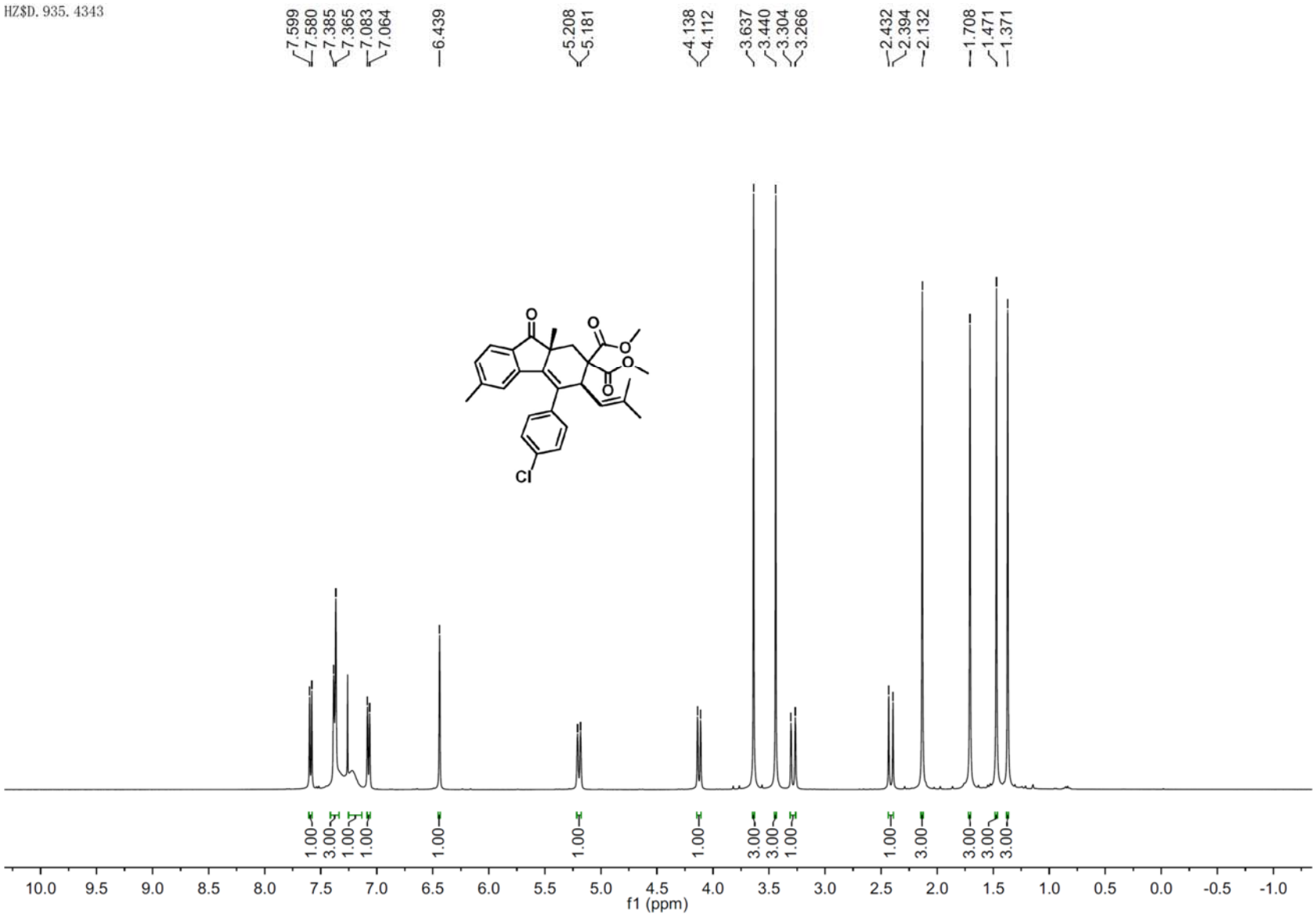
^{13}C NMR Spectrum of Compound 3v



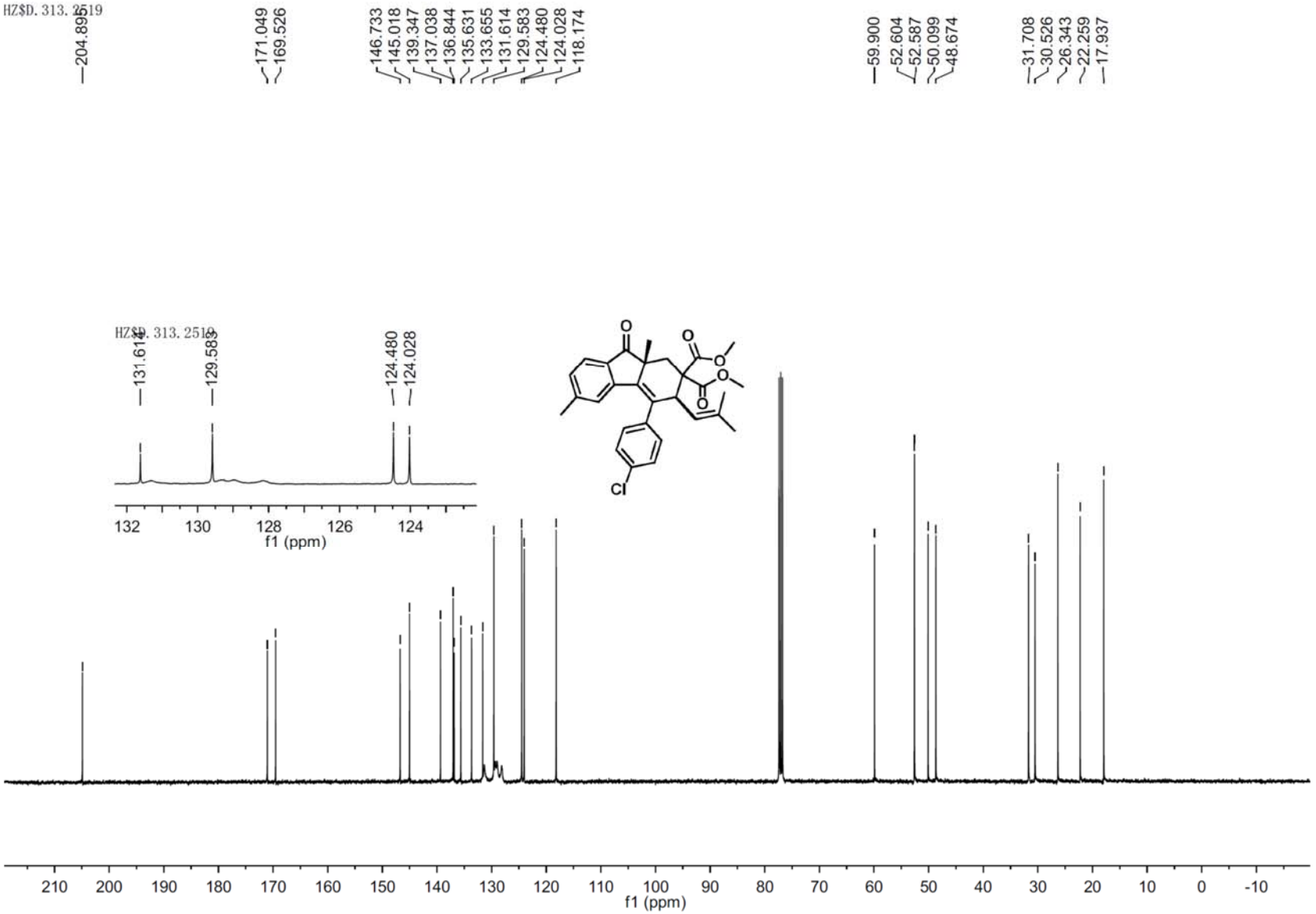
^{1}H NMR Spectrum of Compound 3w



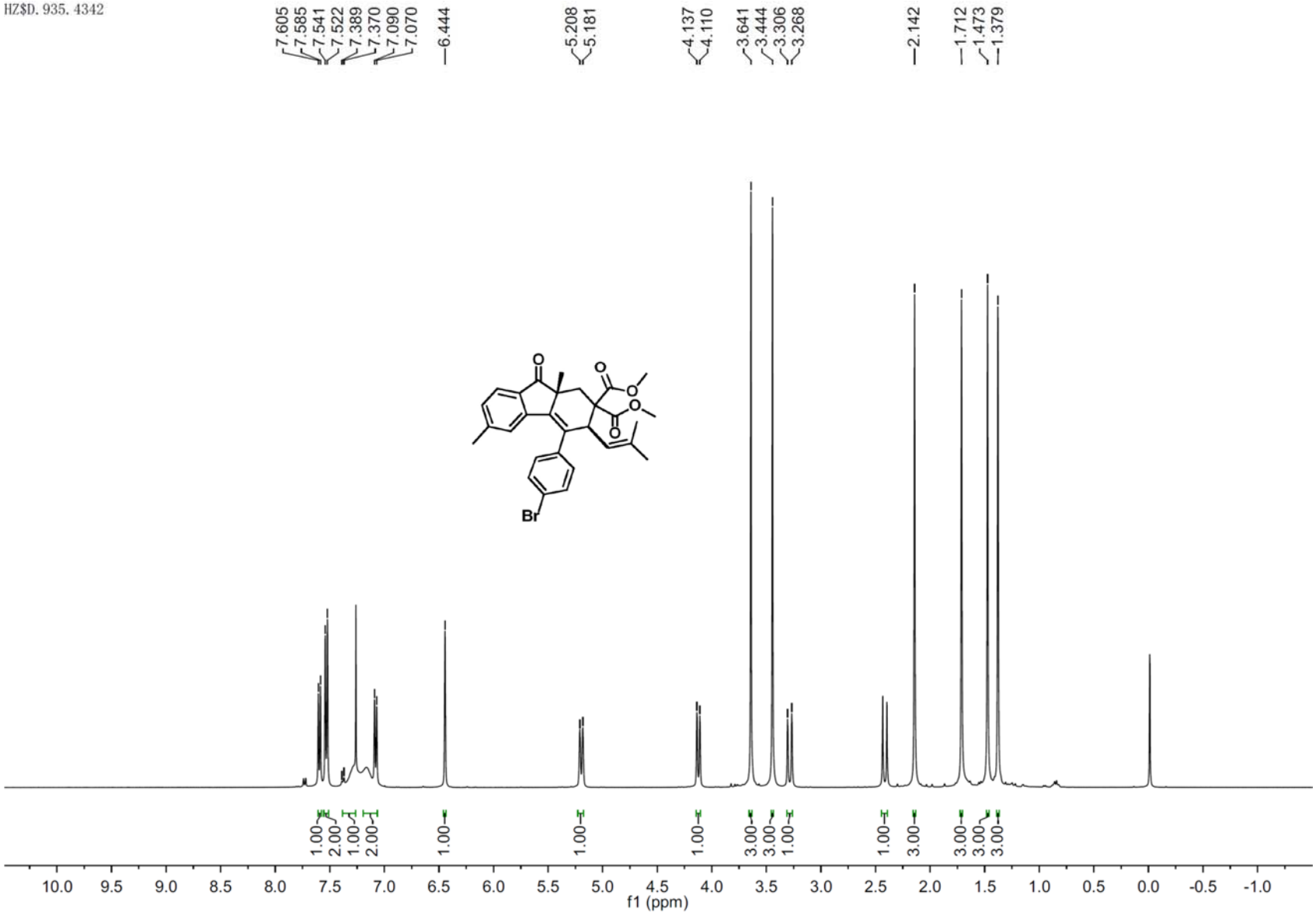
^{13}C NMR Spectrum of Compound 3w



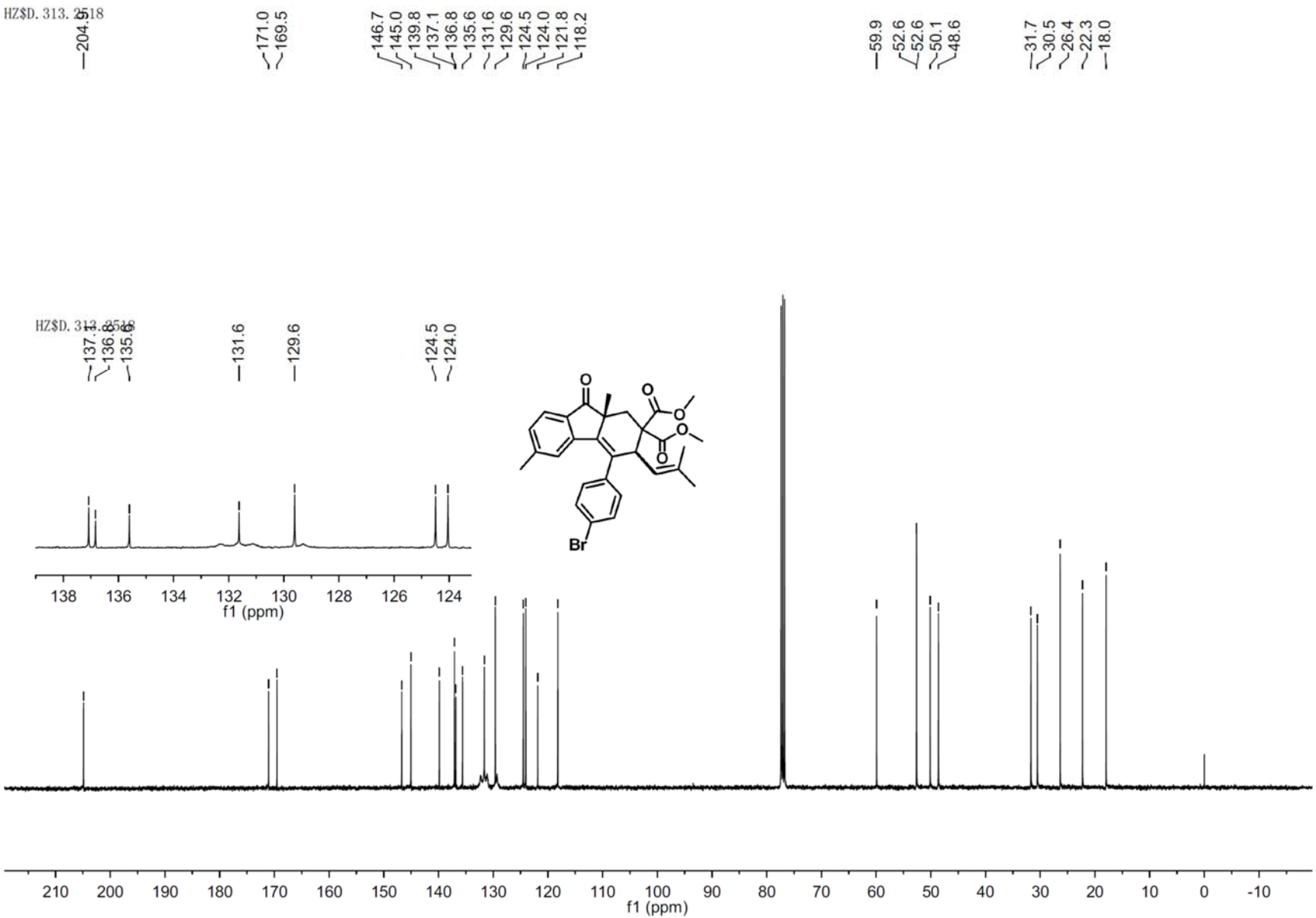
^{1}H NMR Spectrum of Compound 3x



