Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information for

Merging radical-polar crossover/cycloisomerization processes: access to polyfunctional furans enabled by metallaphotoredox catalysis

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1 General information

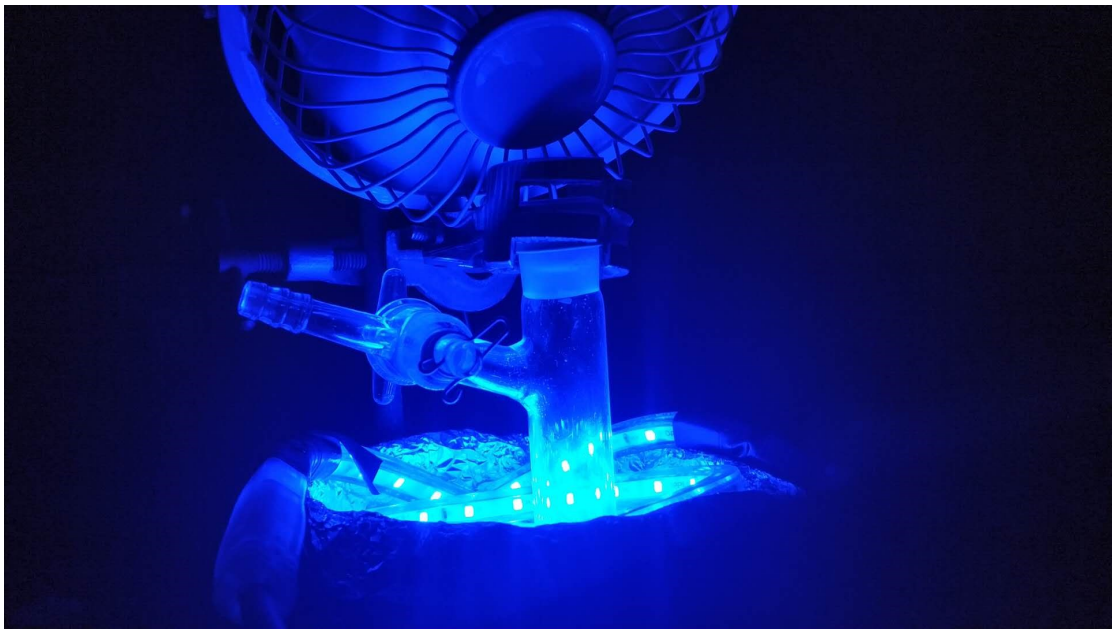
1.1 Solvents, reagents, and starting materials

All reactions were carried out under an atmosphere of nitrogen in oven-dried glassware. Photocatalysts $Ir[dF(CF_3)ppy)]_2(dtbbpy)PF_6$,[1a] 4CzIPN,[1b] and $Ru(bpz)_3(PF_6)_2$[1c] were prepared according to published procedures. Alkyl bis(catecholato)silicates **2** were reported in our previous literatures.[2] 4-Alkyldihydropyridines **7**,[3a] 4-benzoyl-1,4-dihydropyridine,[3b] 4-carbamoyl-1,4-dihydropyridine,[3c] and Hantzsch nitrile **8**[3a] were prepared using available protocols. Dried solvents were obtained from commercial sources and used without further purification unless otherwise noted.

1.2 Instruments

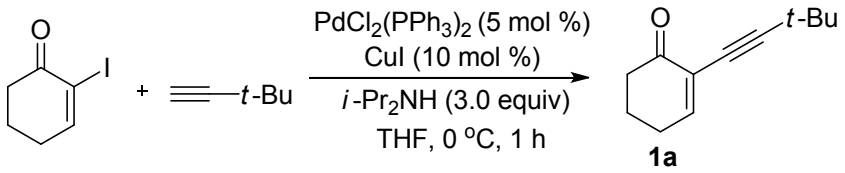
Hydrogen-1 and carbon-13 nuclear magnetic resonance spectra were recorded on Bruker Avance 500 spectrometer (500 MHz)，Agilent 400MHz NMR Spectrometer, Bruker Ultrashield 400 PLUS, Varian AS400 (400MHz). Fluorine-19 nuclear magnetic resonance spectra were recorded on an Agilent 400MHz NMR Spectrometer. Deuterium-2 nuclear magnetic resonance spectra were recorded on Bruker Ultrashield 400 PLUS, Varian AS400 (400MHz).Chemical shifts were reported in ppm downfield from tetramethylsilane, and calibrated using residue undeuterated solvent ($CDCl_3$ at 7.26 ppm ^1H NMR, 77.0 ppm ^{13}C NMR). Spectra were reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q =quartet, m = multiplet, br = broad), coupling constants (Hz) and integration. High resolution mass spectra (HRMS) were recorded on Agilent 6210 ESI/TOF MS, Thermo Q Exactive Plus, and Waters G2-Xs QTOF mass spectrometers. Analytical thin layer chromatography was performed on Polygram SIL G/UV_{254} plates. Visualization was accomplished with short wave UV light, or $KMnO_4$ staining solutions. Flash column chromatography was performed using silica gel (300-400 mesh) with solvents to use.

1.3 Picture of a typical reaction setup



2 Synthesis of various 2-(1-alkynyl)-2-alken-1-ones

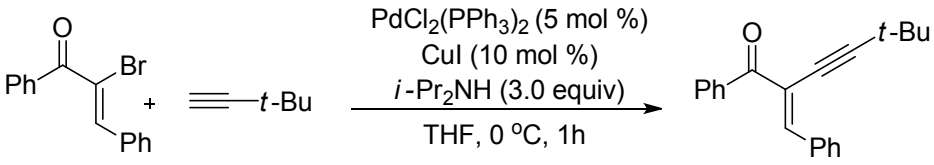
2.1 General procedure for the preparation of various 2-(1-alkynyl)-2-alken-1-ones 1a-v



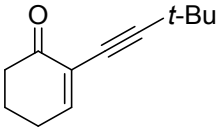
A solution of 2-iodo-cyclohexenone[4] (1.11 g, 5.00 mmol, 1.0 equiv) in THF (25 mL) was treated with $PdCl_2(PPh_3)_2$ (176 mg, 0.25 mmol, 5 mol %) and CuI (95.2 mg, 0.5 mmol, 10 mol %) and cooled down to 0°C under a N_2 atmosphere and in the dark. After 10 min of stirring, 3,3-dimethylbut-1-yne (822.0 mg, 10.0 mmol, 2.0 equiv) and diisopropylamine (1.52 g, 15.0 mmol, 3.0 equiv) were added, and the resulting yellow to dark brown solution was stirred at 0 °C for 1 h. The reaction mixture was partitioned between ethyl acetate and 0.5 N aqueous HCl solution. The aqueous layer was extracted three times with ethyl acetate and the combined organic phases were washed with brine, dried over $MgSO_4$, filtered and concentrated under vacuum. The

crude product was purified by flash column chromatography to yield alkyne **1a** as a yellow solid.

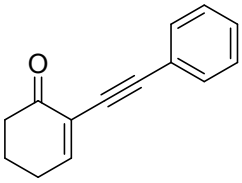
2.2 General procedure for the preparation of various 2-(1-alkynyl)-2-alken-1-ones 1w-x.



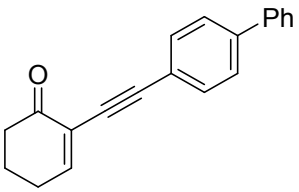
A solution of 2-bromo-1,3-diphenylprop-2-en-1-one[5] (1.44 g, 5.00 mmol, 1.0 equiv) in THF (25 mL) was treated with $PdCl_2(PPh_3)_2$ (176 mg, 0.25 mmol, 5 mol %) and CuI (95.2 mg, 0.5 mmol, 10 mol %) and cooled down to 0°C under a N_2 atmosphere and in the dark. After 10 min of stirring, 3,3-dimethylbut-1-yne (822.0 mg, 10.0 mmol, 2.0 equiv) and diisopropylamine (1.52 g, 15.0 mmol, 3.0 equiv) were added, and the resulting dark brown solution was stirred at 0 °C for 1 h. The reaction mixture was partitioned between ethyl acetate and 0.5 N aqueous HCl solution. The aqueous layer was extracted three times with ethyl acetate and the combined organic phases were washed with brine, dried over $MgSO_4$, filtered and concentrated under vacuum. The crude product was purified by flash column chromatography to yield alkyne **1w** as a yellow liquid.



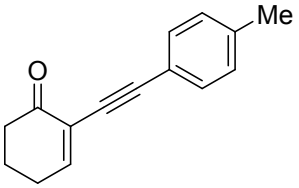
2-(3,3-dimethylbut-1-yn-1-yl)cyclohex-2-en-1-one (1a).[6] ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.17 (t, J = 4.5 Hz, 1H), 2.48-2.43 (m, 2H), 2.43-2.37 (m, 2H), 2.02-1.96 (m, 2H), 1.27 (s, 9H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 195.9, 152.8, 125.5, 101.4, 73.4, 38.2, 30.9, 27.9, 26.3, 22.5.



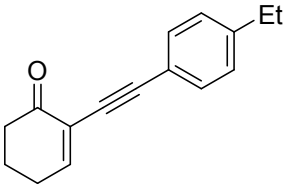
2-(phenylethynyl)cyclohex-2-en-1-one (1b).[7] ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.52-7.47 (m, 2H), 7.36 (t, J = 4.5 Hz, 1H), 7.34-7.28 (m, 3H), 2.56-2.47 (m, 4H), 2.09-2.04 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 195.5, 154.1, 131.8, 128.4, 128.2, 125.3, 122.9, 92.1, 83.8, 38.2, 26.5, 22.4.



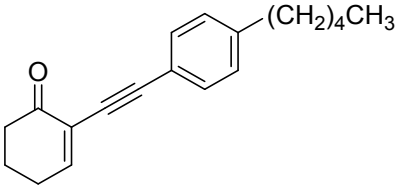
2-([1,1'-biphenyl]-4-ylethynyl)cyclohex-2-en-1-one (1c).[8] ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.64-7.57 (m, 6H), 7.48-7.44 (m, 2H), 7.42-7.36 (m, 2H), 2.59-2.53 (m, 4H), 2.13-2.07 (m, 2H).^{13}C NMR (100 MHz, $CDCl_3$) δ 195.3, 154.1, 141.0, 140.2, 132.1, 128.8, 127.6, 126.9, 126.8, 125.3, 121.7, 92.0, 84.6, 38.3, 26.7, 22.6.



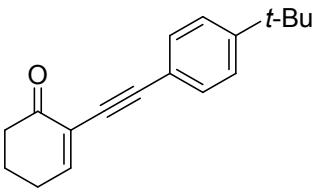
2-(*p*-tolylethynyl)cyclohex-2-en-1-one (1d).[7] ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 (d, J = 7.9 Hz, 2H), 7.34 (t, J = 4.4 Hz, 1H), 7.12 (d, J = 8.1 Hz, 2H), 2.57-2.45 (m, 4H), 2.34 (s, 3H), 2.09-2.04 (m, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 195.6, 153.8, 138.5, 131.7, 129.0, 125.4, 119.8, 92.3, 83.1, 38.2, 26.5, 22.4, 21.5.



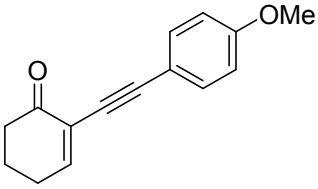
2-((4-ethylphenyl)ethynyl)cyclohex-2-en-1-one (1e). The product **1e** was obtained in 29% yield as a pale yellow solid after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42 (d, J = 8.1 Hz, 2H), 7.35 (t, J = 4.5 Hz, 1H), 7.14 (d, J = 8.1 Hz, 2H), 2.66-2.62 (m, 2H), 2.55-2.49 (m, 4H), 2.09-2.04 (m, 2H), 1.22 (t, J = 7.6 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 195.7, 153.8, 144.8, 131.8, 127.8, 125.4, 120.0, 92.3, 83.1, 38.2, 28.8, 26.5, 22.4, 15.3.HRMS (ESI) $[M+H]^+$: calculated for $C_{16}H_{17}O$: 225.1274 found 225.1287.



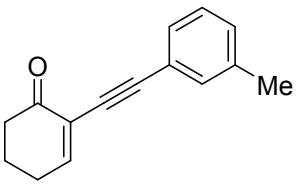
2-((4-pentylphenyl)ethynyl)cyclohex-2-en-1-one (1f).[9] ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 (d, J = 8.1 Hz, 2H), 7.33 (t, J = 4.5 Hz, 1H), 7.12 (d, J = 8.1 Hz, 2H), 2.60-2.57 (m, 2H), 2.55-2.48 (m, 4H), 2.09-2.04 (m, 2H), 1.63-1.58 (m, 2H), 1.33-1.29 (m, 4H), 0.88 (t, J = 7.0 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 195.8, 153.9, 143.6, 131.9, 128.5, 125.7, 120.1, 92.5, 83.3, 38.3, 36.0, 31.6, 31.0, 26.7, 22.7, 22.6, 14.2. HRMS (ESI) $[M+H]^+$: calculated for $C_{19}H_{23}O$: 267.1743, found 267.1741.



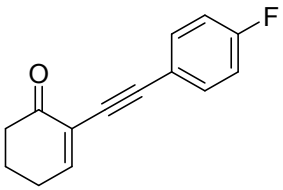
2-((4-(*tert*-butyl)phenyl)ethynyl)cyclohex-2-en-1-one (1g).[7] ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45 (d, *J* = 8.5 Hz, 2H), 7.37-7.32 (m, 3H), 2.47-2.44 (m, 2H), 2.42-2.39 (m, 2H), 2.10-2.05 (m, 2H), 1.32 (s, 9H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 195.6, 153.8, 151.7, 131.5, 125.4, 125.2, 119.8, 92.3, 83.2, 38.2, 34.8, 31.2, 26.5, 22.4.



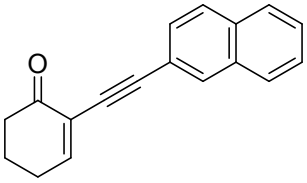
2-((4-methoxyphenyl)ethynyl)cyclohex-2-en-1-one (1h).[7] ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44-7.42 (m, 2H), 7.34-7.29 (m, 1H), 6.85-6.83 (m, 2H), 3.81 (s, 3H), 2.55-2.48 (m, 4H), 2.08-2.03 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 195.7, 159.7, 153.4, 133.3, 125.5, 115.0, 113.9, 92.1, 82.5, 55.3, 38.2, 26.5, 22.5.



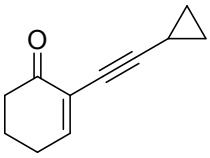
2-(*m*-tolylethynyl)cyclohex-2-en-1-one (1i).[7] ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38-7.35 (m, 2H), 7.32 (d, *J* = 7.6 Hz, 1H), 7.22 (t, *J* = 7.6 Hz, 1H), 7.15 (d, *J* = 7.8 Hz, 1H), 2.58-2.50 (m, 4H), 2.35 (s, 3H), 2.12-2.05 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 195.5, 153.9, 137.9, 132.4, 129.3, 128.8, 128.1, 125.4, 122.7, 92.3, 83.4, 38.2, 26.5, 22.5, 21.2.



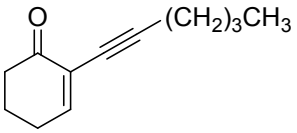
2-((4-fluorophenyl)ethynyl)cyclohex-2-en-1-one (1j).[7] ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50- 7.45 (m, 2H), 7.35 (t, *J* = 4.5 Hz, 1H), 7.03- 6.98 (m, 2H), 2.58- 2.46 (m, 4H), 2.10- 2.04 (m, 2H).^{13}C NMR (100 MHz, $CDCl_3$) δ 195.2 , 162.3 (d, *J* = 249.2 Hz), 154.0 , 133.5 (d, *J* = 8.4 Hz), 125.0 , 118.8 (d, *J* = 3.5 Hz), 115.4 (d, *J* = 21.9 Hz) , 90.9 , 83.4 (d, *J* = 1.9 Hz), 38.2 , 26.6 , 22.5 .



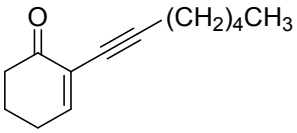
2-(naphthalen-2-ylethynyl)cyclohex-2-en-1-one (1k).[9] ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (s, 1H), 7.81-7.76 (m, 3H), 7.54 (d, *J* = 8.6 Hz, 1H), 7.49-7.47 (m, 2H), 7.40 (t, *J* = 4.4 Hz, 1H), 2.58-2.50 (m, 4H), 2.12-2.05 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 195.7, 154.3, 132.9(2), 132.8(8), 131.8, 128.5, 127.9, 127.8(4), 127.7(5), 126.7(3), 126.5, 125.4, 120.2, 92.5, 84.1, 38.2, 26.6, 22.5.



2-(cyclopropylethynyl)cyclohex-2-en-1-one (1l).[8] ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.18 (t, *J* = 4.5 Hz, 1H), 2.49-2.46 (m, 2H), 2.43-2.40 (m, 2H), 2.03-1.98 (m, 2H), 1.45-1.39 (m, 1H), 0.85-0.80 (m, 2H), 0.79-0.75 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 196.4, 153.3, 125.6, 96.6, 70.2, 38.3, 26.5, 22.6, 8.9, 0.4.

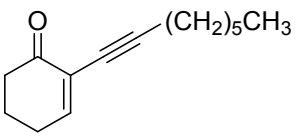


2-(hex-1-yn-1-yl)cyclohex-2-en-1-one (1m).[8] ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.20 (t, *J* = 4.4 Hz, 1H), 2.50-2.47 (m, 2H), 2.43 (q, *J* = 5.6 Hz, 2H), 2.37 (t, *J* = 7.1 Hz, 2H), 2.04-1.99 (m, 2H), 1.58-1.52 (m, 2H), 1.47-1.40 (m, 2H), 0.92 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 196.4, 153.2, 125.7, 93.6, 75.0, 38.3, 30.9, 26.5, 22.6, 22.2, 19.3, 13.8.

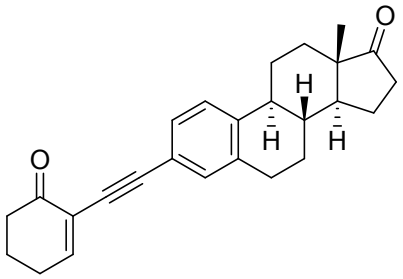


2-(hept-1-yn-1-yl)cyclohex-2-en-1-one (1n).[10] ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.21 (t, *J* = 4.4 Hz, 1H), 2.49 (d, *J* = 6.7 Hz, 2H), 2.44 (q, *J* = 5.7 Hz, 2H), 2.37 (t, *J* = 7.2 Hz, 2H), 2.05-2.00 (m, 2H), 1.60-1.55 (m, 2H), 1.44-1.29 (m, 4H), 0.91 (t, *J* = 7.1 Hz, 3H).

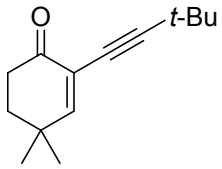
^{13}C NMR (125 MHz, $CDCl_3$) δ 196.2, 153.1, 125.6, 93.5, 74.9, 38.1, 31.1, 28.4, 26.3, 22.5, 22.2, 19.4, 14.0.



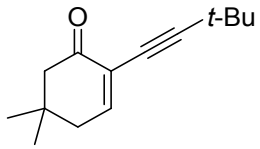
2-(oct-1-yn-1-yl)cyclohex-2-en-1-one (1o).[11] ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.21 (t, *J* = 4.4 Hz, 1H), 2.50 (t, *J* = 6.8 Hz, 2H), 2.45-2.42 (m, 2H), 2.38 (t, *J* = 7.2 Hz, 2H), 2.06-2.00 (m, 2H), 1.60-1.54 (m, 2H), 1.45-1.39 (m, 2H), 1.34-1.27 (m, 4H), 0.90 (t, *J* = 6.8 Hz, 3H).^{13}C NMR (125 MHz, $CDCl_3$) δ 196.2, 153.1, 125.6, 93.5, 74.9, 38.1, 31.4, 28.6(4), 28.6(1), 26.3, 22.5(5), 22.5(0), 19.5, 14.1.



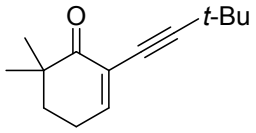
(8R,9S,13S,14S)-13-methyl-3-((6-oxocyclohex-1-en-1-yl)ethynyl)-6,7,8,9,11,12,13,14,15,16-decahydro-17H-cyclopenta[*a*]phenanthren-17-one (1p). The product **1p** was obtained in 53% yield as a pale yellow solid after column chromatography. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 (t, *J* = 4.5 Hz, 1H), 7.28-7.20 (m, 3H), 2.89-2.85 (m, 2H), 2.55-2.44 (m, 4H), 2.42-2.35 (m, 1H), 2.34-2.22 (m, 1H), 2.18-1.93 (m, 6H), 1.65-1.39 (m, 7H), 0.90 (s, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ 220.7, 195.6, 153.7, 140.4, 136.5, 132.3, 129.1, 125.5, 125.2, 120.2, 92.3, 83.2, 50.5, 47.9, 44.5, 38.2, 37.9, 35.8, 31.6, 29.0, 26.5, 26.3, 25.6, 22.5, 21.6, 13.8. HRMS (ESI) $[M+H]^+$: calculated for $C_{26}H_{29}O_2$: 373.2162, found 373.2160



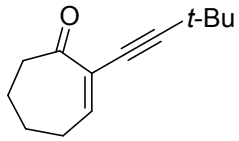
2-(3,3-dimethylbut-1-yn-1-yl)-4,4-dimethylcyclohex-2-en-1-one (1q).[12] ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.86 (s, 1H), 2.49 (t, *J* = 6.8 Hz, 2H), 1.85 (t, *J* = 6.8 Hz, 2H), 1.27 (s, 9H), 1.17 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.7, 161.6, 122.7, 101.2, 73.4, 35.7, 34.4, 33.5, 30.9, 27.9, 27.7.



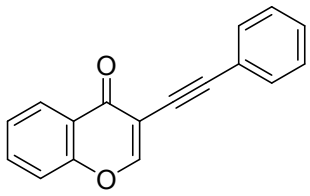
2-(3,3-dimethylbut-1-yn-1-yl)-5,5-dimethylcyclohex-2-en-1-one (1r).[12] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.02 (t, *J* = 4.3 Hz, 1H), 2.33-2.24 (m, 4H), 1.26 (s, 9H), 1.02 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.1, 150.6, 124.8, 101.6, 73.3, 51.8, 40.5, 34.0, 31.0, 28.4, 28.0.



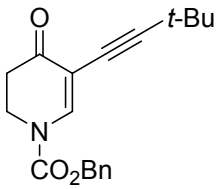
2-(3,3-dimethylbut-1-yn-1-yl)-6,6-dimethylcyclohex-2-en-1-one (1s).[12] ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.06 (t, *J* = 4.4 Hz, 1H), 2.43-2.39 (m, 2H), 1.82 (t, *J* = 6.1 Hz, 2H), 1.27 (s, 9H), 1.12 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.6, 150.9, 123.7, 100.8, 74.1, 41.7, 36.1, 31.1, 28.1, 24.4, 23.7.



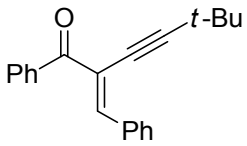
2-(3,3-dimethylbut-1-yn-1-yl)cyclohept-2-en-1-one (1t). The product **1t** was obtained in 44% yield as a pale yellow oil after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.97 (t, *J* = 6.5 Hz, 1H), 2.64-2.61 (m, 2H), 2.50-2.39 (m, 2H), 1.83-1.74 (m, 4H), 1.26 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.0, 149.2, 128.7, 99.2, 75.8, 42.3, 31.0, 28.3, 28.0, 25.0, 21.6. HRMS (ESI) $[M+H]^+$: calculated for $C_{13}H_{19}O$: 191.1430, found 191.1431



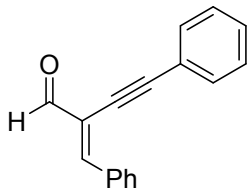
3-(phenylethynyl)-4H-chromen-4-one (1u).[13] ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.33-8.27 (m, 1H), 8.27 (s, 1H), 7.74-7.71 (m, 1H), 7.61-7.59 (m, 2H), 7.52-7.46 (m, 2H), 7.38-7.37 (m, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 175.3, 157.9, 156.0, 134.0, 131.8, 128.6, 128.3, 126.3, 125.8, 123.6, 122.7, 118.2, 111.5, 95.0, 79.5.



benzyl 5-(3,3-dimethylbut-1-yn-1-yl)-4-oxo-3,4-dihydropyridine-1(2H)-carboxylate (1v). The product **1v** was obtained in 21% yield as a gray solid after column chromatography .^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (s, 1H), 7.38 (s, 5H), 5.25 (s, 2H), 4.00 (t, *J* = 7.3 Hz, 2H), 2.56 (t, *J* = 7.3 Hz, 2H), 1.25 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 189.9, 151.8, 145.1, 134.6, 128.7, 128.6, 128.4, 104.8, 100.7, 71.3, 69.4, 42.6, 35.6, 31.1, 28.1. HRMS (ESI) $[M+H]^+$: calculated for $C_{19}H_{22}NO_3$: 312.1594, found 312.1591.

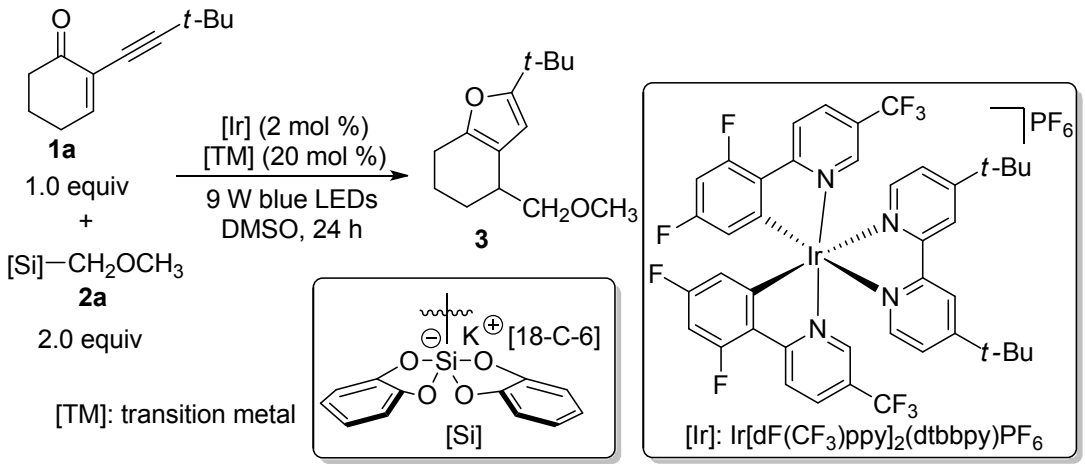


(*E*)-2-benzylidene-5,5-dimethyl-1-phenylhex-3-yn-1-one (1w). The product **1w** was obtained in 52% yield as a pale yellow oil after column chromatography. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09-8.06 (m, 2H), 7.94 (d, J = 8.0 Hz, 2H), 7.57-7.53 (m, 1H), 7.50 (s, 1H), 7.46-7.40 (m, 5H), 1.26 (s, 9H).^{13}C NMR (125 MHz, $CDCl_3$) δ 194.0, 143.6, 137.3, 135.0, 132.2, 130.2, 130.1, 129.7, 128.3, 127.8, 121.5, 110.7, 77.2, 30.3, 28.7. HRMS (ESI) $[M+H]^+$: calculated for $C_{21}H_{21}O$ 289.1587, found 289.1590.



(*E*)-2-benzylidene-4-phenylbut-3-ynal (1x).[14] ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.68 (s, 1H), 8.22-8.13 (m, 2H), 7.64-7.62 (m, 2H), 7.57 (s, 1H), 7.53-7.51 (m, 3H), 7.43-7.40 (m, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 190.9, 151.2, 134.2, 131.9, 131.6, 130.7, 129.1, 128.8, 128.5, 122.7, 122.5, 100.9, 83.2.

3 Further screening of transition metal catalyst



Entry	Deviation from standard conditions	Yield of **3** (%)
1	$In(OTf)_3$ instead of $Cu(OTf)_2$	8
2	$Sc(OTf)_3$ instead of $Cu(OTf)_2$	0
3	$Ni(OTf)_2$ instead of $Cu(OTf)_2$	0

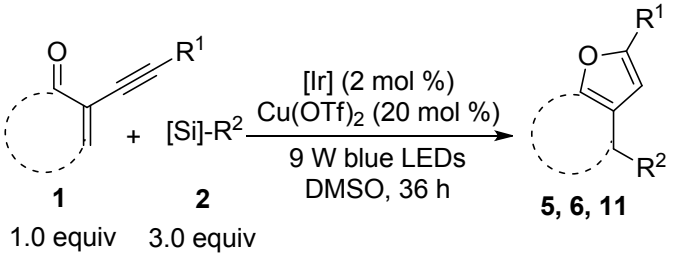
4	$Ce(OTf)_3$ instead of $Cu(OTf)_2$	0
5	$Y(OTf)_3$ instead of $Cu(OTf)_2$	0

4 General procedures of dual photoredox/copper-catalyzed reactions

4.1 General procedure for the preparation of polyfunctional furans 3, 5a, 6a, 6e, 10a-o, 11a-c, and 11f-i.

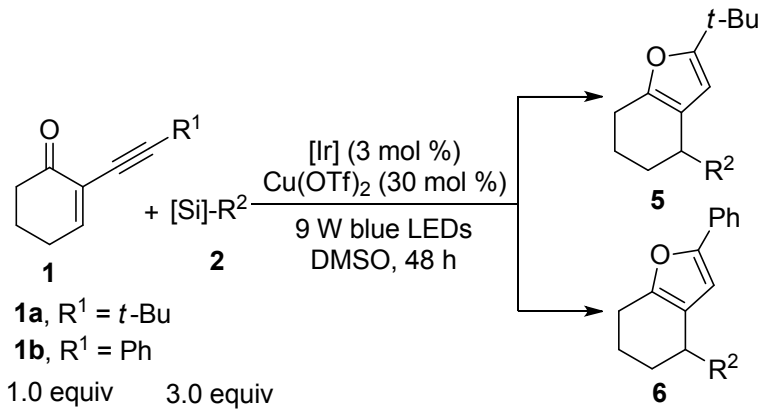
To an oven dried transparent 10 mL Schlenk tube equipped with stirring bar, $Ir[dF(CF_3)ppy)]_2(dtbbpy)PF_6$ (4.5 mg, 0.004 mmol, 2 mol %) and the 2-(1-alkynyl)-2-alken-1-ones **1** (0.2 mmol, 1.0 equiv) were added. In a glovebox, potassium [18-Crown-6] bis(catecholato) alkylsilicate **2** (0.4 mmol, 2.0 equiv) and $Cu(OTf)_2$ (14.4 mg, 0.04 mmol, 20 mol %) were added in the tube. The tube was sealed with a rubber septum and removed from the glovebox. The tube was then charged with degassed DMSO (6.0 mL, 0.033 M) via a syringe under N_2. The tube was irradiated with a 9 W blue LEDs strip spiraled within a bowel for 24 h (cooling with a fan). After the reaction was complete, the reaction solution was diluted with saturated Na_2CO_3 aqueous solution, and was extracted with EtOAc (4 x 5 mL). The organic layer was washed with brine, dried over $MgSO_4$, filtered, and solvent was evaporated to obtain crude product. Flash chromatography over silica gel afforded the product (eluent = petroleum ether / ethyl acetate 100:1 v/v).

4.2 General procedure for the preparation of polyfunctional furans 5c, 5e, 5g , 6c, 6f, 6h, and 11e.



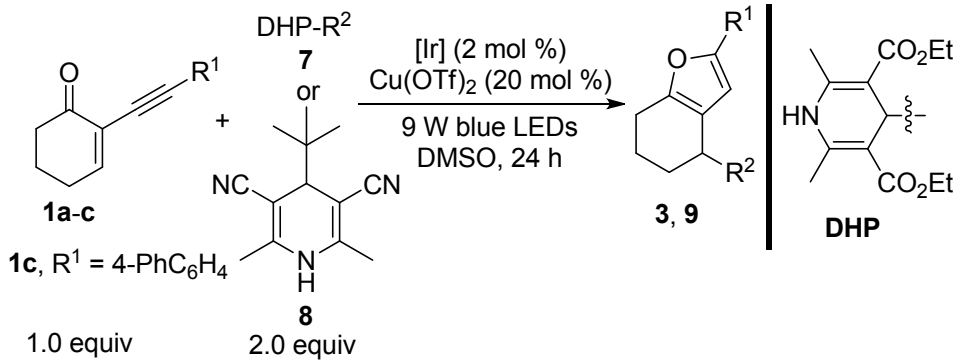
To an oven dried transparent 10 mL Schlenk tube equipped with stirring bar, $Ir[dF(CF_3)ppy)]_2(dtbbpy)PF_6$ (4.5 mg, 0.004 mmol, 2 mol %) and the 2-(1-alkynyl)-2-alken-1-ones **1** (0.2 mmol, 1.0 equiv) were added. In a glovebox, potassium [18-Crown-6] bis(catecholato) alkylsilicate **2** (0.6 mmol, 3.0 equiv) and $Cu(OTf)_2$(14.4 mg, 0.04 mmol, 20 mol %) were added in the tube. The tube was sealed with a rubber septum and removed from the glovebox. The tube was then charged with degassed DMSO (6.0 mL, 0.033 M) via a syringe under N_2. The tube was irradiated with a 9 W blue LEDs strip spiraled within a bowel for 36 h (cooling with a fan). After the reaction was complete, the reaction solution was diluted with saturated Na_2CO_3 aqueous solution, and was extracted with EtOAc (4 x 5 mL). The organic layer was washed with brine, dried over $MgSO_4$, filtered, and solvent was evaporated to obtain crude product. Flash chromatography over silica gel afforded the product (eluent = petroleum ether / ethyl acetate 100:1 v/v).

4.3 General procedure for the preparation of polyfunctional furans 5b, 5d, 5f, 6b, 6d, and 6g.



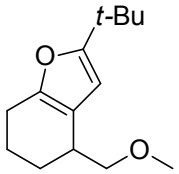
To an oven dried transparent 10 mL Schlenk tube equipped with stirring bar, $Ir[dF(CF_3)ppy)]_2(dtbbpy)PF_6$ (4.5 mg, 0.004 mmol, 2 mol %) and the 2-alkynyl-cyclohexenones **1** (0.2 mmol, 1.0 equiv) were added. In a glovebox, potassium [18-Crown-6] bis(catecholato) alkylsilicate **2** (0.4 mmol, 2.0 equiv) and $Cu(OTf)_2$(14.4 mg, 0.04 mmol, 20 mol %) were added in the tube. The tube was sealed with a rubber septum and removed from the glovebox. The tube was then charged with degassed DMSO (6.0 mL, 0.033 M) via a syringe under N_2. The tube was irradiated with a 9 W blue LEDs strip spiraled within a bowel for 24 h (cooling with a fan). After 24 h, an additional portion of $Ir[dF(CF_3)ppy)]_2(dtbbpy)PF_6$ (2.2 mg, 0.002 mmol, 1 mol %), potassium [18-Crown-6] bis(catecholato) alkylsilicate **2** (0.2 mmol, 1.0 equiv), and $Cu(OTf)_2$ (7.7 mg, 0.02 mmol, 10 mol %) were added under N_2, and the reaction was stirred for an additional 24 h under irradiation. After the reaction was complete, the reaction solution was diluted with saturated Na_2CO_3 aqueous solution, and was extracted with EtOAc (4 x 5 mL). The organic layer was washed with brine, dried over $MgSO_4$, filtered, and solvent was evaporated to obtain crude product. Flash chromatography over silica gel afforded the product (eluent = petroleum ether / ethyl acetate 100:1 v/v).

4.4 General procedure for the preparation of polyfunctional furans 9a-j

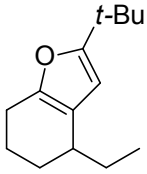


To an oven dried transparent 10 mL Schlenk tube equipped with stirring bar, $Ir[dF(CF_3)ppy)]_2(dtbbpy)PF_6$ (4.5 mg, 0.004 mmol, 2 mol %), Hantzsch ester **7** or Hantzsch nitrile **8** (0.4 mmol, 2.0 equiv) and 2-alkynyl-cyclohexenones **1** (0.2 mmol, 1.0 equiv) were added. In a glovebox, $Cu(OTf)_2$(14.4 mg, 0.04 mmol, 20 mol %) was added in the tube. The tube was sealed with a rubber septum and removed from the glovebox. The tube was then charged with degassed DMSO (6.0 mL, 0.033 M) via a syringe under N_2. The tube was irradiated with a 9 W blue LEDs strip spiraled within a bowel for 24 h (cooling with a fan). After the reaction was complete, the reaction

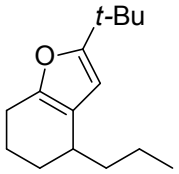
solution was diluted with saturated Na_2CO_3 aqueous solution, and was extracted with EtOAc (4 x 5 mL). The organic layer was washed with brine, dried over $MgSO_4$, filtered, and solvent was evaporated to obtain crude product. Flash chromatography over silica gel afforded the product (eluent = petroleum ether / ethyl acetate 100:1 v/v).



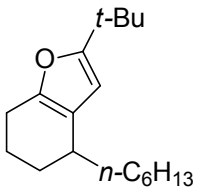
2-(*tert*-butyl)-4-(methoxymethyl)-4,5,6,7-tetrahydrobenzofuran (3). The product **3** was obtained in 87% (39.0 mg) yield as a colorless oil after column chromatography . ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.86 (s, 1H), 3.50-3.47 (m, 1H), 3.38 (s, 3H), 3.35-3.32 (m, 1H), 2.84-2.81 (m, 1H), 2.55-2.53 (m, 2H), 1.95-1.83 (m, 2H), 1.77-1.69 (m, 1H), 1.51-1.45 (m, 1H), 1.25 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 162.1, 149.1, 117.9, 101.6, 76.8, 58.8, 33.7, 32.5, 29.2, 26.4, 23.1, 21.1. HRMS (ESI) $[M+Na]^+$: calculated for $C_{14}H_{22}NaO_2$: 245.1512, found 245.1521.



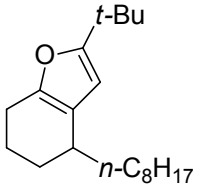
2-(*tert*-butyl)-4-ethyl-4,5,6,7-tetrahydrobenzofuran (5a). The product **5a** was obtained in 73% (30.2 mg) yield as a colorless oil after column chromatography; ^{1}HNMR (500 MHz, $CDCl_3$) δ 5.82 (s, 1H), 2.55-2.52 (m, 2H), 2.44-2.39 (m, 1H), 1.95-1.86 (m, 2H), 1.72-1.67 (m, 2H), 1.38-1.31 (m, 2H), 1.26 (s, 9H), 0.96 (t, J = 7.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 161.9, 148.2, 121.3, 101.6, 34.8, 32.5, 29.3, 28.6, 28.3, 23.2, 21.6, 11.8. HRMS (ESI) $[M+H]^+$: calculated for $C_{14}H_{23}O$: 207.1743, found 207.1740.



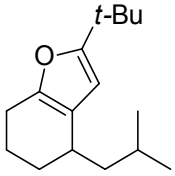
2-(*tert*-butyl)-4-propyl-4,5,6,7-tetrahydrobenzofuran (5b). The product **5b** was obtained in 86% (37.8 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.81 (s, 1H), 2.54-2.46 (m, 3H), 1.95-1.83 (m, 2H), 1.73-1.58 (m, 2H), 1.49-1.41 (m, 1H), 1.39-1.29 (m, 3H), 1.25 (s, 9H), 0.94 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.9, 148.1, 121.5, 101.6, 38.1, 32.9, 32.5, 29.3, 29.2, 23.2, 21.6, 20.5, 14.4. HRMS (ESI) [M+H]: calculated for $C_{15}H_{25}O$: 221.1900, found 221.1897.



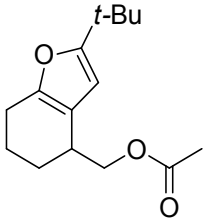
2-(*tert*-butyl)-4-hexyl-4,5,6,7-tetrahydrobenzofuran (5c); The product **5c** was obtained in 68% (35.7 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.81 (s, 1H), 2.54-2.52 (m, 2H), 2.48-2.46 (m, 1H), 1.95-1.84 (m, 2H), 1.72-1.60 (m, 2H), 1.41– 1.28 (m, 10H), 1.25 (s, 9H), 0.89 (t, J = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.7, 148.0, 121.4, 101.5, 35.8, 33.3, 32.6, 32.0, 29.7, 29.4, 29.3, 27.5, 23.4, 22.8, 21.7, 14.3. HRMS (ESI) [M+H]: calculated for $C_{18}H_{31}O$: 263.2369, found 263.2369.



2-(*tert*-butyl)-4-octyl-4,5,6,7-tetrahydrobenzofuran (5d). The product **5d** was obtained in 62% (36.1 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.82 (s, 1H), 2.54-2.48 (m, 3H), 1.94-1.85 (m, 2H), 1.73-1.61 (m, 2H), 1.42-1.28 (m, 14H), 1.26 (s, 9H), 0.91-0.88 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.6, 148.0, 121.4, 101.5, 35.8, 33.3, 32.6, 32.0, 30.1, 29.8, 29.5, 29.4, 29.3, 27.5, 23.4, 22.8, 21.7, 14.3. HRMS (ESI) [M+H]: calculated for $C_{20}H_{35}O$: 291.2682, found 291.2694.

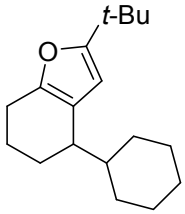


2-(*tert*-butyl)-4-isobutyl-4,5,6,7-tetrahydrobenzofuran (5e). The product **5e** was obtained in 73% (34.4 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.80 (s, 1H), 2.54-2.52 (m, 2H), 2.49-2.46(m, 1H), 1.96-1.84 (m, 2H), 1.79-1.65 (m, 2H), 1.47-1.42 (m, 1H), 1.30-1.27 (m, 1H), 1.25 (s, 9H), 1.24-1.20 (m, 1H), 0.94 (d, J = 6.6 Hz, 3H), 0.91 (d, J = 6.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.7, 147.9, 121.5, 101.5, 45.4, 32.6, 30.7, 29.4, 25.5, 23.8, 23.4, 22.2, 21.6. HRMS (ESI) $[M+H]^+$: calculated for $C_{16}H_{27}O$: 235.2056, found 235.2061.

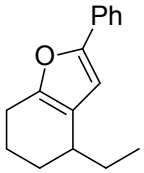


(2-(*tert*-butyl)-4,5,6,7-tetrahydrobenzofuran-4-yl)methyl acetate (5f). The product **5f** was obtained in 87% (43.5 mg) yield as a colorless oil after column

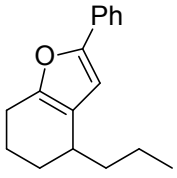
chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.83 (s, 1H), 4.19-4.15 (m, 1H), 4.03-3.99 (m, 1H), 2.90-2.84 (m, 1H), 2.56-2.53 (m, 2H), 2.09 (s, 3H), 1.96-1.84 (m, 2H), 1.78-1.70 (m, 1H), 1.50-1.46 (m, 1H), 1.25 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.1, 162.4, 149.4, 116.9, 101.5, 67.8, 32.8, 32.5, 29.2, 26.2, 23.0, 21.0, 20.8. HRMS (ESI) $[M+H]^+$: calculated for $C_{15}H_{23}O_3$: 251.1642, found 251.1643.



2-(*tert*-butyl)-4-cyclohexyl-4,5,6,7-tetrahydrobenzofuran (5g). The product **5g** was obtained in 56% (29.0 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.78 (s, 1H), 2.54-2.48 (m, 2H), 2.44-2.37 (m, 1H), 1.97-1.87 (m, 1H), 1.81-1.46 (m, 10H), 1.26 (s, 9H), 1.23-1.13 (m, 3H), 1.01-0.93 (m, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 161.6, 148.7, 120.0, 102.1, 41.5, 39.0, 32.5, 31.5, 29.3, 29.2, 27.0, 26.9, 26.8, 24.9, 23.3, 21.9. HRMS (ESI) $[M+H]^+$: calculated for $C_{18}H_{29}O$: 261.2213, found 261.2209.

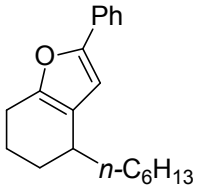


4-ethyl-2-phenyl-4,5,6,7-tetrahydrobenzofuran (6a). The product **6a** was obtained in 70% (3.8 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.62 (d, J = 7.3 Hz, 2H), 7.36-7.32 (m, 2H), 7.21-7.18 (m, 1H), 6.55 (s, 1H), 2.68-2.63 (m, 2H), 2.54-2.49 (m, 1H), 2.02-1.95 (m, 1H), 1.94-1.88 (m, 1H), 1.79-1.71 (m, 2H), 1.47-1.35 (m, 2H), 1.00 (t, J = 7.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 151.5, 150.6, 131.4, 128.5, 126.5, 123.5, 123.2, 105.1, 34.6, 28.5, 28.2, 23.4, 21.4, 11.6; HRMS (ESI) $[M+Na]^+$: calculated for $C_{16}H_{18}NaO$: 249.1250, found 249.1256.

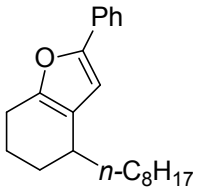


2-phenyl-4-propyl-4,5,6,7-tetrahydrobenzofuran (6b). The product **6b** was obtained in 37% (17.9 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.61 (d, J = 7.5 Hz, 2H), 7.35-7.32 (m, 2H), 7.20-7.18 (m, 1H), 6.54 (s, 1H), 2.65-2.62 (m, 2H), 2.60-2.56 (m, 1H), 2.01-1.94 (m, 1H), 1.94-1.88 (m, 1H), 1.78-1.70 (m, 1H), 1.69-1.63 (m, 1H), 1.51-1.33 (m, 4H), 0.96 (t, J = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 151.5, 150.5, 131.4, 128.5, 126.5, 123.7, 123.2,

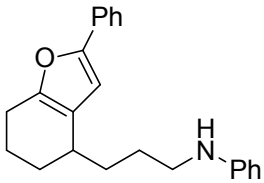
105.1, 38.0, 32.8, 29.0, 23.4, 21.4, 20.4, 14.4; HRMS (ESI) $[M+Na]^+$: calculated for $C_{17}H_{20}NaO$: 263.1406, found 263.1408.



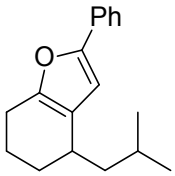
4-hexyl-2-phenyl-4,5,6,7-tetrahydrobenzofuran (6c). The product **6c** was obtained in 55% (31.2 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.62 (d, J = 7.7 Hz, 2H), 7.36-7.33 (m, 2H), 7.21-7.18 (m, 1H), 6.54 (s, 1H), 2.66-2.61 (m, 2H), 2.58-2.56 (m, 1H), 2.02-1.95 (m, 1H), 1.94-1.88 (m, 1H), 1.79-1.66 (m, 2H), 1.45-1.27 (m, 10H), 0.92-0.90 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 151.5, 150.5, 131.5, 128.5, 126.5, 123.7, 123.2, 105.1, 35.7, 33.1, 31.9, 29.6, 29.0, 27.2, 23.4, 22.7, 21.4, 14.1; HRMS (ESI) $[M+H]^+$: calculated for $C_{20}H_{27}O$: 283.2055, found 283.2055.



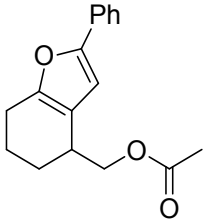
4-octyl-2-phenyl-4,5,6,7-tetrahydrobenzofuran (6d). The product **6d** was obtained in 41% (25.5 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.64-7.62 (m, 2H), 7.37-7.32 (m, 2H), 7.22-7.16 (m, 1H), 6.54 (s, 1H), 2.67-2.62 (m, 2H), 2.62-2.55 (m, 1H), 2.02-1.88 (m, 2H), 1.78-1.66 (m, 2H), 1.41-1.30 (m, 14H), 0.90 (t, J = 6.9 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.3, 150.3, 131.3, 128.4, 126.4, 123.6, 123.1, 105.0, 35.8, 33.2, 32.0, 30.1, 29.8, 29.5, 29.1, 27.4, 23.5, 22.8, 21.6, 14.3. HRMS (ESI) $[M+H]^+$: calculated for $C_{22}H_{31}O$: 311.2369, found 311.2365.



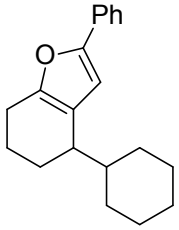
N-(3-(2-phenyl-4,5,6,7-tetrahydrobenzofuran-4-yl)propyl)aniline (6e). The product **6e** was obtained in 38% (25.1 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.61 (d, J = 7.8 Hz, 2H), 7.36-7.33 (m, 2H), 7.22-7.17 (m, 3H), 6.72-6.70 (m, 1H), 6.62 (d, J = 8.0 Hz, 2H), 6.53 (s, 1H), 3.64 (br, 1H), 3.19-3.13 (m, 2H), 2.70-2.61 (m, 3H), 2.01-1.91 (m, 2H), 1.85-1.69 (m, 4H), 1.55-1.51 (m, 1H), 1.44-1.40 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 151.7, 150.7, 148.4, 131.3, 129.2, 128.5, 126.6, 123.2, 123.1, 117.2, 112.7, 104.9, 44.3, 33.1, 32.9, 29.0, 27.2, 23.3, 21.4; HRMS (ESI) $[M+H]^+$: calculated for $C_{23}H_{26}NO$: 332.2009, found 332.2012.



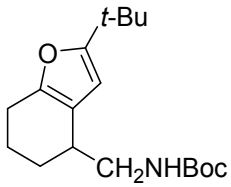
4-isobutyl-2-phenyl-4,5,6,7-tetrahydrobenzofuran (6f). The product **6f** was obtained in 22% (11.4 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.62 (d, *J* = 8.4 Hz, 2H), 7.35-7.32 (m, 2H), 7.20-7.18 (m, 1H), 6.53 (s, 1H), 2.69-2.61 (m, 3H), 2.00-1.88 (m, 2H), 1.83-1.71 (m, 2H), 1.54-1.48 (m, 1H), 1.37-1.27 (m, 2H), 0.97 (d, *J* = 6.6 Hz,3H), 0.95 (d, *J* = 6.6 Hz,3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 151.5, 150.5, 131.4, 128.5, 126.5, 123.8, 123.2, 105.1, 45.3, 30.5, 29.2, 25.4, 23.5, 23.4, 22.1, 21.2. HRMS (ESI) $[M+H]^+$: calculated for $C_{18}H_{23}O$: 255.1743, found 255.1742.



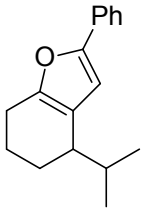
(2-phenyl-4,5,6,7-tetrahydrobenzofuran-4-yl)methyl acetate (6g). The product **6g** was obtained in 45% (24.2 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.61 (d, *J* = 7.3 Hz, 2H), 7.36-7.33 (m, 2H), 7.22-7.19 (m, 1H), 6.56 (s, 1H), 4.21-4.18 (m, 1H), 4.14-4.09 (m, 1H), 2.99-2.95 (m, 1H), 2.68-2.65 (m, 2H), 2.12-2.10 (m, 3H), 2.01-1.89 (m, 2H), 1.83-1.78 (m, 1H), 1.56-1.52 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.1, 152.0, 151.5, 131.1, 128.6, 126.8, 123.3, 119.3, 104.8, 67.6, 32.8, 26.1, 23.2, 21.0, 20.8; HRMS (ESI) $[M+Na]^+$: calculated for $C_{17}H_{18}NaO_3$: 293.1148, found 293.1148.



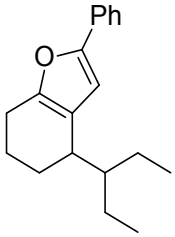
4-cyclohexyl-2-phenyl-4,5,6,7-tetrahydrobenzofuran (6h). The product **6h** was obtained in 51% (28.8 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.63 (d, *J* = 7.8 Hz, 2H), 7.36-7.32 (m, 2H), 7.21-7.18 (m, 1H), 6.53 (s, 1H), 2.66-2.61 (m, 2H), 2.52-2.48 (m, 1H), 2.01-1.95 (m, 1H), 1.80-1.58 (m, 8H), 1.30-1.14 (m, 5H), 1.06-0.98 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 151.3, 151.2, 131.5, 128.5, 126.5, 123.2, 122.2, 105.6, 41.6, 38.9, 31.4, 29.1, 27.0, 26.9, 26.8, 24.8, 23.4, 21.8. HRMS (ESI) $[M+H]^+$: calculated for $C_{20}H_{25}O$: 281.1900, found 281.1896.



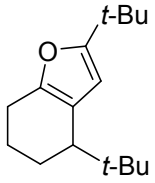
***tert*-butyl ((2-(*tert*-butyl)-4,5,6,7-tetrahydrobenzofuran-4-yl)methyl)carbamate (9a).** The product **9a** was obtained in 39% (23.8 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.82 (s, 1H), 4.60 (br, 1H), 3.45-3.31 (m, 1H), 3.17-3.08 (m, 1H), 2.75-2.65 (m, 1H), 2.53 (t, *J* = 6.3 Hz, 2H), 1.95-1.88 (m, 1H), 1.86-1.79 (m, 1H), 1.76-1.66 (m, 2H), 1.45 (s, 9H), 1.24 (s, 9H).^{13}C NMR (100 MHz, $CDCl_3$) δ 162.2, 155.9, 149.2, 117.8, 101.1, 79.1, 44.8, 33.9, 32.6, 29.3, 28.6, 27.0, 23.2, 21.3. HRMS (ESI) $[M+H]^+$: calculated for $C_{18}H_{30}NO_3$: 308.2220 found 308.2212.



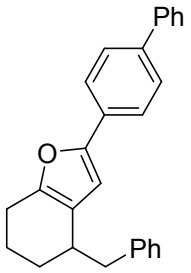
4-isopropyl-2-phenyl-4,5,6,7-tetrahydrobenzofuran (9b). The product **9b** was obtained in 50% (24.0 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.63 (d, *J* = 8.3 Hz, 2H), 7.36-7.33 (m, 2H), 7.21-7.18 (m, 1H), 6.54 (s, 1H), 2.66-2.62 (m, 2H), 2.55-2.51 (m, 1H), 2.05-1.98 (m, 2H), 1.79-1.68 (m, 2H), 1.50-1.43 (m, 1H), 1.02 (d, *J* = 6.9 Hz, 3H), 0.86 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 151.4, 151.3, 131.5, 128.5, 126.5, 123.2, 122.4, 105.4, 39.4, 30.9, 24.0, 23.4, 21.9, 20.6, 18.3; HRMS (ESI) $[M+Na]^+$: calculated for $C_{17}H_{20}NaO$: 263.1406, found 263.1402.



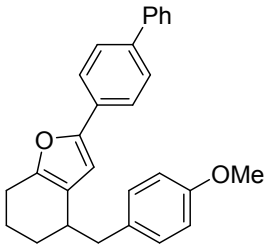
4-(pentan-3-yl)-2-phenyl-4,5,6,7-tetrahydrobenzofuran (9c). The product **9c** was obtained in 25% (13.4 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.65-7.60 (m, 2H), 7.37-7.32 (m, 2H), 7.22-7.17 (m, 1H), 6.51 (s, 1H), 2.82-2.76 (m, 1H), 2.70-2.57 (m, 2H), 2.07-1.98 (m, 1H), 1.79-1.65 (m, 2H), 1.54-1.43 (m, 2H), 1.40-1.26 (m, 3H), 1.16-1.06 (m, 1H), 0.97 (t, *J* = 7.3 Hz, 3H), 0.88 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.3, 151.2, 131.3, 128.4, 126.3, 123.1, 122.5, 104.8, 44.9, 35.3, 24.2, 23.9, 23.5, 23.3, 22.5, 13.1, 12.7. HRMS (ESI) $[M+H]^+$: calculated for $C_{19}H_{25}O$: 269.1900, found 269.1899.



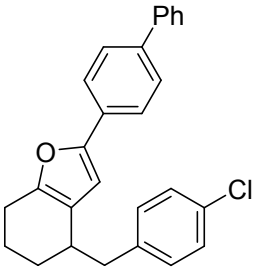
2,4-di-tert-butyl-4,5,6,7-tetrahydrobenzofuran (9d). The product **9d** was obtained in 33% (15.4 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.92 (s, 1H), 2.51-2.48 (m, 2H), 2.39-2.35 (m, 1H), 1.99-1.92 (m, 1H), 1.84-1.76 (m, 1H), 1.66-1.59 (m, 1H), 1.48-1.39 (m, 1H), 1.25 (s, 9H), 0.96 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.8, 149.7, 118.4, 103.6, 44.1, 34.1, 32.5, 29.4, 28.7, 26.1, 23.5, 22.7. HRMS (ESI) $[M+H]^+$: calculated for $C_{16}H_{27}O$: 235.2056, found 235.2051.



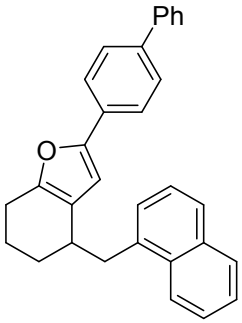
2-([1,1'-biphenyl]-4-yl)-4-benzyl-4,5,6,7-tetrahydrobenzofuran (9e). The product **9e** was obtained in 45% (32.9 mg) yield as a pale yellow solid after column chromatography; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67-7.56 (m, 6H), 7.47-7.41 (m, 2H), 7.37-7.29 (m, 3H), 7.26-7.20 (m, 3H), 6.38 (s, 1H), 3.08-3.00 (m, 1H), 2.97-2.88 (m, 1H), 2.71-2.62 (m, 3H), 2.01-1.92 (m, 1H), 1.83-1.69 (m, 2H), 1.47-1.38 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.3, 151.1, 140.7(3), 140.6(8), 139.2, 130.3, 129.2, 128.8, 128.3, 127.2(2), 127.1(8), 126.8, 126.0, 123.6, 123.1, 105.3, 42.1, 35.0, 28.8, 23.4, 21.1. HRMS (ESI) $[M+H]^+$: calculated for $C_{27}H_{25}O$: 365.1900, found 365.1905.



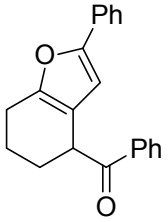
2-([1,1'-biphenyl]-4-yl)-4-(4-methoxybenzyl)-4,5,6,7-tetrahydrobenzofuran (9f). The product **9f** was obtained in 31% (24.4 mg) yield as a pale yellow solid after column chromatography; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67-7.56 (m, 6H), 7.47-7.41 (m, 2H), 7.36-7.31 (m, 1H), 7.15 (d, J = 8.6 Hz, 2H), 6.87 (d, J = 8.6 Hz, 2H), 6.39 (s, 1H), 3.82 (s, 3H), 3.00-2.94 (m, 1H), 2.91-2.84 (m, 1H), 2.70-2.64 (m, 2H), 2.63-2.57 (m, 1H), 2.00-1.93 (m, 1H), 1.81-1.71 (m, 2H), 1.45-1.39 (m, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 157.9, 151.2, 151.1, 140.7, 139.1, 132.7, 130.3, 130.1, 128.8, 127.2(1), 127.1(6), 126.8, 123.6, 123.1, 113.6, 105.3, 55.3, 41.2, 35.1, 28.8, 23.4, 21.1. HRMS (ESI) $[M+H]^+$: calculated for $C_{28}H_{27}O_2$: 395.2006, found 395.2014.



2-([1,1'-biphenyl]-4-yl)-4-(4-chlorobenzyl)-4,5,6,7-tetrahydrobenzofuran (9g). The product **9g** was obtained in 46% (36.9 mg) yield as a pale yellow solid after column chromatography; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69-7.56 (m, 6H), 7.46-7.42 (m, 2H), 7.36-7.31 (m, 1H), 7.29 (d, *J* = 8.2 Hz, 2H), 7.16 (d, *J* = 8.3 Hz, 2H), 6.37 (s, 1H), 3.03-2.97 (m, 1H), 2.93-2.85 (m, 1H), 2.70-2.59 (m, 3H), 2.00-1.92 (m, 1H), 1.81-1.72 (m, 2H), 1.42-1.35 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.4, 151.1, 140.7, 139.3, 139.1, 131.8, 130.6, 130.2, 128.8, 128.4, 127.3, 127.2, 126.8, 123.7, 122.7, 105.1, 41.4, 34.9, 28.8, 23.4, 21.1. HRMS (ESI) $[M+H]^+$: calculated for $C_{27}H_{24}ClO$: 399.1510, found 399.1508.

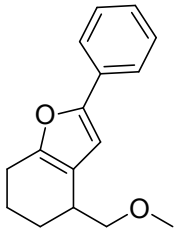


2-([1,1'-biphenyl]-4-yl)-4-(naphthalen-1-ylmethyl)-4,5,6,7-tetrahydrobenzofuran (9h). The product **9h** was obtained in 16% (13.5 mg) yield as a pale yellow solid after column chromatography; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (d, *J* = 8.2 Hz, 1H), 7.90 (d, *J* = 7.9 Hz, 1H), 7.78 (d, *J* = 8.3 Hz, 1H), 7.71-7.58 (m, 6H), 7.56-7.48 (m, 2H), 7.47-7.40 (m, 3H), 7.33 (d, *J* = 7.3 Hz, 2H), 6.49 (s, 1H), 3.59 (dd, *J* = 13.3, 5.1 Hz, 1H), 3.16-3.08 (m, 1H), 3.06-2.99 (m, 1H), 2.74-2.65 (m, 2H), 2.05-1.97 (m, 1H), 1.77-1.69 (m, 2H), 1.53-1.47 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.4, 151.1, 140.7, 139.2, 136.6, 134.0, 132.1, 130.3, 128.9, 128.8, 127.4, 127.2(4), 127.1(8), 126.9, 126.8, 125.8, 125.5, 125.3, 123.9, 123.7, 123.3, 105.3, 39.3, 34.0, 29.1, 23.5, 21.1. HRMS (ESI) $[M+H]^+$: calculated for $C_{31}H_{27}O$: 415.2056, found 415.2065.

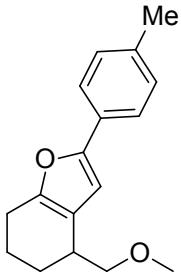


phenyl(2-phenyl-4,5,6,7-tetrahydrobenzofuran-4-yl)methanone (9i). The product **9i** was obtained in 25% (15.2 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.05 (d, *J* = 7.8 Hz, 2H), 7.63-7.60 (m, 1H), 7.57-7.51 (m, 4H), 7.33-7.30 (m, 2H), 7.20-7.17 (m, 1H), 6.31 (s, 1H), 4.61-

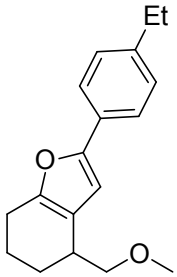
4.58 (m, 1H), 2.80-2.70 (m, 2H), 2.16-2.08 (m, 2H), 2.04-1.99 (m, 1H), 1.94-1.87 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 200.9, 152.1, 151.9, 136.6, 133.2, 131.1, 128.7, 128.6, 128.5, 126.8, 123.4, 117.1, 105.3, 41.8, 27.1, 23.0, 21.4; HRMS (ESI) $[M+Na]^+$: calculated for $C_{21}H_{18}NaO_2$: 325.1199, found 325.1197.



4-(methoxymethyl)-2-phenyl-4,5,6,7-tetrahydrobenzofuran (10a). The product **10a** was obtained in 57% (27.8 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.63-7.61 (m, 2H), 7.35-7.32 (m, 2H), 7.21-7.18 (m, 1H), 6.60 (s, 1H), 3.51-3.48 (m, 1H), 3.43-3.39 (m, 4H), 2.92-2.87 (m, 1H), 2.67-2.64 (m, 2H), 2.01-1.95 (m, 1H), 1.93-1.87 (m, 1H), 1.83-1.74 (m, 1H), 1.55-1.49 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 151.8, 151.3, 131.3, 128.5, 126.59, 123.3, 120.4, 105.2, 76.7, 58.9, 33.7, 26.3, 23.3, 21.1; HRMS (ESI) $[M+Na]^+$: calculated for $C_{16}H_{18}NaO_2$: 265.1199, found 265.1196.

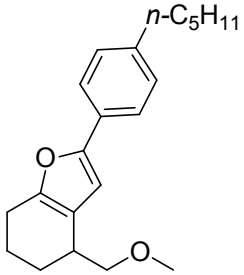


4-(methoxymethyl)-2-(*p*-tolyl)-4,5,6,7-tetrahydrobenzofuran (10b). The product **10b** was obtained in 69% (35.3 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.52 (d, J = 8.2 Hz, 2H), 7.15 (d, J = 7.9 Hz, 2H), 6.54 (s, 1H), 3.52-3.49 (m, 1H), 3.42-3.39 (m, 4H), 2.92-2.87 (m, 1H), 2.68-2.62 (m, 2H), 2.34 (s, 3H), 2.01-1.94 (m, 1H), 1.93-1.87 (m, 1H), 1.82-1.74 (m, 1H), 1.55-1.49 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 152.0, 150.8, 136.3, 129.2, 128.6, 123.2, 120.2, 104.4, 76.8, 58.9, 33.7, 26.3, 23.2, 21.2, 21.1; HRMS (ESI) $[M+Na]^+$: calculated for $C_{17}H_{20}NaO_2$: 279.1356, found 279.1359.

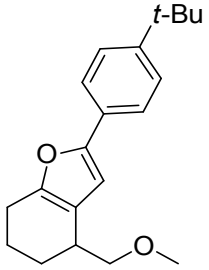


2-(4-ethylphenyl)-4-(methoxymethyl)-4,5,6,7-tetrahydrobenzofuran (10c). The product **10c** was obtained in 60% (32.3 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.53 (d, J = 8.2 Hz, 2H), 7.17 (d, J = 7.9 Hz, 2H), 6.53 (s, 1H), 3.52-3.49 (m, 1H), 3.42-3.39 (m, 4H), 2.92-2.86 (m,1H),

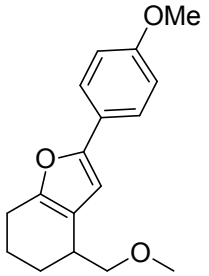
2.66-2.62 (m, 4H), 2.01-1.94 (m, 1H), 1.93-1.87 (m, 1H), 1.82-1.74 (m, 1H), 1.53-1.49 (m, 1H), 1.24 (t, J = 7.6 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 152.1, 150.9, 142.8, 128.9, 128.0, 123.4, 120.2, 104.5, 76.8, 58.9, 33.7, 28.6, 26.3, 23.3, 21.1, 15.5; HRMS (ESI) $[M+H]^+$: calculated for $C_{18}H_{23}O_2$: 271.1693, found 271.1705.



