Supplementary Information

Anodic Dearomatization of 2-Alkynylanilines for the Synthesis of Multi-functionalized Indoles

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Supplementary Data

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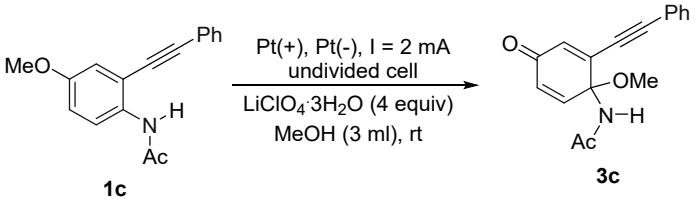
1. General Information

All reactions were performed in Schlenk tubes. Flash column chromatography was performed using silica gel (60-A pore size, 32–63 μm, standard grade). Analytical thin–layer chromatography was performed using glass plates pre-coated with 0.25 mm 230–400 mesh silica gel impregnated with a fluorescent indicator (254 nm). Thin layer chromatography plates were visualized by exposure to ultraviolet light. Organic solutions were concentrated on rotary evaporators at ~20 Torr (house vacuum) at 25–35 °C. Commercial reagents and solvents were used as received. Nuclear magnetic resonance (NMR) spectra are recorded in parts per million from internal tetramethylsilane on the δ scale.

2. Optimization of the Reaction Conditions

Table 1 Reaction Optimization with Substrate 1c

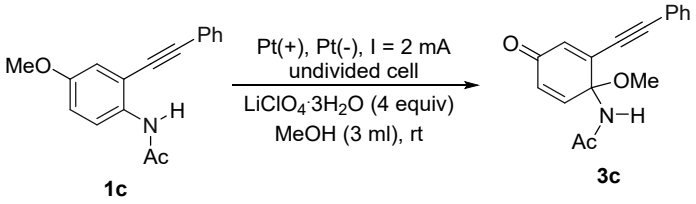


entry	variation from standard conditions[a]	**3c** yield[b]（%）
1	**none**	**98**
2	*n*-Bu_4NClO_4 instead of $LiClO_4$	75
3	*n*-Bu_4NBF_4 instead of $LiClO_4$	63
4	LiCl instead of $LiClO_4$	48
5	RVC (+) instead of Pt (+)	43
6	1.5 mA instead of 2.0 mA	62
7	3 mA instead of 2.0 mA	78
8	no electric current	0
9	at 0 °C	73
10	at 50 °C	81
11	gram scale	96

[a]Standard conditions: **1c** (0.1 mmol), $LiClO_4·3H_2O$ (4.0 equiv.), and MeOH (3 mL), in an undivided cell with two platinum plate electrodes (each 1.0 × 1.0 cm^2), constant current = 2 mA, room temperature, 3.5 h. [b]Isolated yield.

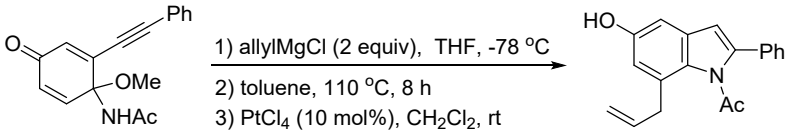
3. Experimental Procedures

Representative Procedure



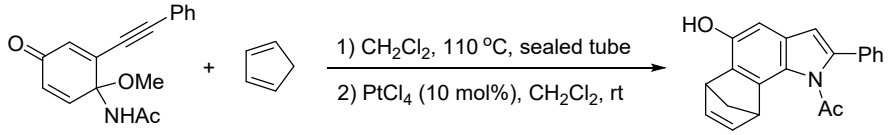
1c **3c**

The electrolysis was carried out in an undivided cell equipped with two platinum electrodes (1.0×1.0 cm^2). A mixture of *N*-(4-methoxy-2-(phenylethynyl)phenyl)acetamide **1c** (26 mg, 0.1 mmol, 1.0 equiv), $LiClO_4 \cdot 3H_2O$ (64 mg, 0.4 mmol, 4.0 equiv) and MeOH (3 mL) were added to the electrochemical cell. Electrolysis was started at 25 °C with a constant current of 2 mA maintained for 3.5 h. After the material was consumed completely (monitored by TLC analysis), the reaction mixture was washed with water, dried over Na_2SO_4, filtered and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 1/2) to furnish the desired compound **3c**.



3c **32**

A stirred solution of *N*-(1-methoxy-4-oxo-2-(phenylethynyl)cyclohexa-2,5-dien-1-yl)acetamide **3c** (14 mg, 0.05 mmol, 1.0 equiv) in dry THF (1 mL) was cooled to -78 °C under nitrogen atmosphere. AllylMgCl (0.05 mL, 2 mol/L in THF, 2.0 equiv) was added dropwise to this solution. After stirring at -78 °C for 1 h, the reaction was allowed to warm to room temperature and quenched with MeOH. the reaction mixture was passed through a short silica gel column to remove Magnesium salt with DCM as flushing agent and then concentrated in vacuo. The residue was dissolved in toluene (1 mL) and stirred at 110 °C for 8 h. The mixture was concentrated in vacuo and mixed with $PtCl_4$ (2 mg, 0.005 mmol, 0.1 equiv) in DCM (1.0 mL) at 25 °C for 8 h. After the intermediate was consumed completely (monitored by TLC analysis), the mixture was concentrated in vacuo. The residue was purified by flash column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 2/1) to furnish the desired compound **32**.

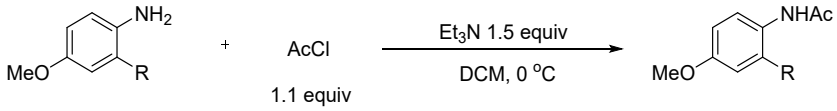


3c 5.0 equiv **48**

N-(1-methoxy-4-oxo-2-(phenylethynyl)cyclohexa-2,5-dien-1-yl)acetamide **3c** (9 mg, 0.03 mmol, 1.0 equiv) was dissolved in DCM (1 mL) in a sealed tube. Cyclopentadiene (10 mg, 0.15 mmol, 5.0 equiv) was added to this solution. After stirring at 110 °C for 24 h, the reaction mixture was cooled down to the room temperature and mixed with $PtCl_4$ (1 mg, 0.003 mmol, 0.1 equiv) for 8 h. After the intermediate was consumed completely (monitored by TLC analysis), the mixture was concentrated in vacuo. The residue

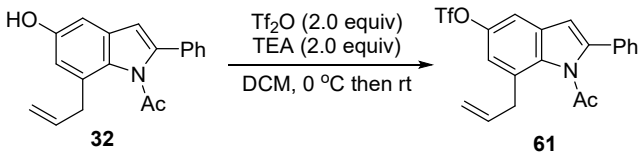
was purified by flash column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 2/1) to furnish the desired compound **48**.

The procedure for synthesis of a series of starting material



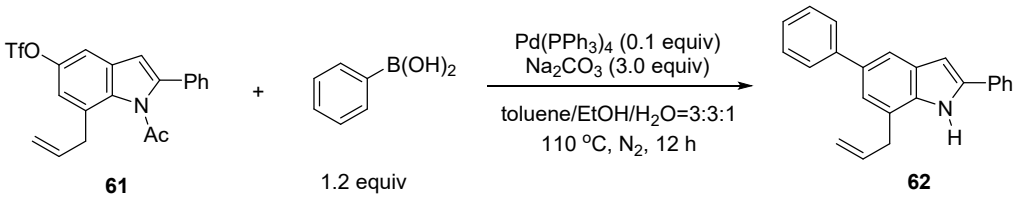
To a stirred solution of arylamine (1.0 equiv) in DCM (10 mL) was added AcCl (1.1 equiv) and Et_3N (1.5 equiv). The resulted mixture was stirred at 0 °C for 4 h. Then the reaction was quenched by saturated NH_4Cl solution and the reaction mixture was extracted with CH_2Cl_2 (3 x 5 mL). The combined organic layer was dried over anhydrous Na_2SO_4 and concentrated. The residue was purified by flash column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 2/1) to furnish the desired compound.

The procedure[1] for synthesis of compound 61



A stirred solution of 1-(7-allyl-5-hydroxy-2-phenyl-1*H*-indol-1-yl)ethan-1-one **32** (29 mg, 0.1 mmol, 1.0 equiv) in DCM (1 mL) cooled to 0 °C. Et_3N (20 mg, 0.2 mmol, 2 equiv) was added dropwise to this solution followed by Tf_2O (57 mg, 0.2 mmol, 2 equiv). After stirring at 0 °C for 10 min, the reaction was allowed to warm to room temperature and stirred for 2 h, quenched with saturated aqueous $NaHCO_3$ solution, and extracted by ethyl acetate (10 mL × 3). The combined organic layers were washed with brine, dried over Na_2SO_4, filtered and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 10/1) to furnish the desired compound **61** (94%).

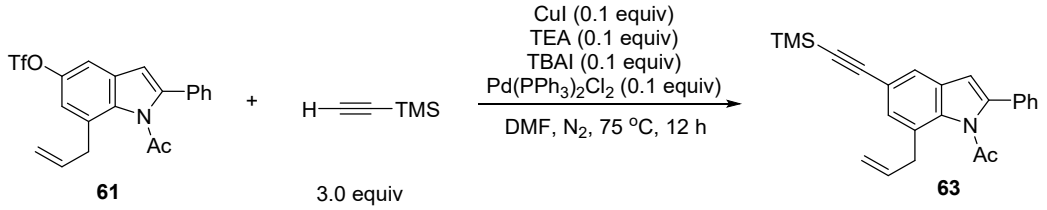
The procedure[2] for synthesis of compound 62



1-acetyl-7-allyl-2-phenyl-1*H*-indol-5-yl trifluoromethanesulfonate **61** (21 mg, 0.05 mmol, 1.0 equiv), phenylboronic acid (7 mg, 0.06 mmol, 1.2 equiv), tetrakis(triphenylphosphine)palladium (6 mg, 0.005 mmol, 0.1equiv), sodium carbonate (16 mg, 0.15 mmol, 3.0 equiv), toluene (0.3 mL), ethanol (0.3 mL) and water (0.1 mL) were mixed under nitrogen atmosphere. After stirring at 110 °C for 12 h the mixture was cooled down to the room temperature and treated with 2 M HCl (1 mL) and water (1 mL)., The aqueous phase was extracted with ethyl acetate (10 mL × 3). The combined organic layers were washed with brine, dried over Na_2SO_4 filtered and concentrated in vacuo. The residue was purified by flash column

chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 10/1) to furnish the desired compound **62** (83%).

The procedure[3] for synthesis of compound 63



To a stirred solution of 1-acetyl-7-allyl-2-phenyl-1*H*-indol-5-yl trifluoromethanesulfonate **61** (21mg, 0.05 mmol, 1.0 equiv), CuI (1 mg, 0.005 mmol, 0.1 equiv), TBAI (2 mg, 0.005 mmol, 0.1 equiv) and $Pd(PPh_3)Cl_2$ (4 mg, 0.005 mmol, 0.1 equiv) in dry DMF (1 mL) were added trimethylsilylacetylene (15 mg, 0.3 mmol, 3.0 equiv) and TEA (1 mg, 0.005 mmol, 0.1 equiv) under nitrogen atmosphere. After stirring for 12 h at 70 °C, the reaction mixture was poured into saturated aqueous NH_4Cl solution and extracted with ethyl acetate (10 mL × 3). The combined organic layers were washed with water, brine, dried over Na_2SO_4, filtered and concentrated in vacuo to give a residue, which was purified by flash column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 10/1) to furnish the desired compound **63** (79%).

Supplementary References

1. Y. Wang, Q. Q. He and R. H. Fan, *Org. Chem. Front*., 2021, **8**, 3004-3007
2. M. Sera, H. Mizufune, T. Ueda, M. Mineno and A. Zanka, *Tetrahedron*, 2017, **73**, 5946-5658.
3. X. M. Zheng and M. A. Kerr, *Org. Lett.*, 2006, **8**, 3777-3779

4. Crystal details of compound 3c

Sample preparation: A solution of compound **3c** (0.1 mmol) in a mixed solvent of dichloromethane (1 mL) and petroleum ethe (2 mL) was placed in a vial (10 mL). The single crystal **3c** was obtained by slowly evaporating solvent at room temperature under the air conditions.

Crystal measurement: X-ray crystal structures of **3c** were determined at 173 K by using D8 VENTURE MetalJet microfocal single crystal diffractometer.

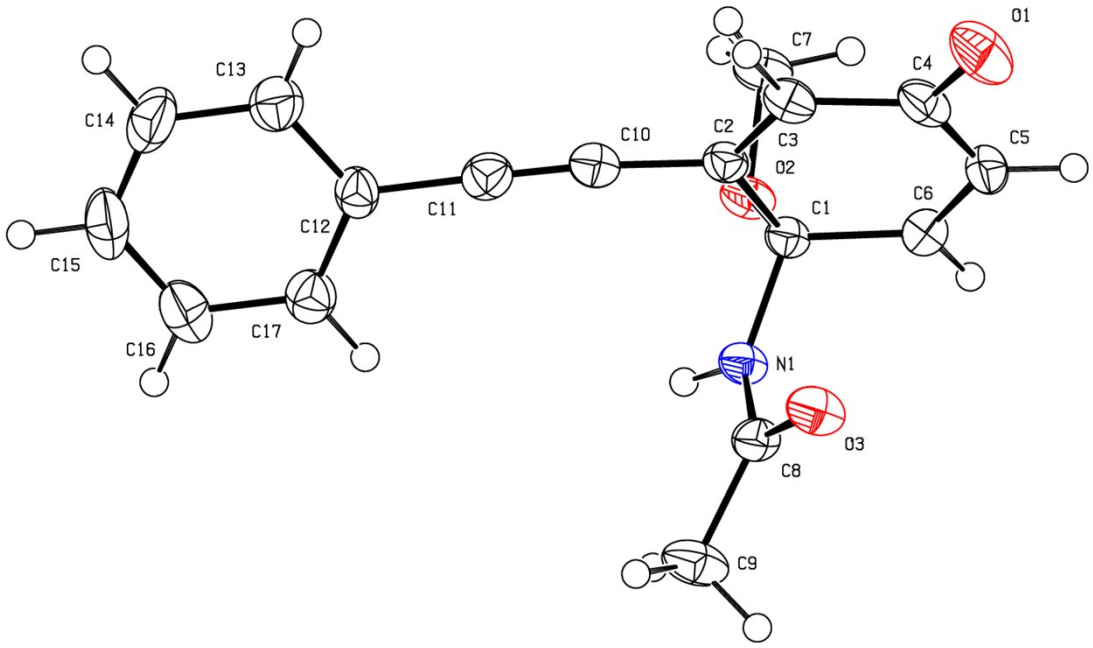


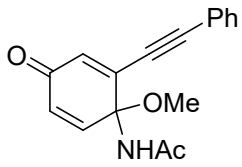
Figure 1. ORTEP diagram of compound 3c (The ellipsoid contour of probability level is 50%)

Table 2. Crystal data and structure refinement for compound 3c

Identification code	3c
Empirical formula	C17 H15 N O3
Formula weight	281.30
Temperature	173(2) K
Wavelength	1.34138 Å
Crystal system	Orthorhombic
Space group	$Pca2_1$
Unit cell dimensions	a = 17.0936(9) Å
	b = 9.1661(5) Å
	c = 9.2363(5) Å
Volume	1447.16(13) $Å^3$

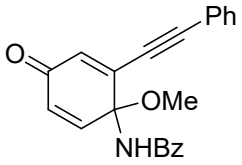
Z	4
Density (calculated)	1.291 Mg/m^3
Absorption coefficient	0.462 mm^{-1}
F(000)	592
Crystal size	0.280 x 0.170 x 0.080 mm^3
Theta range for data collection	4.501 to 57.983°.
Index ranges	-21<=h<=21, -11<=k<=11, -11<=l<=11
Reflections collected	24217
Independent reflections	3063 [R(int) = 0.0568]
Completeness to theta = 53.594°	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.751 and 0.633
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3063 / 1 / 195
Goodness-of-fit on F^2	1.050
Final R indices [I>2sigma(I)]	R^1 = 0.0308, wR^2 = 0.0776
R indices (all data)	R^1 = 0.0325, wR^2 = 0.0794
Absolute structure parameter	-0.07(11)
Extinction coefficient	n/a
Largest diff. peak and hole	0.191 and -0.153 $e.Å^{-3}$

5. Characterization of Products



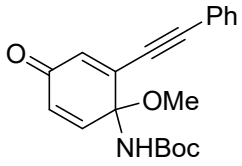
3c

***N*-(1-methoxy-4-oxo-2-(phenylethynyl)cyclohexa-2,5-dien-1-yl)acetamide 3c**: 27.5 mg, 98% yield. Yellow solid, mp:182-183 ℃. ^{1}H NMR (400 MHz, Chloroform-d) δ 7.53-7.51 (m, 2H), 7.42-7.36 (m, 3H), 6.89 (d, *J* = 10.3 Hz, 1H), 6.66 (d, *J* = 1.9 Hz, 1H), 6.45 (dd, *J* = 10.3, 1.9 Hz, 1H), 6.41 (s, 1H), 3.24 (s, 3H), 2.03 (s, 3H); ^{13}C NMR (101 MHz, CDCl3) δ 184.6, 168.9, 144.5, 138.9, 134.6, 132.1, 131.0, 129.9, 128.6, 121.5, 100.9, 84.1, 80.5, 51.0, 23.6; HRMS (m/z): [M+H]+ calcd. for $C_{17}H_{16}NO_3$, 282.1125; found, 282.1125.



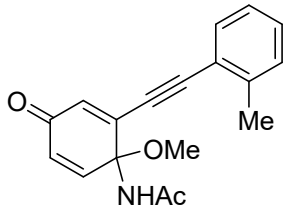
3d

***N*-(1-methoxy-4-oxo-2-(phenylethynyl)cyclohexa-2,5-dien-1-yl)benzamide 3d**: 10.8 mg, 21% yield. White solid, mp:191-192 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.81 (d, *J* = 7.6 Hz, 2H), 7.52-7.50 (m, 1H), 7.45-7.43 (m, 4H), 7.37-7.36 (m, 1H), 7.33 (d, *J* = 7.5 Hz, 2H), 6.95 (d, *J* = 10.3 Hz, 1H), 6.91 (s, 1H), 6.72 (d, *J* = 1.7 Hz, 1H), 6.52 (dd, *J* = 10.2, 1.7 Hz, 1H), 3.31 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 184.6, 165.8, 144.4, 138.9, 134.7, 133.5, 132.2, 132.1, 131.3, 129.8, 128.8, 128.6, 127.1, 121.5, 101.1, 84.1, 81.1, 51.1; HRMS (m/z): $[M+H]^+$ calcd. for $C_{22}H_{18}NO_3$, 344.1281; found, 344.1284.



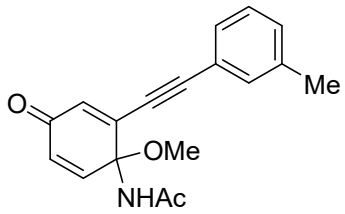
3e

tert-butyl (1-methoxy-4-oxo-2-(phenylethynyl)cyclohexa-2,5-dien-1-yl)carbamate 3e: 12.1 mg, 24% yield. White solid, mp:164-165 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.56-7.54 (m, 2H), 7.41-7.38 (m, 3H), 7.09-7.08 (m, 1H), 6.62 (d, *J* = 2.0 Hz, 1H), 6.42 (dd, *J* = 10.3, 2.0 Hz, 1H), 5.44 (s, 1H), 3.24 (s, 3H), 1.42 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 184.6, 153.0, 144.9, 134.0, 132.2, 130.4, 129.9, 128.6, 121.5, 101.4, 84.0, 81.1, 80.2, 51.3, 28.2; HRMS (m/z): $[M+H]^+$ calcd. for $C_{20}H_{22}NO_4$, 340.1543; found, 340.1539.



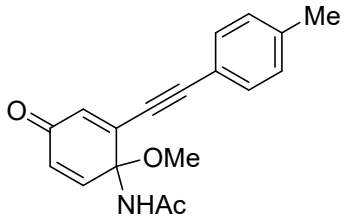
4

***N*-(1-methoxy-4-oxo-2-(*o*-tolylethynyl)cyclohexa-2,5-dien-1-yl)acetamide 4**: 26.8 mg, 88% yield. Yellow solid, mp:132-133 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.48 (d, *J* = 7.5 Hz, 1H), 7.31 (t, *J* = 7.3 Hz, 1H), 7.25 (d, *J* = 7.4 Hz, 1H), 7.20 (t, *J* = 7.4 Hz, 1H), 6.89 (d, *J* = 10.3 Hz, 1H), 6.65 (d, *J* = 1.7 Hz, 1H), 6.44 (dd, *J* = 10.3, 1.7 Hz, 1H), 6.40 (s, 1H), 3.25 (s, 3H), 2.48 (s, 3H), 2.01 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 184.6, 168.7, 144.5, 140.9, 139.1, 134.2, 132.4, 131.0, 130.0, 129.8, 125.9, 121.4, 100.1, 88.0, 80.6, 51.0, 23.6, 20.7; HRMS (m/z): $[M+H]^+$ calcd. for $C_{18}H_{18}NO_3$, 296.1281; found, 296.1282.



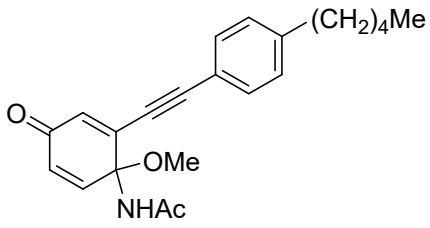
5

***N*-(1-methoxy-4-oxo-2-(*m*-tolylethynyl)cyclohexa-2,5-dien-1-yl)acetamide 5**: 25.4 mg, 87% yield. Yellow solid, mp:134-135 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.33-7.31 (m, 2H), 7.26 (t, *J* = 7.5 Hz, 1H), 7.21 (d, *J* = 7.5 Hz, 1H), 6.88 (d, *J* = 10.3 Hz, 1H), 6.64 (d, *J* = 1.9 Hz, 1H), 6.57 (s, 1H), 6.43 (dd, *J* = 10.3, 1.9 Hz, 1H), 3.23 (s, 3H), 2.35 (s, 3H), 2.02 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 184.7, 169.0, 144.7, 139.2, 138.4, 134.4, 132.6, 130.9, 130.8, 129.3, 128.5, 121.4, 101.3, 83.8, 80.6, 51.0, 23.6, 21.2; HRMS (m/z): $[M+H]^+$ calcd. for $C_{18}H_{18}NO_3$, 296.1281; found, 295.1279.