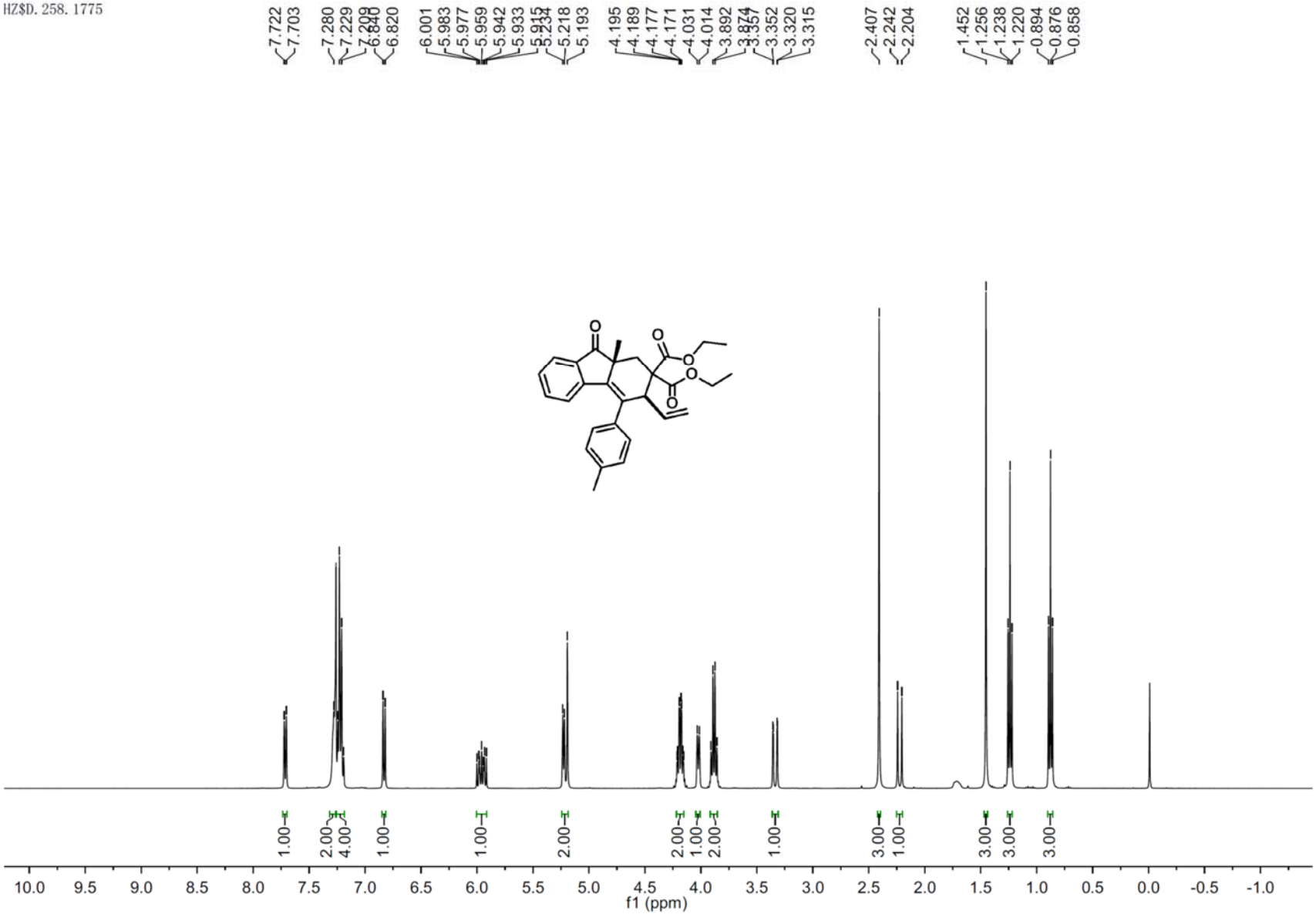
^{13}C NMR Spectrum of Compound 3x



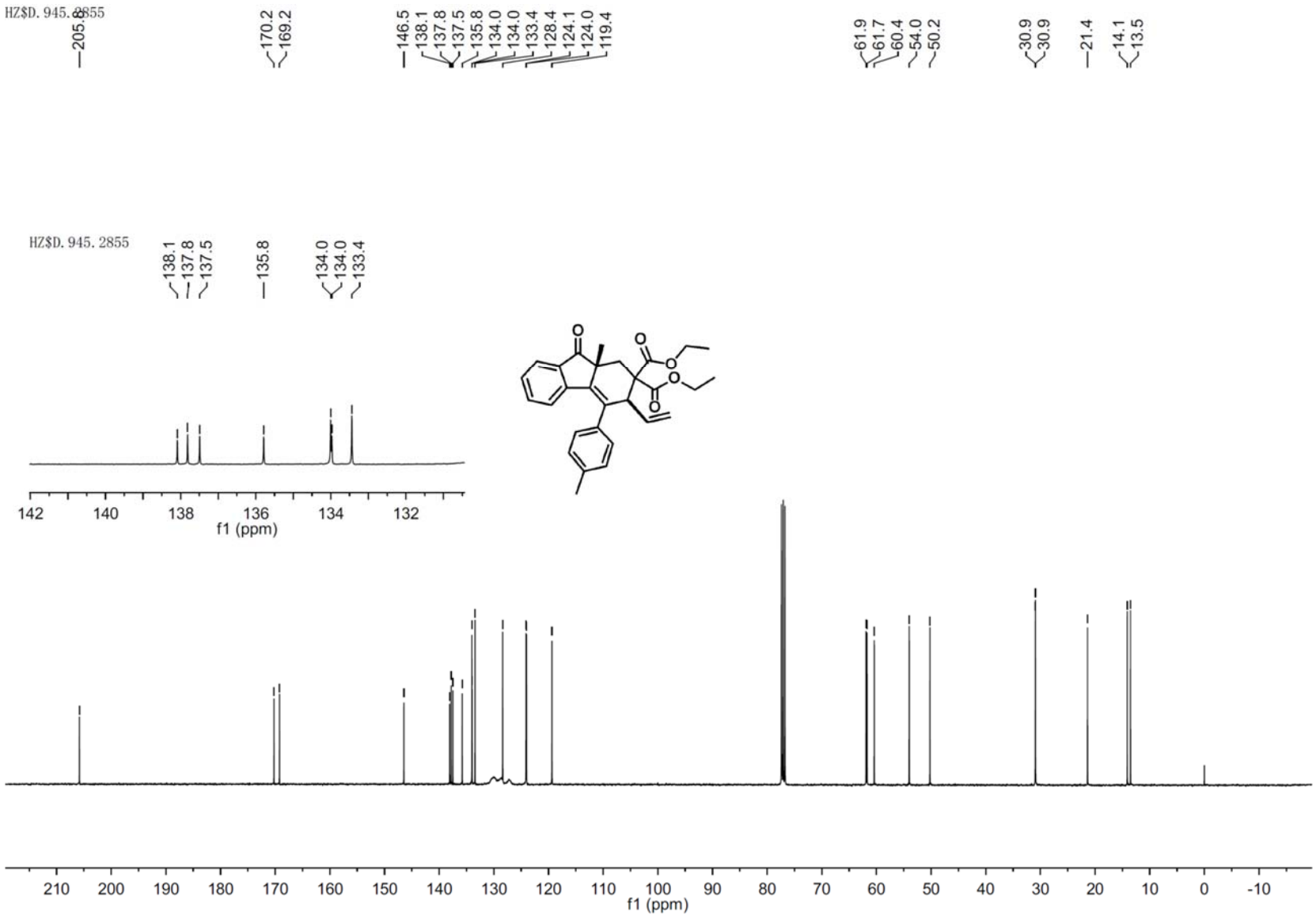
^{1}H NMR Spectrum of Compound 3y



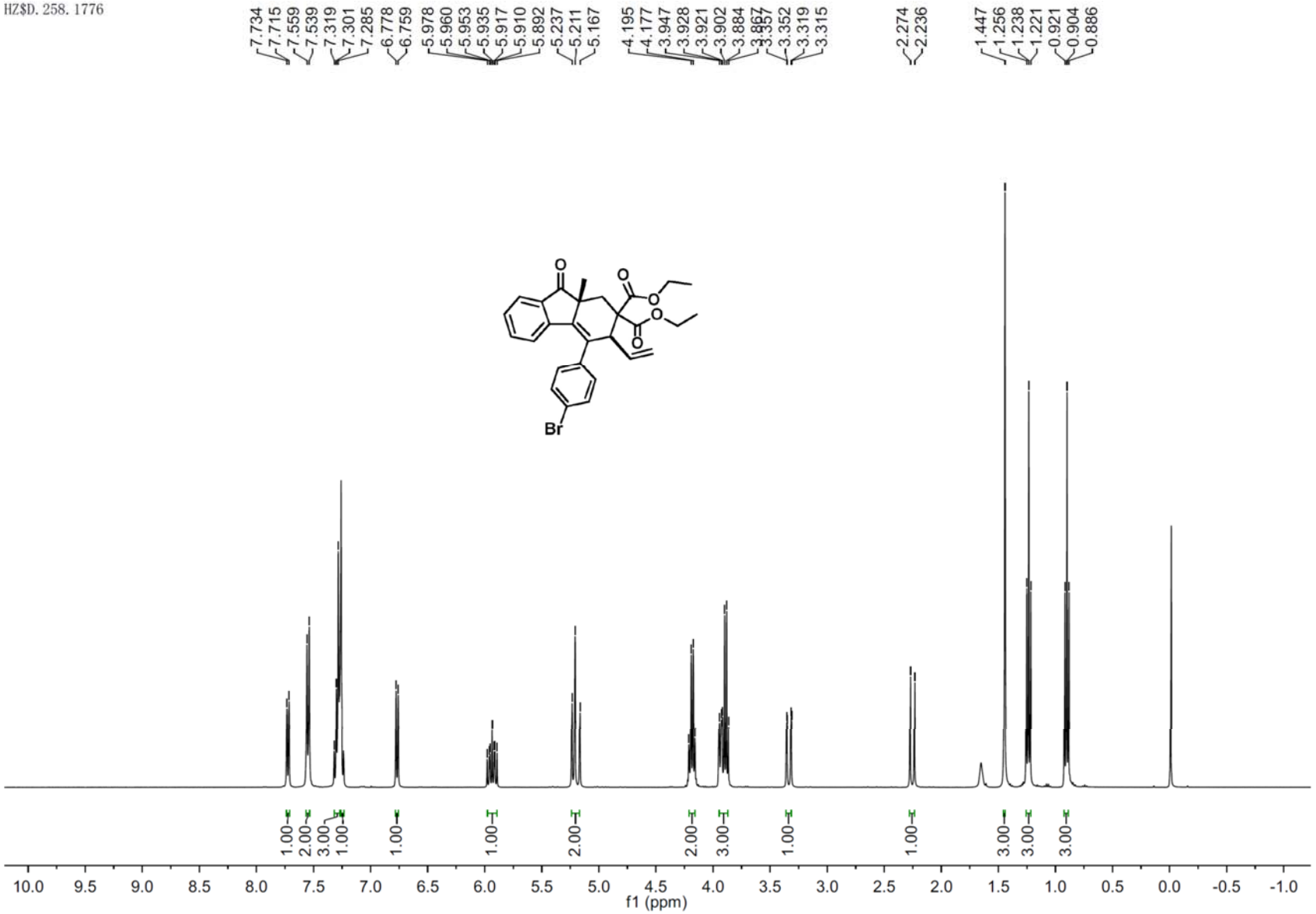
^{13}C NMR Spectrum of Compound 3y



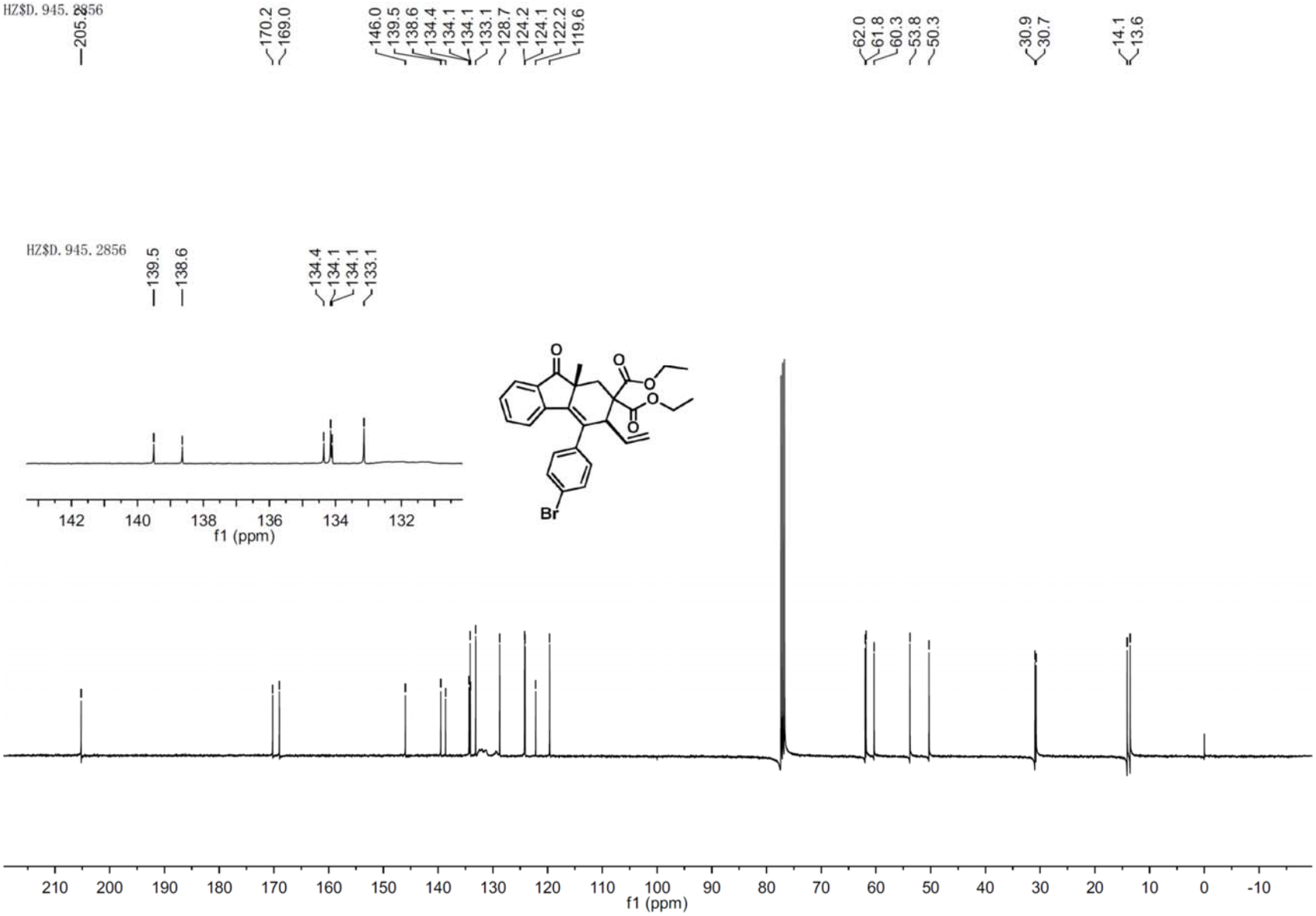
^{1}H NMR Spectrum of Compound 3aa



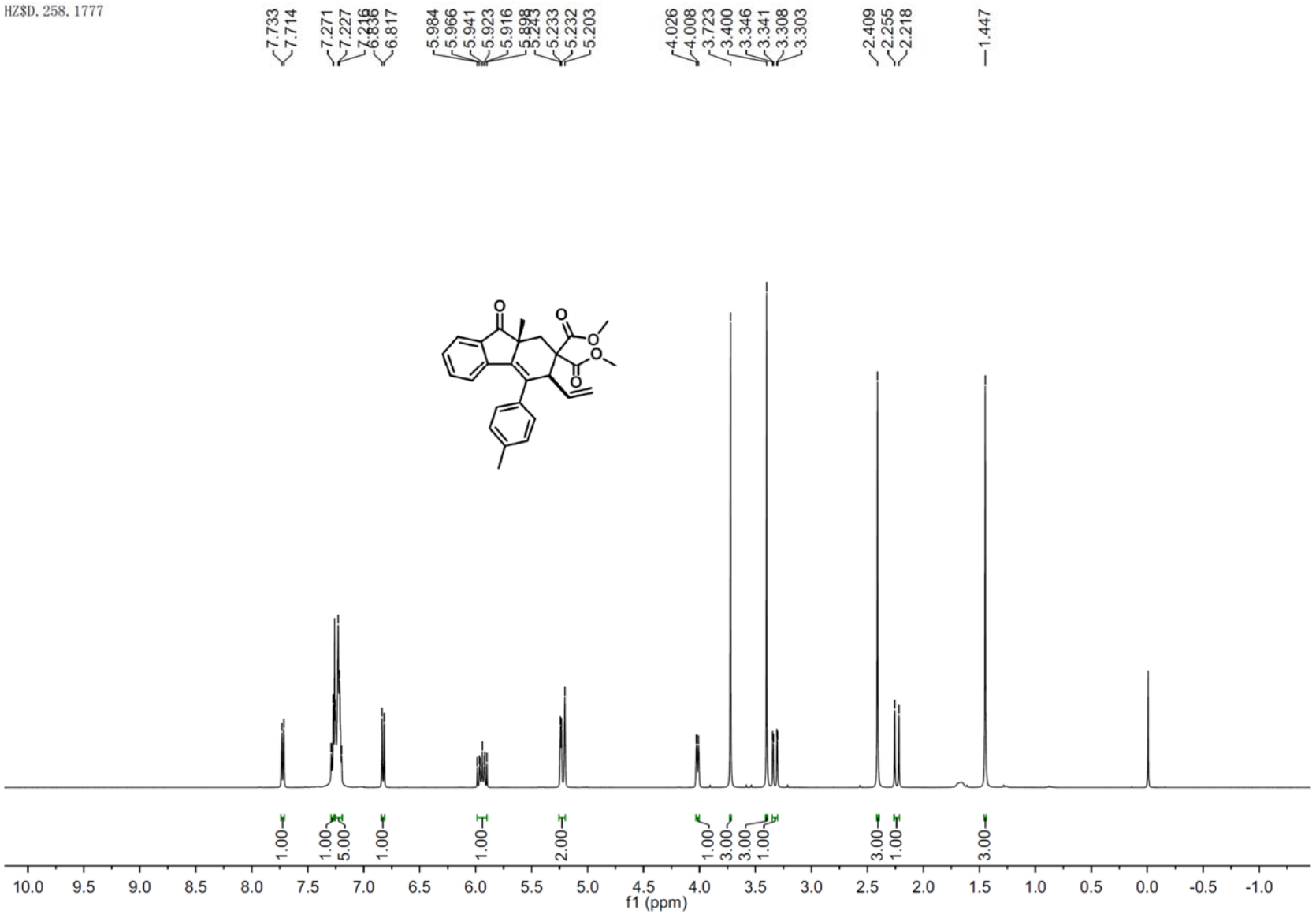