5-(methoxymethyl)-2-(4-pentylphenyl)-4,5,6,7-tetrahydrobenzofuran (10d). The product **10d** was obtained in 52% (32.4 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.53 (d, J = 7.5 Hz, 2H), 7.16 (d, J = 7.7 Hz, 2H), 6.54 (s, 1H), 3.52-3.49 (m, 1H), 3.42-3.39 (m, 4H), 2.91-2.89 (m, 1H), 2.66-2.64 (m, 2H), 2.61-2.58 (m, 2H), 2.00-1.95 (m, 1H), 1.93-1.88 (m, 1H), 1.82-1.75 (m, 1H), 1.65-1.61 (m, 2H), 1.56-1.49 (m, 1H), 1.38-1.31 (m, 4H), 0.91-0.88 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 152.1, 150.9, 141.5, 128.9, 128.6, 123.3, 120.2, 104.5, 76.8, 58.9, 35.7, 33.7, 31.5, 31.1, 26.3, 23.3, 22.5, 21.1, 14.0. HRMS (ESI) $[M+H]^+$: calculated for $C_{21}H_{29}O_2$: 313.2162, found 313.2167.

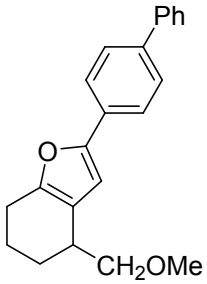


2-(4-(*tert*-butyl)phenyl)-4-(methoxymethyl)-4,5,6,7-tetrahydrobenzofuran (10e). The product **10e** was obtained in 68% (40.5 mg) yield as a pale yellow solid after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.55 (d, J = 8.5 Hz, 2H), 7.37 (d, J = 8.5 Hz, 2H), 6.55 (s, 1H), 3.53-3.50 (m, 1H), 3.43-3.40 (m, 4H), 2.93-2.87 (m, 1H), 2.67-2.65 (m, 2H), 2.02-1.95 (m, 1H), 1.94-1.88 (m, 1H), 1.83-1.75 (m, 1H), 1.56-1.50 (m, 1H), 1.34 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 152.0, 150.9, 149.6, 128.7, 125.5, 123.1, 120.2, 104.6, 76.8, 58.9, 34.6, 33.7, 31.3, 26.3, 23.3, 21.1. HRMS (ESI) $[M+H]^+$: calculated for $C_{20}H_{27}O_2$: 299.2006, found 299.2009.

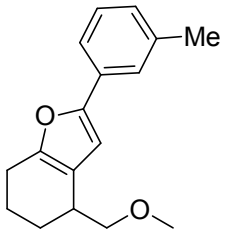


4-(methoxymethyl)-2-(4-methoxyphenyl)-4,5,6,7-tetrahydrobenzofuran (10f). The product **10f** was obtained in 60% (32.6 mg) yield as a colorless oil after column

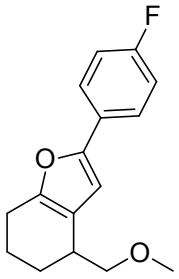
chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.55 (d, *J* = 8.8 Hz, 2H), 6.89 (d, *J* = 8.8 Hz, 2H), 6.47 (s, 1H), 3.82 (s, 3H), 3.52-3.49 (m, 1H), 3.42-3.39 (m, 4H), 2.92-2.85 (m, 1H), 2.68-2.62 (m, 2H), 2.01-1.94 (m, 1H), 1.94-1.86 (m, 1H), 1.83-1.74 (m, 1H), 1.56-1.48 (m, 1H).^{13}C NMR (125 MHz, $CDCl_3$) δ 158.6, 151.9, 150.6, 124.7, 124.5, 120.2, 114.0, 103.6, 76.8, 58.9, 55.3, 33.7, 26.3, 23.3, 21.2. HRMS (ESI) $[M+H]^+$: calculated for $C_{17}H_{21}O_3$: 273.1485, found 273.1483.



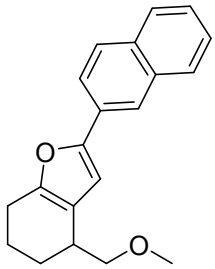
2-([1,1'-biphenyl]-4-yl)-4-(methoxymethyl)-4,5,6,7-tetrahydrobenzofuran (10g). The product **10g** was obtained in 66% (42.2 mg) yield as a pale yellow solid after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.69 (d, *J* = 8.4 Hz, 2H), 7.63-7.58 (m, 4H), 7.46-7.42 (m, 2H), 7.35-7.32 (m, 1H), 6.65 (s, 1H), 3.53-3.49 (m, 1H), 3.44-3.41 (m, 4H), 2.94-2.89 (m, 1H), 2.70-2.66 (m, 2H), 2.02-1.95 (m, 1H), 1.95-1.88 (m, 1H), 1.84-1.76 (m, 1H), 1.56-1.49 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.6, 151.5, 140.7, 139.2, 130.3, 128.7, 127.2(1), 127.1(7), 126.8, 123.7, 120.6, 105.5, 76.8, 58.9, 33.7, 26.3, 23.3, 21.1. HRMS (ESI) $[M+H]^+$: calculated for $C_{22}H_{23}O_2$: 319.1693, found 319.1689.



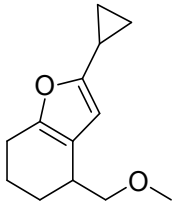
4-(methoxymethyl)-2-(*m*-tolyl)-4,5,6,7-tetrahydrobenzofuran (10h). The product **10h** was obtained in 56% (28.8 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46-7.42 (m, 2H), 7.26-7.22 (m, 1H), 7.02 (d, *J* = 7.5 Hz, 1H), 6.59 (s, 1H), 3.52-3.49 (m, 1H), 3.43-3.40 (m, 4H), 2.91-2.88 (m, 1H), 2.67-2.65 (m, 2H), 2.37 (s, 3H), 2.02-1.95 (m, 1H), 1.93-1.88 (m, 1H), 1.83-1.75 (m, 1H), 1.56-1.49 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 151.9, 151.1, 138.1, 131.2, 128.4, 127.4, 123.9, 120.5, 120.3, 105.1, 76.8, 58.9, 33.7, 26.3, 23.5, 21.5, 21.1. HRMS (ESI) $[M+Na]^+$: calculated for $C_{17}H_{20}NaO_2$: 279.1356, found 279.1359.



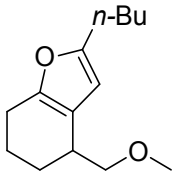
2-(4-fluorophenyl)-4-(methoxymethyl)-4,5,6,7-tetrahydrobenzofuran (10i). The product **10i** was obtained in 58% (30.0 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59-7.56 (m, 2H), 7.05-7.02 (m, 2H), 6.53 (s, 1H), 3.50-3.47 (m, 1H), 3.43-3.40 (m, 4H), 2.91-2.86 (m, 1H), 2.68-2.61 (m, 2H), 2.00-1.94 (m, 1H), 1.92-1.87 (m, 1H), 1.82-1.74 (m, 1H), 1.55-1.48 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 161.7 (d, J = 244.4 Hz), 151.3, 150.9, 127.7 (d, J = 3.1 Hz), 124.9 (d, J = 7.8 Hz), 120.5, 115.50 (d, J = 21.9 Hz), 104.9, 58.9, 33.7, 26.2, 23.2, 21.1, one carbon atom was not assigned due to the overlap with the peaks of $CDCl_3$; ^{19}F NMR (376 MHz, $CDCl_3$) δ -115.5. HRMS (ESI) $[M+Na]^+$: calculated for $C_{16}H_{17}FNaO_2$: 283.1105, found 283.1109.



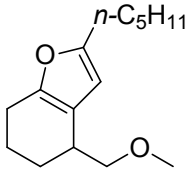
4-(methoxymethyl)-2-(naphthalen-2-yl)-4,5,6,7-tetrahydrobenzofuran (10j). The product **10j** was obtained in 76% (44.5 mg) yield as a colorless oil after column chromatography;^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (s, 1H), 7.86-7.72 (m, 4H), 7.49-7.39 (m, 2H), 6.74 (s, 1H), 3.56-3.52 (m, 1H), 3.47-3.43 (m, 4H), 2.97-2.90 (m, 1H), 2.75-2.67 (m, 2H), 2.06-1.98 (m, 1H), 1.97-1.89 (m, 1H), 1.87-1.76 (m, 1H), 1.60-1.50 (m, 1H).^{13}C NMR (125 MHz, $CDCl_3$) δ 151.9, 151.7, 133.6, 132.3, 128.6, 128.2, 128.0, 127.7, 126.3, 125.5, 122.2, 121.2, 120.6, 106.0, 76.8, 58.9, 33.7, 26.3, 23.3, 21.1.HRMS (ESI) $[M+H]^+$: calculated for $C_{20}H_{21}O_2$: 293.1536, found 293.1537.



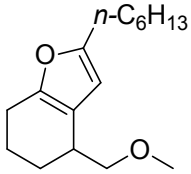
2-cyclopropyl-4-(methoxymethyl)-4,5,6,7-tetrahydrobenzofuran (10k). The product **10k** was obtained in 58% (24.0 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.87 (s, 1H), 3.45-3.42 (m, 1H), 3.37 (s, 3H), 3.34-3.31 (m, 1H), 2.81-2.76 (m, 1H), 2.53-2.50 (m, 2H), 1.94-1.79 (m, 3H), 1.75-1.67 (m, 1H), 1.50-1.43 (m, 1H), 0.83-0.79 (m, 2H), 0.74-0.68 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 155.2, 149.1, 118.5, 103.5, 77.2, 58.8, 33.7, 26.4, 23.1, 21.1, 8.8, 6.4, 6.3; HRMS (ESI) $[M+Na]^+$: calculated for $C_{13}H_{18}NaO_2$: 229.1199, found 229.1205.



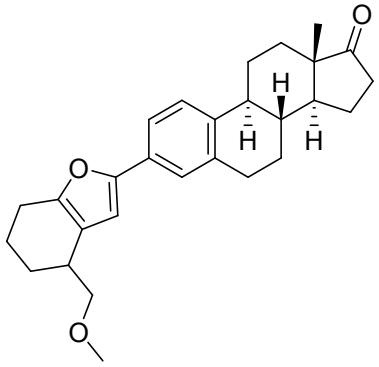
2-butyl-4-(methoxymethyl)-4,5,6,7-tetrahydrobenzofuran (10l). The product **10l** was obtained in 19% (8.6 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.88 (s, 1H), 3.47-3.44 (m, 1H), 3.38 (s, 3H), 3.35-3.32 (m, 1H), 2.83-2.78 (m, 1H), 2.57-2.52 (m, 4H), 1.95-1.82 (m, 2H), 1.76-1.68 (m, 1H), 1.62-1.56 (m, 2H), 1.51-1.44 (m, 1H), 1.39-1.35 (m, 2H), 0.92 (t, J = 7.3 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 154.4, 149.2, 118.3, 104.4, 76.8, 58.8, 33.7, 30.3, 27.9, 26.4, 23.1, 22.4, 21.1, 13.9; HRMS (ESI) $[M+H]^+$: calculated for $C_{14}H_{23}O_2$: 223.1693, found 223.1692.



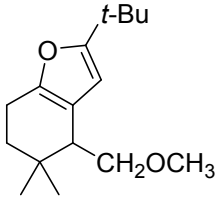
4-(methoxymethyl)-2-pentyl-4,5,6,7-tetrahydrobenzofuran (10m). The product **10m** was obtained in 46% (21.7 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.89 (s, 1H), 3.47-3.44 (m, 1H), 3.38 (s, 3H), 3.36-3.32 (m, 1H), 2.84-2.79 (m, 1H), 2.56-2.52 (m, 4H), 1.95-1.83 (m, 2H), 1.76-1.69 (m, 1H), 1.64-1.60 (m, 2H), 1.52-1.43 (m, 1H), 1.35-1.32 (m, 4H), 0.92-0.89 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 154.4, 149.2, 118.4, 104.4, 76.8, 58.8, 33.7, 31.5, 28.2, 27.9, 26.4, 23.1, 22.5, 21.2, 14.0. HRMS (ESI) $[M+H]^+$: calculated for $C_{15}H_{25}O_2$: 237.1849, found 237.1848.



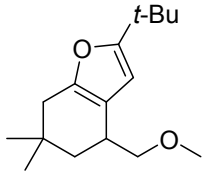
2-hexyl-4-(methoxymethyl)-4,5,6,7-tetrahydrobenzofuran (10n). The product **10n** was obtained in 70% (35.2 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.89 (s, 1H), 3.48-3.43 (m, 1H), 3.38 (s, 3H), 3.36-3.32 (m, 1H), 2.84-2.78 (m, 1H), 2.56-2.51 (m, 4H), 1.95-1.82 (m, 2H), 1.77-1.68 (m, 1H), 1.64-1.57 (m, 2H), 1.52-1.45 (m, 1H), 1.38-1.25 (m, 6H), 0.89 (t, J = 6.5 Hz, 3H).^{13}C NMR (125 MHz, $CDCl_3$) δ 154.4, 149.2, 118.4, 104.4, 76.8, 58.8, 33.7, 31.6, 29.0, 28.2(3), 28.2(1), 26.4, 23.1, 22.6, 21.2, 14.1. HRMS (ESI) $[M+H]^+$: calculated for $C_{16}H_{27}O_2$: 251.2006, found 251.2011.



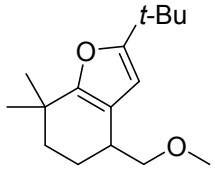
(8R,9S,13S,14S)-3-(4-(methoxymethyl)-4,5,6,7-tetrahydrobenzofuran-2-yl)-13-methyl-6,7,8,9,11,12,13,14,15,16-decahydro-17H-cyclopenta[*a*]phenanthren-17-one (10o). The product **10o** was obtained in 66% (55.2 mg) yield as a pale yellow solid after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44-7.36 (m, 2H), 7.29-7.28(s, 1H), 6.57 (s, 1H), 3.55-3.50 (m, 1H), 3.44-3.41 (m, 4H), 2.99-2.87 (m, 3H), 2.71-2.63 (m, 2H), 2.57-2.49 (m, 1H), 2.49-2.42 (m, 1H), 2.37-2.28 (m, 1H), 2.22-2.13 (m, 1H), 2.13-1.97 (m, 4H), 1.96-1.89 (m, 1H), 1.85-1.75 (m, 1H), 1.70-1.46 (m, 7H), 0.95 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 220.5, 151.7, 150.8, 138.1, 136.4, 128.8, 125.4, 123.6, 120.8, 120.1, 104.6, 76.8, 58.9, 50.6, 48.1, 44.5, 38.2, 35.9, 33.8, 31.7, 29.5, 26.6, 26.4, 25.8, 23.4, 21.7, 21.2, 14.0. HRMS (ESI) $[M+H]^+$: calculated for $C_{28}H_{35}O_3$: 419.2581, found 419.2583.



2-(*tert*-butyl)-4-(methoxymethyl)-5,5-dimethyl-4,5,6,7-tetrahydrobenzofuran (11a). The product **11a** was obtained in 86% (43.1 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.89 (s, 1H), 3.60- 3.54 (m, 1H), 3.39-3.32 (m, 4H), 2.54 -2.44 (m, 3H), 1.60-1.57 (m, 2H), 1.25 (s, 9H), 1.06 (s, 3H), 0.85 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.3, 147.3, 118.5, 102.6, 74.7, 58.6, 43.3, 36.9, 32.5, 32.4, 29.2, 28.6, 22.2, 20.5. HRMS (ESI) $[M+H]^+$: calculated for $C_{16}H_{27}O_2$: 251.2006, found 251.2002.

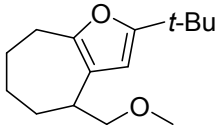


2-(*tert*-butyl)-4-(methoxymethyl)-6,6-dimethyl-4,5,6,7-tetrahydrobenzofuran (11b). The product **11b** was obtained in 64% (32.1 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.87 (s, 1H), 3.58-3.54 (m, 1H), 3.40 (s, 3H), 3.35-3.30 (m, 1H), 2.82-2.74 (m, 1H), 2.41-2.27 (m, 2H), 1.62-1.57 (m, 1H), 1.24 (s, 9H), 1.21-1.14 (m, 1H), 1.08 (s, 3H), 0.94 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 162.5, 148.8, 116.5, 101.0, 77.3, 58.9, 41.0, 36.9, 32.5, 31.8, 31.7, 31.4, 29.2, 25.6. HRMS (ESI) $[M+H]^+$: calculated for $C_{16}H_{27}O_2$: 251.2006, found 251.2006.

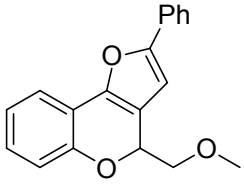


2-(*tert*-butyl)-4-(methoxymethyl)-7,7-dimethyl-4,5,6,7-tetrahydrobenzofuran (11c). The product **11c** was obtained in 64% (32.0 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.79 (s, 1H), 3.50-3.47 (m,

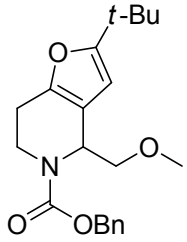
1H), 3.38 (s, 3H), 3.38-3.28 (m, 1H), 2.81-2.76 (m, 1H), 1.88-1.82 (m, 1H), 1.69-1.63 (m, 1H), 1.59-1.52 (m, 2H), 1.24 (s, 9H), 1.22 (s, 3H), 1.20 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.6, 155.5, 115.9, 101.1, 76.8, 58.8, 37.4, 34.3, 32.7, 32.2, 29.2, 27.8, 23.9. HRMS (ESI) $[M+H]^+$: calculated for $C_{16}H_{27}O_2$: 251.2006, found 251.2016.



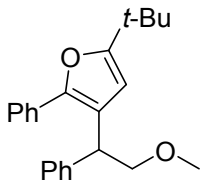
2-(*tert*-butyl)-4-(methoxymethyl)-5,6,7,8-tetrahydro-4H-cyclohepta[b]furan (11d). The product **11d** was obtained in 32% (15.2 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.76 (s, 1H), 3.51-3.48 (m, 1H), 3.47-3.40 (m, 1H), 3.38 (s, 3H), 2.81-2.76 (m, 1H), 2.75-2.69 (m, 2H), 1.89-1.65 (m, 6H), 1.23 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.0, 150.8, 120.8, 103.6, 75.5, 58.7, 36.4, 32.4, 30.9, 29.3, 28.9, 27.3, 26.6. HRMS (ESI) $[M+H]^+$: calculated for $C_{15}H_{25}O_2$: 237.1849, found 237.1855.



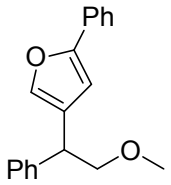
4-(methoxymethyl)-2-phenyl-4H-furo[3,2-*c*]chromene (11f). The product **11f** was obtained in 11% (6.4 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.71 (d, J = 7.5 Hz, 2H), 7.48 (d, J = 7.5 Hz, 1H), 7.42-7.39 (m, 2H), 7.30-7.27 (m, 1H), 7.14-7.11 (m, 1H), 6.98-6.94 (m, 2H), 6.58 (s, 1H), 5.70-5.67 (m, 1H), 3.84-3.80 (m, 1H), 3.73-3.70 (m, 1H), 3.48 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 154.4, 152.3, 145.8, 130.3, 128.7, 128.6, 127.6, 123.7, 121.4, 119.4, 116.5, 116.2, 116.0, 103.4, 75.8, 75.1, 59.6. HRMS (ESI) $[M+H]^+$: calculated for $C_{19}H_{17}O_3$: 293.1172, found 293.1179.



benzyl-2-(*tert*-butyl)-4-(methoxymethyl)-6,7-dihydrofuro[3,2-*c*]pyridine-5(4H)-carboxylate (11g). The product **11g** was obtained in 76% (54.2 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$, isolated as a mixture of rotamers) δ 7.42-7.29 (m, 5H), 5.85-5.83 (m, 1H), 5.27-5.10 (m, 3H), 4.59-4.36 (m, 1H), 3.68-3.48 (m, 2H), 3.38 (m, 3H), 3.30-3.17 (m, 1H), 2.78 (br, 1H), 2.60-2.50 (m, 1H), 1.27 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$, observed as a mixture of rotamers) δ 163.0, 155.4, 155.2, 147.0, 146.5, 136.6, 128.3 , 127.8, 127.7, 115.8, 115.5, 100.8, 74.1, 73.8, 67.3 , 59.1 , 50.8 , 38.9, 38.6, 32.7 , 29.2 , 24.0, 23.7. HRMS (ESI) $[M+H]^+$: calculated for $C_{21}H_{28}NO_4$: 358.2013, found 358.2013.



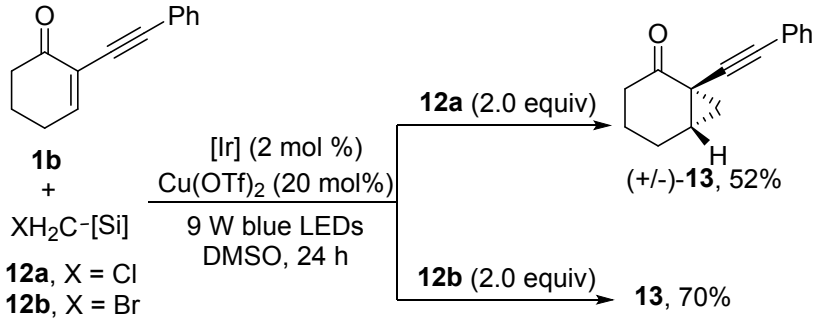
5-(*tert*-butyl)-3-(2-methoxy-1-phenylethyl)-2-phenylfuran (11h). The product **11h** was obtained in 8% (5.9 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.53-7.51 (m, 2H), 7.36-7.32 (m, 6H), 7.26-7.21 (m, 2H), 6.10 (s, 1H), 4.45 (t, J = 7.1 Hz, 1H), 3.88-3.77 (m, 2H), 3.34 (s, 3H), 1.34 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.9, 147.2, 142.0, 131.5, 128.3, 128.2, 128.0, 126.6, 126.4, 125.7, 121.3, 104.1, 76.9, 58.9, 42.3, 32.8, 29.3. HRMS (ESI) $[M+H]^+$: calculated for $C_{23}H_{27}O_2$: 335.2006, found 335.2003.



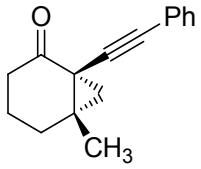
5-(2-methoxy-1-phenylethyl)-2-phenylfuran (11i). The product **11i** was obtained in 26% (14.6 mg) yield as a colorless oil after column chromatography; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.64-7.62 (m, 2H), 7.37-7.22 (m, 9H), 6.54 (s, 1H), 4.16 (t, J = 7.0 Hz, 1H), 3.88-3.79 (m, 2H), 3.40 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 153.9, 141.5, 139.1, 130.9, 128.6, 128.5, 128.2, 128.1, 127.2, 126.8, 123.7, 106.1, 76.5, 58.9, 42.8. HRMS (ESI) $[M+H]^+$: calculated for $C_{19}H_{19}O_2$: 279.1380, found 279.1383.

5 Mechanistic experiments

5.1 Cyclopropanation *vs* cycloisomerization

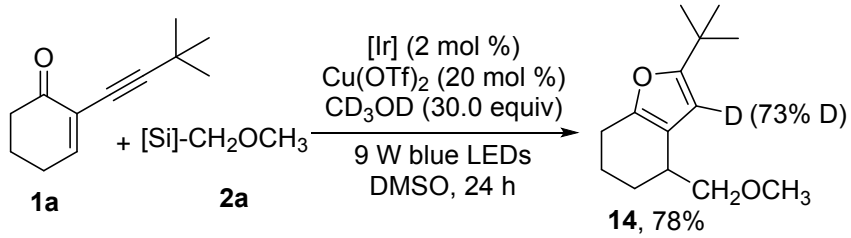


To an oven dried transparent 10 mL Schlenk tube equipped with stirring bar, $Ir[dF(CF_3)ppy)]_2(dtbbpy)PF_6$ (4.5 mg, 0.004 mmol, 2 mol %) and the 2-(3,3-dimethylbut-1-yn-1-yl)cyclohex-2-en-1-one **1a** (39.2mg, 0.2 mmol, 1.0 equiv) were added. In a glovebox, **12a** or **12b** (0.4 mmol, 2.0 equiv) and $Cu(OTf)_2$ (14.4 mg, 0.04 mmol, 20 mol %) were added in the tube. The tube was sealed with a rubber septum and removed from the glovebox. The tube was then charged with degassed DMSO (6.0 mL, 0.033 M) via a syringe under N_2. The tube was irradiated with a 9 W blue LEDs strip spiraled within a bowel for 24 h (cooling with a fan). After the reaction was complete, the reaction solution was diluted with saturated Na_2CO_3 aqueous solution, and was extracted with EtOAc (4 x 5 mL). The organic layer was washed with brine, dried over $MgSO_4$, filtered, and solvent was evaporated to obtain crude product. Flash chromatography over silica gel afforded the product **13** (eluent = petroleum ether /ethyl acetate 20:1 v/v).



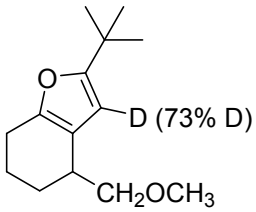
(1*R, 6*S**)-6-methyl-1-(phenylethynyl)bicyclo[4.1.0]heptan-2-one (13)**.[15] The product **13** was obtained as a pale yellow solid after column chromatography; ^1H NMR (500 MHz, $CDCl_3$) δ 7.46-7.43 (m, 2H), 7.30 -7.27 (m, 3H), 2.44-2.39 (m, 1H), 2.26-2.19 (m, 2H), 2.13-2.02 (m, 2H), 1.82-1.77 (m, 2H), 1.69-1.64 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 203.8, 131.9, 128.1, 127.9, 123.2, 89.2, 79.8, 36.4, 29.4, 27.0, 21.3(3), 21.2(7), 18.3.

5.2 Deuteration study



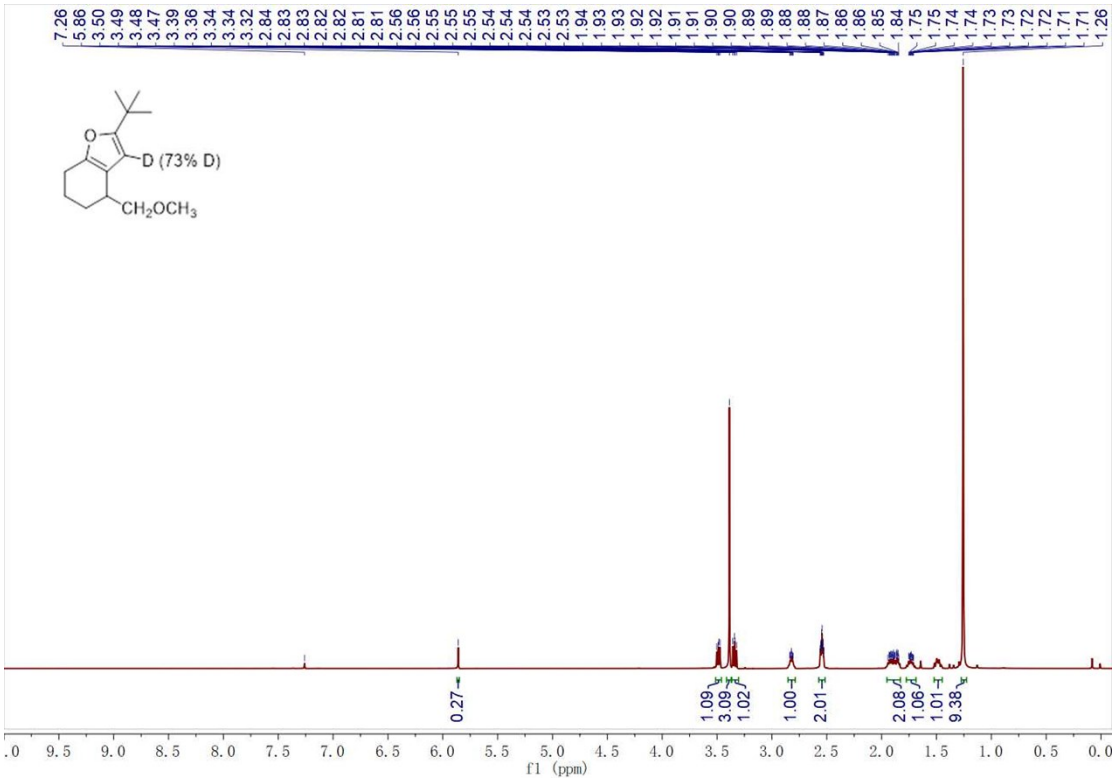
1a **2a** **14**, 78%

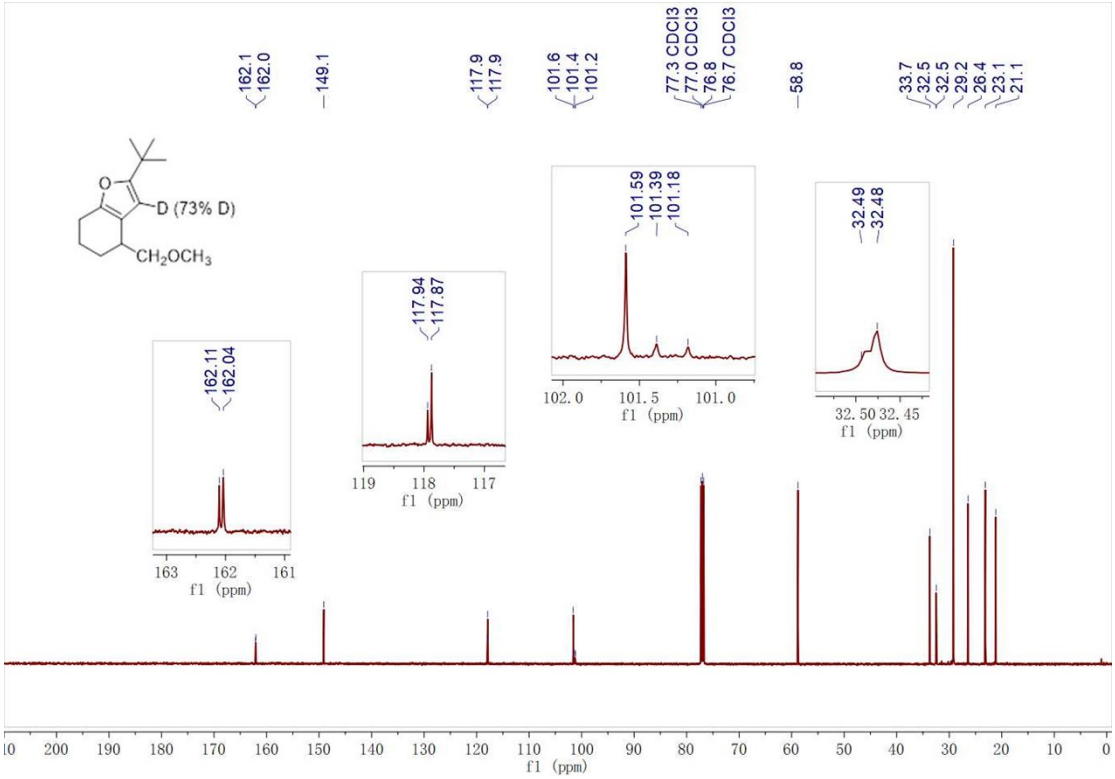
To an oven dried transparent 10 mL Schlenk tube equipped with stirring bar, $Ir[dF(CF_3)ppy)]_2(dtbbpy)PF_6$ (4.5 mg, 0.004 mmol, 2 mol %) and the 2-(3,3-dimethylbut-1-yn-1-yl)cyclohex-2-en-1-one **1a** (0.2 mmol, 1.0 equiv) were added. In a glovebox, potassium [18-Crown-6] bis(catecholato) alkylsilicate **2a** (238.9 mg, 0.4 mmol, 2.0 equiv) and $Cu(OTf)_2$(14.4 mg, 0.04 mmol, 20 mol %) were added in the tube. The tube was sealed with a rubber septum and removed from the glovebox. The tube was then charged with degassed DMSO (6.0 mL, 0.033 M) via a syringe under N_2. Then CD_3OD (244.0 μL, 6 mmol, 30.0 equiv) was added via a micro syringe. The tube was irradiated with a 9 W blue LEDs strip spiraled within a bowel for 24 (cooling with a fan). After the reaction was complete, the reaction solution was diluted with saturated Na_2CO_3 aqueous solution, and was extracted with EtOAc (4 x 5 mL). The organic layer was washed with brine, dried over $MgSO_4$, filtered, and solvent was evaporated to obtain crude product. Flash chromatography over silica gel afforded the product **14** in 78% yield (33.1 mg) (eluent = petroleum ether /ethyl acetate 100:1 v/v). The product **14** with 73% D-incorporation was determined by ^1H NMR.

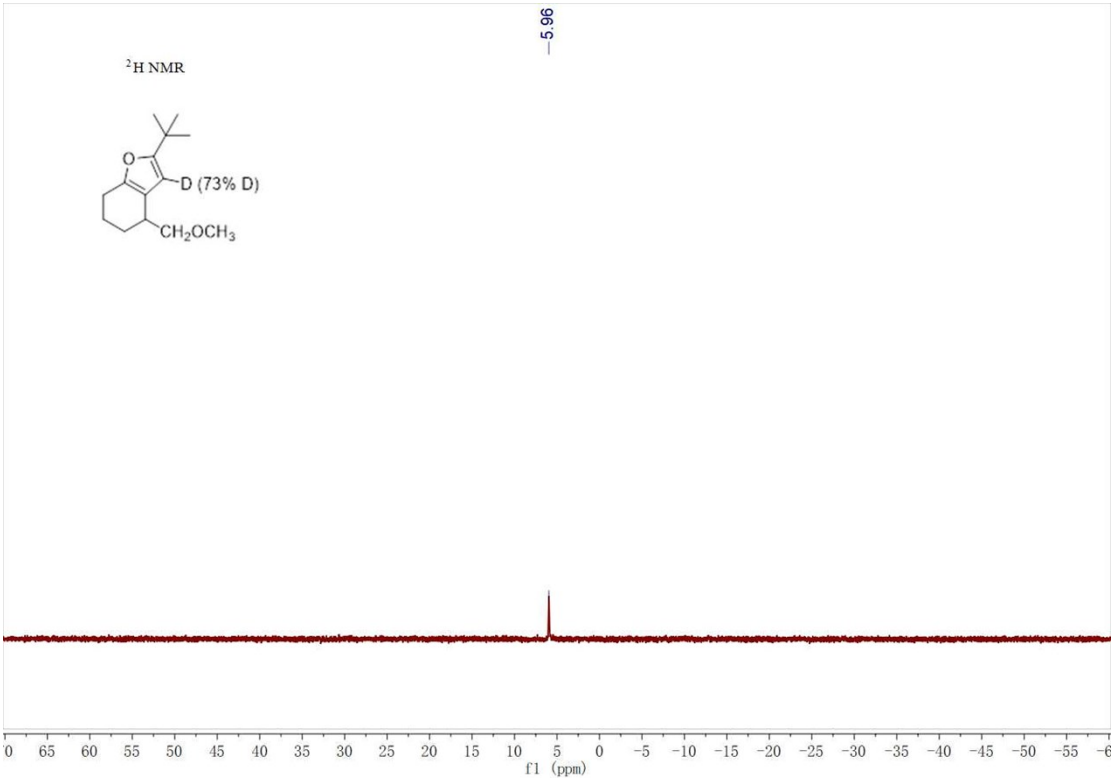


2-(*tert*-butyl)-4-(methoxymethyl)-4,5,6,7-tetrahydrobenzofuran-3-*d* (14), colorless oil. The product **14** was obtained as an inseparable mixture after column chromatography; ^1H NMR (500 MHz, $CDCl_3$) δ 5.84 (s, 0.27H), 3.49-3.45 (m, 1H), 3.37 (s, 3H), 3.34-3.29 (m, 1H), 2.83-2.76 (m, 1H), 2.52 (t, J = 5.7 Hz, 2H), 1.95-1.79 (m, 2H), 1.78-1.65 (m, 1H), 1.51-1.41 (m, 1H), 1.23 (s, 9H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 162.1, 162.0, 149.1, 117.9(4), 117.8(7), 101.6,101.4 (t, J =25.7 Hz, C-D), 76.8, 58.8, 33.7, 32.4(9), 32.4(8), 29.2, 26.4, 23.1, 21.1. ^2H NMR (61 MHz, $CHCl_3$) δ

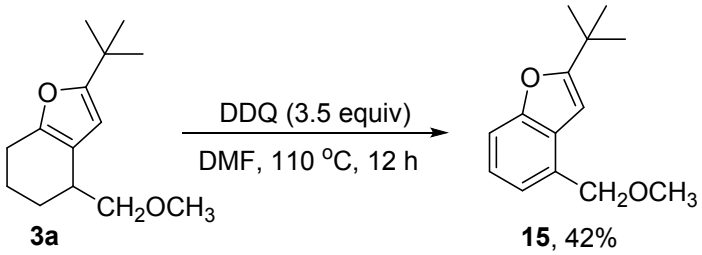
5.96. HRMS (ESI) $[M+H]^+$: calculated for $C_{14}H_{22}DO_2$: 224.1755, found 224.1753







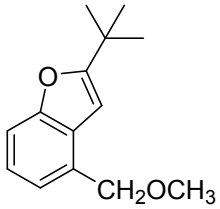
6 Dehydrogenation of furan product



3a **15**, 42%

To an oven dried transparent 10 mL Schlenk tube equipped with stirring bar, 2-(tert-butyl)-4-(methoxymethyl)-4,5,6,7-tetrahydrobenzofuran **3a** (22.3 mg, 0.1 mmol, 1.0 equiv) was added. The tube was evacuated and filled with nitrogen for 3 times, The tube was then charged with DMF (3.0 mL, 0.033 M) via a syringe under N_2. Then DDQ (79.5 mg, 0.35 mmol, 3.5 equiv) was added under N_2. The resulting mixture was stirred at 110 °C for 12 h, After the reaction was complete, the reaction solution was extracted with EtOAc (3 x 5 mL). The organic layer was washed with brine, dried over $MgSO_4$, filtered, and solvent was evaporated to obtain crude product. Flash chromatography over silica gel afforded the product **15** (eluent = petroleum ether

/ethyl acetate 100:1 v/v).



2-(*tert*-butyl)-4-(methoxymethyl)benzofuran (15). The product **15** was obtained in 42% (9.2 mg) yield as a colorless oil after column chromatography. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (d, J = 7.9 Hz, 1H), 7.21-7.07 (m, 2H), 6.46 (s, 1H), 4.65 (s, 2H), 3.40 (s, 3H), 1.37 (s, 9H).^{13}C NMR (125 MHz, $CDCl_3$) δ 167.5, 154.7, 130.1, 127.9, 122.8, 121.8, 110.4, 97.5, 73.0, 58.1, 33.0, 28.8. HRMS (ESI) $[M+H]^+$: calculated for $C_{14}H_{19}O_2$: 219.1380, found 219.1379.

7 Reference

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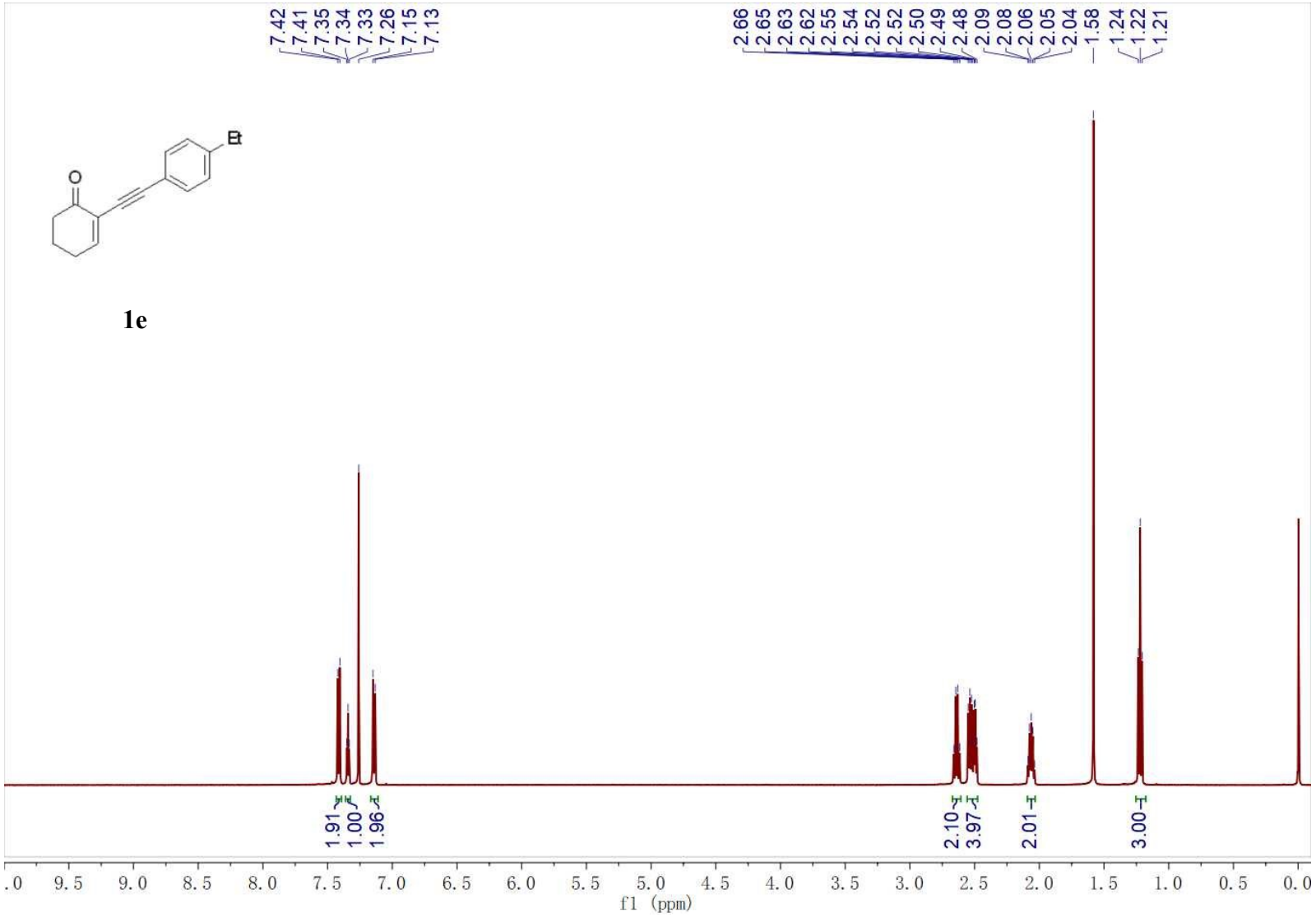
8 NMR spectra of new compounds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1.24
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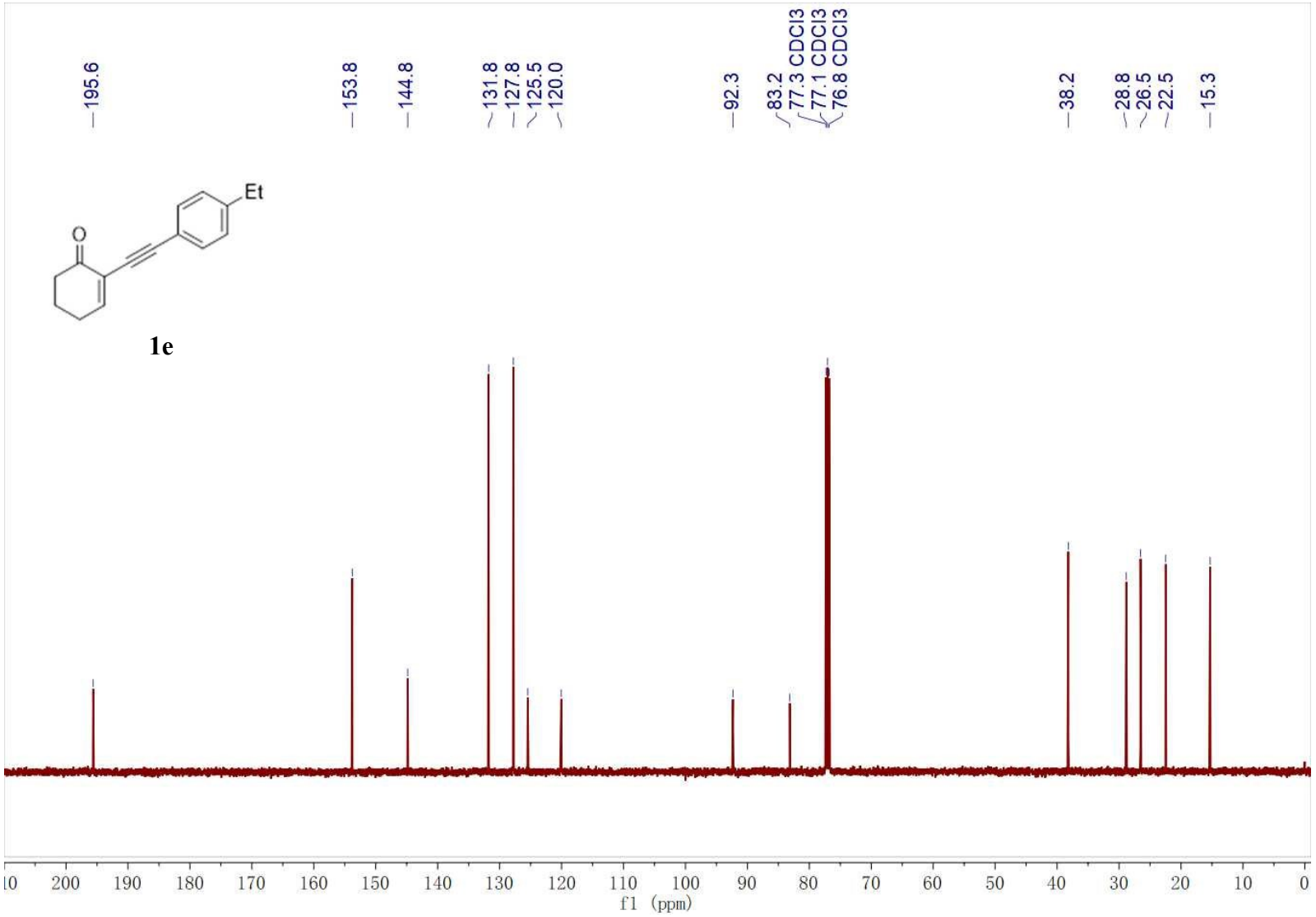
f1 (ppm)

1e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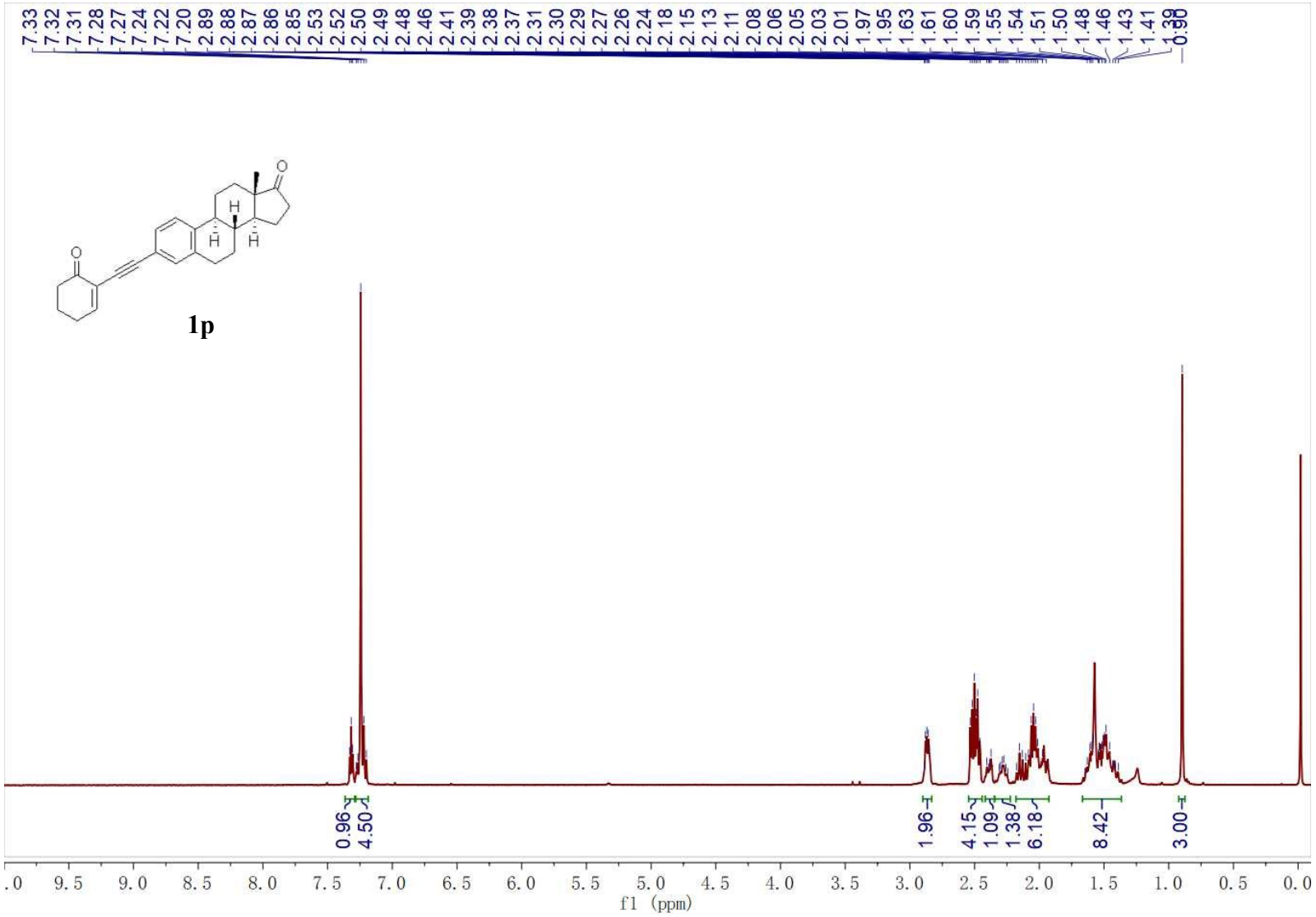
f1 (ppm)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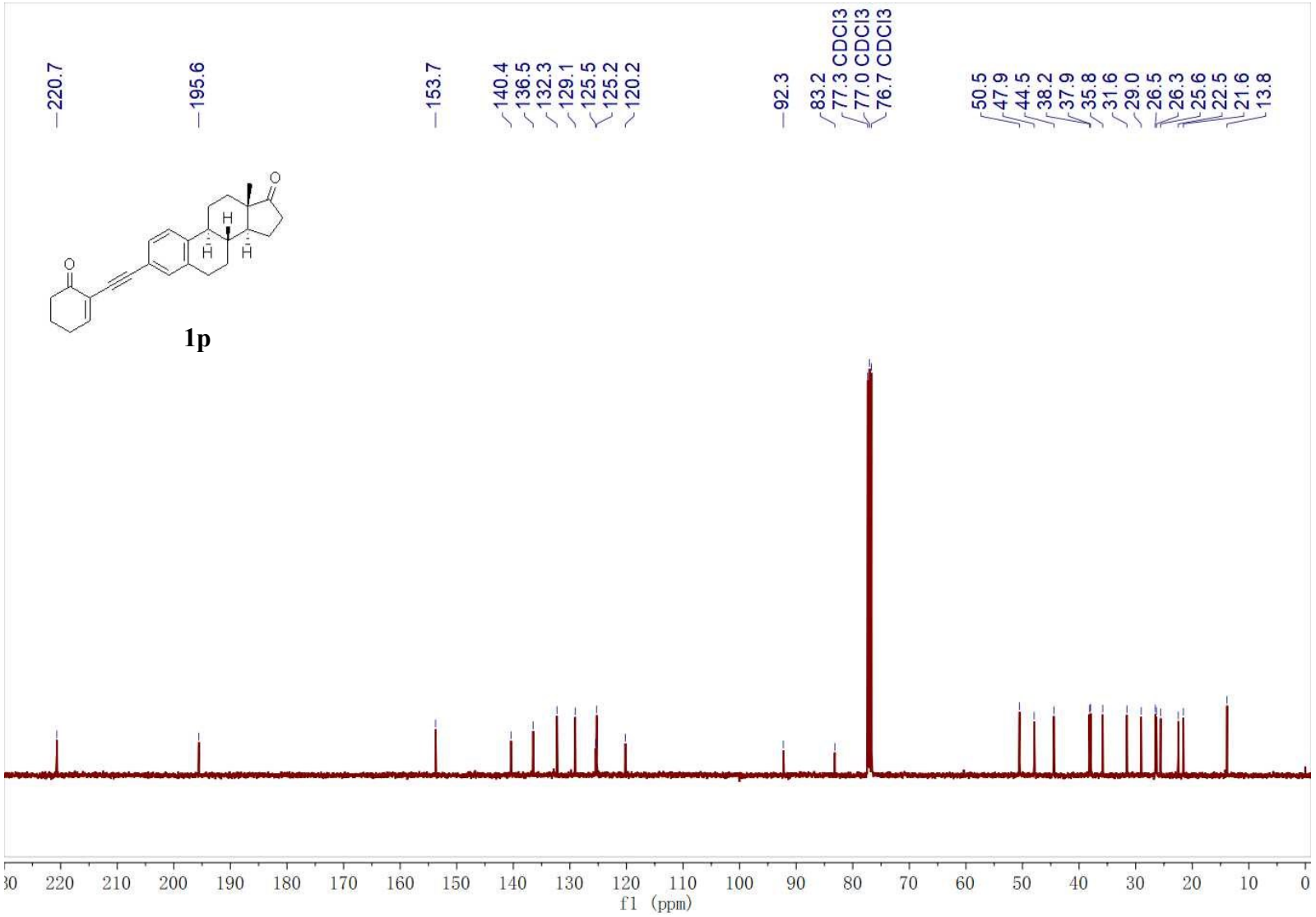
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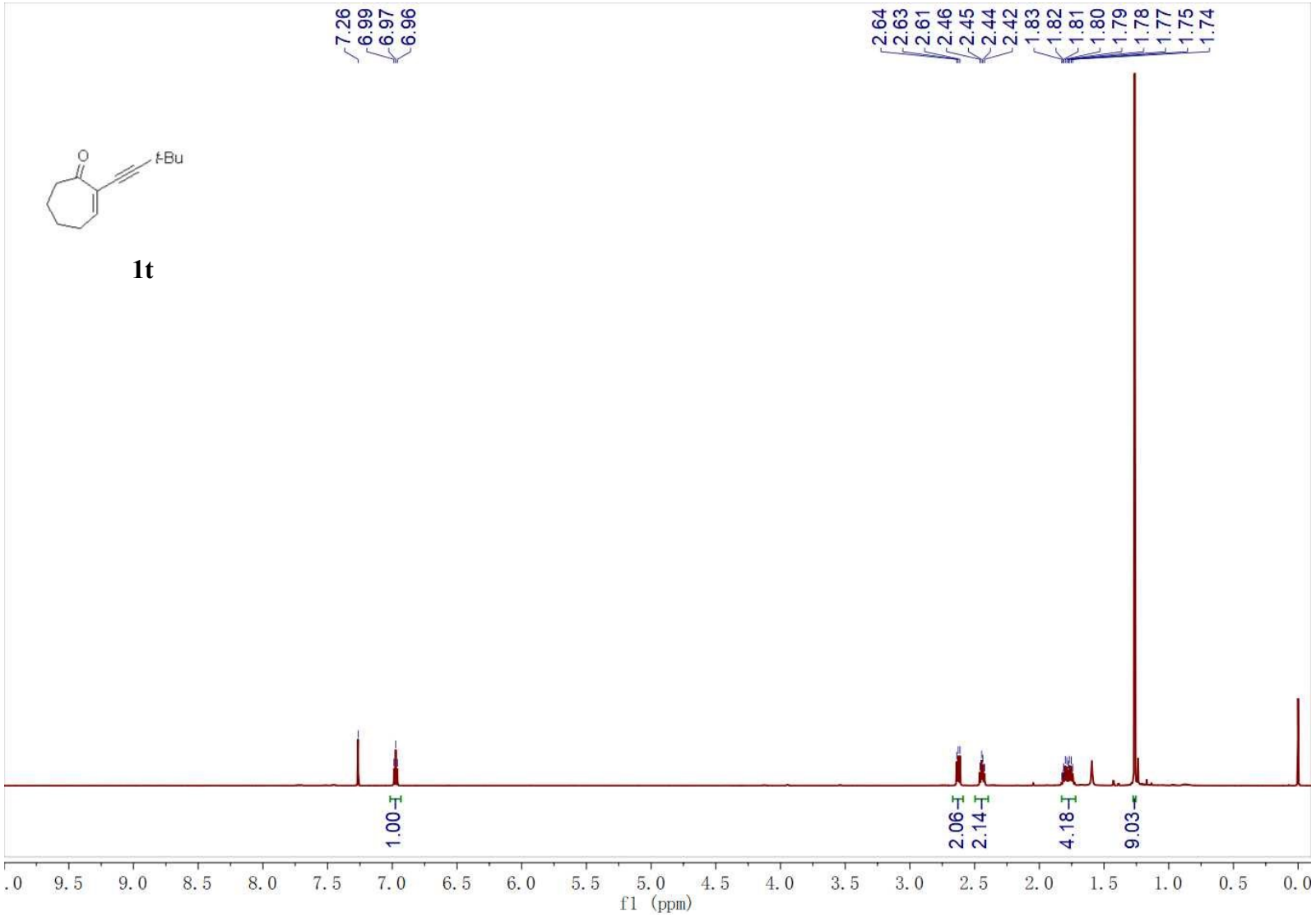
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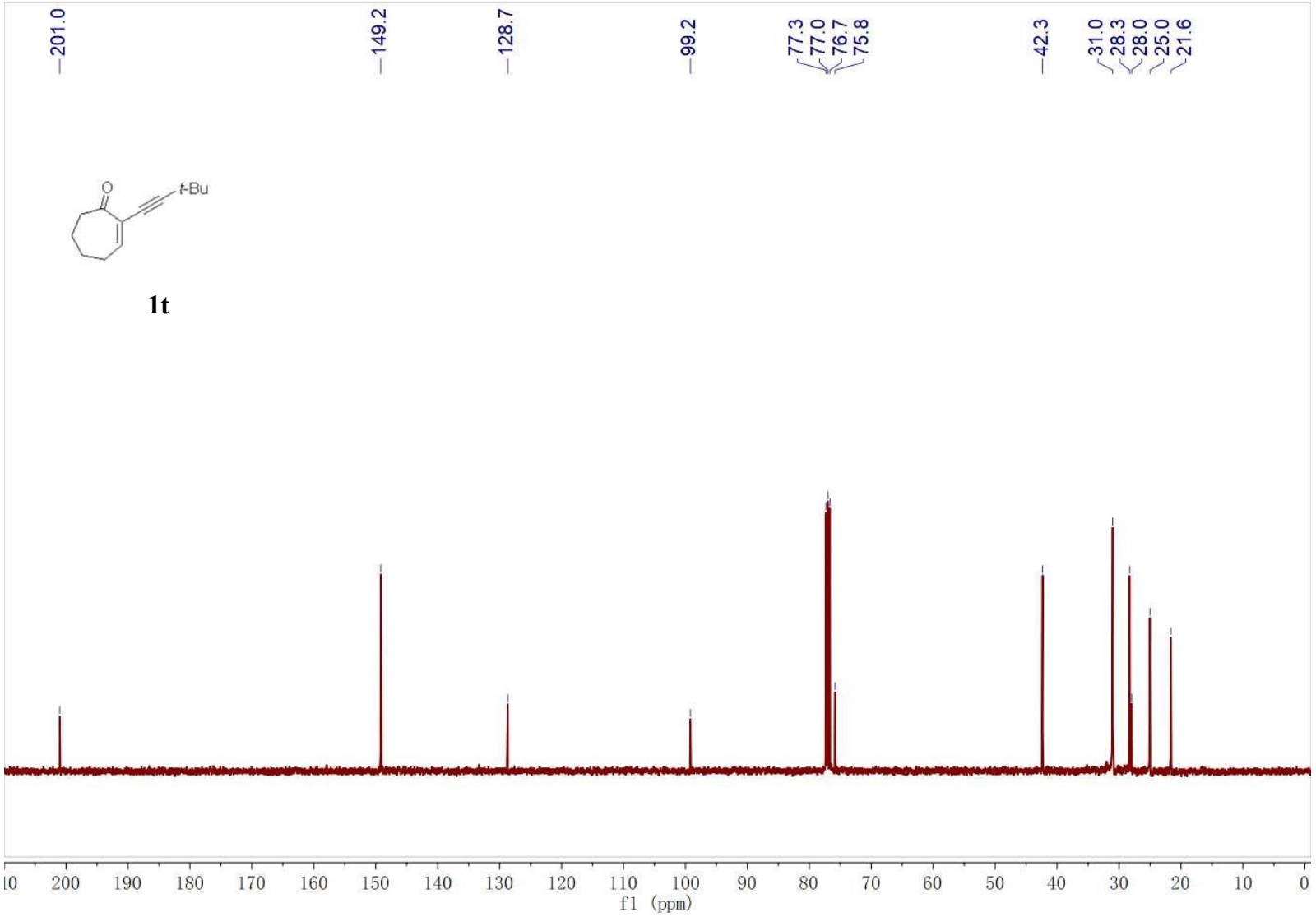
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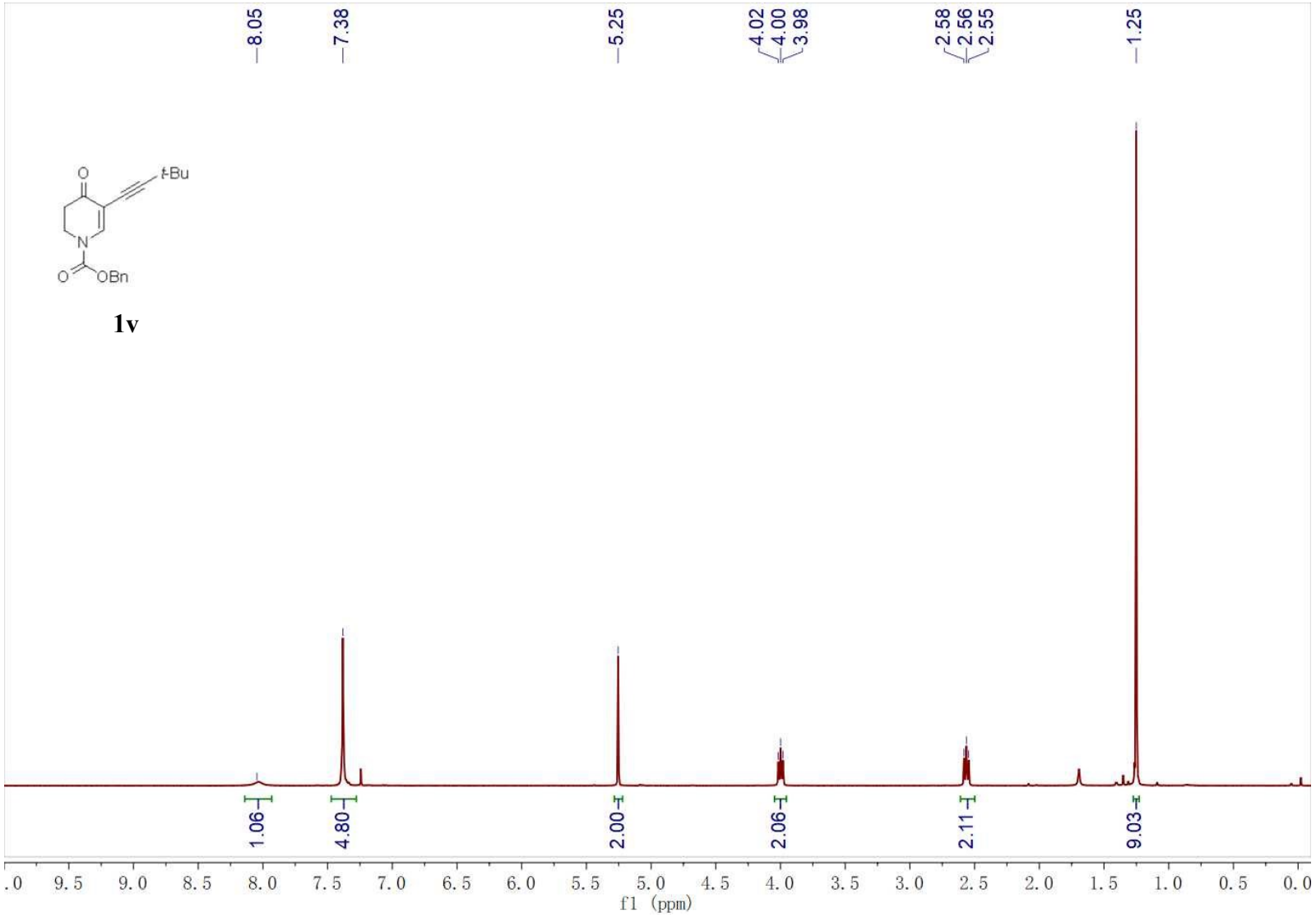
f1 (ppm)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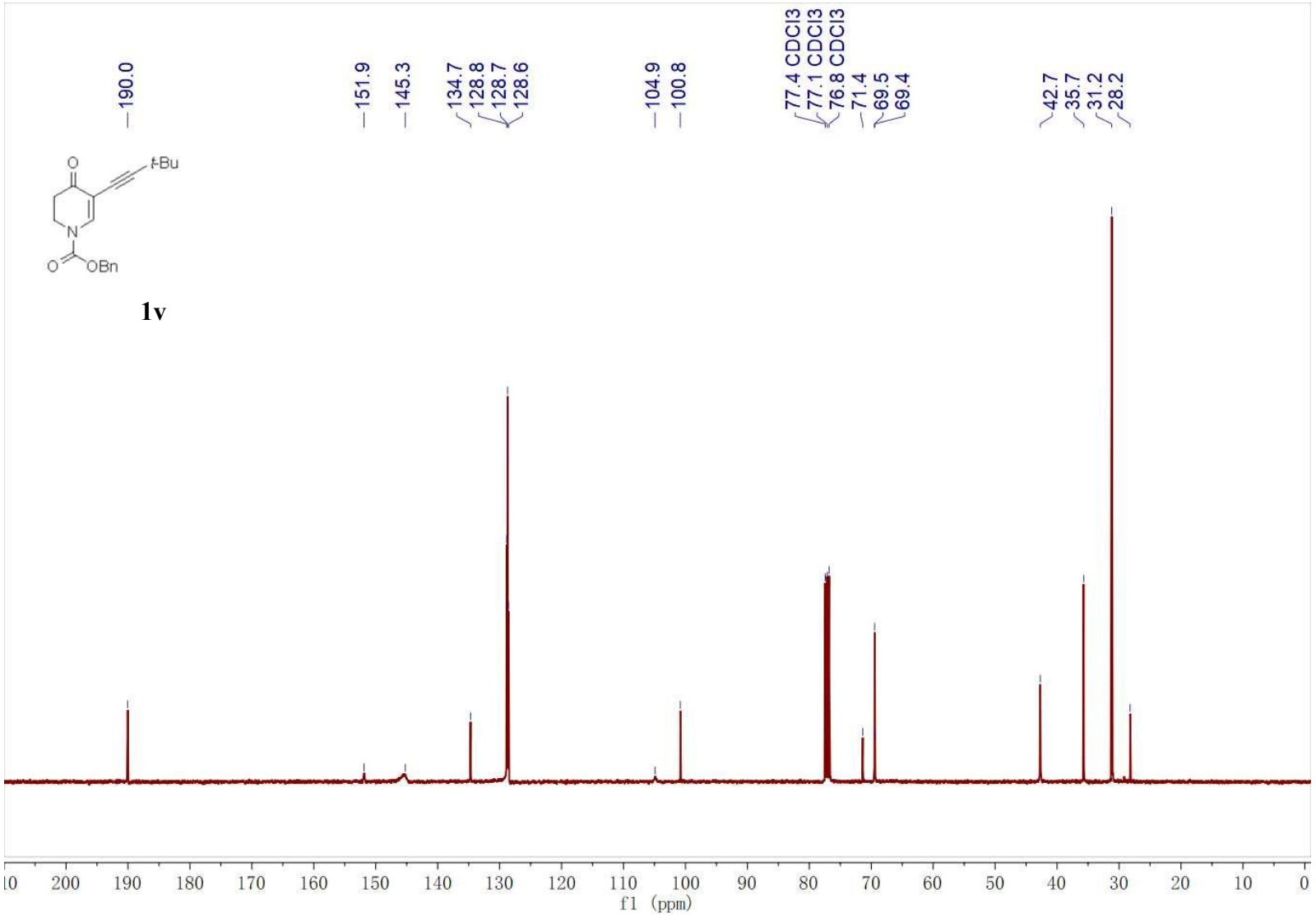
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76.8 CDCl3
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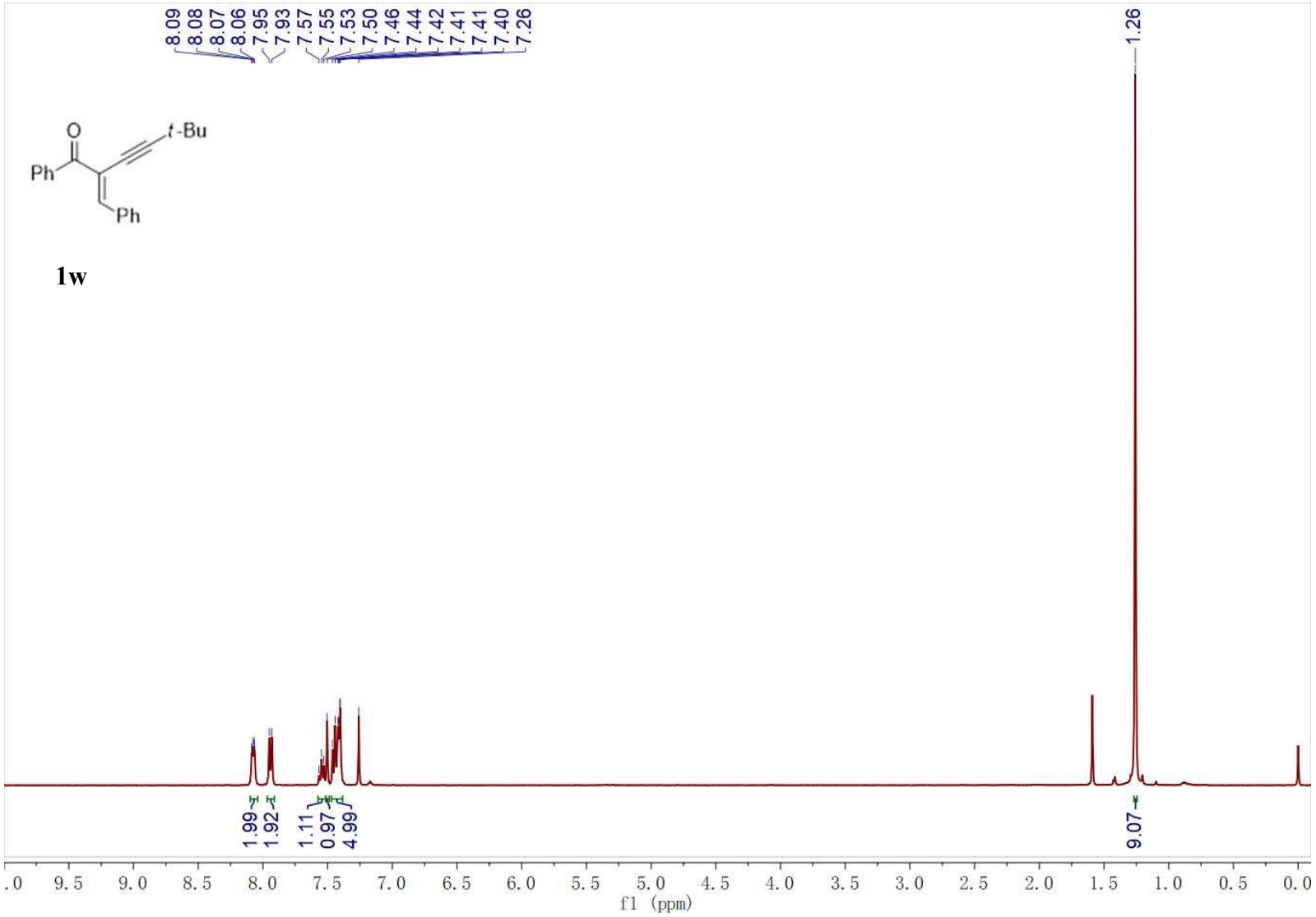
O
N
O
OBn
f1 (ppm)