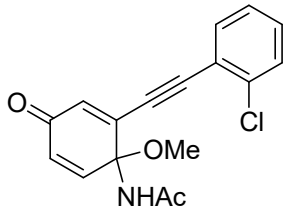
6

***N*-(1-methoxy-4-oxo-2-(*p*-tolylethynyl)cyclohexa-2,5-dien-1-yl)acetamide 6**: 27.1 mg, 91% yield. Yellow solid, mp:148-149 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.41 (d, *J* = 7.9 Hz, 2H), 7.18 (d, *J* = 7.9 Hz, 2H), 6.89 (d, *J* = 10.3 Hz, 1H), 6.63 (d, *J* = 2.1 Hz, 1H), 6.52 (s, 1H), 6.43 (dd, *J* = 10.3, 2.1 Hz, 1H), 3.23 (s, 3H), 2.38 (s, 3H), 2.02 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 184.7, 169.0, 144.6, 140.4, 139.2, 134.2, 132.1, 130.9, 129.4, 118.5, 101.5, 83.7, 80.5, 51.0, 23.6, 21.7; HRMS (m/z): $[M+H]^+$ calcd. for $C_{18}H_{18}NO_3$, 296.1281; found, 295.1277.



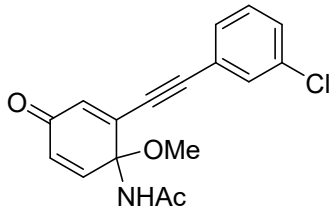
7

***N*-(1-methoxy-4-oxo-2-((4-pentylphenyl)ethynyl)cyclohexa-2,5-dien-1-yl)acetamide 7**: 32.4 mg, 92% yield. Yellow solid, mp:170-171 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.43 (d, *J* = 8.2 Hz, 2H), 7.19 (d, *J* = 8.1 Hz, 2H), 6.92 (d, *J* = 10.3 Hz, 1H), 6.64 (d, *J* = 2.1 Hz, 1H), 6.44 (dd, *J* = 10.3, 2.1 Hz, 1H), 6.37 (s, 1H), 3.24 (s, 3H), 2.62 (t, *J* = 7.7 Hz, 2H), 2.02 (s, 3H), 1.61 (p, *J* = 7.6 Hz, 2H), 1.35-1.29 (m, 4H), 0.89 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 184.6, 168.8, 145.5, 144.4, 139.1, 134.2, 132.1, 130.9, 128.8, 118.7, 101.6, 83.7, 80.5, 51.0, 36.0, 31.4, 30.8, 23.7, 22.5, 14.0; HRMS (m/z): $[M+H]^+$ calcd. for $C_{22}H_{26}NO_3$, 352.1907; found, 352.1907.



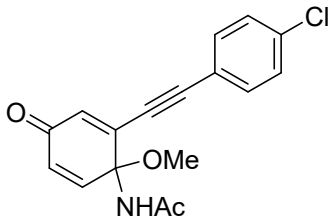
8

***N*-(2-((2-chlorophenyl)ethynyl)-1-methoxy-4-oxocyclohexa-2,5-dien-1-yl)acetamide 8**: 28.9 mg, 97% yield. Yellow solid, mp:178-179 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.55 (d, *J* = 7.2 Hz, 1H), 7.46 (d, *J* = 7.3 Hz, 1H), 7.36 (t, *J* = 7.2 Hz, 1H), 7.29 (t, *J* = 7.3 Hz, 1H), 6.92 (d, *J* = 10.3 Hz, 1H), 6.68 (s, 1H), 6.46-6.44 (m, 2H), 3.25 (s, 3H), 2.03 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 184.5, 168.9, 144.6, 138.5, 136.5, 134.7, 133.7, 130.9, 130.7, 129.5, 126.9, 121.6, 97.2, 88.9, 80.5, 51.0, 23.7; HRMS (m/z): $[M+H]^+$ calcd. for $C_{17}H_{15}ClNO_3$, 316.0735; found, 316.0731.



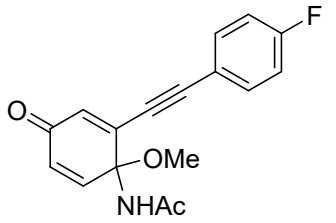
9

***N*-(2-((3-chlorophenyl)ethynyl)-1-methoxy-4-oxocyclohexa-2,5-dien-1-yl)acetamide 9**: 26.8 mg, 91% yield. Yellow solid, mp:189-190 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.50 (s, 1H), 7.41-7.38 (m, 2H), 7.33 (d, *J* = 7.8 Hz, 1H), 6.84 (d, *J* = 10.3 Hz, 1H), 6.67 (d, *J* = 2.0 Hz, 1H), 6.45 (dd, *J* = 10.3, 2.0 Hz, 1H), 6.31 (s, 1H), 3.24 (s, 3H), 2.03 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 184.4, 168.7, 144.6, 138.5, 135.2, 134.5, 131.9, 131.1, 130.2, 130.1, 129.9, 123.3, 98.8, 85.0, 80.5, 51.0, 23.6; HRMS (m/z): $[M+H]^+$ calcd. for $C_{17}H_{15}ClNO_3$, 316.0735; found, 316.0736.



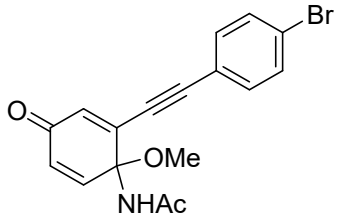
10

***N*-(2-((4-chlorophenyl)ethynyl)-1-methoxy-4-oxocyclohexa-2,5-dien-1-yl)acetamide 10**: 29.5 mg, 98% yield. Yellow solid, mp:176-177 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.45 (d, J = 8.6 Hz, 2H), 7.36 (d, J = 8.6 Hz, 2H), 6.85 (d, J = 10.3 Hz, 1H), 6.66 (d, J = 2.0 Hz, 1H), 6.45 (dd, J = 10.3, 2.0 Hz, 1H), 6.26 (s, 1H), 3.24 (s, 3H), 2.02 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 184.4, 168.7, 144.4, 138.6, 136.1, 134.9, 133.3, 131.1, 129.0, 120.0, 99.4, 84.9, 80.5, 51.0, 23.6; HRMS (m/z): $[M+H]^+$ calcd. for $C_{17}H_{15}ClNO_3$, 316.0735; found, 316.0734.



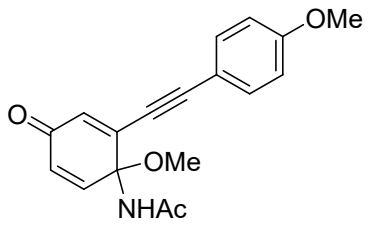
11

***N*-(2-((4-fluorophenyl)ethynyl)-1-methoxy-4-oxocyclohexa-2,5-dien-1-yl)acetamide 11**: 28.2 mg, 97% yield. Yellow solid, mp:182-183 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.51 (dd, *J* = 8.6, 5.5 Hz, 2H), 7.08 (t, *J* = 8.6 Hz, 2H), 6.86 (d, *J* = 10.3 Hz, 1H), 6.65 (d, *J* = 2.0 Hz, 1H), 6.46-6.43 (m, 2H), 3.24 (s, 3H), 2.02 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 184.5, 168.8, 163.4 (d, *J* = 252.7 Hz), 144.5, 138.8, 134.6, 134.3 (d, *J* = 8.7 Hz), 131.0, 117.7 (d, *J* = 3.7 Hz), 116.1 (d, *J* = 22.3 Hz), 99.7, 83.9, 80.5, 51.0, 23.6; HRMS (m/z): $[M+H]^+$ calcd. for $C_{17}H_{15}FNO_3$, 300.1030; found, 300.1032.



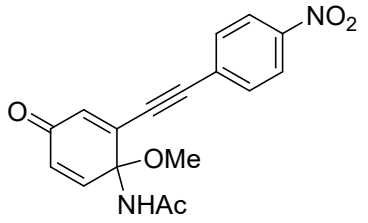
12

***N*-(2-((4-bromophenyl)ethynyl)-1-methoxy-4-oxocyclohexa-2,5-dien-1-yl)acetamide 12**: 34.5 mg, 91% yield. Yellow solid, mp:147-148 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.52 (d, *J* = 8.4 Hz, 2H), 7.38 (d, *J* = 8.4 Hz, 2H), 6.85 (d, *J* = 10.3 Hz, 1H), 6.66 (d, *J* = 1.9 Hz, 1H), 6.45 (dd, *J* = 10.3, 1.9 Hz, 1H), 6.24 (s, 1H), 3.24 (s, 3H), 2.02 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 184.4, 168.6, 144.4, 138.6, 134.9, 133.5, 132.0, 131.1, 124.4, 120.5, 99.4, 85.1, 80.5, 51.1, 23.6; HRMS (m/z): $[M+H]^+$ calcd. for $C_{17}H_{15}BrNO_3$, 360.0230; found, 360.0229.



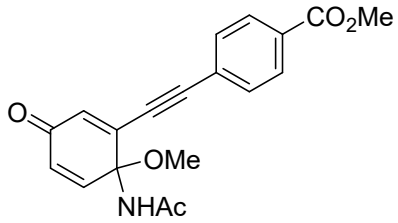
13

***N*-(1-methoxy-2-((4-methoxyphenyl)ethynyl)-4-oxocyclohexa-2,5-dien-1-yl)acetamide 13**: 23.6 mg, 85% yield. Yellow solid, mp:165-166 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.47 (d, *J* = 8.7 Hz, 2H), 6.95 (d, *J* = 10.3 Hz, 1H), 6.90 (d, *J* = 8.7 Hz, 2H), 6.62 (d, *J* = 2.0 Hz, 1H), 6.43 (dd, *J* = 10.3, 2.0 Hz, 1H), 6.28 (s, 1H), 3.85 (s, 3H), 3.24 (s, 3H), 2.02 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 184.6, 168.8, 160.9, 144.3, 139.2, 133.9, 133.8, 130.9, 114.3, 113.5, 101.7, 83.3, 80.5, 55.4, 51.1, 23.7; HRMS (m/z): $[M+H]^+$ calcd. for $C_{18}H_{18}NO_4$, 312.1230; found, 312.1233.



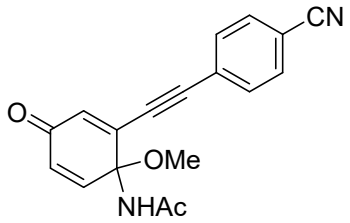
14

***N*-(1-methoxy-2-((4-nitrophenyl)ethynyl)-4-oxocyclohexa-2,5-dien-1-yl)acetamide 14**: 17.4 mg, 58% yield. Yellow solid, mp:161-162 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.25 (d, *J* = 8.8 Hz, 2H), 7.67 (d, *J* = 8.8 Hz, 2H), 6.78 (d, *J* = 10.3 Hz, 1H), 6.72 (d, *J* = 2.1 Hz, 1H), 6.48 (dd, *J* = 10.3, 2.1 Hz, 1H), 6.14 (s, 1H), 3.27 (s, 3H), 2.03 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 184.1, 168.5, 144.5, 137.4, 136.0, 134.9, 132.9, 131.3, 128.2, 123.8, 97.3, 88.3, 80.5, 51.1, 23.6; HRMS (m/z): $[M+H]^+$ calcd. for $C_{17}H_{15}N_2O_5$, 327.0975; found, 327.0971.



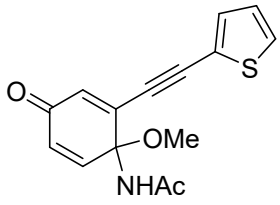
15

methyl 4-((6-acetamido-6-methoxy-3-oxocyclohexa-1,4-dien-1-yl)ethynyl)benzoate 15: 20.7 mg, 60% yield. White solid, mp:139-140 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.04 (d, *J* = 8.4 Hz, 2H), 7.57 (d, *J* = 8.3 Hz, 2H), 6.83 (d, *J* = 10.3 Hz, 1H), 6.69 (d, *J* = 1.9 Hz, 1H), 6.46 (dd, *J* = 10.3, 1.9 Hz, 1H), 6.34 (s, 1H), 3.94 (s, 3H), 3.25 (s, 3H), 2.03 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 184.4, 168.7, 166.2, 144.5, 138.4, 135.3, 132.0, 131.1, 130.9, 129.7, 126.1, 99.3, 86.4, 80.5, 52.4, 51.0, 23.6; HRMS (m/z): $[M+H]^+$ calcd. for $C_{19}H_{18}NO_5$, 340.1179; found, 340.1181.



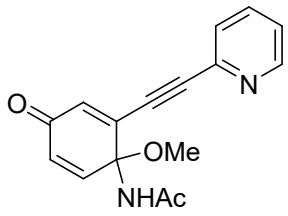
16

***N*-(2-((4-cyanophenyl)ethynyl)-1-methoxy-4-oxocyclohexa-2,5-dien-1-yl)acetamide 16**: 20.9 mg, 67% yield. White solid, mp:172-173 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.68 (d, *J* = 8.2 Hz, 2H), 7.61 (d, *J* = 8.1 Hz, 2H), 6.79 (d, *J* = 10.3 Hz, 1H), 6.70 (d, *J* = 2.0 Hz, 1H), 6.48 (dd, *J* = 10.3, 2.0 Hz, 1H), 6.17 (s, 1H), 3.25 (s, 3H), 2.02 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 184.1, 168.5, 144.4, 137.9, 135.9, 132.6, 132.2, 131.3, 126.3, 118.0, 113.1, 97.7, 87.6, 80.5, 51.1, 23.6; HRMS (m/z): $[M+H]^+$ calcd. for $C_{18}H_{15}N_2O_3$, 307.1077; found, 307.1076.



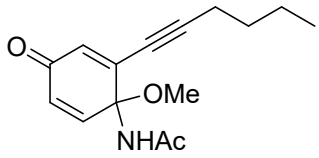
17

***N*-(1-methoxy-4-oxo-2-(thiophen-2-ylethynyl)cyclohexa-2,5-dien-1-yl)acetamide 17**: 18.3 mg, 64% yield. Yellow solid, mp: 138-139 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.43 (d, *J* = 4.9 Hz, 1H), 7.37 (d, *J* = 3.0 Hz, 1H), 7.08-7.04 (m, 1H), 6.87 (d, *J* = 10.3 Hz, 1H), 6.64 (d, *J* = 2.0 Hz, 1H), 6.45 (dd, *J* = 10.3, 2.0 Hz, 1H), 6.25 (s, 1H), 3.24 (s, 3H), 2.03 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 184.4, 168.7, 144.3, 138.6, 134.2, 134.1, 131.1, 129.8, 127.6, 121.4, 94.2, 88.2, 80.5, 51.1, 23.6; HRMS (m/z): $[M+H]^+$ calcd. for $C_{15}H_{14}NO_3S$, 288.0689; found, 288.0691.



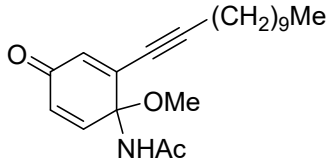
18

***N*-(1-methoxy-4-oxo-2-(pyridin-2-ylethynyl)cyclohexa-2,5-dien-1-yl)acetamide 18**: 23.8 mg, 85% yield. Black solid, mp:154-155 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.65 (ddd, *J* = 4.9, 1.8, 1.0 Hz, 1H), 7.74 (td, *J* = 7.8, 1.7 Hz, 1H), 7.55 (dt, *J* = 7.8, 1.1 Hz, 1H), 7.33 (ddd, *J* = 7.6, 4.9, 1.0 Hz, 1H), 6.85 (d, *J* = 10.3 Hz, 1H), 6.73 (d, *J* = 2.0 Hz, 1H), 6.70 (s, 1H), 6.46 (dd, *J* = 10.3, 2.0 Hz, 1H), 3.26 (s, 3H), 2.03 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 184.4, 169.0, 150.3, 144.9, 141.9, 138.2, 136.6, 135.7, 130.8, 128.0, 124.1, 98.5, 83.4, 80.6, 51.0, 23.5. HRMS (m/z): $[M+H]^+$ calcd. for $C_{16}H_{15}N_2O_3$, 283.1077; found, 283.1074.



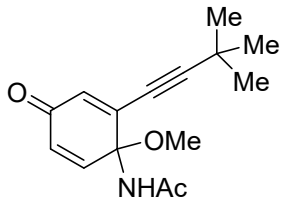
19

***N*-(2-(hex-1-yn-1-yl)-1-methoxy-4-oxocyclohexa-2,5-dien-1-yl)acetamide 19**: 26.1 mg, 98% yield. White solid, mp:169-170 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 6.93 (d, *J* = 10.3 Hz, 1H), 6.50 (d, *J* = 2.0 Hz, 1H), 6.40 (dd, *J* = 10.3, 2.0 Hz, 1H), 6.24 (s, 1H), 3.19 (s, 3H), 2.46 (t, *J* = 7.0 Hz, 2H), 2.00 (s, 3H), 1.59 (p, *J* = 7.0 Hz, 3H), 1.51-1.40 (m, 2H), 0.95 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 184.8, 168.7, 144.3, 139.5, 134.2, 130.7, 103.7, 80.4, 75.9, 50.9, 30.3, 23.7, 22.0, 19.6, 13.5; HRMS (m/z): $[M+H]^+$ calcd. for $C_{15}H_{20}NO_3$, 262.1438; found, 262.1439.



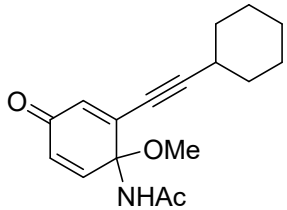
20

***N*-(2-(dodec-1-yn-1-yl)-1-methoxy-4-oxocyclohexa-2,5-dien-1-yl)acetamide 20**: 28.9 mg, 83% yield. Brown oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 6.93 (d, *J* = 10.2 Hz, 1H), 6.50 (s, 1H), 6.39 (d, *J* = 10.3 Hz, 1H), 6.26 (s, 1H), 3.19 (s, 3H), 2.44 (t, *J* = 6.8 Hz, 2H), 2.00 (s, 3H), 1.60 (p, *J* = 6.7 Hz, 2H), 1.42 (p, *J* = 6.9 Hz, 2H), 1.33-1.27 (m, 12H), 0.88 (t, *J* = 5.9 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 184.8, 168.7, 144.3, 139.5, 134.2, 130.7, 103.8, 80.4, 75.8, 51.0, 31.9, 29.6, 29.5, 29.3, 29.1, 28.9, 28.3, 23.7, 22.7, 19.9, 14.1; HRMS (m/z): $[M+H]^+$ calcd. for $C_{21}H_{32}NO_3$, 346.2377; found, 346.2374.



21

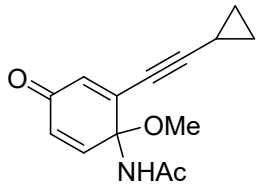
***N*-(2-(3,3-dimethylbut-1-yn-1-yl)-1-methoxy-4-oxocyclohexa-2,5-dien-1-yl)acetamide 21**: 25.7 mg, 98% yield. White solid, mp:170-171 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 6.90 (d, *J* = 10.3 Hz, 1H), 6.48 (d, *J* = 2.0 Hz, 1H), 6.39 (dd, *J* = 10.3, 2.0 Hz, 1H), 6.23 (s, 1H), 3.17 (s, 3H), 2.00 (s, 3H), 1.31 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 184.9, 168.7, 144.4, 139.5, 133.9, 130.7, 111.6, 80.3, 74.4, 50.8, 30.6, 28.5, 23.6; HRMS (m/z): $[M+H]^+$ calcd. for $C_{15}H_{20}NO_3$, 262.1438; found, 262.1436.



22

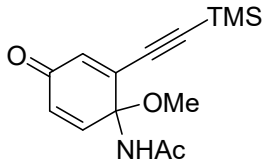
***N*-(2-(cyclohexylethynyl)-1-methoxy-4-oxocyclohexa-2,5-dien-1-yl)acetamide 22**: 26.7 mg, 97% yield. White solid, mp:166-167 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 6.93 (d, *J* = 10.3 Hz, 1H), 6.49 (d, *J* = 2.0 Hz, 1H), 6.39 (dd, *J* = 10.3, 2.0 Hz, 1H), 6.25 (s, 1H), 3.18 (s, 3H), 2.66 (tt, *J* = 8.8, 3.9 Hz,

1H), 2.00 (s, 3H), 1.89-1.80 (m, 2H), 1.77-1.66 (m, 2H), 1.60-1.47 (m, 3H), 1.43-1.34 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 184.9, 168.8, 144.4, 139.6, 133.9, 130.7, 107.7, 80.4, 76.0, 50.9, 32.1, 29.9, 25.7, 24.6, 23.6; HRMS (m/z): $[M+H]^+$ calcd. for $C_{17}H_{22}NO_3$, 288.1594; found, 288.1594.



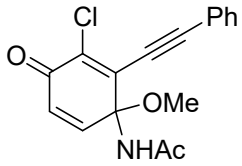
23

***N*-(2-(cyclopropylethynyl)-1-methoxy-4-oxocyclohexa-2,5-dien-1-yl)acetamide 23**: 24.1 mg, 99% yield. White solid, mp:152-153 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 6.92 (d, *J* = 10.3 Hz, 1H), 6.46 (d, *J* = 2.0 Hz, 1H), 6.39 (dd, *J* = 10.3, 2.0 Hz, 1H), 6.29 (s, 1H), 3.18 (s, 3H), 2.01 (s, 3H), 1.49 (tt, *J* = 8.2, 5.1 Hz, 1H), 0.98 (dt, *J* = 8.2, 3.3 Hz, 2H), 0.89-0.83 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 184.8, 168.8, 144.3, 139.6, 133.8, 130.7, 107.3, 80.4, 71.1, 50.9, 23.7, 9.7, 0.8; HRMS (m/z): $[M+H]^+$ calcd. for $C_{14}H_{16}NO_3$, 246.1125; found, 246.1122.