^{13}C NMR Spectrum of Compound 3aa



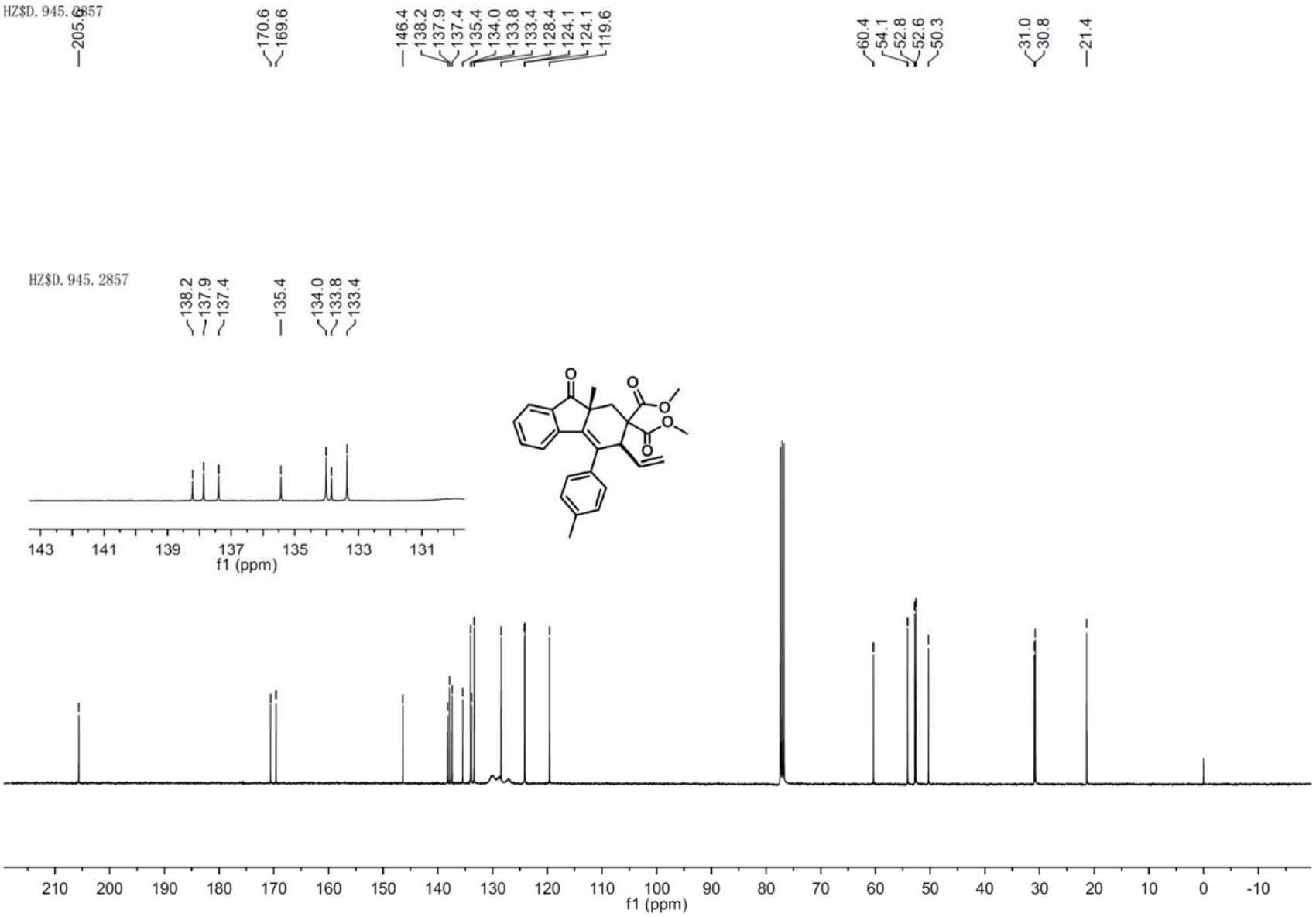
^{1}H NMR Spectrum of Compound 3bb



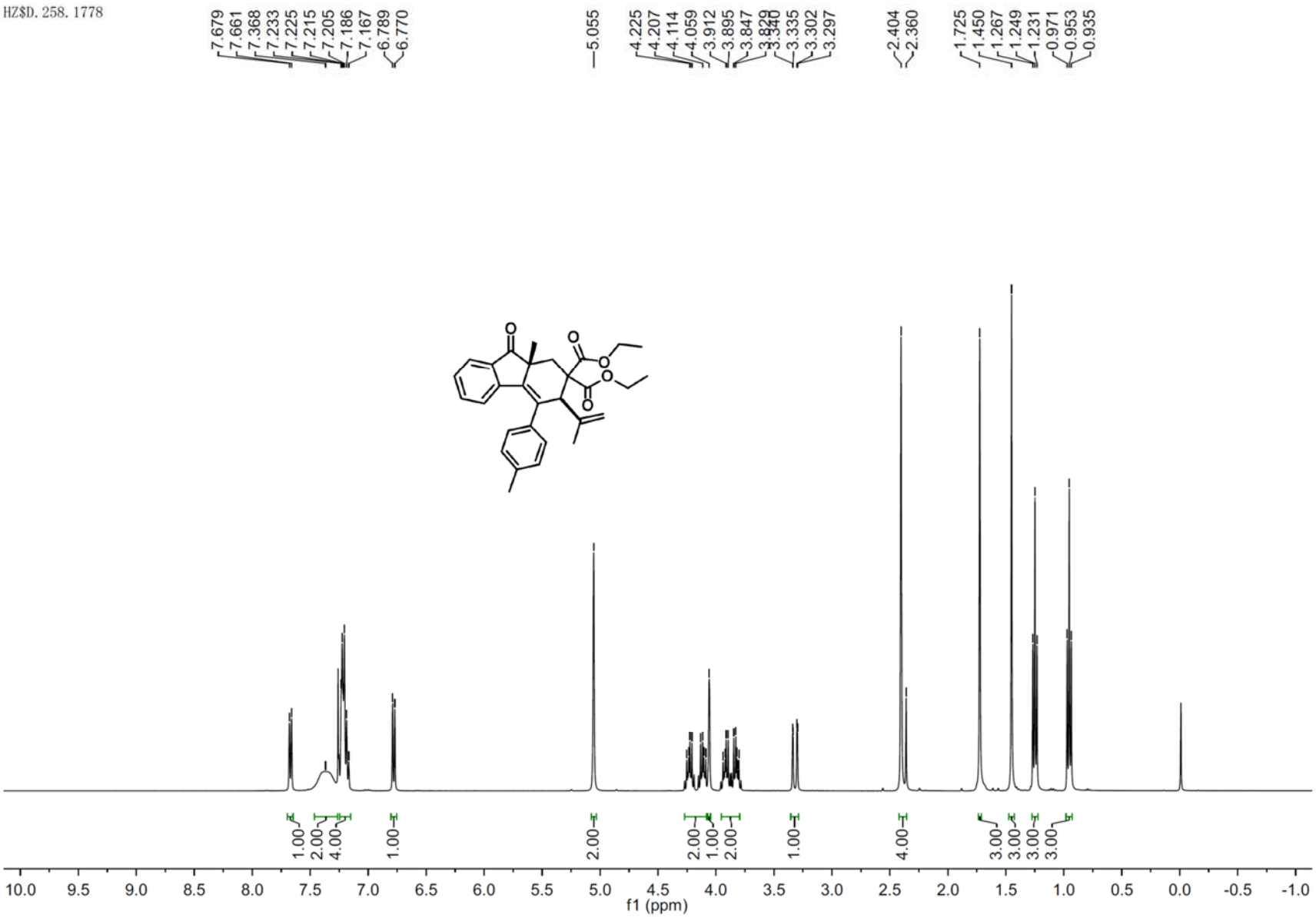
^{13}C NMR Spectrum of Compound 3bb



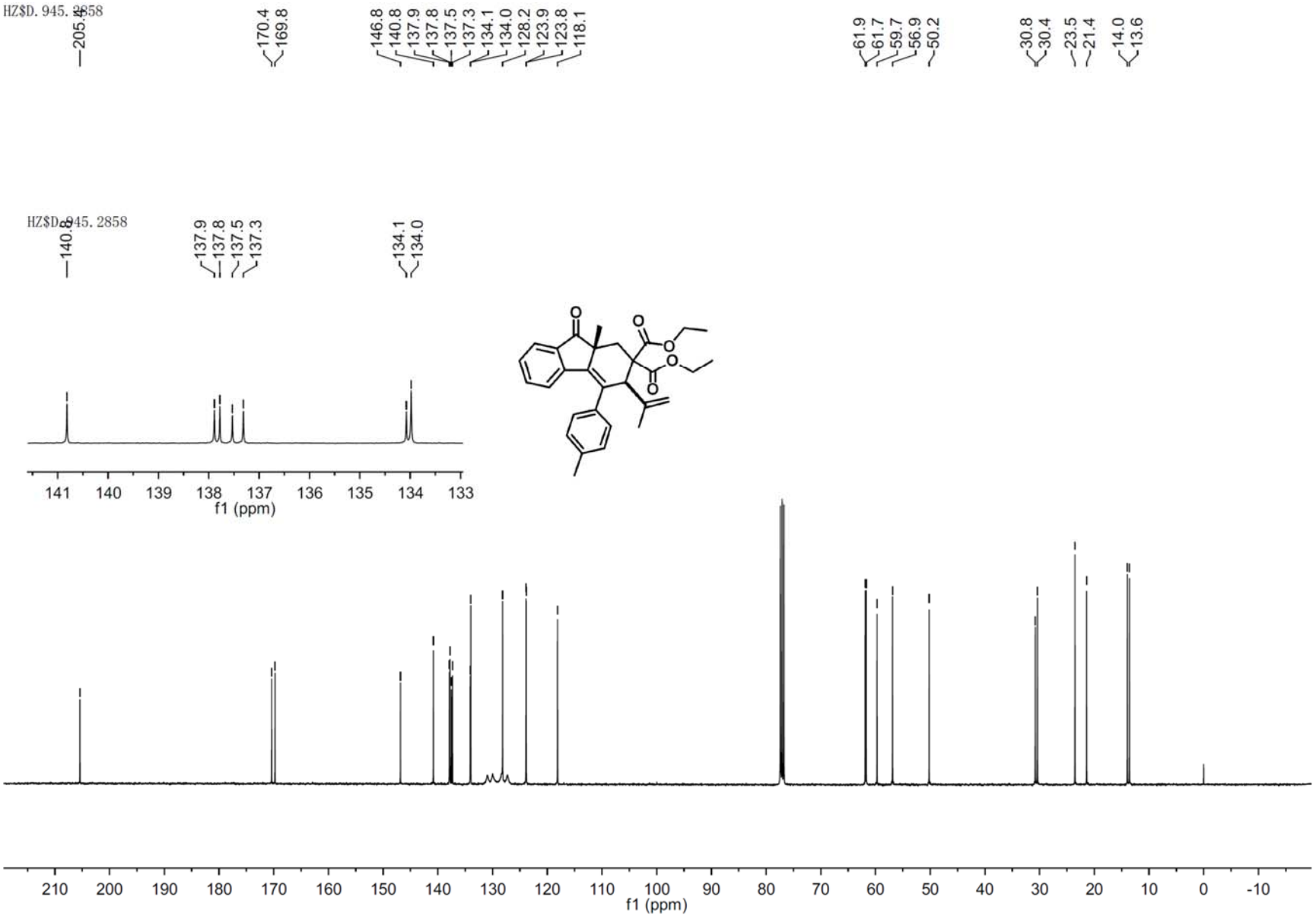
^{1}H NMR Spectrum of Compound 3cc



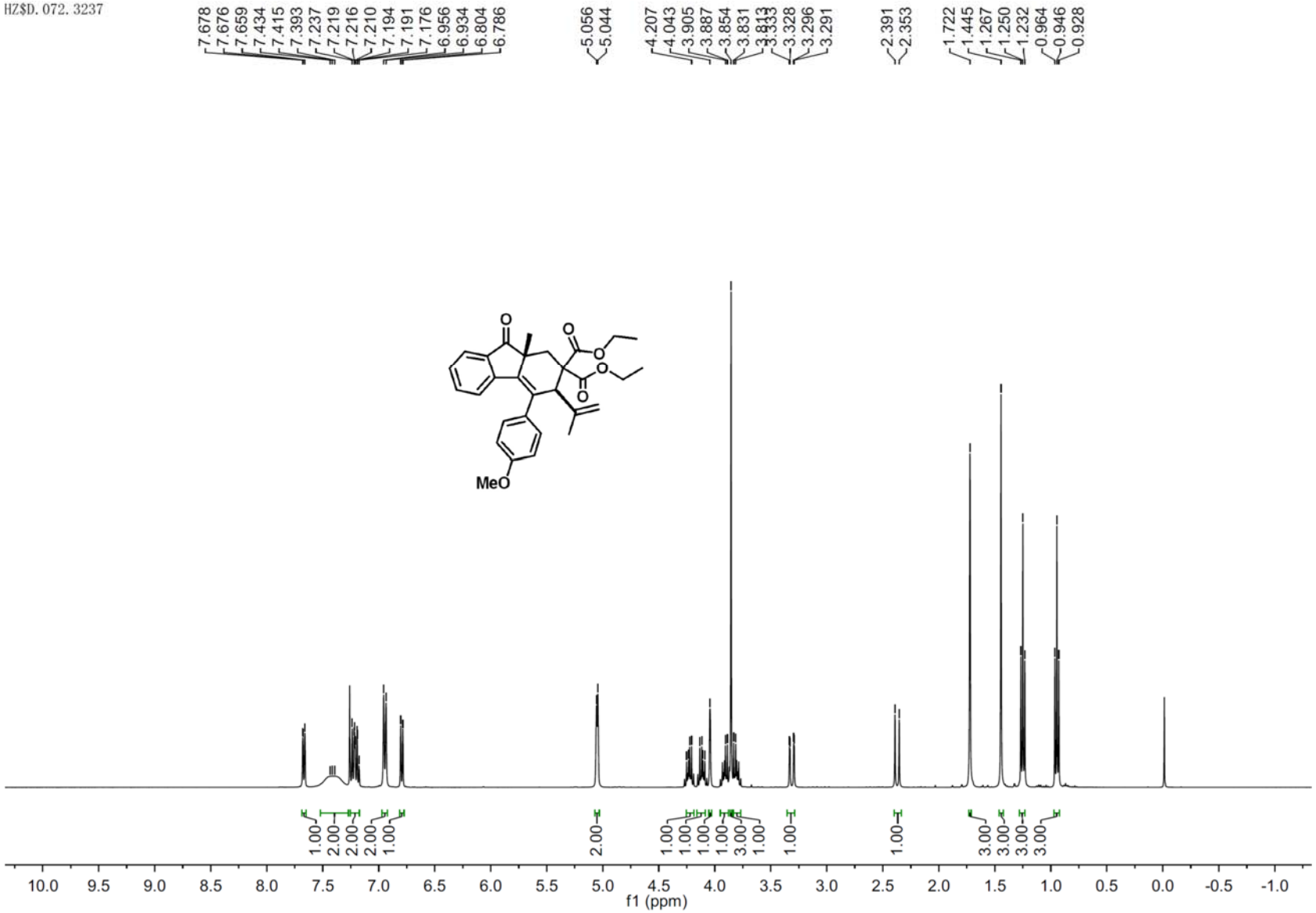