1v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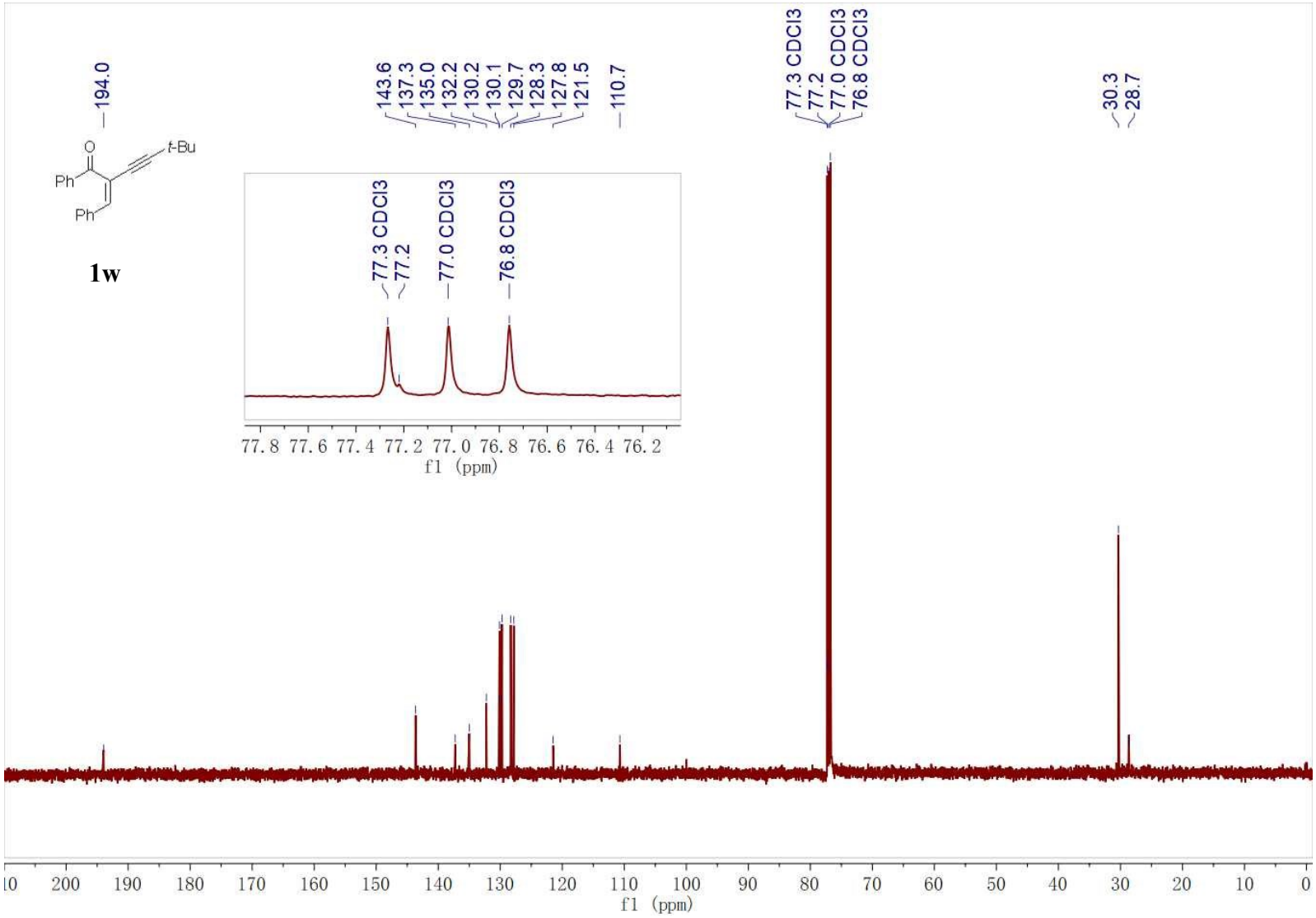
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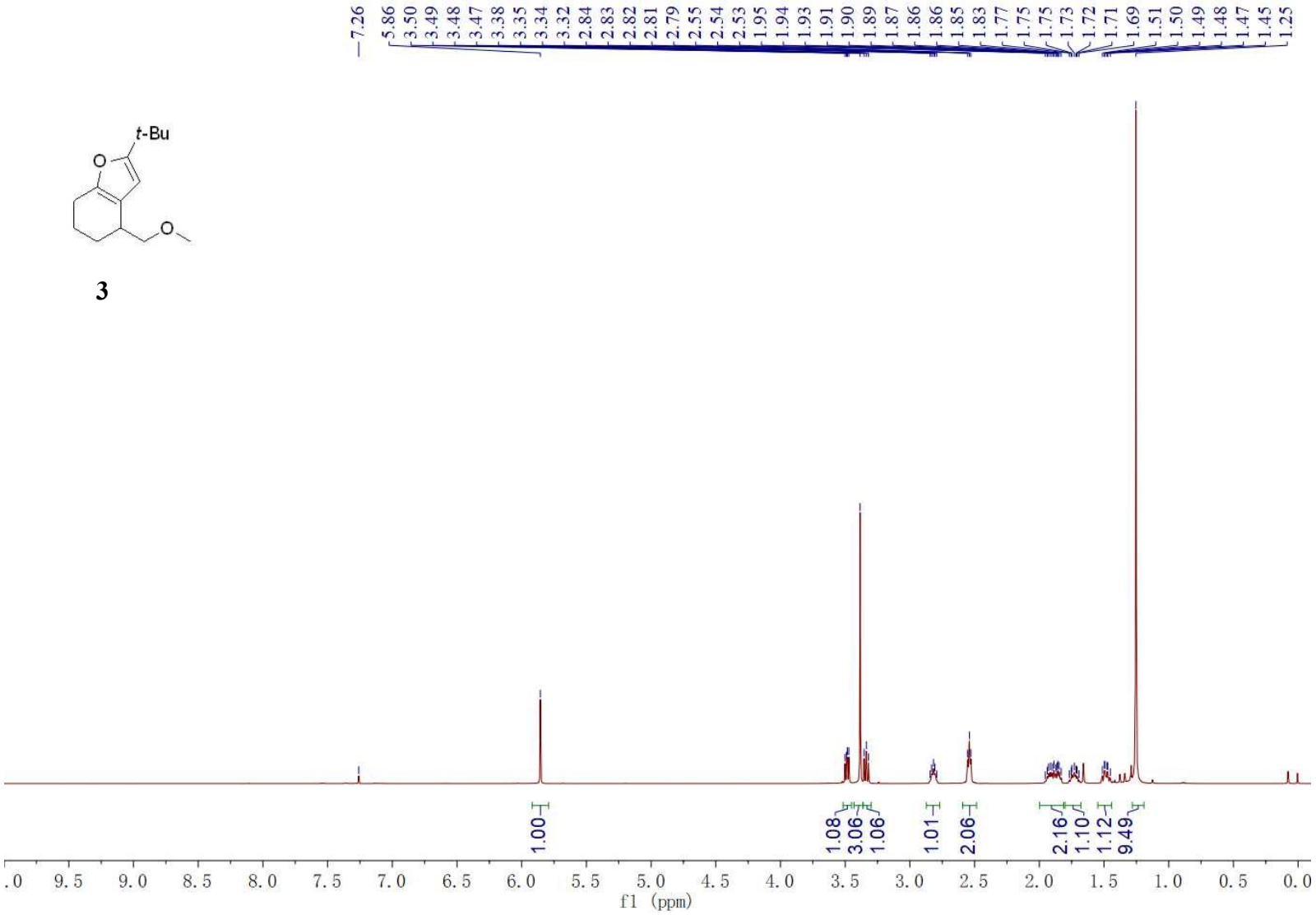
f1 (ppm)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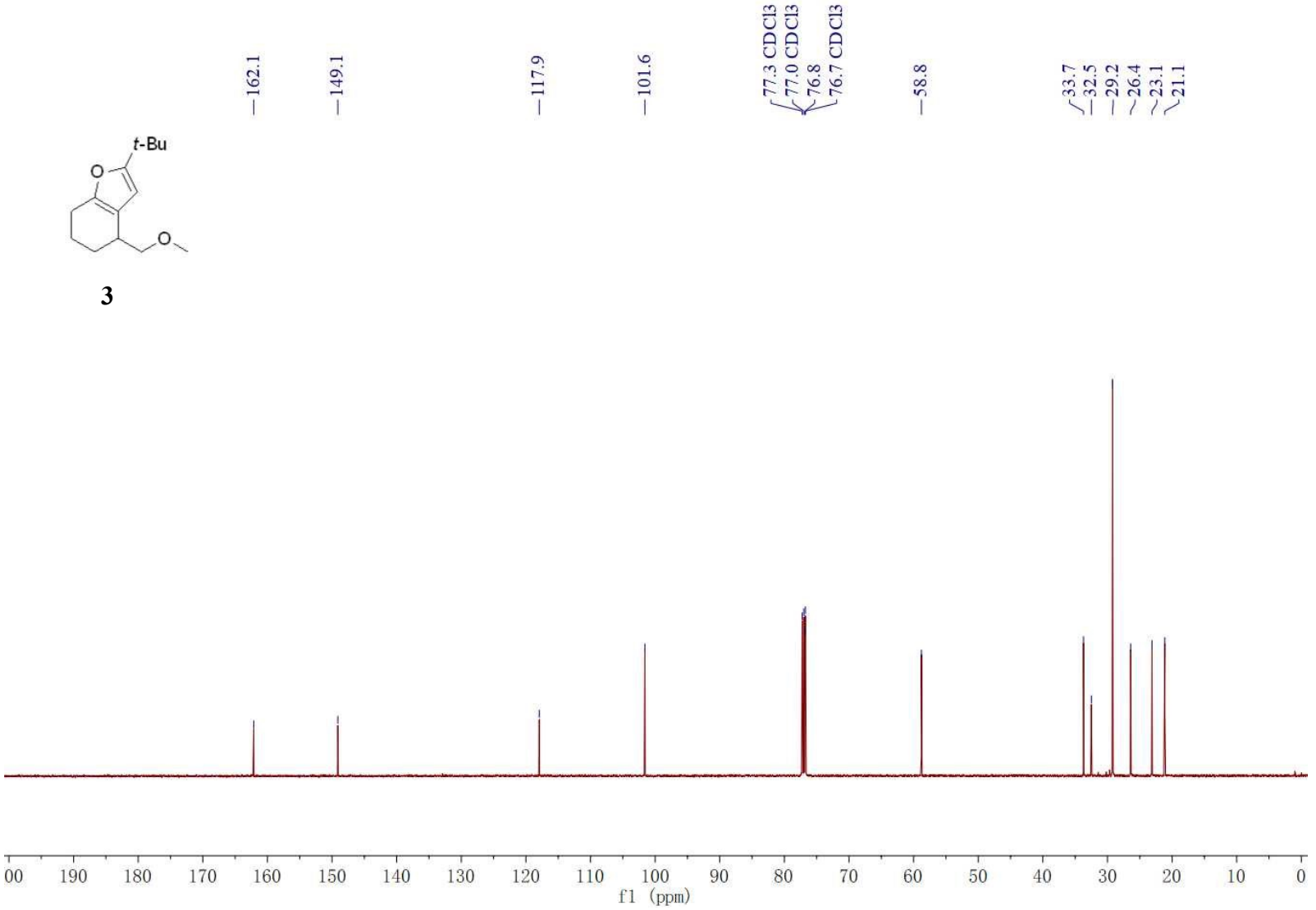
3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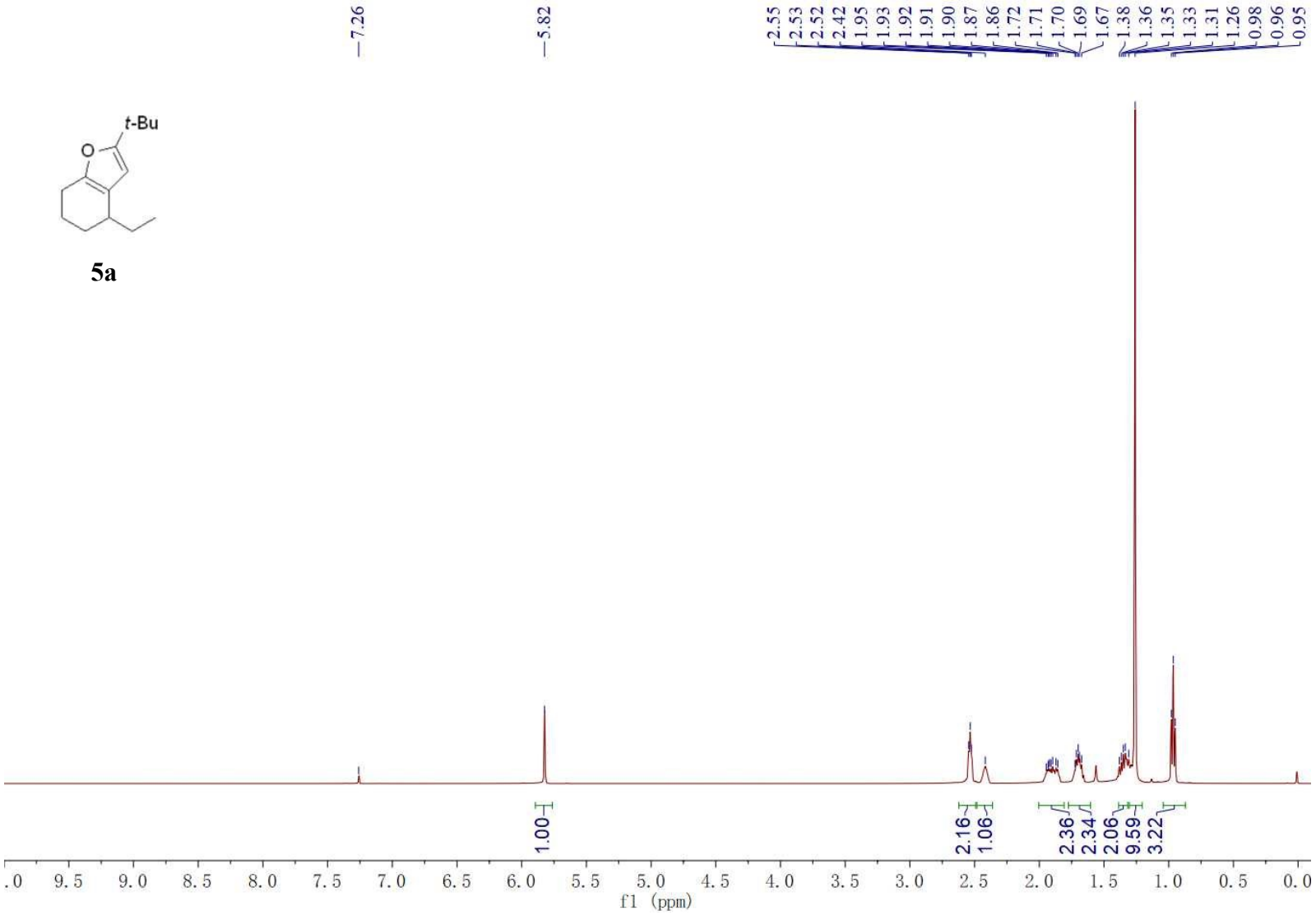
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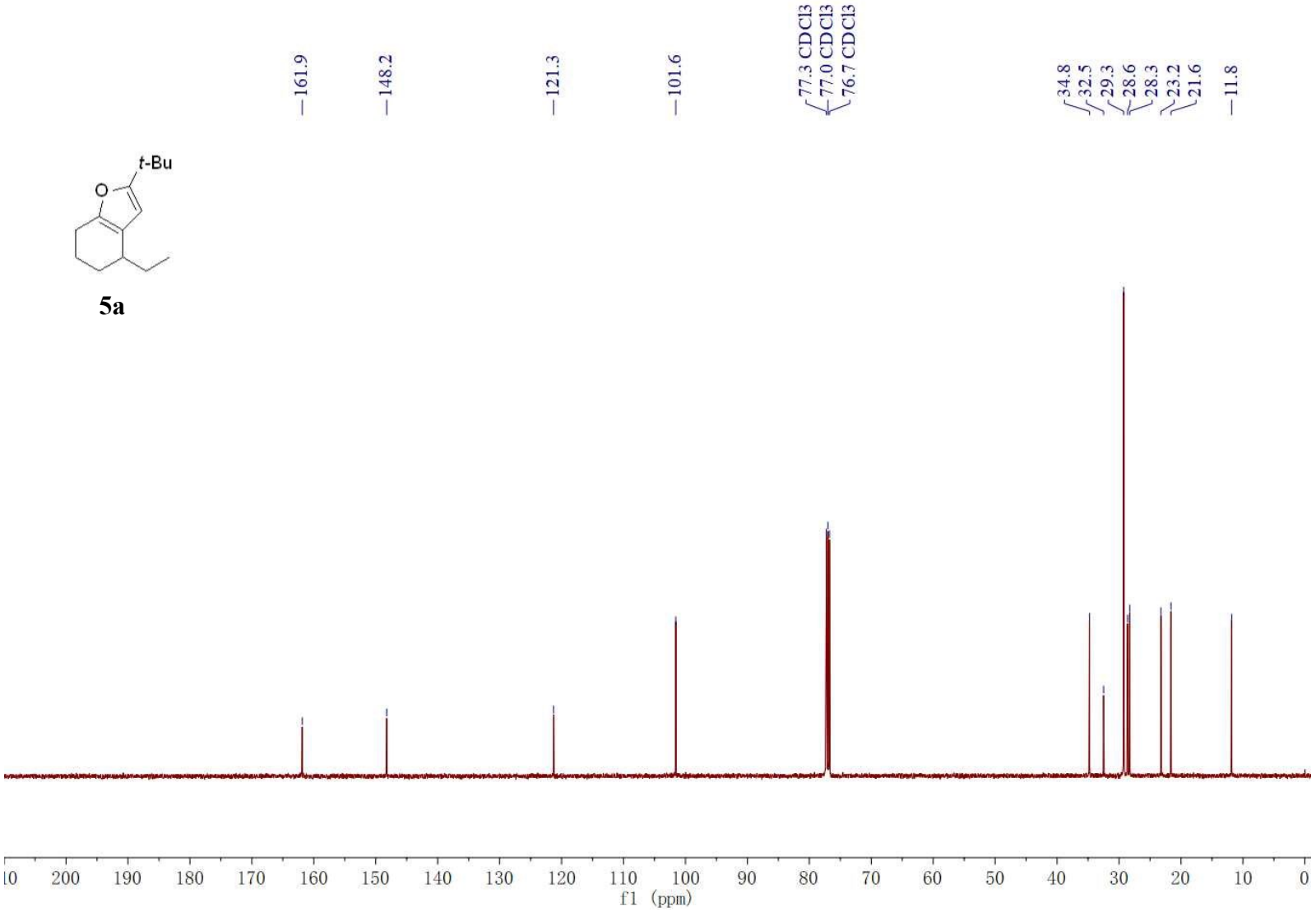
5a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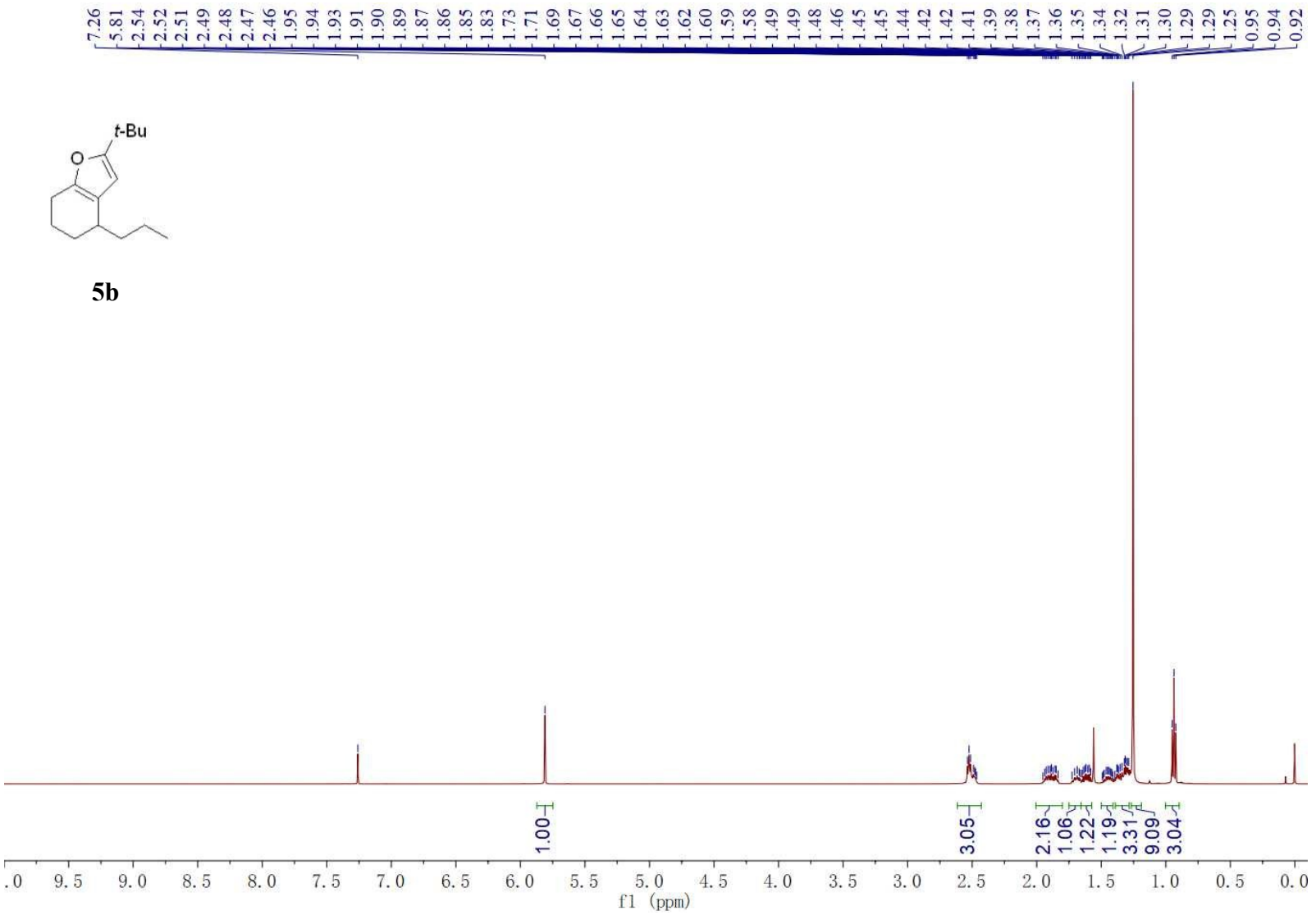
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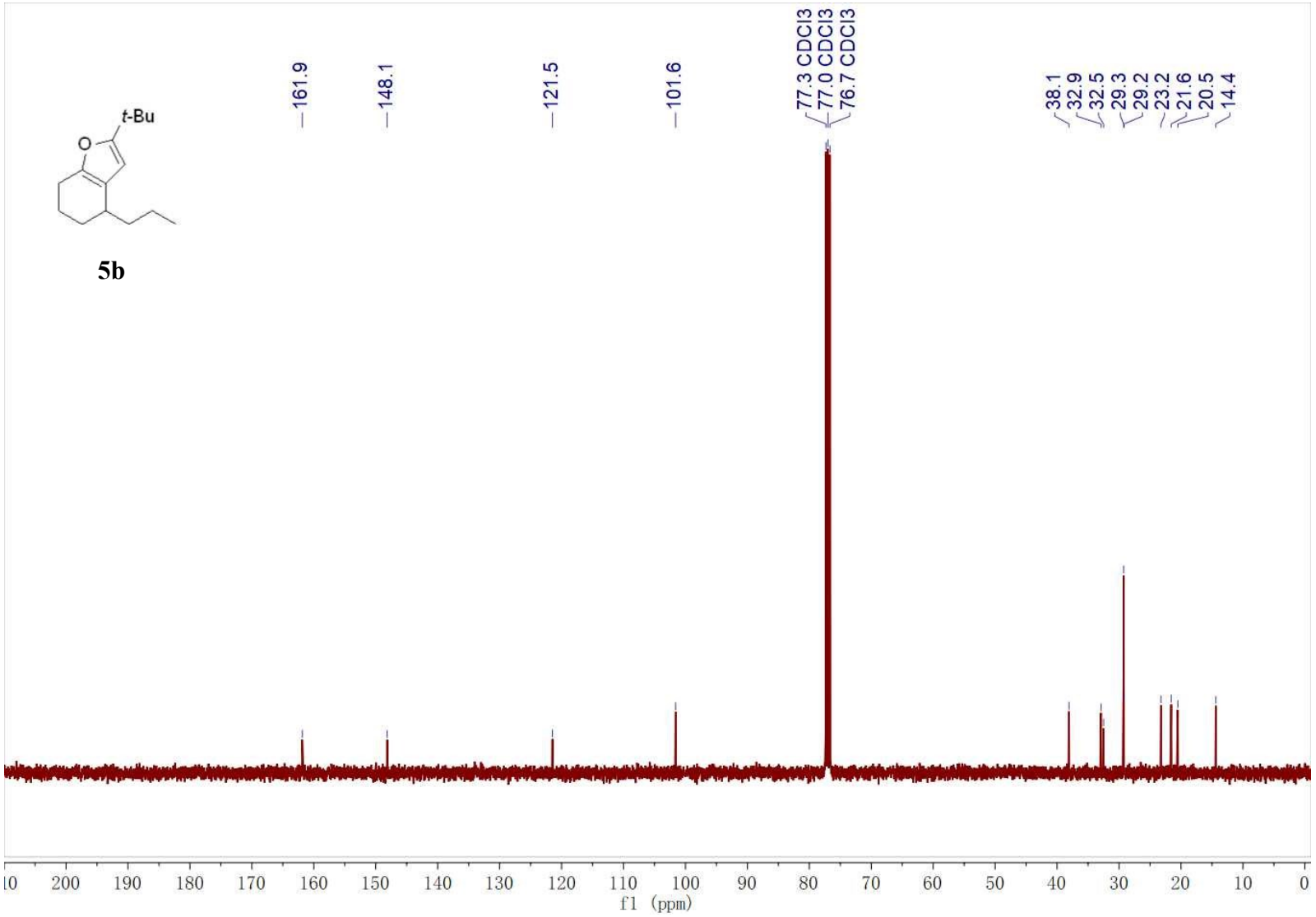
5b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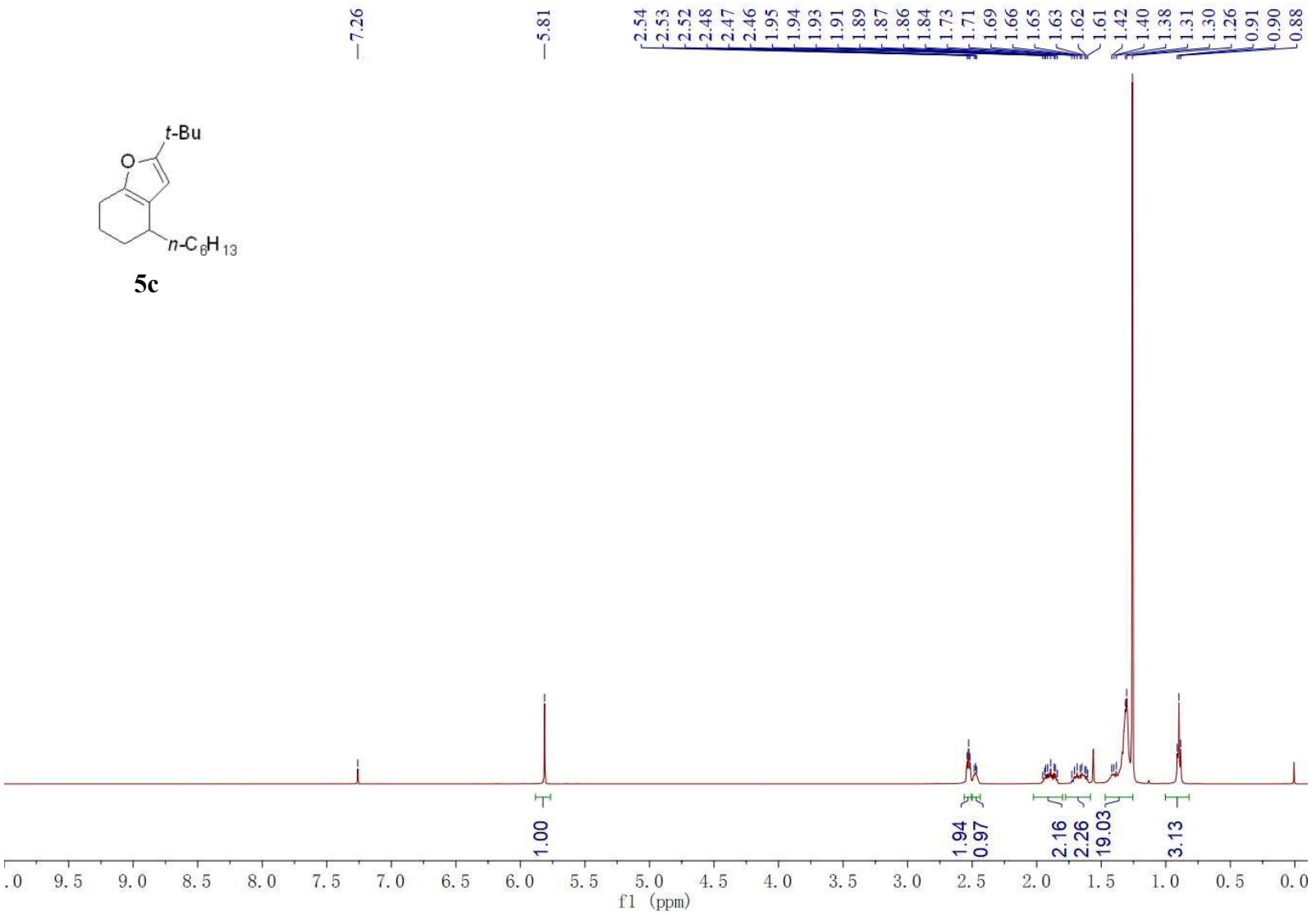
5b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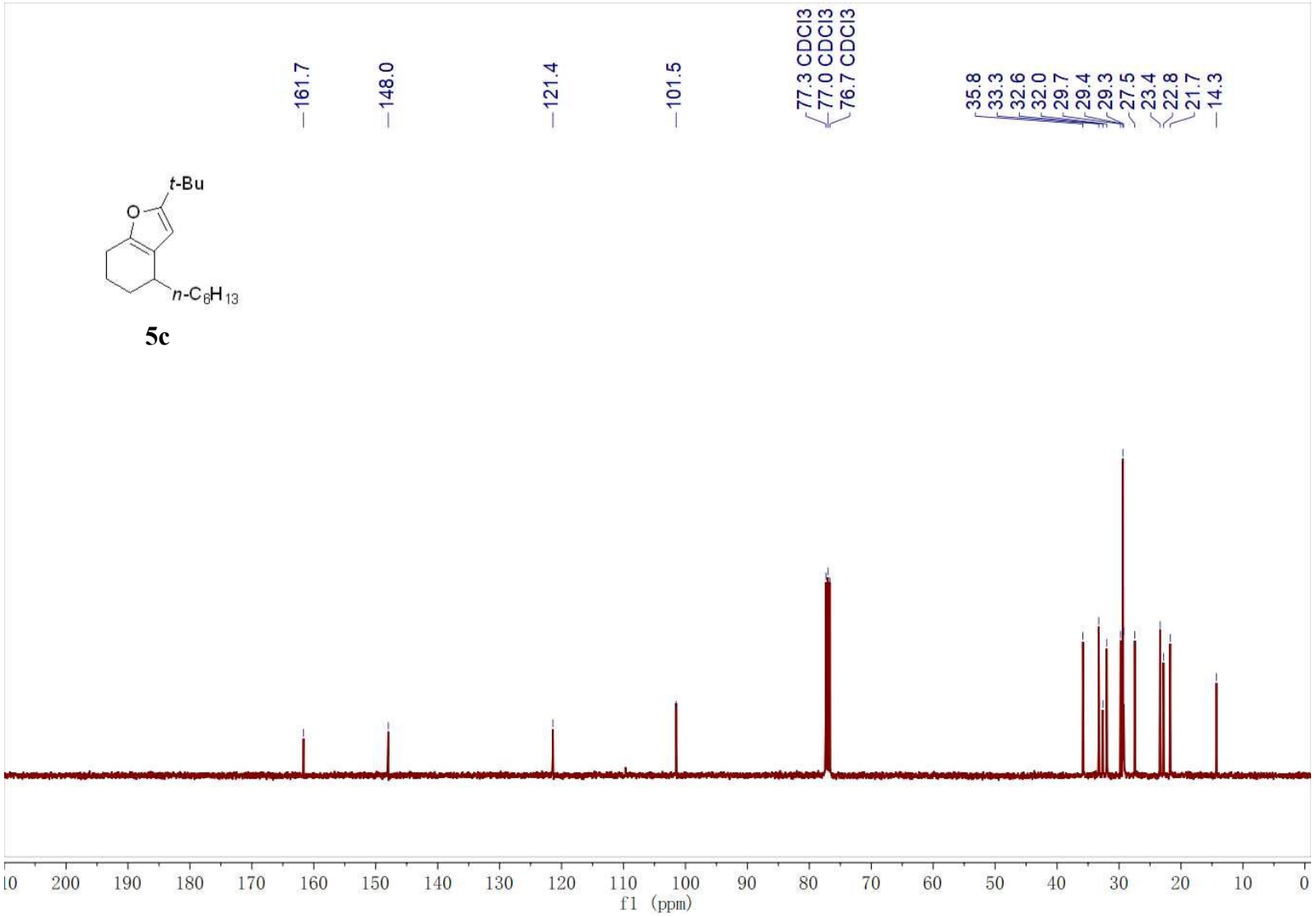
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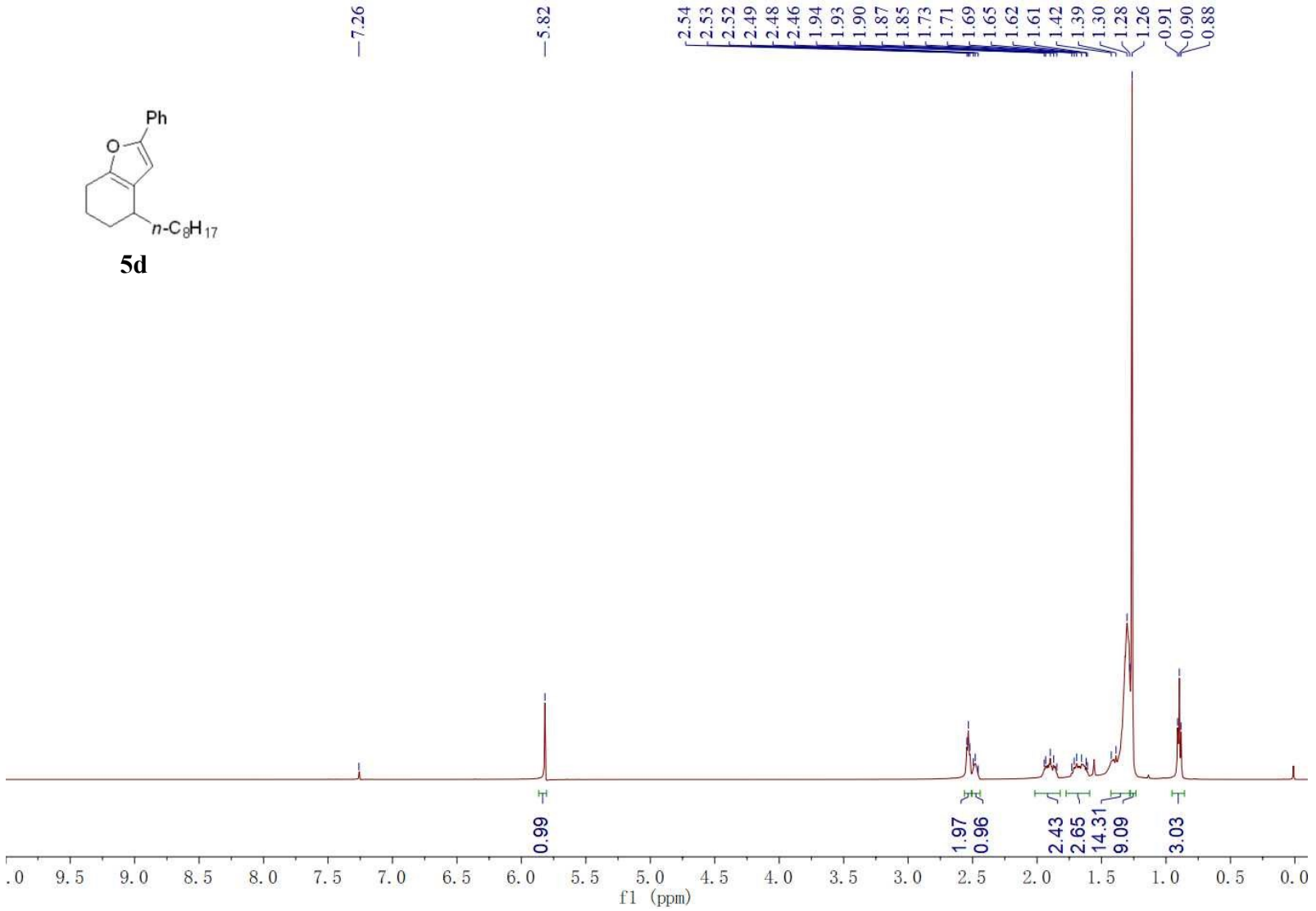
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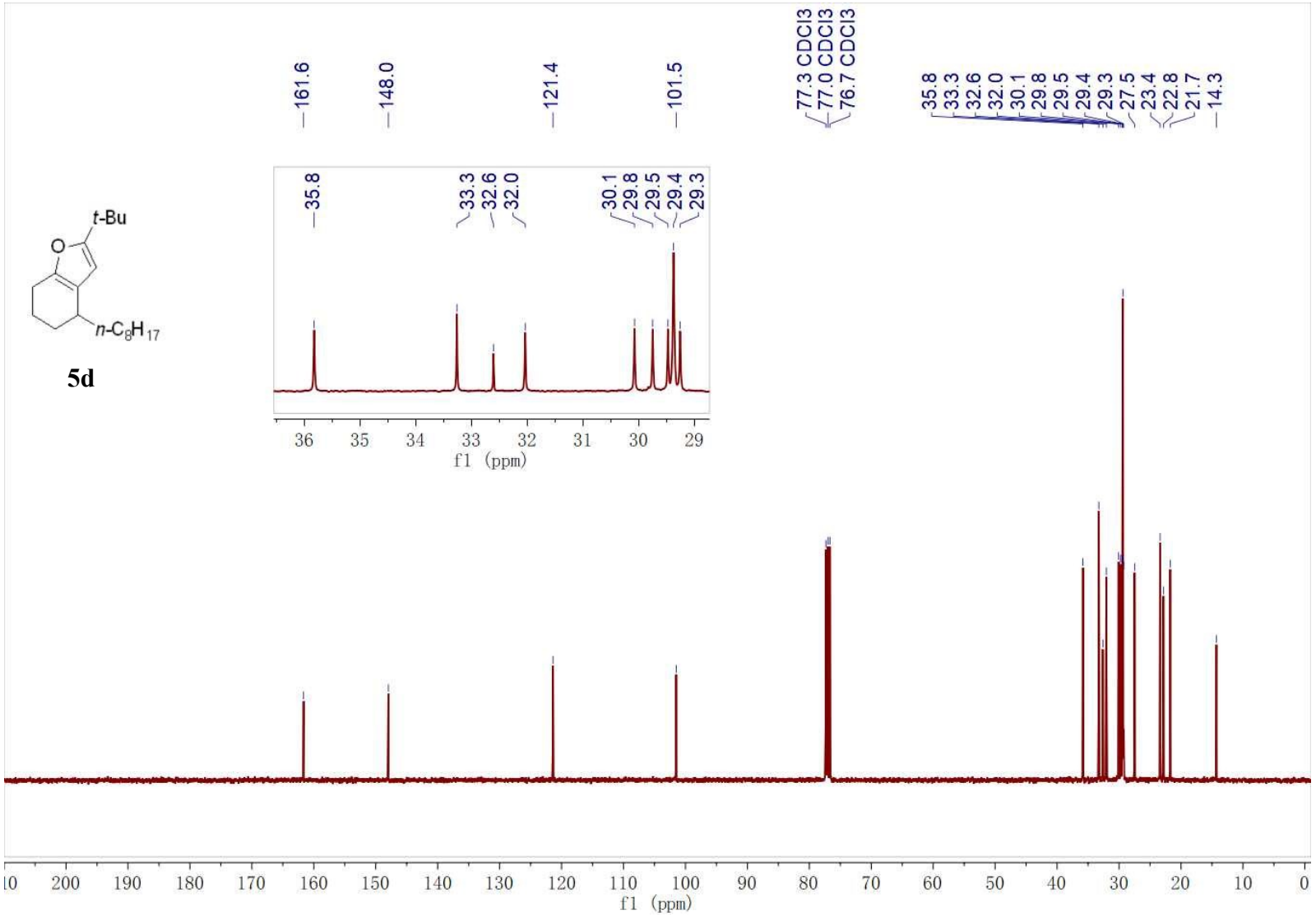
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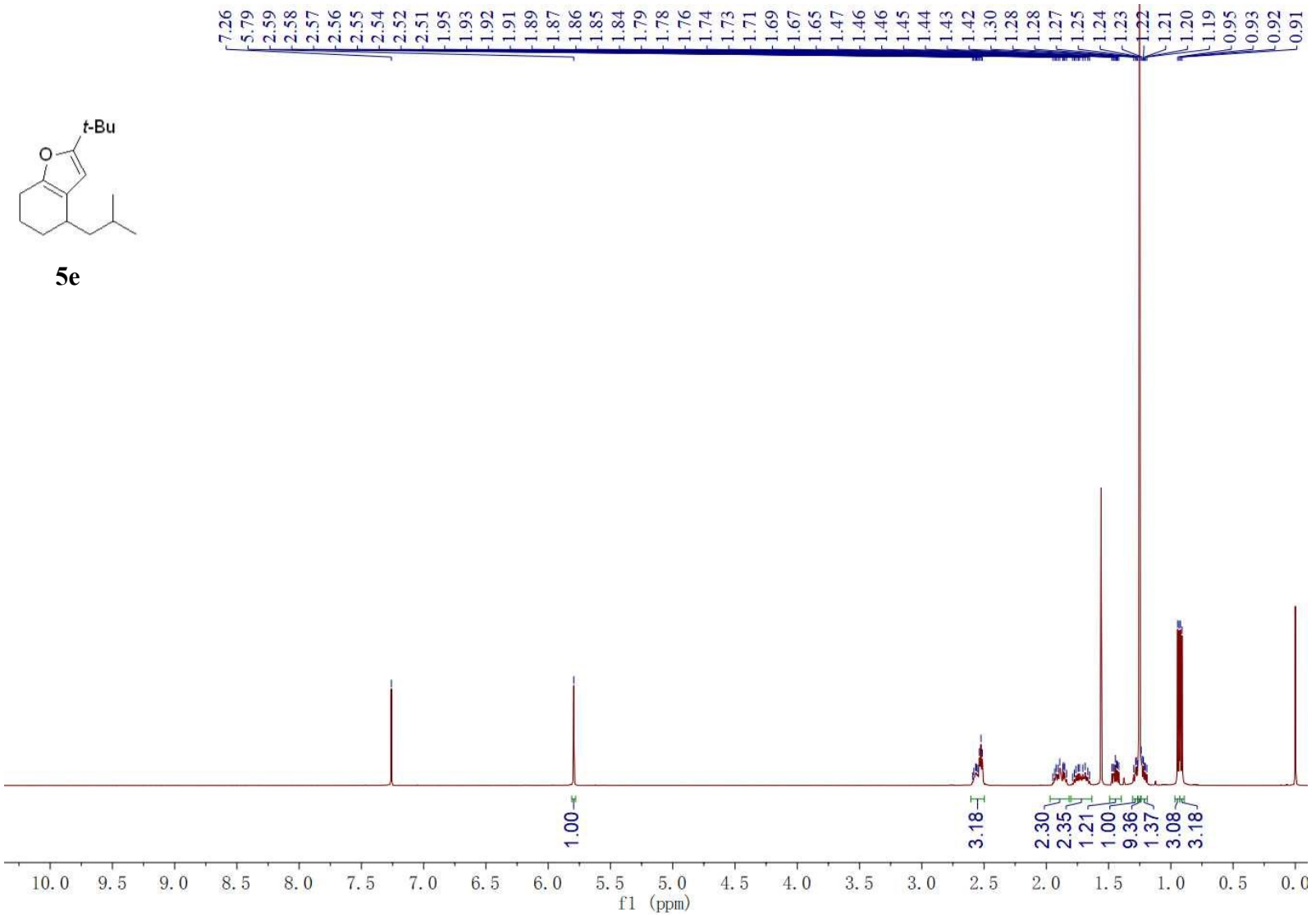
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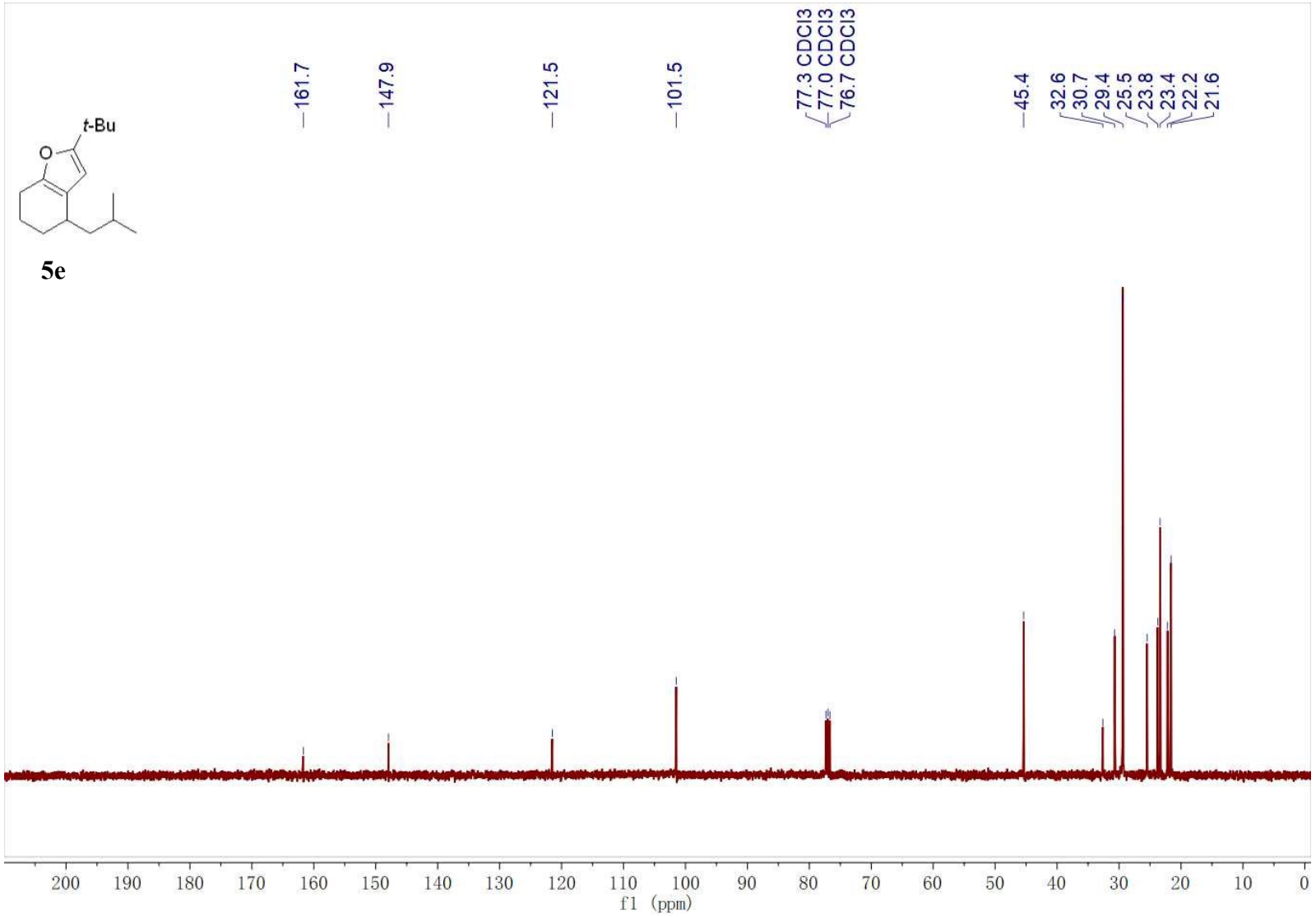
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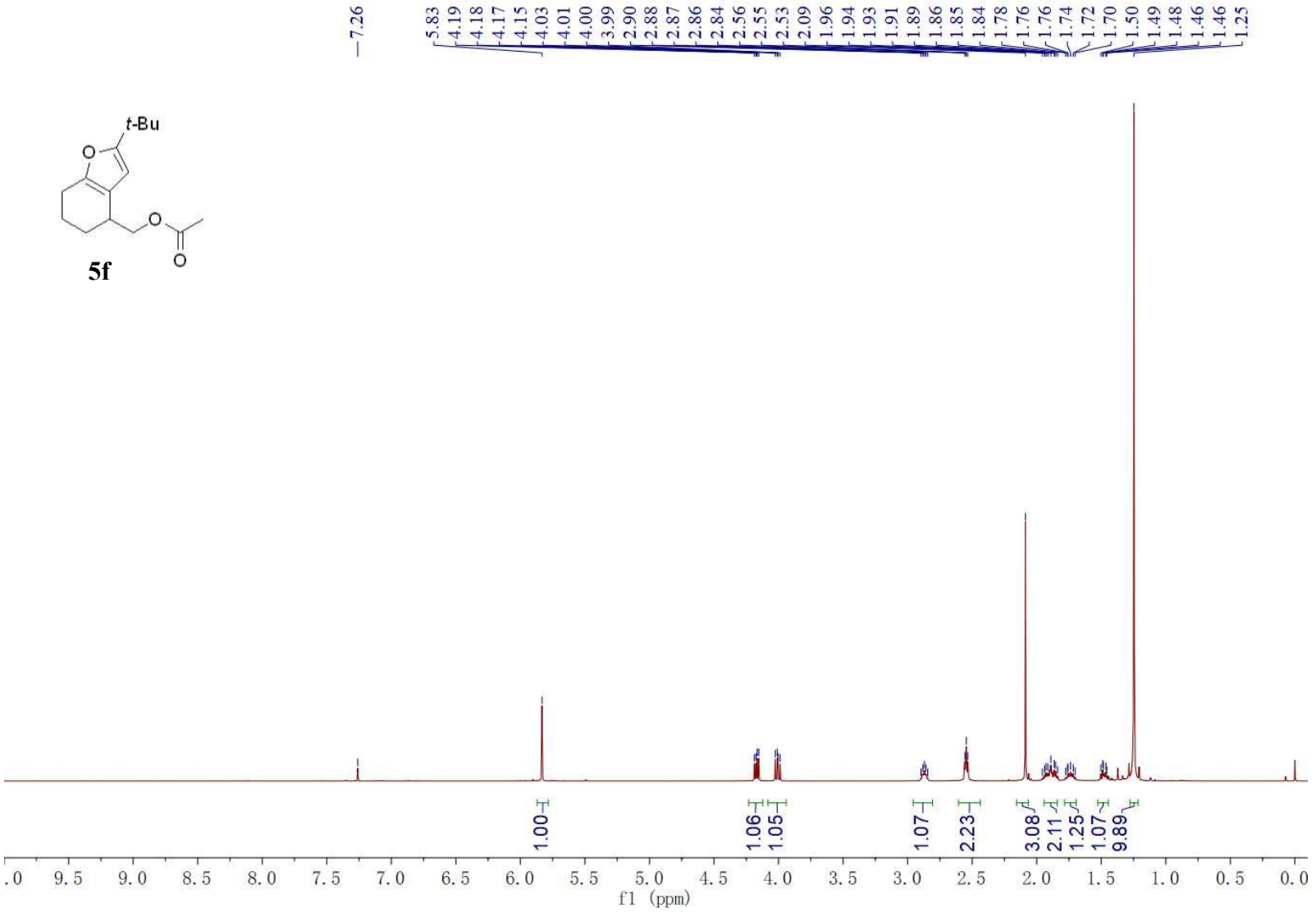
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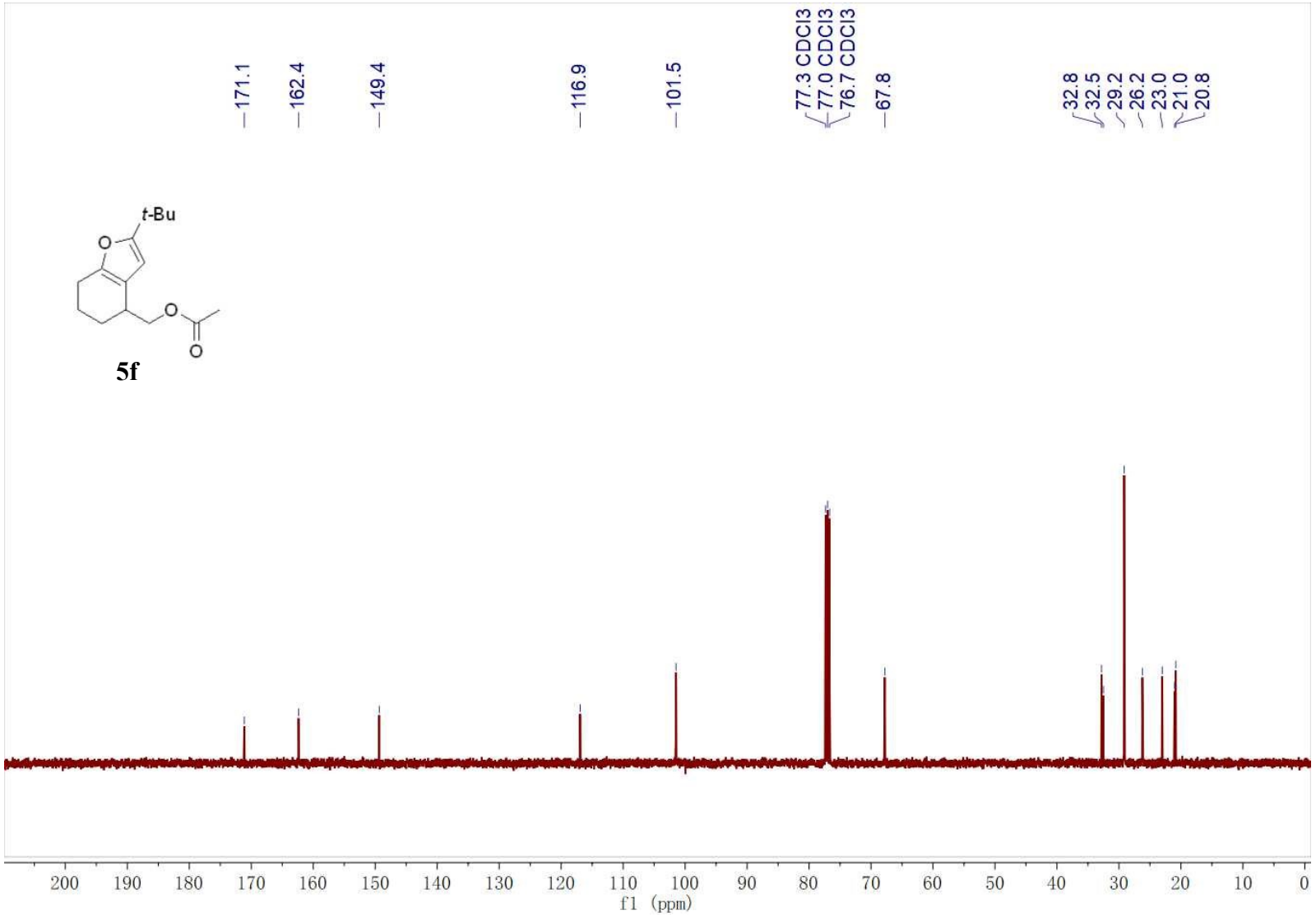
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O
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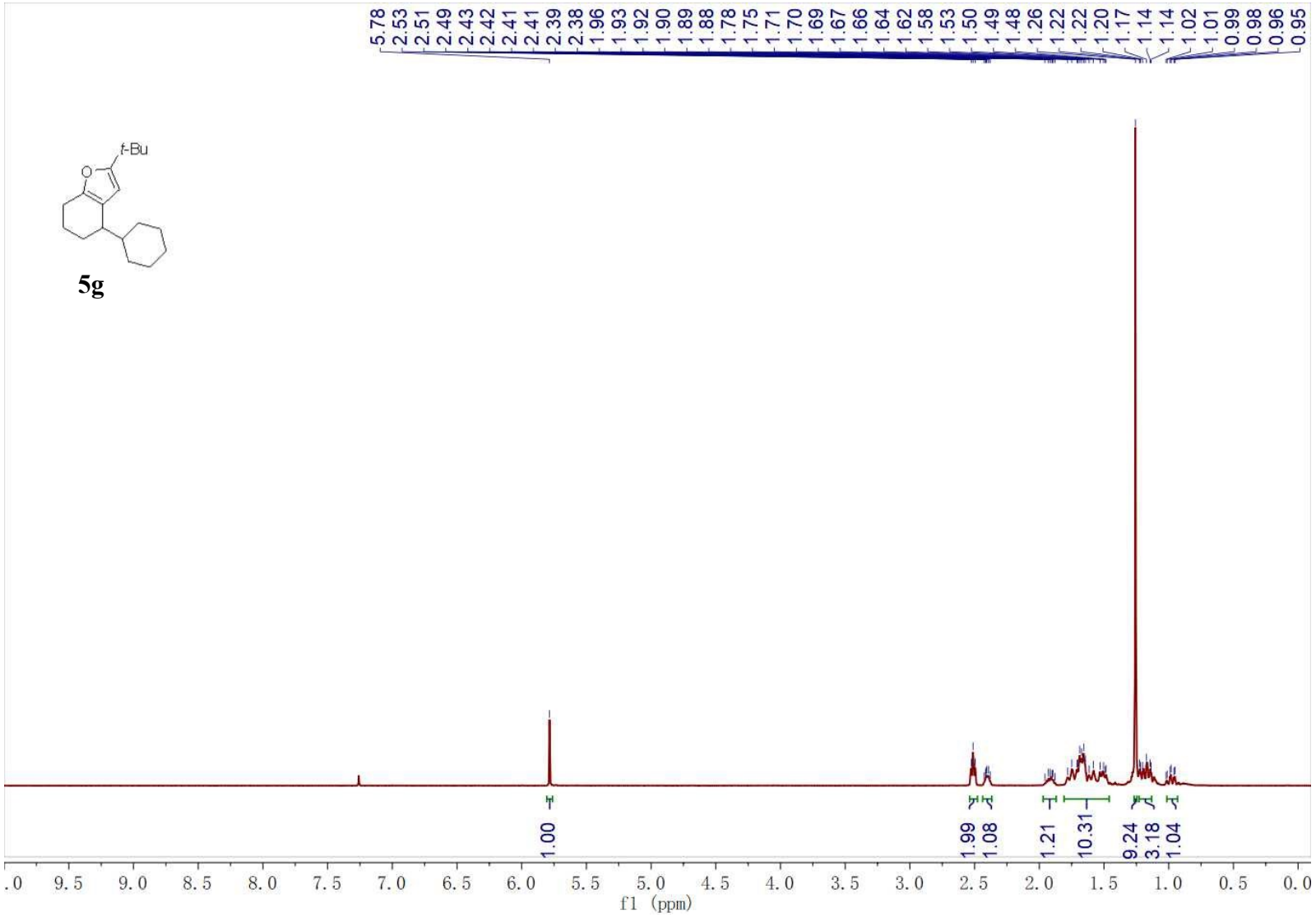
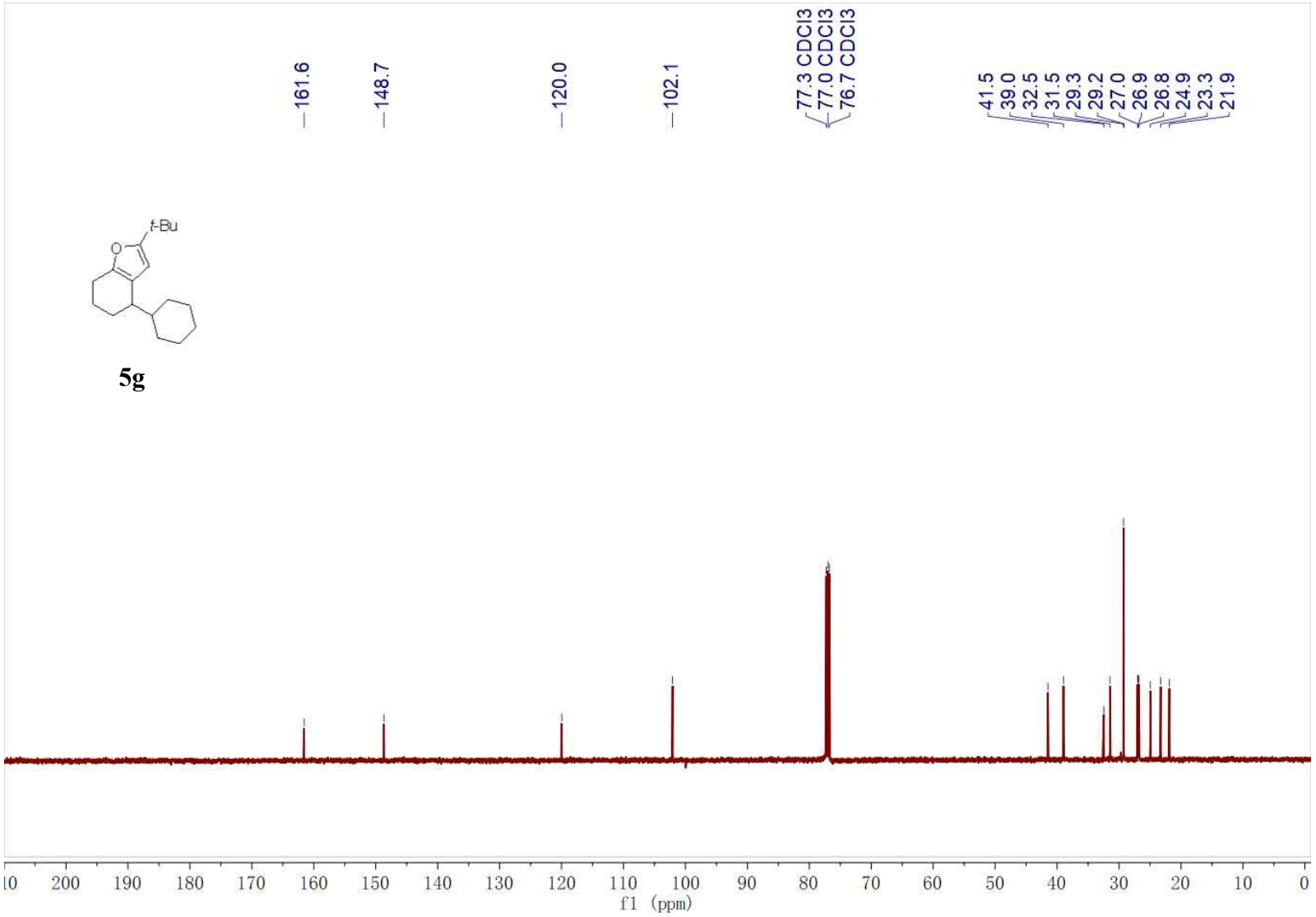
f1 (ppm)