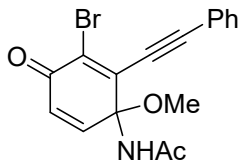
24

***N*-(1-methoxy-4-oxo-2-((trimethylsilyl)ethynyl)cyclohexa-2,5-dien-1-yl)acetamide 24**: 19.8 mg, 65% yield. White solid, mp:185-186 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 6.88 (d, *J* = 10.3 Hz, 1H), 6.56 (d, *J* = 2.0 Hz, 1H), 6.41 (dd, *J* = 10.3, 2.0 Hz, 1H), 6.11 (s, 1H), 3.19 (s, 3H), 2.00 (s, 3H), 0.25 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 184.6, 168.7, 144.4, 138.4, 135.2, 130.8, 108.1, 98.8, 80.2, 51.0, 23.6, -0.4; HRMS (m/z): $[M+H]^+$ calcd. for $C_{14}H_{20}NO_3Si$, 278.1207; found, 278.1207.



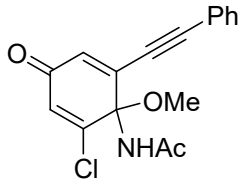
26

***N*-(3-chloro-1-methoxy-4-oxo-2-(phenylethynyl)cyclohexa-2,5-dien-1-yl)acetamide 26**: 17.1 mg, 55% yield. White solid, mp:174-175 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.59-7.57 (m, 2H), 7.45-7.38 (m, 3H), 6.90 (d, *J* = 10.2 Hz, 1H), 6.55 (d, *J* = 10.2 Hz, 1H), 6.35 (s, 1H), 3.25 (s, 3H), 2.02 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 177.5, 168.7, 144.8, 137.7, 135.7, 132.3, 130.3, 129.8, 128.7, 128.7, 121.5, 107.3, 82.8, 82.3, 51.2, 23.6; HRMS (m/z): $[M]^+$ calcd. for $C_{17}H_{15}ClNO_3$, 316.0735; found, 316.0730.



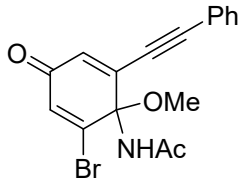
27

***N*-(3-bromo-1-methoxy-4-oxo-2-(phenylethynyl)cyclohexa-2,5-dien-1-yl)acetamide 27**: 24.2 mg, 67% yield. White solid, mp:184-185 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.60-7.58 (m, 2H), 7.47-7.38 (m, 3H), 6.91 (d, *J* = 10.2 Hz, 1H), 6.57 (d, *J* = 10.2 Hz, 1H), 6.30 (s, 1H), 3.26 (s, 3H), 2.02 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 177.5, 168.6, 144.6, 139.4, 132.3, 131.4, 130.3, 129.4, 128.7, 121.5, 106.6, 85.0, 82.4, 51.2, 23.6; HRMS (m/z): $[M+H]^+$ calcd. for $C_{17}H_{15}BrNO_3$, 360.0230; found, 360.0233.



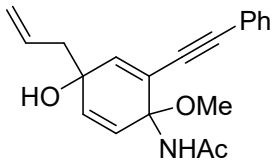
28

***N*-(2-chloro-1-methoxy-4-oxo-6-(phenylethynyl)cyclohexa-2,5-dien-1-yl)acetamide 28**: 19.1 mg, 61% yield. White solid, mp:188-189 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.54-7.51 (m, 2H), 7.44-7.37 (m, 3H), 7.08 (s, 1H), 6.74 (s, 1H), 6.21 (s, 1H), 3.28 (s, 3H), 2.03 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 177.6, 168.7, 140.7, 139.5, 134.8, 133.3, 132.2, 130.1, 128.7, 121.3, 102.2, 83.5, 82.1, 51.3, 23.6; HRMS (m/z): $[M]^+$ calcd. for $C_{17}H_{15}ClNO_3$, 316.0735; found, 316.0732.



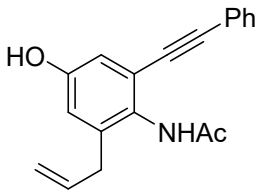
29

***N*-(2-bromo-1-methoxy-4-oxo-6-(phenylethynyl)cyclohexa-2,5-dien-1-yl)acetamide 29**: 18.5 mg, 53% yield. White solid, mp:171-172 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.53-7.51 (m, 2H), 7.44-7.37 (m, 3H), 7.34 (s, 1H), 6.76 (s, 1H), 6.20 (s, 1H), 3.29 (s, 3H), 2.03 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 177.5, 168.6, 145.1, 139.4, 132.8, 132.2, 130.1, 128.7, 126.4, 121.3, 102.1, 83.6, 82.4, 51.4, 23.6; HRMS (m/z): $[M+H]^+$ calcd. for $C_{17}H_{15}BrNO_3$, 360.0230; found, 360.0232.



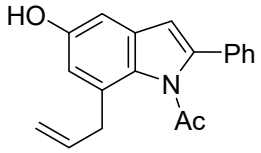
30

***N*-(4-allyl-4-hydroxy-1-methoxy-2-(phenylethynyl)cyclohexa-2,5-dien-1-yl)acetamide 30**: 28.7 mg, 94% yield. Colorless oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.47-7.45 (m, 2H), 7.33-7.31 (m, 3H), 6.58 (d, *J* = 2.2 Hz, 1H), 6.24 (s, 1H), 6.23 (dd, *J* = 10.0, 2.1 Hz, 1H), 5.73 (ddt, *J* = 17.3, 10.1, 7.2 Hz, 1H), 5.59 (d, *J* = 10.0 Hz, 1H), 5.13 (dd, *J* = 17.3, 1.8 Hz, 1H), 5.10-5.06 (m, 1H), 4.57 (s, 1H), 3.18 (s, 3H), 2.49 (d, *J* = 7.3 Hz, 2H), 1.94 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.3, 144.5, 138.2, 132.3, 131.8, 128.5, 128.3, 127.5, 122.9, 122.1, 118.7, 90.8, 85.1, 80.9, 66.6, 50.4, 44.2, 24.0; HRMS (m/z): $[M+H]^+$ calcd. for $C_{20}H_{22}NO_3$, 324.1594; found, 324.1596.



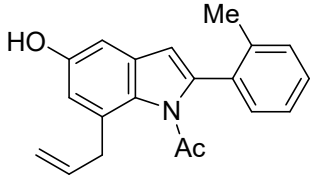
31

***N*-(2-allyl-4-hydroxy-6-(phenylethynyl)phenyl)acetamide 31**: 19.1 mg, 78% yield. Yellow solid, mp:171-172 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.02 (s, 1H), 7.77 (s, 1H), 7.49-7.47 (m, 2H), 7.36-7.34 (m, 3H), 6.95 (s, 1H), 6.58 (s, 1H), 5.98 (ddt, *J* = 16.8, 10.2, 6.4 Hz, 1H), 5.14-5.09 (m, 2H), 3.38 (d, *J* = 6.6 Hz, 2H), 2.22 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.5, 150.7, 135.9, 131.9, 131.5, 128.8, 128.6, 128.4, 128.1, 122.5, 118.1, 116.6, 112.1, 95.6, 84.5, 35.1, 24.7; HRMS (m/z): $[M+H]^+$ calcd. for $C_{19}H_{18}NO_2$, 292.1332; found, 292.1333.



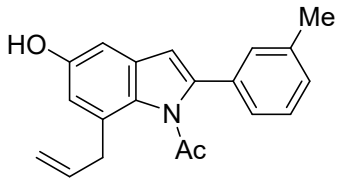
32

1-(7-allyl-5-hydroxy-2-phenyl-1*H*-indol-1-yl)ethan-1-one 32: 18.5 mg, 75% yield. White solid, mp:186-187 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.19 (s, 1H), 7.46-7.41 (m, 5H), 6.98 (s, 1H), 6.50 (s, 1H), 6.08 (ddt, *J* = 16.6, 9.6, 6.3 Hz, 1H), 5.22-5.19 (m, 1H), 5.18-5.16 (m, 1H), 5.07 (s, 1H), 3.55 (d, *J* = 6.3 Hz, 2H), 2.04 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.3, 151.1, 140.0, 136.8, 134.3, 132.9, 129.0, 128.8, 128.7, 128.6, 123.9, 117.7, 116.5, 111.3, 106.1, 36.2, 27.8; HRMS (m/z): $[M+H]^+$ calcd. for $C_{19}H_{18}NO_2$, 292.1332; found, 292.1332.



33

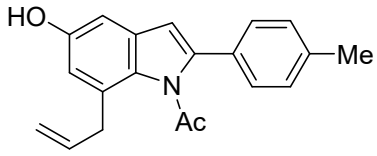
1-(7-allyl-5-hydroxy-2-(*o*-tolyl)-1*H*-indol-1-yl)ethan-1-one 33: 8.5 mg, 66% yield. Colorless oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.28 (s, 1H), 7.36-7.27 (m, 4H), 6.98 (s, 1H), 6.40 (s, 1H), 6.09 (ddt, *J* = 17.2, 10.0, 6.3 Hz, 1H), 5.21 (dd, *J* = 17.1, 1.7 Hz, 1H), 5.18 (dd, *J* = 10.0, 1.6 Hz, 1H), 5.01 (s, 1H), 3.56 (d, *J* = 6.3 Hz, 2H), 2.21 (s, 3H), 1.90 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.7, 151.1, 138.8, 137.5, 136.8, 134.1, 132.3, 130.4, 130.4, 129.2, 129.0, 126.1, 123.6, 118.4, 116.5, 111.0, 106.0, 36.2, 26.4, 20.0; HRMS (m/z): $[M+H]^+$ calcd. for $C_{20}H_{20}NO_2$, 306.1489; found, 306.1489.



34

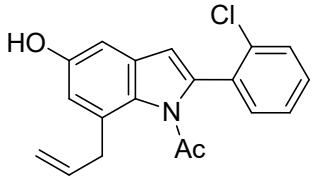
1-(7-allyl-5-hydroxy-2-(*m*-tolyl)-1*H*-indol-1-yl)ethan-1-one 34: 8.1 mg, 63% yield. Yellow oil. ^{1}H

NMR (400 MHz, Chloroform-*d*) δ 8.18 (s, 1H), 7.33 (t, *J* = 7.5 Hz, 1H), 7.27-7.21 (m, 3H), 6.97 (s, 1H), 6.47 (s, 1H), 6.08 (ddt, *J* = 17.2, 10.1, 6.3 Hz, 1H), 5.19 (dd, *J* = 17.1, 1.7 Hz, 1H), 5.17 (dd, *J* = 10.0, 1.6 Hz, 1H), 5.09 (s, 1H), 3.55 (d, *J* = 6.3 Hz, 2H), 2.41 (s, 3H), 2.05 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.4, 151.1, 140.2, 138.5, 136.8, 134.2, 132.8, 129.6, 129.3, 128.8, 128.6, 126.1, 123.8, 117.7, 116.4, 111.1, 106.0, 36.1, 27.8, 21.4; HRMS (m/z): $[M+H]^+$ calcd. for $C_{20}H_{20}NO_2$, 306.1489; found, 306.1490.



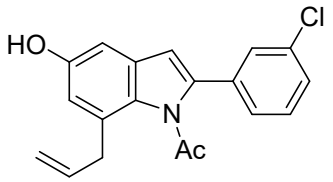
35

1-(7-allyl-5-hydroxy-2-(*p*-tolyl)-1*H*-indol-1-yl)ethan-1-one 35: 9.7 mg, 74% yield. Yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.18 (s, 1H), 7.33 (d, *J* = 8.0 Hz, 2H), 7.25 (d, *J* = 8.0 Hz, 2H), 6.96 (s, 1H), 6.46 (s, 1H), 6.08 (ddt, *J* = 16.6, 10.1, 6.3 Hz, 1H), 5.03 (s, 1H), 5.19 (dd, *J* = 16.7, 1.6 Hz, 1H), 5.17 (dd, *J* = 10.1, 1.7 Hz, 1H), 3.55 (d, *J* = 6.2 Hz, 2H), 2.42 (s, 3H), 2.04 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.4, 151.0, 140.2, 138.6, 136.8, 132.8, 131.3, 129.4, 128.9, 128.8, 123.6, 117.7, 116.4, 110.9, 106.0, 36.2, 27.8, 21.3; HRMS (m/z): $[M+H]^+$ calcd. for $C_{20}H_{20}NO_2$, 306.1489; found, 306.1487.



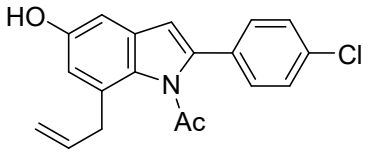
36

1-(7-allyl-2-(2-chlorophenyl)-5-hydroxy-1*H*-indol-1-yl)ethan-1-one 36: 9.8 mg, 68% yield. Yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.25 (s, 1H), 7.50-7.45 (m, 2H), 7.42-7.35 (m, 2H), 6.99 (s, 1H), 6.48 (s, 1H), 6.09 (ddt, *J* = 17.2, 10.1, 6.3 Hz, 1H), 5.24-5.17 (m, 2H), 3.56 (d, *J* = 6.3 Hz, 2H), 2.05 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.2, 151.1, 136.7, 136.3, 134.7, 133.8, 132.3, 131.7, 130.4, 129.9, 128.7, 127.1, 124.2, 118.2, 116.5, 112.1, 106.2, 36.2, 26.1; HRMS (m/z): $[M+H]^+$ calcd. for $C_{19}H_{17}ClNO_2$, 326.0942; found, 326.0941.



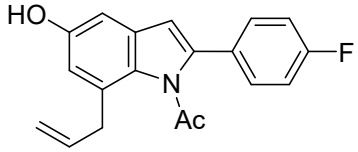
37

1-(7-allyl-2-(3-chlorophenyl)-5-hydroxy-1*H*-indol-1-yl)ethan-1-one 37: 9.4 mg, 66% yield. Yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.15 (s, 1H), 7.47 (s, 1H), 7.39-7.38 (m, 2H), 7.33-7.31 (m, 1H), 6.98 (s, 1H), 6.52 (s, 1H), 6.08 (ddt, *J* = 16.6, 10.2, 6.3 Hz, 1H), 5.22-5.16 (m, 2H), 3.55 (d, *J* = 6.2 Hz, 2H), 2.10 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.8, 151.2, 138.4, 136.6, 136.0, 134.7, 132.9, 129.9, 128.8, 128.6, 128.5, 127.1, 124.4, 117.6, 116.5, 112.0, 106.2, 36.1, 27.9; HRMS (m/z): $[M+H]^+$ calcd. for $C_{19}H_{17}ClNO_2$, 326.0942; found, 326.0940.



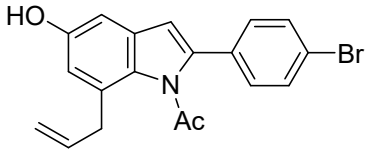
38

1-(7-allyl-2-(4-chlorophenyl)-5-hydroxy-1*H*-indol-1-yl)ethan-1-one 38: 10.5 mg, 70% yield. Yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.15 (s, 1H), 7.43 (d, *J* = 8.5 Hz, 2H), 7.37 (d, *J* = 8.6 Hz, 2H), 6.97 (s, 1H), 6.49 (s, 1H), 6.07 (ddt, *J* = 16.5, 10.2, 6.3 Hz, 1H), 5.21-5.16 (m, 2H), 5.10 (s, 1H), 3.54 (d, *J* = 6.2 Hz, 2H), 2.08 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.9, 151.2, 138.7, 136.7, 134.7, 132.9, 132.7, 130.1, 129.0, 128.6, 124.3, 117.6, 116.5, 111.7, 106.1, 36.1, 27.9; HRMS (m/z): $[M+H]^+$ calcd. for $C_{19}H_{17}ClNO_2$, 326.0942; found, 326.0942.



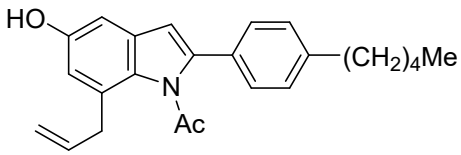
39

1-(7-allyl-2-(4-fluorophenyl)-5-hydroxy-1*H*-indol-1-yl)ethan-1-one 39: 7.9 mg, 62% yield. Yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.16 (s, 1H), 7.44-7.40 (m, 2H), 7.17-7.13 (m, 2H), 6.97 (s, 1H), 6.48 (s, 1H), 6.08 (ddt, *J* = 16.5, 10.2, 6.3 Hz, 1H), 5.22-5.16 (m, 2H), 3.55 (d, *J* = 6.2 Hz, 2H), 2.05 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.9, 162.9 (d, *J* = 249.1 Hz), 151.1, 138.8, 136.7, 132.8, 130.7 (d, *J* = 8.1 Hz), 130.4 (d, *J* = 3.6 Hz), 128.7, 124.0, 117.7, 116.5, 115.9 (d, *J* = 22.3 Hz), 111.4, 106.1, 36.2, 27.8; HRMS (m/z): $[M+H]^+$ calcd. for $C_{19}H_{17}FNO_2$, 310.1238; found, 310.1240.



40

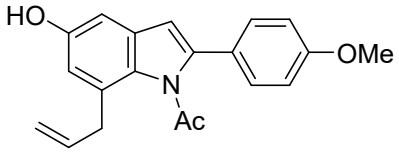
1-(7-allyl-2-(4-bromophenyl)-5-hydroxy-1*H*-indol-1-yl)ethan-1-one 40: 9.5 mg, 60% yield. White solid, mp:174-175 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.14 (s, 1H), 7.58 (d, *J* = 8.3 Hz, 2H), 7.32 (d, *J* = 8.3 Hz, 2H), 6.97 (s, 1H), 6.50 (s, 1H), 6.07 (ddt, *J* = 16.6, 10.3, 6.3 Hz, 1H), 5.22-5.16 (m, 2H), 5.04 (s, 1H), 3.55 (d, *J* = 6.2 Hz, 2H), 2.09 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.8, 151.2, 138.7, 136.6, 133.2, 132.9, 132.0, 130.4, 128.6, 124.2, 122.8, 117.6, 116.5, 111.7, 106.1, 36.1, 27.9; HRMS (m/z): $[M+H]^+$ calcd. for $C_{19}H_{17}BrNO_2$, 369.0364; found, 369.0365.



41

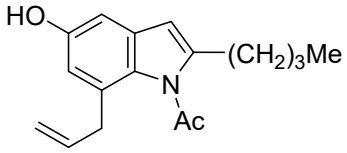
1-(7-allyl-5-hydroxy-2-(4-pentylphenyl)-1*H*-indol-1-yl)ethan-1-one 41: 9.1 mg, 58% yield. Yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.18 (s, 1H), 7.34 (d, *J* = 8.1 Hz, 2H), 7.25 (d, *J* = 8.0 Hz, 2H), 6.96 (s, 1H), 6.47 (s, 1H), 6.08 (ddt, *J* = 16.5, 10.1, 6.3 Hz, 1H), 5.22-5.16 (m, 2H), 4.97 (s, 1H), 3.55 (d, *J* = 6.2 Hz, 2H), 2.68-2.64 (m, 2H), 2.03 (s, 3H), 1.66 (p, *J* = 7.3 Hz, 2H), 1.38-1.33 (m, 4H), 0.91 (t,

J = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.4, 151.0, 143.7, 140.2, 136.8, 132.8, 131.5, 128.9, 128.8, 128.7, 123.6, 117.7, 116.4, 110.9, 106.0, 36.2, 35.7, 31.5, 31.0, 27.7, 22.5, 14.0; HRMS (m/z): $[M+H]^+$ calcd. for $C_{24}H_{28}NO_2$, 361.2115; found, 361.2115.



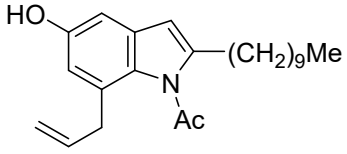
42

1-(7-allyl-5-hydroxy-2-(4-methoxyphenyl)-1*H*-indol-1-yl)ethan-1-one 42: 8.3 mg, 75% yield. Yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.18 (s, 1H), 7.35 (d, *J* = 8.8 Hz, 2H), 6.97 (d, *J* = 8.8 Hz, 2H), 6.96 (s, 1H), 6.44 (s, 1H), 6.08 (ddt, *J* = 17.2, 10.1, 6.3 Hz, 1H), 5.19 (dd, *J* = 17.1, 1.7 Hz, 1H), 5.17 (dd, *J* = 10.1, 1.6 Hz, 1H), 3.86 (s, 3H), 3.54 (d, *J* = 6.2 Hz, 2H), 2.04 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.3, 160.0, 151.0, 139.9, 136.8, 132.7, 130.3, 128.8, 126.5, 123.5, 117.7, 116.4, 114.2, 110.7, 105.9, 55.4, 36.2, 27.7; HRMS (m/z): $[M+H]^+$ calcd. for $C_{20}H_{20}NO_3$, 322.1438; found, 322.1437.



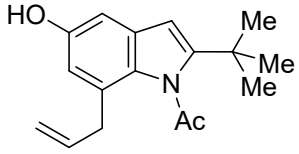
43

1-(7-allyl-2-butyl-5-hydroxy-1*H*-indol-1-yl)ethan-1-one 43: 9.9 mg, 65% yield. White solid, mp:169-170 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.67 (s, 1H), 6.89 (s, 1H), 6.28 (s, 1H), 6.06 (ddt, *J* = 17.9, 9.7, 6.3 Hz, 1H), 5.18-5.14 (m, 2H), 3.51 (d, *J* = 6.2 Hz, 2H), 2.95 (t, *J* = 7.6 Hz, 2H), 2.71 (s, 3H), 1.68 (p, *J* = 7.5 Hz, 2H), 1.44 (h, *J* = 7.4 Hz, 2H), 0.96 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.1, 150.5, 143.0, 136.8, 131.6, 129.7, 122.0, 116.7, 116.4, 107.9, 105.9, 36.0, 31.1, 30.4, 27.5, 22.6, 14.0; HRMS (m/z): $[M+H]^+$ calcd. for $C_{17}H_{22}NO_2$, 272.1645; found, 272.1645.