^{13}C NMR Spectrum of Compound 3cc



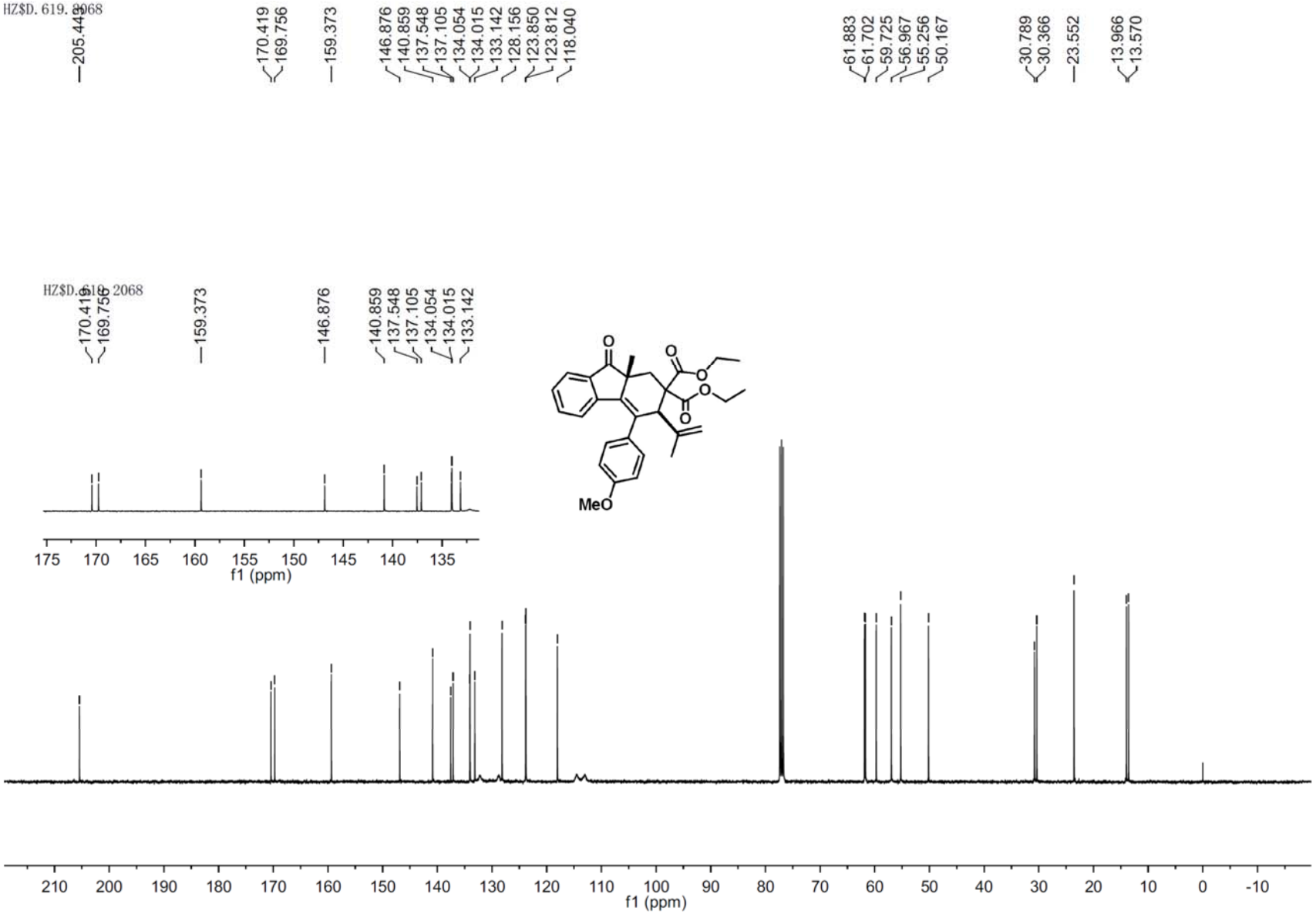
^{1}H NMR Spectrum of Compound 3dd



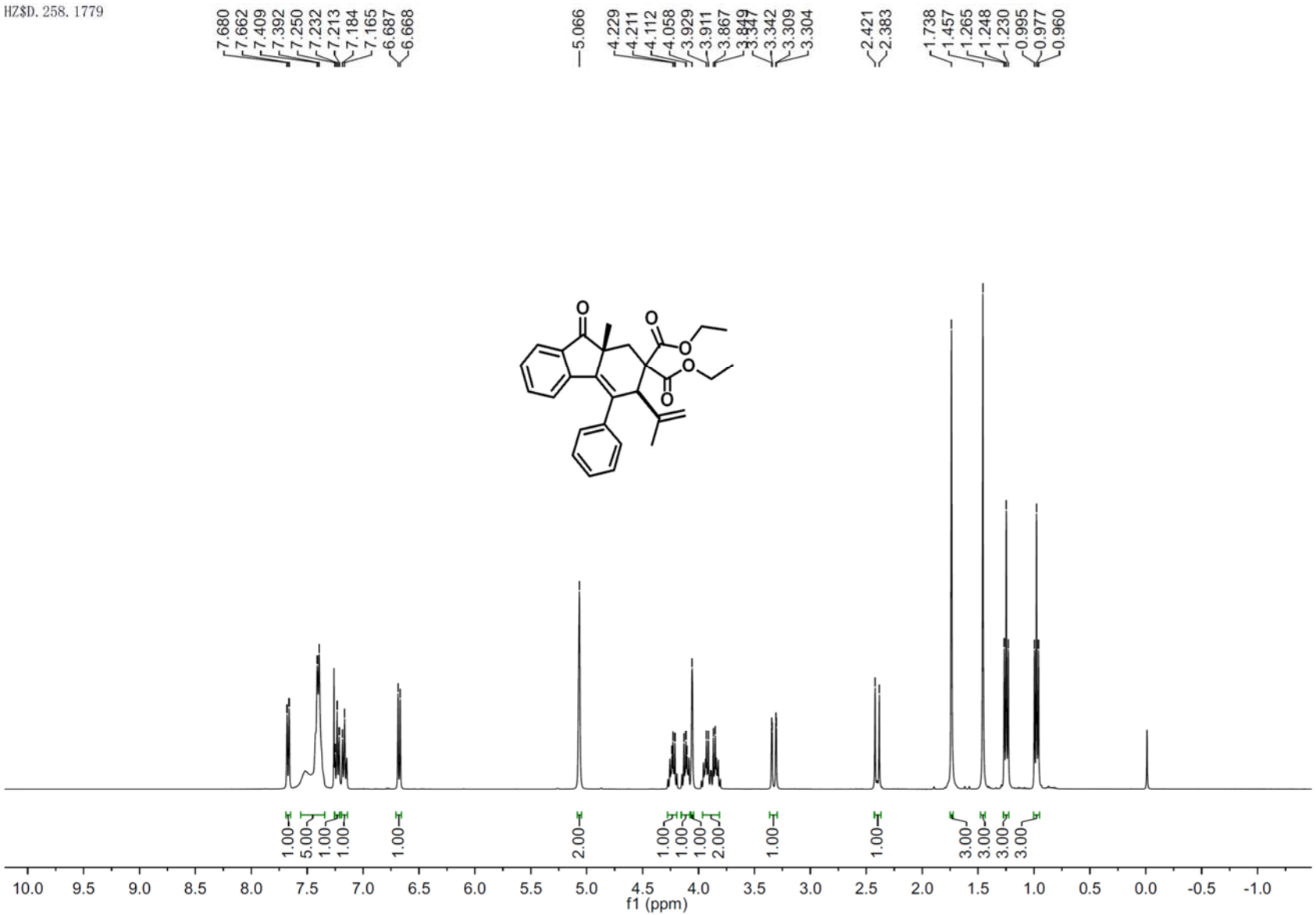
^{13}C NMR Spectrum of Compound 3dd



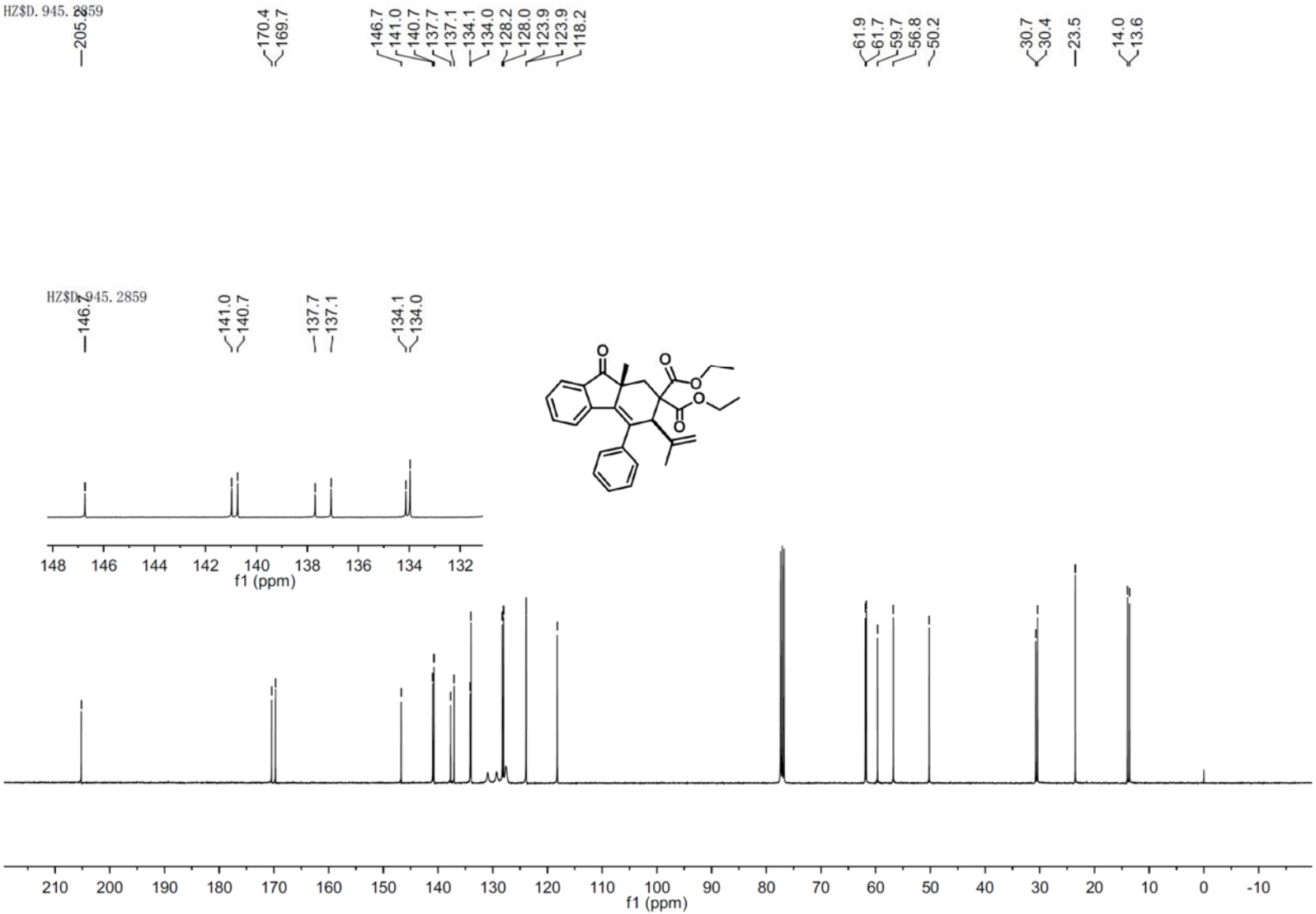
^{1}H NMR Spectrum of Compound 3ee



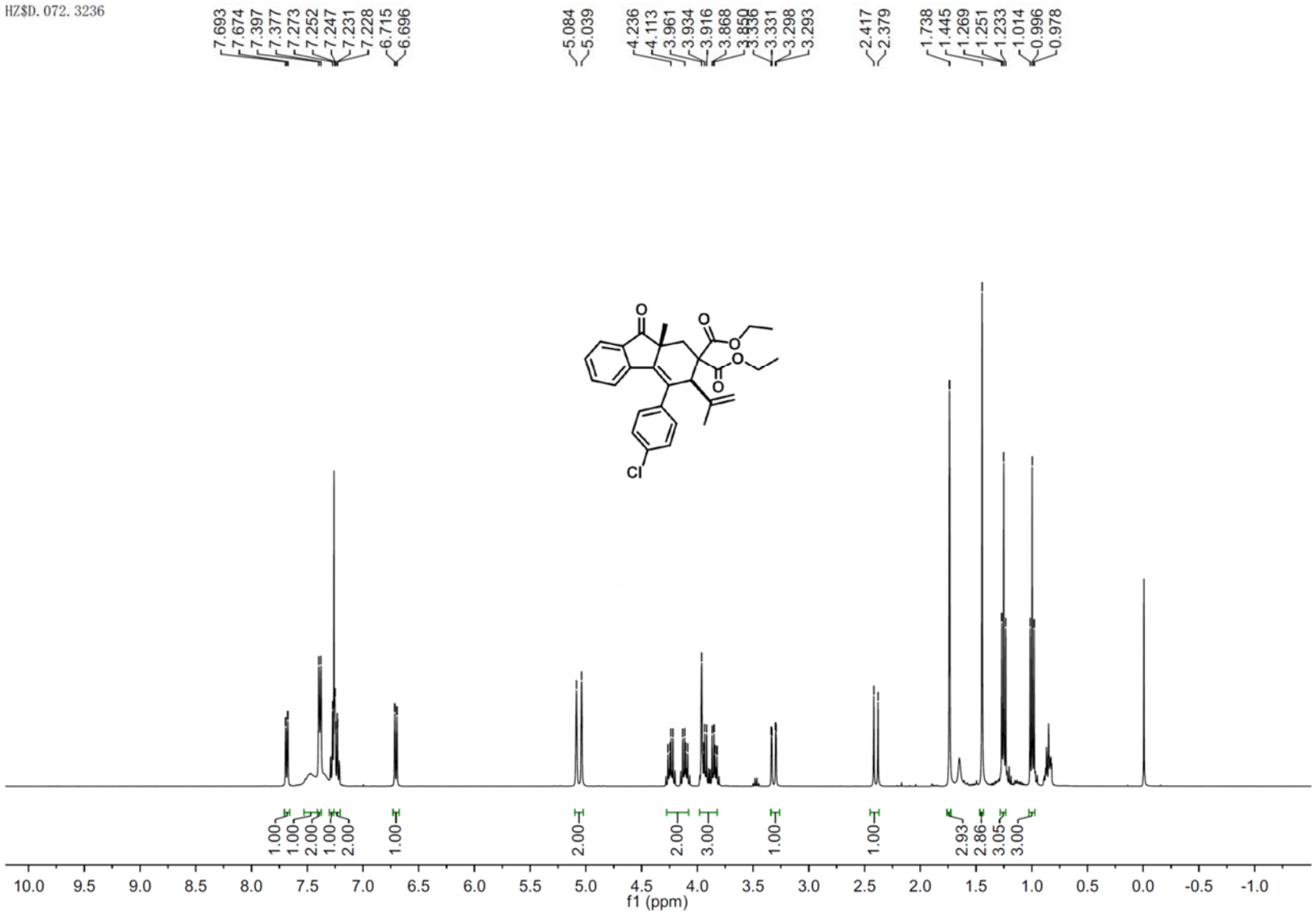