5f


5.78
2.53
2.51
2.49
2.43
2.42
2.41
2.41
2.39
2.38
1.96
1.93
1.92
1.90
1.89
1.88
1.78
1.75
1.71
1.70
1.69
1.67
1.66
1.64
1.62
1.58
1.53
1.50
1.49
1.48
1.26
1.22
1.22
1.20
1.17
1.14
1.14
1.02
1.01
0.99
0.98
0.96
0.95
t-Bu
O
1.00
1.99
1.08
1.21
10.31
9.24
3.18
1.04
.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

5g


161.6
148.7
120.0
102.1
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
41.5
39.0
32.5
31.5
29.3
29.2
27.0
26.9
26.8
24.9
23.3
21.9
t-Bu
O
f1 (ppm)

5g


7.63
7.61
7.36
7.34
7.33
7.26
7.21
7.19
7.18
6.55
2.66
2.65
2.64
2.64
2.64
2.63
2.54
2.52
2.51
2.50
2.01
1.99
1.98
1.97
1.95
1.94
1.93
1.91
1.90
1.88
1.79
1.78
1.77
1.75
1.74
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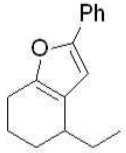
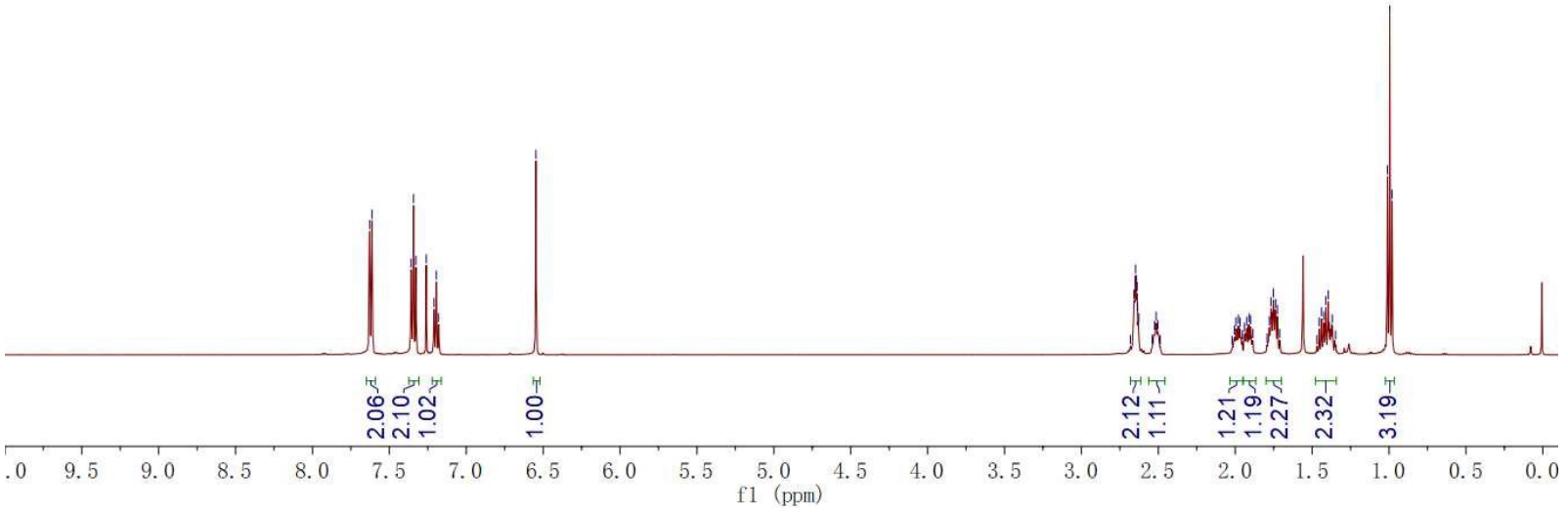
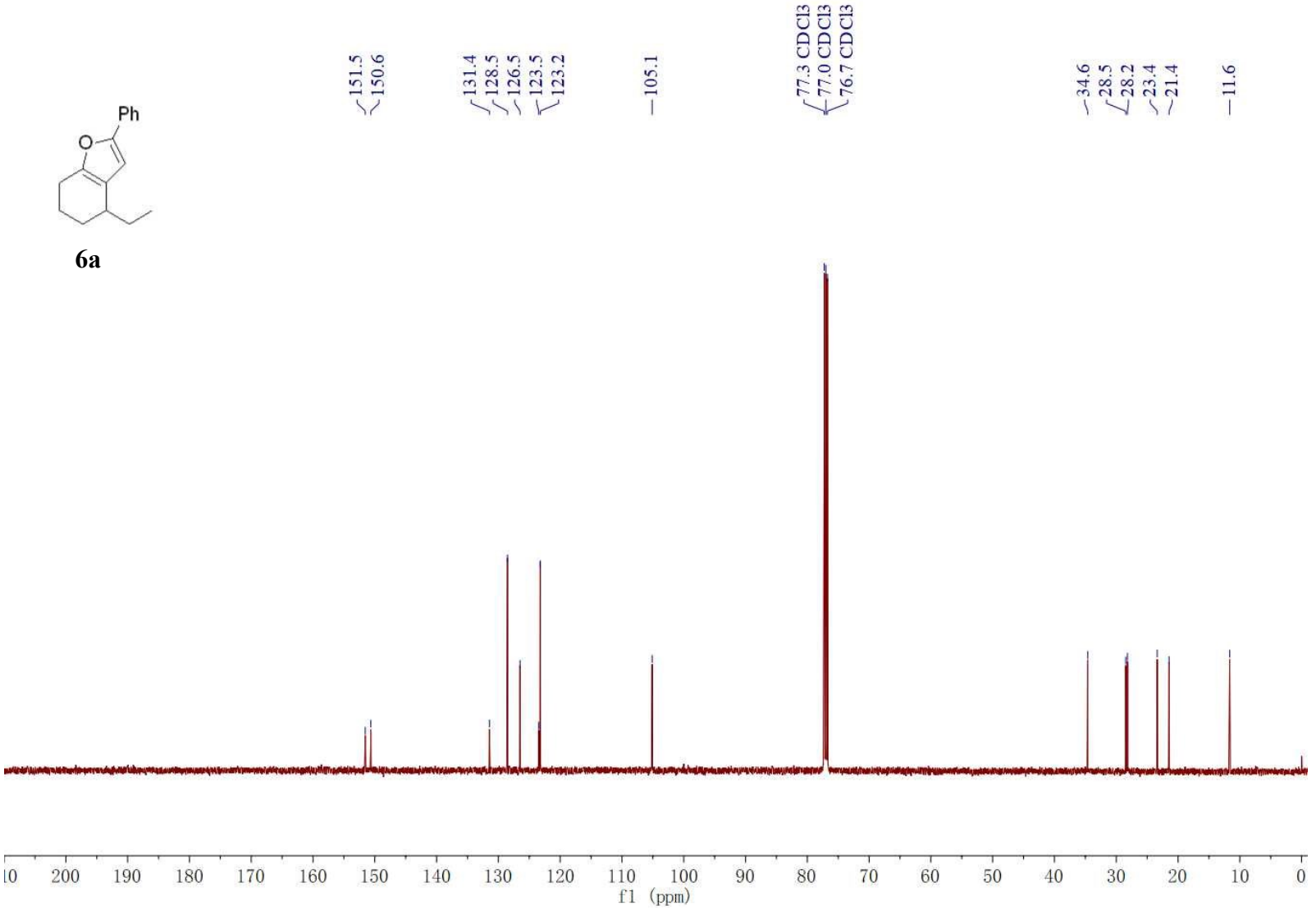
1.41
1.40
1.37
1.35
1.01
1.00

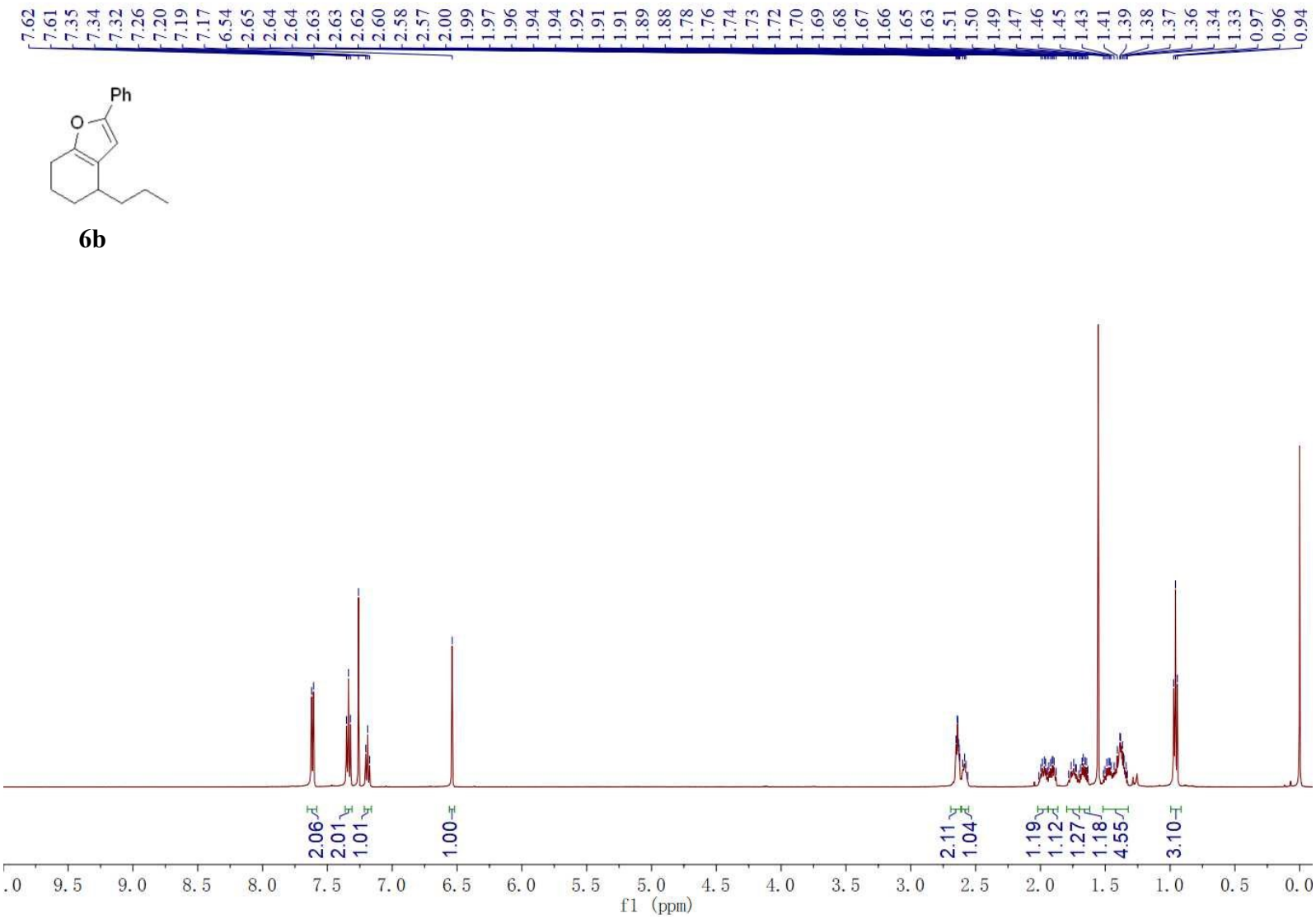

Ph
O

6a

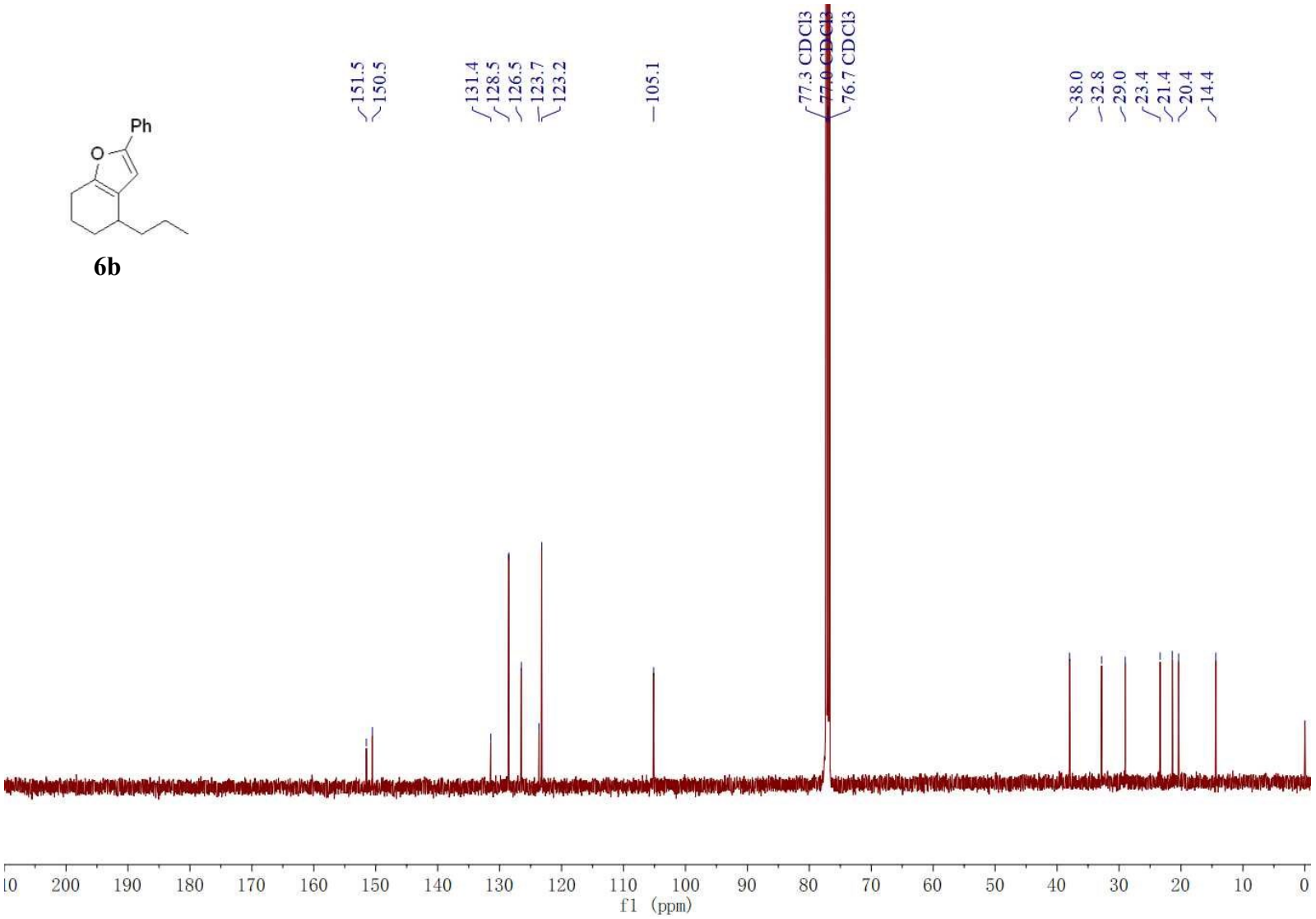

2.06
2.10
1.02
1.00
2.12
1.11
1.21
1.19
2.27
2.32
3.19
.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


Ph
O
151.5
150.6
131.4
128.5
126.5
123.5
123.2
105.1
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
34.6
28.5
28.2
23.4
21.4
11.6
f1 (ppm)

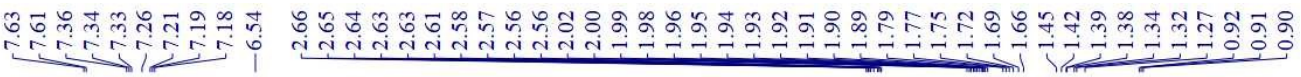
6a

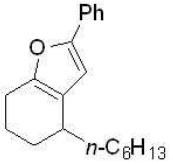

7.62
7.61
7.35
7.34
7.32
7.26
7.20
7.19
7.17
6.54
2.65
2.64
2.64
2.63
2.63
2.62
2.60
2.58
2.57
2.00
1.99
1.97
1.96
1.94
1.94
1.92
1.91
1.91
1.89
1.88
1.78
1.76
1.74
1.73
1.72
1.70
1.69
1.68
1.67
1.66
1.65
1.63
1.51
1.50
1.49
1.47
1.46
1.45
1.43
1.41
1.39
1.38
1.37
1.36
1.34
1.33
0.97
0.96
0.94
Ph
O
2.06
2.01
1.01
1.00
2.11
1.04
1.19
1.12
1.27
1.18
4.55
3.10
f1 (ppm)

6b

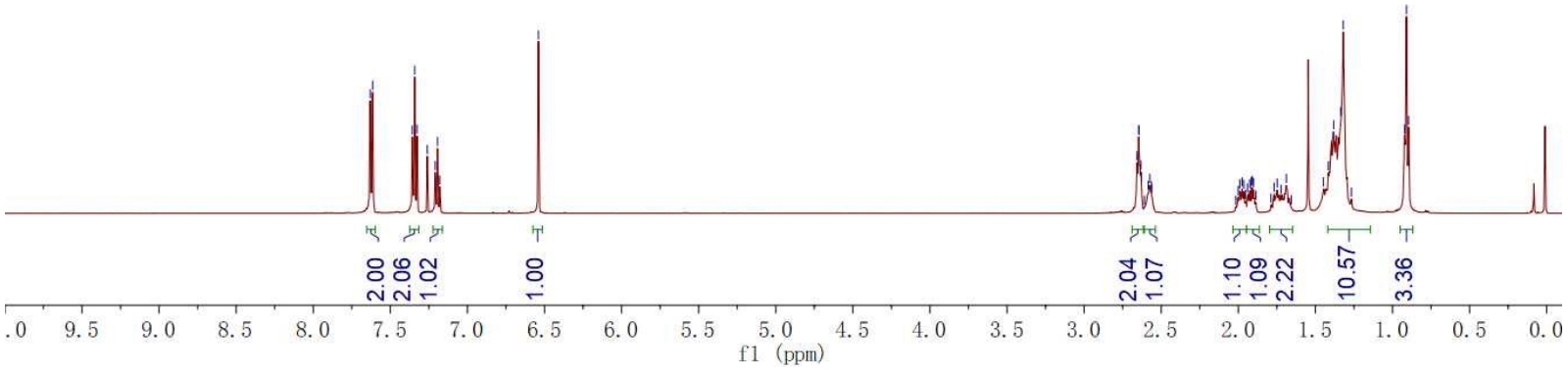

Ph
O
151.5
150.5
131.4
128.5
126.5
123.7
123.2
105.1
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
38.0
32.8
29.0
23.4
21.4
20.4
14.4
f1 (ppm)

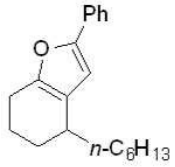
6b


7.63
7.61
7.36
7.34
7.33
7.26
7.21
7.19
7.18
6.54
2.66
2.65
2.64
2.63
2.63
2.61
2.58
2.57
2.56
2.56
2.02
2.00
1.99
1.98
1.96
1.95
1.94
1.93
1.92
1.91
1.90
1.89
1.79
1.77
1.75
1.72
1.69
1.66
1.45
1.42
1.39
1.38
1.34
1.32
1.27
0.92
0.91
0.90


Ph
O
n-C6H13

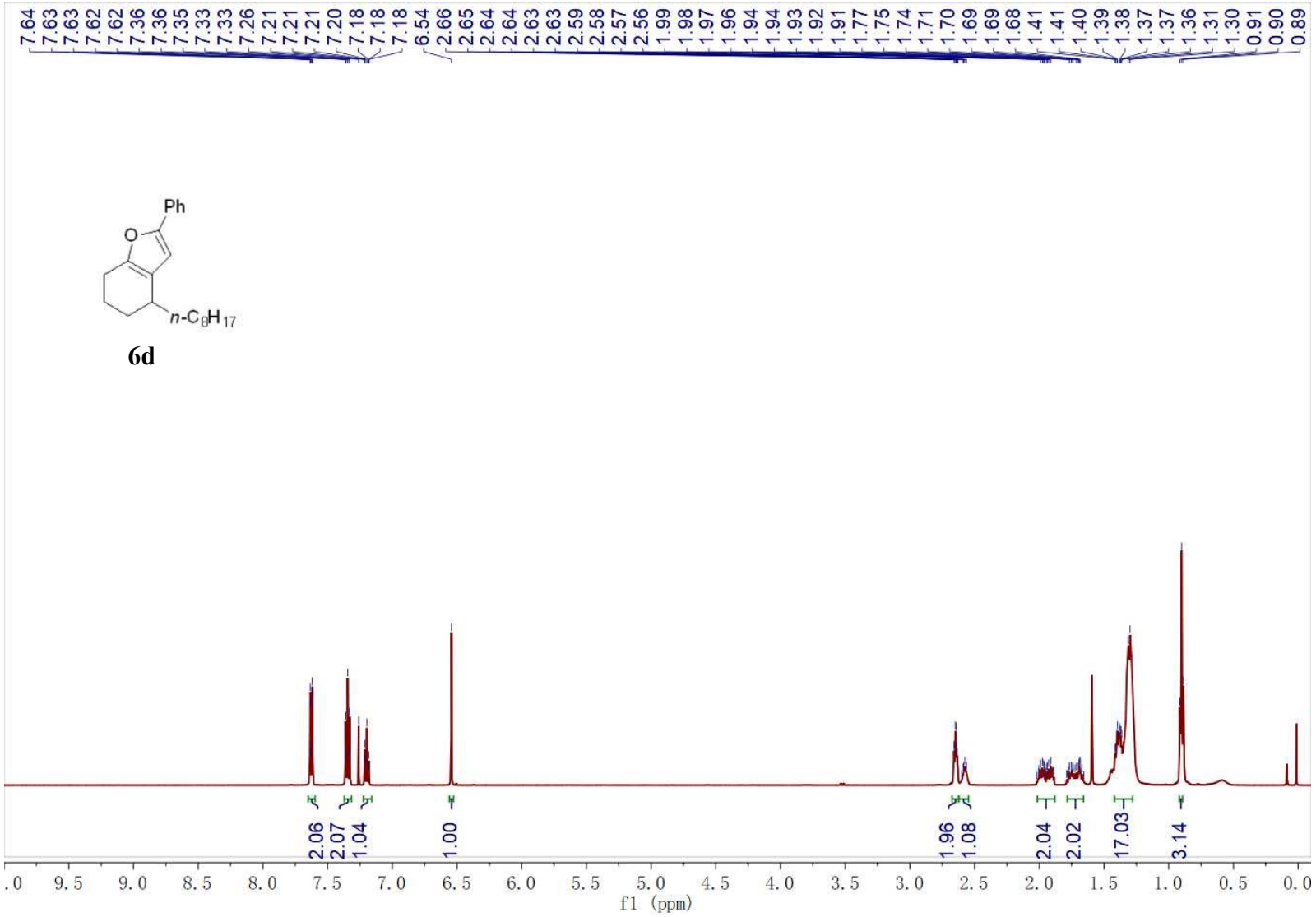
6c


2.00
2.06
1.02
1.00
2.04
1.07
1.10
1.09
2.22
10.57
3.36
.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

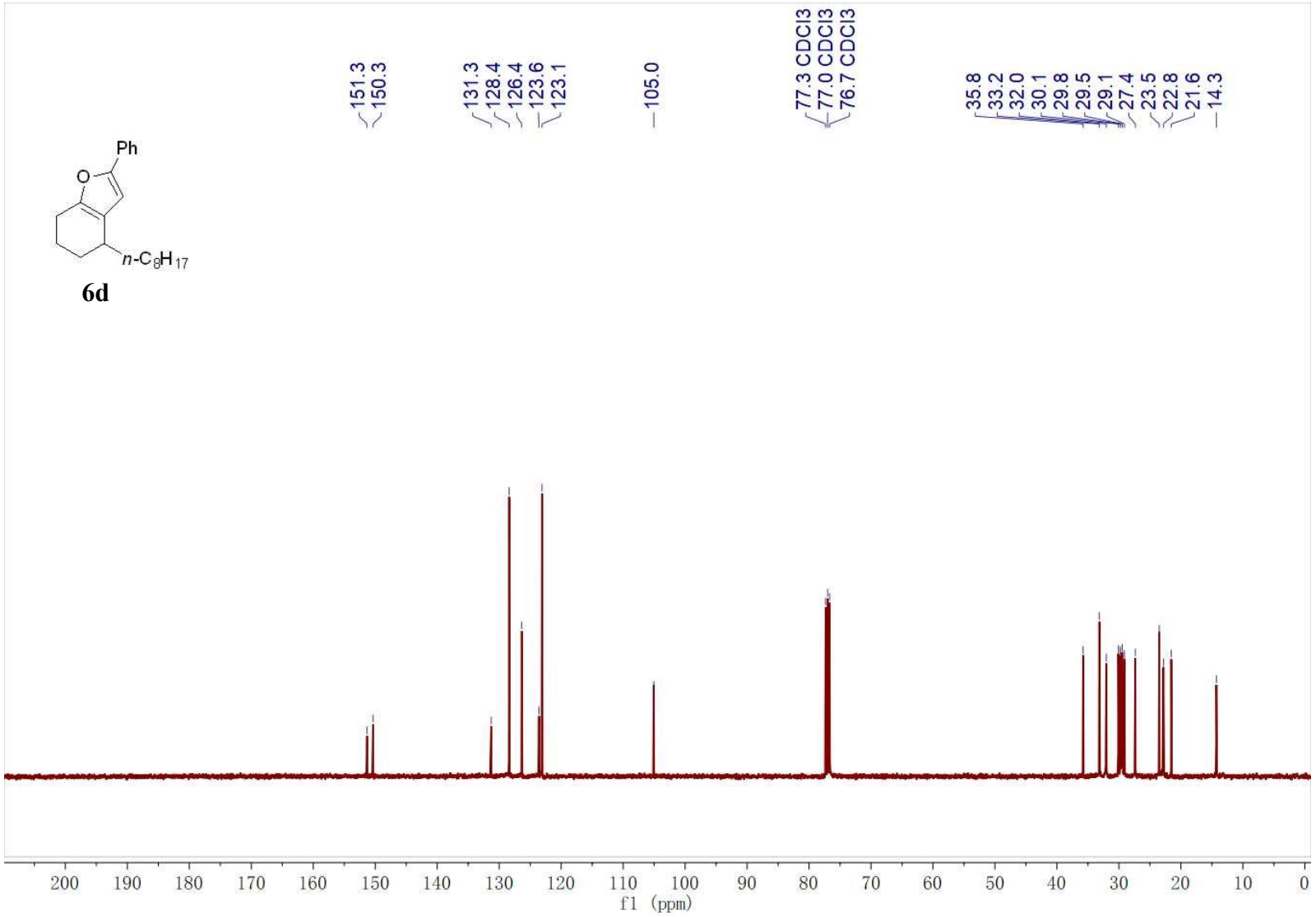

Ph
O
n-C6H13

6c

151.5
150.5
131.5
128.5
126.5
123.7
123.2
105.1
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
35.7
33.1
31.9
29.6
29.0
27.2
23.4
22.7
21.4
14.1
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.64 7.63 7.63 7.62 7.62 7.36 7.36 7.35 7.33 7.33 7.26 7.21 7.21 7.21 7.20 7.18 7.18 7.18 6.54 2.66 2.65 2.64 2.64 2.63 2.63 2.59 2.58 2.57 2.56 1.99 1.98 1.97 1.96 1.94 1.94 1.93 1.92 1.91 1.77 1.75 1.74 1.71 1.70 1.69 1.69 1.68 1.41 1.41 1.40 1.39 1.38 1.37 1.37 1.36 1.31 1.30 0.91 0.90 0.89
Ph
O
n-C8H17
2.06 2.07 1.04 1.00 1.96 1.08 2.04 2.02 17.03 3.14
f1 (ppm)

6d


151.3
150.3
131.3
128.4
126.4
123.6
123.1
105.0
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
35.8
33.2
32.0
30.1
29.8
29.5
29.1
27.4
23.5
22.8
21.6
14.3
Ph
O
n-C8H17
6d
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.62
7.60
7.36
7.34
7.33
7.26
7.22
7.20
7.18
7.17
6.72
6.70
6.69
6.63
6.62
6.53
3.64
3.19
3.17
3.16
3.13
2.70
2.69
2.67
2.65
2.62
2.61
2.01
1.99
1.97
1.95
1.91
1.85
1.82
1.79
1.77
1.74
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1.69
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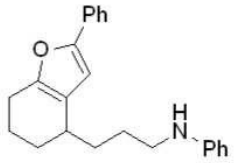
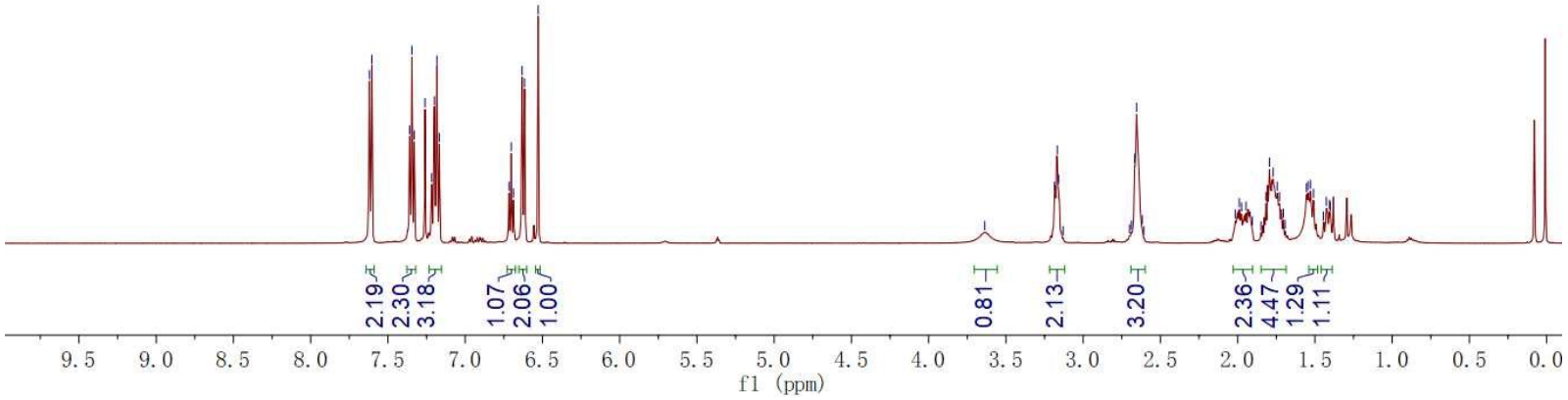
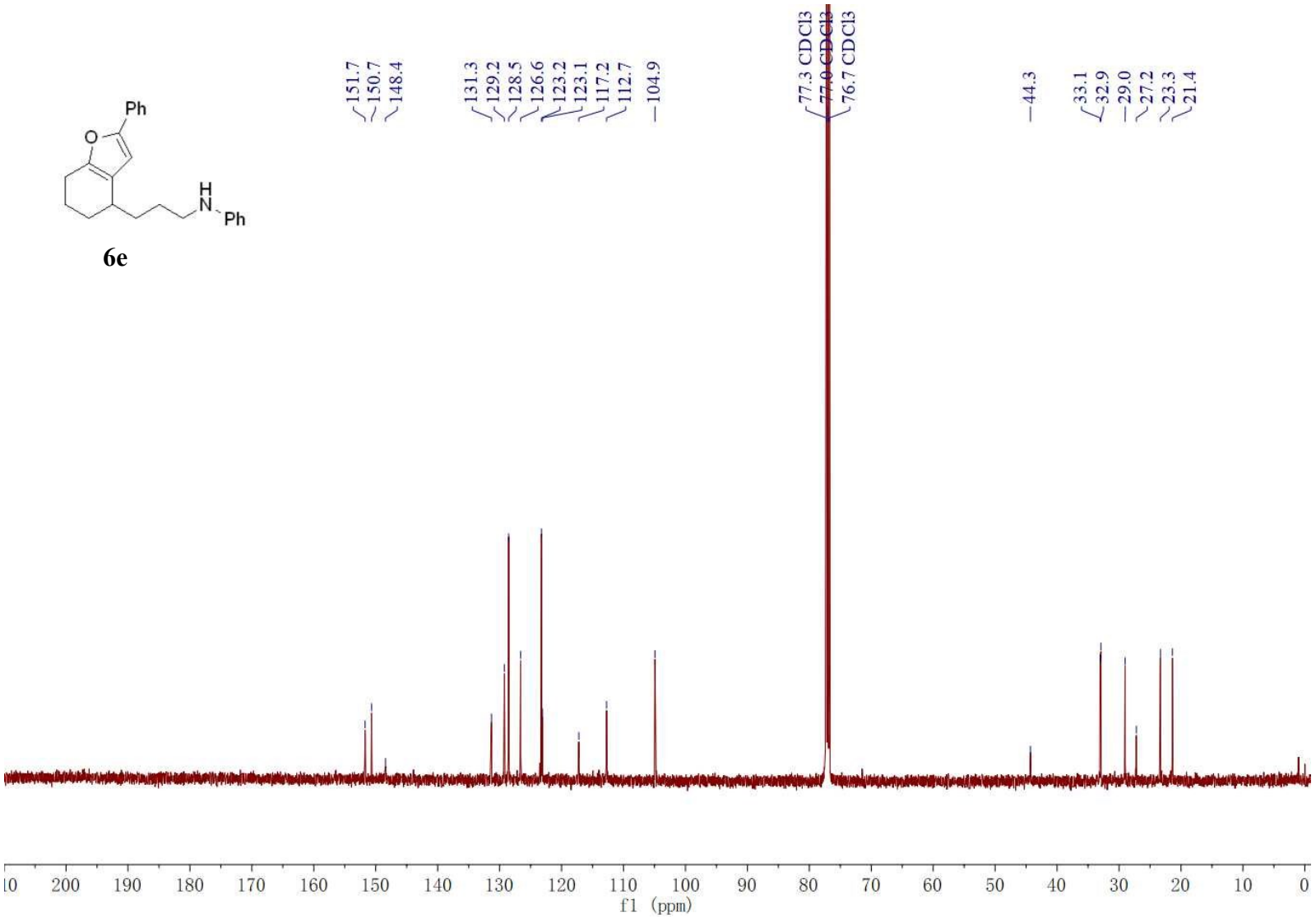
1.53
1.51
1.45
1.43
1.41
1.40

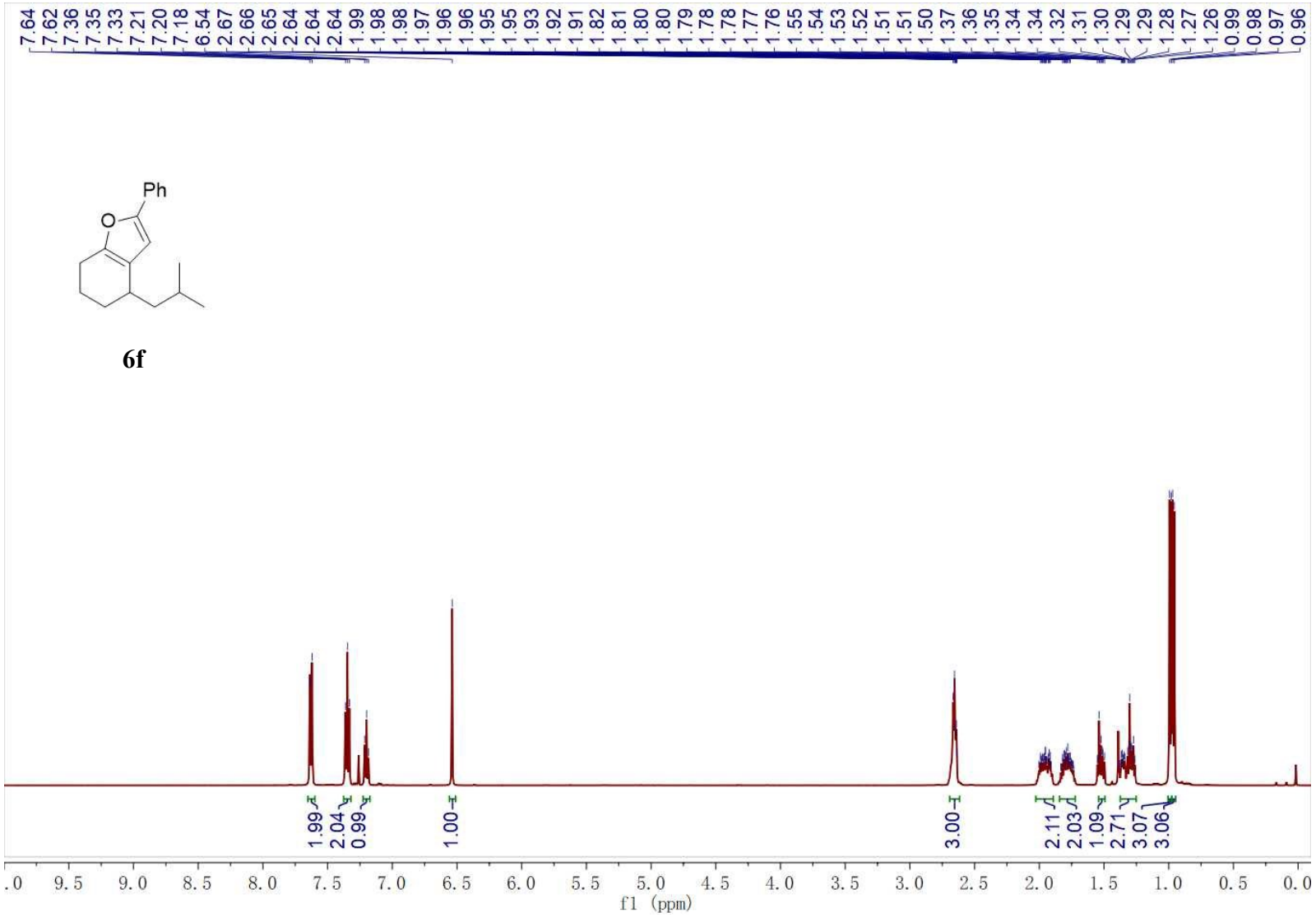

Ph
O
H
N
Ph

6e

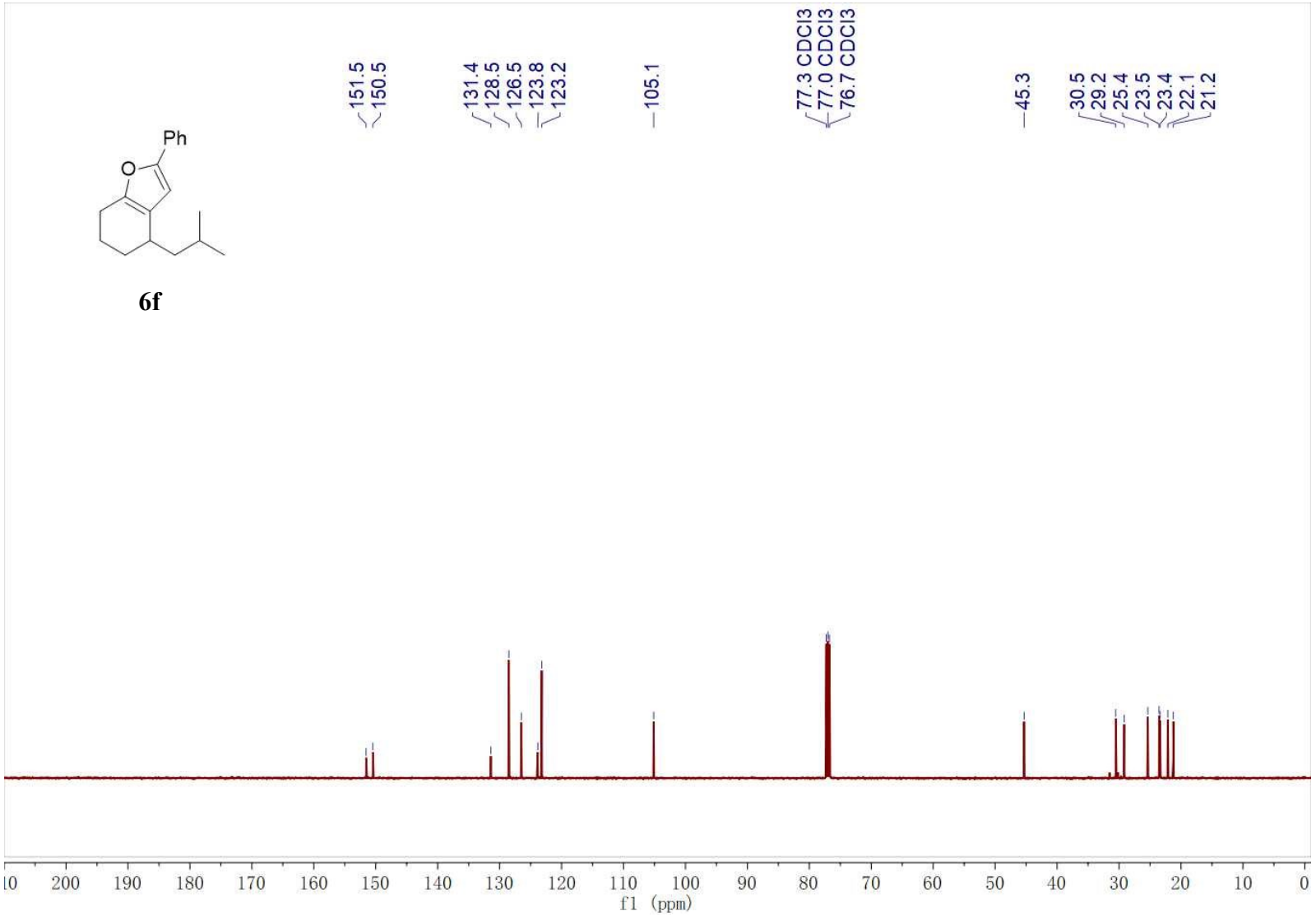

2.19
2.30
3.18
1.07
2.06
1.00
0.81
2.13
3.20
2.36
4.47
1.29
1.11
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

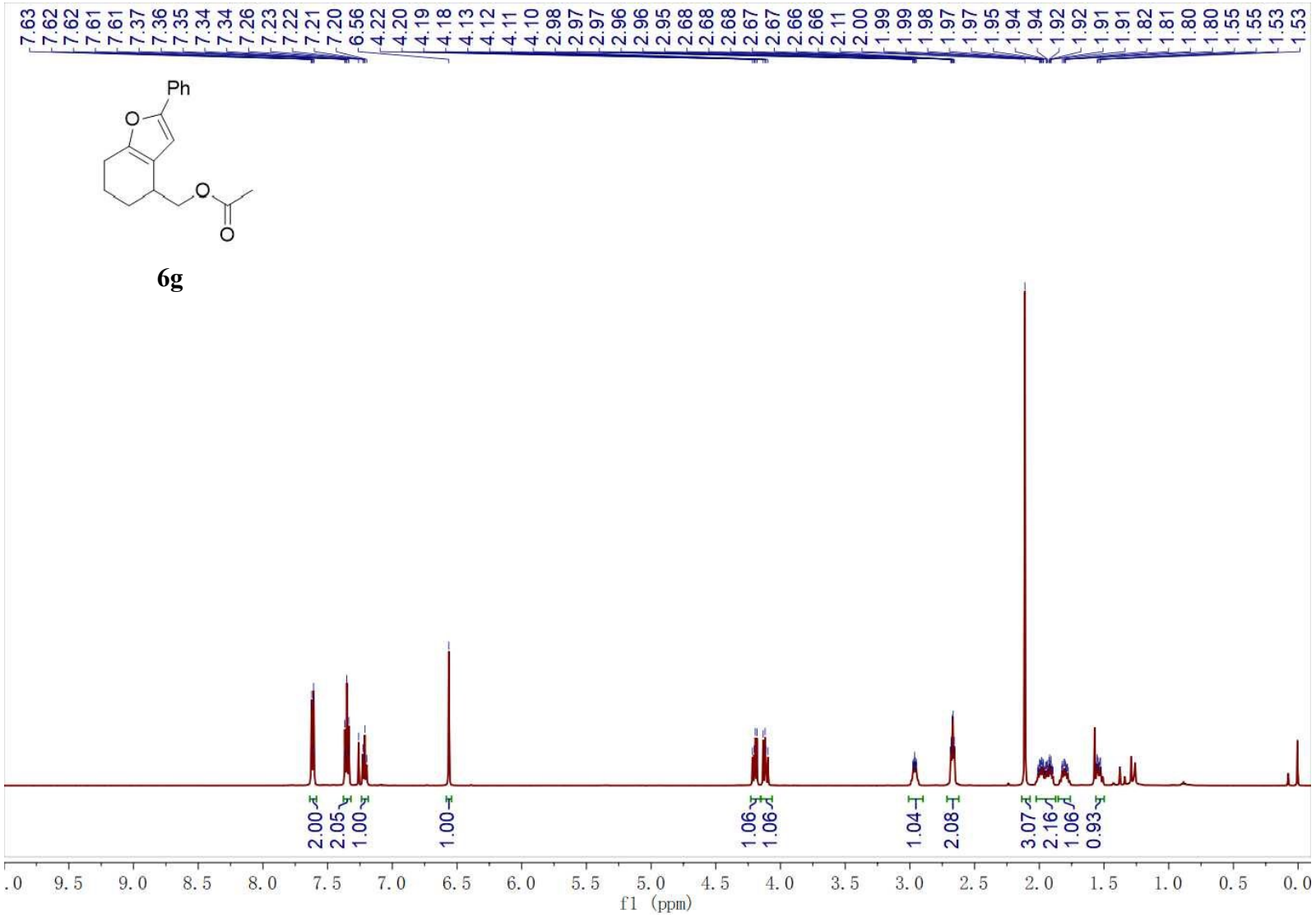

Ph
O
H
N
Ph
151.7
150.7
148.4
131.3
129.2
128.5
126.6
123.2
123.1
117.2
112.7
104.9
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
44.3
33.1
32.9
29.0
27.2
23.3
21.4
f1 (ppm)

6e

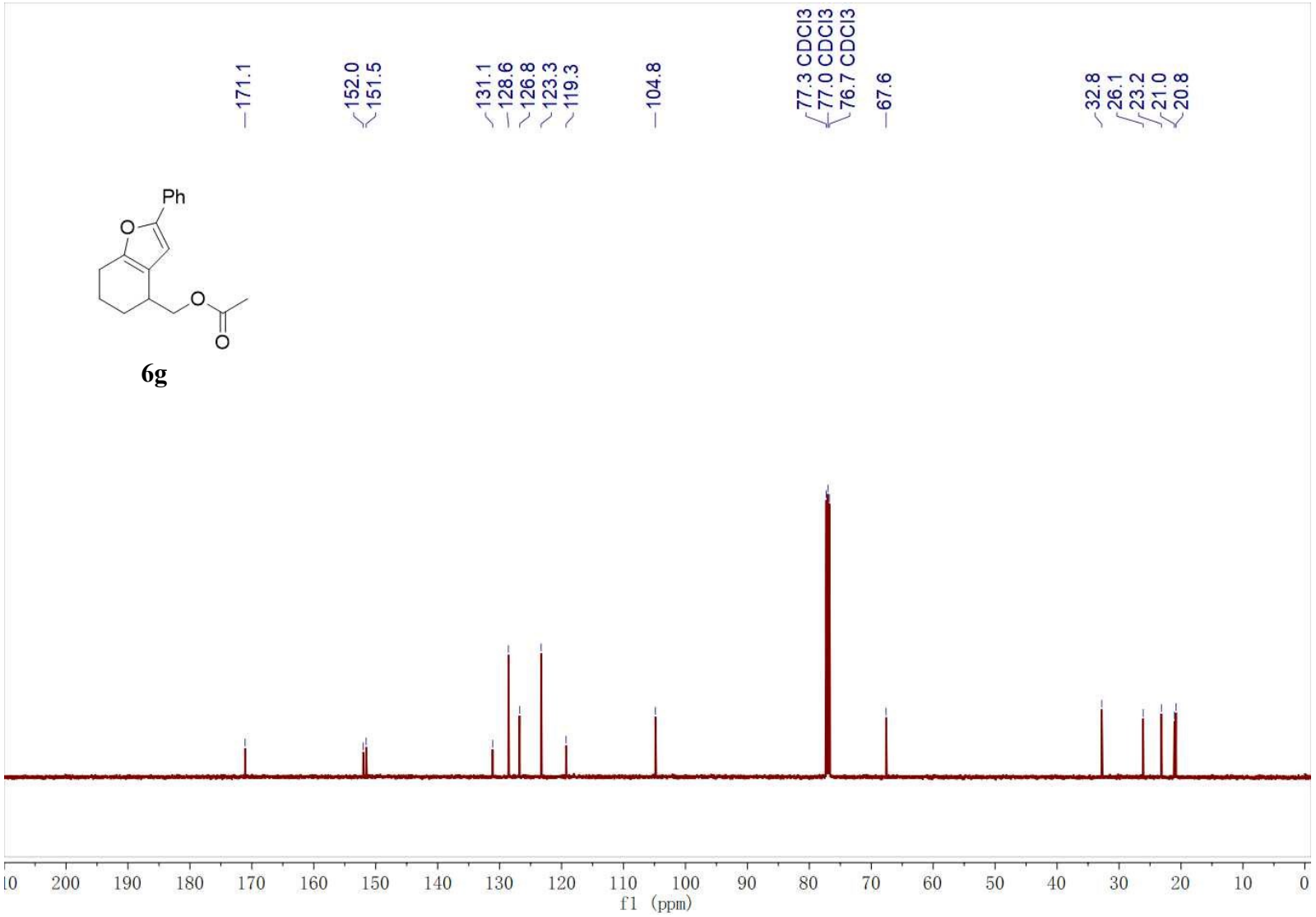

7.64 7.62 7.36 7.35 7.33 7.21 7.20 7.18 6.54 2.67 2.66 2.65 2.64 2.64 2.64 1.99 1.98 1.98 1.97 1.96 1.96 1.95 1.95 1.93 1.92 1.91 1.82 1.81 1.80 1.80 1.79 1.78 1.78 1.77 1.76 1.55 1.54 1.53 1.52 1.51 1.51 1.50 1.37 1.36 1.35 1.34 1.34 1.32 1.31 1.30 1.29 1.29 1.28 1.27 1.26 0.99 0.98 0.97 0.96
Ph
O
1.99 2.04 0.99 1.00 3.00 2.11 2.03 1.09 2.71 3.07 3.06
f1 (ppm)

6f


Ph
O
6f
151.5
150.5
131.4
128.5
126.5
123.8
123.2
105.1
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
45.3
30.5
29.2
25.4
23.5
23.4
22.1
21.2
f1 (ppm)


7.63 7.62 7.62 7.61 7.61 7.37 7.36 7.35 7.34 7.34 7.26 7.23 7.22 7.21 7.20 6.56 4.22 4.20 4.19 4.18 4.13 4.12 4.11 4.10 2.98 2.97 2.97 2.96 2.96 2.95 2.68 2.68 2.68 2.67 2.67 2.66 2.66 2.11 2.00 1.99 1.99 1.98 1.97 1.97 1.95 1.94 1.94 1.92 1.92 1.91 1.91 1.82 1.81 1.80 1.80 1.55 1.55 1.53 1.53
Ph
O
O
O
2.00 2.05 1.00 1.00 1.06 1.06 1.04 2.08 3.07 2.16 1.06 0.93
f1 (ppm)

6g


171.1
152.0
151.5
131.1
128.6
126.8
123.3
119.3
104.8
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
67.6
32.8
26.1
23.2
21.0
20.8
Ph
O
O
O
6g
f1 (ppm)


7.64
7.62
7.36
7.34
7.33
7.26
7.21
7.19
7.18
6.53
2.64
2.63
2.63
2.62
2.52
2.51
2.50
2.48
2.01
1.99
1.97
1.95
1.80
1.77
1.73
1.72
1.70
1.65
1.62
1.60
1.58
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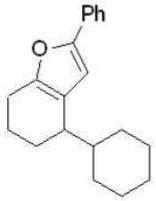
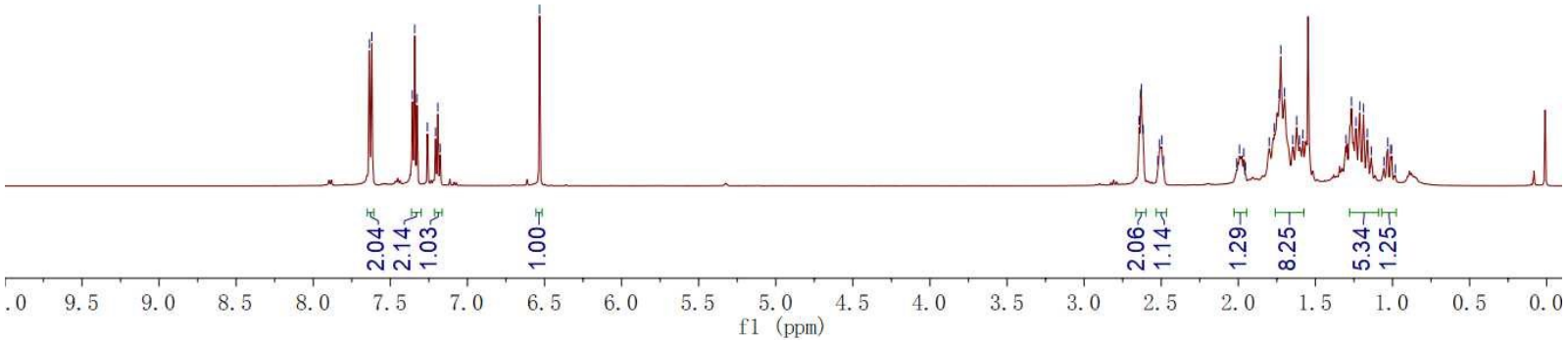
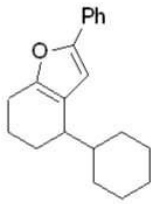
1.14
1.06
1.03
1.01
1.01
0.98


Ph
O

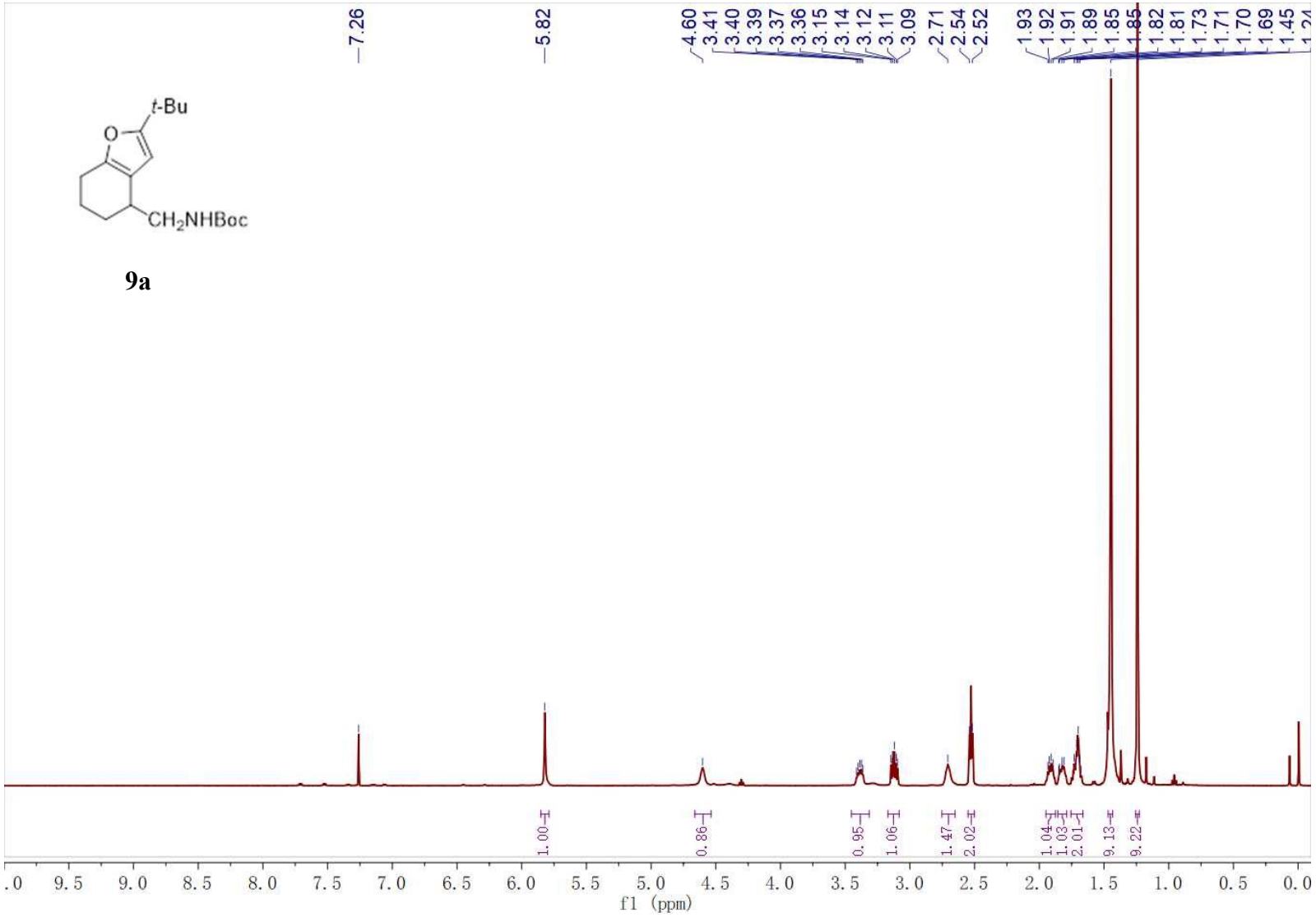
6h


2.04
2.14
1.03
1.00
2.06
1.14
1.29
8.25
5.34
1.25
.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

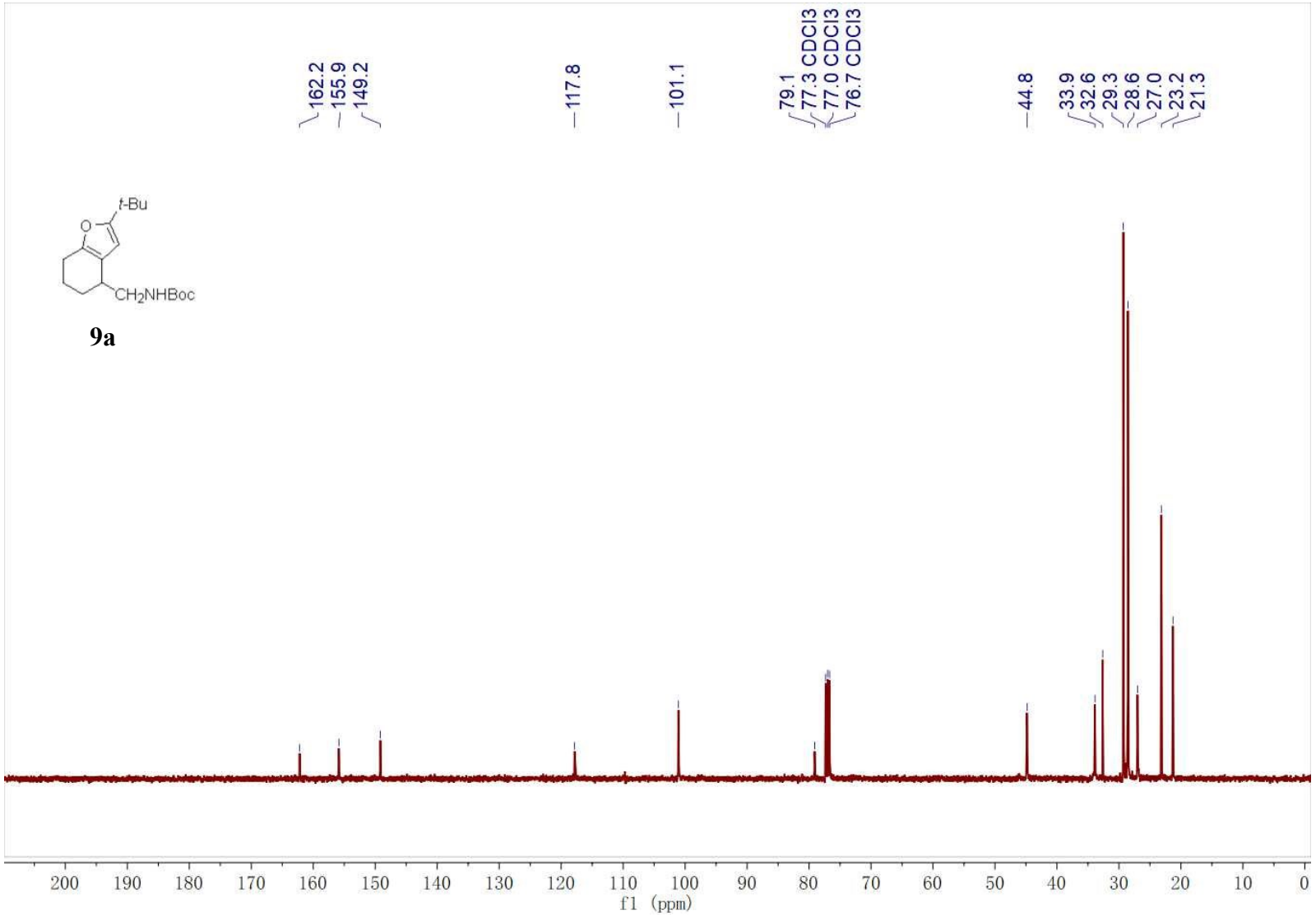

Ph
O

6h

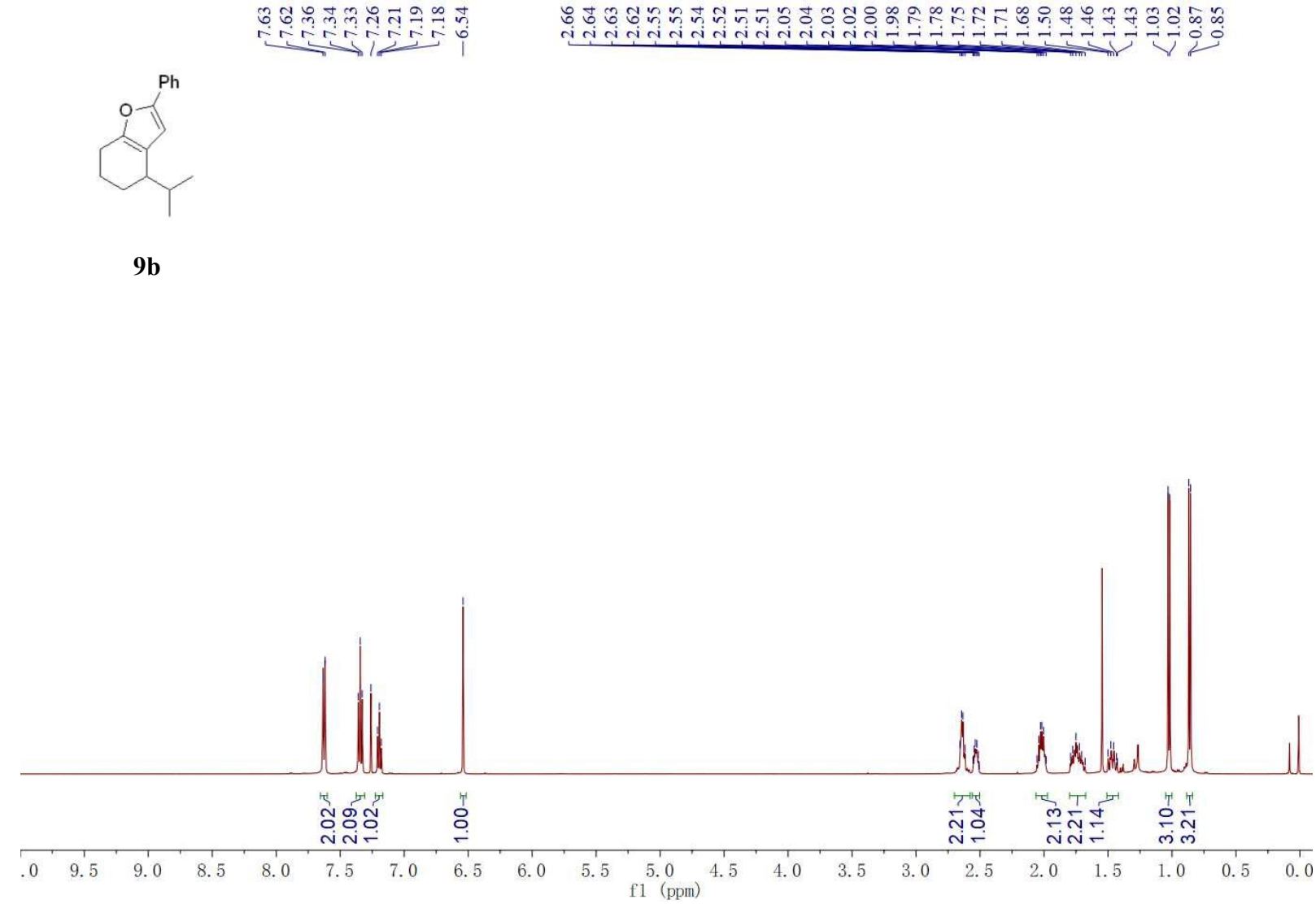
151.3
151.2
131.5
128.5
126.5
123.2
122.2
105.6
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
41.6
38.9
31.4
29.1
27.0
26.9
26.8
24.8
23.4
21.8
f1 (ppm)