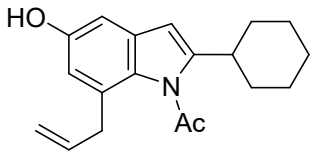
44

1-(7-allyl-2-decyl-5-hydroxy-1*H*-indol-1-yl)ethan-1-one 44: 6.1 mg, 55% yield. Yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.68 (s, 1H), 6.89 (s, 1H), 6.29 (s, 1H), 6.06 (ddt, *J* = 17.8, 9.8, 6.2 Hz, 1H), 5.19-5.14 (m, 2H), 4.86 (s, 1H), 3.51 (d, *J* = 6.2 Hz, 2H), 2.94 (t, *J* = 7.6 Hz, 2H), 2.70 (s, 3H), 1.69 (p, *J* = 7.6 Hz, 2H), 1.32-1.27 (m, 14H), 0.88 (t, *J* = 6.5 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.0, 150.5, 143.1, 136.8, 131.6, 129.7, 121.8, 116.7, 116.4, 107.9, 105.9, 36.0, 31.9, 30.7, 29.7, 29.6, 29.5, 29.5, 29.3, 28.9, 27.5, 22.7, 14.1; HRMS (m/z): $[M+H]^+$ calcd. for $C_{23}H_{34}NO_2$, 356.2584; found, 356.2583.



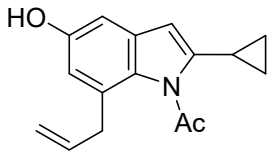
45

1-(7-allyl-2-(tert-butyl)-5-hydroxy-1*H*-indol-1-yl)ethan-1-one 45: 7.4 mg, 50% yield. Yellow solid, mp:171-172 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.22 (s, 1H), 6.91 (s, 1H), 6.40 (s, 1H), 6.05 (ddt, *J* = 16.7, 10.5, 6.2 Hz, 1H), 5.18-5.13 (m, 2H), 4.83 (s, 1H), 3.50 (d, *J* = 6.1 Hz, 2H), 2.77 (s, 3H), 1.45 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.8, 152.3, 149.9, 136.7, 131.8, 129.1, 121.8, 116.5, 114.9, 106.4, 106.2, 35.9, 34.2, 30.5, 28.1; HRMS (m/z): $[M+H]^+$ calcd. for $C_{17}H_{22}NO_2$, 272.1645; found, 272.1647.



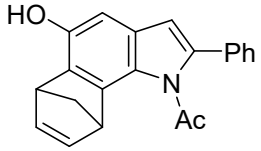
46

1-(7-allyl-2-cyclohexyl-5-hydroxy-1*H*-indol-1-yl)ethan-1-one 46: 10.5 mg, 71% yield. White solid, mp:166-167 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.54 (s, 1H), 6.90 (s, 1H), 6.32 (s, 1H), 6.05 (ddt, *J* = 17.6, 9.7, 6.3 Hz, 1H), 5.19-5.18 (m, 1H), 5.16-5.14 (m, 1H), 4.86 (s, 1H), 3.51 (d, *J* = 6.2 Hz, 2H), 3.27 (tt, *J* = 11.1, 2.8 Hz, 1H), 2.73 (s, 3H), 2.10-2.06 (m, 2H), 1.86-1.81 (m, 2H), 1.46-1.41 (m, 2H), 1.40-1.32 (m, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.2, 150.4, 149.2, 136.7, 131.4, 129.9, 121.8, 116.5, 116.3, 106.2, 105.6, 38.0, 36.0, 33.9, 27.7, 26.6, 26.4; HRMS (m/z): $[M+H]^+$ calcd. for $C_{19}H_{24}NO_2$, 298.1802; found, 298.1804.



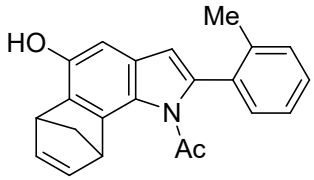
47

1-(7-allyl-2-cyclopropyl-5-hydroxy-1*H*-indol-1-yl)ethan-1-one 47: 10.8 mg, 70% yield. White solid, mp:152-153 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.14 (s, 1H), 6.85 (s, 1H), 6.23 (s, 1H), 6.05 (ddt, *J* = 16.6, 10.2, 6.2 Hz, 1H), 5.19-5.14 (m, 2H), 4.93 (s, 1H), 3.51 (d, *J* = 6.2 Hz, 2H), 2.82 (s, 3H), 2.08-2.01 (m, 1H), 1.08-1.03 (m, 2H), 0.87-0.83 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.4, 150.8, 142.4, 136.9, 132.4, 128.8, 122.7, 118.1, 116.4, 108.5, 105.7, 36.1, 27.0, 11.8, 8.5; HRMS (m/z): $[M+H]^+$ calcd. for $C_{16}H_{18}NO_2$, 256.1332; found, 256.1330.



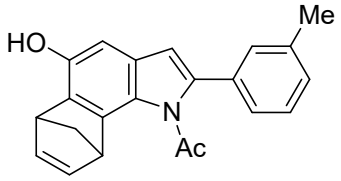
48

1-(5-hydroxy-2-phenyl-6,9-dihydro-1*H*-6,9-methanobenzo[*g*]indol-1-yl)ethan-1-one 48: 27.5 mg, 68% yield. Yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.44-7.39 (m, 5H), 7.01 (dd, *J* = 5.2, 3.2 Hz, 1H), 6.91 (dd, *J* = 5.1, 3.0 Hz, 1H), 6.61 (s, 1H), 6.46 (s, 1H), 4.77-4.75 (m, 1H), 4.70 (s, 1H), 4.19-4.18 (m, 1H) 2.29-2.23 (m, 2H), 2.02 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.1, 147.0, 143.8, 143.2, 140.7, 140.5, 136.5, 134.5, 129.3, 128.8, 128.8, 128.7, 128.5, 112.1, 103.0, 69.0, 52.4, 46.5, 28.0; HRMS (m/z): $[M+H]^+$ calcd. for $C_{21}H_{18}NO_2$, 316.1332; found, 316.1332.



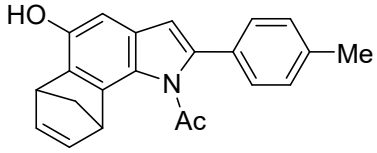
49

1-(5-hydroxy-2-(*o*-tolyl)-6,9-dihydro-1*H*-6,9-methanobenzo[*g*]indol-1-yl)ethan-1-one 49: 8.6 mg, 62% yield. Yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.36-7.27 (m, 4H), 7.03 (dd, *J* = 5.2, 3.1 Hz, 1H), 6.91 (dd, *J* = 5.2, 2.9 Hz, 1H), 6.60 (s, 1H), 6.33 (s, 1H), 4.69-4.67 (m, 1H), 4.21-4.19 (m, 1H), 2.27-2.21 (m, 5H), 1.88 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.4, 147.1, 143.8, 143.3, 141.1, 138.9, 137.5, 136.3, 134.2, 130.5, 130.4, 129.4, 129.1, 127.9, 126.0, 111.6, 102.8, 68.7, 52.6, 46.5, 26.4, 20.1; HRMS (m/z): $[M+H]^+$ calcd. for $C_{22}H_{20}NO_2$, 330.1489; found, 330.1490.



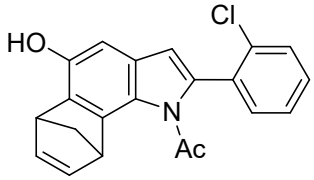
50

1-(5-hydroxy-2-(*m*-tolyl)-6,9-dihydro-1*H*-6,9-methanobenzo[*g*]indol-1-yl)ethan-1-one 50: 5.4 mg, 60% yield. Yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.32 (t, *J* = 7.5 Hz, 1H), 7.26-7.19 (m, 3H), 7.00 (dd, *J* = 5.3, 3.2 Hz, 1H), 6.90 (dd, *J* = 5.3, 2.9 Hz, 1H), 6.60 (s, 1H), 6.44 (s, 1H), 4.90 (s, 1H), 4.78-4.76 (m, 1H), 4.20-4.18 (m, 1H), 2.40 (s, 3H), 2.28-2.22 (m, 2H), 2.03 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.2, 147.0, 143.9, 143.2, 140.7, 140.7, 138.5, 136.4, 134.4, 129.3, 129.3, 129.2, 128.8, 128.6, 125.9, 111.9, 103.0, 69.0, 52.4, 46.5, 28.0, 21.4; HRMS (m/z): $[M+H]^+$ calcd. for $C_{22}H_{20}NO_2$, 330.1489; found, 330.1487.



51

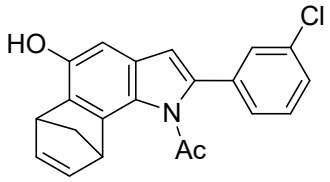
1-(5-hydroxy-2-(*p*-tolyl)-6,9-dihydro-1*H*-6,9-methanobenzo[*g*]indol-1-yl)ethan-1-one 51: 7.1 mg, 66% yield. Yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.33 (d, *J* = 8.0 Hz, 2H), 7.24 (d, *J* = 7.8 Hz, 2H), 7.00 (dd, *J* = 5.2, 3.2 Hz, 1H), 6.90 (dd, *J* = 5.3, 3.0 Hz, 1H), 6.59 (s, 1H), 6.42 (s, 1H), 4.76-4.74 (m, 1H), 4.19-4.17 (m, 1H), 2.41 (s, 3H), 2.28-2.21 (m, 2H), 2.02 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.1, 146.9, 143.8, 143.1, 140.7, 138.5, 136.2, 131.6, 129.4, 129.4, 128.8, 128.7, 111.6, 102.8, 69.0, 52.4, 46.5, 28.0, 21.3; HRMS (m/z): $[M+H]^+$ calcd. for $C_{22}H_{20}NO_2$, 330.1489; found, 330.1491.



52

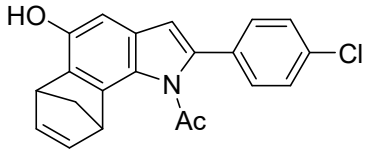
1-(2-(2-chlorophenyl)-5-hydroxy-6,9-dihydro-1*H*-6,9-methanobenzo[*g*]indol-1-yl)ethan-1-one 52: 5.4 mg, 64% yield. Yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.49 (dd, *J* = 7.6, 1.7 Hz, 1H),

7.44-7.37 (m, 3H), 7.04 (dd, *J* = 5.3, 3.2 Hz, 1H), 6.91 (dd, *J* = 5.3, 3.0 Hz, 1H), 6.62 (s, 1H), 6.43 (s, 1H), 4.76 (s, 1H), 4.70-4.68 (m, 1H), 4.20-4.19 (m, 1H), 2.28-2.23 (m, 2H), 2.01 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.8, 147.0, 143.8, 143.2, 141.1, 136.8, 136.4, 133.9, 131.9, 130.4, 130.3, 129.9, 129.9, 129.1, 127.0, 112.7, 103.0, 68.8, 52.6, 46.5, 26.1; HRMS (m/z): $[M+H]^+$ calcd. for $C_{21}H_{17}ClNO_2$, 350.0942; found, 350.0945.



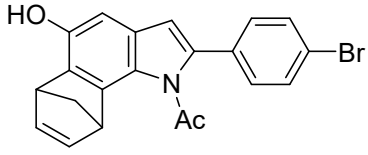
53

1-(2-(3-chlorophenyl)-5-hydroxy-6,9-dihydro-1*H*-6,9-methanobenzo[*g*]indol-1-yl)ethan-1-one 53: 7.9 mg, 60% yield. Yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.48-7.47 (m, 1H), 7.38-7.31 (m, 3H), 7.00 (dd, *J* = 5.2, 3.2 Hz, 1H), 6.90 (dd, *J* = 5.3, 3.0 Hz, 1H), 6.61 (s, 1H), 6.49 (s, 1H), 4.93 (s, 1H), 4.77-4.75 (m, 1H), 4.21-4.19 (m, 1H), 2.29-2.22 (m, 2H), 2.06 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.6, 147.1, 143.8, 143.1, 140.9, 138.9, 137.1, 136.9, 136.2, 134.8, 130.0, 129.0, 128.5, 128.5, 126.8, 112.9, 103.2, 69.1, 52.4, 46.6, 28.1; HRMS (m/z): $[M+H]^+$ calcd. for $C_{21}H_{17}ClNO_2$, 350.0942; found, 350.0944.



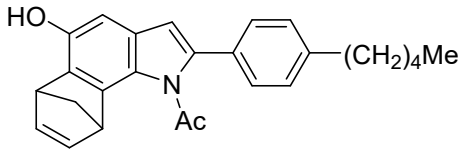
54

1-(2-(4-chlorophenyl)-5-hydroxy-6,9-dihydro-1*H*-6,9-methanobenzo[*g*]indol-1-yl)ethan-1-one 54: 6.8 mg, 68% yield. Yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.43 (d, *J* = 8.8 Hz, 2H), 7.39 (d, *J* = 8.6 Hz, 2H), 7.00 (dd, *J* = 5.3, 3.1 Hz, 1H), 6.90 (dd, *J* = 5.2, 3.0 Hz, 1H), 6.61 (s, 1H), 6.46 (s, 1H), 4.75-4.74 (m, 1H), 4.20-4.17 (m, 1H), 2.29-2.22 (m, 2H), 2.04 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.7, 147.0, 143.8, 143.1, 140.8, 139.2, 136.8, 134.6, 132.9, 129.9, 129.1, 129.0, 129.0, 112.5, 103.0, 69.1, 52.4, 46.5, 28.1; HRMS (m/z): $[M+H]^+$ calcd. for $C_{21}H_{17}ClNO_2$, 350.0942; found, 350.0941.



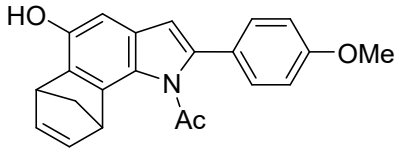
55

1-(2-(4-bromophenyl)-5-hydroxy-6,9-dihydro-1*H*-6,9-methanobenzo[*g*]indol-1-yl)ethan-1-one 55: 6.3 mg, 63% yield. Yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.58 (d, *J* = 8.4 Hz, 2H), 7.33 (d, *J* = 8.4 Hz, 2H), 7.00 (dd, *J* = 5.3, 3.1 Hz, 1H), 6.90 (dd, *J* = 5.2, 3.0 Hz, 1H), 6.61 (s, 1H), 6.46 (s, 1H), 4.74-4.76 (m, 1H), 4.63 (s, 1H), 4.19-4.17 (m, 1H), 2.29-2.21 (m, 2H), 2.05 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.6, 147.0, 143.8, 143.1, 140.8, 136.8, 133.4, 132.0, 130.2, 130.1, 129.1, 122.7, 112.5, 103.1, 69.1, 52.4, 46.5, 28.2; HRMS (m/z): $[M+H]^+$ calcd. for $C_{21}H_{17}BrNO_2$, 394.0437; found, 394.0438.



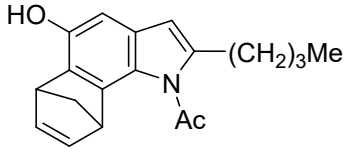
56

1-(5-hydroxy-2-(4-pentylphenyl)-6,9-dihydro-1*H*-6,9-methanobenzo[*g*]indol-1-yl)ethan-1-one 56: 6.7 mg, 63% yield. Yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.34 (d, *J* = 8.1 Hz, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 7.00 (dd, *J* = 5.3, 3.1 Hz, 1H), 6.90 (dd, *J* = 5.2, 3.0 Hz, 1H), 6.59 (s, 1H), 6.43 (s, 1H), 4.76-4.74 (m, 1H), 4.72 (s, 1H), 4.19-4.18 (m, 1H), 2.67-2.63 (m, 2H), 2.28-2.22 (m, 2H), 2.02 (s, 3H), 1.65 (p, *J* = 7.5 Hz, 2H), 1.37-1.33 (m, 4H), 0.91 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.1, 146.9, 143.8, 143.6, 143.1, 140.7, 140.6, 136.2, 131.7, 129.4, 128.7, 128.7, 111.6, 102.8, 69.0, 52.4, 46.5, 35.7, 31.5, 31.0, 28.0, 22.5, 14.0; HRMS (m/z): $[M+H]^+$ calcd. for $C_{26}H_{28}BrNO_2$, 386.2115; found, 386.2117.



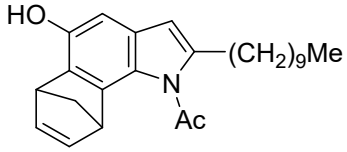
57

1-(5-hydroxy-2-(4-methoxyphenyl)-6,9-dihydro-1*H*-6,9-methanobenzo[*g*]indol-1-yl)ethan-1-one 57: 4.6 mg, 56% yield. Yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.36 (d, *J* = 8.7 Hz, 2H), 7.00 (dd, *J* = 5.3, 3.2 Hz, 1H), 6.97 (d, *J* = 8.7 Hz, 2H), 6.90 (dd, *J* = 5.2, 3.0 Hz, 1H), 4.75-4.73 (m, 1H), 4.65 (s, 1H), 4.19-4.17 (m, 1H), 3.86 (s, 3H), 2.28-2.20 (m, 2H), 2.02 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.6, 146.9, 143.8, 143.1, 140.4, 134.9, 130.2, 130.1, 126.8, 125.6, 114.4, 114.1, 111.3, 105.4, 102.7, 68.9, 55.4, 52.4, 46.5, 27.9; HRMS (m/z): $[M+H]^+$ calcd. for $C_{22}H_{20}NO_3$, 346.1438; found, 346.1438.



58

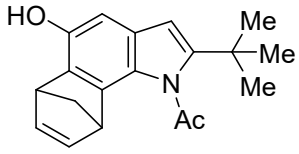
1-(2-butyl-5-hydroxy-6,9-dihydro-1*H*-6,9-methanobenzo[*g*]indol-1-yl)ethan-1-one 58: 7.8 mg, 70% yield. Colorless oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 6.97-6.92 (m, 2H), 6.52 (s, 1H), 6.21 (s, 1H), 4.76 (s, 1H), 4.35-4.33 (m, 1H), 4.19-4.16 (m, 1H), 2.86-2.79 (m, 2H), 2.62 (s, 3H), 2.27-2.25 (m, 1H), 2.16-2.13 (m, 1H), 1.71-1.66 (m, 2H), 1.44 (h, *J* = 7.4 Hz, 2H), 0.96 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.8, 146.6, 144.0, 142.1, 141.7, 139.4, 134.4, 129.7, 127.9, 107.5, 102.3, 69.2, 51.7, 46.4, 31.1, 29.6, 27.0, 22.5, 13.9; HRMS (m/z): $[M+H]^+$ calcd. for $C_{19}H_{22}NO_2$, 296.1645; found, 296.1642.



59

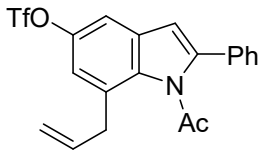
1-(2-decyl-5-hydroxy-6,9-dihydro-1*H*-6,9-methanobenzo[*g*]indol-1-yl)ethan-1-one 59: 5.4 mg, 60% yield. Yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 6.97-6.92 (m, 2H), 6.52 (s, 1H), 6.21 (s,

1H), 4.35-4.33 (m, 1H), 4.18-4.16 (m, 1H), 2.85-2.78 (m, 2H), 2.61 (s, 3H), 2.27-2.24 (m, 1H), 2.16-2.13 (m, 1H), 1.73-1.65 (m, 2H), 1.33-1.27 (m, 14H), 0.88 (t, J = 6.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.8, 149.7, 149.0, 146.6, 146.6, 144.0, 142.1, 141.8, 129.7, 127.9, 107.4, 102.3, 69.2, 51.7, 46.4, 31.9, 29.9, 29.6, 29.6, 29.5, 29.3, 29.0, 27.0, 22.7, 14.1; HRMS (m/z): $[M+H]^+$ calcd. for $C_{25}H_{34}NO_2$, 380.2584; found, 380.2585.



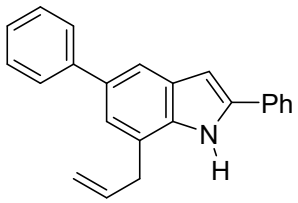
60

1-(2-(tert-butyl)-5-hydroxy-6,9-dihydro-1*H*-6,9-methanobenzo[*g*]indol-1-yl)ethan-1-one 60: 6.1 mg, 62% yield. Colorless oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 6.98 (dd, J = 5.4, 3.1 Hz, 1H), 6.89 (dd, J = 5.3, 2.9 Hz, 1H), 6.58 (s, 1H), 6.23 (s, 1H), 4.67 (s, 1H), 4.22-4.20 (m, 1H), 4.07-4.09 (m, 1H), 2.57 (s, 3H), 2.34-2.31 (m, 1H), 2.18-2.21 (m, 1H), 1.42 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.4, 151.0, 146.1, 145.0, 140.6, 140.6, 136.8, 133.6, 128.6, 103.4, 102.4, 70.8, 50.1, 46.6,33.5, 30.1, 29.0; HRMS (m/z): $[M+H]^+$ calcd. for $C_{19}H_{22}NO_2$, 296.1645; found, 296.1644.



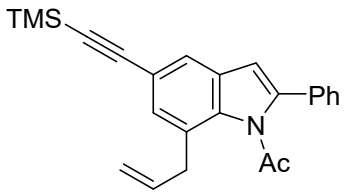
61

1-acetyl-7-allyl-2-phenyl-1*H*-indol-5-yl trifluoromethanesulfonate 61: 30.7 mg, 94% yield. Colorless oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.35 (s, 1H), 7.49-7.44 (m, 6H), 6.61 (s, 1H), 6.00 (ddt, J = 17.7, 9.3, 6.5 Hz, 1H), 5.18-5.14 (m, 2H), 3.60 (d, J = 6.5 Hz, 2H), 2.05 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.4, 144.4, 141.5, 136.7, 135.2, 133.4, 129.5, 129.1, 129.0, 128.9, 128.2, 118.7 (q, J = 320.2 Hz), 118.2, 117.1, 112.6, 110.6, 35.0, 27.8; HRMS (m/z): $[M+H]^+$ calcd. for $C_{20}H_{17}F_3NO_4S$, 424.0825; found, 424.0823.