^{13}C NMR Spectrum of Compound 3ee



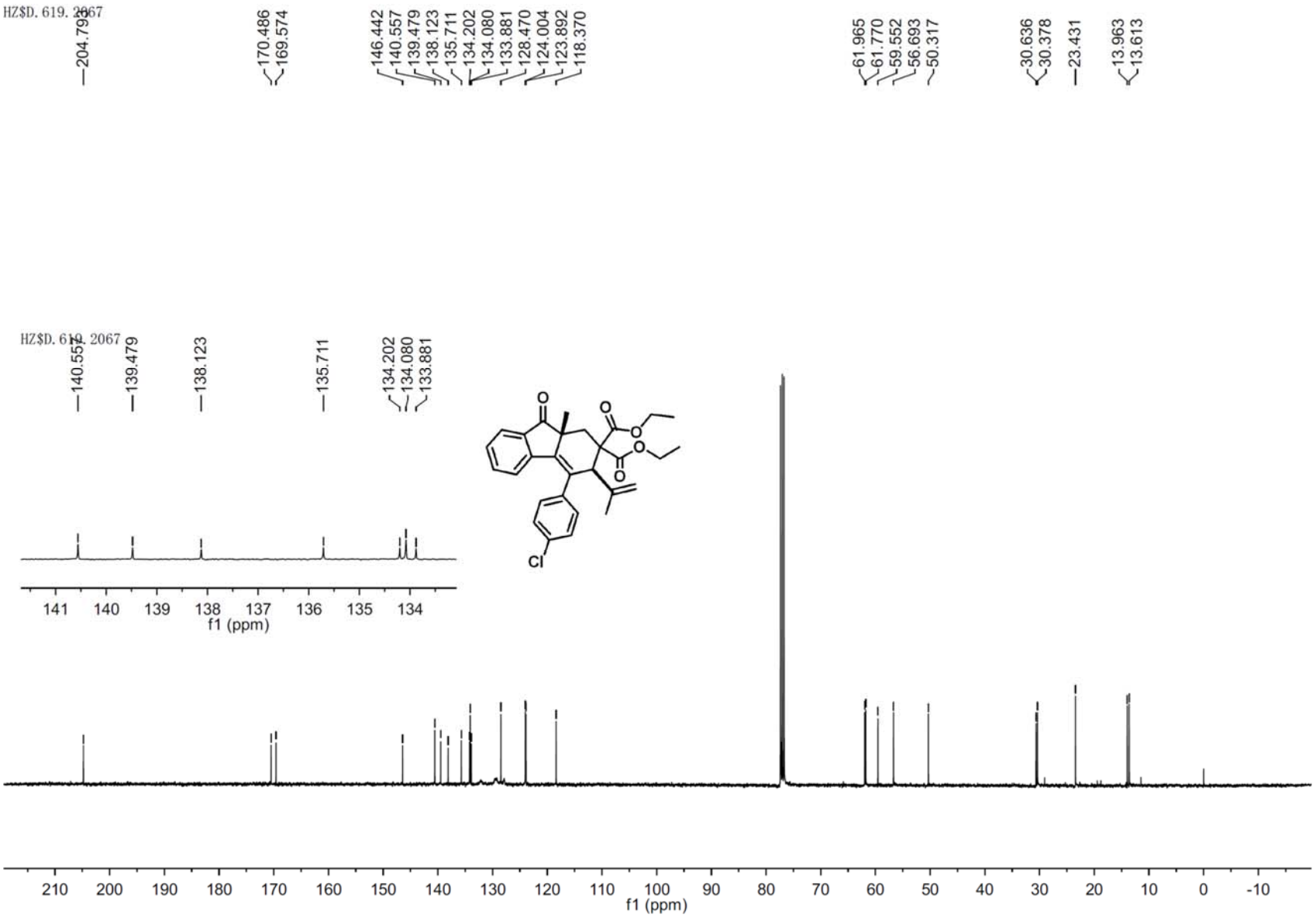
^{1}H NMR Spectrum of Compound 3ff



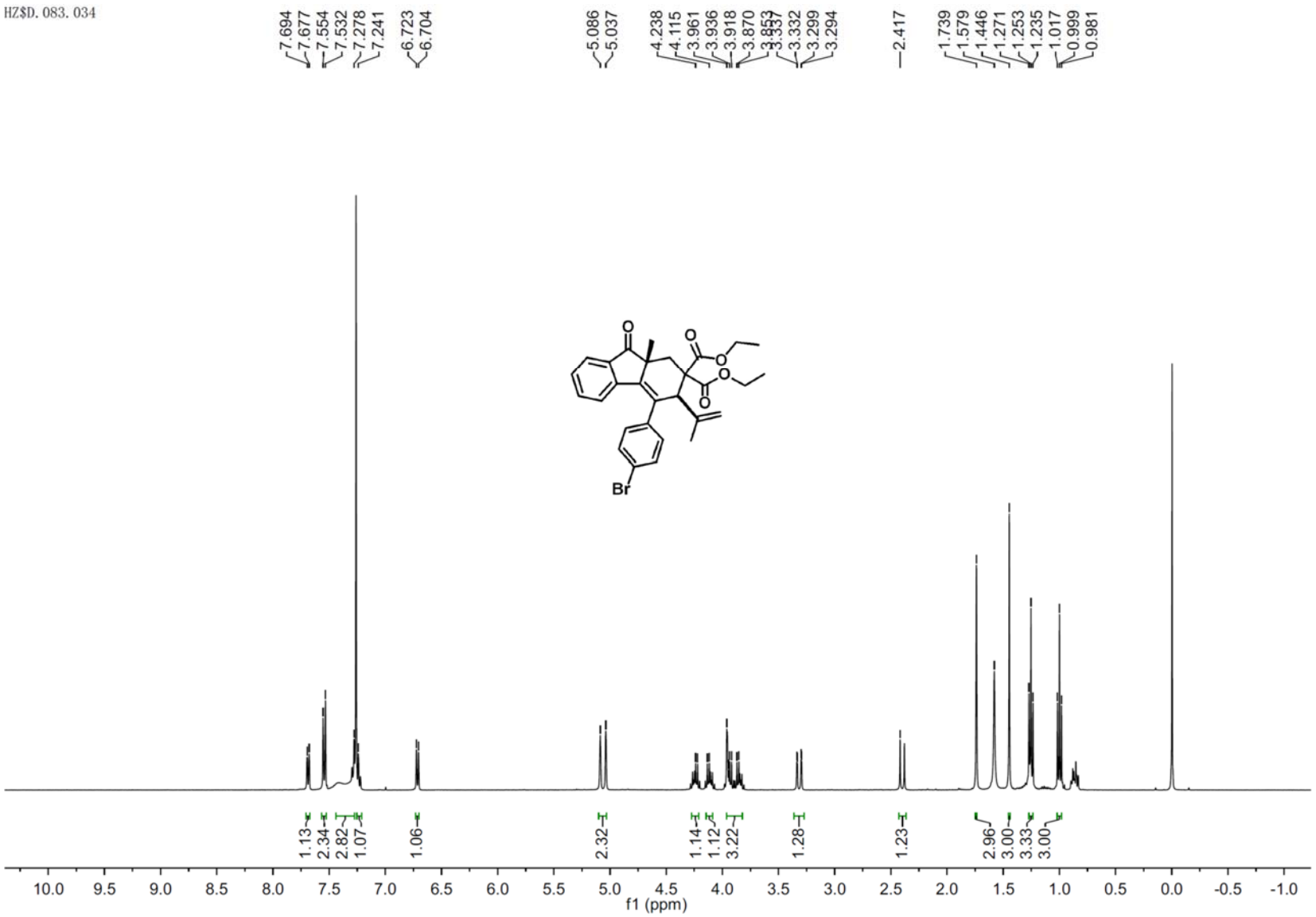
^{13}C NMR Spectrum of Compound 3ff



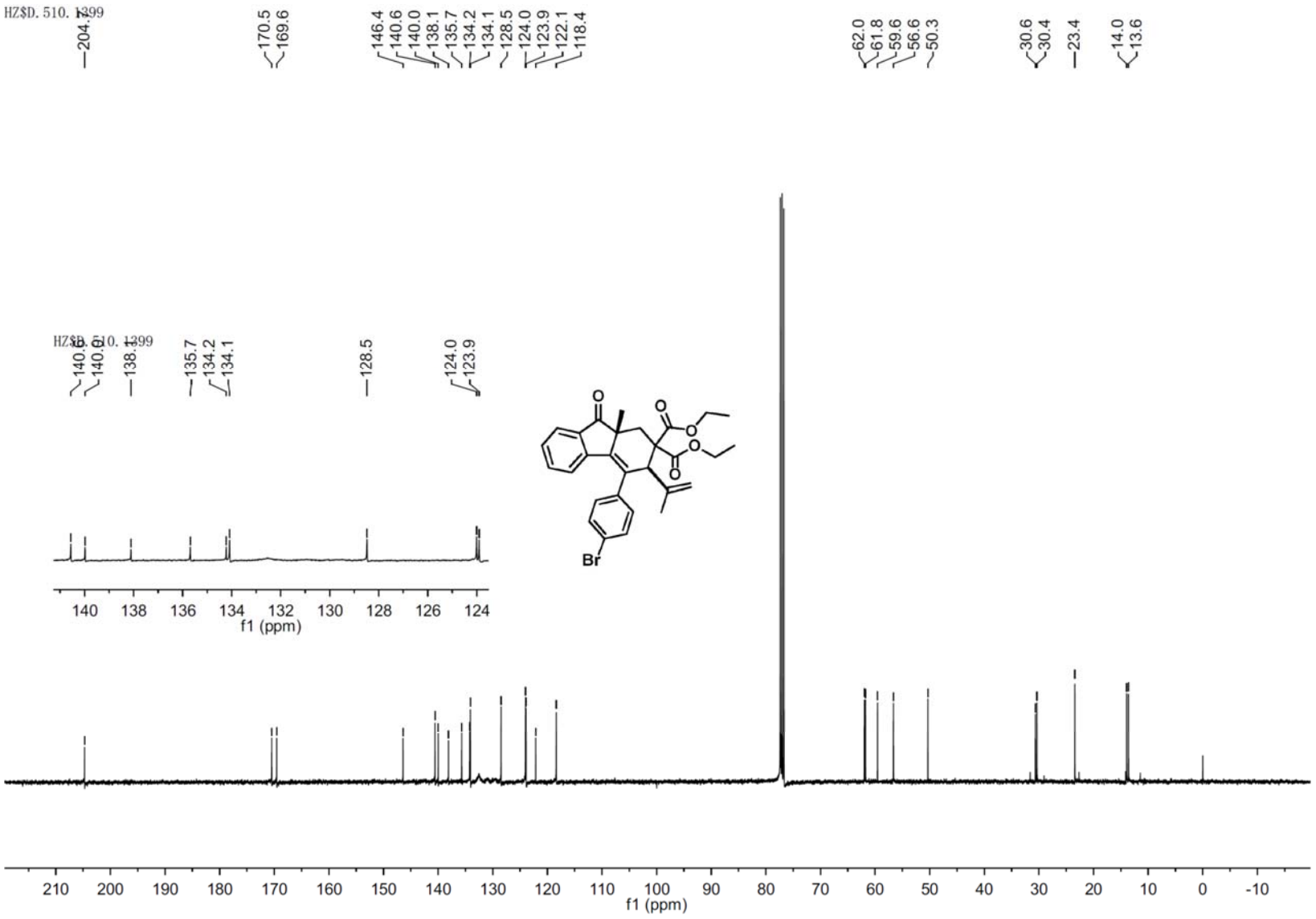
^{1}H NMR Spectrum of Compound 3gg



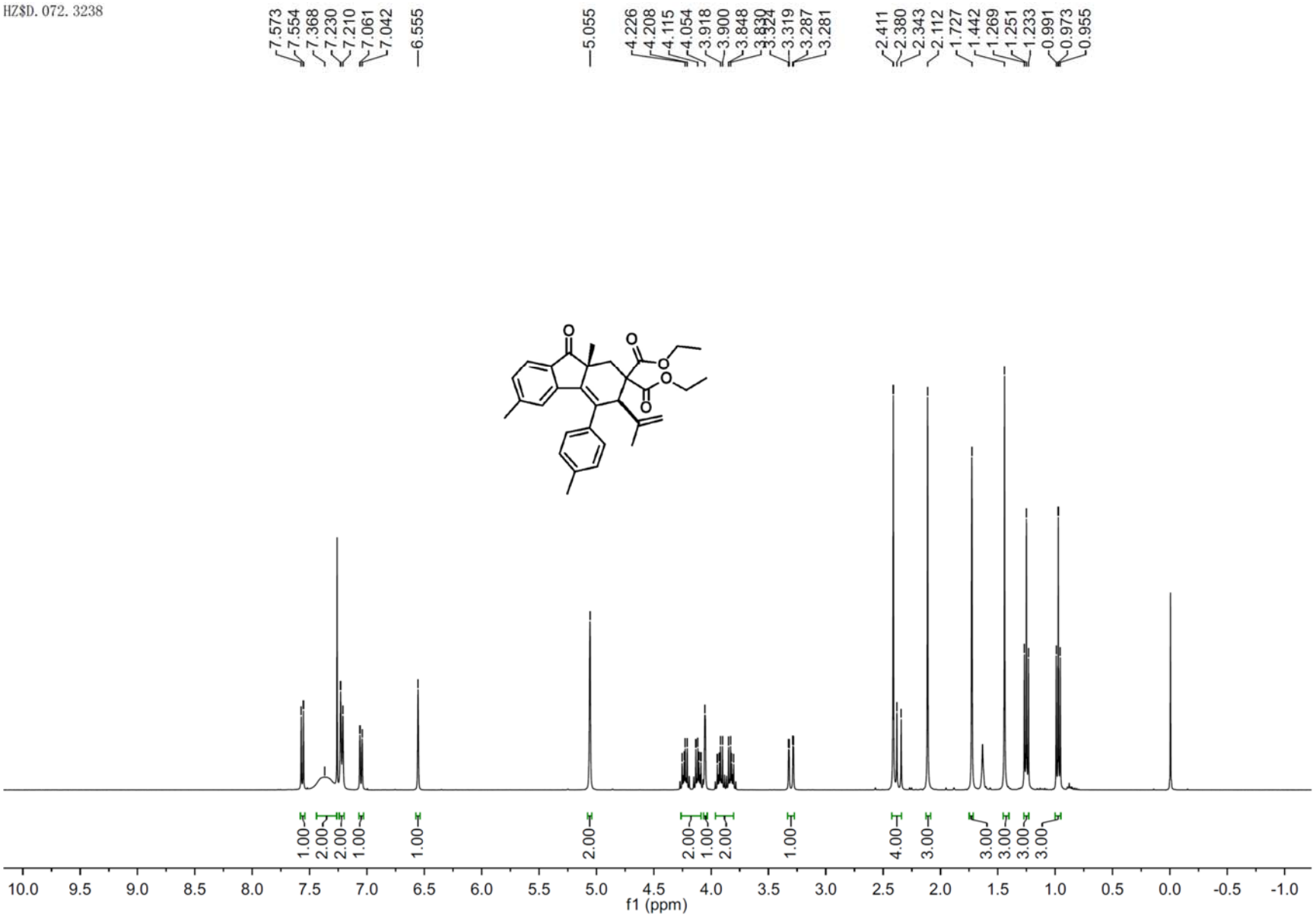
^{13}C NMR Spectrum of Compound 3gg