7.26
5.82
4.60
3.41
3.40
3.39
3.37
3.36
3.15
3.14
3.12
3.11
3.09
2.71
2.54
2.52
1.93
1.92
1.91
1.89
1.85
1.85
1.82
1.81
1.73
1.71
1.70
1.69
1.45
t-Bu
O
CH2NHBoc
1.00
0.86
0.95
1.06
1.47
2.02
1.04
1.03
2.01
9.13
9.22
f1 (ppm)

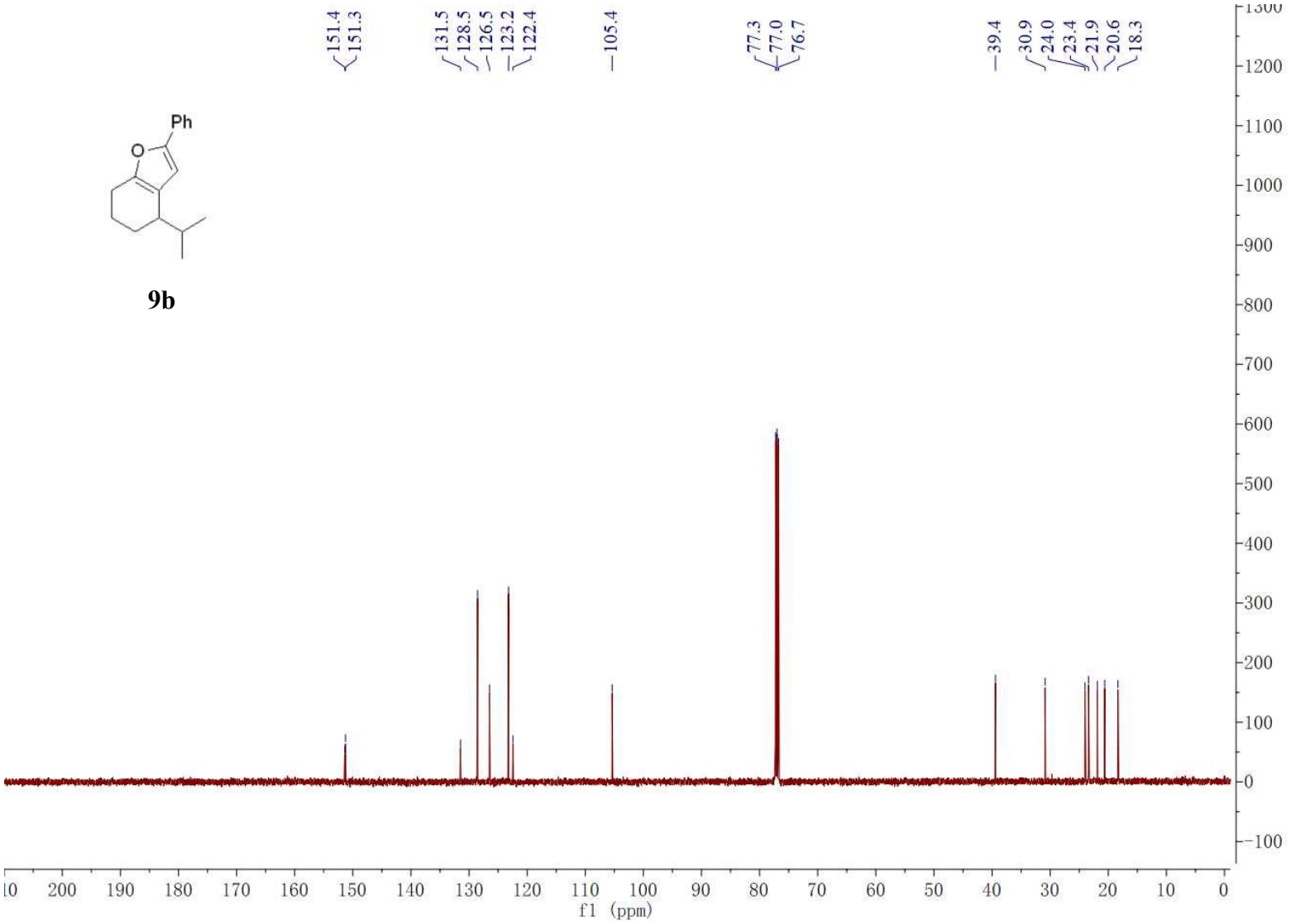
9a


162.2
155.9
149.2
117.8
101.1
79.1
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
44.8
33.9
32.6
29.3
28.6
27.0
23.2
21.3
t-Bu
O
CH2NHBoc
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

9a


7.63
7.62
7.36
7.34
7.33
7.26
7.21
7.19
7.18
6.54
2.66
2.64
2.63
2.62
2.55
2.55
2.54
2.52
2.51
2.51
2.05
2.04
2.03
2.02
2.00
1.98
1.79
1.78
1.75
1.72
1.71
1.68
1.50
1.48
1.46
1.43
1.43
1.03
1.02
0.87
0.85
Ph
O
2.02
2.09
1.02
1.00
2.21
1.04
2.13
2.21
1.14
3.10
3.21
f1 (ppm)

9b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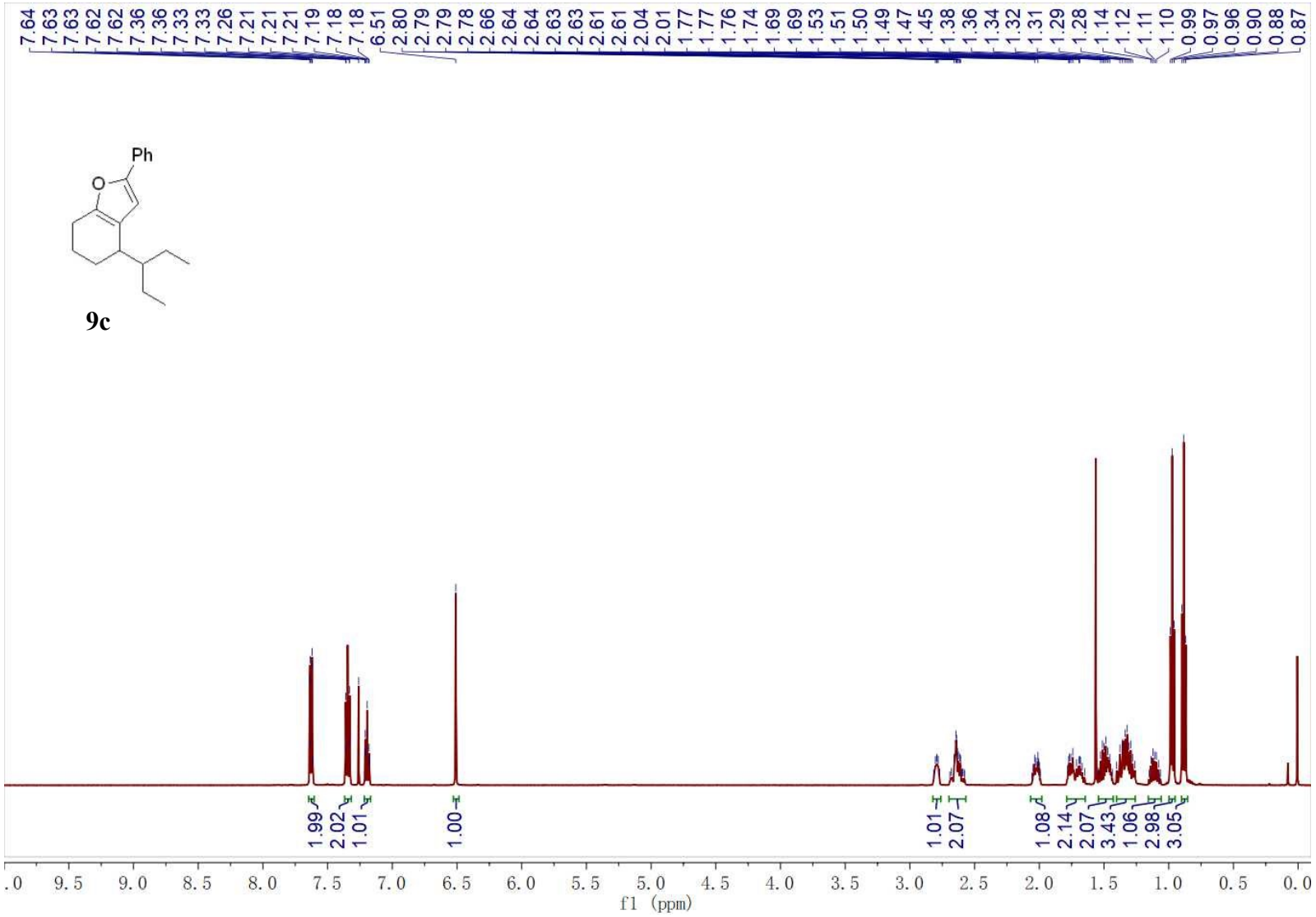
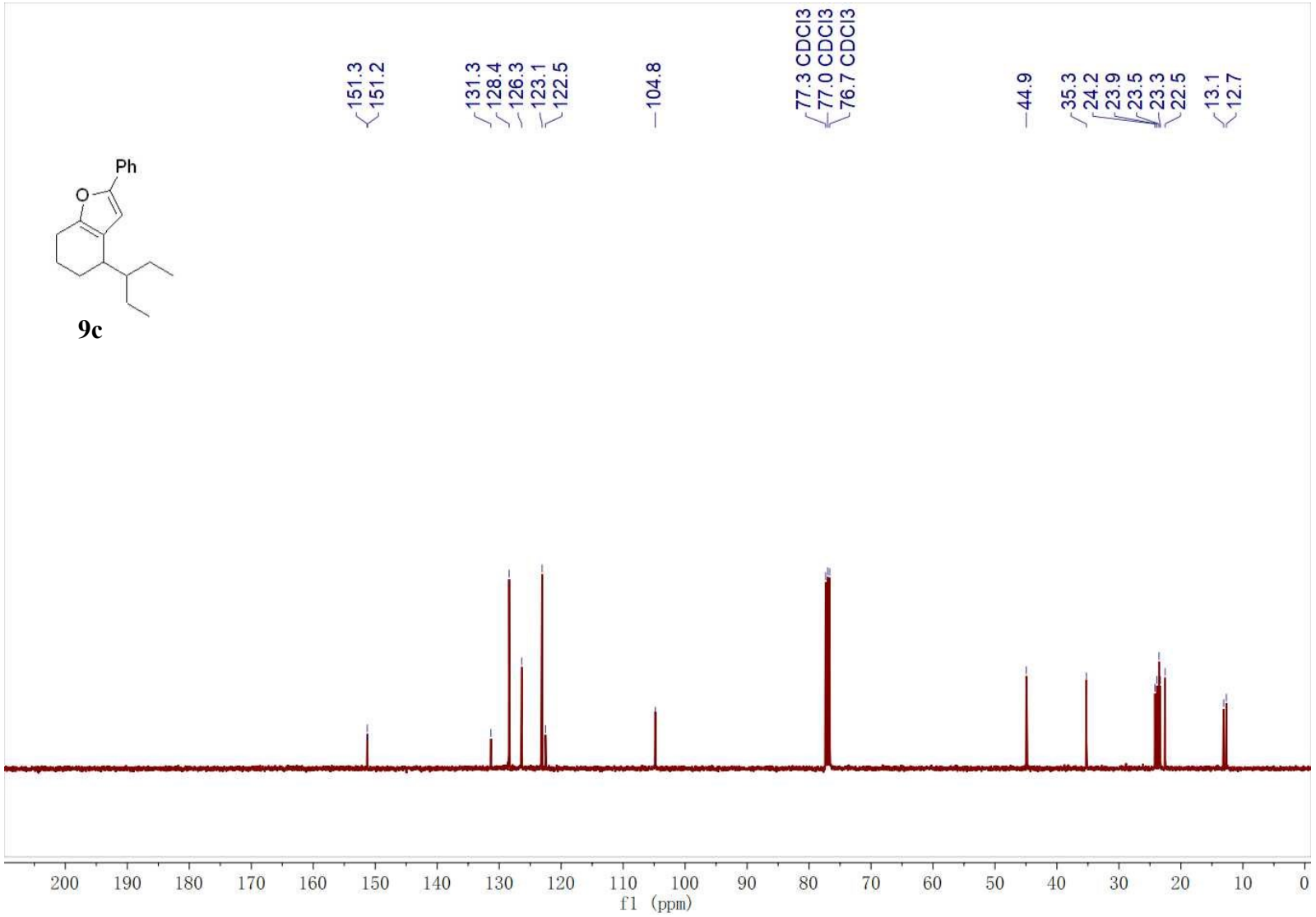
151.3
131.5
128.5
126.5
123.2
122.4
105.4
77.3
77.0
76.7
39.4
30.9
24.0
23.4
21.9
20.6
18.3
Ph
O
f1 (ppm)

9b


7.64 7.63 7.63 7.62 7.62 7.36 7.36 7.33 7.33 7.26 7.21 7.21 7.21 7.19 7.18 7.18 6.51 2.80 2.79 2.79 2.78 2.66 2.64 2.64 2.63 2.63 2.61 2.61 2.04 2.01 1.77 1.77 1.76 1.74 1.69 1.69 1.53 1.51 1.50 1.49 1.47 1.45 1.38 1.36 1.34 1.32 1.31 1.29 1.28 1.14 1.12 1.11 1.10 0.99 0.97 0.96 0.90 0.88 0.87
Ph
O
1.99 2.02 1.01 1.00 1.01 2.07 1.08 2.14 2.07 3.43 1.06 2.98 3.05
f1 (ppm)

9c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151.2
131.3
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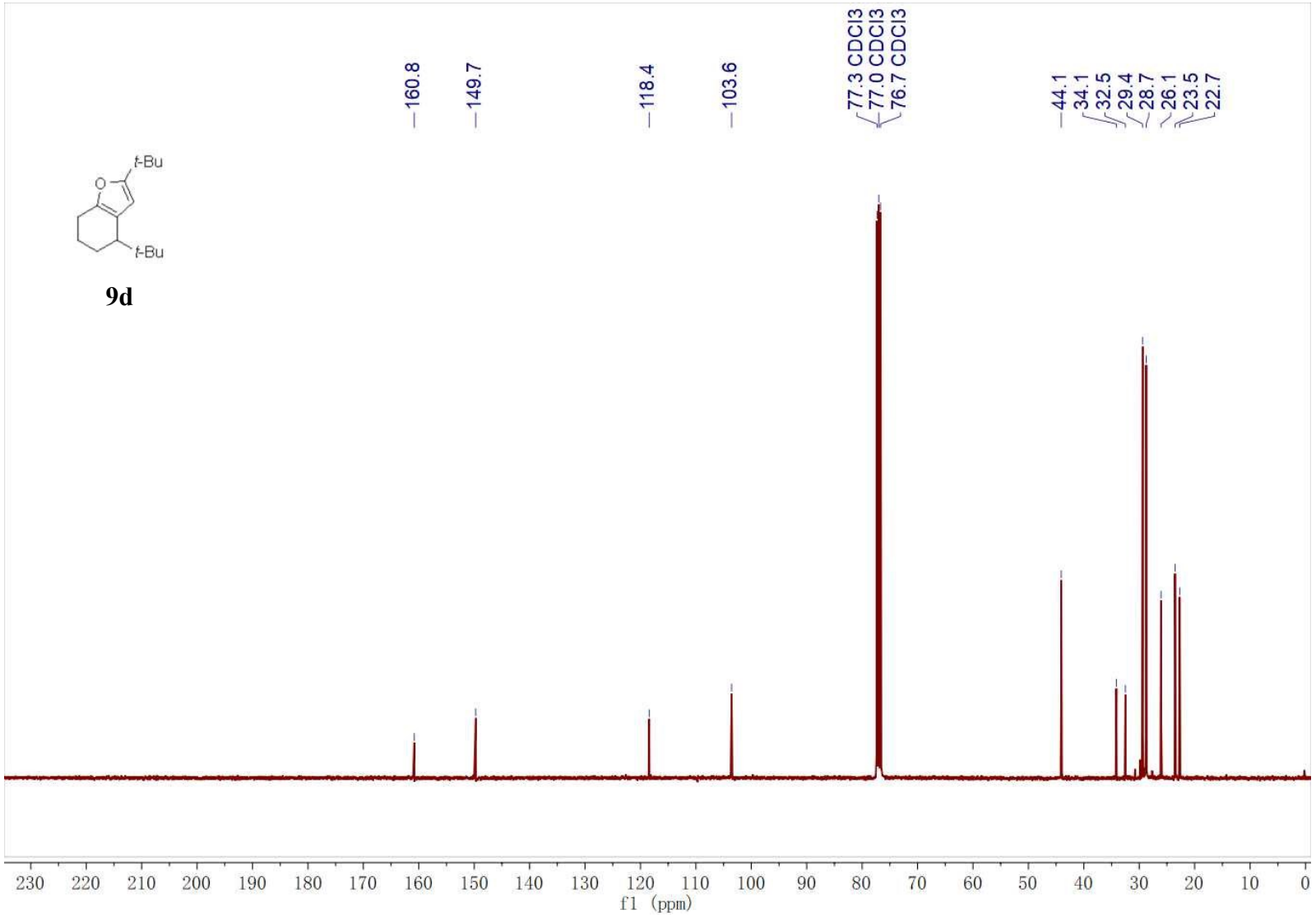
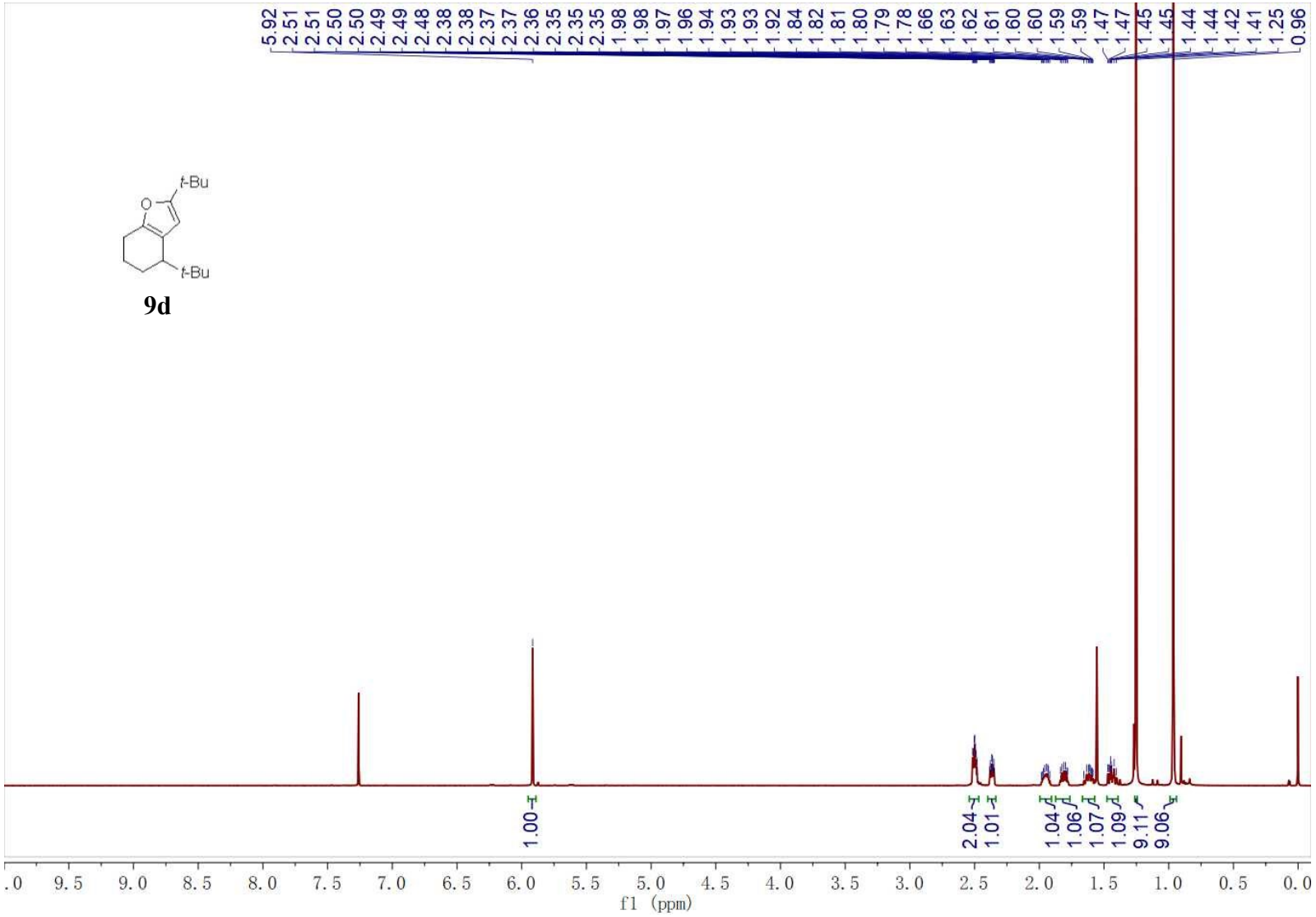
126.3
123.1
122.5
104.8
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
44.9
35.3
24.2
23.9
23.5
23.3
22.5
13.1
12.7
Ph
O
9c
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


160.8
149.7
118.4
103.6
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
44.1
34.1
32.5
29.4
28.7
26.1
23.5
22.7
t-Bu
t-Bu
O
9d
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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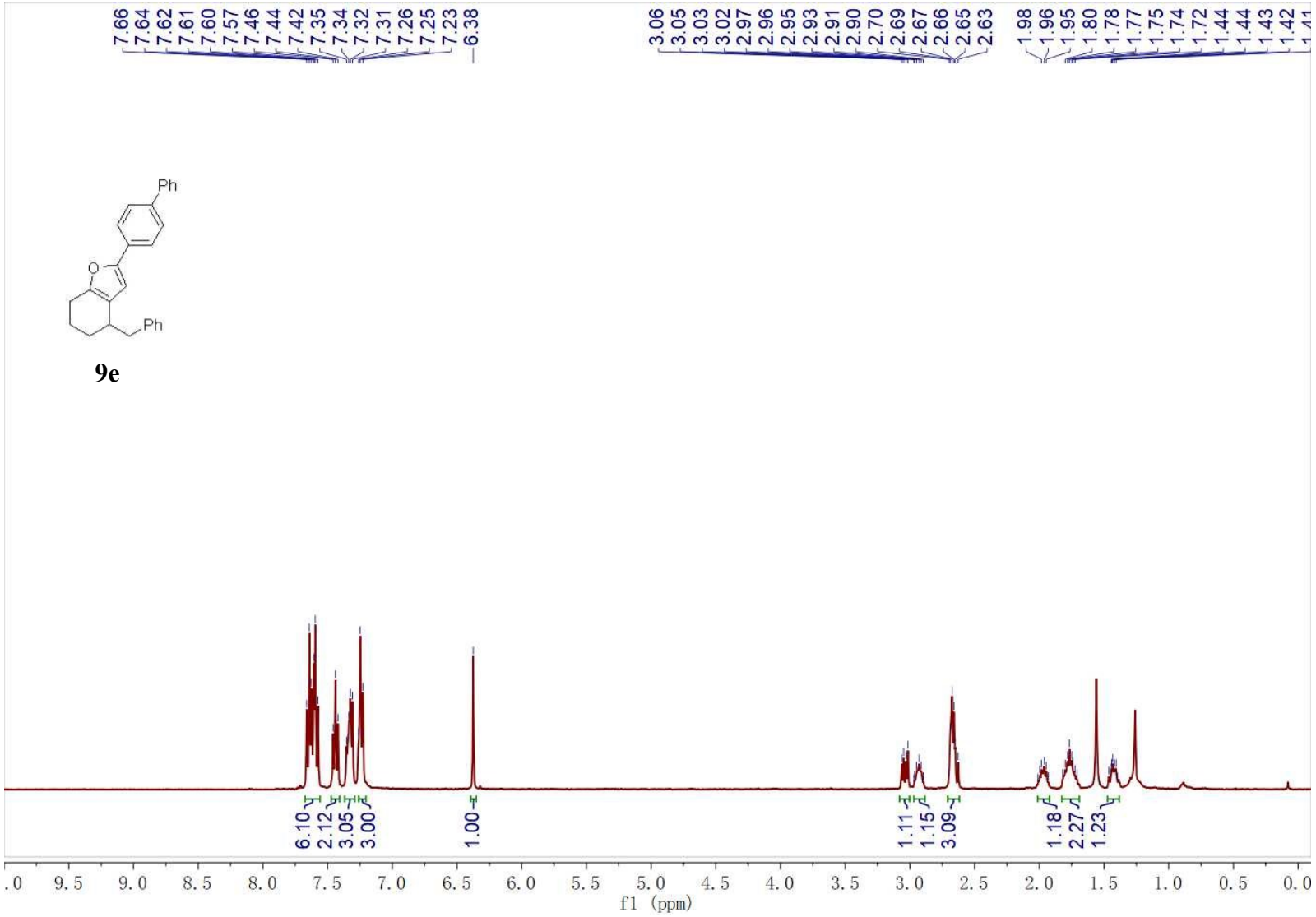
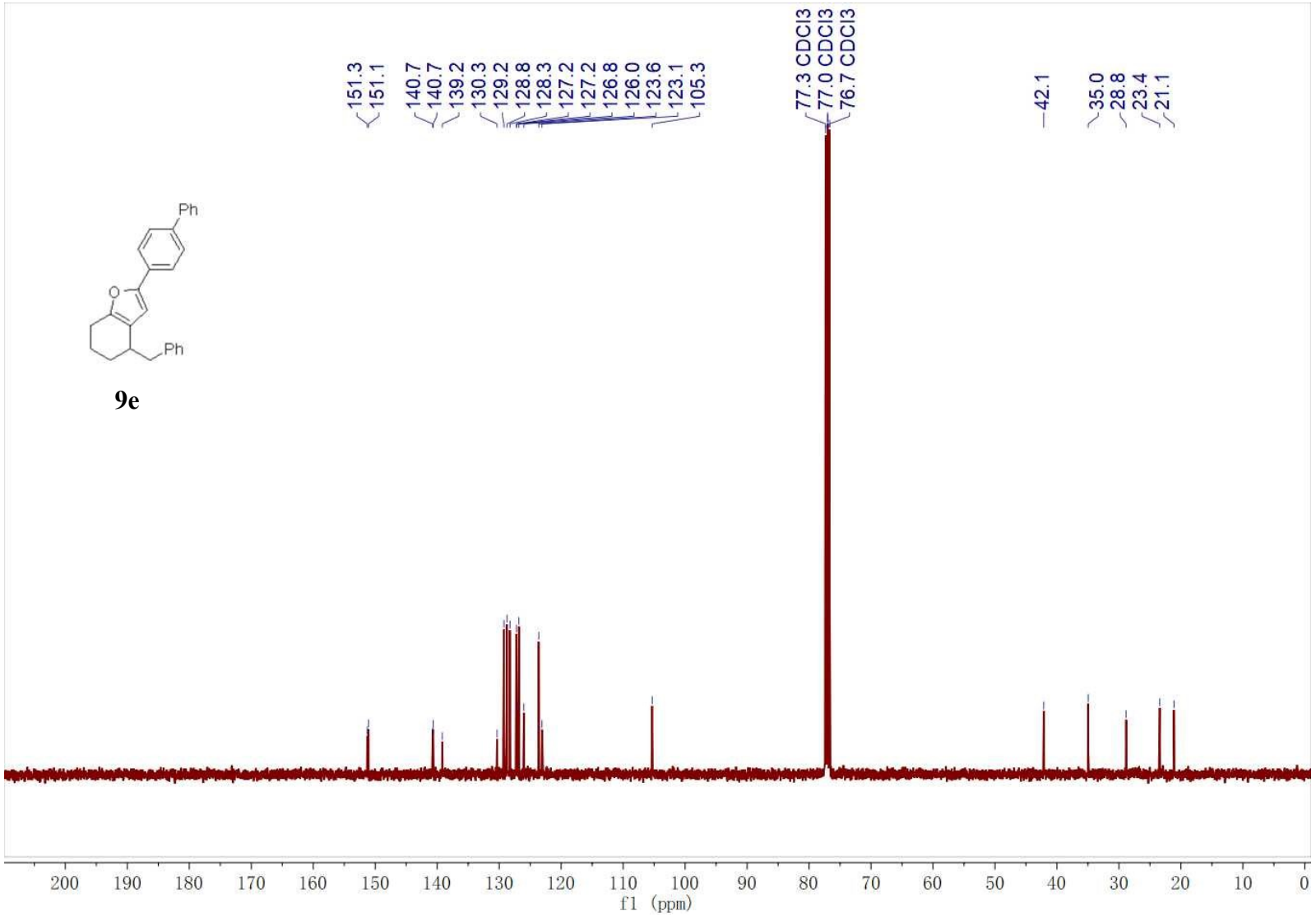
2.51
2.50
2.50
2.49
2.49
2.48
2.38
2.38
2.37
2.37
2.36
2.35
2.35
2.35
1.98
1.98
1.97
1.96
1.94
1.93
1.93
1.92
1.84
1.82
1.81
1.80
1.79
1.78
1.66
1.63
1.62
1.61
1.60
1.60
1.59
1.59
1.47
1.47
1.45
1.45
1.44
1.44
1.42
1.41
1.25
0.96
t-Bu
t-Bu
1.00
2.04
1.01
1.04
1.06
1.07
1.09
9.11
9.06
f1 (ppm)

9d


7.66
7.64
7.62
7.61
7.60
7.57
7.46
7.44
7.42
7.35
7.34
7.32
7.31
7.26
7.25
7.23
6.38
3.06
3.05
3.03
3.02
2.97
2.96
2.95
2.93
2.91
2.90
2.70
2.69
2.67
2.66
2.65
2.63
1.98
1.96
1.95
1.80
1.78
1.77
1.75
1.74
1.72
1.44
1.44
1.43
1.42
Ph
O
Ph
6.10
2.12
3.05
3.00
1.00
1.11
1.15
3.09
1.18
2.27
1.23
f1 (ppm)

9e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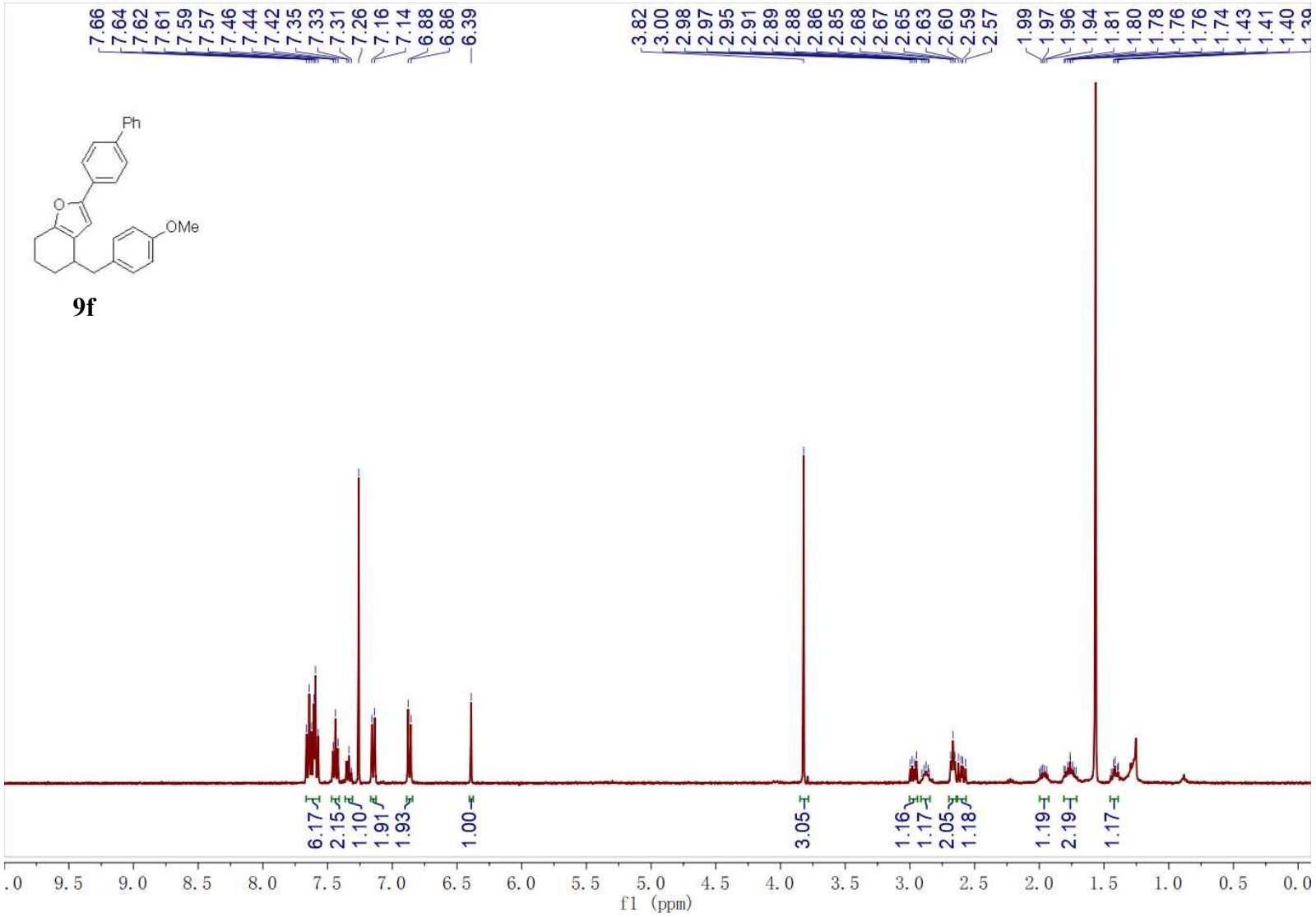
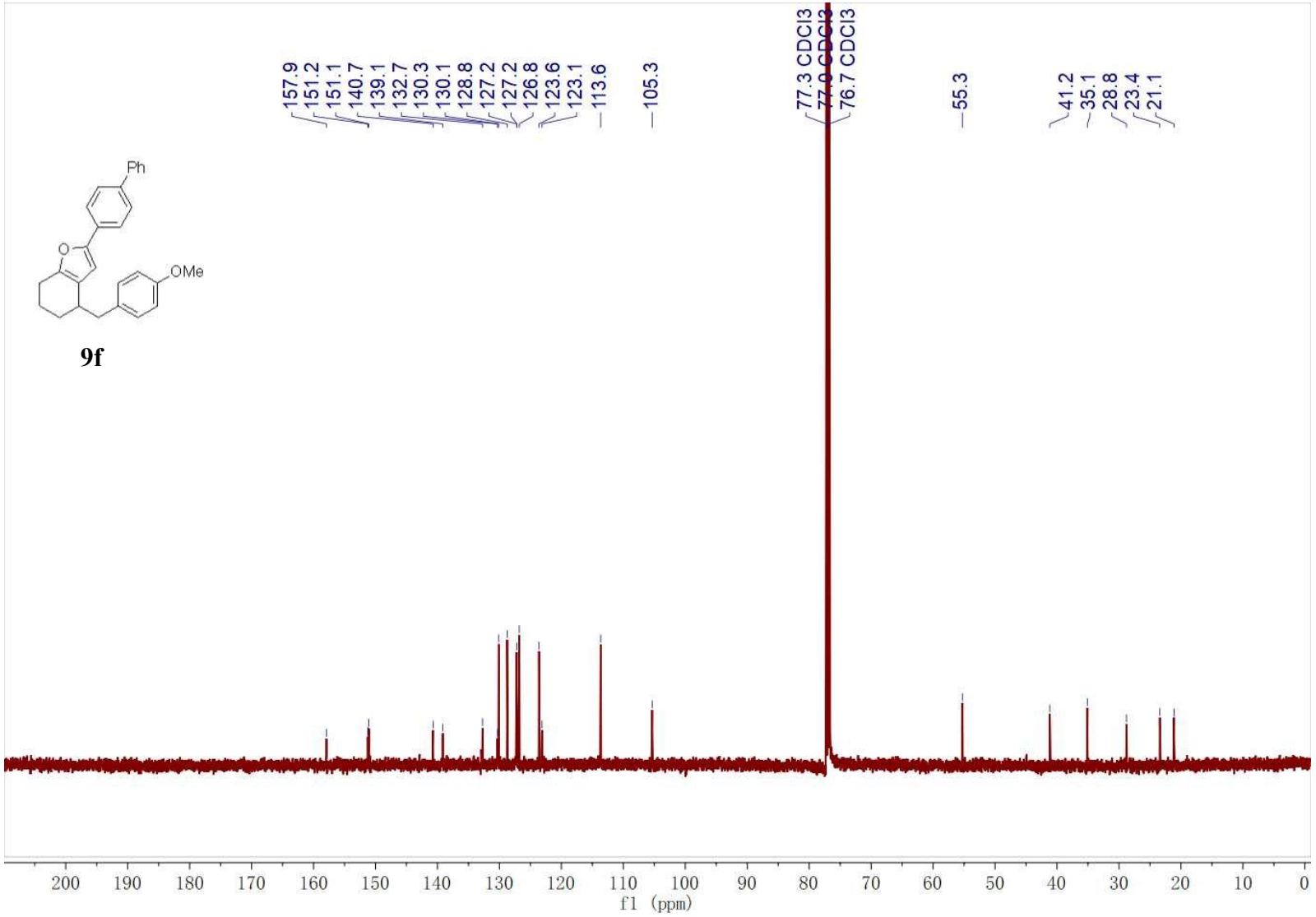
151.1
140.7
140.7
139.2
130.3
129.2
128.8
128.3
127.2
127.2
126.8
126.0
123.6
123.1
105.3
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
42.1
35.0
28.8
23.4
21.1
Ph
O
Ph
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

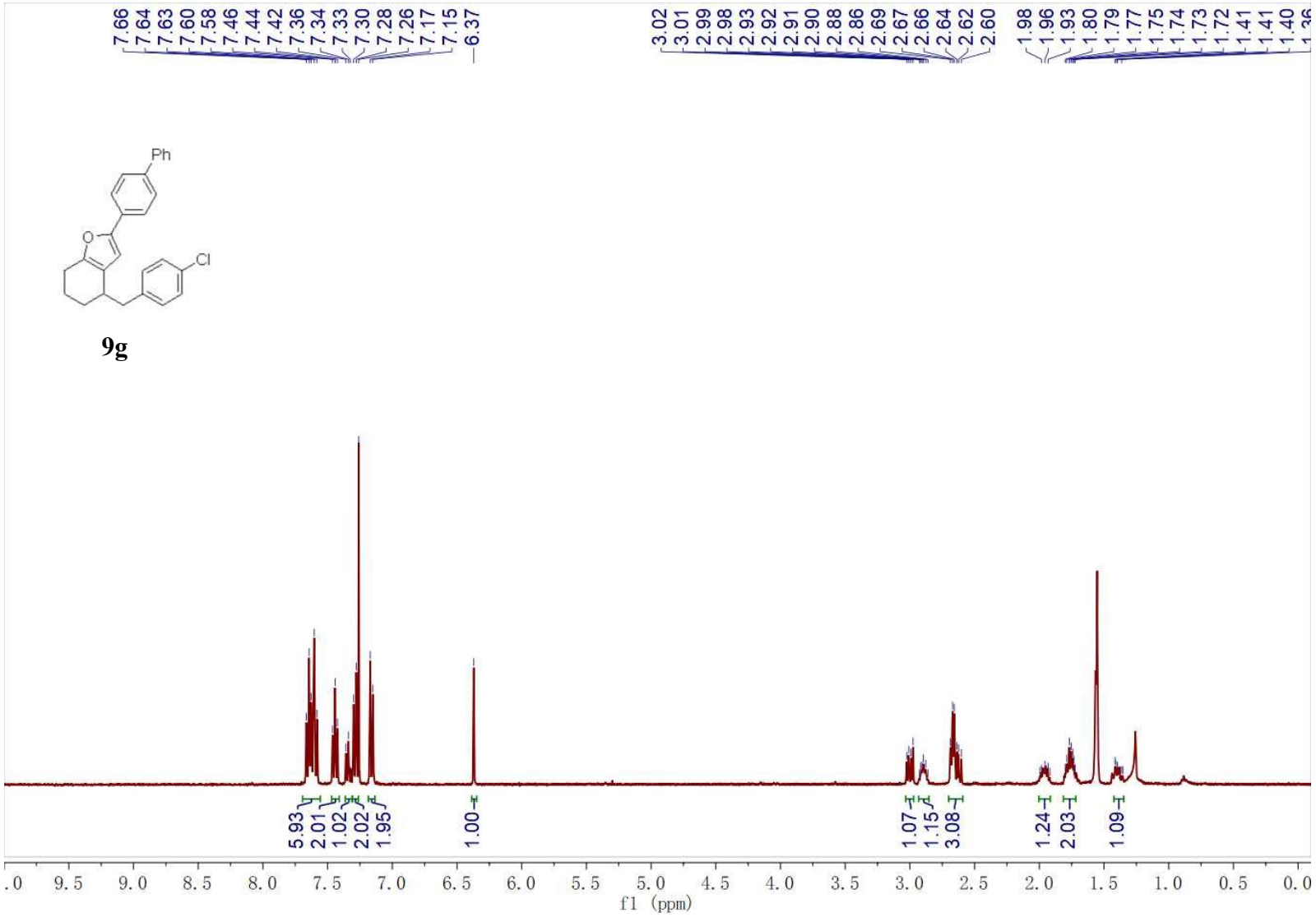
9e


7.66
7.64
7.62
7.61
7.59
7.57
7.46
7.44
7.42
7.35
7.33
7.31
7.26
7.16
7.14
6.88
6.86
6.39
3.82
3.00
2.98
2.97
2.95
2.91
2.89
2.88
2.86
2.85
2.68
2.67
2.65
2.63
2.60
2.59
2.57
1.99
1.97
1.96
1.94
1.81
1.80
1.78
1.76
1.76
1.74
1.43
1.41
1.40
Ph
O
OMe
6.17
2.15
1.10
1.91
1.93
1.00
3.05
1.16
1.17
2.05
1.18
1.19
2.19
1.17
f1 (ppm)

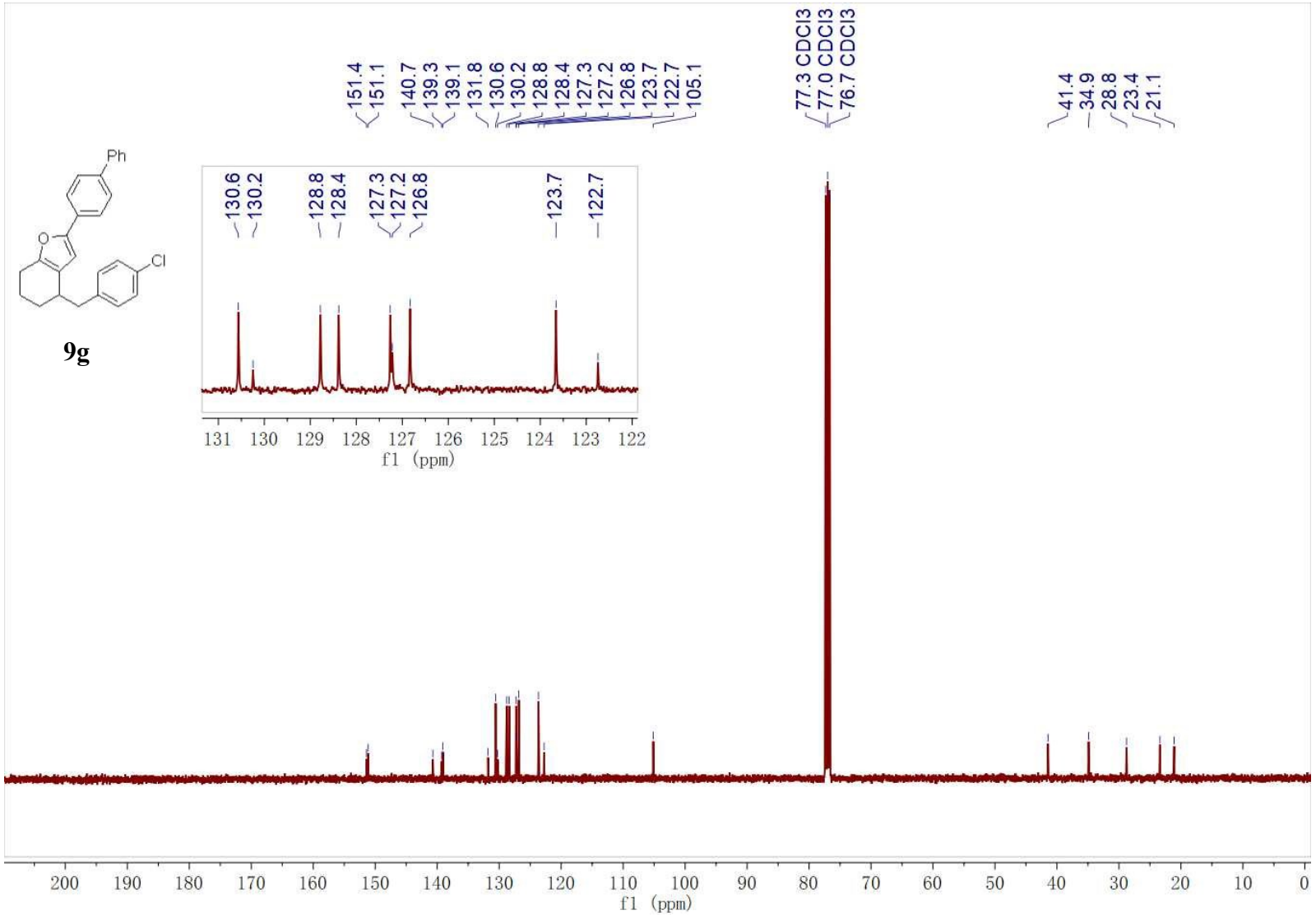
9f

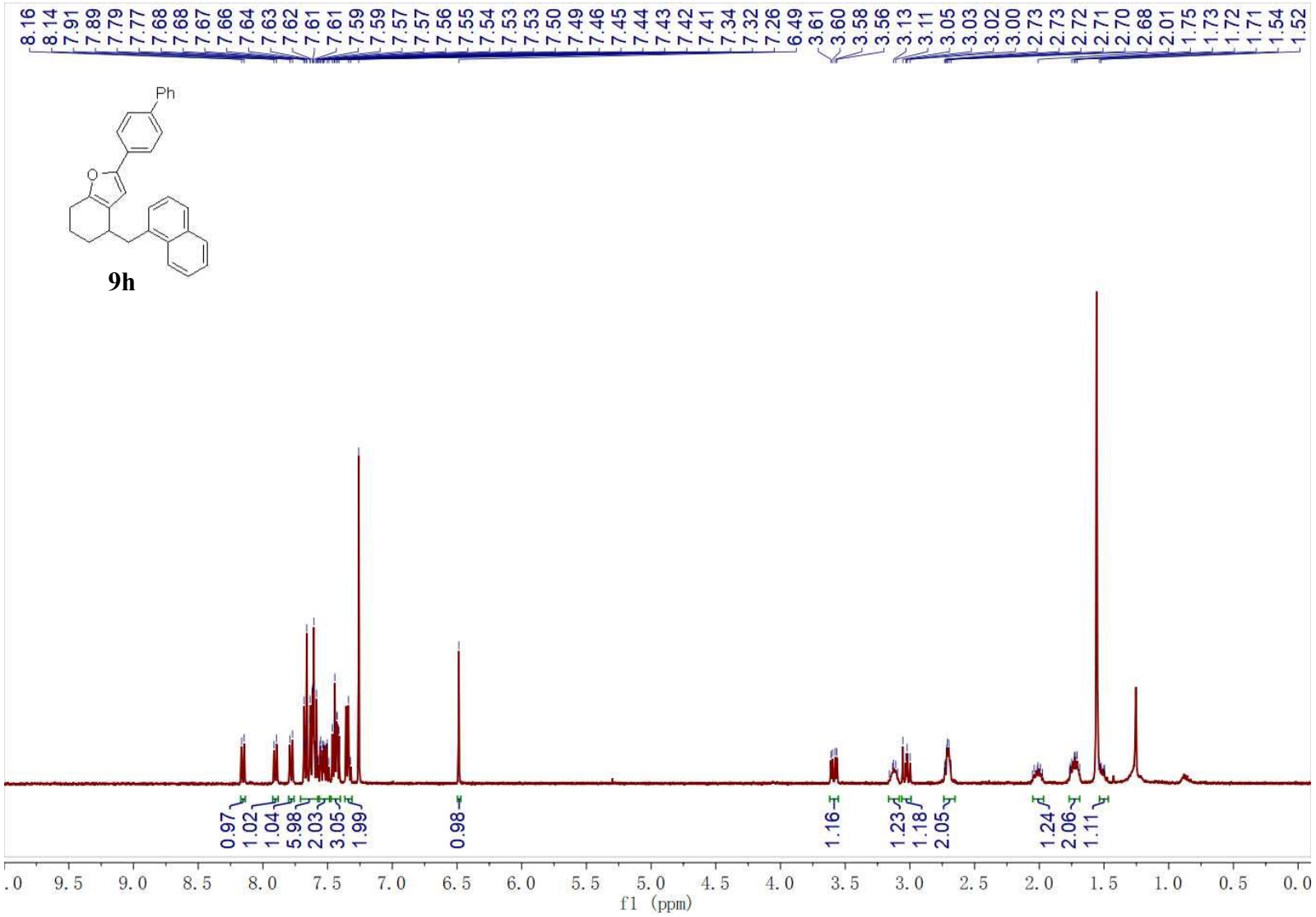

157.9
151.2
151.1
140.7
139.1
132.7
130.3
130.1
128.8
127.2
127.2
126.8
123.6
123.1
113.6
105.3
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
55.3
41.2
35.1
28.8
23.4
21.1
Ph
O
OMe
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

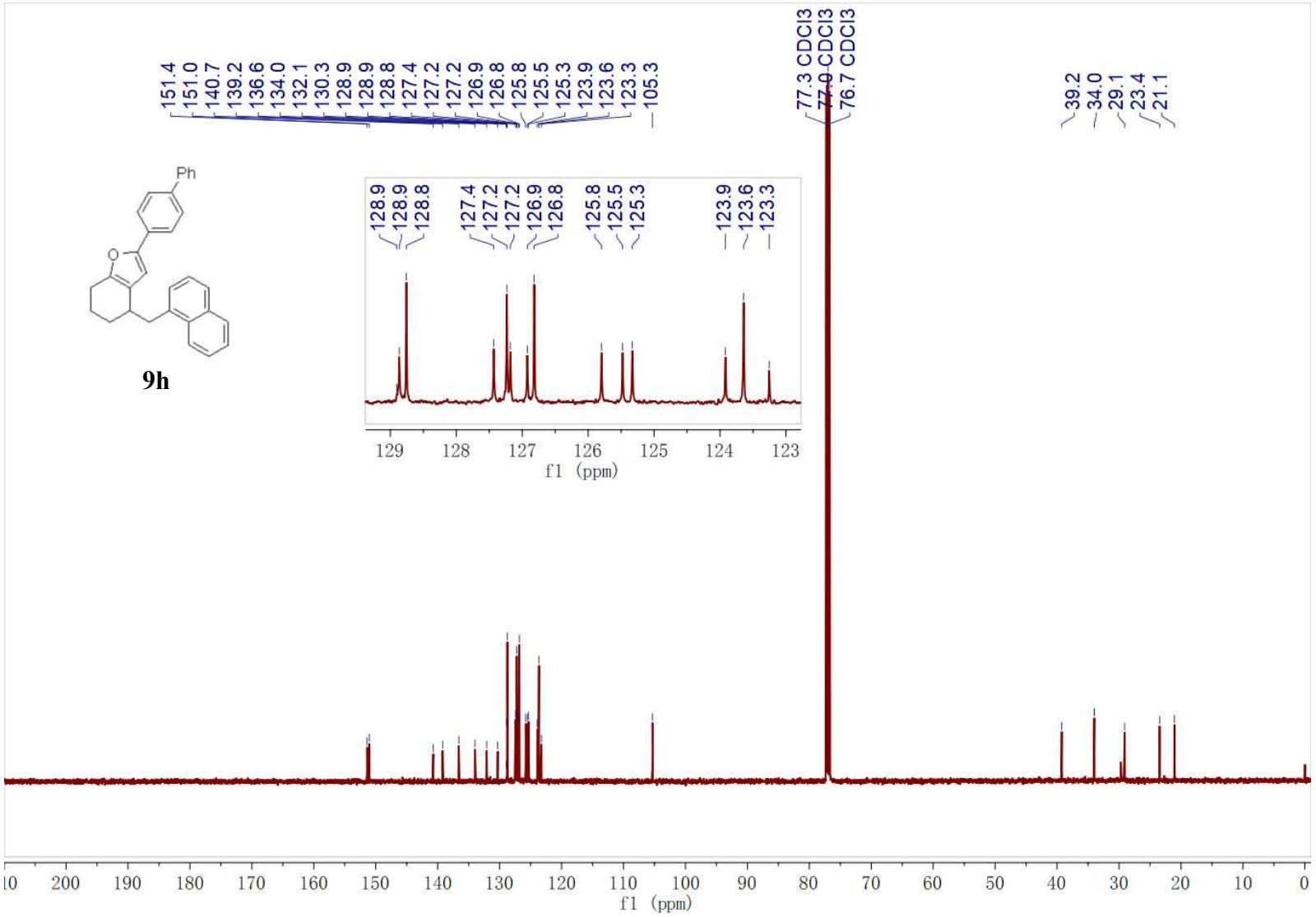
9f


7.66
7.64
7.63
7.60
7.58
7.46
7.44
7.42
7.36
7.34
7.33
7.30
7.28
7.26
7.17
7.15
6.37
3.02
3.01
2.99
2.98
2.93
2.92
2.91
2.90
2.88
2.86
2.69
2.67
2.66
2.64
2.62
2.60
1.98
1.96
1.93
1.80
1.79
1.77
1.75
1.74
1.73
1.72
1.41
1.41
1.40
Ph
O
Cl
5.93
2.01
1.02
2.02
1.95
1.00
1.07
1.15
3.08
1.24
2.03
1.09
f1 (ppm)

9g


151.4
151.1
140.7
139.3
139.1
131.8
130.6
130.2
128.8
128.4
127.3
127.2
126.8
123.7
122.7
105.1
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
41.4
34.9
28.8
23.4
21.1
Ph
Cl
O
9g
f1 (ppm)


8.16 8.14 7.91 7.89 7.79 7.77 7.68 7.68 7.67 7.66 7.64 7.63 7.62 7.61 7.61 7.59 7.59 7.57 7.57 7.56 7.55 7.54 7.53 7.53 7.50 7.49 7.46 7.45 7.44 7.43 7.42 7.41 7.34 7.32 7.26 6.49 3.61 3.60 3.58 3.56 3.13 3.11 3.05 3.03 3.02 3.00 2.73 2.73 2.72 2.71 2.70 2.68 2.01 1.75 1.73 1.72 1.71 1.54 1.52
Ph
O
9h
0.97 1.02 1.04 5.98 2.03 3.05 1.99 0.98 1.16 1.23 1.18 2.05 1.24 2.06 1.11
f1 (ppm)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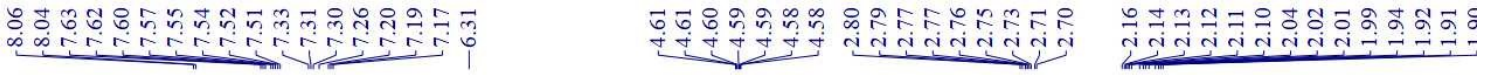
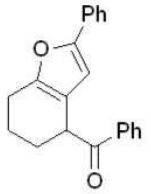
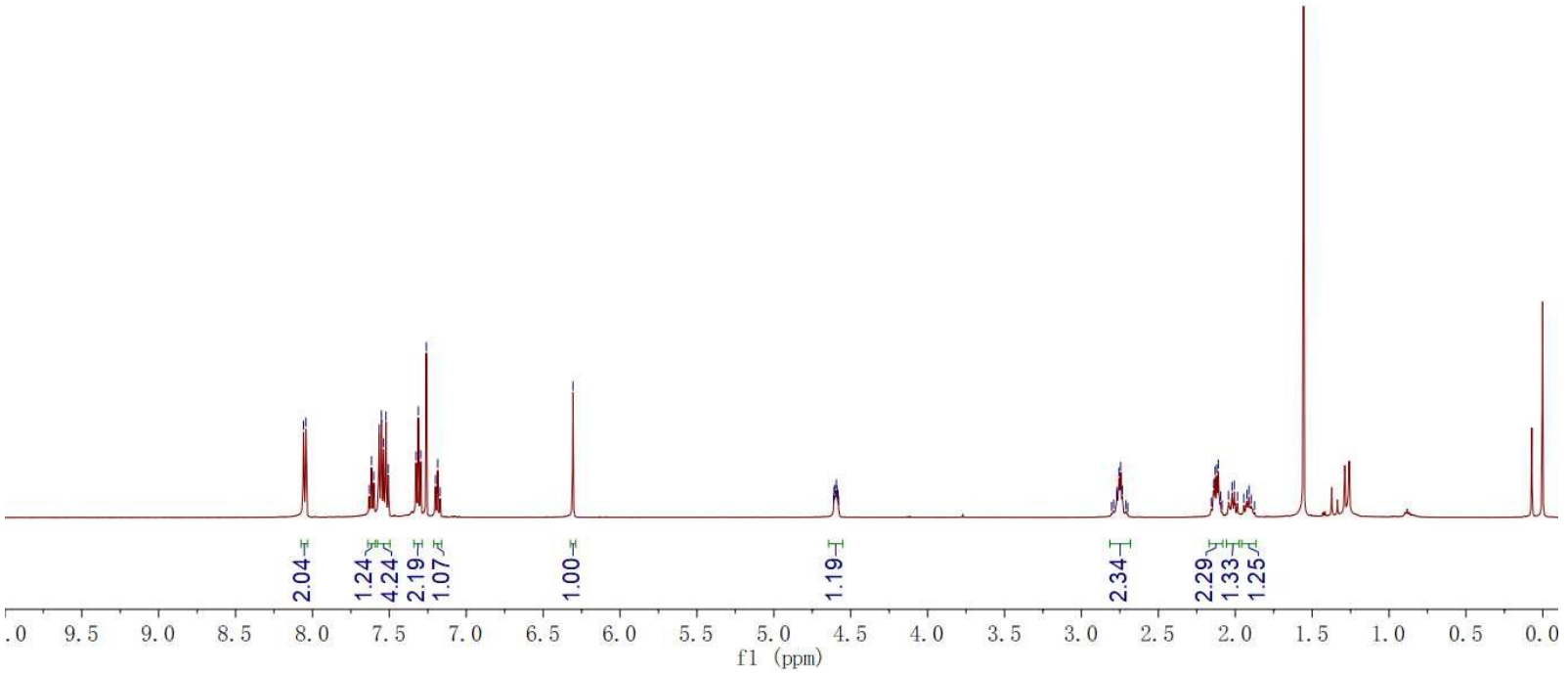
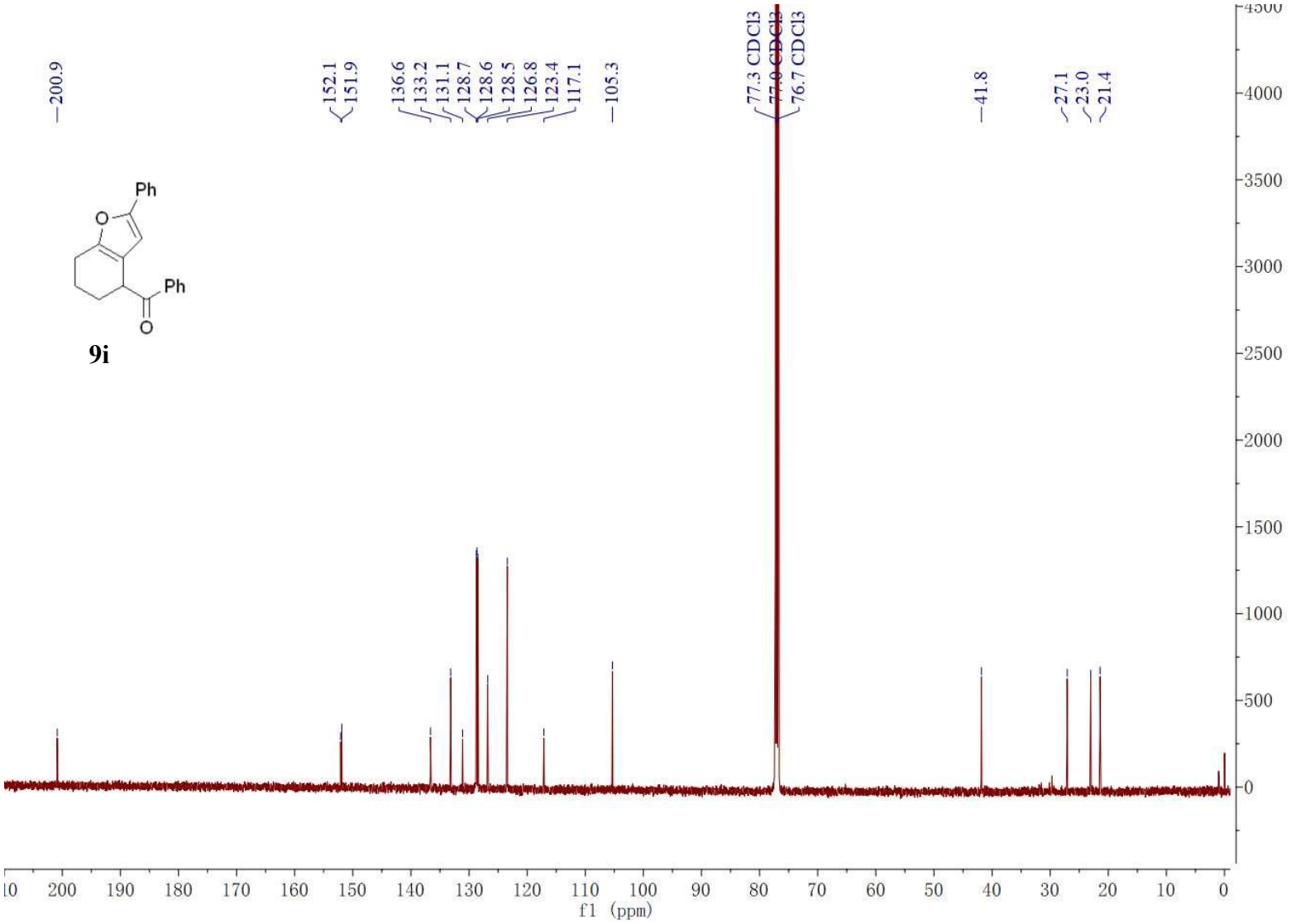
151.0
140.7
139.2
136.6
134.0
132.1
130.3
128.9
128.9
128.8
127.4
127.2
127.2
126.9
126.8
125.8
125.5
125.3
123.9
123.6
123.3
105.3
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
39.2
34.0
29.1
23.4
21.1
Ph
O
9h
f1 (ppm)

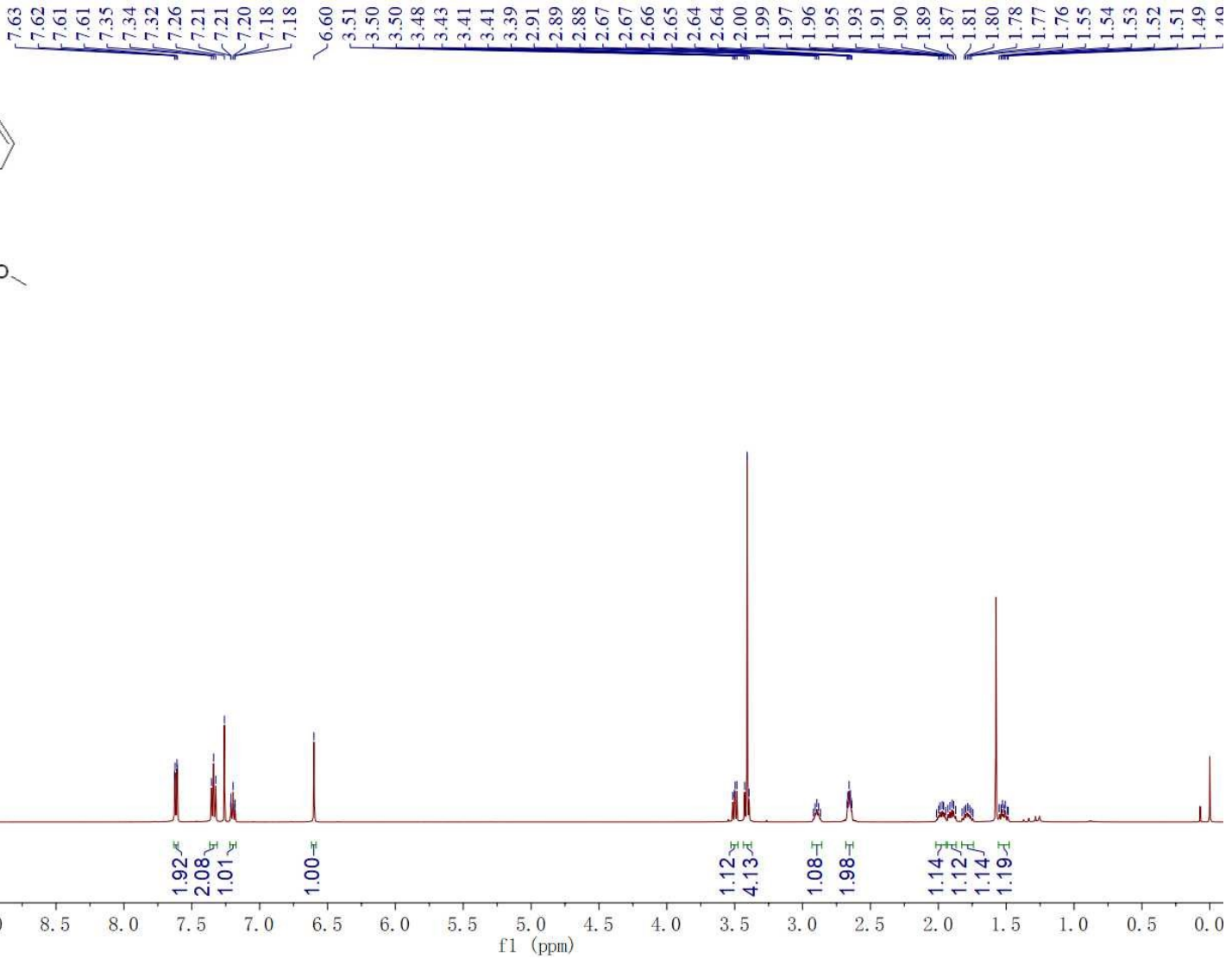

8.06
8.04
7.63
7.62
7.60
7.57
7.55
7.54
7.52
7.51
7.33
7.31
7.30
7.26
7.20
7.19
7.17
6.31
4.61
4.61
4.60
4.59
4.59
4.58
4.58
2.80
2.79
2.77
2.77
2.76
2.75
2.73
2.71
2.70
2.16
2.14
2.13
2.12
2.11
2.10
2.04
2.02
2.01
1.99
1.94
1.92
1.91