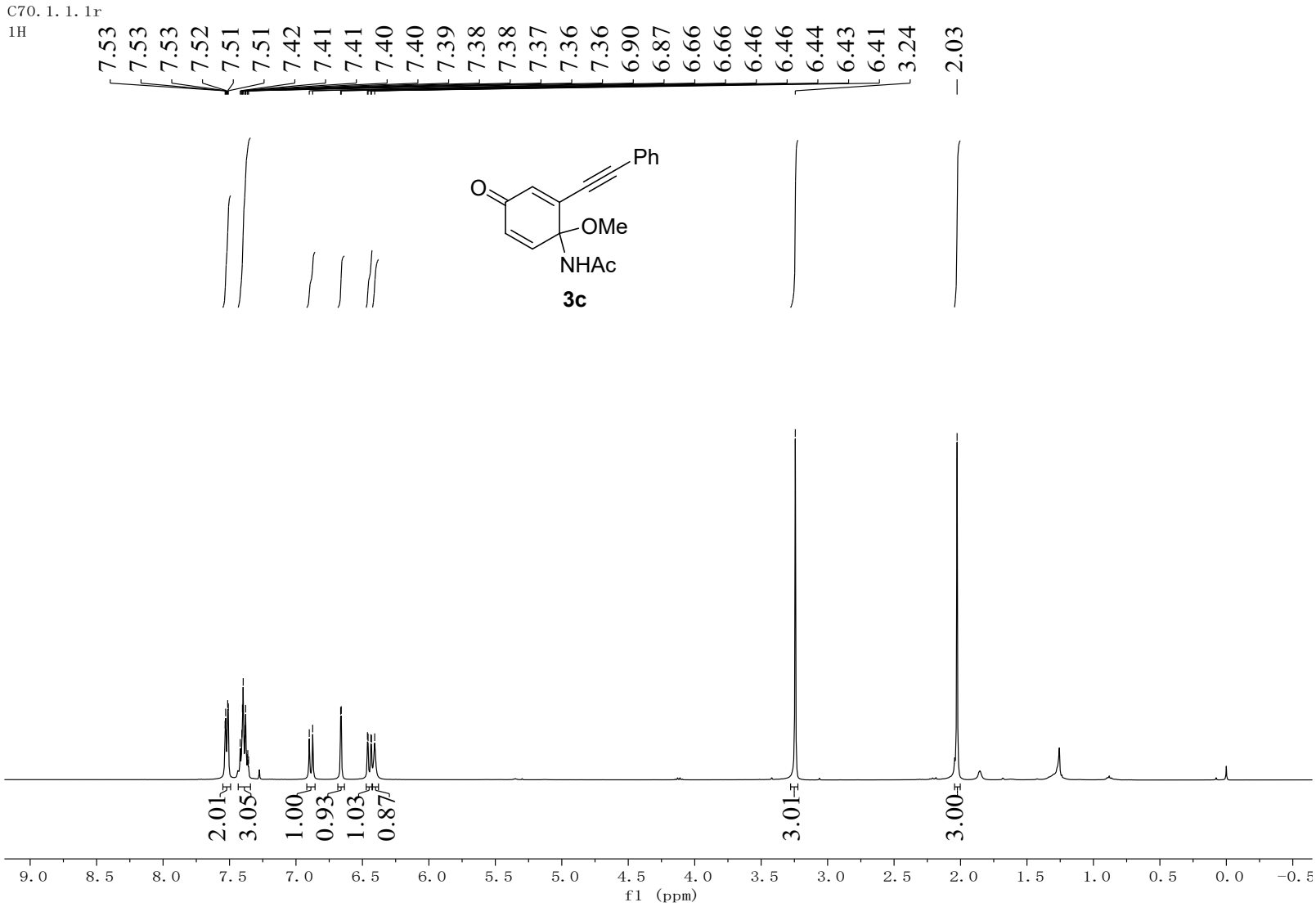
62

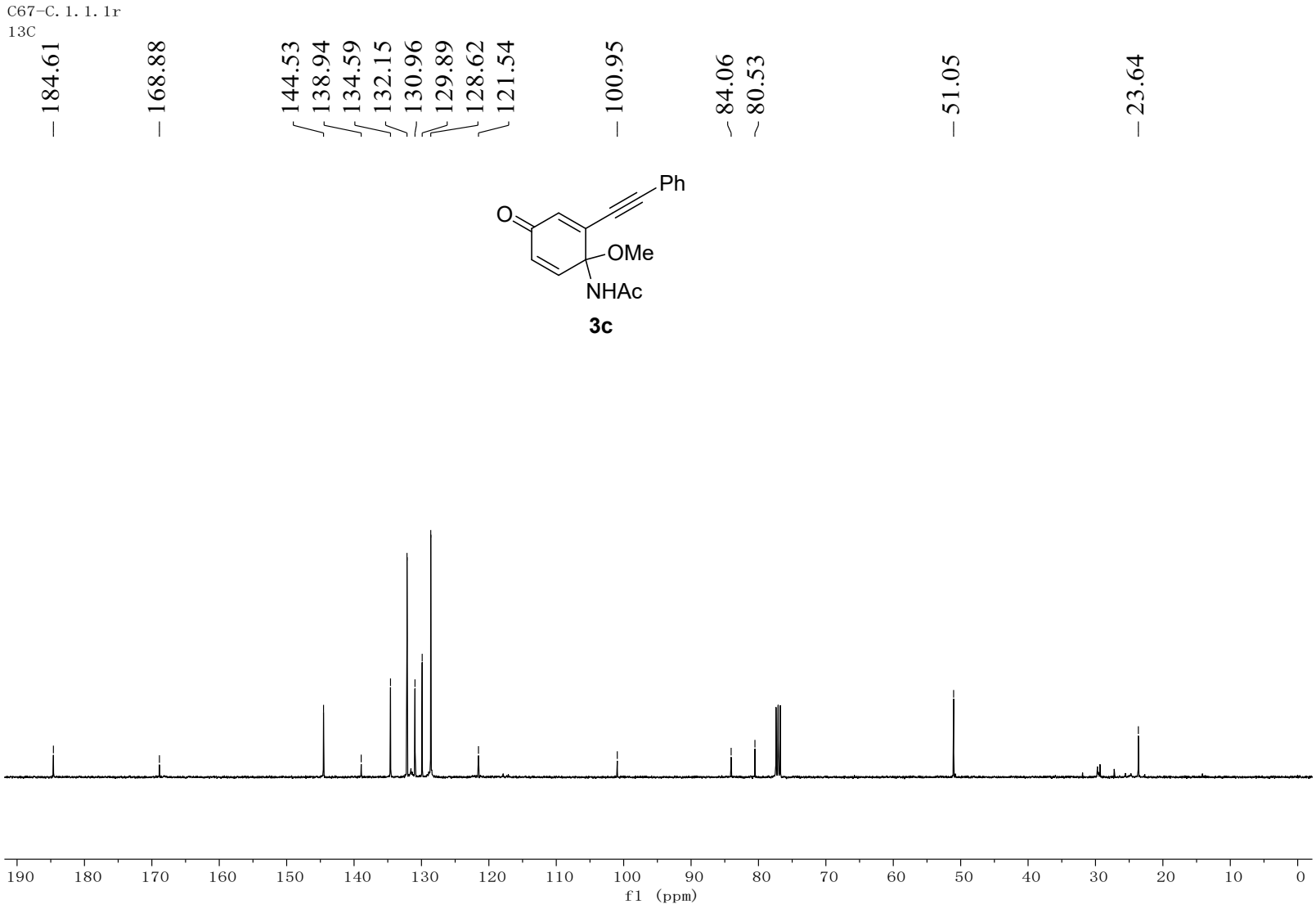
7-allyl-2,5-diphenyl-1*H*-indole 62: 7.5 mg, 83% yield. White solid, mp:171-172 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.27 (s, 1H), 7.67-7.65 (m, 2H), 7.48 (s, 1H), 7.46-7.40 (m, 4H), 7.39 (s, 1H), 7.38 (s, 1H), 7.36-7.31 (m, 4H), 6.80-6.79 (m, 1H), 5.93 (ddt, J = 16.7, 10.1, 6.5 Hz, 1H), 5.01 (dd, J = 10.1, 1.8 Hz, 1H), 4.95 (dd, J = 17.0, 1.7 Hz, 1H), 3.42 (d, J = 6.4 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 142.8, 138.3, 138.3, 136.7, 135.2, 132.5, 132.2, 129.9, 129.1, 127.9, 127.7, 127.7, 126.4, 125.1, 121.8, 115.5, 111.3, 99.9, 38.1; HRMS (m/z): $[M+H]^+$ calcd. for $C_{23}H_{20}N$, 310.1590; found, 310.1590.

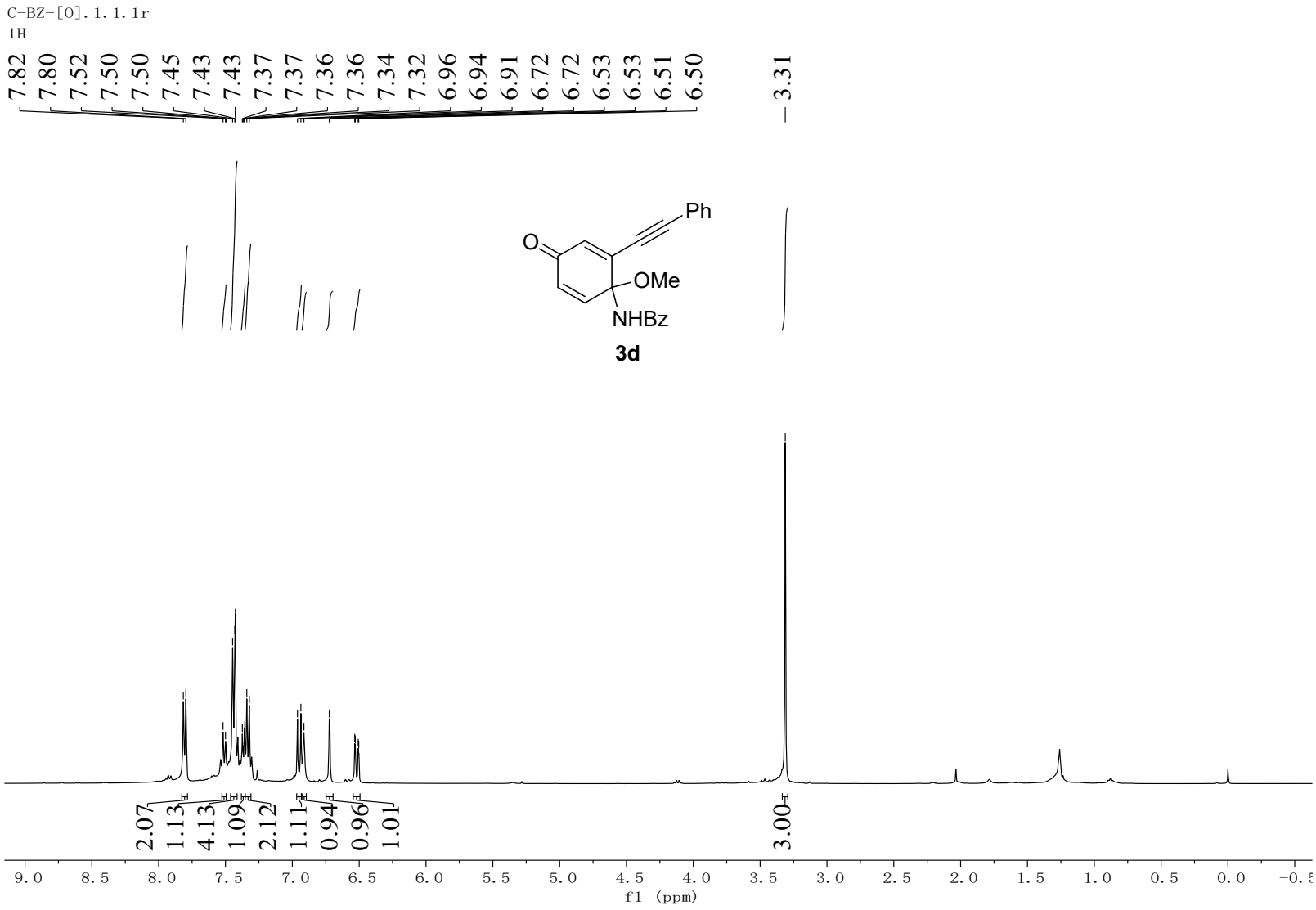


63

1-(7-allyl-2-phenyl-5-((trimethylsilyl)ethynyl)-1*H*-indol-1-yl)ethan-1-one 63: 8.5 mg, 79% yield. White solid, mp:171-172 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.21 (s, 1H), 7.67 (s, 1H), 7.48-7.41 (m, 6H), 6.54 (s, 1H), 6.05 (ddt, *J* = 16.8, 10.0, 6.7 Hz, 1H), 5.15 (dd, *J* = 17.0, 1.8 Hz, 1H), 5.08 (dd, *J* = 10.0, 1.8 Hz, 1H), 3.69 (d, *J* = 6.7 Hz, 2H), 2.05 (s, 3H), 0.27 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.4, 140.2, 139.8, 137.9, 136.9, 133.9, 128.9, 128.8, 128.7, 127.2, 124.6, 118.3, 116.1, 115.8, 111.0, 104.4, 96.9, 39.5, 27.9, 0.1; HRMS (m/z): $[M+H]^+$ calcd. for $C_{24}H_{26}NOSi$, 372.1778; found, 372.1775.

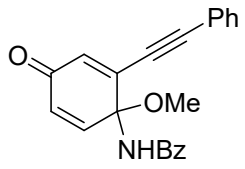

C70.1.1.1r
1H
7.53
7.53
7.53
7.52
7.51
7.51
7.42
7.41
7.41
7.40
7.40
7.39
7.38
7.38
7.37
7.36
7.36
6.90
6.87
6.66
6.66
6.46
6.46
6.44
6.43
6.41
3.24
2.03
Ph
O
OMe
NHAc
3c
2.01
3.05
1.00
0.93
1.03
0.87
3.01
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
f1 (ppm)


C67-C.1.1.1r
13C
184.61
168.88
144.53
138.94
134.59
132.15
130.96
129.89
128.62
121.54
100.95
84.06
80.53
51.05
23.64
O
Ph
OMe
NHAc
3c
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

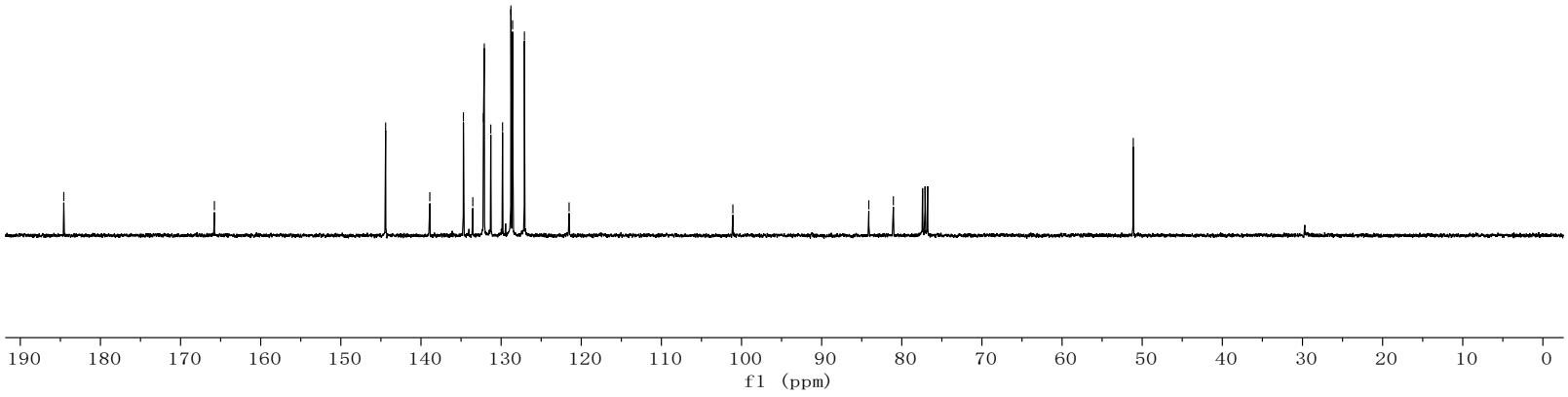

C-BZ-[O].1.1.1r
1H
7.82
7.80
7.52
7.50
7.50
7.45
7.43
7.43
7.37
7.37
7.36
7.36
7.34
7.32
6.96
6.94
6.91
6.72
6.72
6.53
6.53
6.51
6.50
3.31
Ph
O
OMe
NHBz
3d
2.07
1.13
4.13
1.09
2.12
1.11
0.94
0.96
1.01
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

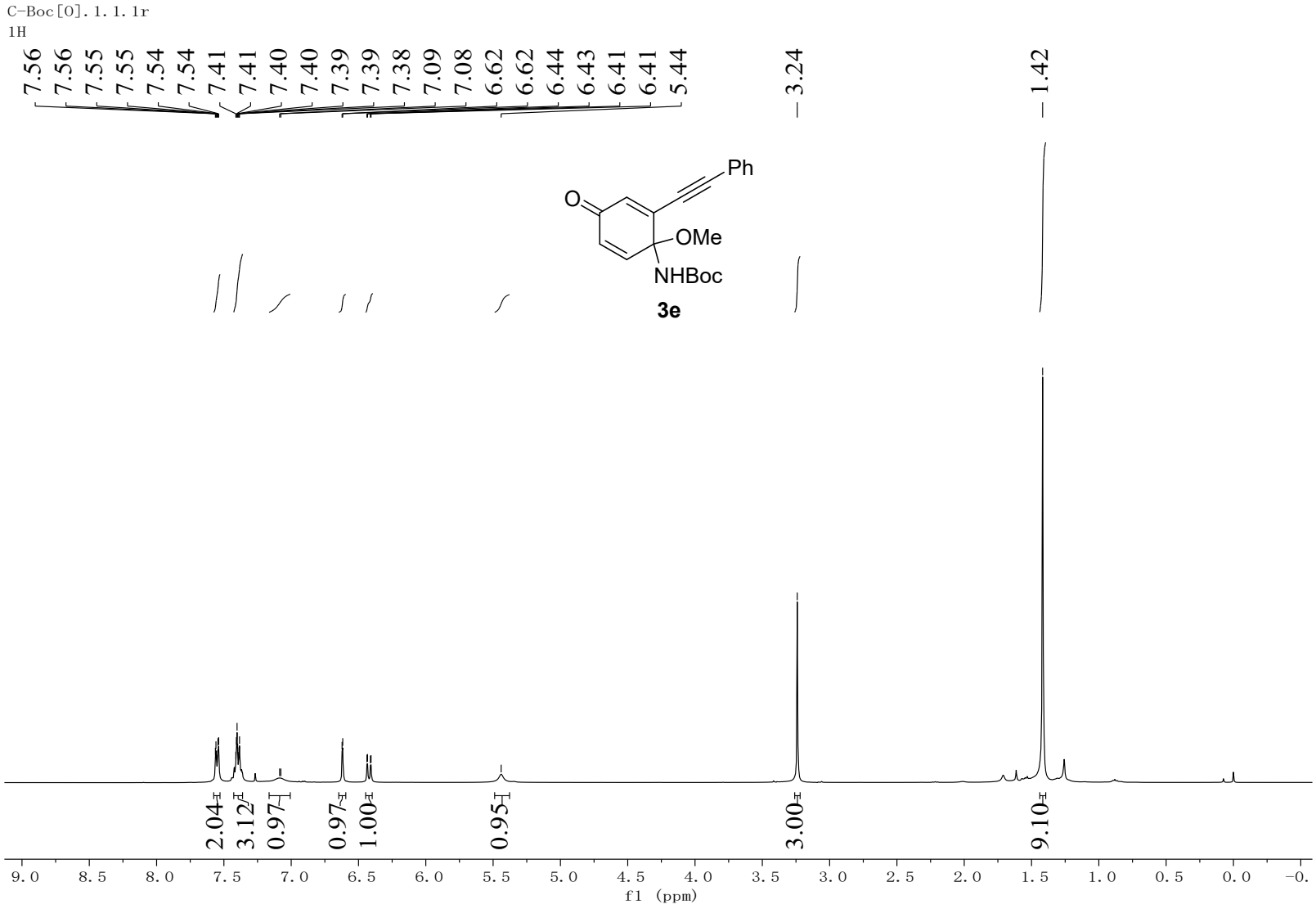
C-BZ-[O]-C.1.1.1r
13C

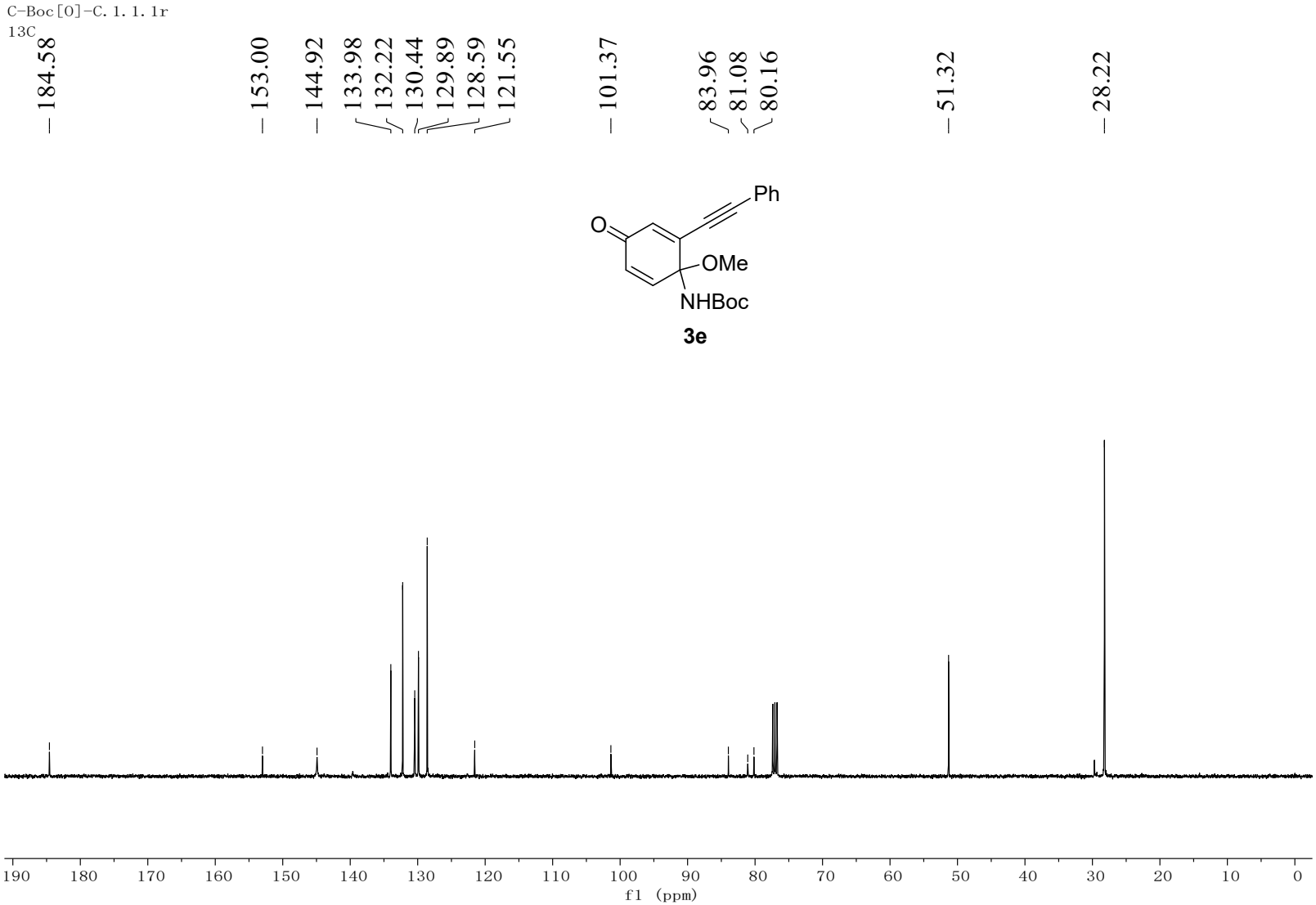
184.57
165.78
144.41
138.90
134.70
133.53
132.21
132.10
131.29
129.82
128.78
128.57
127.09
121.52
101.08
84.12
81.05
51.11