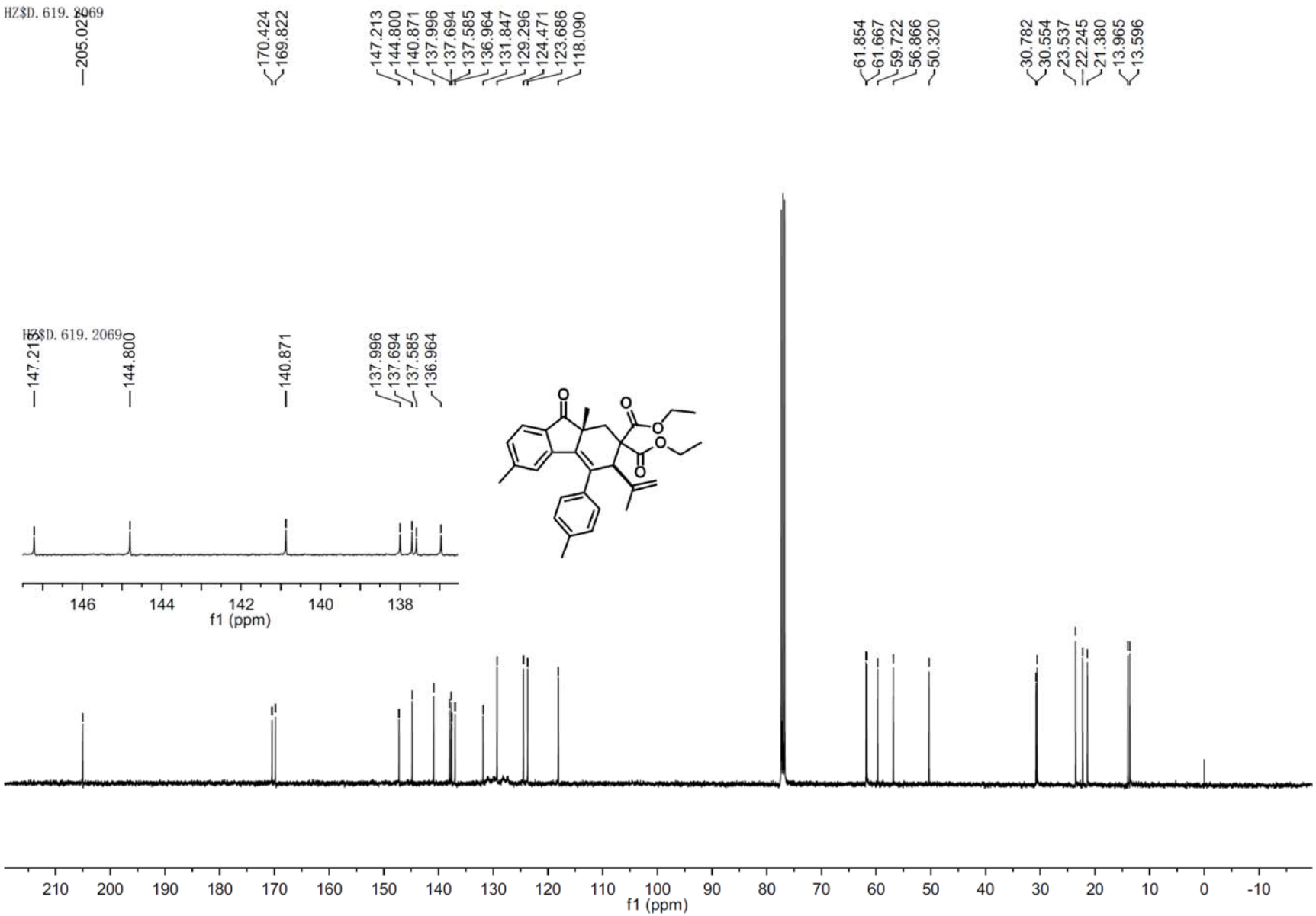
^{1}H NMR Spectrum of Compound 3hh



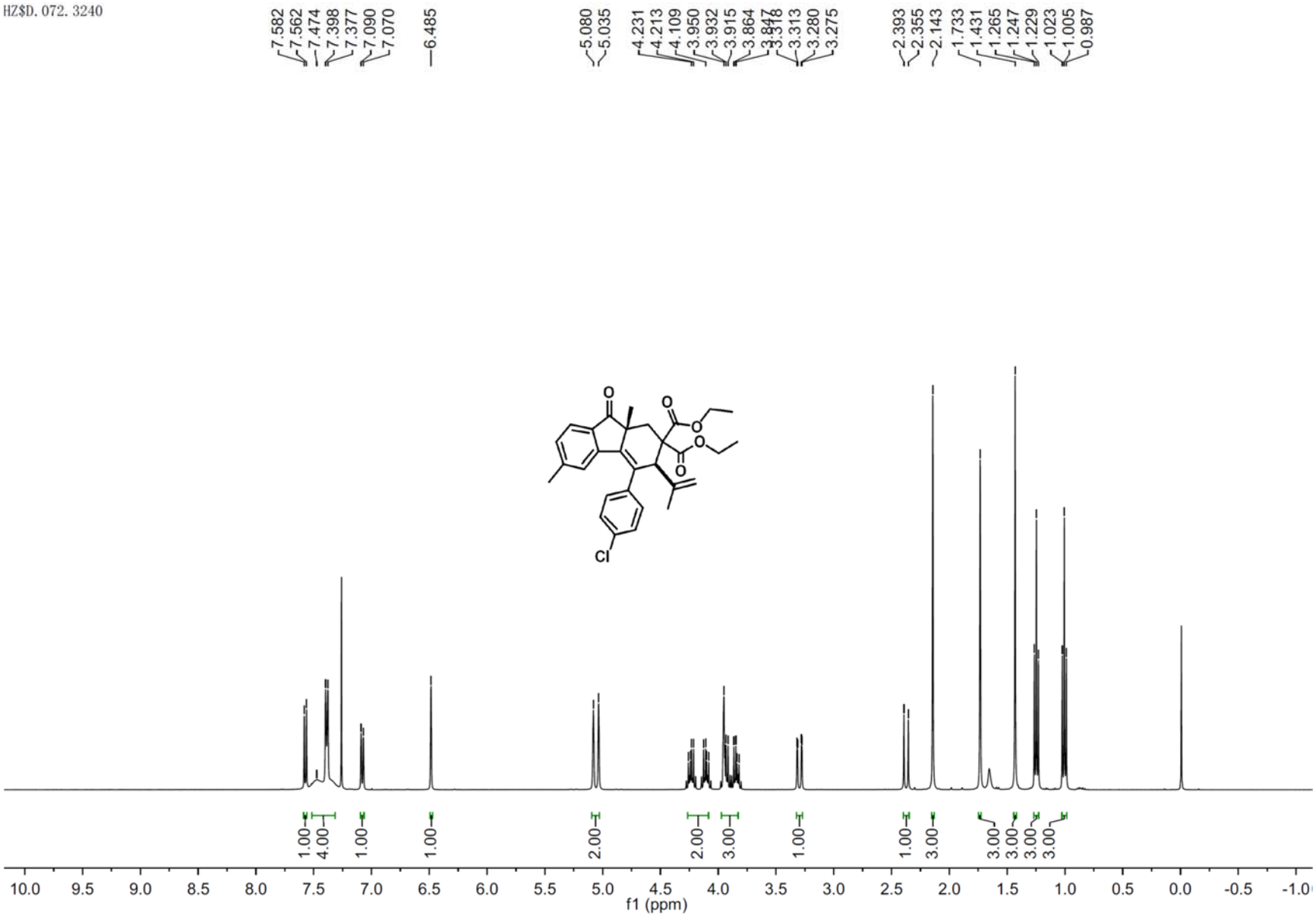
^{13}C NMR Spectrum of Compound 3hh



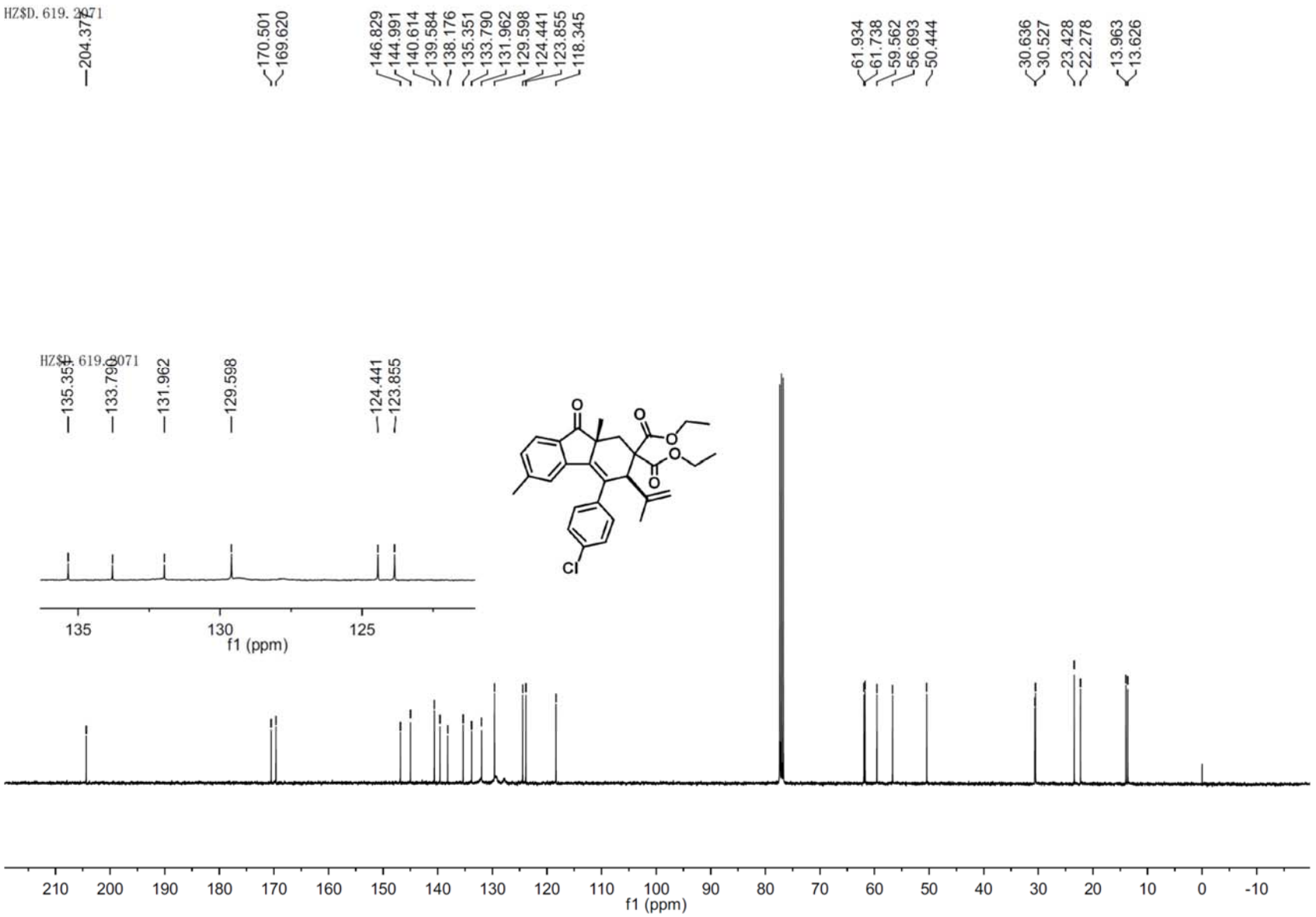
^{1}H NMR Spectrum of Compound 3ii



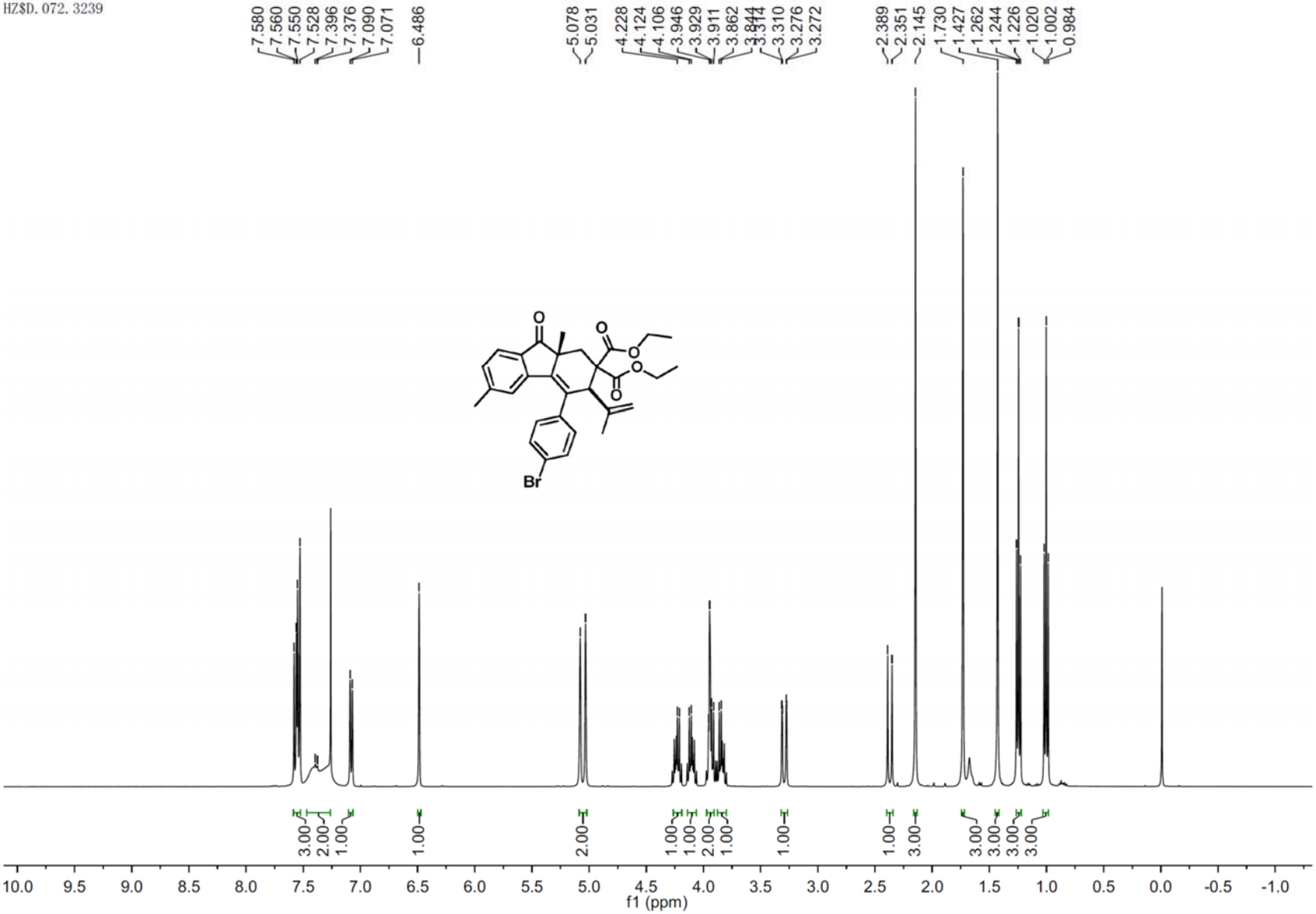
^{13}C NMR Spectrum of Compound 3ii