Ph
O
Ph
O

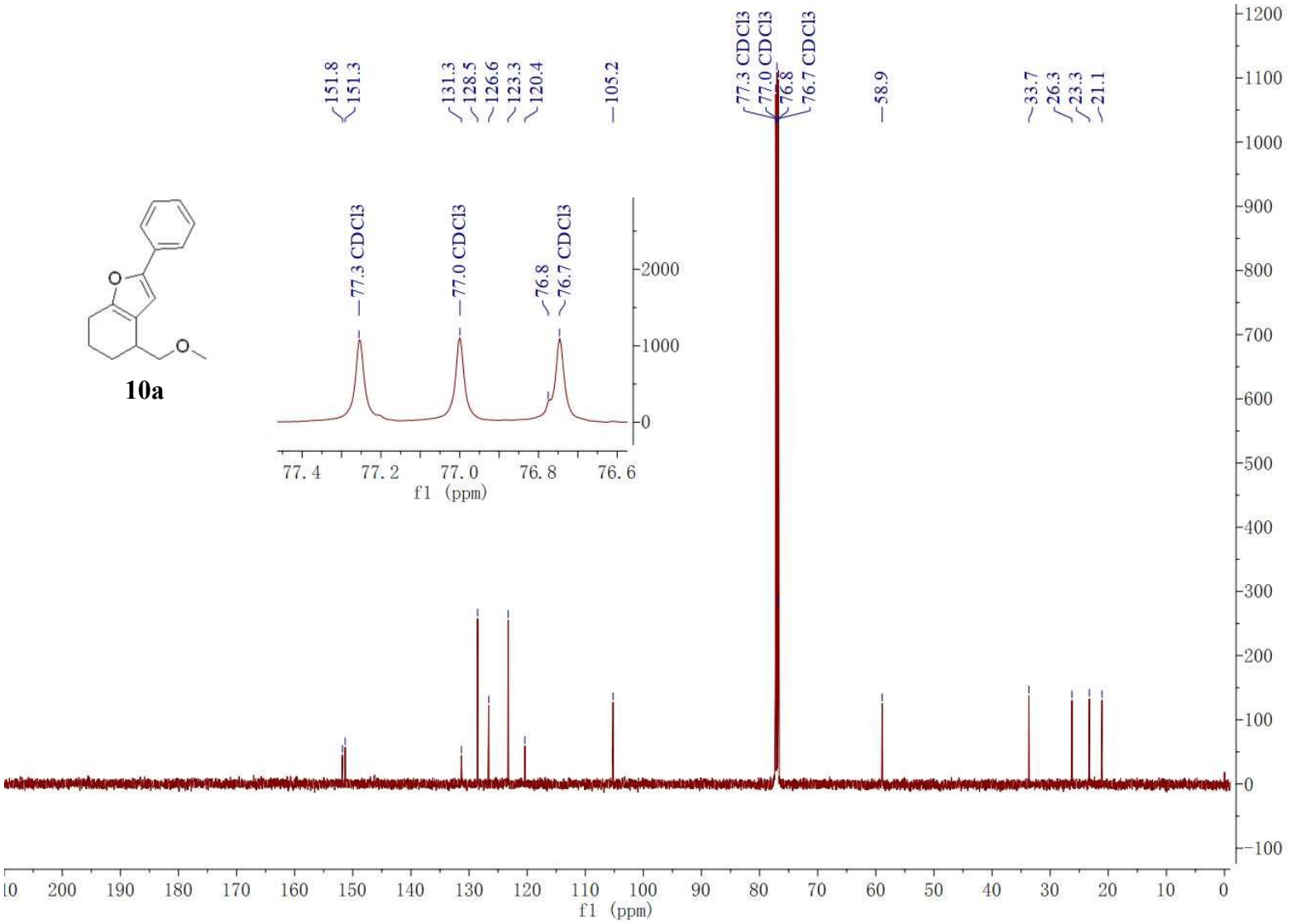
9i

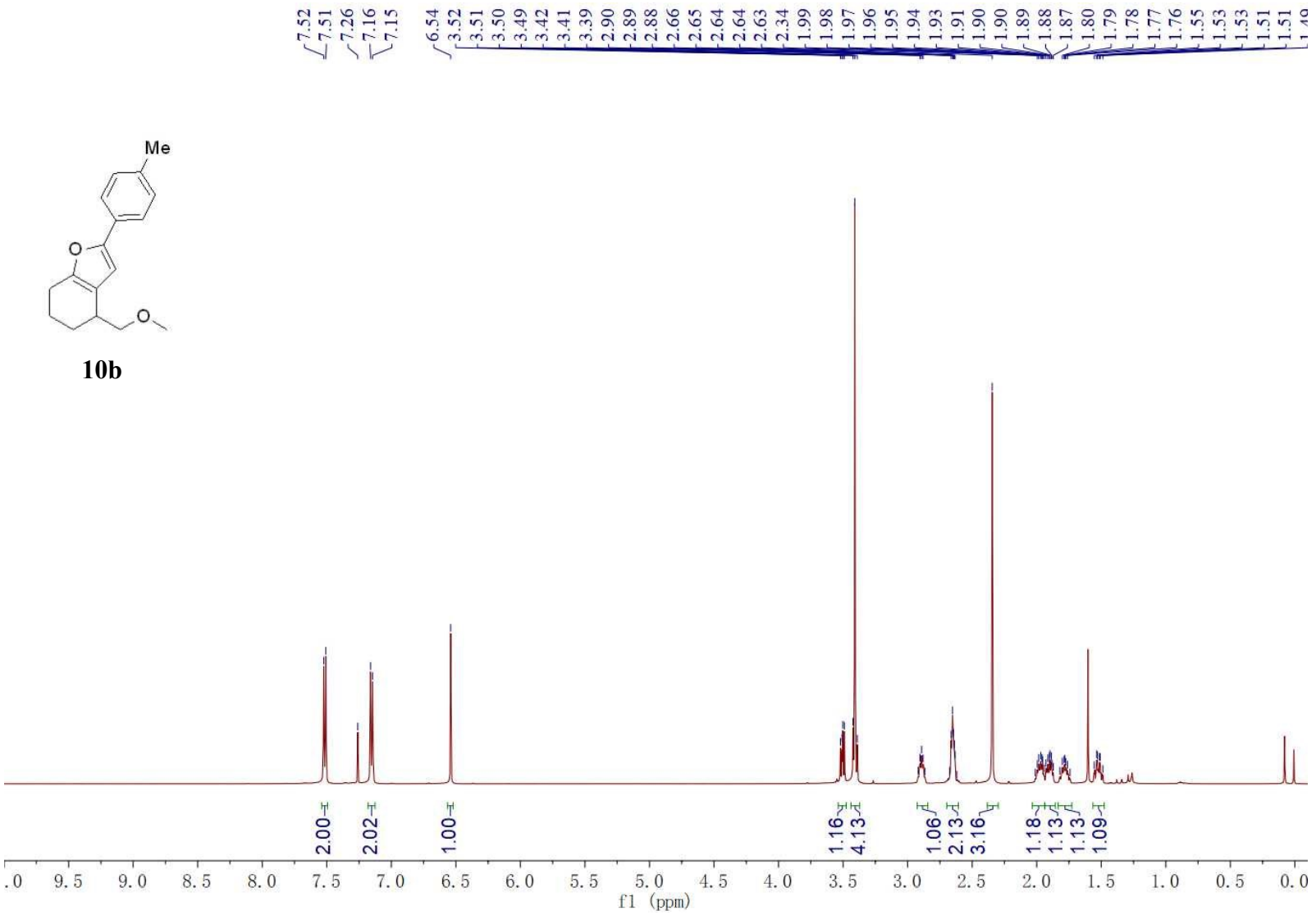

2.04
1.24
4.24
2.19
1.07
1.00
1.19
2.34
2.29
1.33
1.25
.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

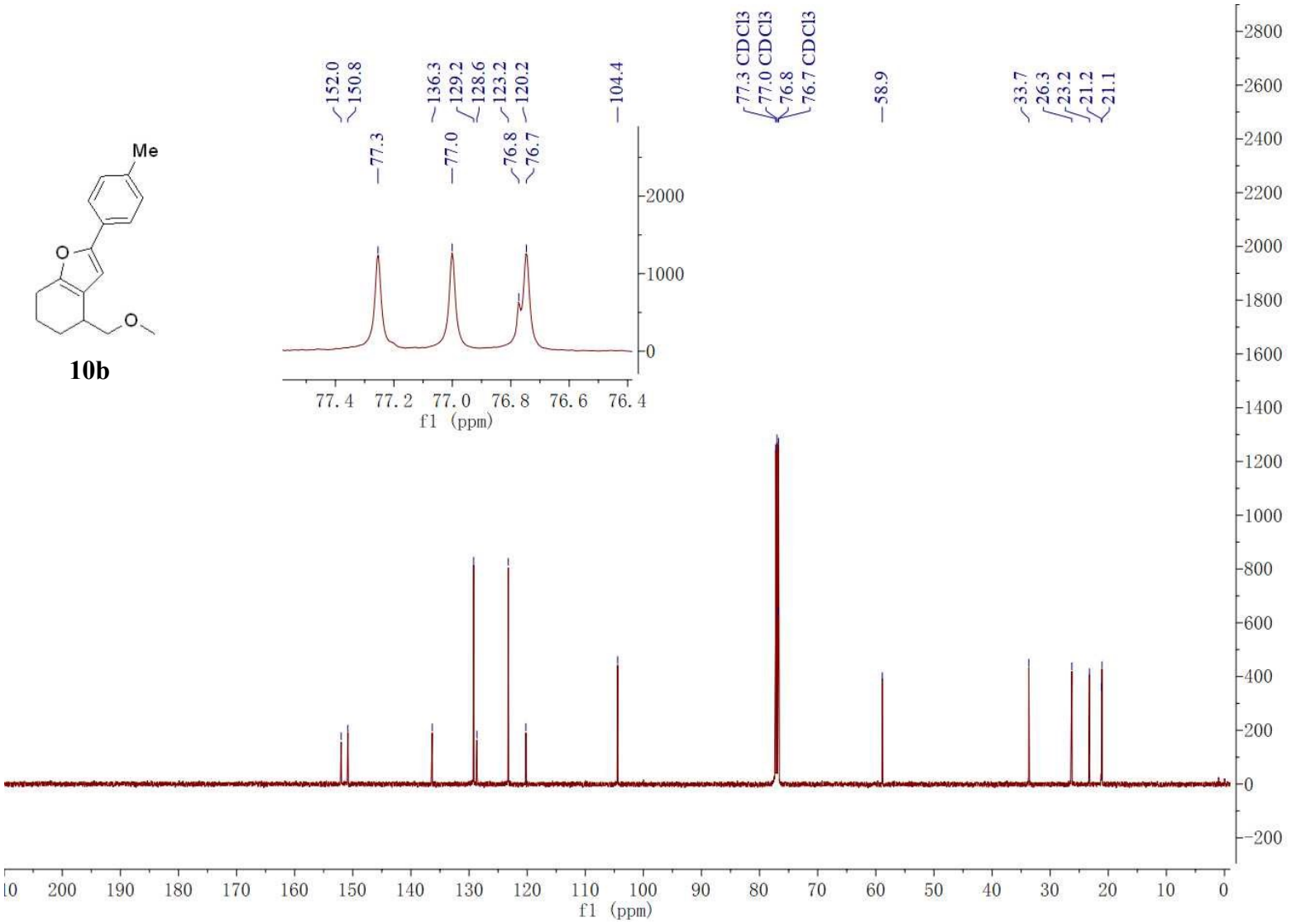

200.9
152.1
151.9
136.6
133.2
131.1
128.7
128.6
128.5
126.8
123.4
117.1
105.3
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
41.8
27.1
23.0
21.4
Ph
O
Ph
O
9i
f1 (ppm)

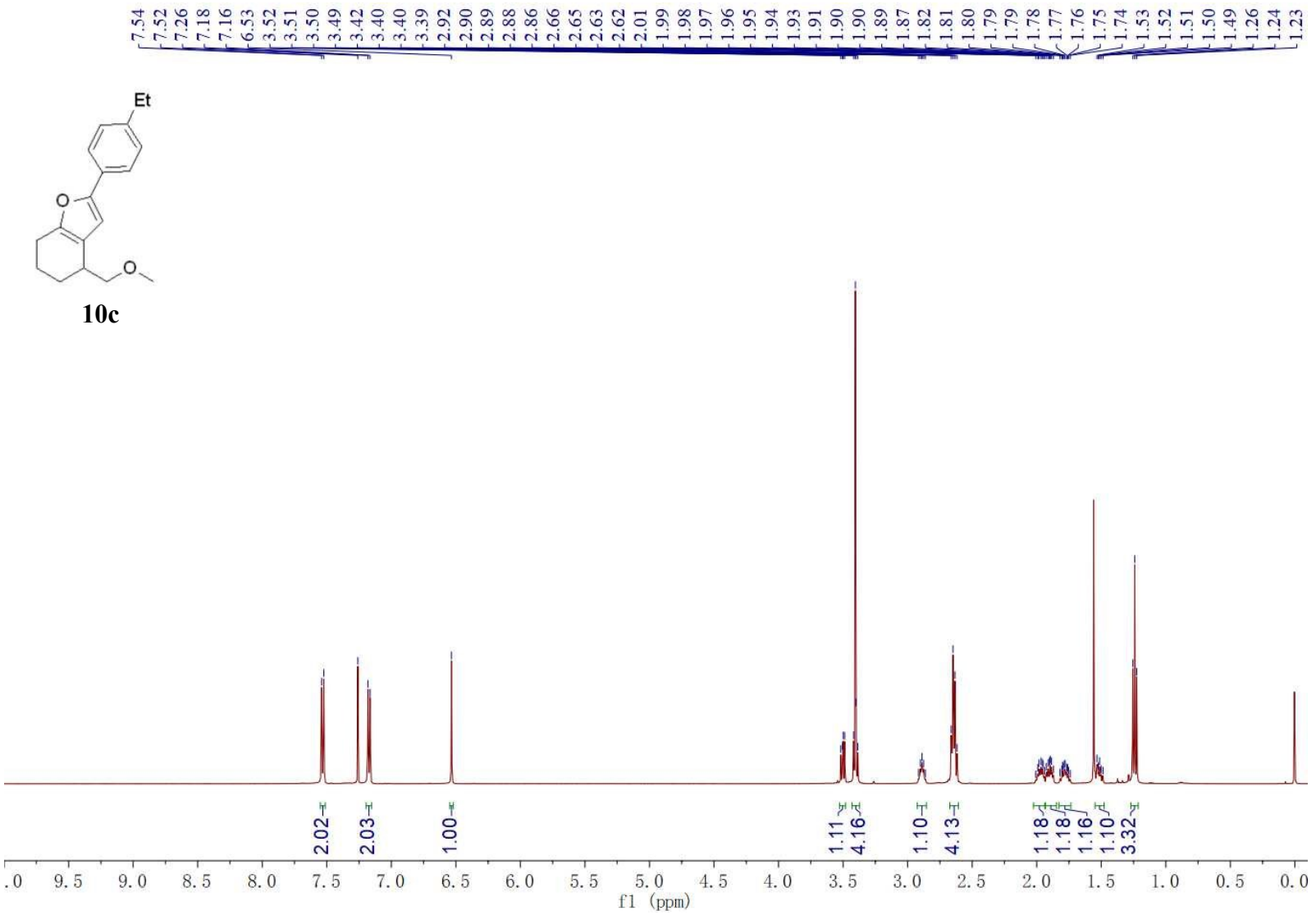

7.63
7.62
7.61
7.61
7.35
7.34
7.32
7.26
7.21
7.21
7.20
7.18
7.18
6.60
3.51
3.50
3.50
3.48
3.43
3.41
3.41
3.39
2.91
2.89
2.88
2.67
2.67
2.66
2.65
2.64
2.64
2.00
1.99
1.97
1.96
1.95
1.93
1.91
1.90
1.89
1.87
1.81
1.80
1.78
1.77
1.76
1.55
1.54
1.53
1.52
1.51
1.49
O
O
1.92
2.08
1.01
1.00
1.12
4.13
1.08
1.98
1.14
1.12
1.14
1.19
f1 (ppm)

10a

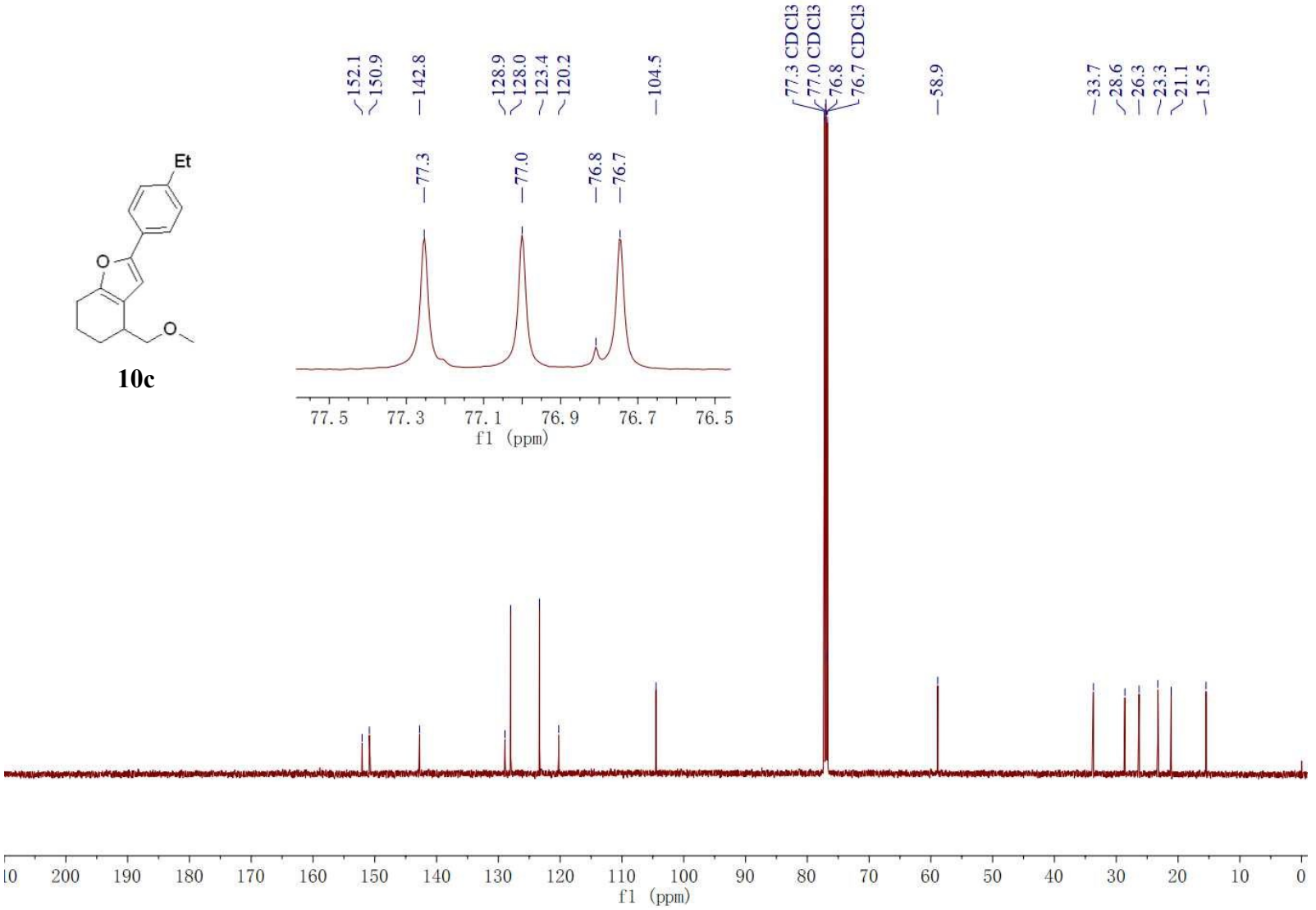

10a
151.8
151.3
131.3
128.5
126.6
123.3
120.4
105.2
77.3 CDCl3
77.0 CDCl3
76.8
76.7 CDCl3
58.9
33.7
26.3
23.3
21.1
f1 (ppm)

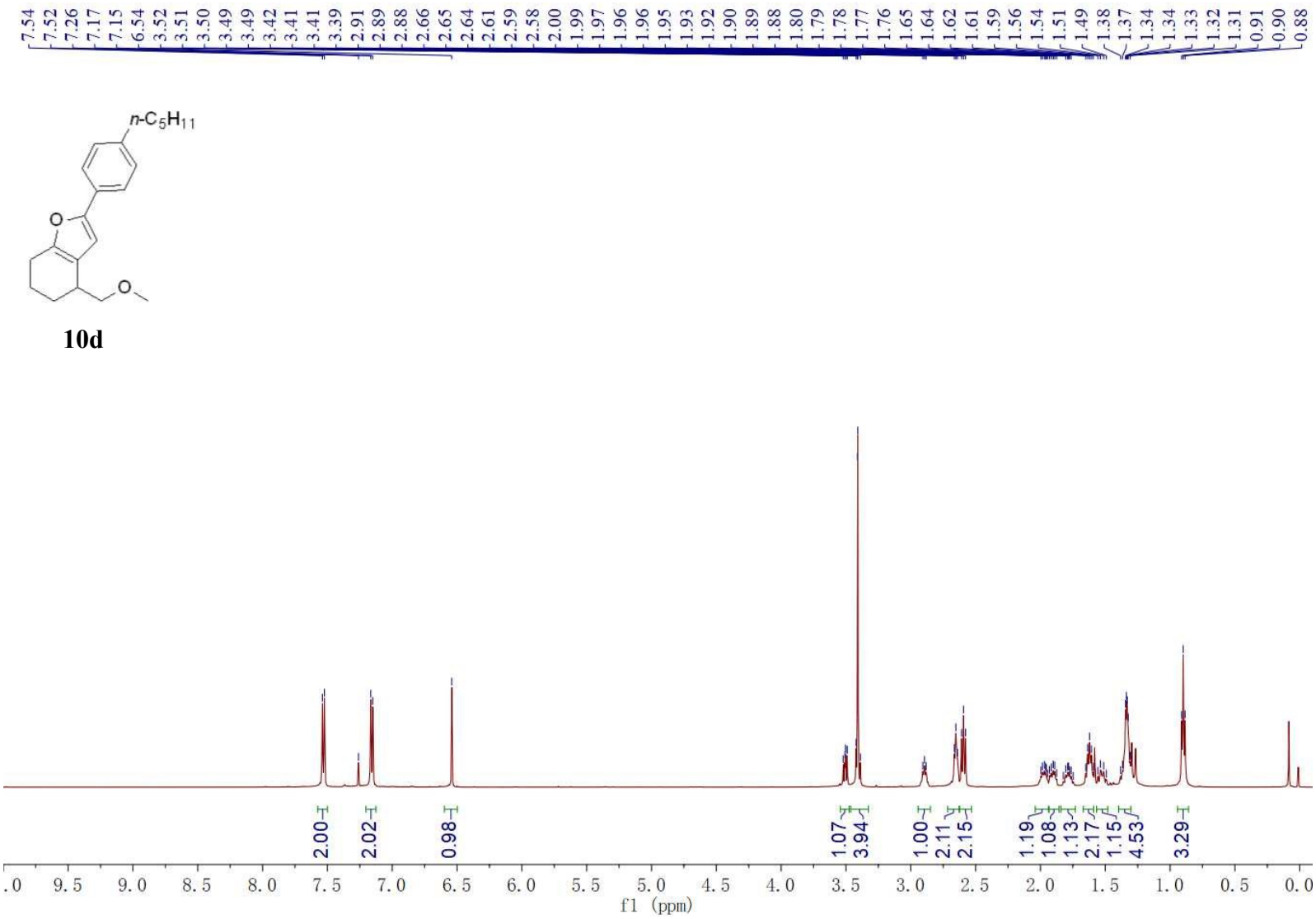

Me
O
O
10b
7.52
7.51
7.26
7.16
7.15
6.54
3.52
3.51
3.50
3.49
3.42
3.41
3.39
2.90
2.89
2.88
2.66
2.65
2.64
2.64
2.63
2.34
1.99
1.98
1.97
1.96
1.95
1.94
1.93
1.91
1.90
1.90
1.89
1.88
1.87
1.80
1.79
1.78
1.77
1.76
1.55
1.53
1.53
1.51
1.51
2.00
2.02
1.00
1.16
4.13
1.06
2.13
3.16
1.18
1.13
1.13
1.09
f1 (ppm)


Me
O
O
10b
152.0
150.8
136.3
129.2
128.6
123.2
120.2
104.4
77.3
77.0
76.8
76.7
77.3 CDCl3
77.0 CDCl3
76.8
76.7 CDCl3
58.9
33.7
26.3
23.2
21.2
21.1
f1 (ppm)
f1 (ppm)

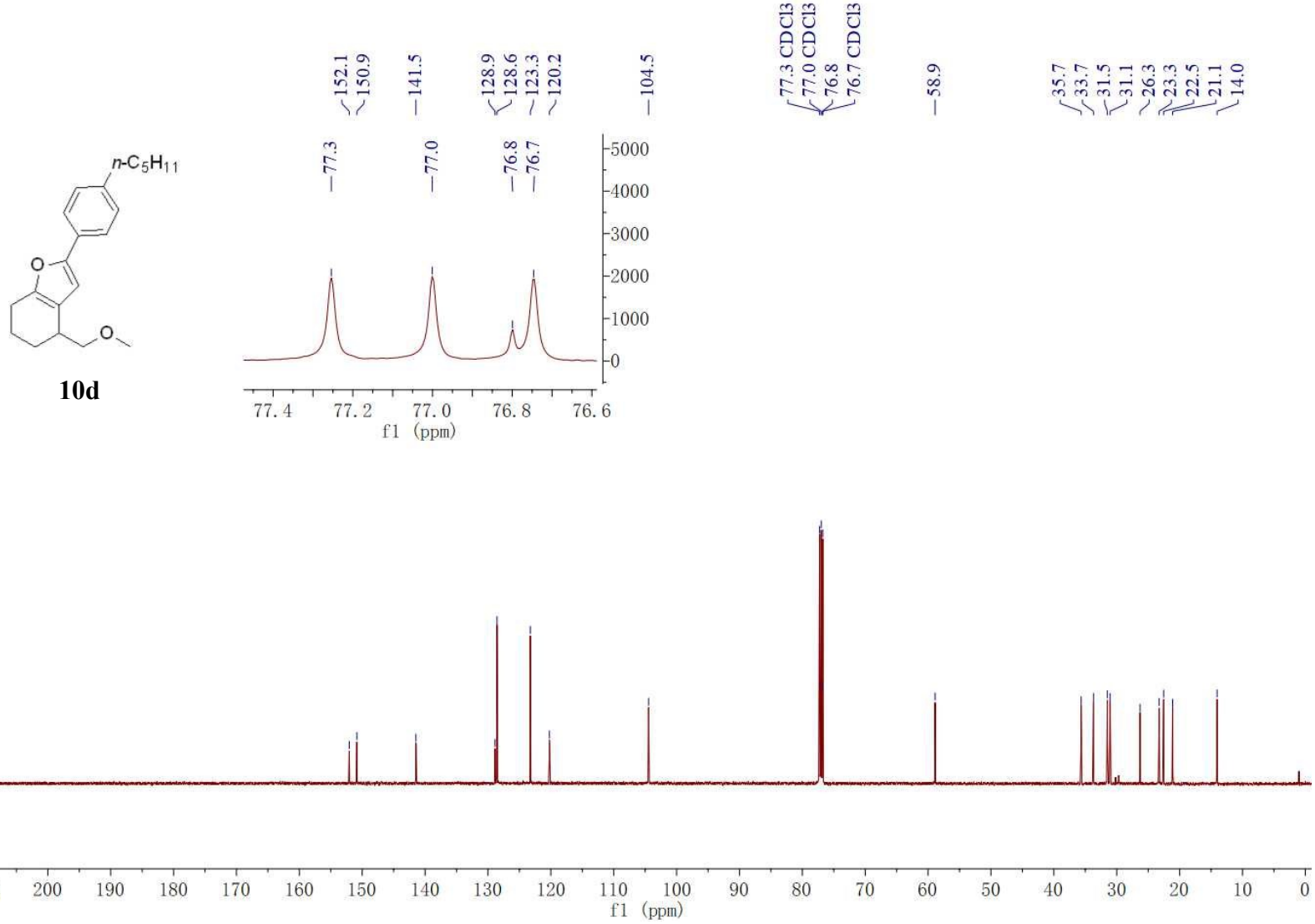

7.54
7.52
7.26
7.18
7.16
6.53
3.52
3.51
3.50
3.49
3.42
3.40
3.40
3.39
2.92
2.90
2.89
2.88
2.86
2.66
2.65
2.63
2.62
2.01
1.99
1.98
1.97
1.96
1.95
1.94
1.93
1.91
1.90
1.90
1.89
1.87
1.82
1.81
1.80
1.79
1.79
1.78
1.77
1.76
1.75
1.74
1.53
1.52
1.51
1.50
1.49
1.26
1.24
1.23
Et
O
O
2.02
2.03
1.00
1.11
4.16
1.10
4.13
1.18
1.18
1.16
1.10
3.32
f1 (ppm)

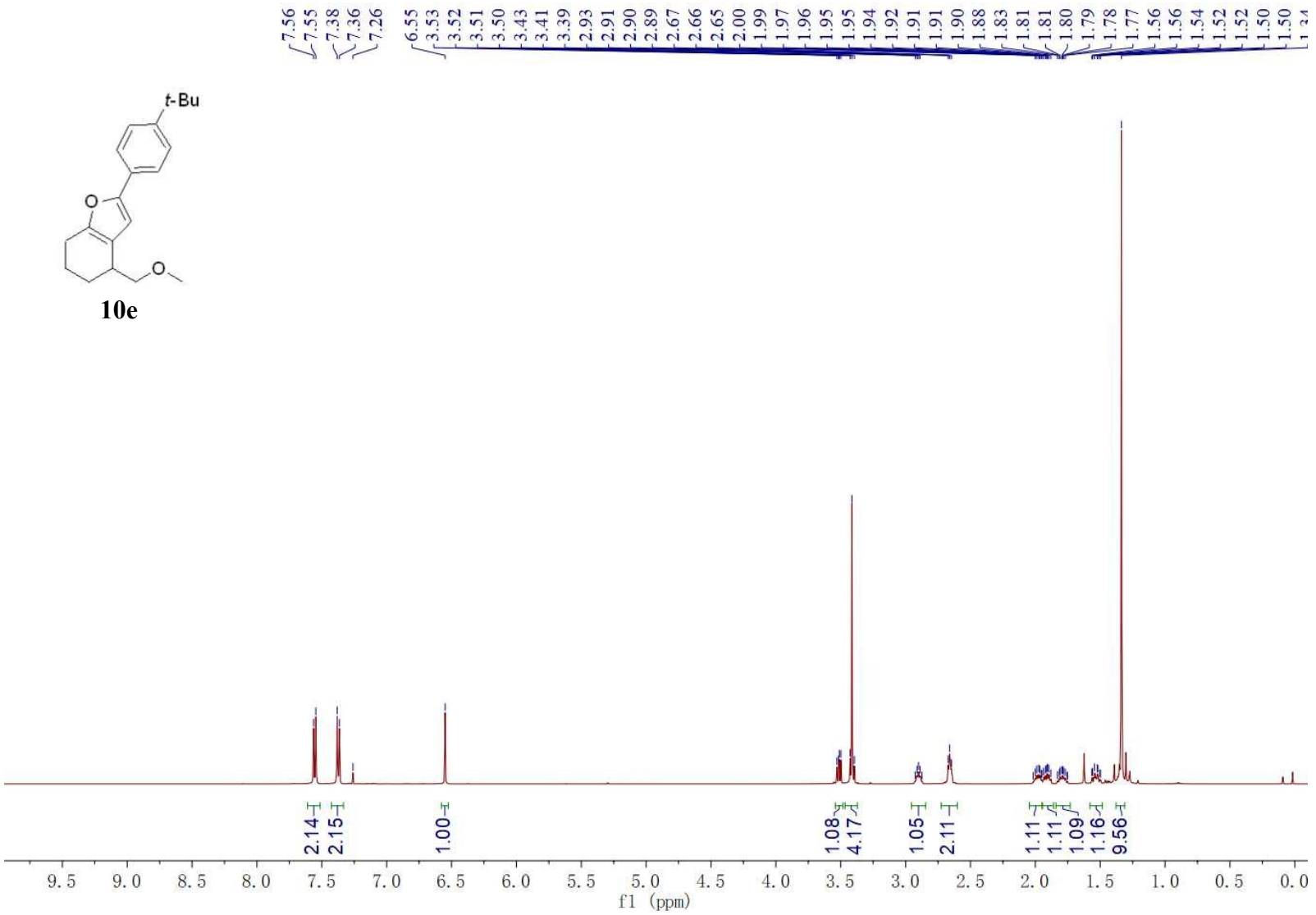
10c


Et
O
O
10c
152.1
150.9
142.8
128.9
128.0
123.4
120.2
104.5
77.3 CDCl3
77.0 CDCl3
76.8
76.7 CDCl3
58.9
33.7
28.6
26.3
23.3
21.1
15.5
77.3
77.0
76.8
76.7
77.5 77.3 77.1 76.9 76.7 76.5
f1 (ppm)
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

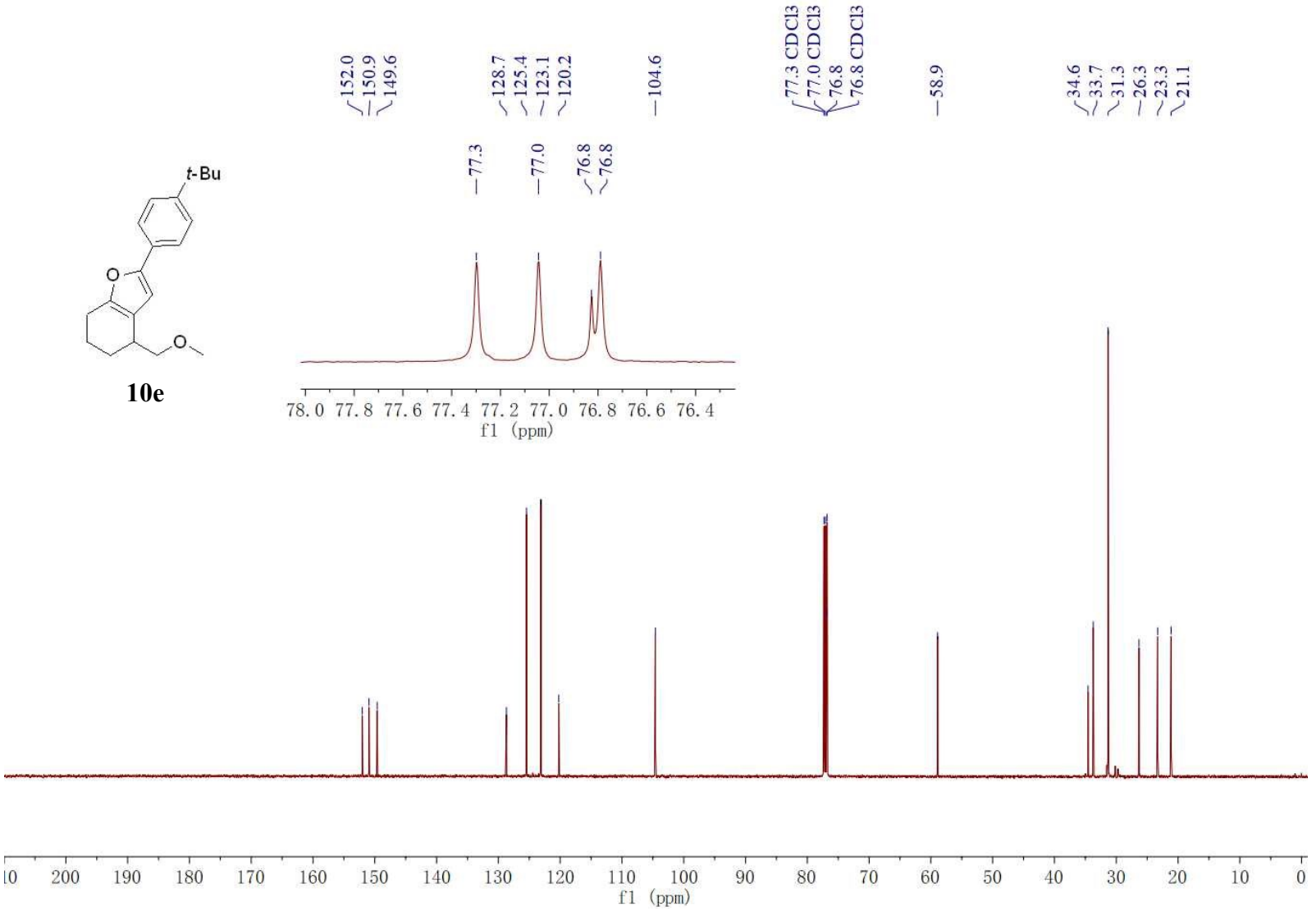

7.54
7.52
7.26
7.17
7.15
6.54
3.52
3.51
3.50
3.49
3.49
3.42
3.41
3.41
3.39
2.91
2.89
2.88
2.66
2.65
2.64
2.61
2.59
2.58
2.00
1.99
1.97
1.96
1.96
1.95
1.93
1.92
1.90
1.89
1.88
1.80
1.79
1.78
1.77
1.76
1.65
1.64
1.62
1.61
1.59
1.56
1.54
1.51
1.49
1.38
1.37
1.34
1.34
1.33
1.32
1.31
0.91
0.90
0.88
n-C5H11
O
O
2.00
2.02
0.98
1.07
3.94
1.00
2.11
2.15
1.19
1.08
1.13
2.17
1.15
4.53
3.29
f1 (ppm)

10d

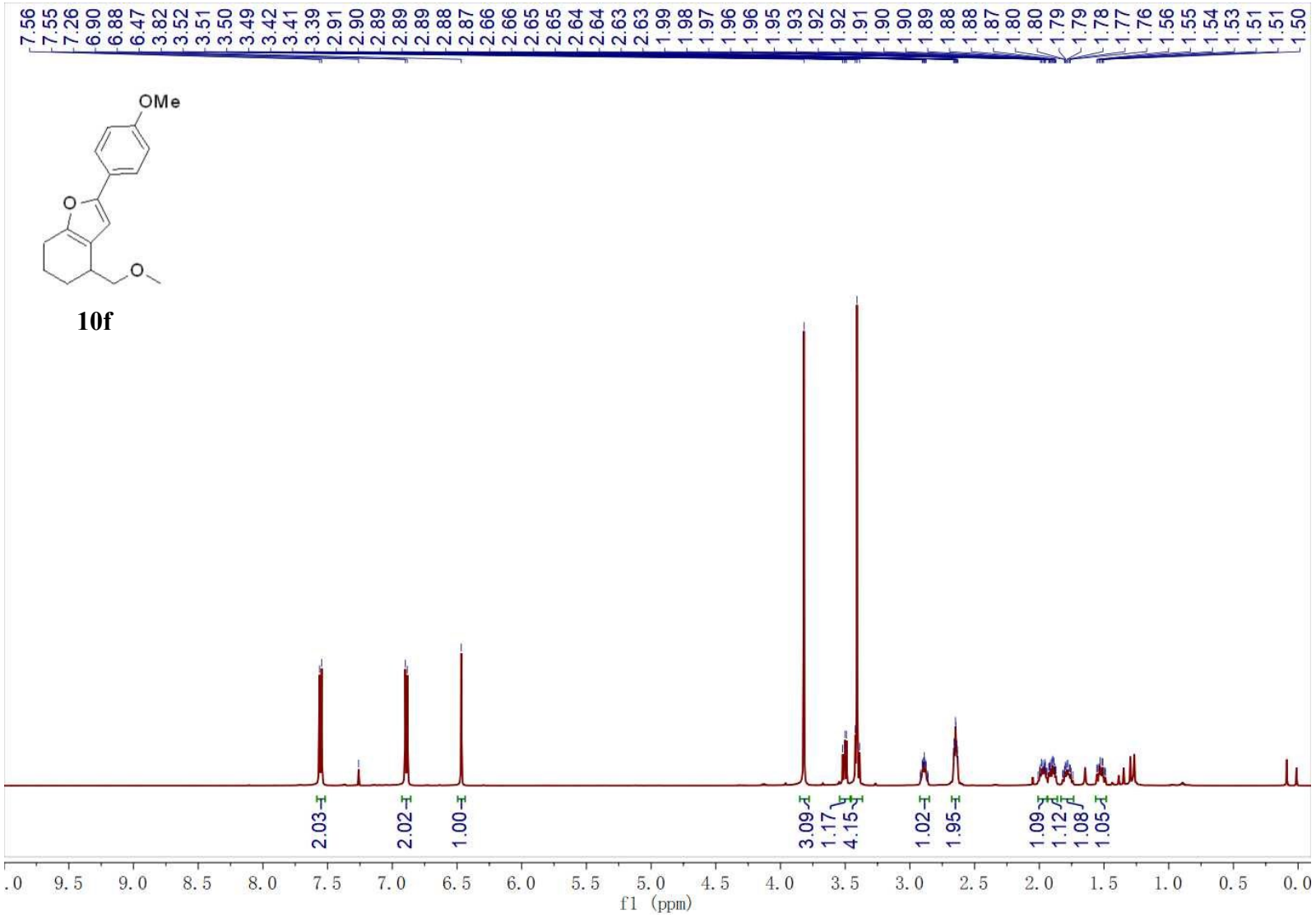

n-C5H11
O
O
10d
152.1
150.9
141.5
128.9
128.6
123.3
120.2
104.5
77.3 CDCl3
77.0 CDCl3
76.8
76.7 CDCl3
58.9
35.7
33.7
31.5
31.1
26.3
23.3
22.5
21.1
14.0
77.3
77.0
76.8
76.7
5000
4000
3000
2000
1000
0
77.4
77.2
77.0
76.8
76.6
f1 (ppm)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.56
7.55
7.38
7.36
7.26
6.55
3.53
3.52
3.51
3.50
3.43
3.41
3.39
2.93
2.91
2.90
2.89
2.67
2.66
2.65
2.00
1.99
1.97
1.96
1.95
1.95
1.94
1.92
1.91
1.91
1.90
1.88
1.83
1.81
1.81
1.80
1.79
1.78
1.77
1.56
1.56
1.54
1.52
1.52
1.50
1.50
t-Bu
O
O
2.14
2.15
1.00
1.08
4.17
1.05
2.11
1.11
1.11
1.09
1.16
9.56
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

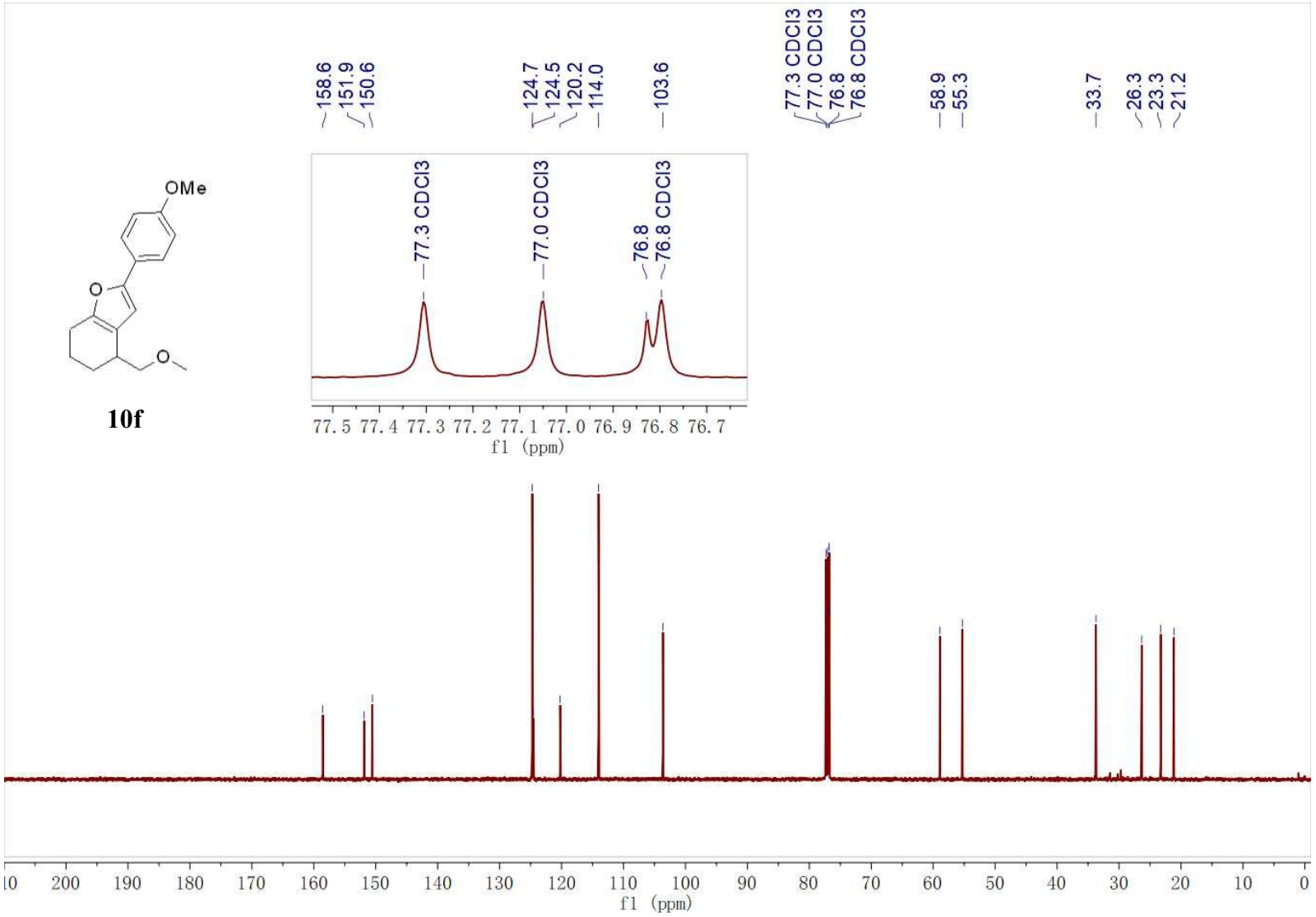
10e


152.0
150.9
149.6
128.7
125.4
123.1
120.2
104.6
77.3 CDCl3
77.0 CDCl3
76.8
76.8 CDCl3
58.9
34.6
33.7
31.3
26.3
23.3
21.1
77.3
77.0
76.8
76.8
t-Bu
O
O
78.0 77.8 77.6 77.4 77.2 77.0 76.8 76.6 76.4
f1 (ppm)
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

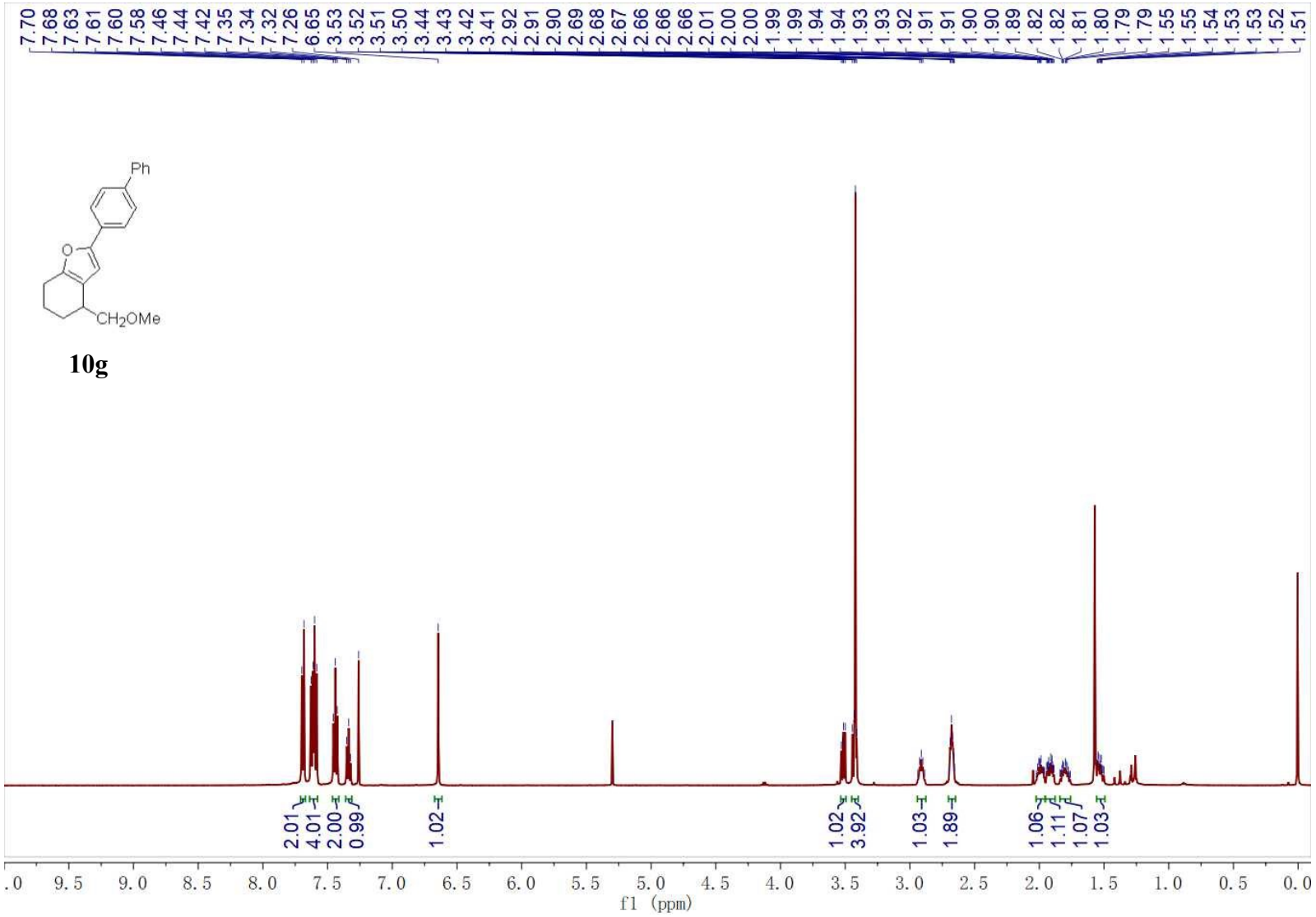
10e

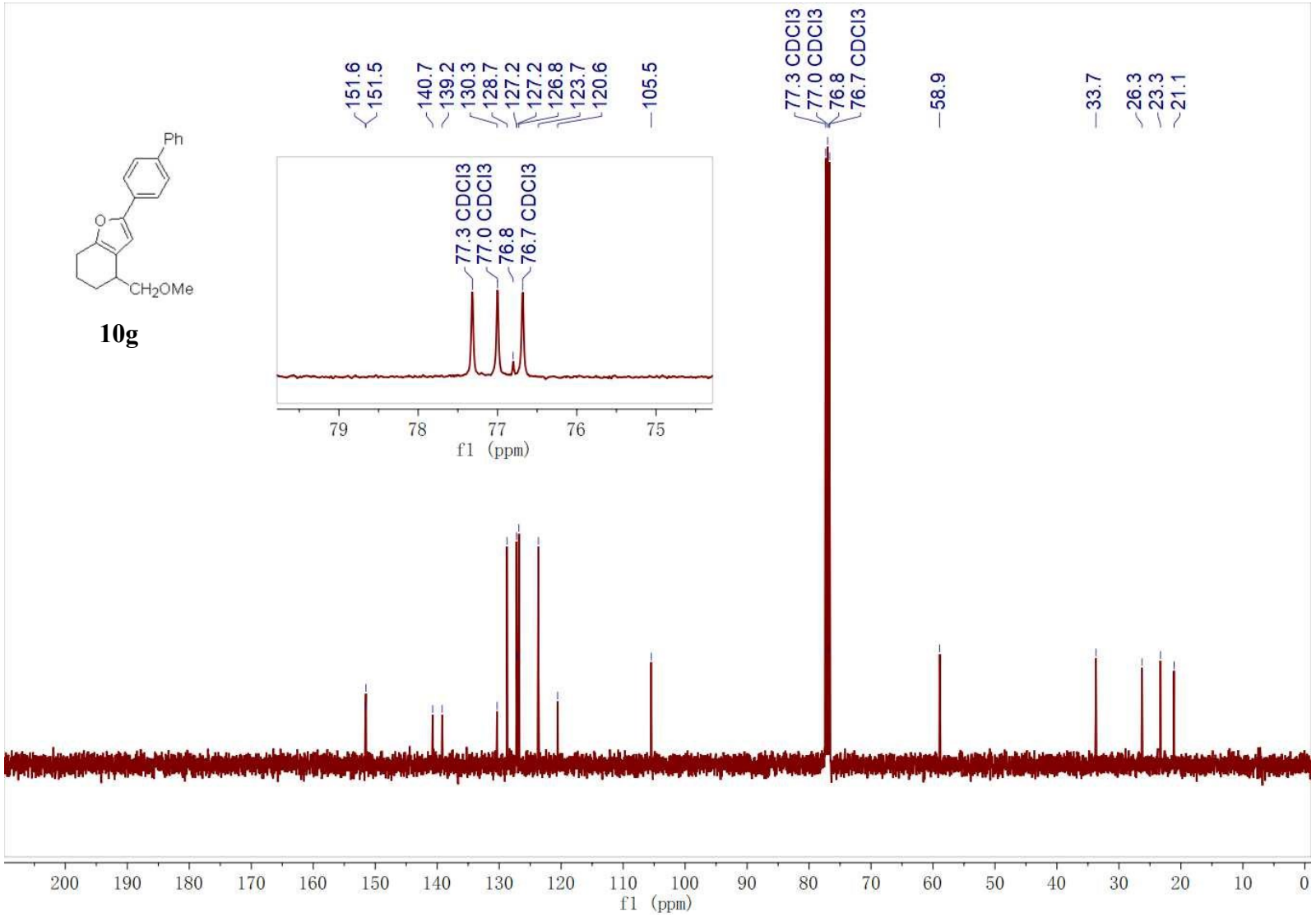

7.56 7.55 7.26 6.90 6.88 6.47 3.82 3.52 3.51 3.50 3.49 3.42 3.41 3.39 2.91 2.90 2.89 2.89 2.89 2.88 2.87 2.66 2.66 2.65 2.65 2.64 2.64 2.63 2.63 1.99 1.98 1.97 1.96 1.96 1.95 1.93 1.92 1.92 1.91 1.90 1.90 1.89 1.88 1.88 1.87 1.80 1.80 1.79 1.79 1.78 1.77 1.76 1.56 1.55 1.54 1.53 1.51 1.51 1.50
OMe
O
O
2.03
2.02
1.00
3.09
1.17
4.15
1.02
1.95
1.09
1.12
1.08
1.05
f1 (ppm)

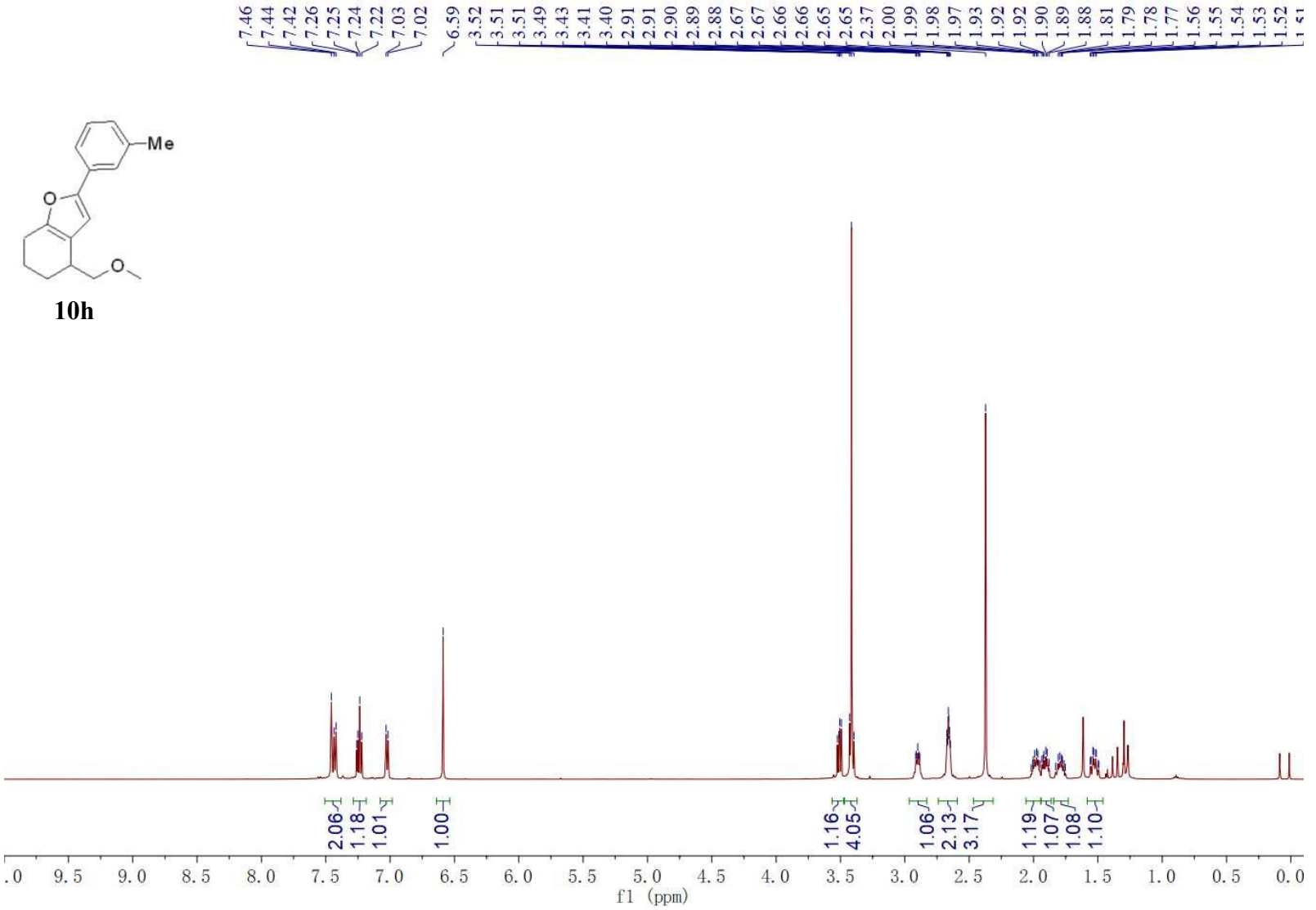
10f

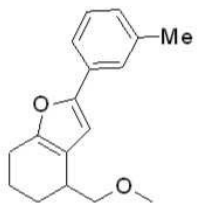

158.6
151.9
150.6
124.7
124.5
120.2
114.0
103.6
77.3 CDCl3
77.0 CDCl3
76.8
76.8 CDCl3
58.9
55.3
33.7
26.3
23.3
21.2
OMe
O
O
77.3 CDCl3
77.0 CDCl3
76.8
76.8 CDCl3
77.5 77.4 77.3 77.2 77.1 77.0 76.9 76.8 76.7
f1 (ppm)
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

10f

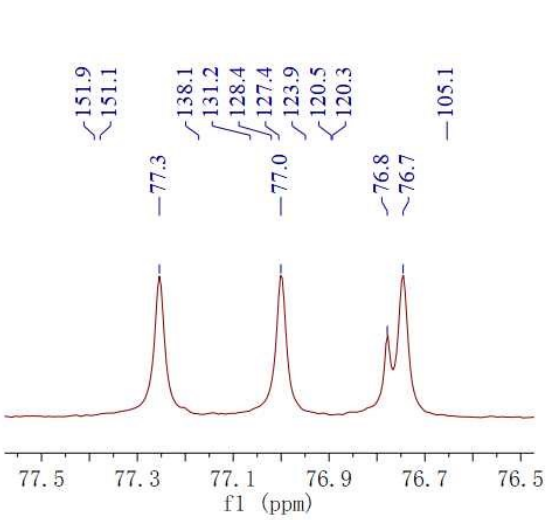

7.70
7.68
7.63
7.61
7.60
7.58
7.46
7.44
7.42
7.35
7.34
7.32
7.26
6.65
3.53
3.52
3.51
3.50
3.44
3.43
3.42
3.41
2.92
2.91
2.90
2.69
2.68
2.67
2.66
2.66
2.66
2.01
2.00
2.00
1.99
1.99
1.94
1.94
1.93
1.93
1.92
1.91
1.91
1.90
1.90
1.89
1.82
1.82
1.81
1.80
1.79
1.79
1.55
1.55
1.54
1.53
1.53
1.52
1.51
Ph
O
CH2OMe
10g
2.01
4.01
2.00
0.99
1.02
1.02
3.92
1.03
1.89
1.06
1.11
1.07
1.03
f1 (ppm)

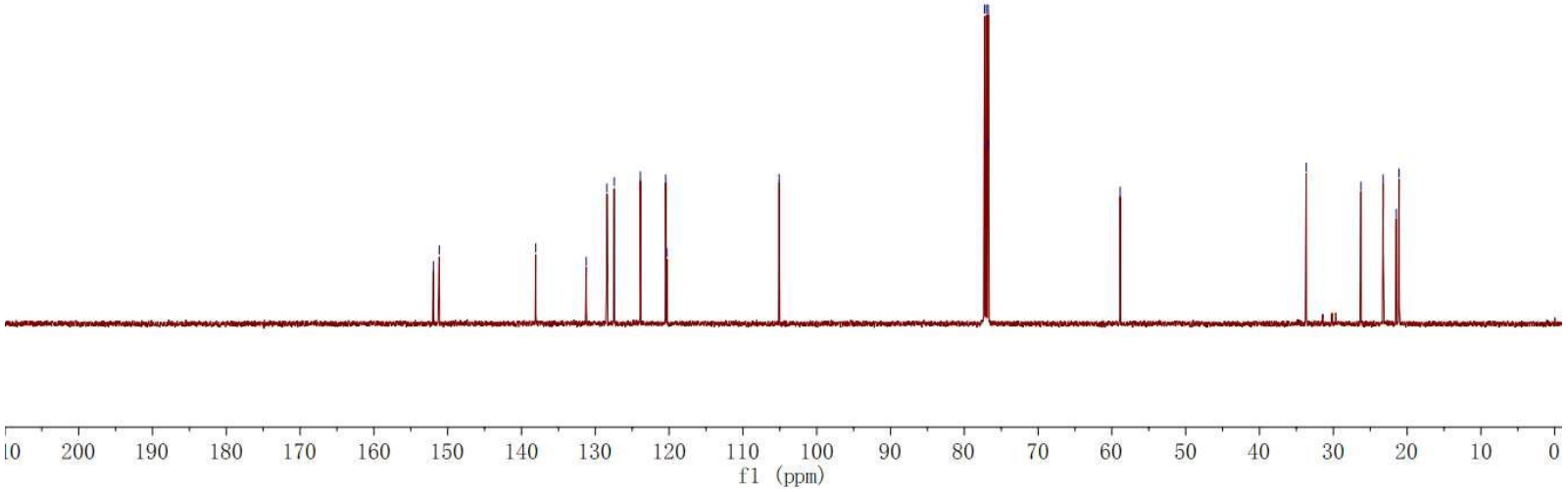

Ph
O
CH2OMe
10g
151.6
151.5
140.7
139.2
130.3
128.7
127.2
127.2
126.8
123.7
120.6
105.5
77.3 CDCl3
77.0 CDCl3
76.8
76.7 CDCl3
58.9
33.7
26.3
23.3
21.1
79
78
77
76
75
f1 (ppm)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

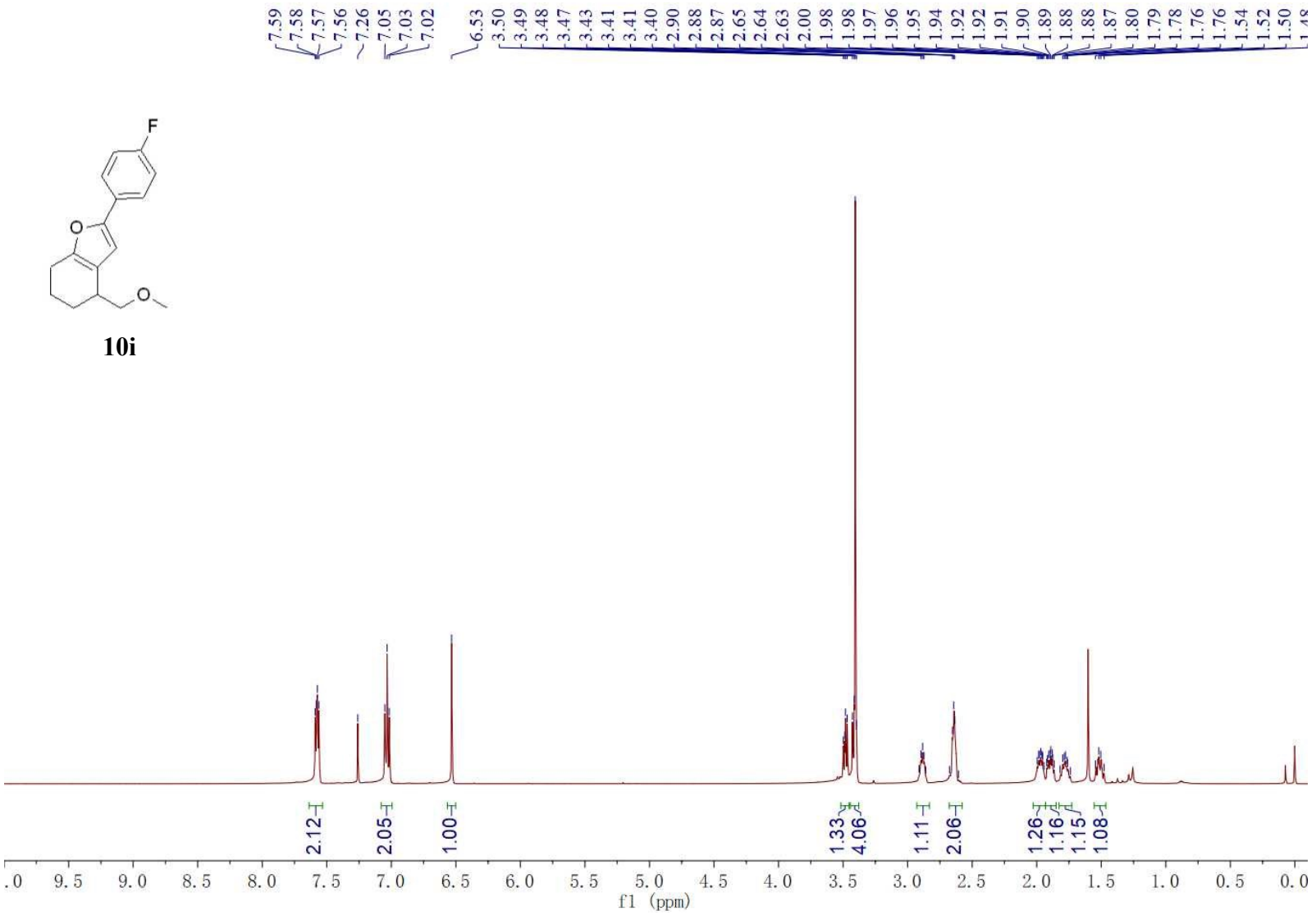

Me
O
O
10h
7.46
7.44
7.42
7.26
7.25
7.24
7.22
7.03
7.02
6.59
3.52
3.51
3.51
3.49
3.43
3.41
3.40
2.91
2.91
2.90
2.89
2.88
2.67
2.67
2.66
2.66
2.65
2.65
2.37
2.00
1.99
1.98
1.97
1.93
1.92
1.92
1.90
1.89
1.88
1.81
1.79
1.78
1.77
1.56
1.55
1.54
1.53
1.52
2.06
1.18
1.01
1.00
1.16
4.05
1.06
2.13
3.17
1.19
1.07
1.08
1.10
f1 (ppm)