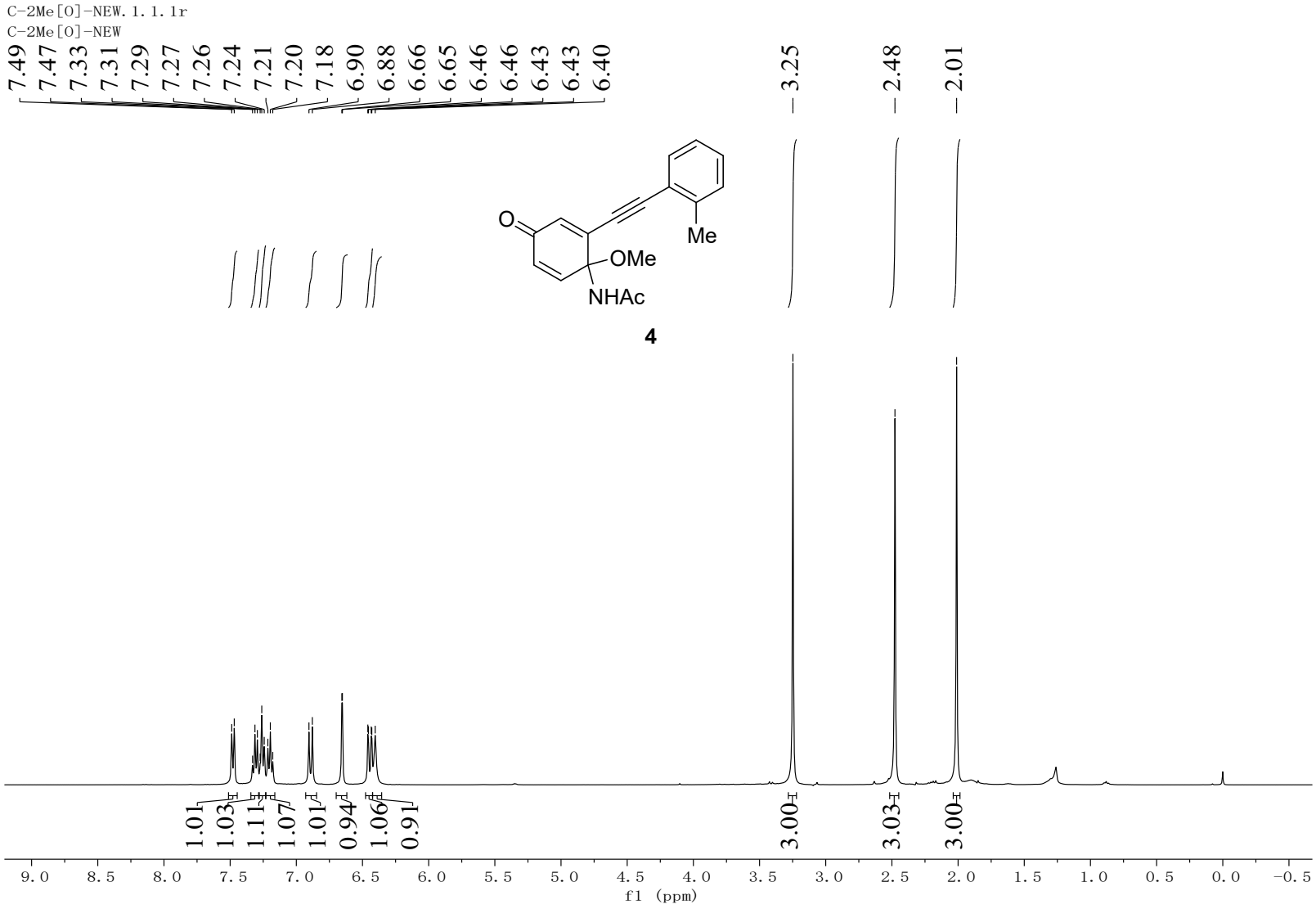


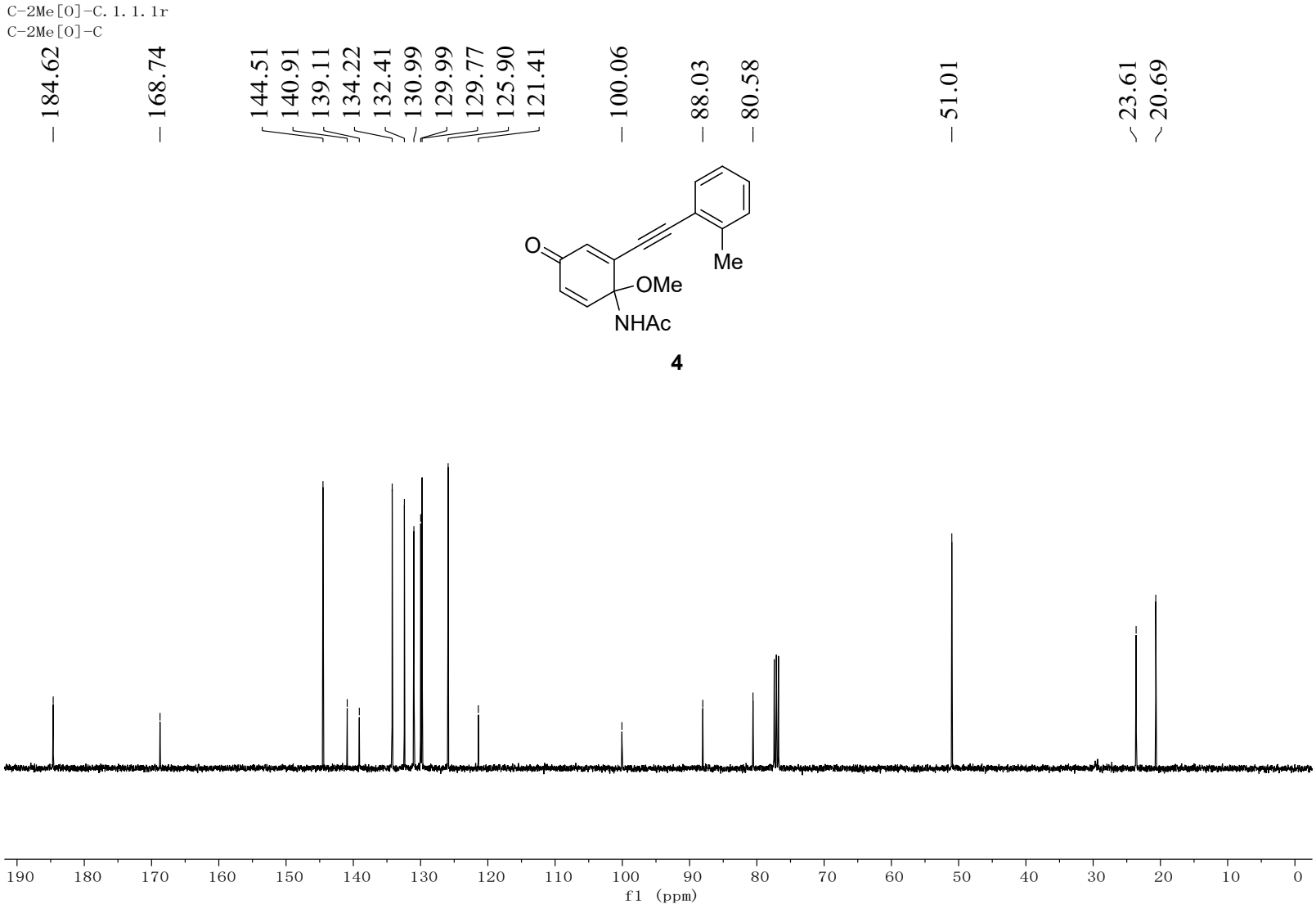
3d

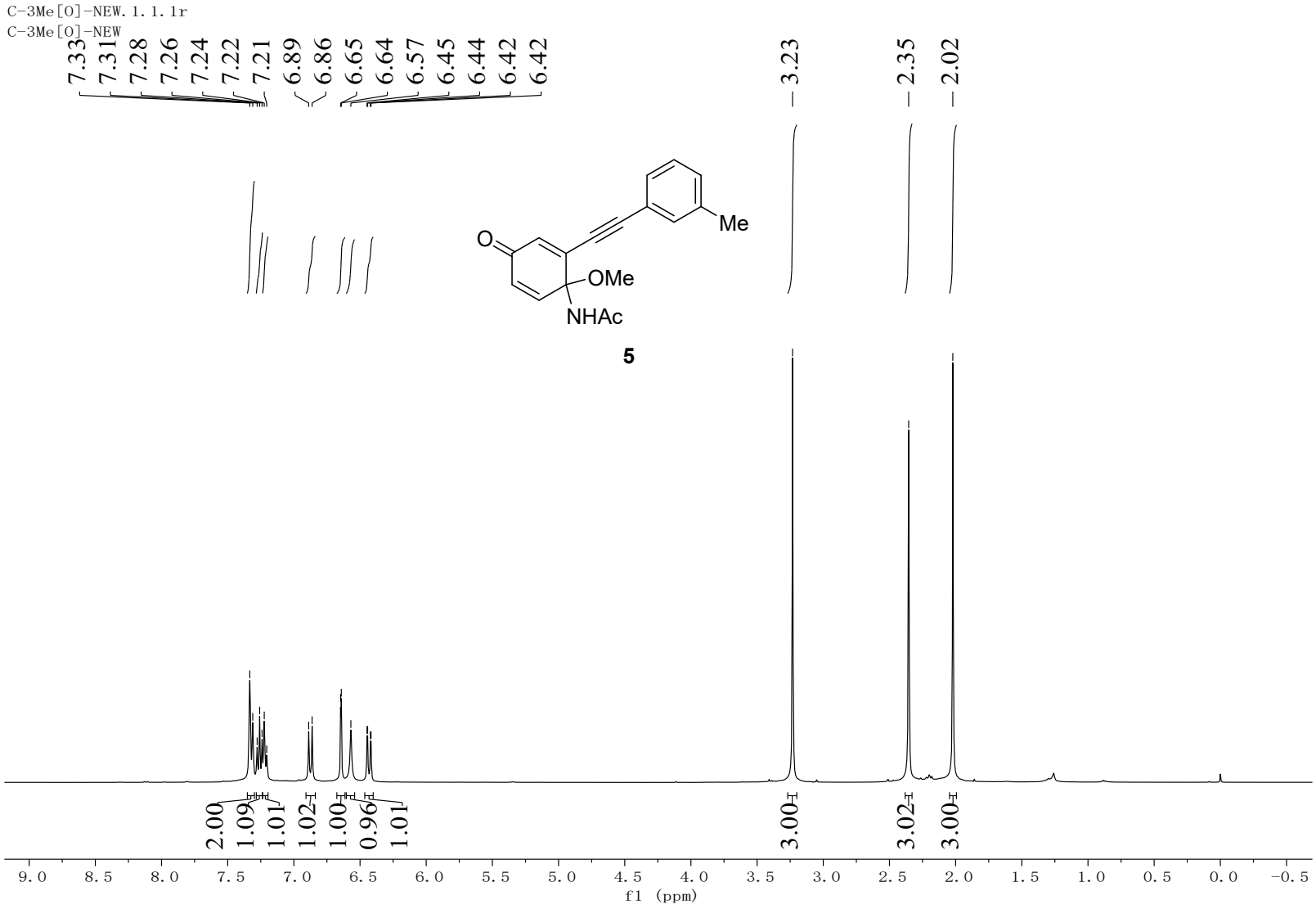


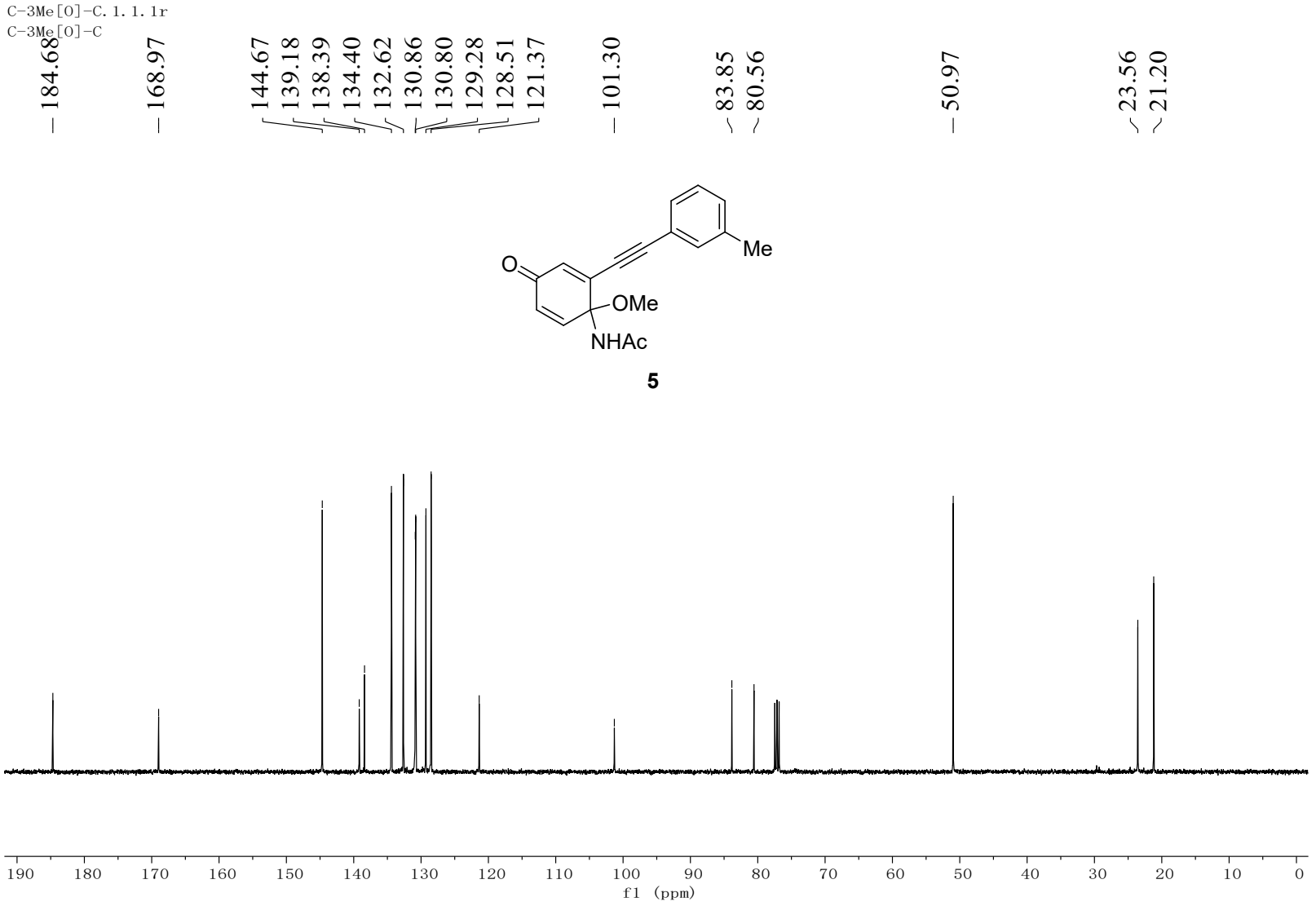

C-Boc[0].1.1.1r
1H
7.56
7.56
7.55
7.55
7.54
7.54
7.41
7.41
7.40
7.40
7.39
7.39
7.38
7.09
7.08
6.62
6.62
6.44
6.43
6.41
6.41
5.44
3.24
1.42
Ph
O
OMe
NHBoc
3e
2.04
3.12
0.97
0.97
1.00
0.95
3.00
9.10
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.
f1 (ppm)

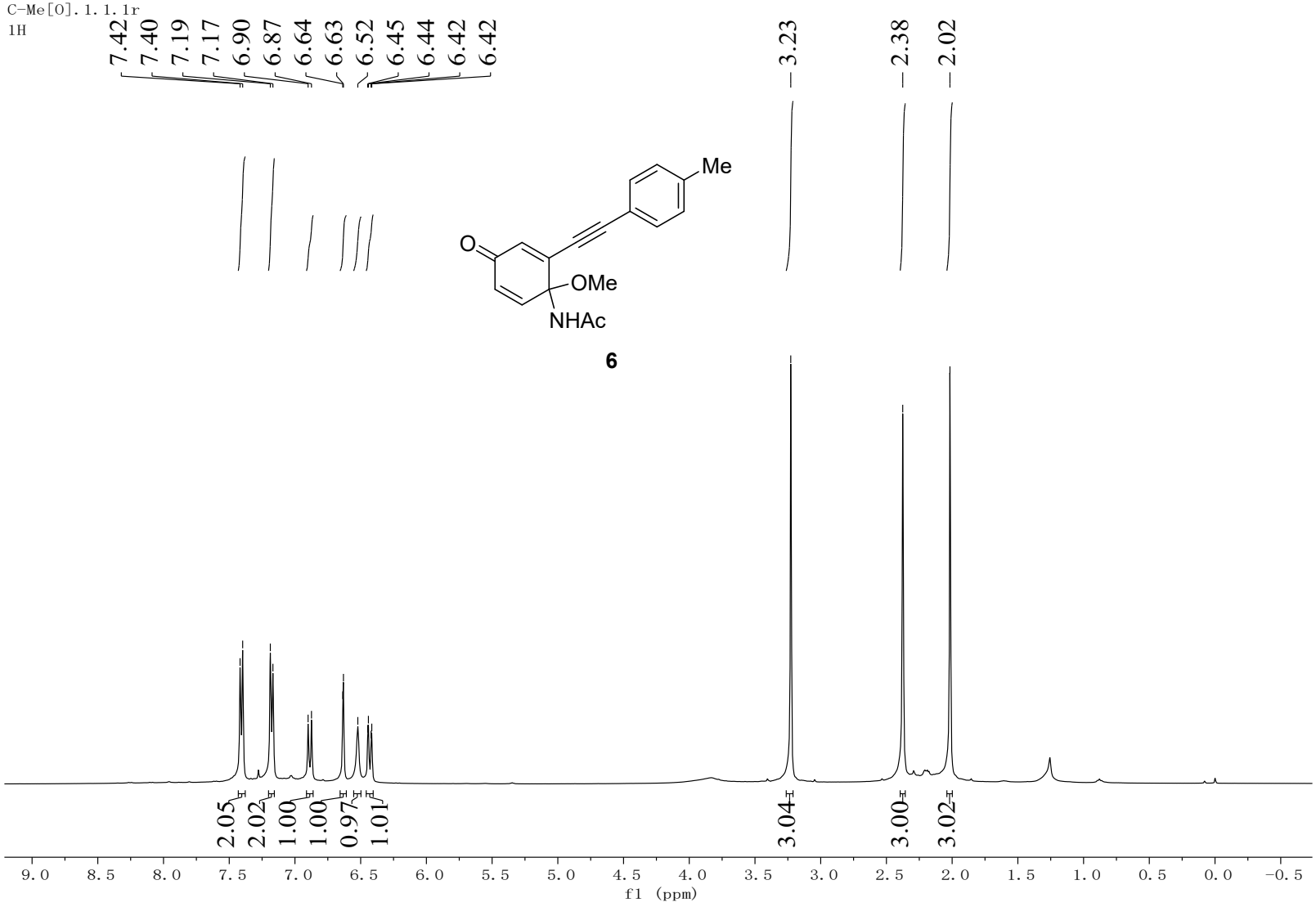

C-Boc[O]-C. 1. 1. 1r
13C
184.58
153.00
144.92
133.98
132.22
130.44
129.89
128.59
121.55
101.37
83.96
81.08
80.16
51.32
28.22
Ph
O
OMe
NHBoc
3e
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