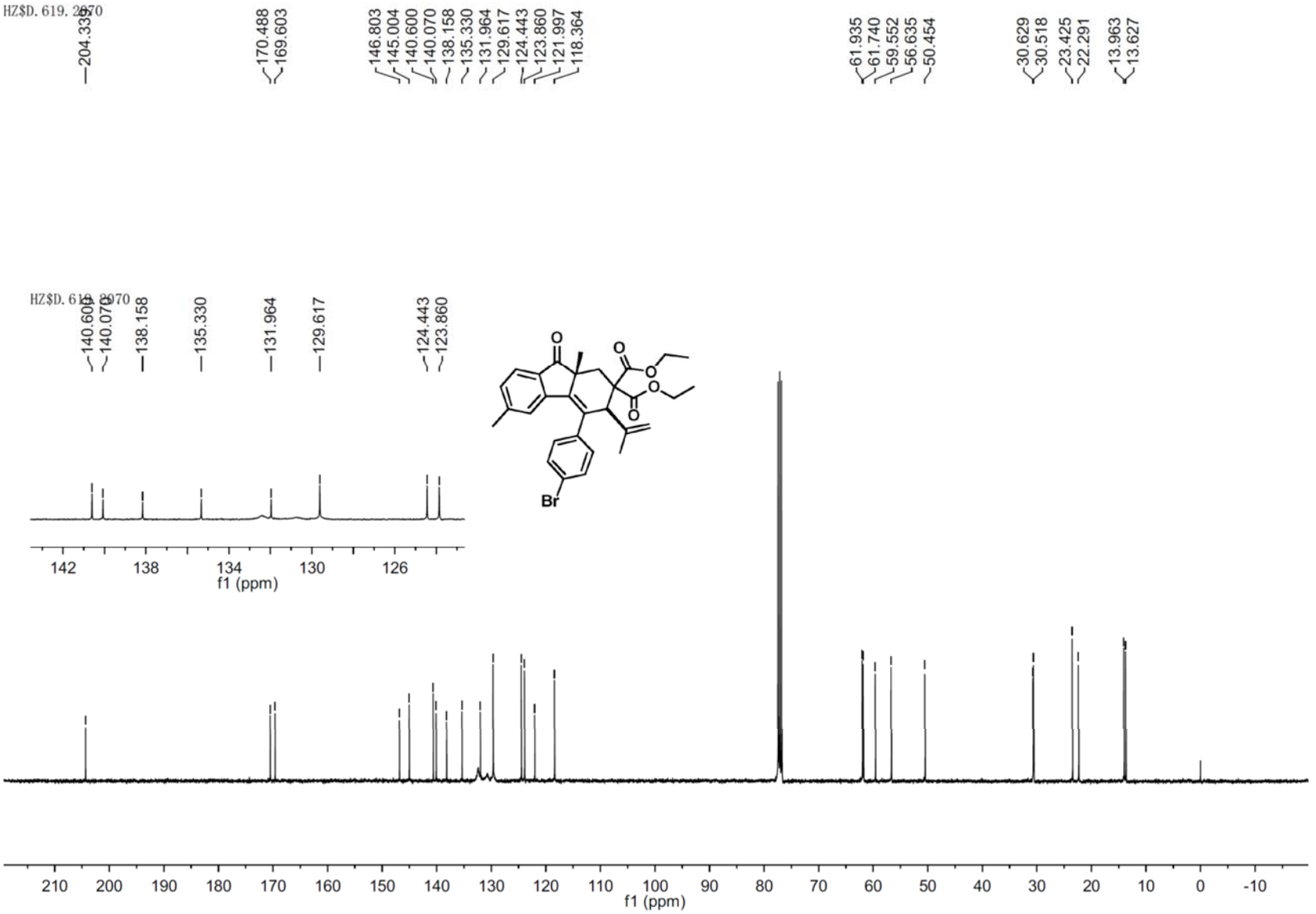
^{1}H NMR Spectrum of Compound 3jj



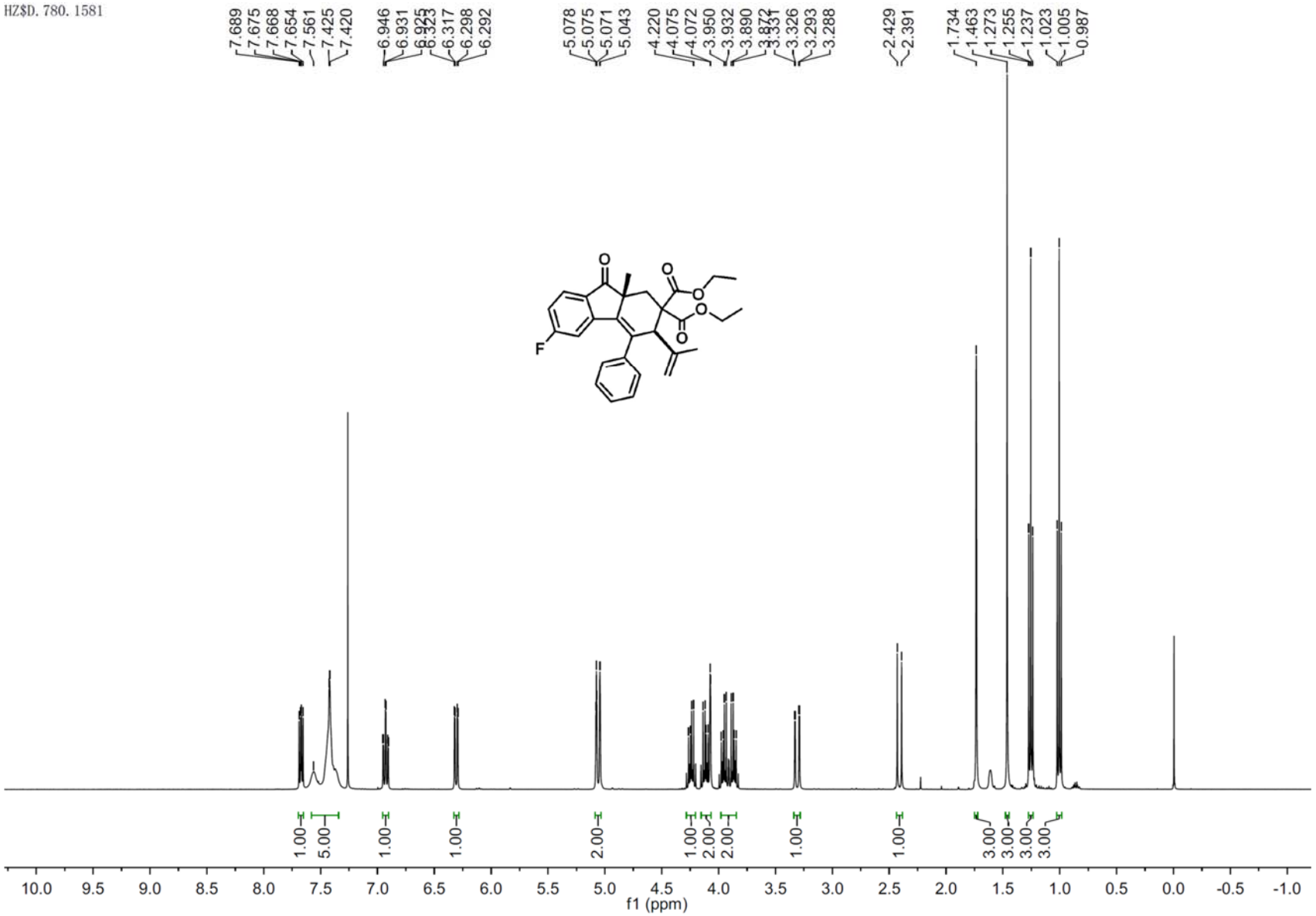
^{13}C NMR Spectrum of Compound 3jj



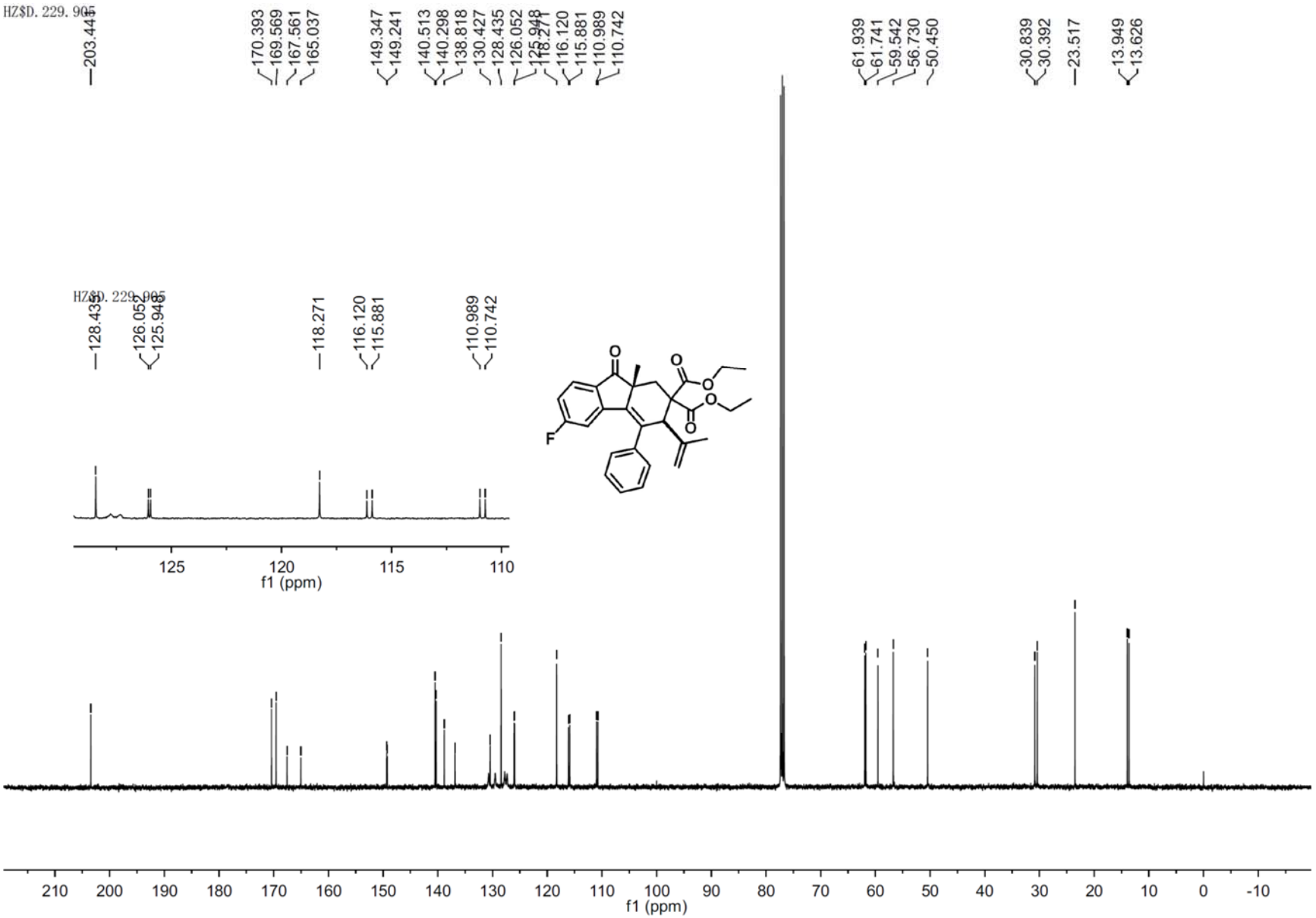
^{1}H NMR Spectrum of Compound 3kk



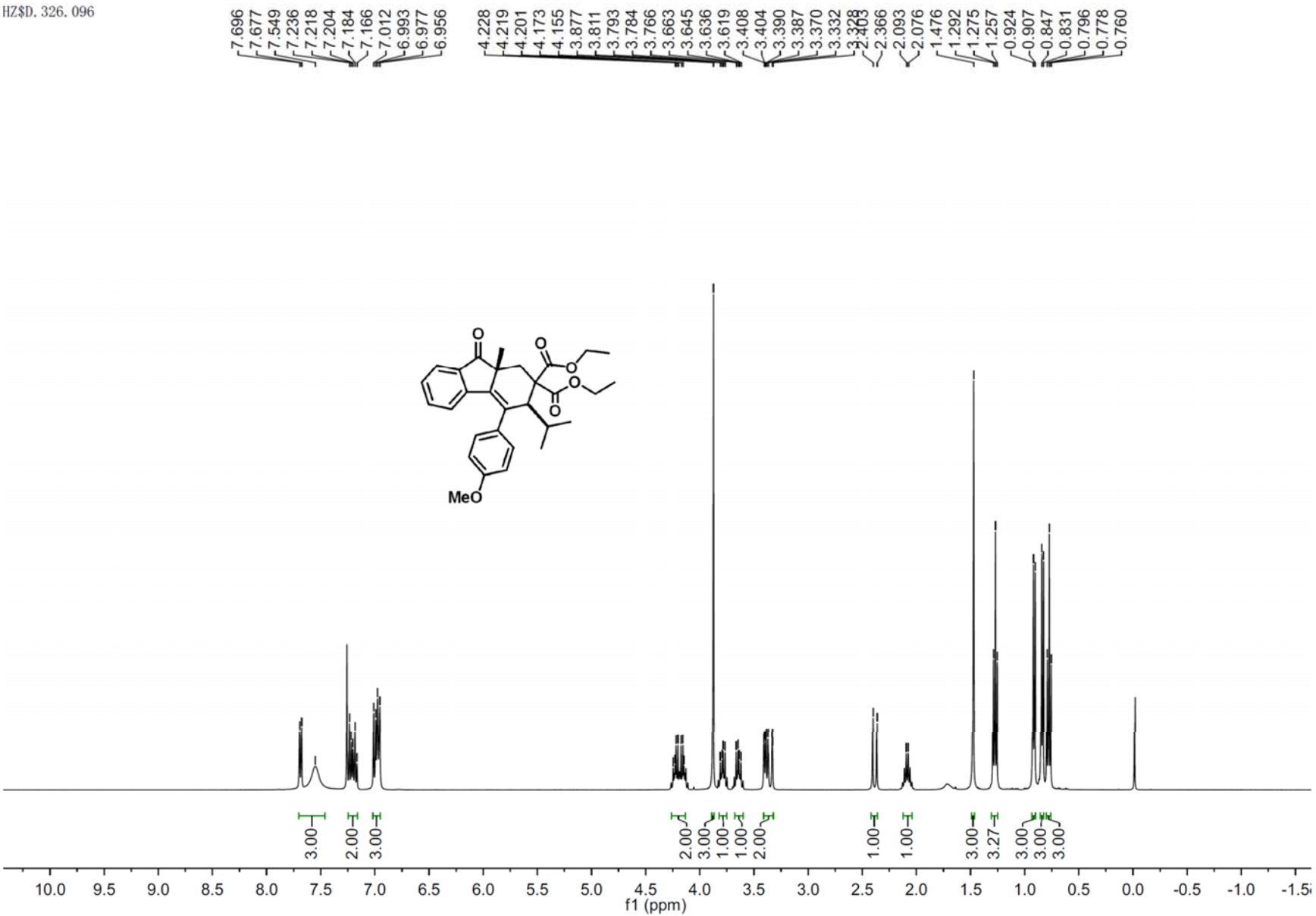
^{13}C NMR Spectrum of Compound 3kk