Me
O
O

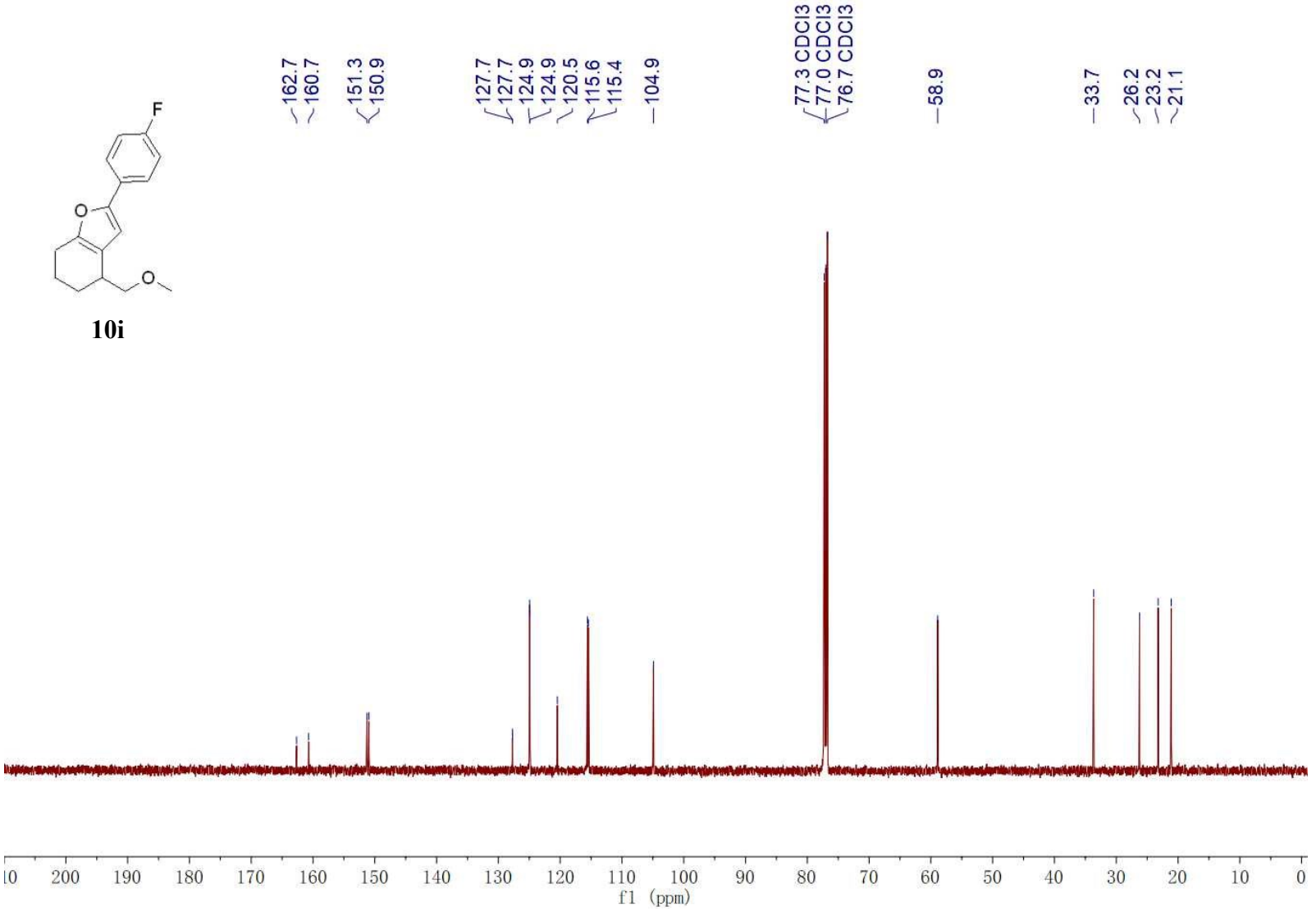
10h

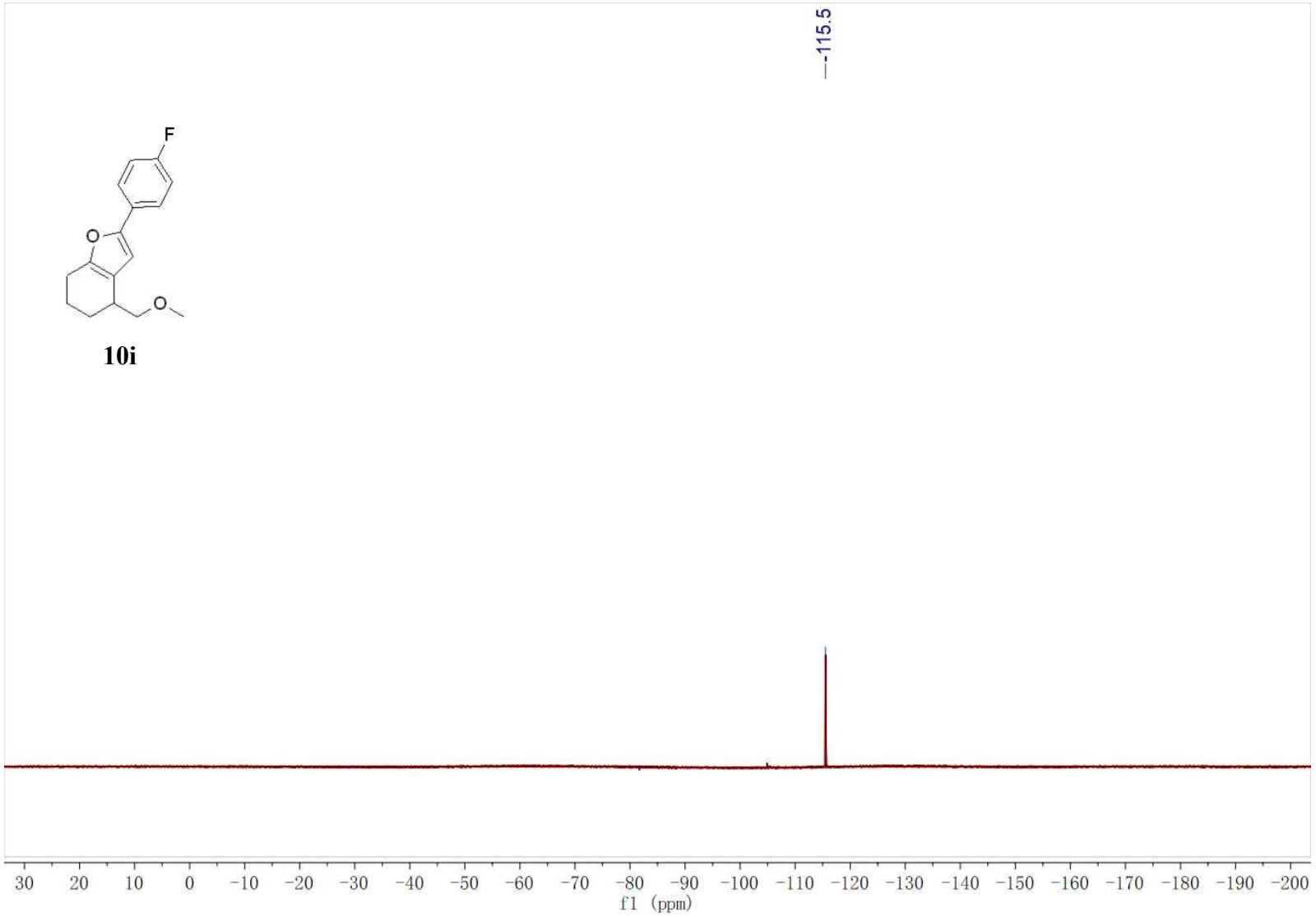

151.9
151.1
138.1
131.2
128.4
127.4
123.9
120.5
120.3
105.1
77.3 CDCl3
77.0 CDCl3
76.8
76.7 CDCl3
58.9
33.7
26.3
23.2
21.5
21.1
77.3
77.0
76.8
76.7
77.5
77.3
77.1
76.9
76.7
76.5
f1 (ppm)

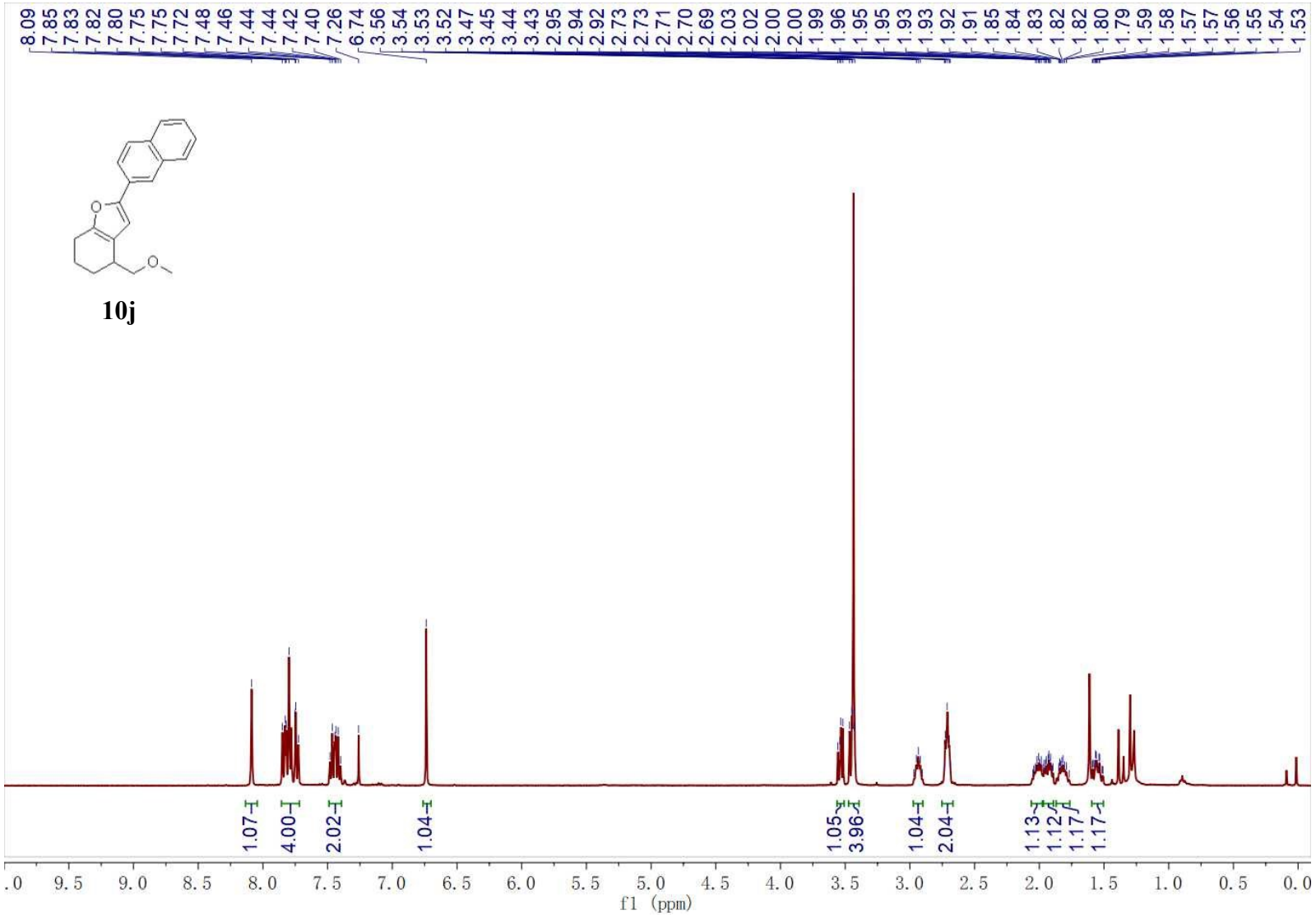

210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.59
7.58
7.57
7.56
7.26
7.05
7.03
7.02
6.53
3.50
3.49
3.48
3.47
3.43
3.41
3.41
3.40
2.90
2.88
2.87
2.65
2.64
2.63
2.00
1.98
1.98
1.97
1.96
1.95
1.94
1.92
1.92
1.91
1.90
1.89
1.88
1.88
1.87
1.80
1.79
1.78
1.76
1.76
1.54
1.52
1.50
F
O
O
2.12
2.05
1.00
1.33
4.06
1.11
2.06
1.26
1.16
1.15
1.08
f1 (ppm)

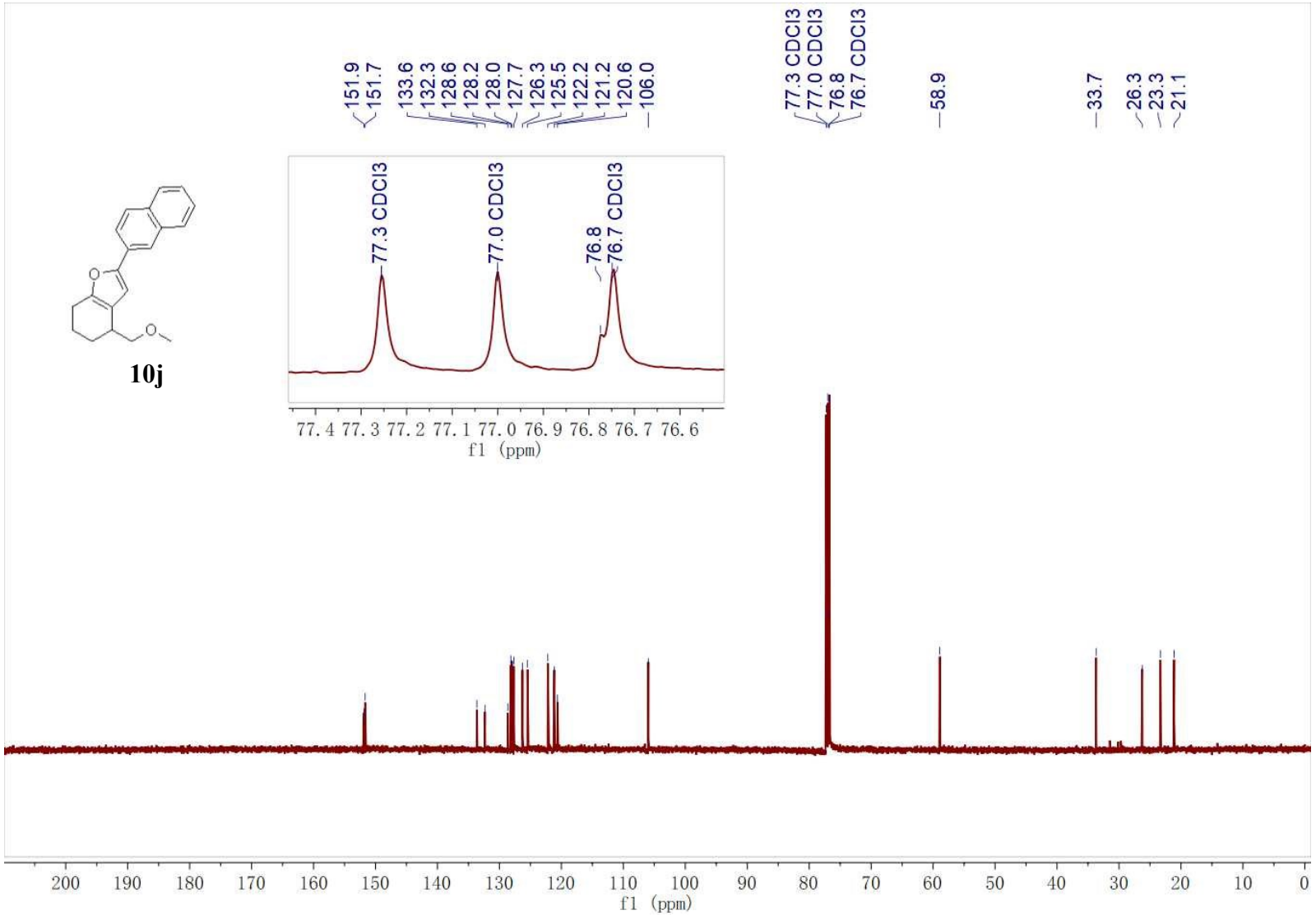
10i


F
O
O
10i
162.7
160.7
151.3
150.9
127.7
127.7
124.9
124.9
120.5
115.6
115.4
104.9
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
58.9
33.7
26.2
23.2
21.1
f1 (ppm)

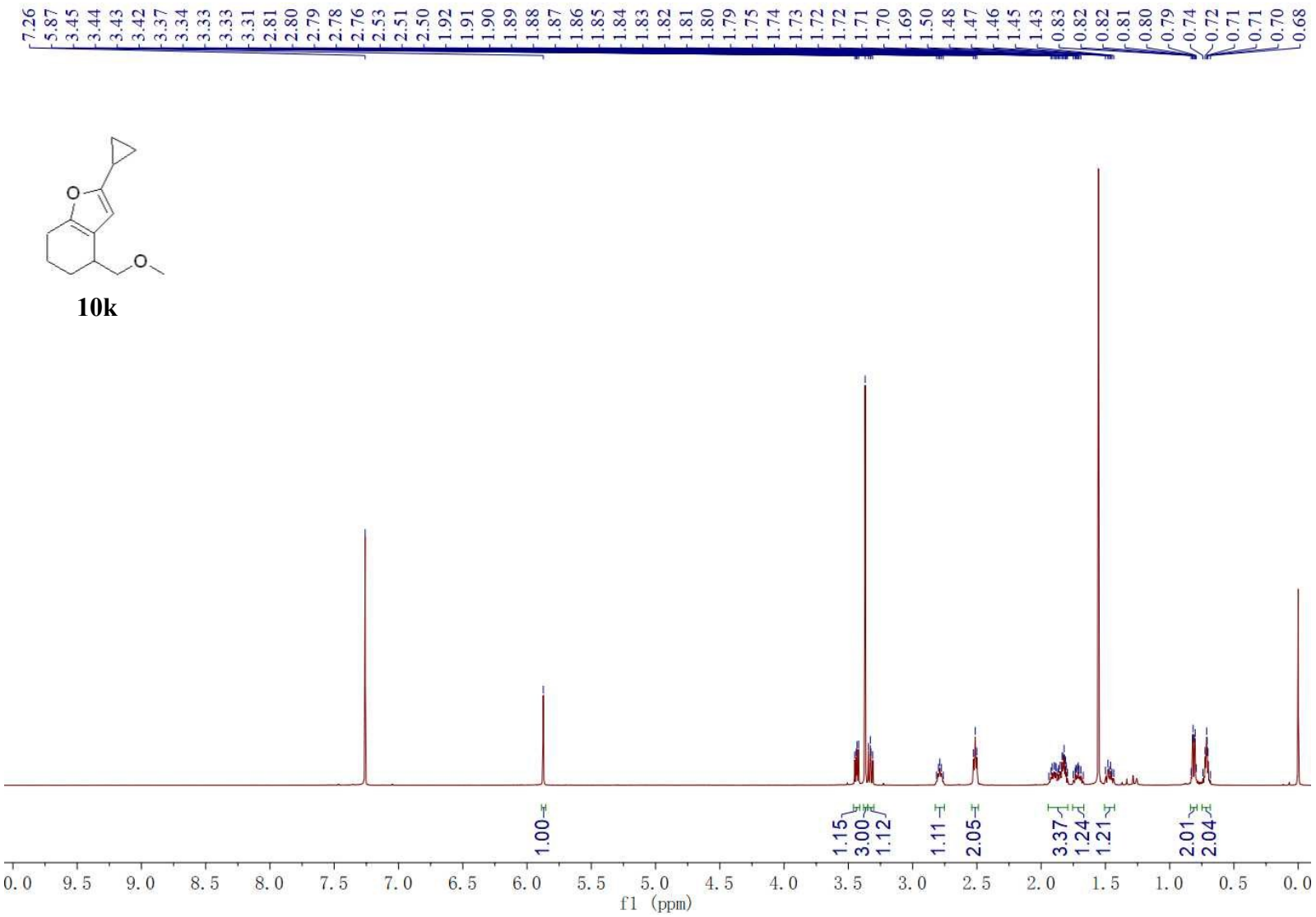

F
O
O
10i
-115.5
30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200
f1 (ppm)


8.09 7.85 7.83 7.82 7.80 7.75 7.75 7.72 7.48 7.46 7.44 7.44 7.42 7.40 7.26 6.74 3.56 3.54 3.53 3.52 3.47 3.45 3.44 3.43 2.95 2.94 2.92 2.73 2.73 2.71 2.70 2.69 2.03 2.02 2.00 2.00 1.99 1.96 1.95 1.95 1.93 1.93 1.92 1.91 1.85 1.84 1.83 1.82 1.82 1.80 1.79 1.59 1.58 1.57 1.57 1.56 1.55 1.54 1.53
1.07 4.00 2.02 1.04 1.05 3.96 1.04 2.04 1.13 1.12 1.17 1.17
f1 (ppm)

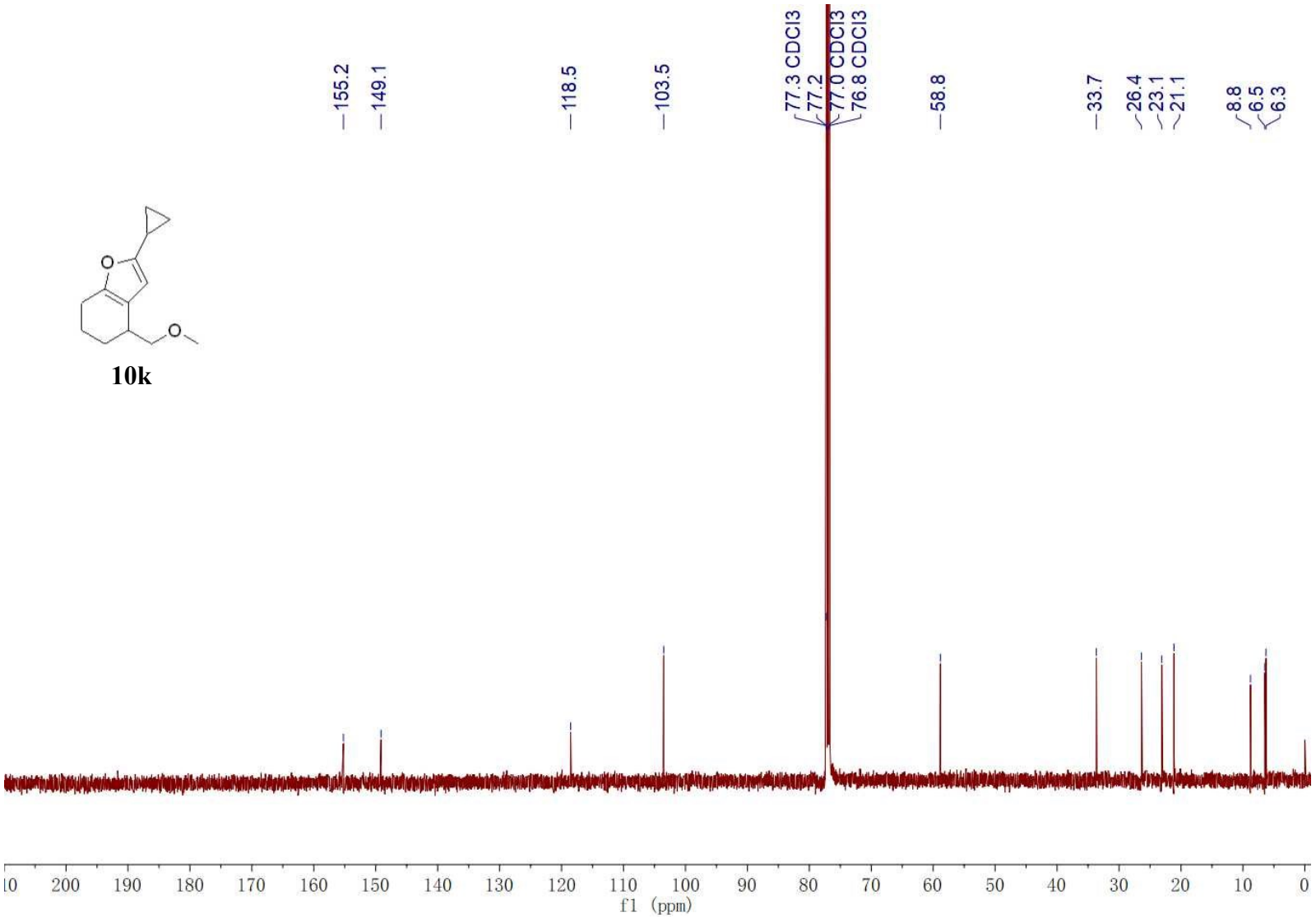
10j

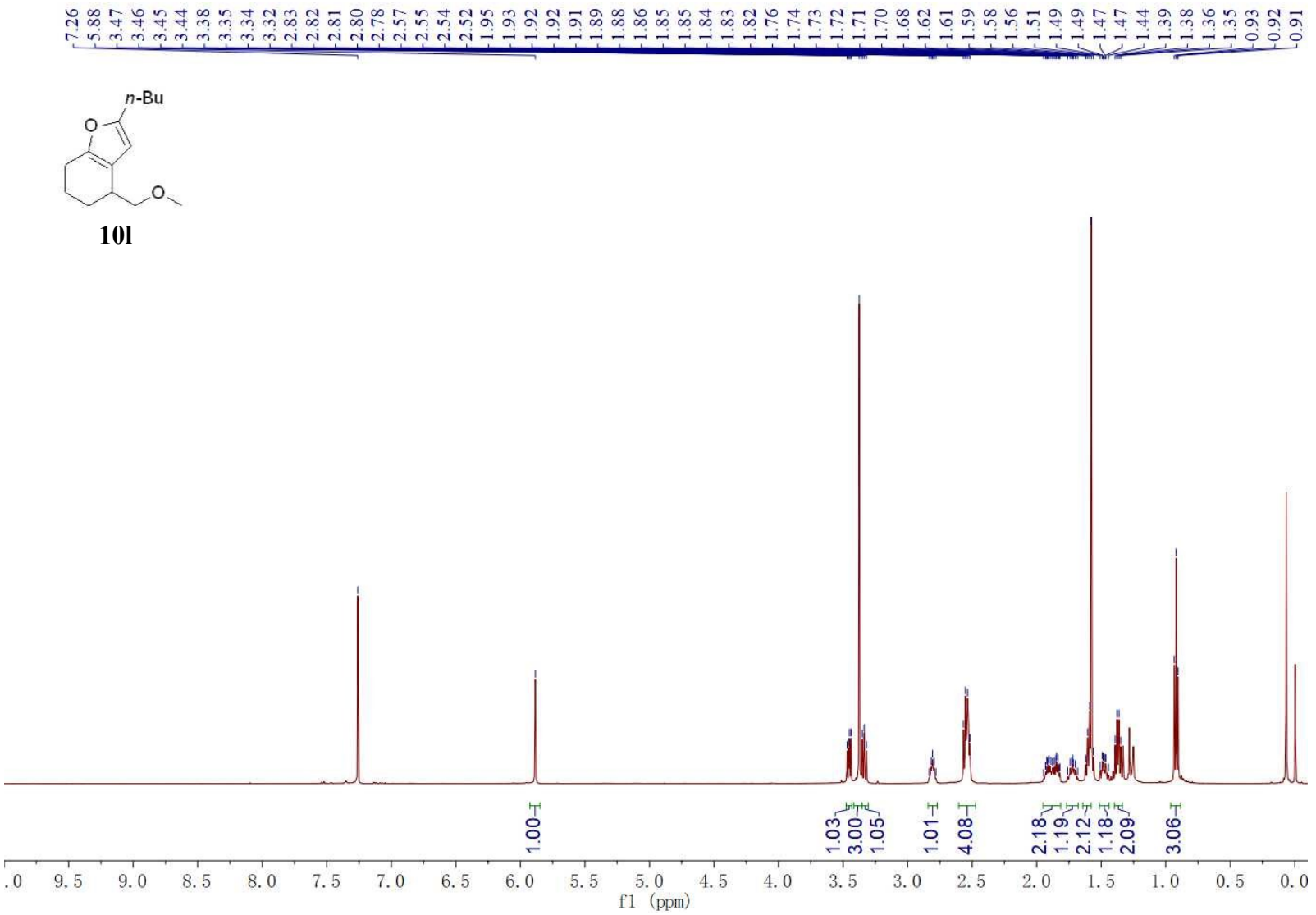

151.9
151.7
133.6
132.3
128.6
128.2
128.0
127.7
126.3
125.5
122.2
121.2
120.6
106.0
77.3 CDCl3
77.0 CDCl3
76.8
76.7 CDCl3
58.9
33.7
26.3
23.3
21.1
77.3 CDCl3
77.0 CDCl3
76.8
76.7 CDCl3
77.4 77.3 77.2 77.1 77.0 76.9 76.8 76.7 76.6
f1 (ppm)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

10j

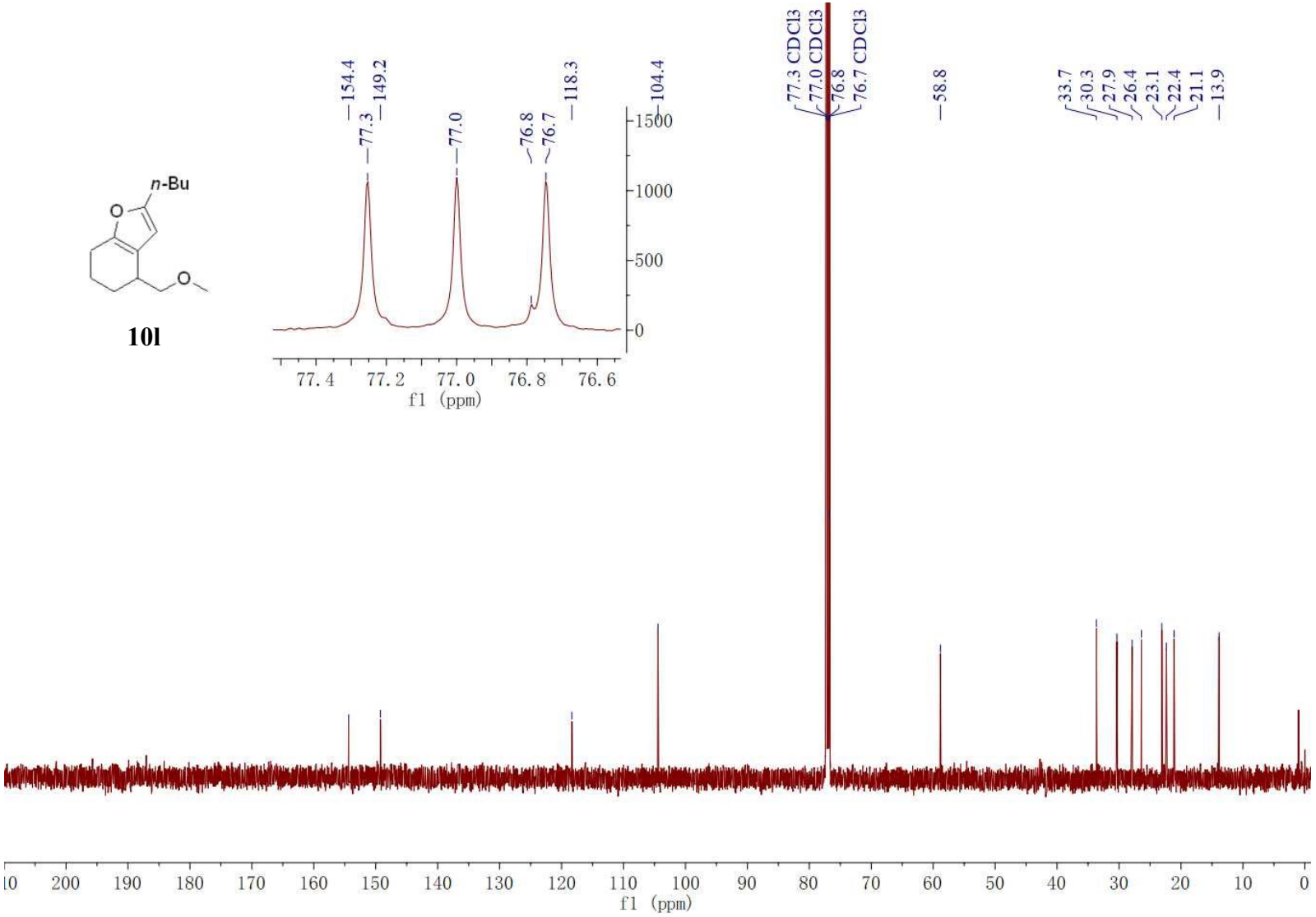

7.26
5.87
3.45
3.44
3.43
3.42
3.37
3.34
3.33
3.33
3.31
2.81
2.80
2.79
2.78
2.76
2.53
2.51
2.50
1.92
1.91
1.90
1.89
1.88
1.87
1.86
1.85
1.84
1.83
1.82
1.81
1.80
1.79
1.75
1.74
1.73
1.72
1.72
1.71
1.70
1.69
1.50
1.48
1.47
1.46
1.45
1.43
0.83
0.82
0.82
0.81
0.80
0.79
0.74
0.72
0.71
0.71
0.70
0.68
O
O
1.00
1.15
3.00
1.12
1.11
2.05
3.37
1.24
1.21
2.01
2.04
0.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

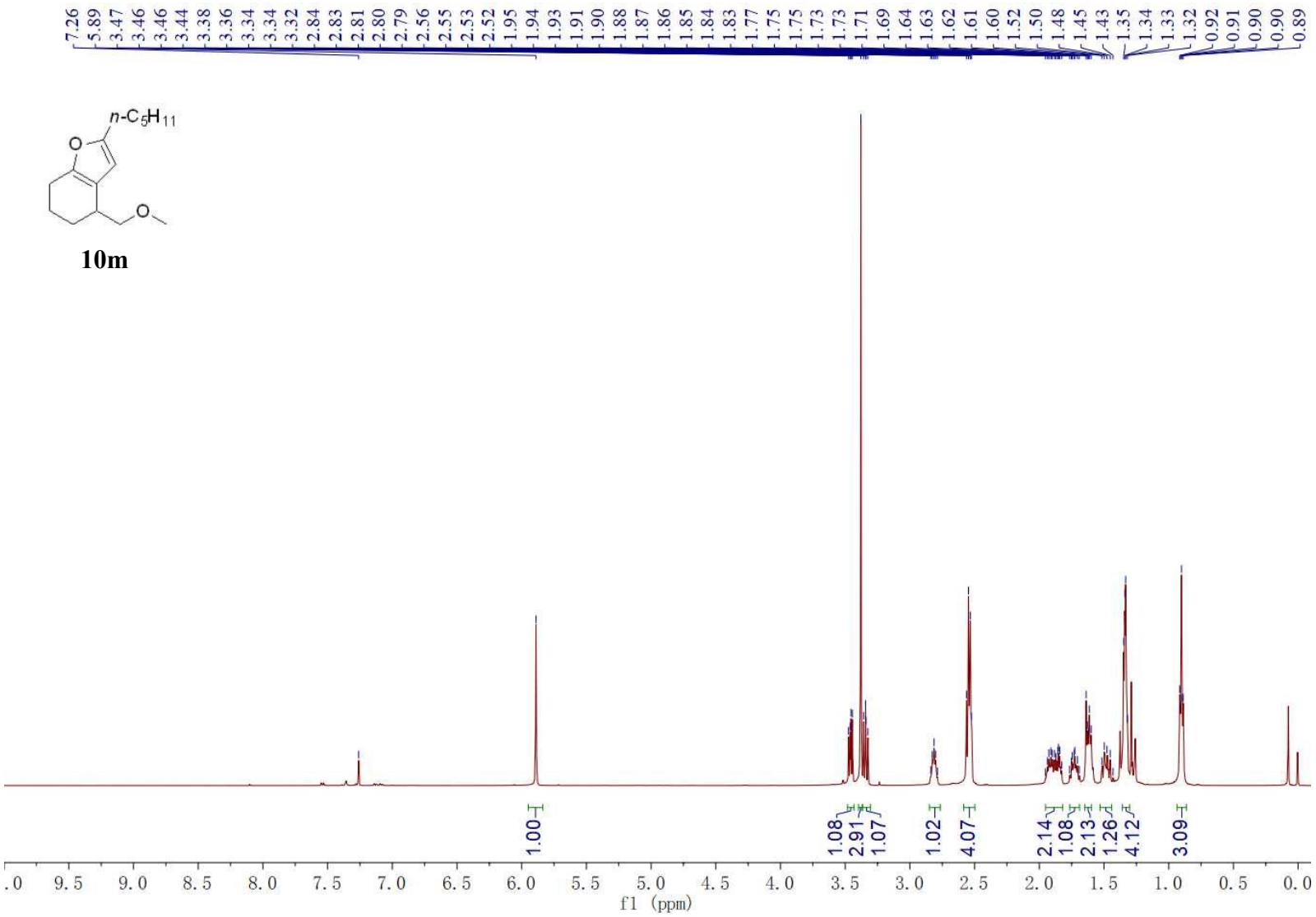
10k


155.2
149.1
118.5
103.5
77.3 CDCl3
77.2
77.0 CDCl3
76.8 CDCl3
58.8
33.7
26.4
23.1
21.1
8.8
6.5
6.3
O
O
10k
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

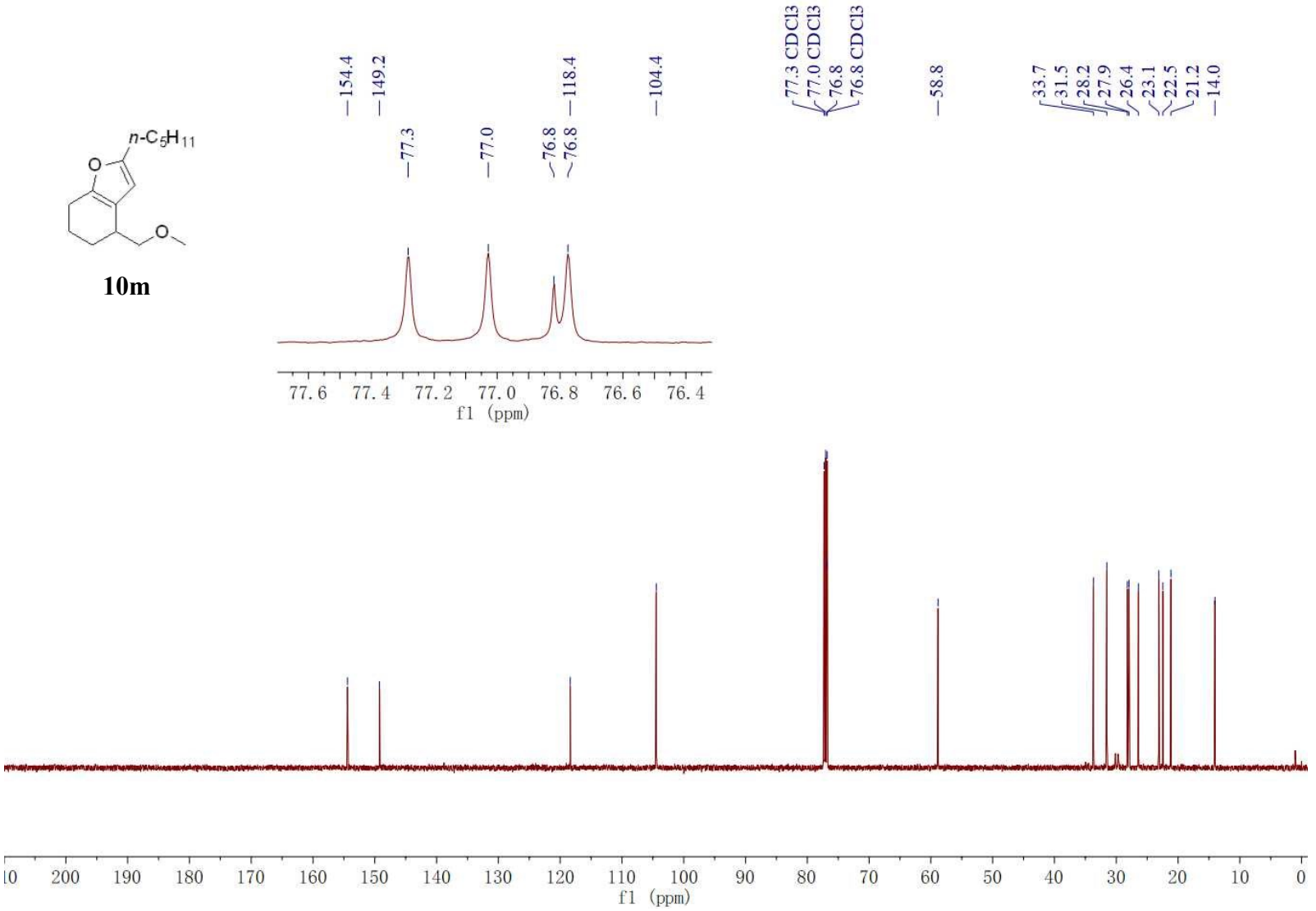

7.26
5.88
3.47
3.46
3.45
3.44
3.38
3.35
3.34
3.32
2.83
2.82
2.81
2.80
2.78
2.57
2.55
2.54
2.52
1.95
1.93
1.92
1.92
1.91
1.89
1.88
1.86
1.85
1.85
1.84
1.83
1.82
1.76
1.74
1.73
1.72
1.71
1.70
1.68
1.62
1.61
1.59
1.58
1.56
1.51
1.49
1.49
1.47
1.47
1.44
1.39
1.38
1.36
1.35
0.93
0.92
0.91
n-Bu
O
O
1.00
1.03
3.00
1.05
1.01
4.08
2.18
1.19
2.12
1.18
2.09
3.06
f1 (ppm)

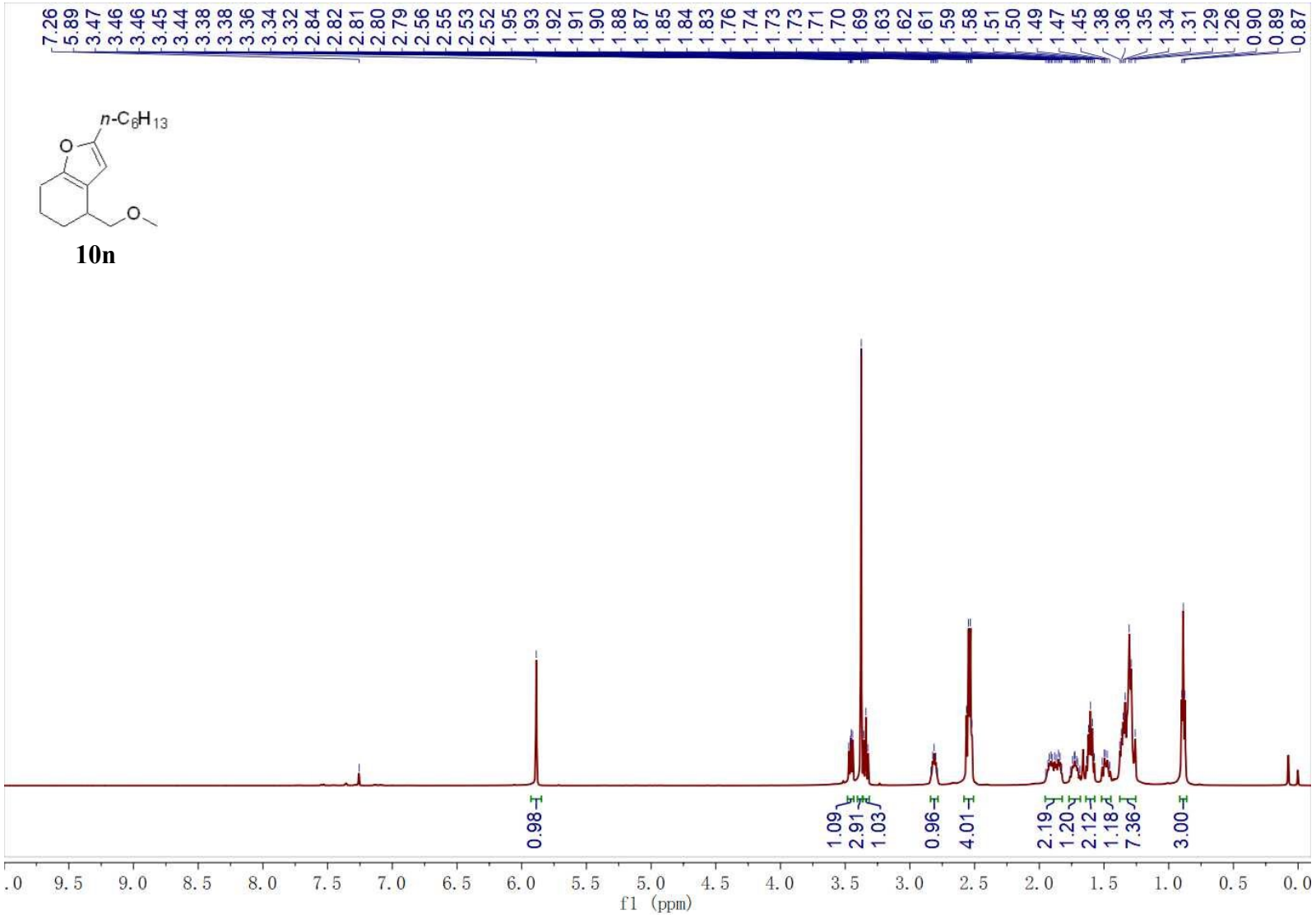
10l


n-Bu
O
O
10l
154.4
149.2
77.3
77.0
76.8
76.7
118.3
104.4
77.3 CDCl3
77.0 CDCl3
76.8
76.7 CDCl3
58.8
33.7
30.3
27.9
26.4
23.1
22.4
21.1
13.9
77.4
77.2
77.0
76.8
76.6
f1 (ppm)
f1 (ppm)

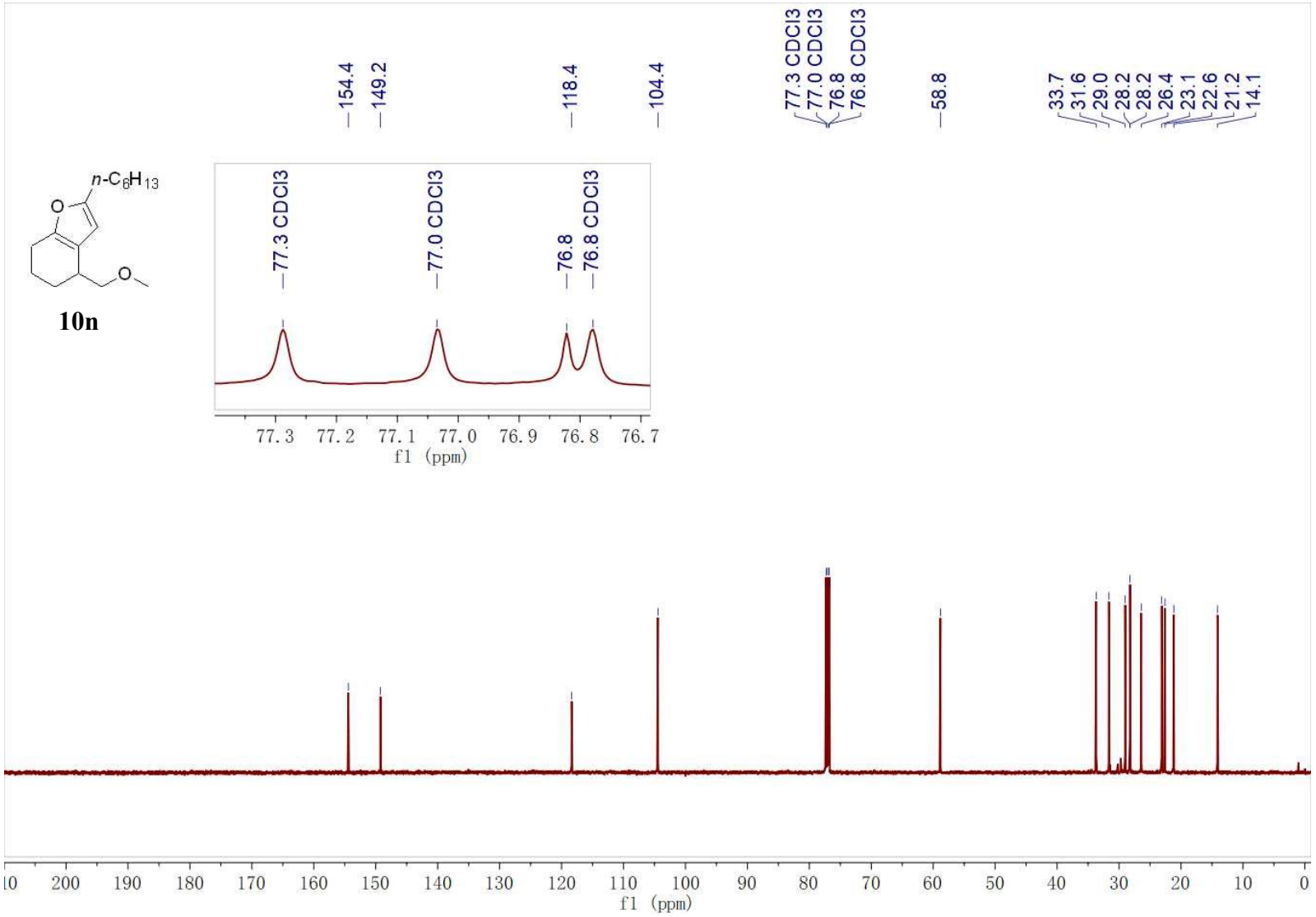

7.26
5.89
3.47
3.46
3.46
3.44
3.38
3.36
3.34
3.34
3.32
2.84
2.83
2.81
2.80
2.79
2.56
2.55
2.53
2.52
1.95
1.94
1.93
1.91
1.90
1.88
1.87
1.86
1.85
1.84
1.83
1.77
1.75
1.75
1.73
1.73
1.71
1.69
1.64
1.63
1.62
1.61
1.60
1.52
1.50
1.48
1.45
1.43
1.35
1.34
1.33
1.32
0.92
0.91
0.90
0.90
0.89
n-C5H11
O
O
1.00
1.08
2.91
1.07
1.02
4.07
2.14
1.08
2.13
1.26
4.12
3.09
f1 (ppm)

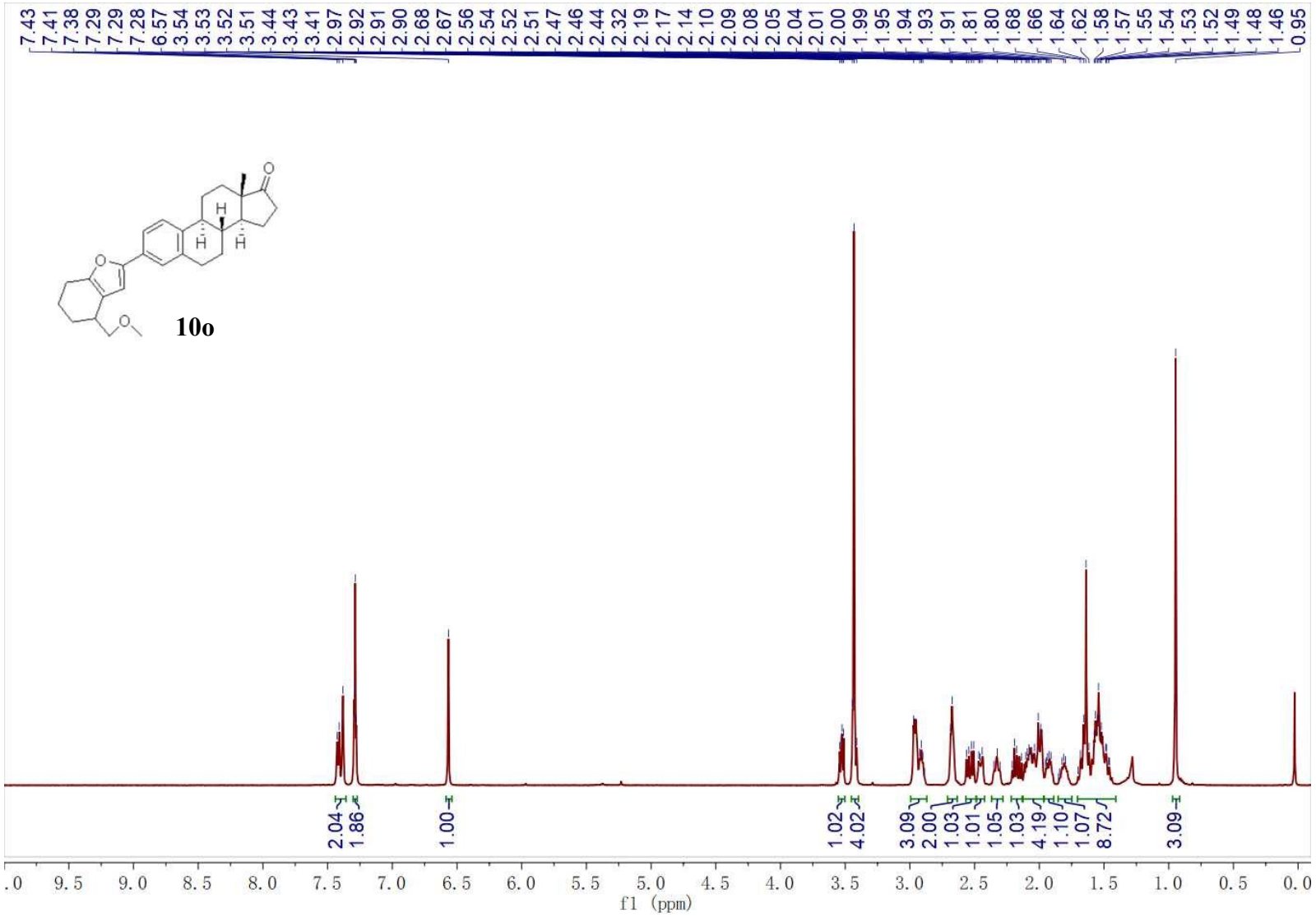
10m

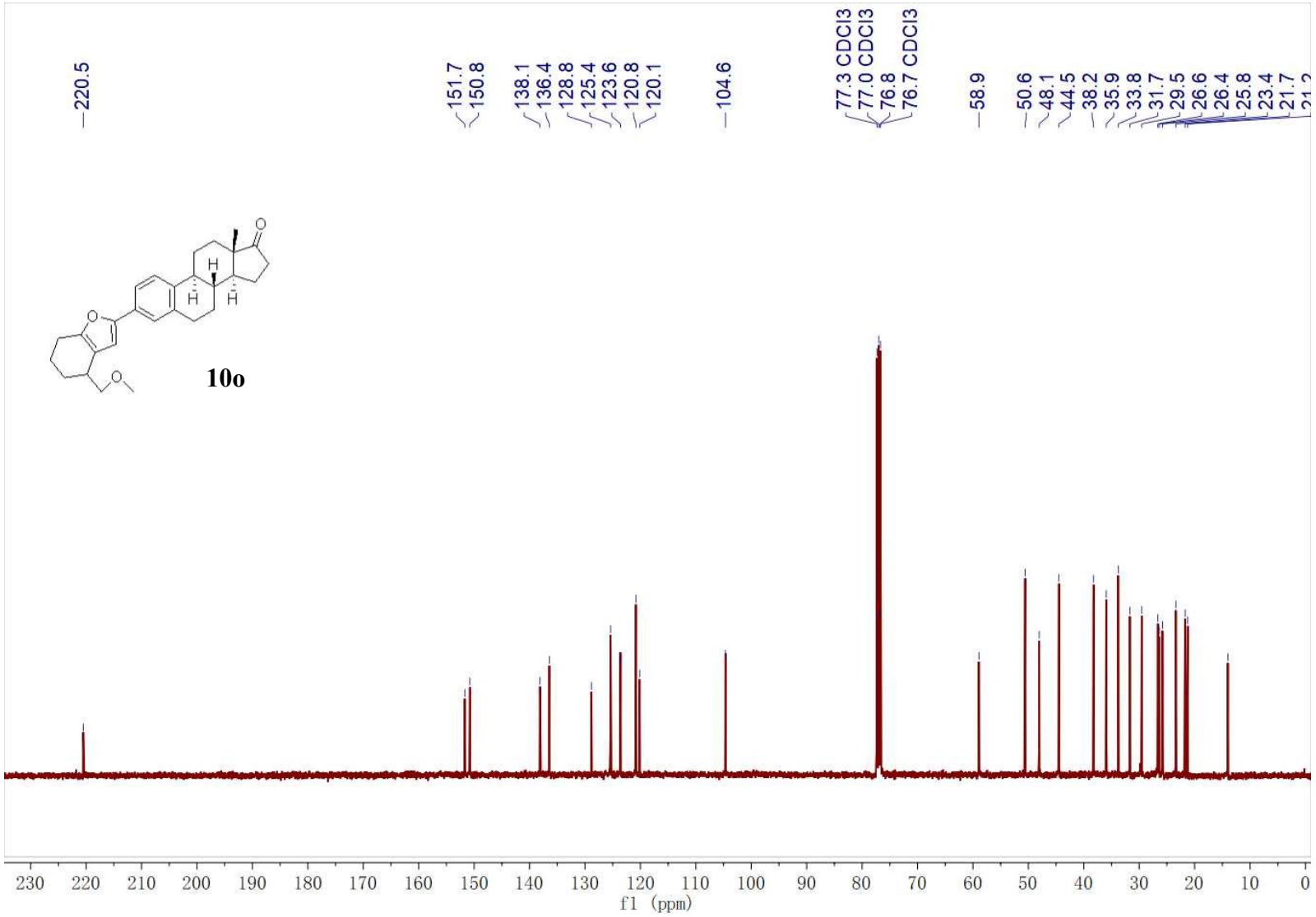

n-C5H11
O
O
10m
154.4
149.2
77.3
77.0
76.8
76.8
118.4
104.4
77.3 CDCl3
77.0 CDCl3
76.8
76.8 CDCl3
58.8
33.7
31.5
28.2
27.9
26.4
23.1
22.5
21.2
14.0
77.6 77.4 77.2 77.0 76.8 76.6 76.4
f1 (ppm)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

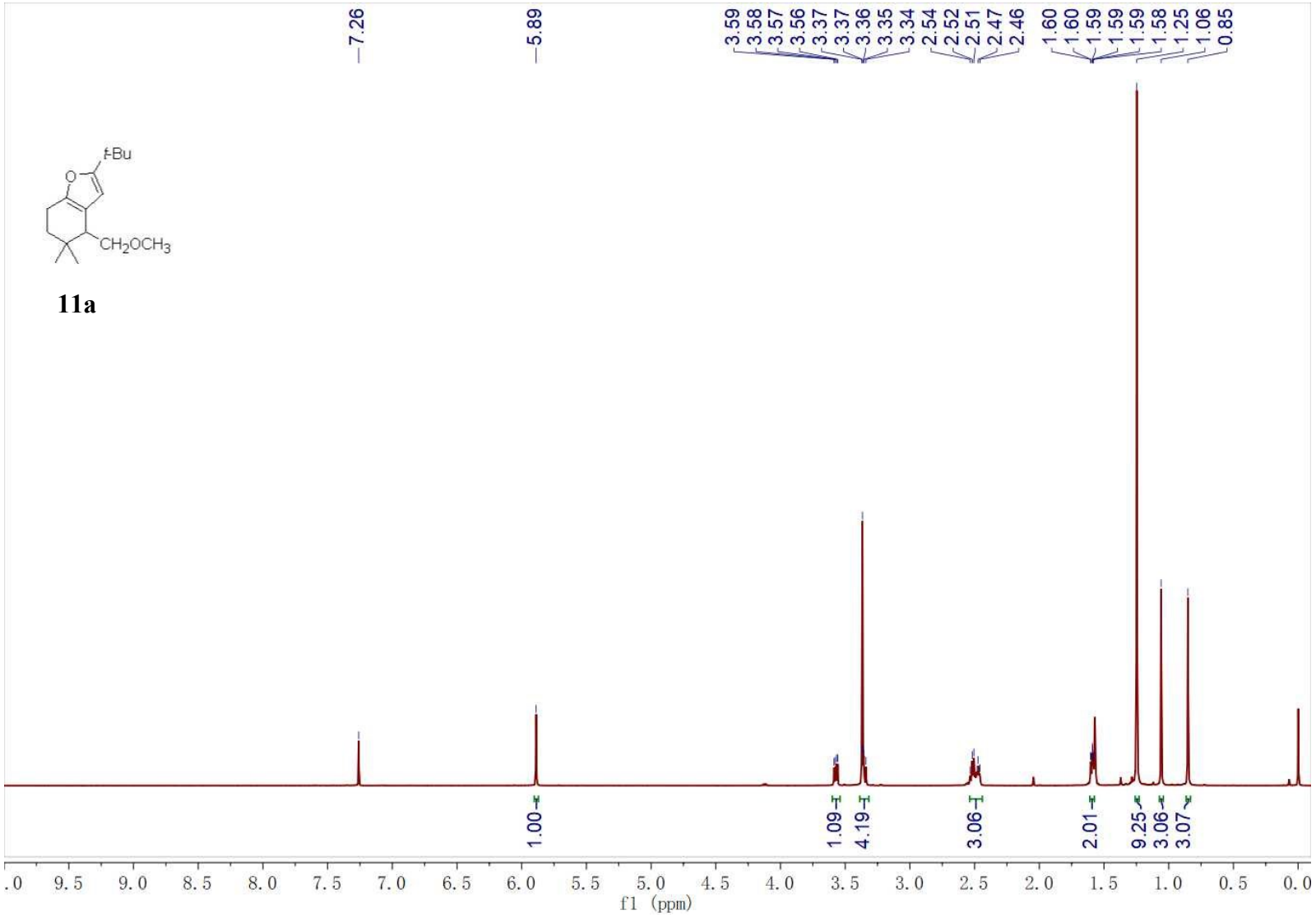

7.26
5.89
3.47
3.46
3.46
3.45
3.44
3.38
3.38
3.36
3.34
3.32
2.84
2.82
2.81
2.80
2.79
2.56
2.55
2.53
2.52
1.95
1.93
1.92
1.91
1.90
1.88
1.87
1.85
1.84
1.83
1.76
1.74
1.73
1.73
1.71
1.70
1.69
1.63
1.62
1.61
1.59
1.58
1.51
1.50
1.49
1.47
1.45
1.38
1.36
1.35
1.34
1.31
1.29
1.26
0.90
0.89
0.87
n-C6H13
O
O
0.98
1.09
2.91
1.03
0.96
4.01
2.19
1.20
2.12
1.18
7.36
3.00
f1 (ppm)

10n

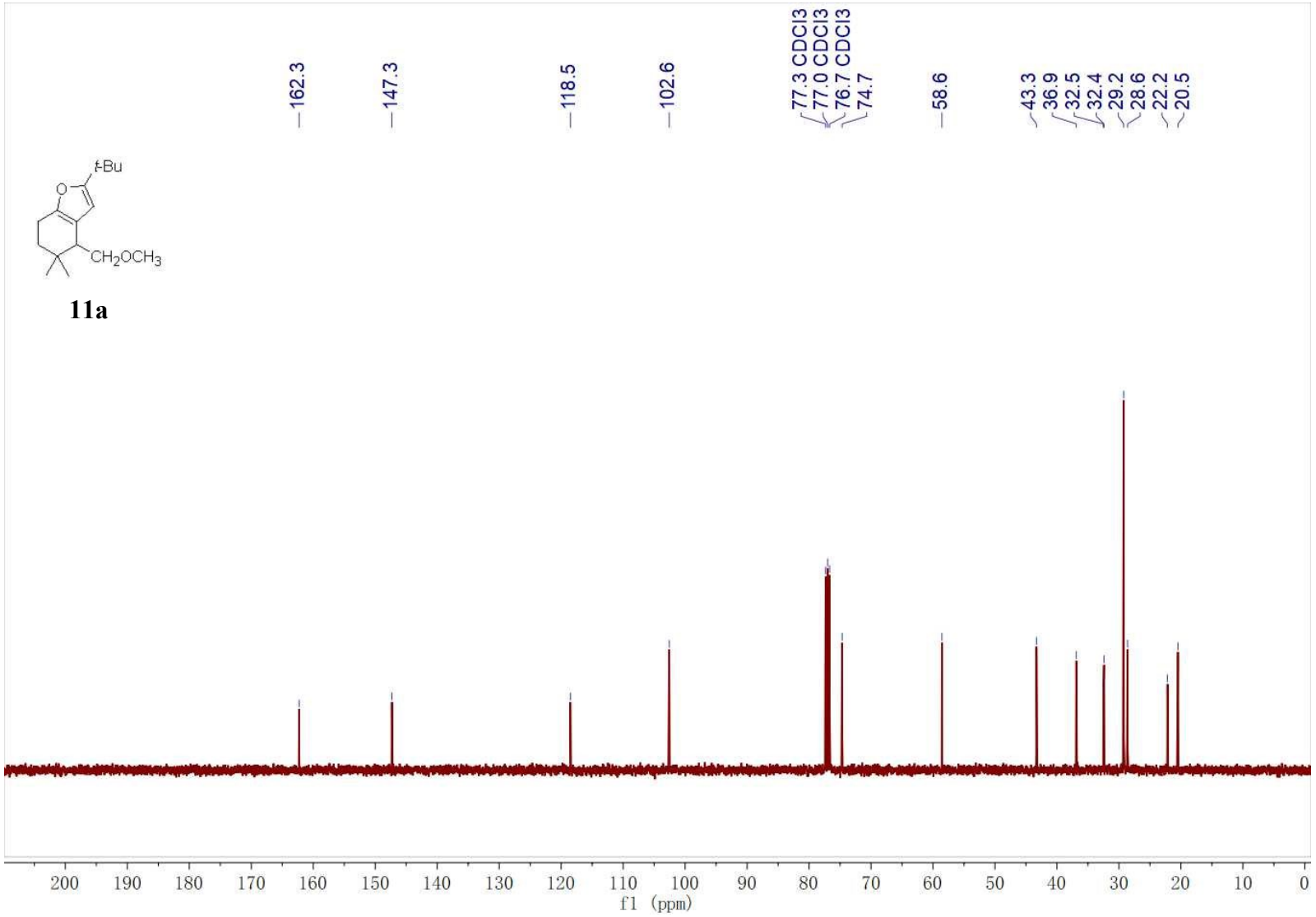

154.4
149.2
118.4
104.4
77.3 CDCl3
77.0 CDCl3
76.8
76.8 CDCl3
58.8
33.7
31.6
29.0
28.2
28.2
26.4
23.1
22.6
21.2
14.1
n-C6H13
O
O
10n
77.3 CDCl3
77.0 CDCl3
76.8
76.8 CDCl3
77.3 77.2 77.1 77.0 76.9 76.8 76.7
f1 (ppm)
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.43 7.41 7.38 7.29 7.29 7.28 6.57 3.54 3.53 3.52 3.51 3.44 3.43 3.41 2.97 2.92 2.91 2.90 2.68 2.67 2.56 2.54 2.52 2.51 2.47 2.46 2.44 2.32 2.19 2.17 2.14 2.10 2.09 2.08 2.05 2.04 2.01 2.00 1.99 1.95 1.94 1.93 1.91 1.81 1.80 1.68 1.66 1.64 1.62 1.58 1.57 1.55 1.54 1.53 1.52 1.49 1.48 1.46 0.95
10o
2.04 1.86 1.00 1.02 4.02 3.09 2.00 1.03 1.01 1.05 1.03 4.19 1.10 1.07 8.72 3.09
f1 (ppm)