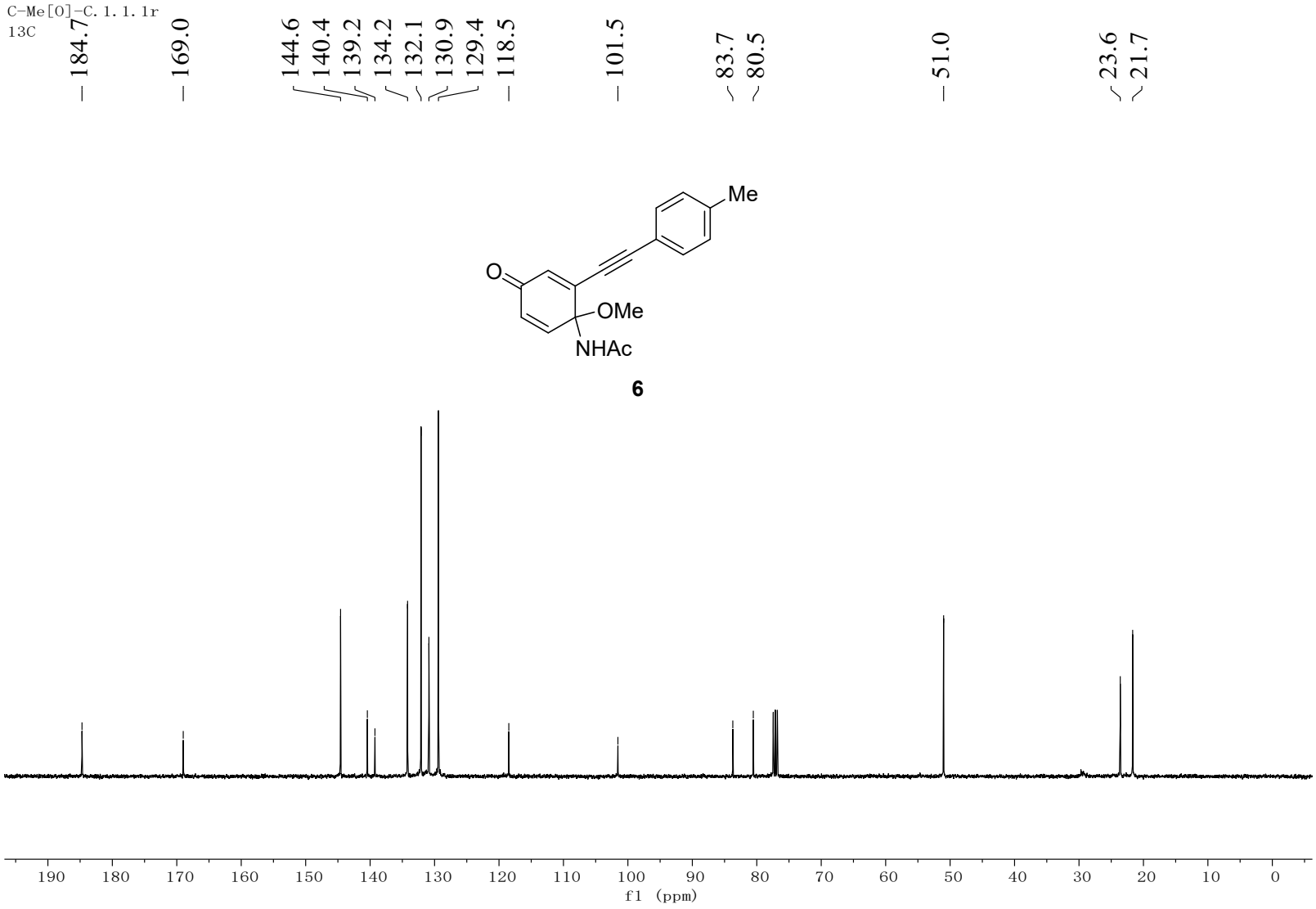

C-2Me[O]-NEW.1.1.1r
C-2Me[O]-NEW
7.49
7.47
7.33
7.31
7.29
7.27
7.26
7.24
7.21
7.20
7.18
6.90
6.88
6.66
6.65
6.46
6.46
6.43
6.43
6.40
3.25
2.48
2.01
O
Me
OMe
NHAc
4
1.01
1.03
1.11
1.07
1.01
0.94
1.06
0.91
3.00
3.03
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

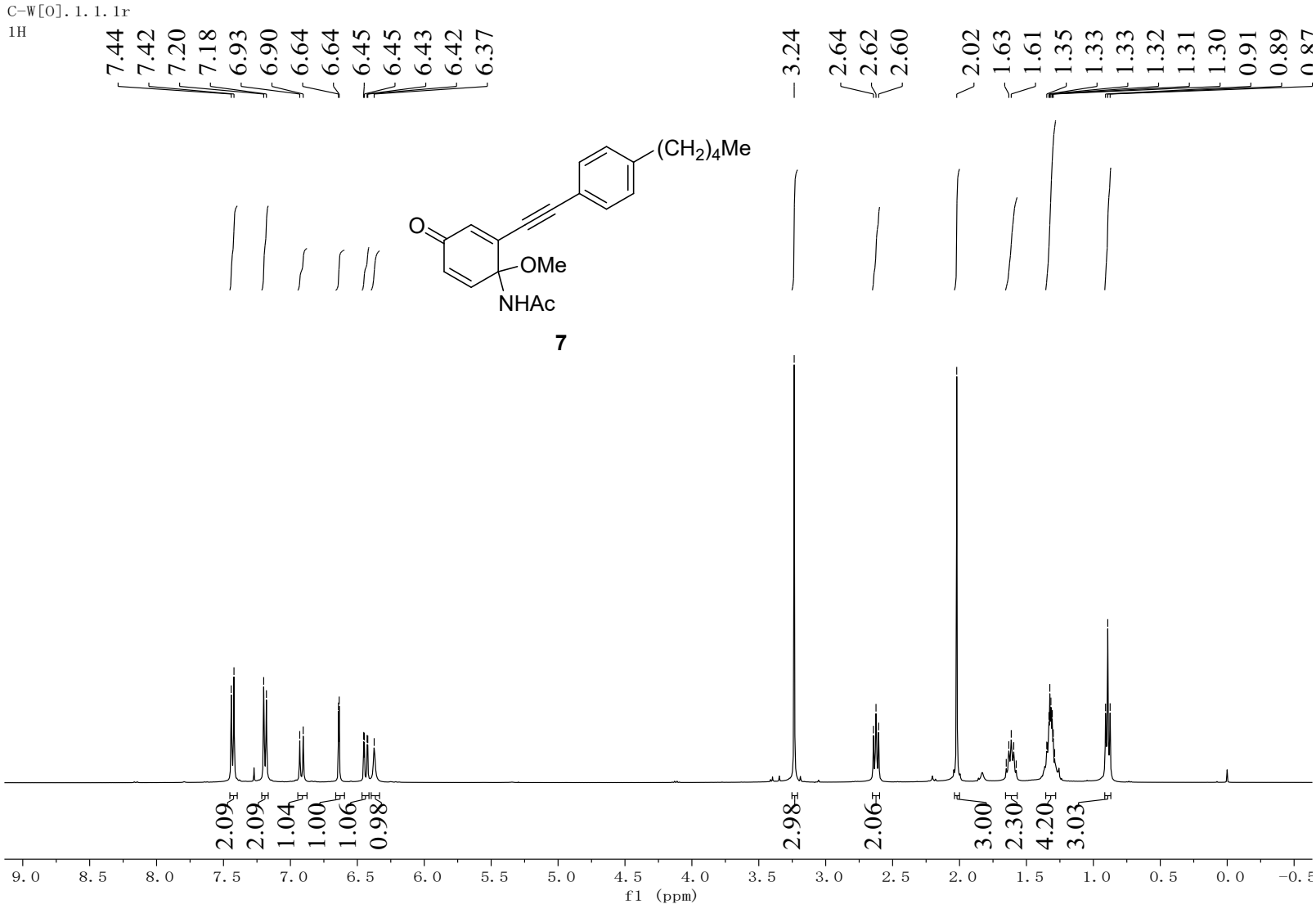

C-2Me[O]-C.1.1.1r
C-2Me[O]-C
184.62
168.74
144.51
140.91
139.11
134.22
132.41
130.99
129.99
129.77
125.90
121.41
100.06
88.03
80.58
51.01
23.61
20.69
O
Me
OMe
NHAc
4
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

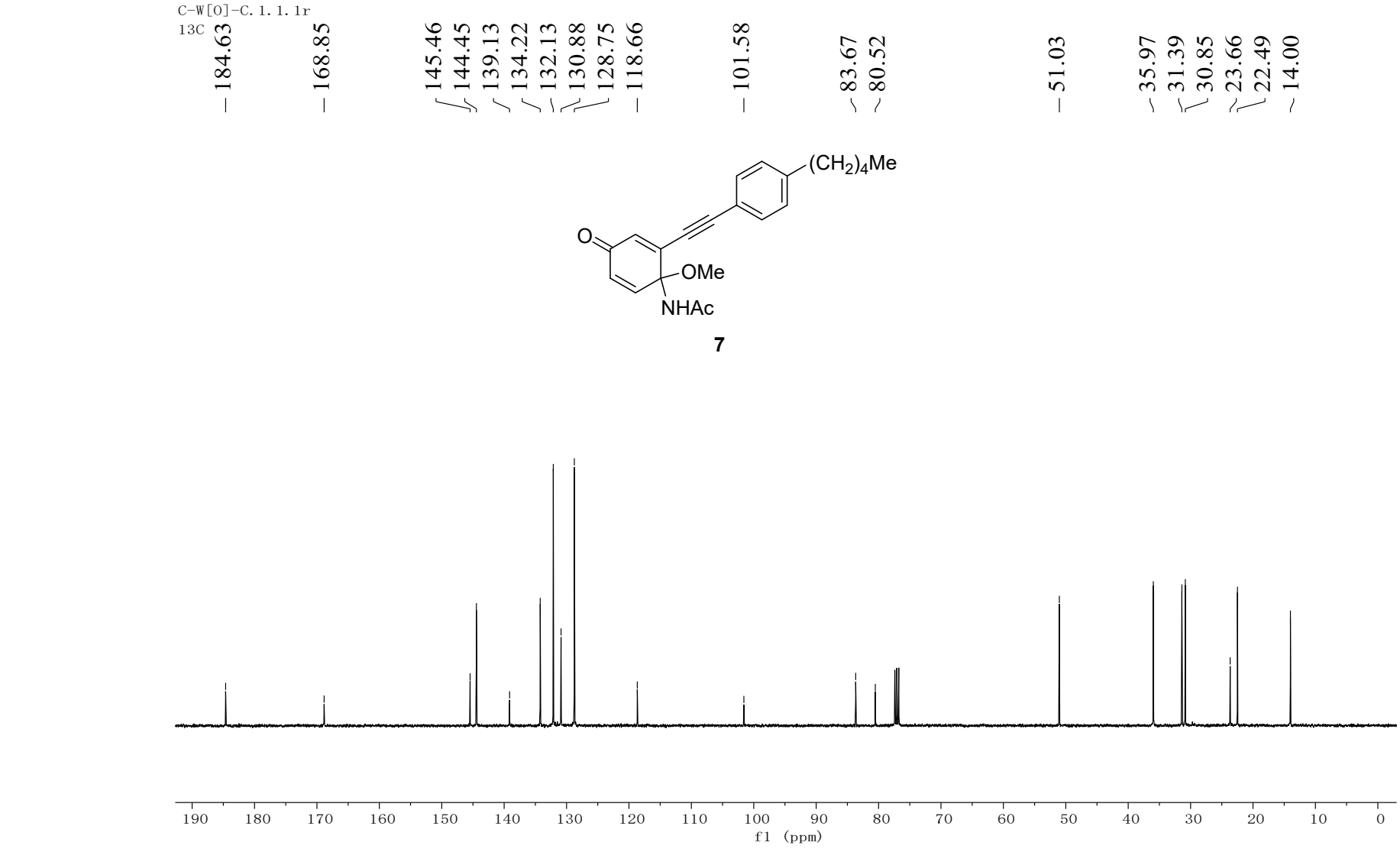

C-3Me[O]-NEW.1.1.1r
C-3Me[O]-NEW
7.33
7.31
7.28
7.26
7.24
7.22
7.21
6.89
6.86
6.65
6.64
6.57
6.45
6.44
6.42
6.42
3.23
2.35
2.02
O
Me
OMe
NHAc
5
2.00
1.09
1.01
1.02
1.00
0.96
1.01
3.00
3.02
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

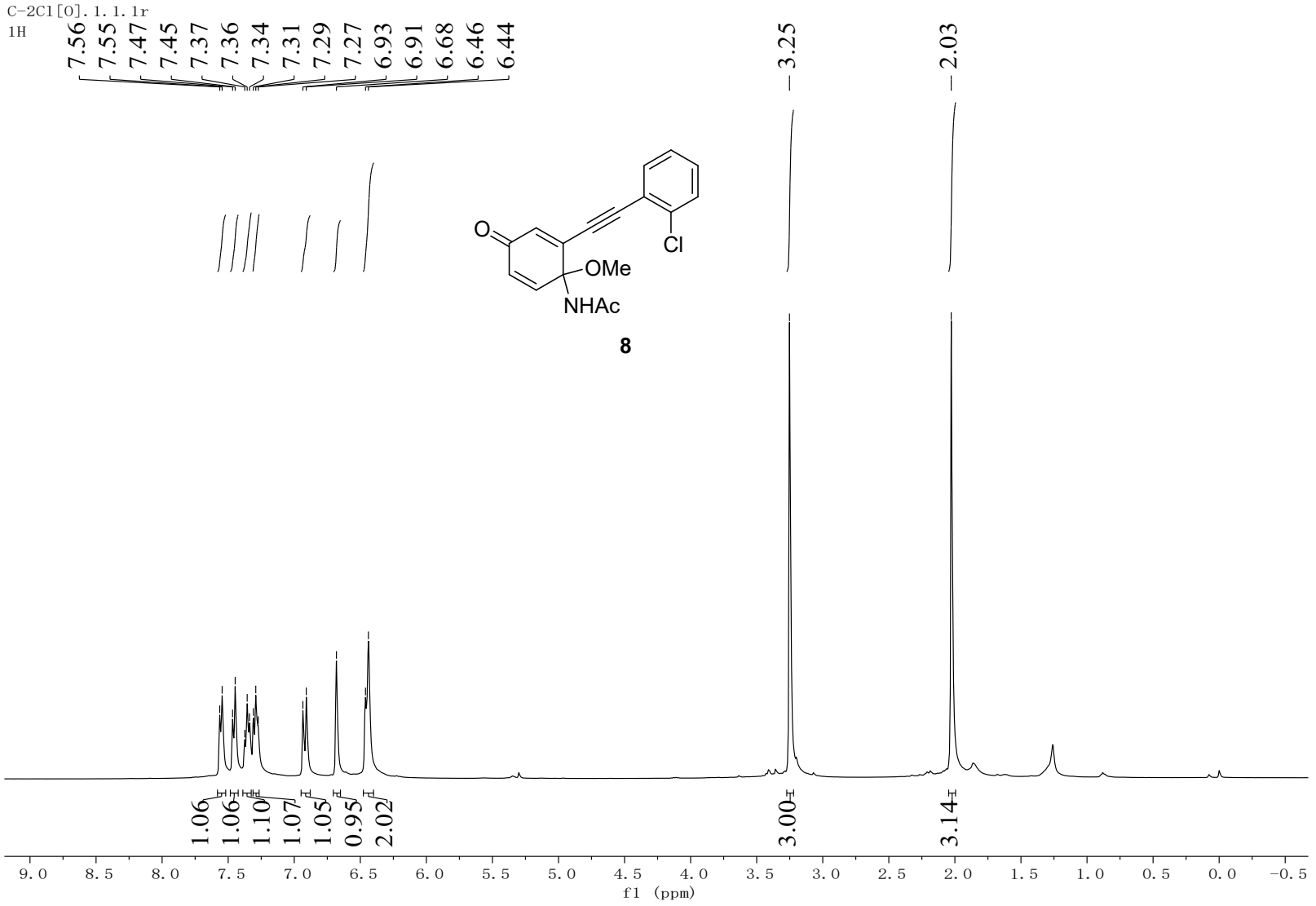

C-3Me[O]-C.1.1.1r
C-3Me[O]-C
184.68
168.97
144.67
139.18
138.39
134.40
132.62
130.86
130.80
129.28
128.51
121.37
101.30
83.85
80.56
50.97
23.56
21.20
O
Me
OMe
NHAc
5
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

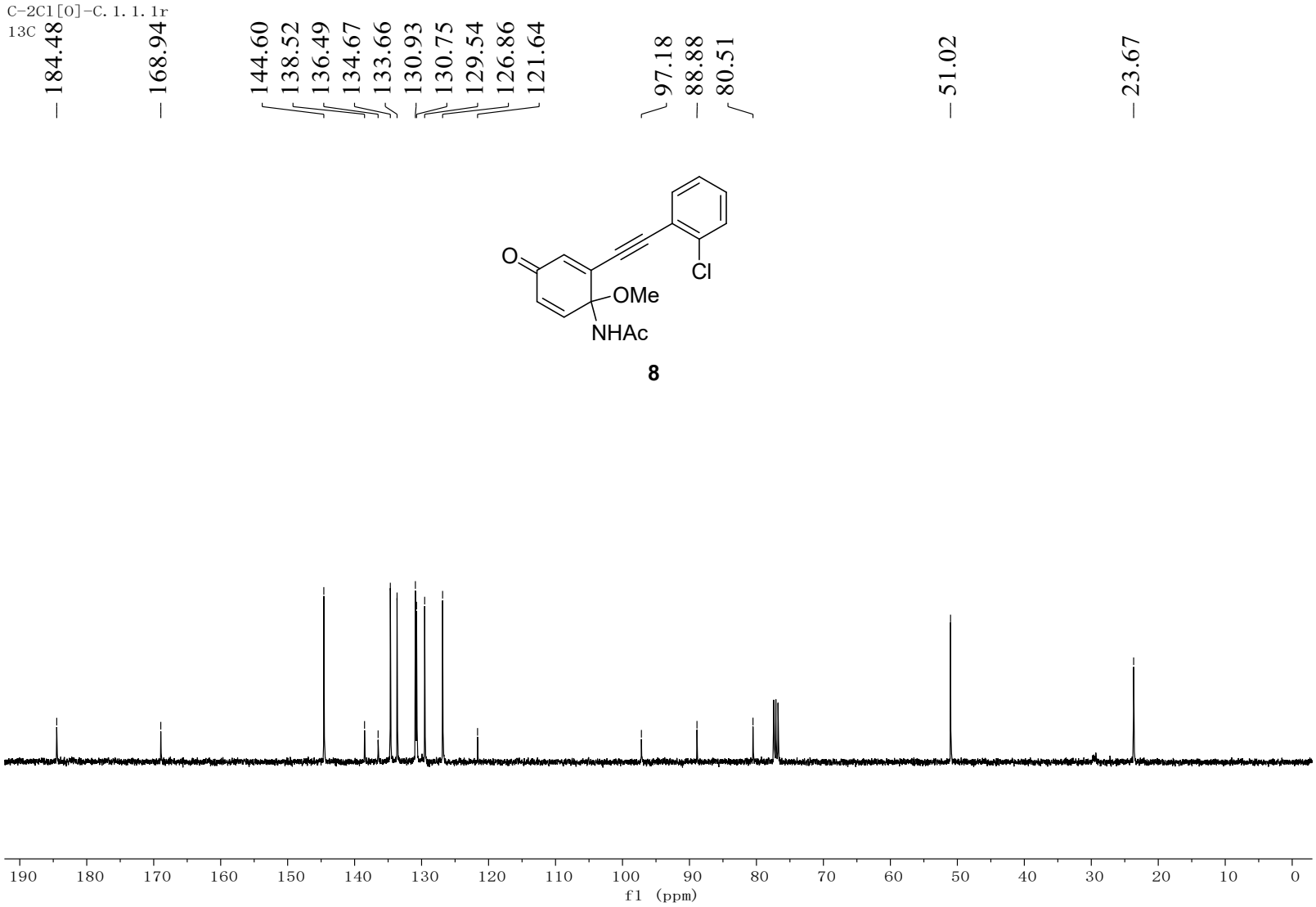

C-Me[O].1.1.1r
1H
7.42
7.40
7.19
7.17
6.90
6.87
6.64
6.63
6.52
6.45
6.44
6.42
6.42
3.23
2.38
2.02
Me
O
OMe
NHAc
6
2.05
2.02
1.00
1.00
0.97
1.01
3.04
3.00
3.02
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

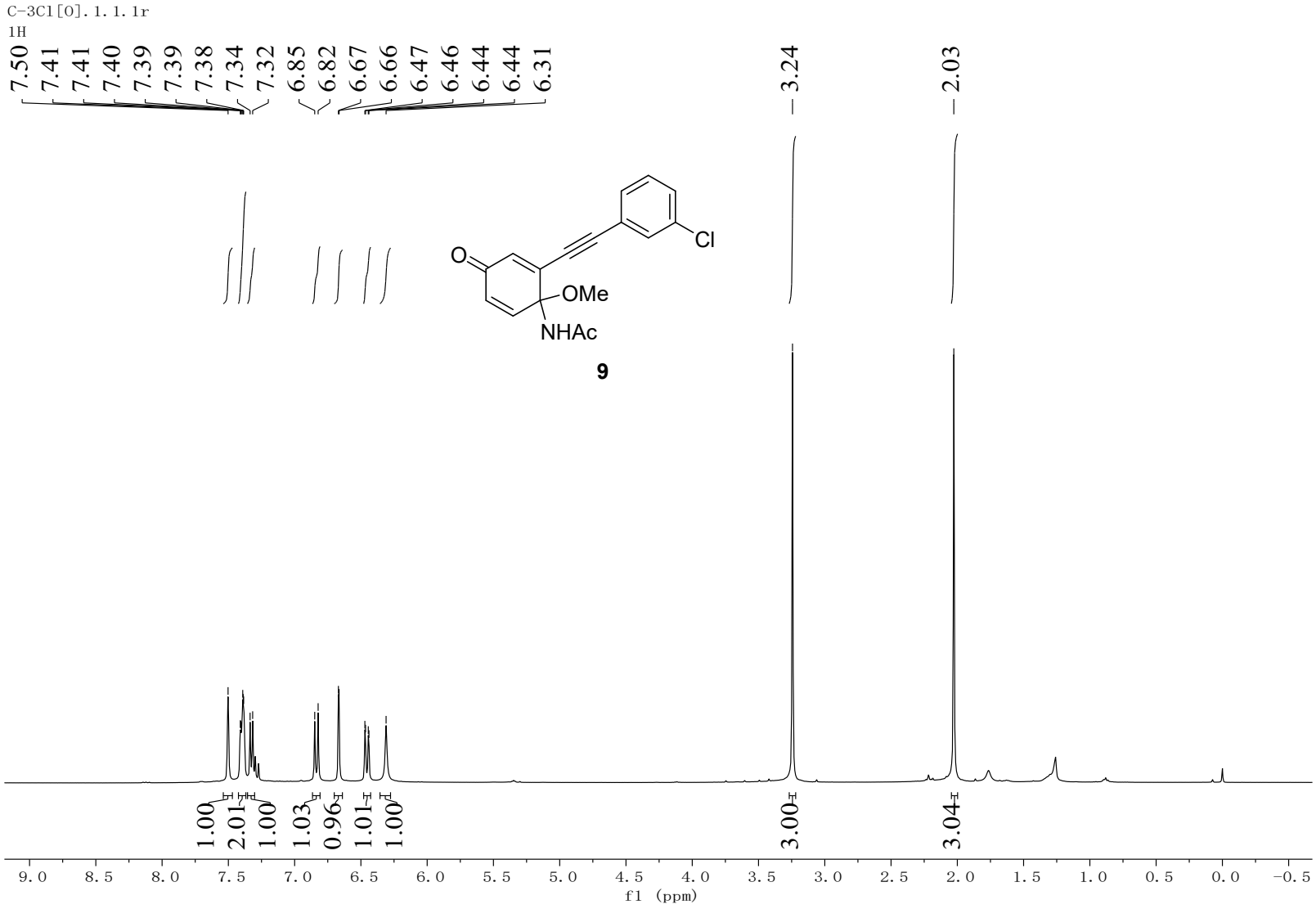

C-Me[O]-C.1.1.1r
13C
184.7
169.0
144.6
140.4
139.2
134.2
132.1
130.9
129.4
118.5
101.5
83.7
80.5
51.0
23.6
21.7
Me
O
OMe
NHAc
6
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