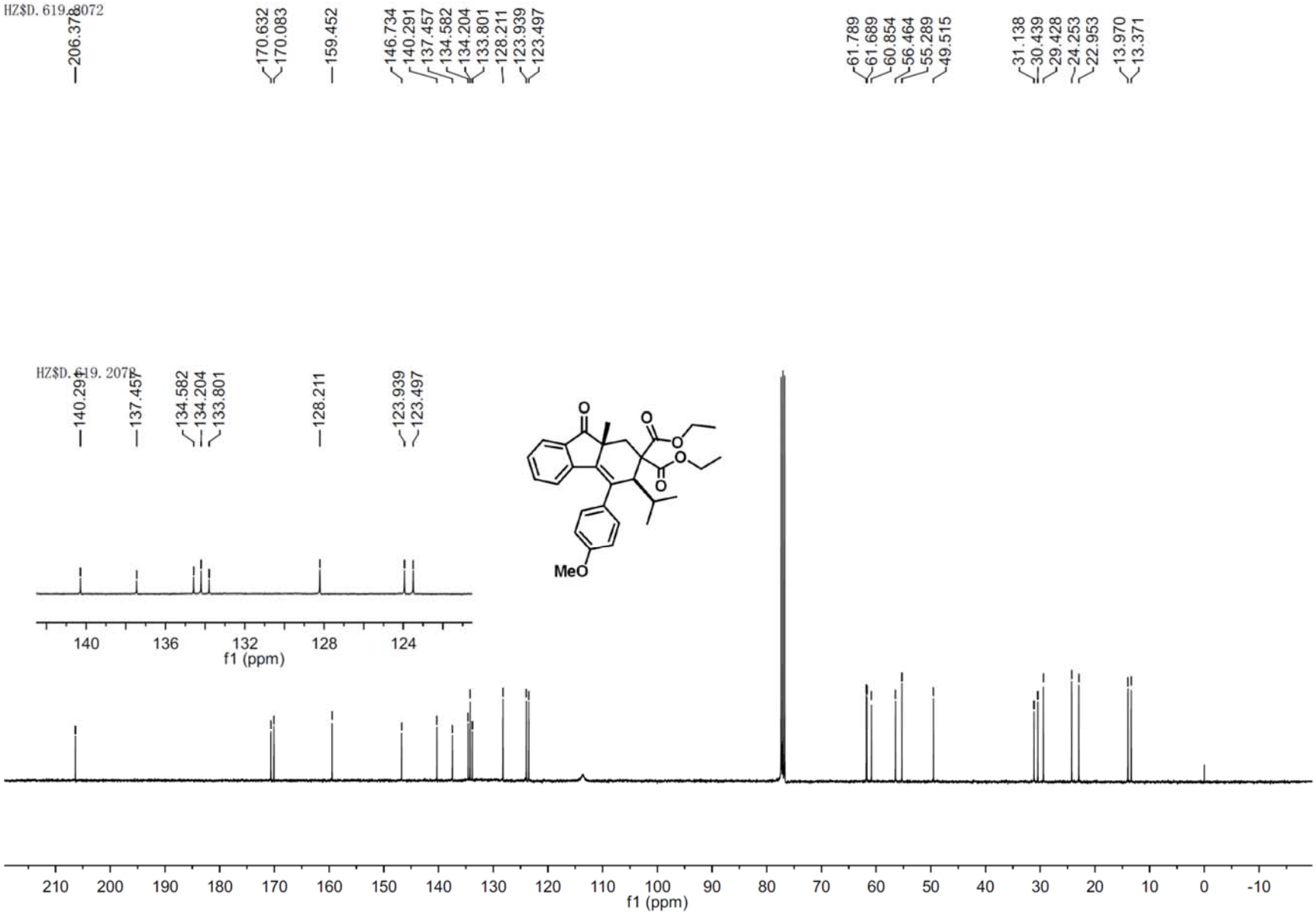
^{1}H NMR Spectrum of Compound 3ll



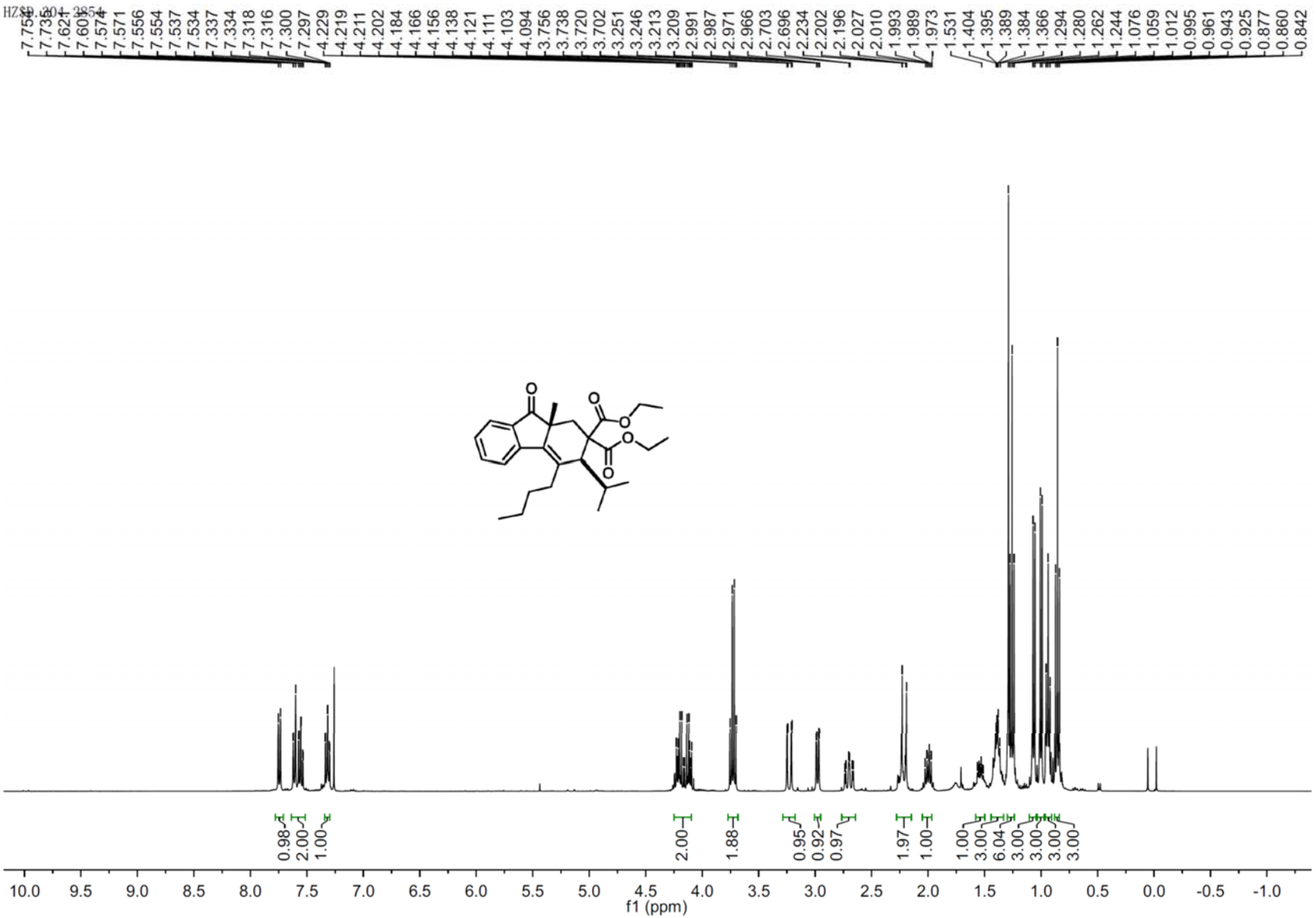
^{13}C NMR Spectrum of Compound 3ll



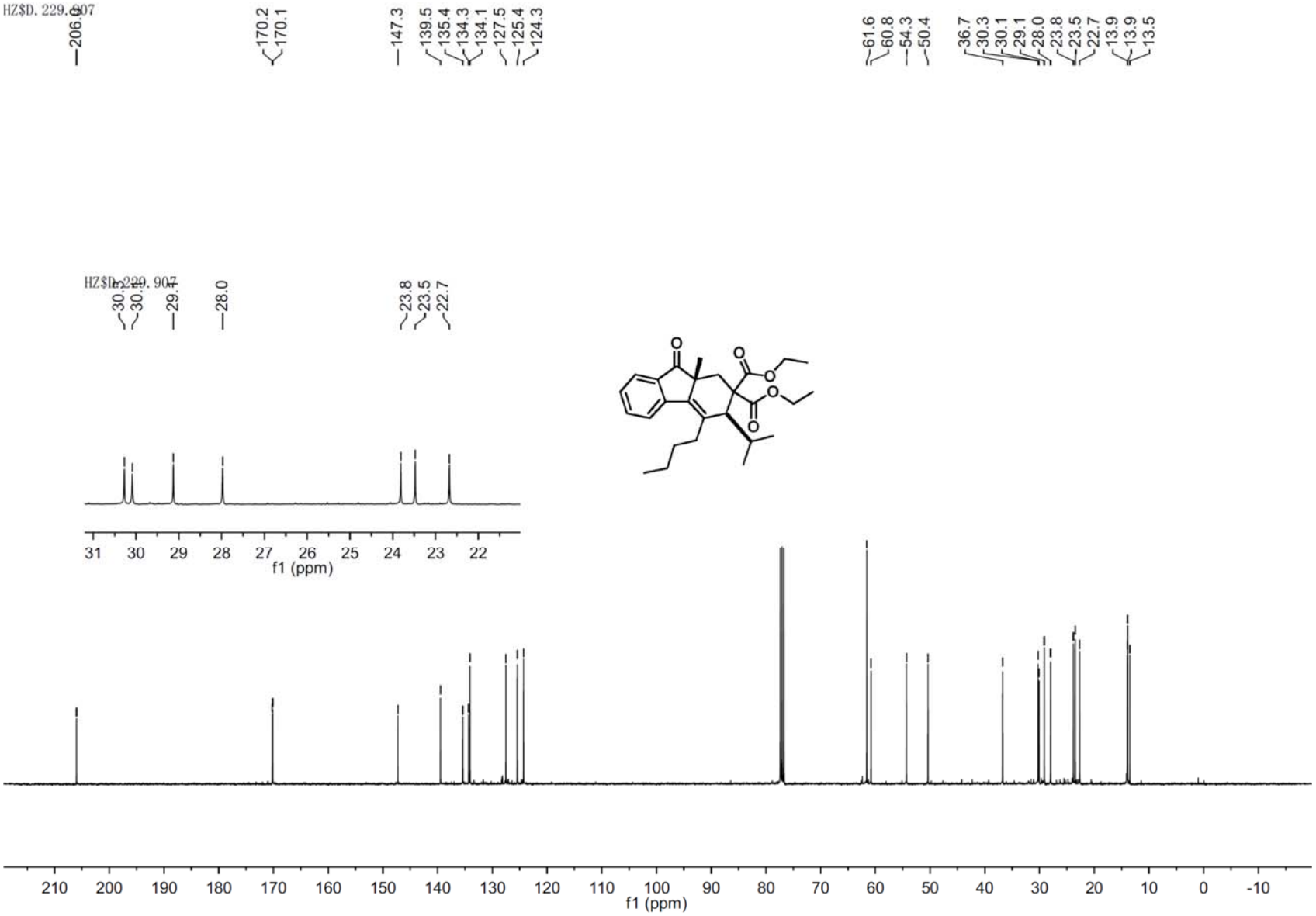
^{1}H NMR Spectrum of Compound 3mm



^{13}C NMR Spectrum of Compound 3mm



^{1}H NMR Spectrum of Compound 3nn



^{13}C NMR Spectrum of Compound 3nn