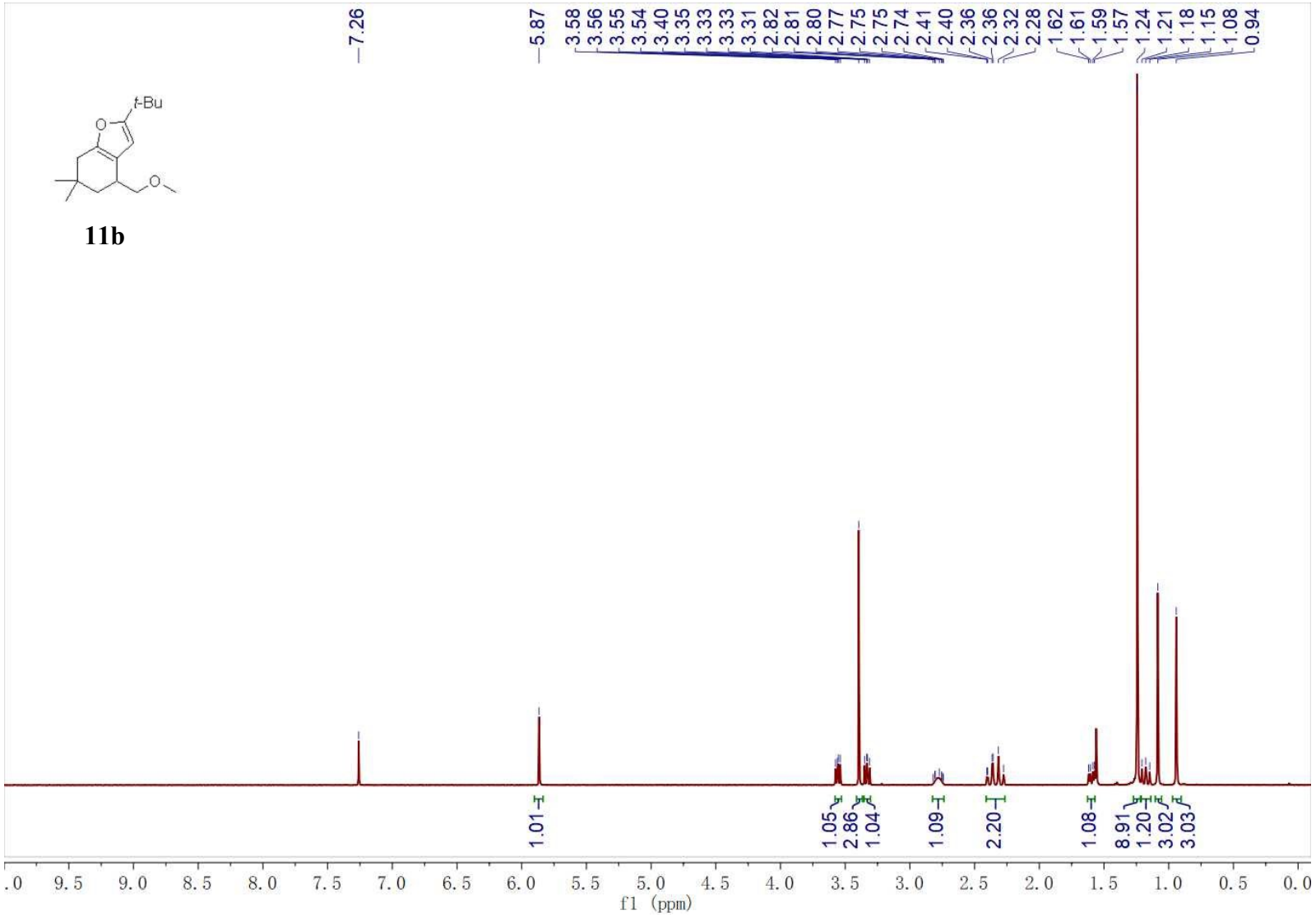

220.5
151.7
150.8
138.1
136.4
128.8
125.4
123.6
120.8
120.1
104.6
77.3 CDCl3
77.0 CDCl3
76.8
76.7 CDCl3
58.9
50.6
48.1
44.5
38.2
35.9
33.8
31.7
29.5
26.6
26.4
25.8
23.4
21.7
10o
f1 (ppm)


7.26
5.89
3.59
3.58
3.57
3.56
3.37
3.37
3.36
3.35
3.34
2.54
2.52
2.51
2.47
2.46
1.60
1.60
1.59
1.59
1.59
1.58
1.25
1.06
0.85
t-Bu
O
CH2OCH3
1.00
1.09
4.19
3.06
2.01
9.25
3.06
3.07
f1 (ppm)

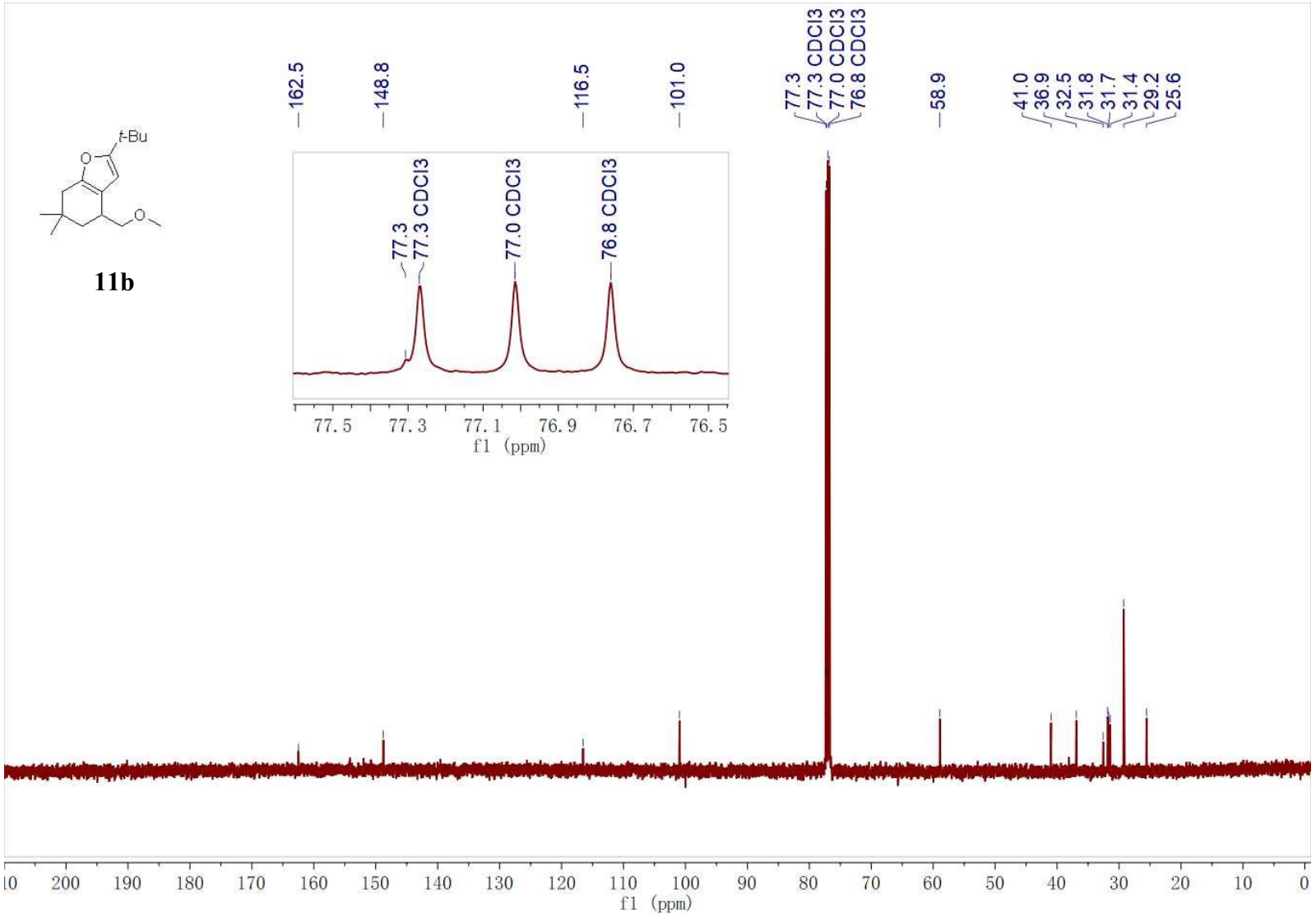
11a

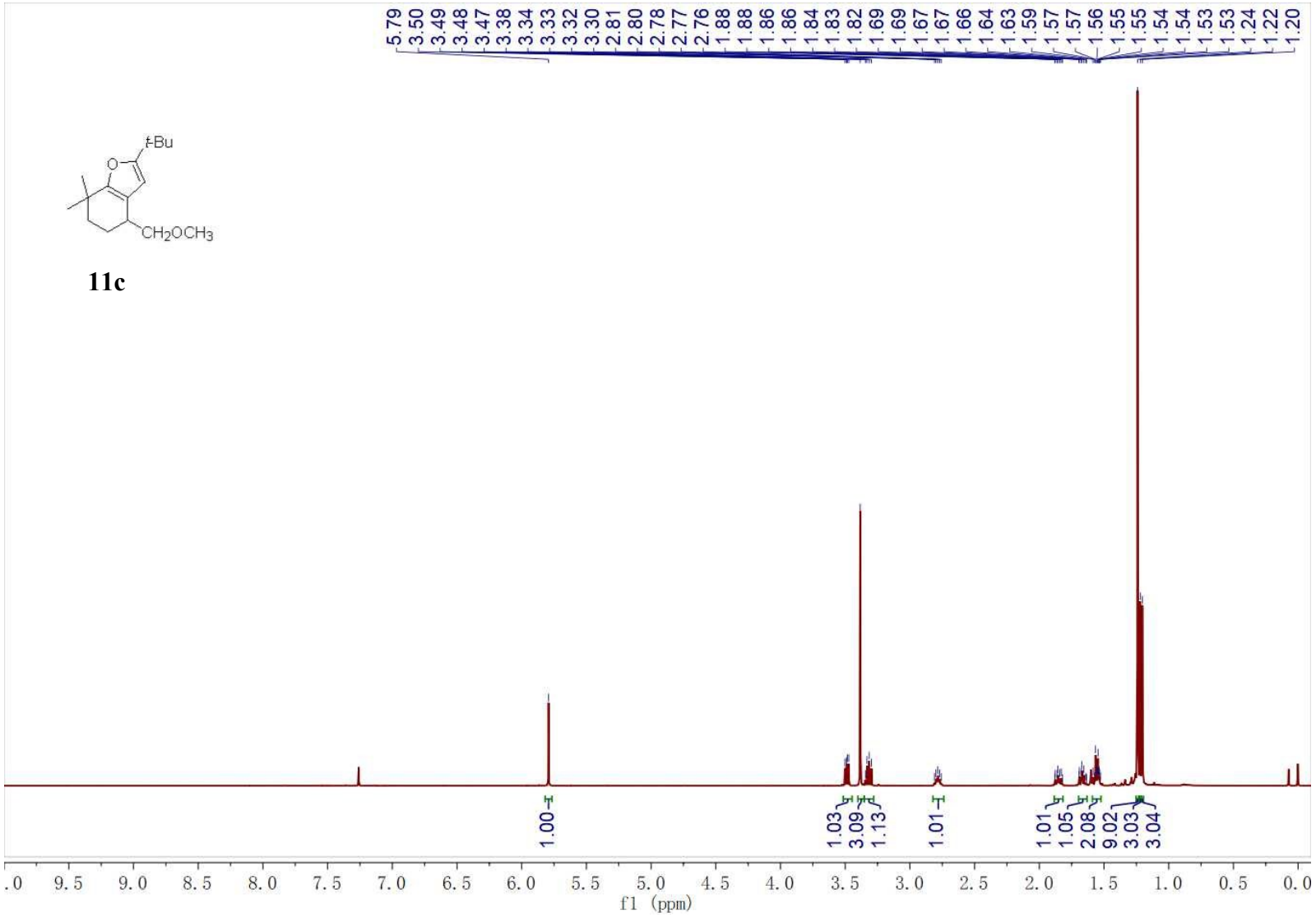

162.3
147.3
118.5
102.6
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
74.7
58.6
43.3
36.9
32.5
32.4
29.2
28.6
22.2
20.5
t-Bu
O
CH2OCH3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

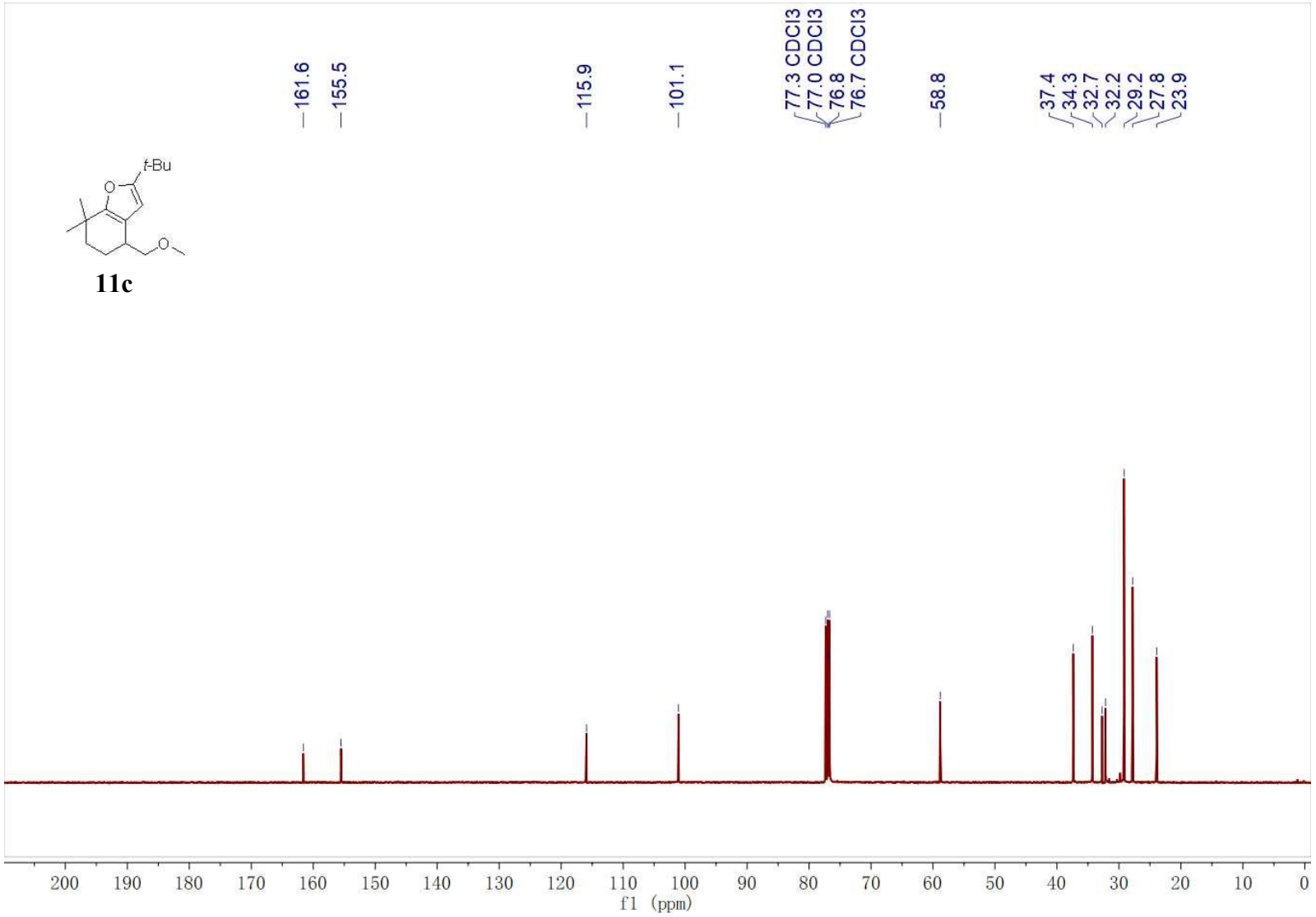
11a


7.26
5.87
3.58
3.56
3.55
3.54
3.40
3.35
3.33
3.33
3.31
2.82
2.81
2.80
2.77
2.75
2.75
2.74
2.41
2.40
2.36
2.36
2.32
2.28
1.62
1.61
1.59
1.57
1.24
1.21
1.18
1.15
1.08
0.94
t-Bu
O
O
1.01
1.05
2.86
1.04
1.09
2.20
1.08
8.91
1.20
3.02
3.03
f1 (ppm)

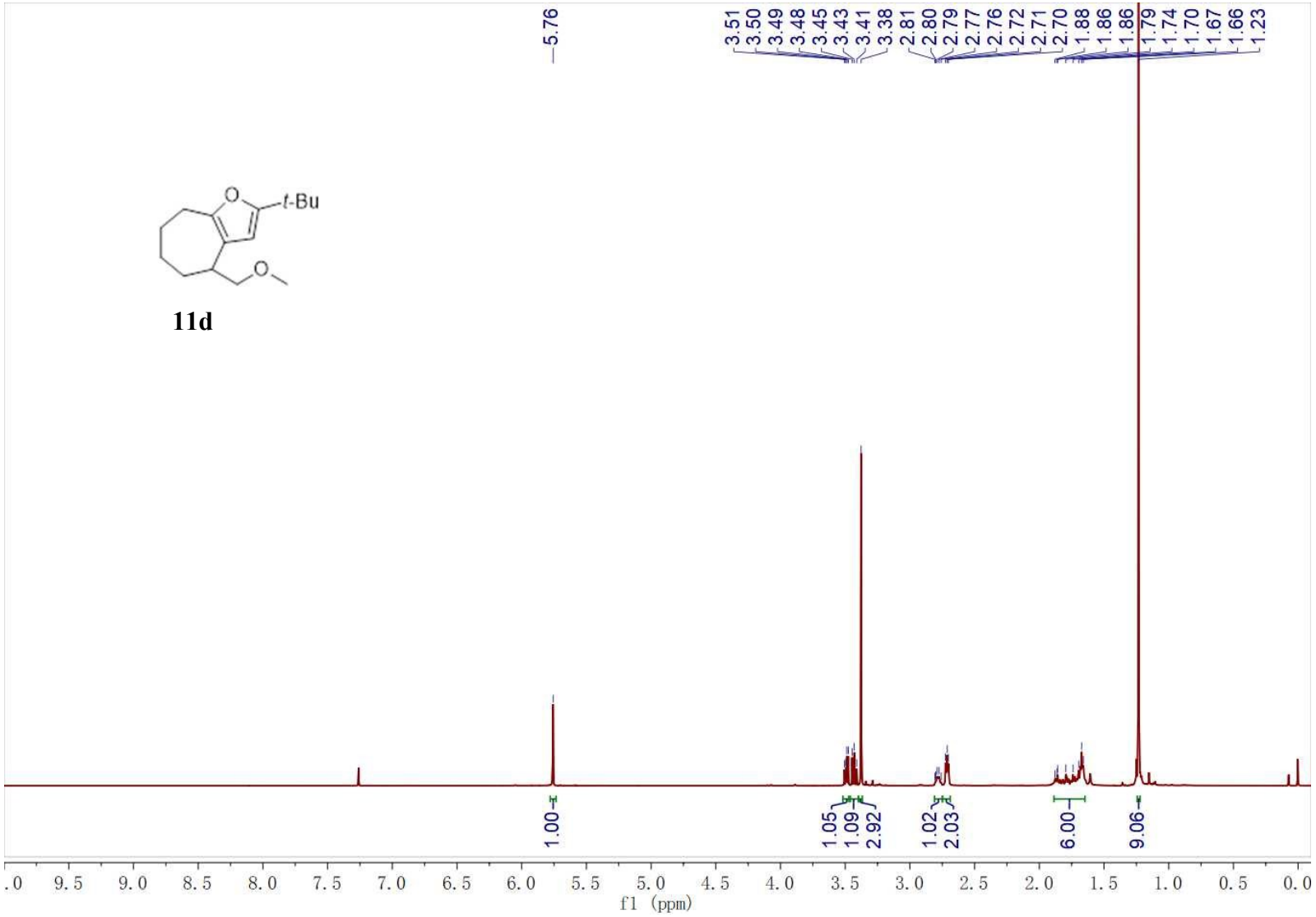
11b


t-Bu
O
O
11b
162.5
148.8
116.5
101.0
77.3
77.3 CDCl3
77.0 CDCl3
76.8 CDCl3
58.9
41.0
36.9
32.5
31.8
31.7
31.4
29.2
25.6
77.5
77.3
77.1
76.9
76.7
76.5
f1 (ppm)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

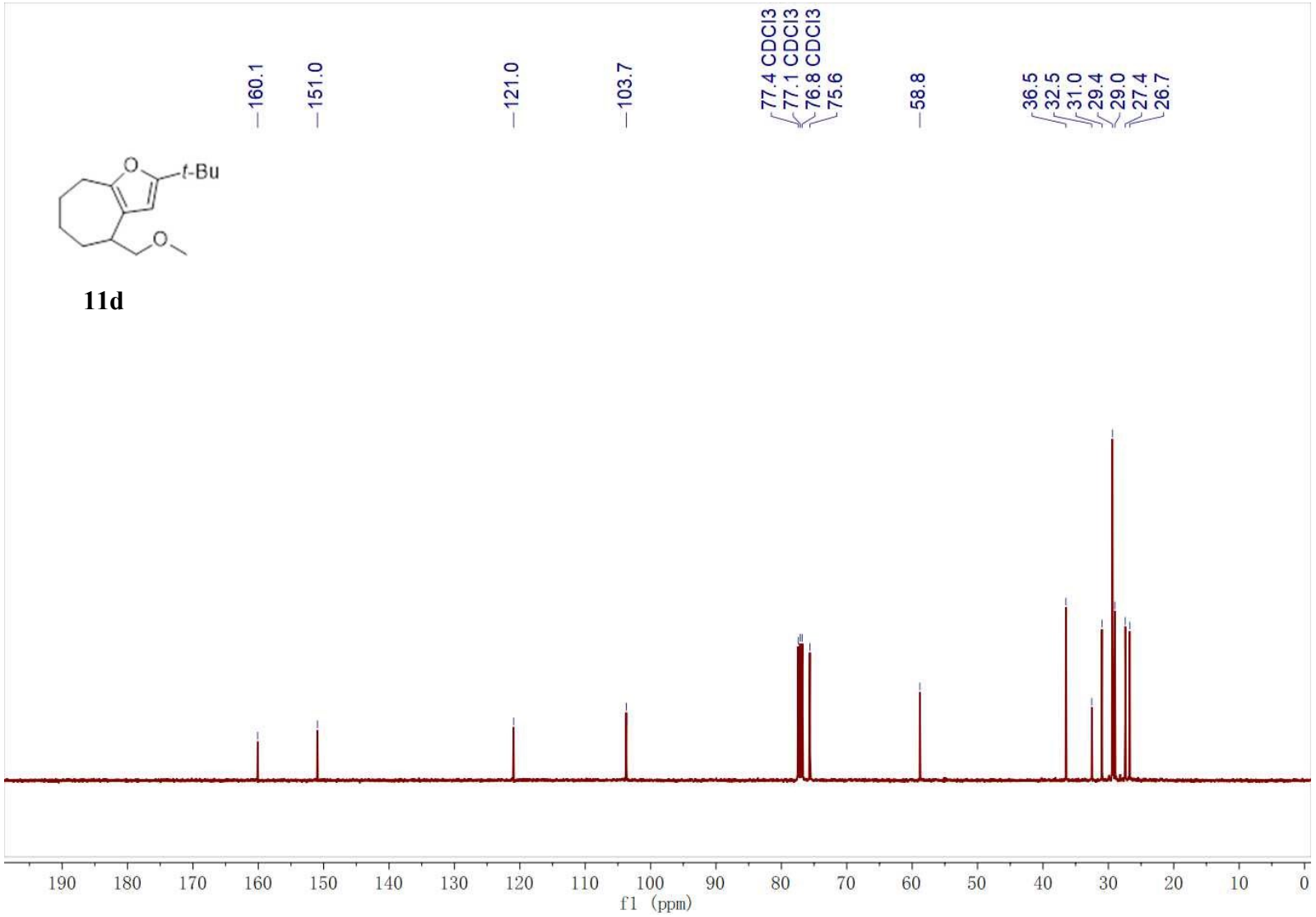

5.79
3.50
3.49
3.48
3.47
3.38
3.34
3.33
3.32
3.30
2.81
2.80
2.78
2.77
2.76
1.88
1.88
1.86
1.86
1.84
1.83
1.82
1.69
1.69
1.67
1.67
1.66
1.64
1.63
1.59
1.57
1.57
1.56
1.55
1.55
1.54
1.54
1.53
1.53
1.24
1.22
1.20
t-Bu
O
CH2OCH3
11c
1.00
1.03
3.09
1.13
1.01
1.01
1.05
2.08
9.02
3.03
3.04
f1 (ppm)

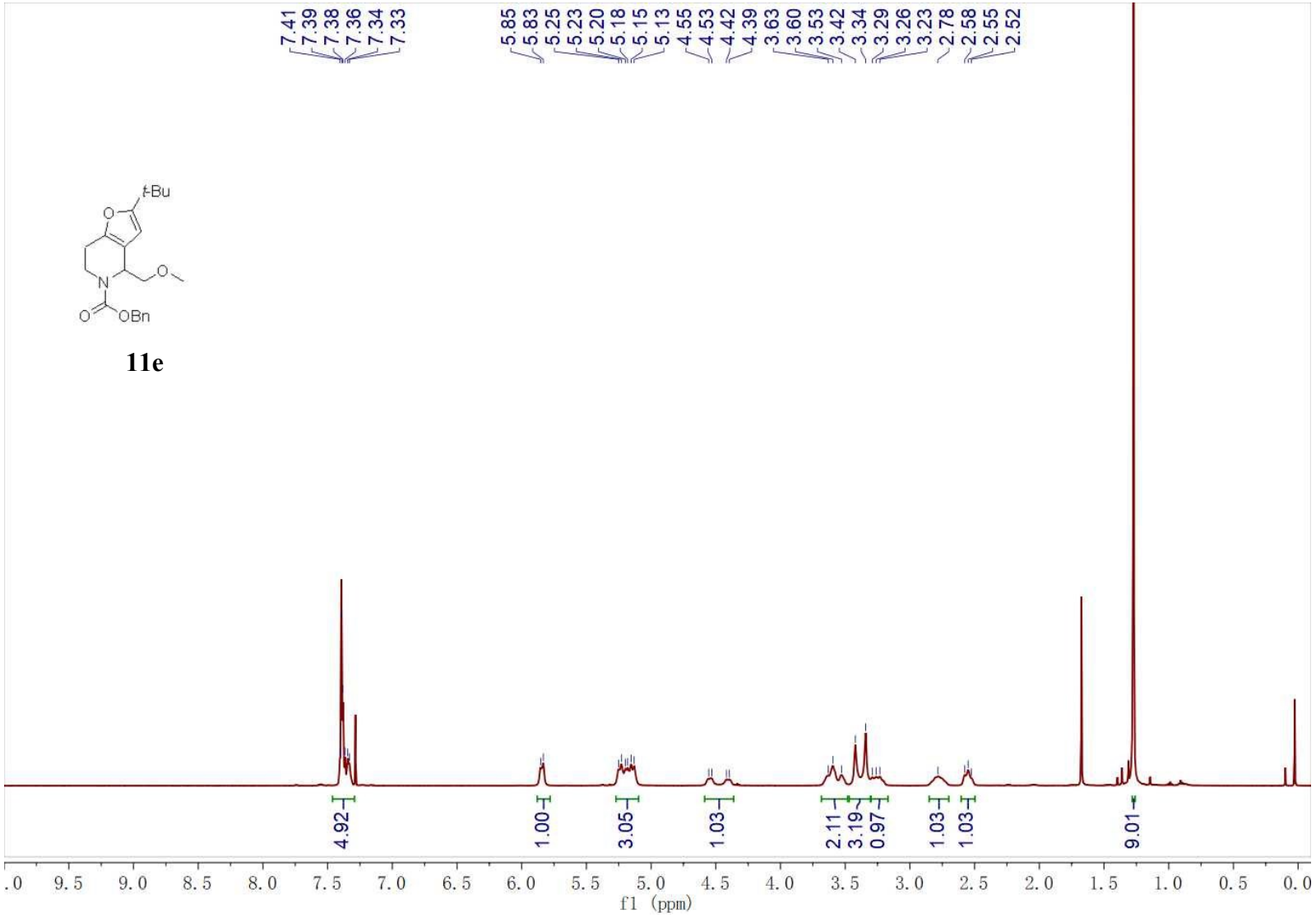

161.6
155.5
115.9
101.1
77.3 CDCl3
77.0 CDCl3
76.8
76.7 CDCl3
58.8
37.4
34.3
32.7
32.2
29.2
27.8
23.9
t-Bu
O
O
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

11c

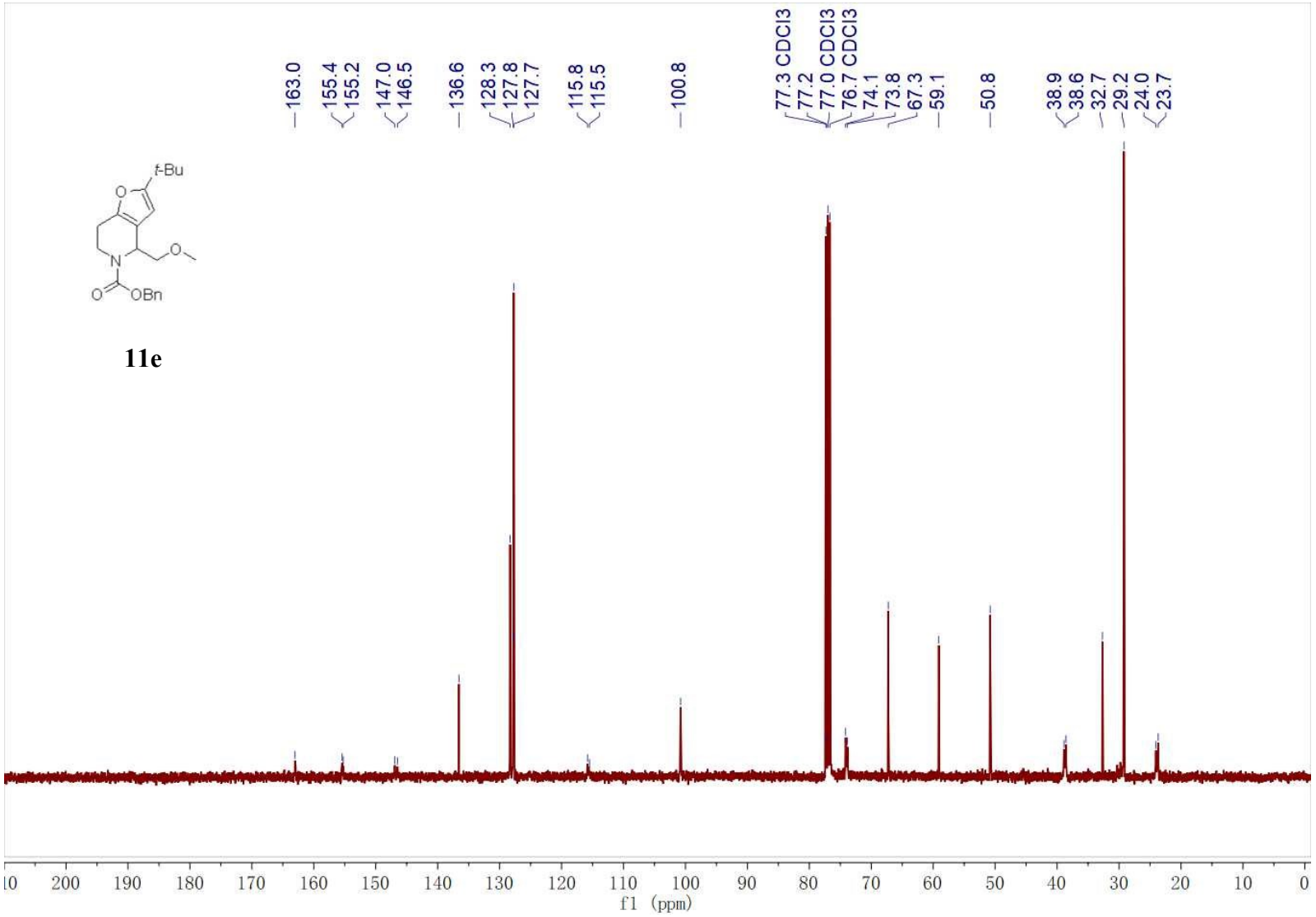

5.76
3.51
3.50
3.49
3.48
3.45
3.43
3.41
3.38
2.81
2.80
2.79
2.77
2.76
2.72
2.71
2.70
1.88
1.86
1.86
1.79
1.74
1.70
1.67
1.66
1.23
t-Bu
O
O
1.00
1.05
1.09
2.92
1.02
2.03
6.00
9.06
f1 (ppm)

11d

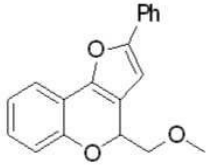

160.1
151.0
121.0
103.7
77.4 CDCl3
77.1 CDCl3
76.8 CDCl3
75.6
58.8
36.5
32.5
31.0
29.4
29.0
27.4
26.7
t-Bu
O
O
11d
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.41
7.39
7.38
7.36
7.34
7.33
5.85
5.83
5.25
5.23
5.20
5.18
5.15
5.13
4.55
4.53
4.42
4.39
3.63
3.60
3.53
3.42
3.34
3.29
3.26
3.23
2.78
2.58
2.55
2.52
t-Bu
O
N
O
OBn
4.92
1.00
3.05
1.03
2.11
3.19
0.97
1.03
1.03
9.01
f1 (ppm)

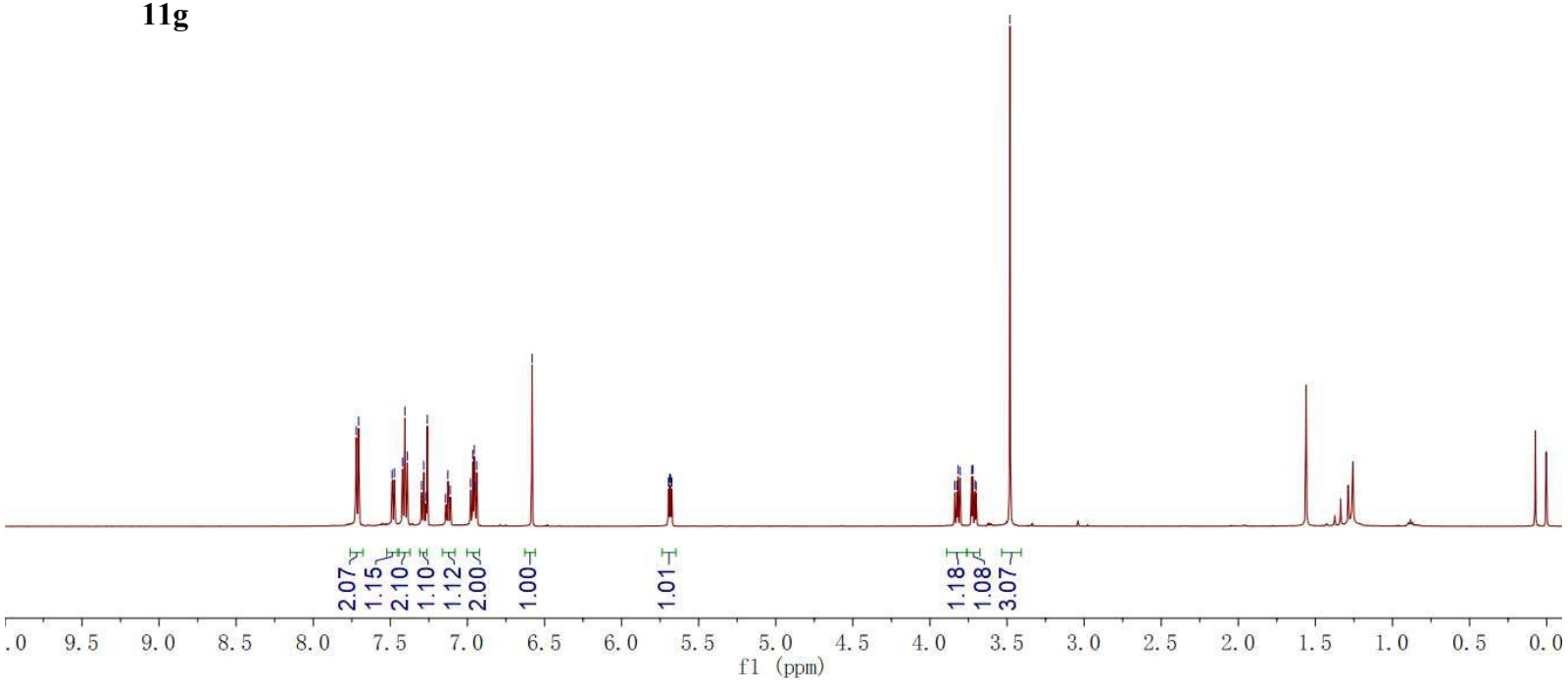
11e

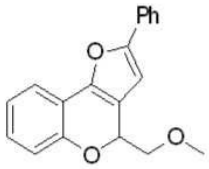

163.0
155.4
155.2
147.0
146.5
136.6
128.3
127.8
127.7
115.8
115.5
100.8
77.3 CDCl3
77.2
77.0 CDCl3
76.7 CDCl3
74.1
73.8
67.3
59.1
50.8
38.9
38.6
32.7
29.2
24.0
23.7
t-Bu
O
N
O
OBn
O
11e
f1 (ppm)


7.72
7.71
7.49
7.47
7.42
7.41
7.39
7.30
7.28
7.27
7.26
7.14
7.13
7.11
6.98
6.96
6.96
6.95
6.94
6.58
5.70
5.69
5.68
5.67
3.84
3.83
3.82
3.80
3.73
3.72
3.71
3.70
3.48


Ph
O
O
O

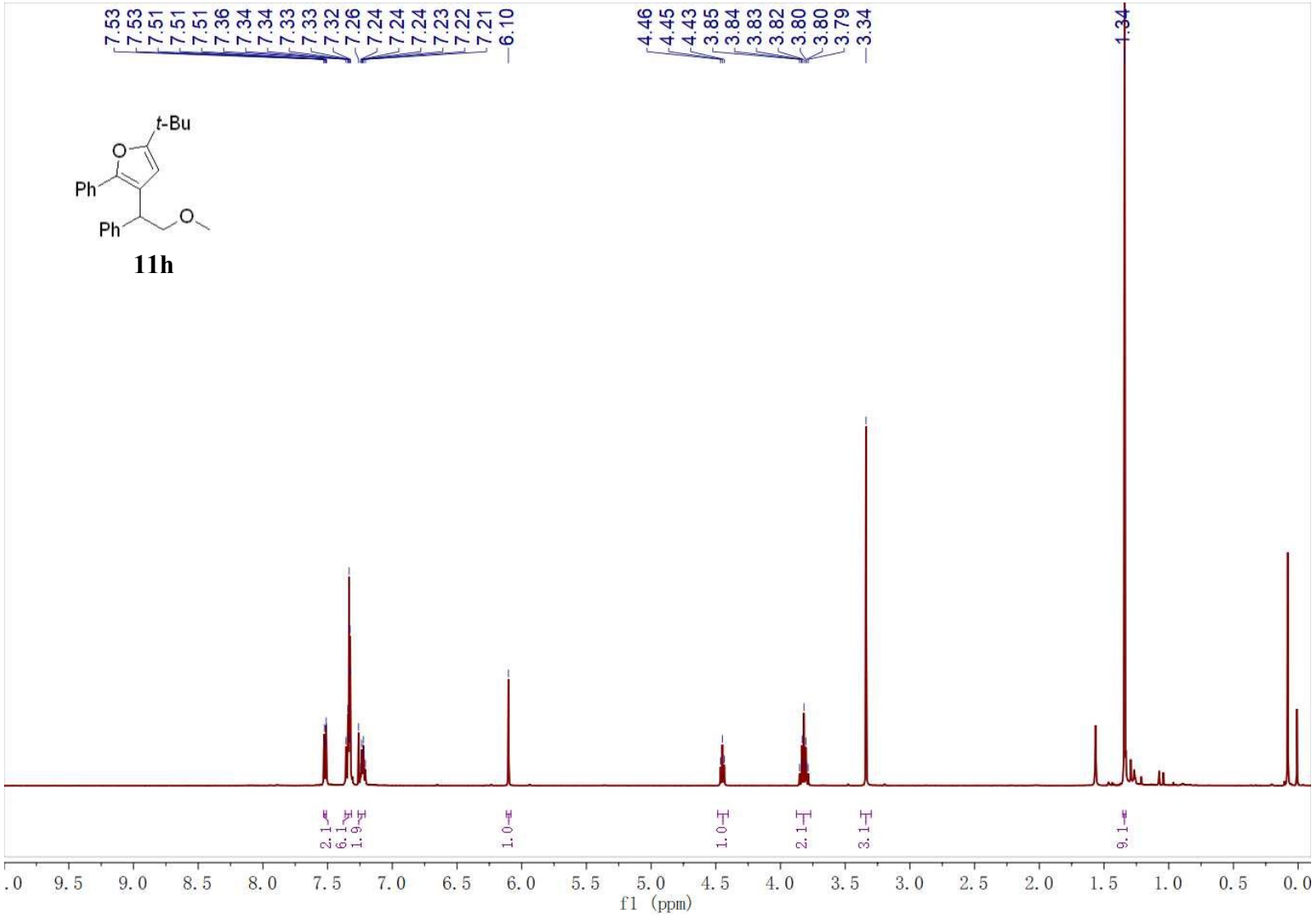
11g

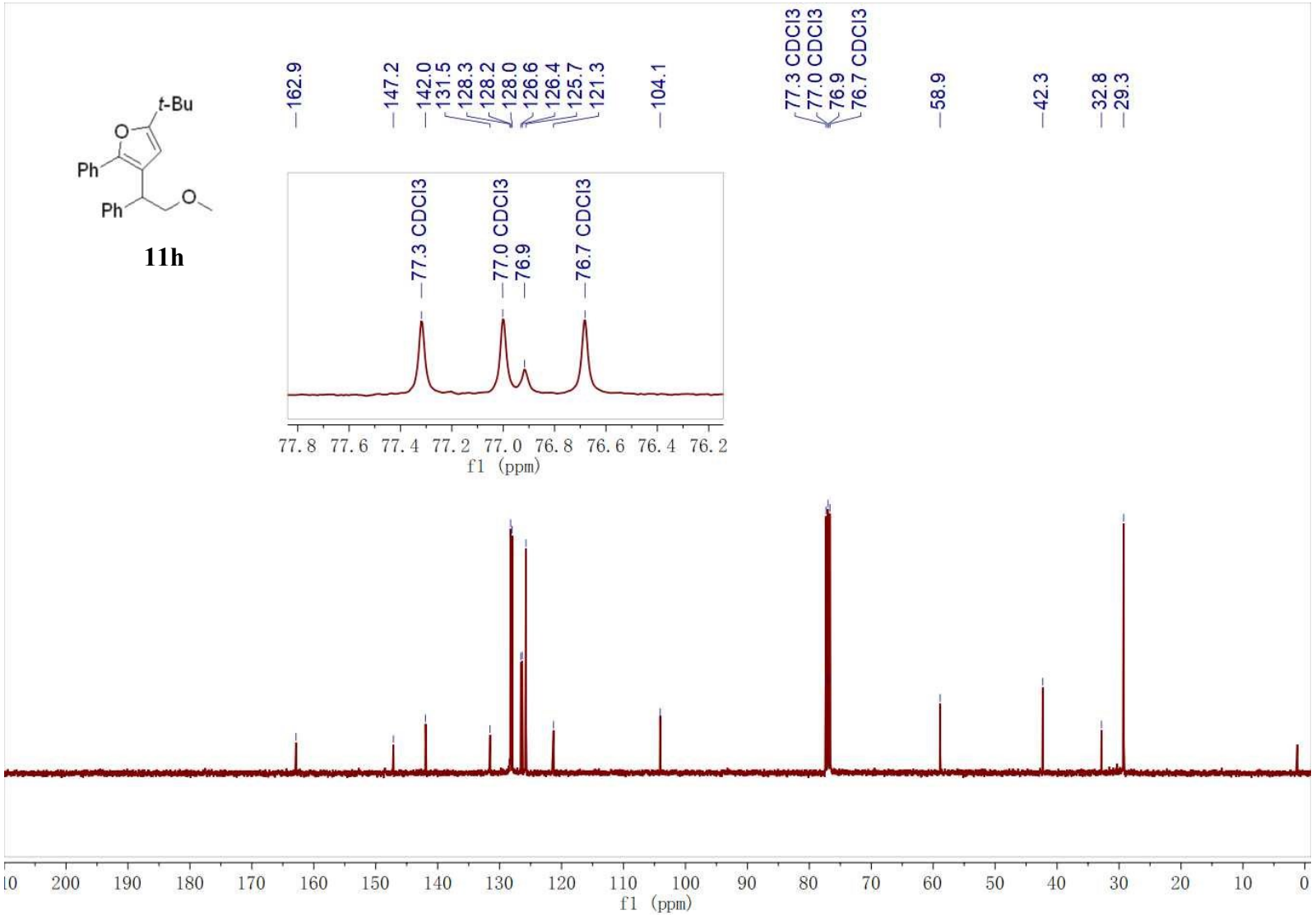

2.07
1.15
2.10
1.10
1.12
2.00
1.00
1.01
1.18
1.08
3.07
.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


Ph
O
O
O

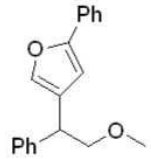
11g

154.4
152.3
145.8
130.4
128.8
128.6
127.6
123.7
121.4
119.4
116.5
116.2
116.1
103.4
77.3 CDCl3
77.0 CDCl3
76.8 CDCl3
75.8
75.1
59.6
f1 (ppm)

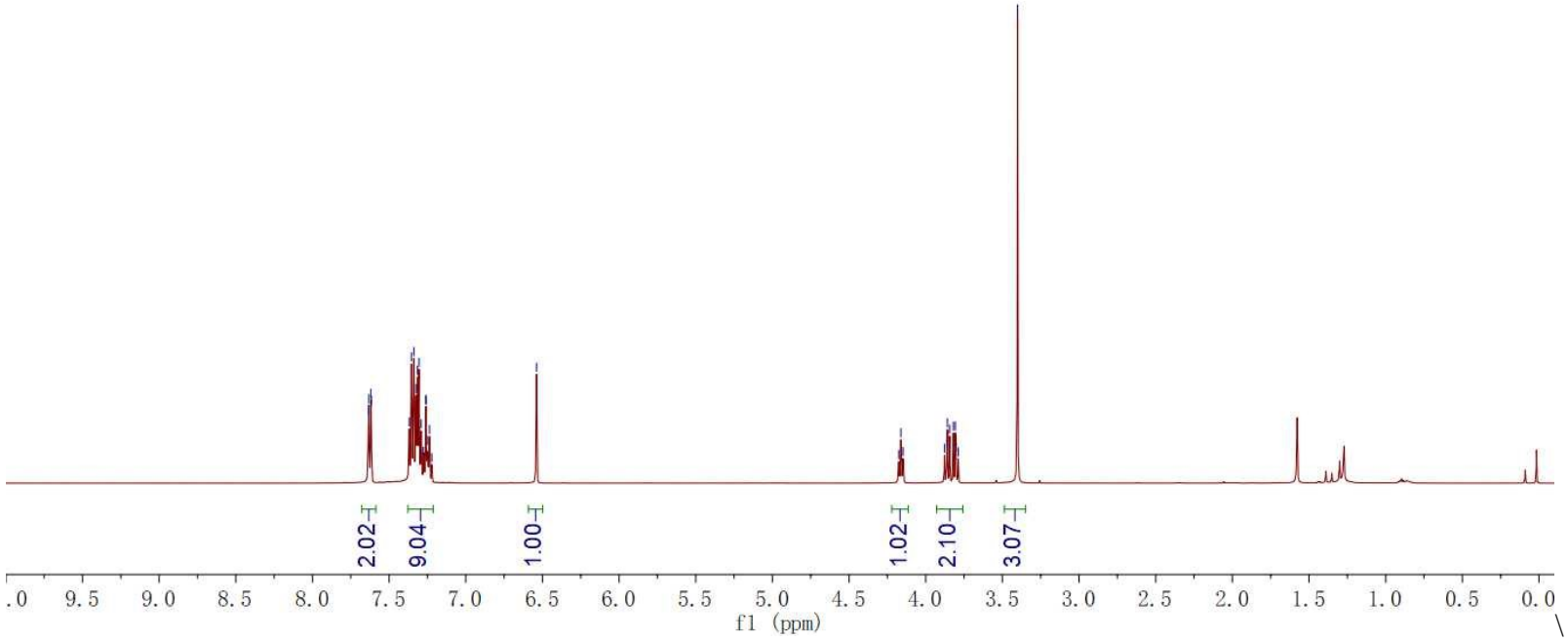

7.53
7.53
7.51
7.51
7.51
7.36
7.34
7.34
7.33
7.33
7.32
7.26
7.24
7.24
7.24
7.23
7.22
7.21
6.10
4.46
4.45
4.43
3.85
3.84
3.83
3.82
3.80
3.80
3.79
3.34
1.34
t-Bu
O
Ph
Ph
O
11h
2.1
6.1
1.9
1.0
1.0
2.1
3.1
9.1
f1 (ppm)

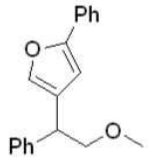

t-Bu
O
Ph
Ph
O
11h
162.9
147.2
142.0
131.5
128.3
128.2
128.0
126.6
126.4
125.7
121.3
104.1
77.3 CDCl3
77.0 CDCl3
76.9
76.7 CDCl3
58.9
42.3
32.8
29.3
77.3 CDCl3
77.0 CDCl3
76.9
76.7 CDCl3
77.8 77.6 77.4 77.2 77.0 76.8 76.6 76.4 76.2
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.64
7.63
7.62
7.62
7.37
7.35
7.34
7.33
7.32
7.30
7.29
7.28
7.26
7.26
7.25
7.24
7.22
6.54
4.18
4.16
4.15
3.88
3.86
3.86
3.84
3.82
3.81
3.80
3.79
3.40


Ph
O
Ph
O

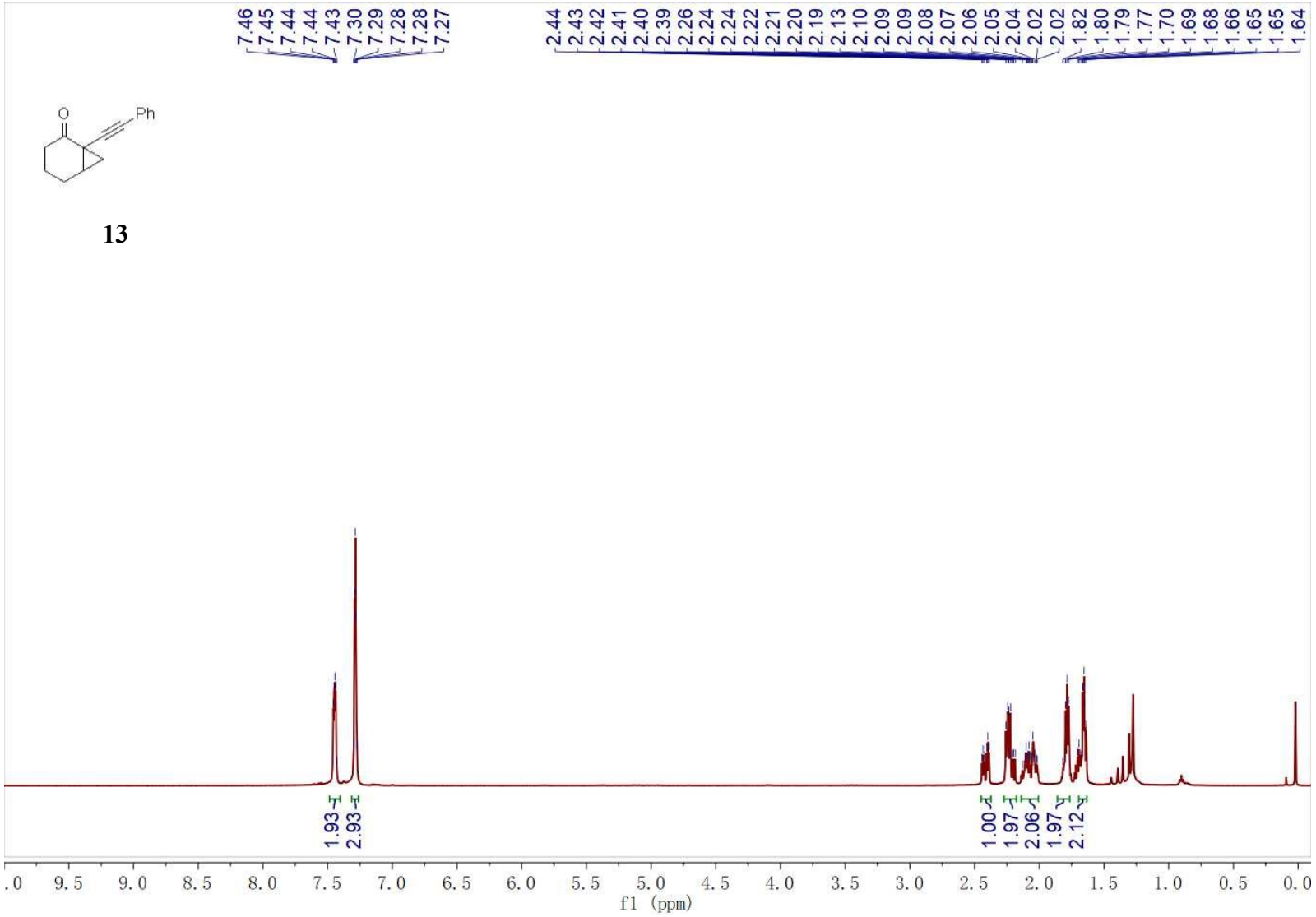
11i


2.02
9.04
1.00
1.02
2.10
3.07
f1 (ppm)

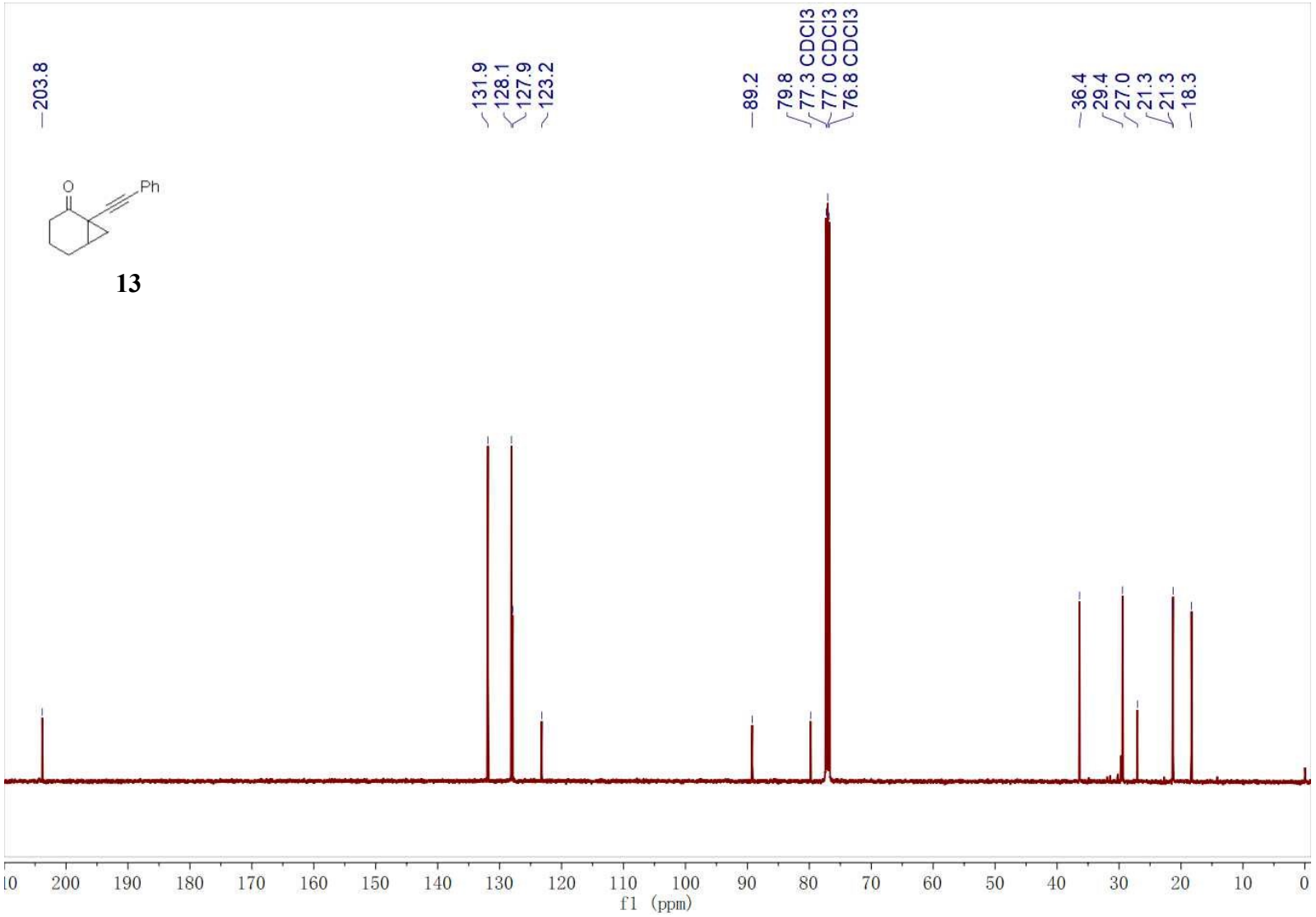

Ph
O
Ph
O

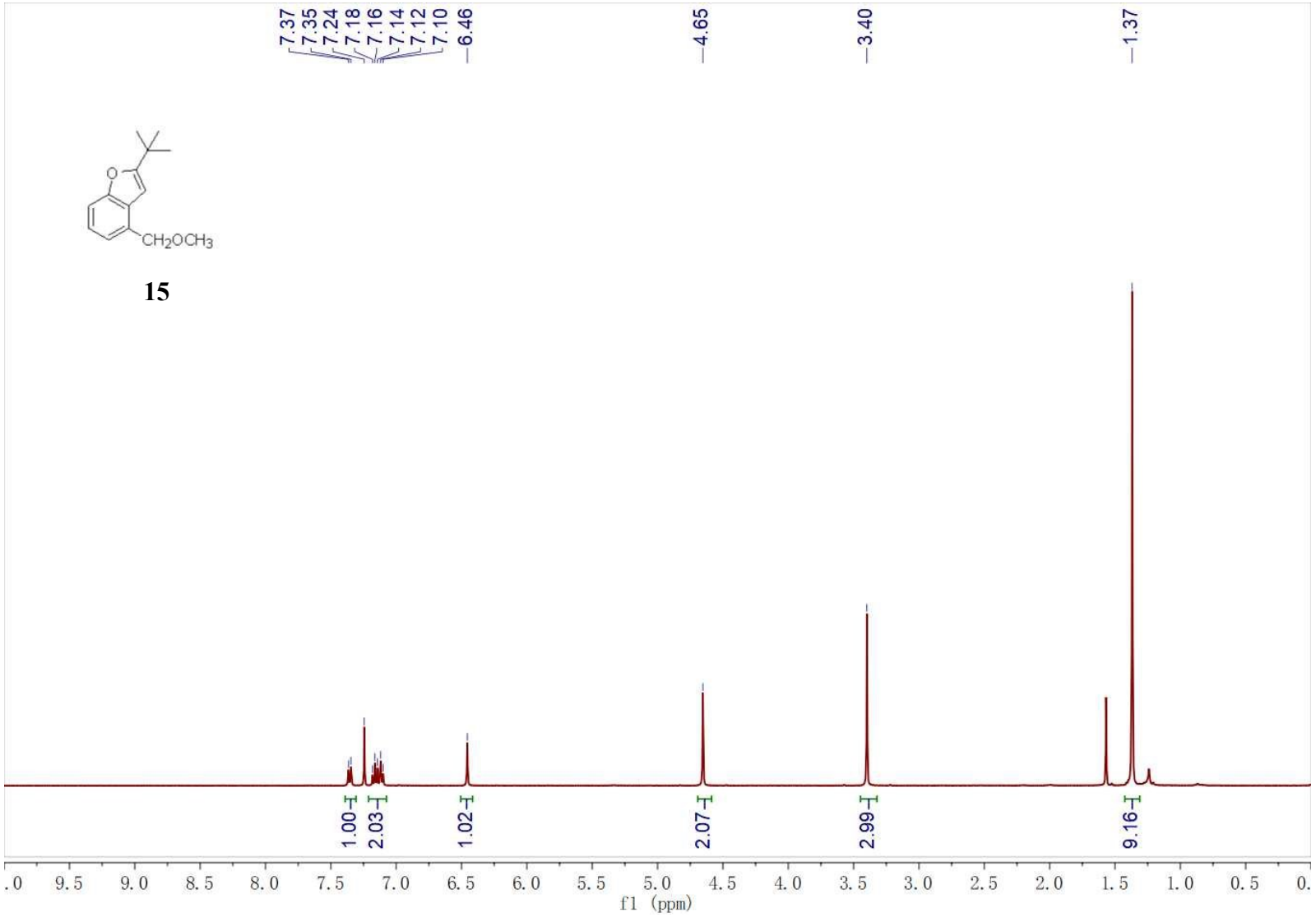
11i

153.9
141.5
139.1
130.9
128.6
128.5
128.2
128.1
127.2
126.8
123.7
106.1
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
76.5
58.9
42.8
f1 (ppm)

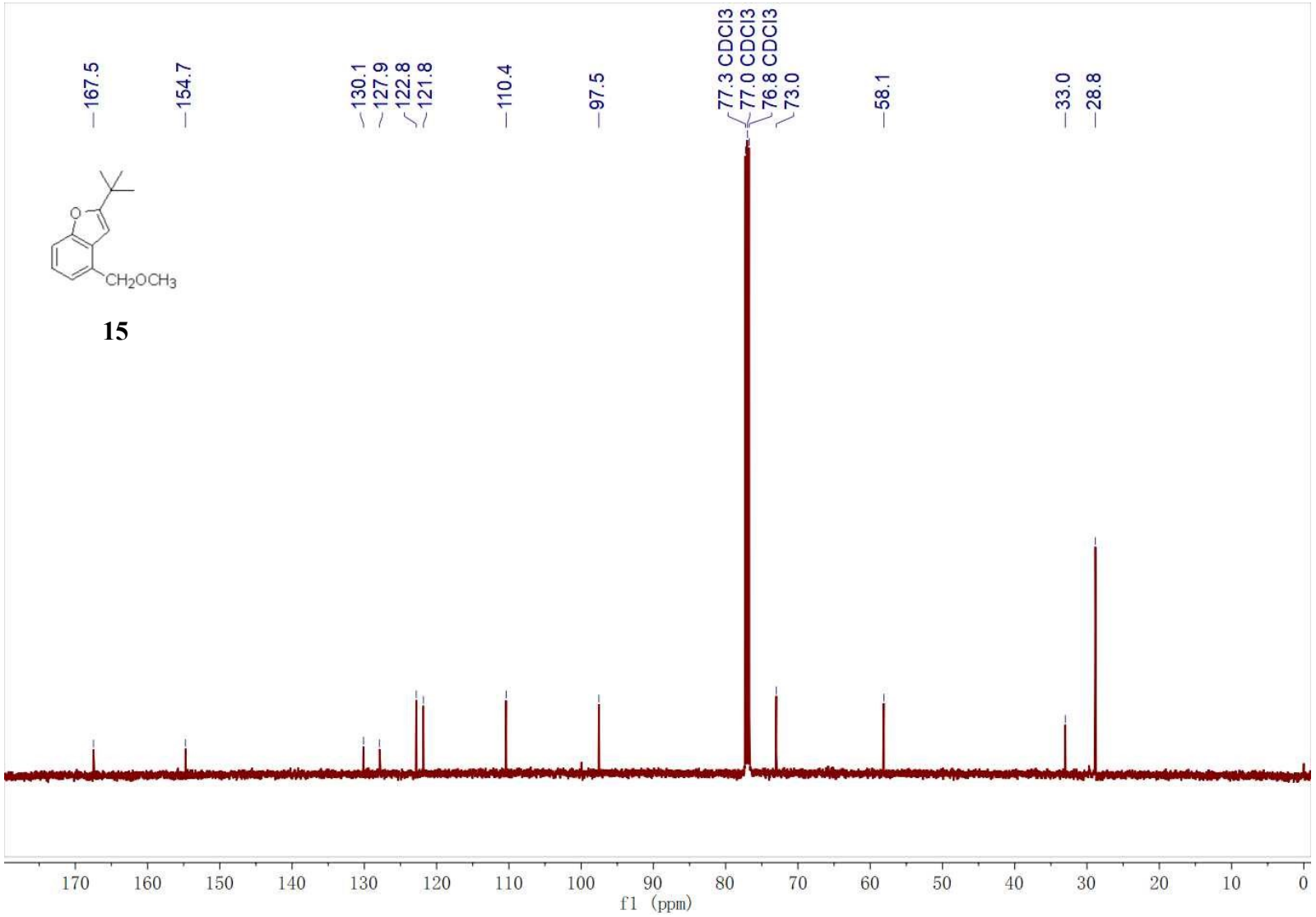

7.46
7.45
7.44
7.44
7.43
7.30
7.29
7.28
7.28
7.27
2.44
2.43
2.42
2.41
2.40
2.39
2.26
2.24
2.24
2.22
2.21
2.20
2.19
2.13
2.10
2.09
2.09
2.08
2.07
2.06
2.05
2.04
2.02
2.02
1.82
1.80
1.79
1.77
1.70
1.69
1.68
1.66
1.65
1.65
1.64
O
Ph
1.93
2.93
1.00
1.97
2.06
1.97
2.12
f1 (ppm)

13


203.8
131.9
128.1
127.9
123.2
89.2
79.8
77.3 CDCl3
77.0 CDCl3
76.8 CDCl3
36.4
29.4
27.0
21.3
21.3
18.3
O
Ph
13
f1 (ppm)


7.37
7.35
7.24
7.18
7.16
7.14
7.12
7.10
6.46
4.65
3.40
1.37
CH2OCH3
1.00
2.03
1.02
2.07
2.99
9.16
f1 (ppm)

15


167.5
154.7
130.1
127.9
122.8
121.8
110.4
97.5
77.3 CDCl3
77.0 CDCl3
76.8 CDCl3
73.0
58.1
33.0
28.8
O
CH2OCH3
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

15