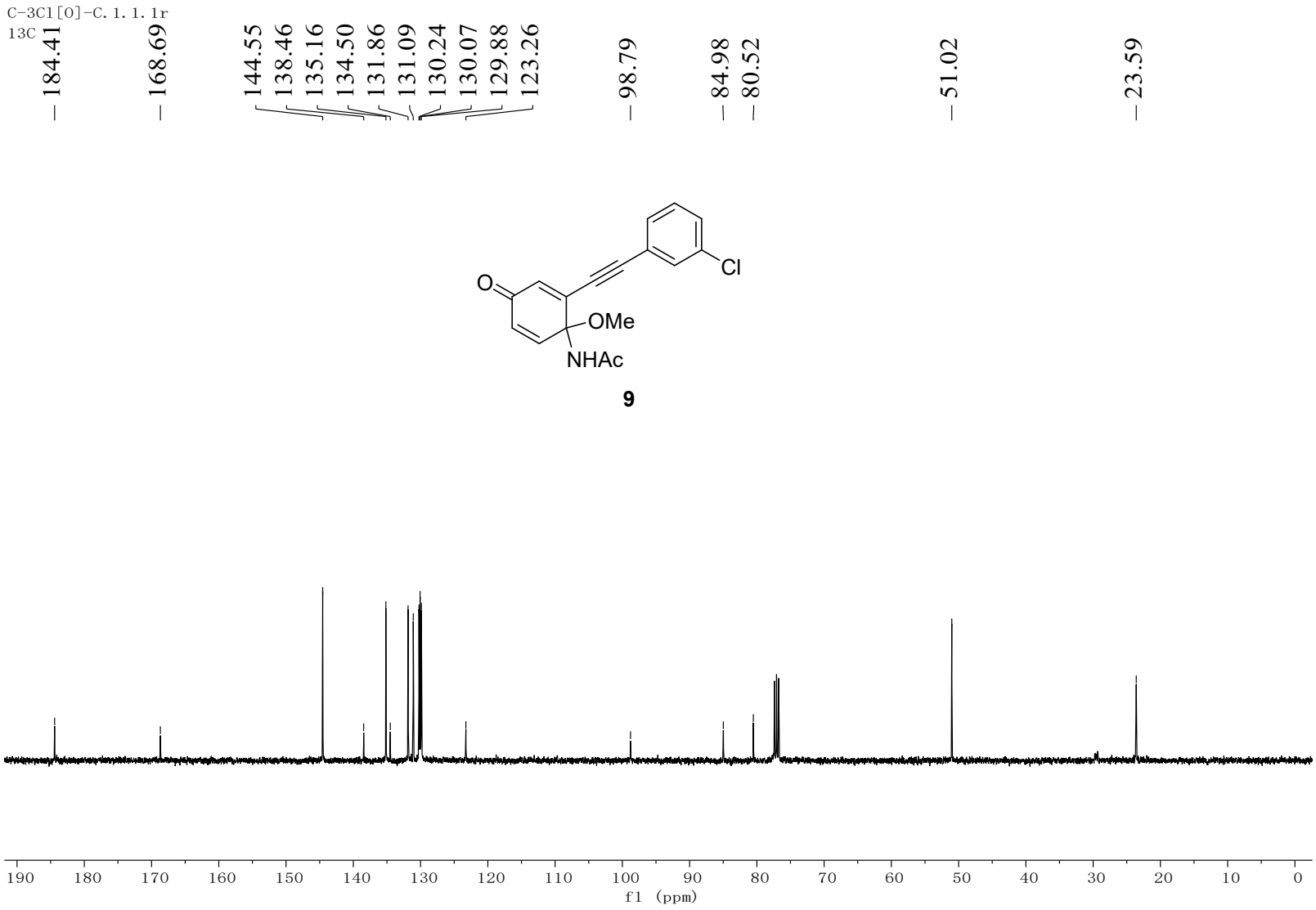

C-W[O].1.1.1r
1H
7.44
7.42
7.20
7.18
6.93
6.90
6.64
6.64
6.45
6.45
6.43
6.42
6.37
3.24
2.64
2.62
2.60
2.02
1.63
1.61
1.35
1.33
1.33
1.32
1.31
1.30
0.91
0.89
(CH2)4Me
O
OMe
NHAc
7
2.09
2.09
1.04
1.00
1.06
0.98
2.98
2.06
3.00
2.30
4.20
3.03
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


C-W[O]-C.1.1.1r
13C
184.63
168.85
145.46
144.45
139.13
134.22
132.13
130.88
128.75
118.66
101.58
83.67
80.52
51.03
35.97
31.39
30.85
23.66
22.49
14.00
(CH2)4Me
O
OMe
NHAc
7
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

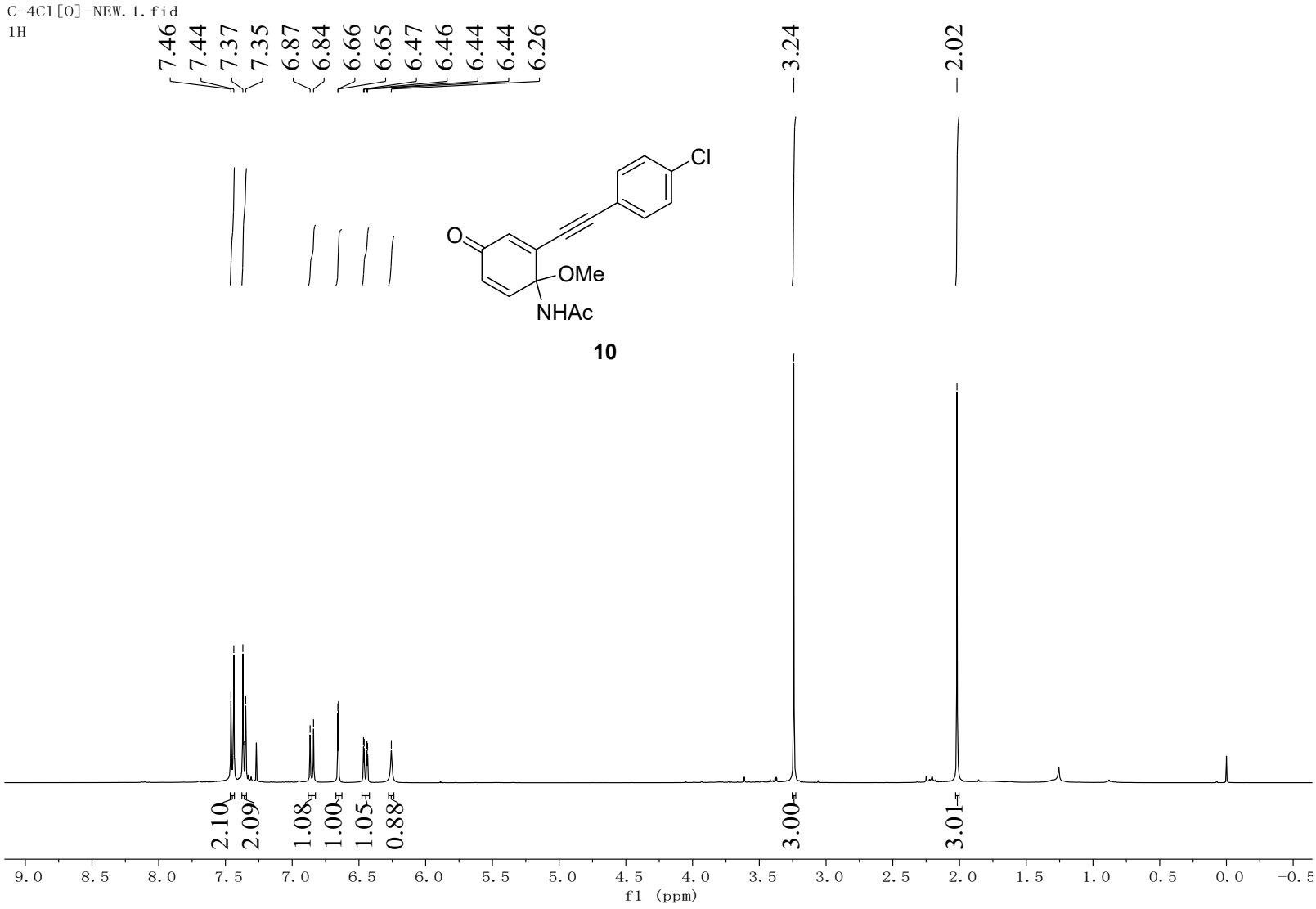

C-2Cl[O].1.1.1r
1H
7.56
7.55
7.47
7.45
7.37
7.36
7.34
7.31
7.29
7.27
6.93
6.91
6.68
6.46
6.44
3.25
2.03
O
Cl
OMe
NHAc
8
1.06
1.06
1.10
1.07
1.05
0.95
2.02
3.00
3.14
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

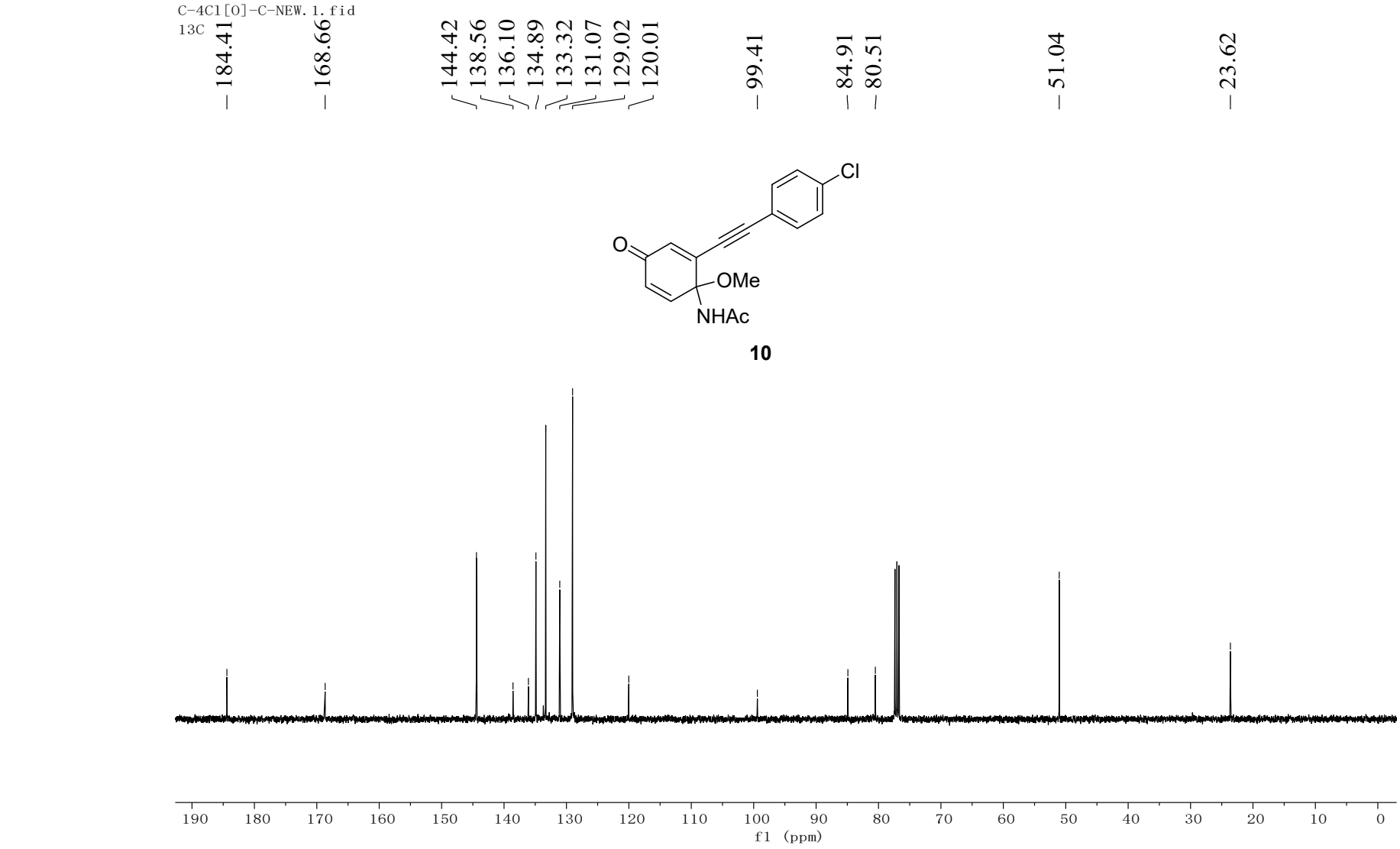

C-2Cl[O]-C. 1. 1. 1r
13C
184.48
168.94
144.60
138.52
136.49
134.67
133.66
130.93
130.75
129.54
126.86
121.64
97.18
88.88
80.51
51.02
23.67
O
Cl
OMe
NHAc
8
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

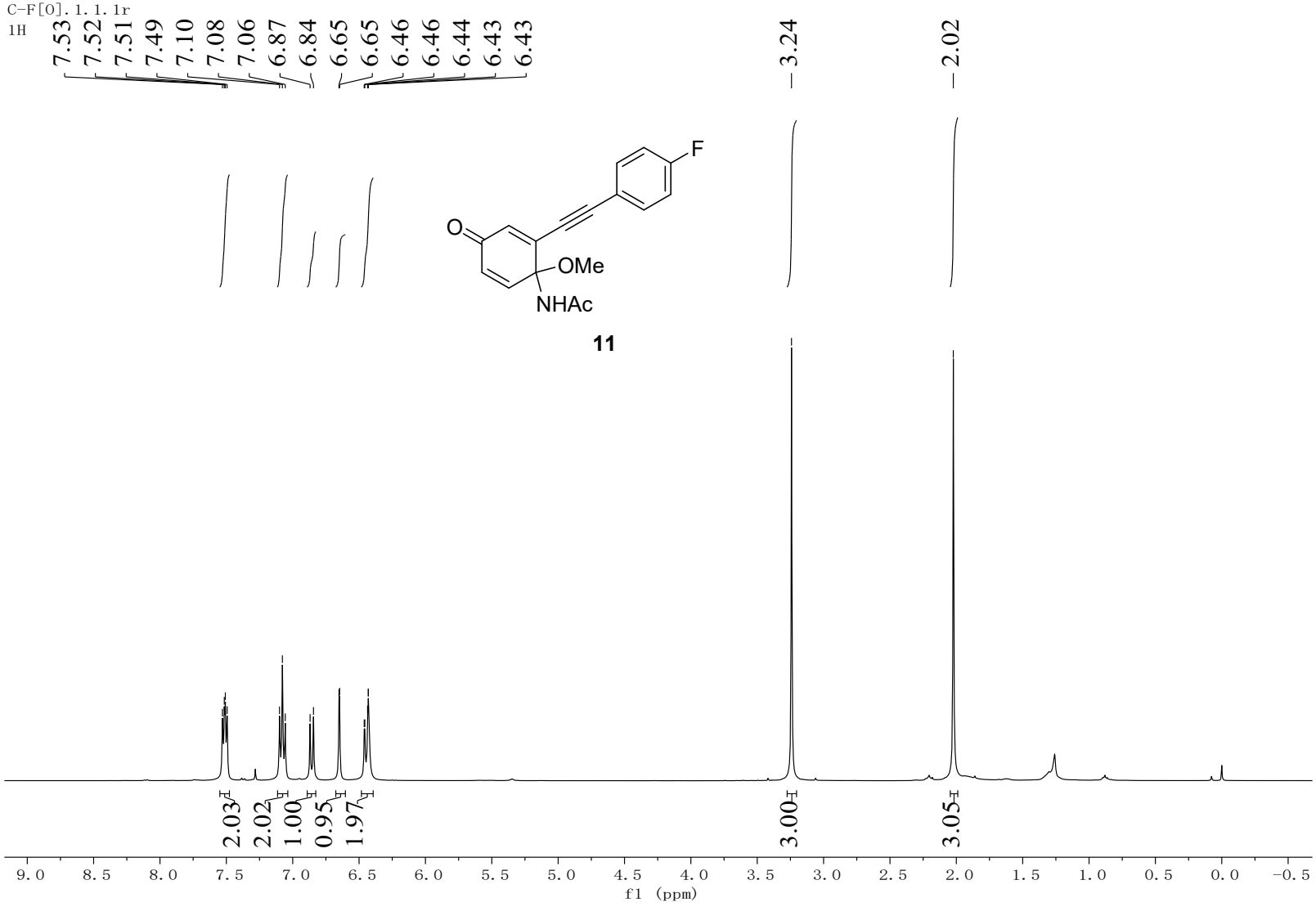

C-3Cl[O].1.1.1r
1H
7.50
7.41
7.41
7.40
7.39
7.39
7.38
7.34
7.32
6.85
6.82
6.67
6.66
6.47
6.46
6.44
6.44
6.31
3.24
2.03
O
Cl
OMe
NHAc
9
1.00
2.01
1.00
1.03
0.96
1.01
1.00
3.00
3.04
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

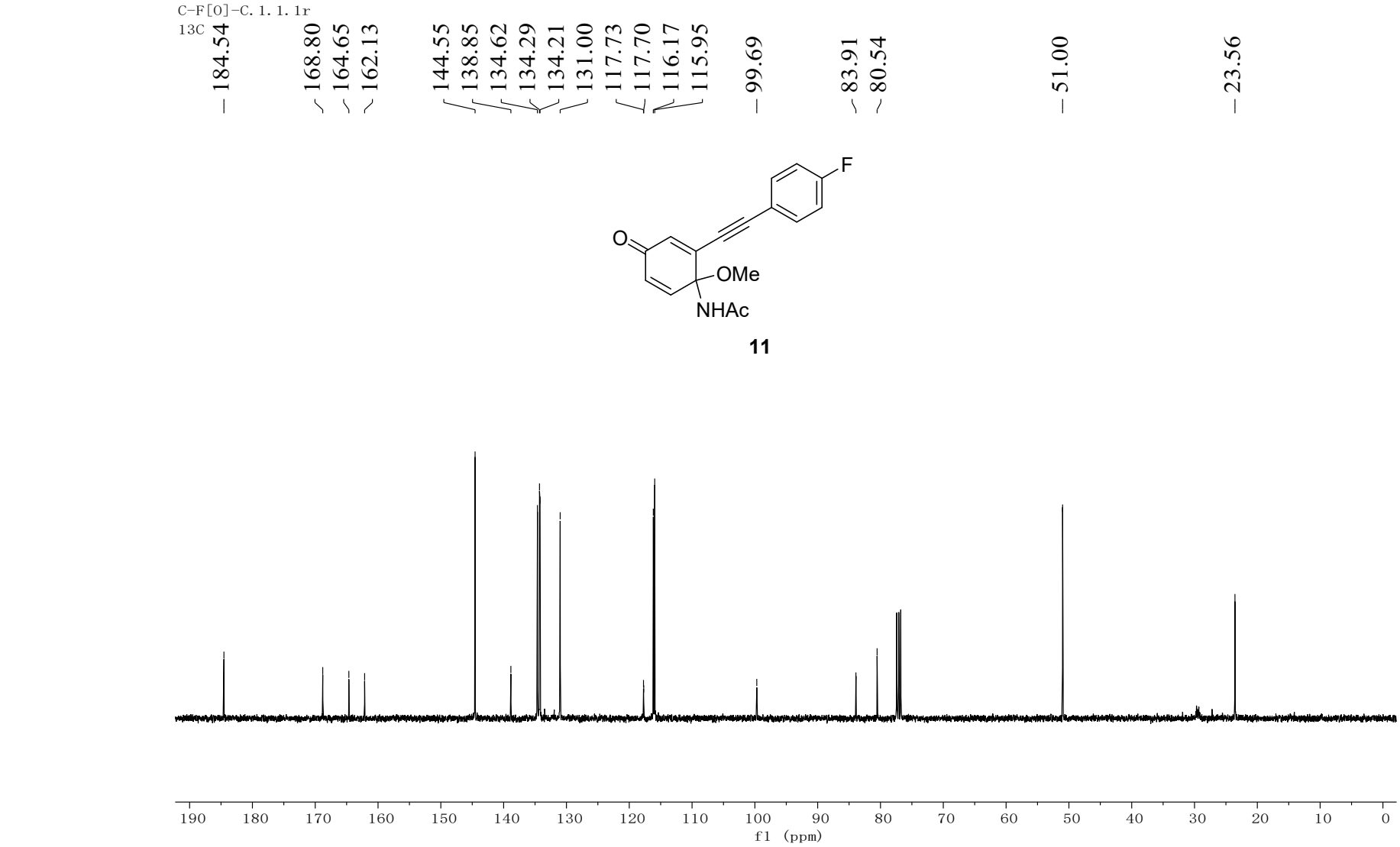

C-3Cl[O]-C.1.1.1r
13C
184.41
168.69
144.55
138.46
135.16
134.50
131.86
131.09
130.24
130.07
129.88
123.26
98.79
84.98
80.52
51.02
23.59
O
Cl
OMe
NHAc
f1 (ppm)

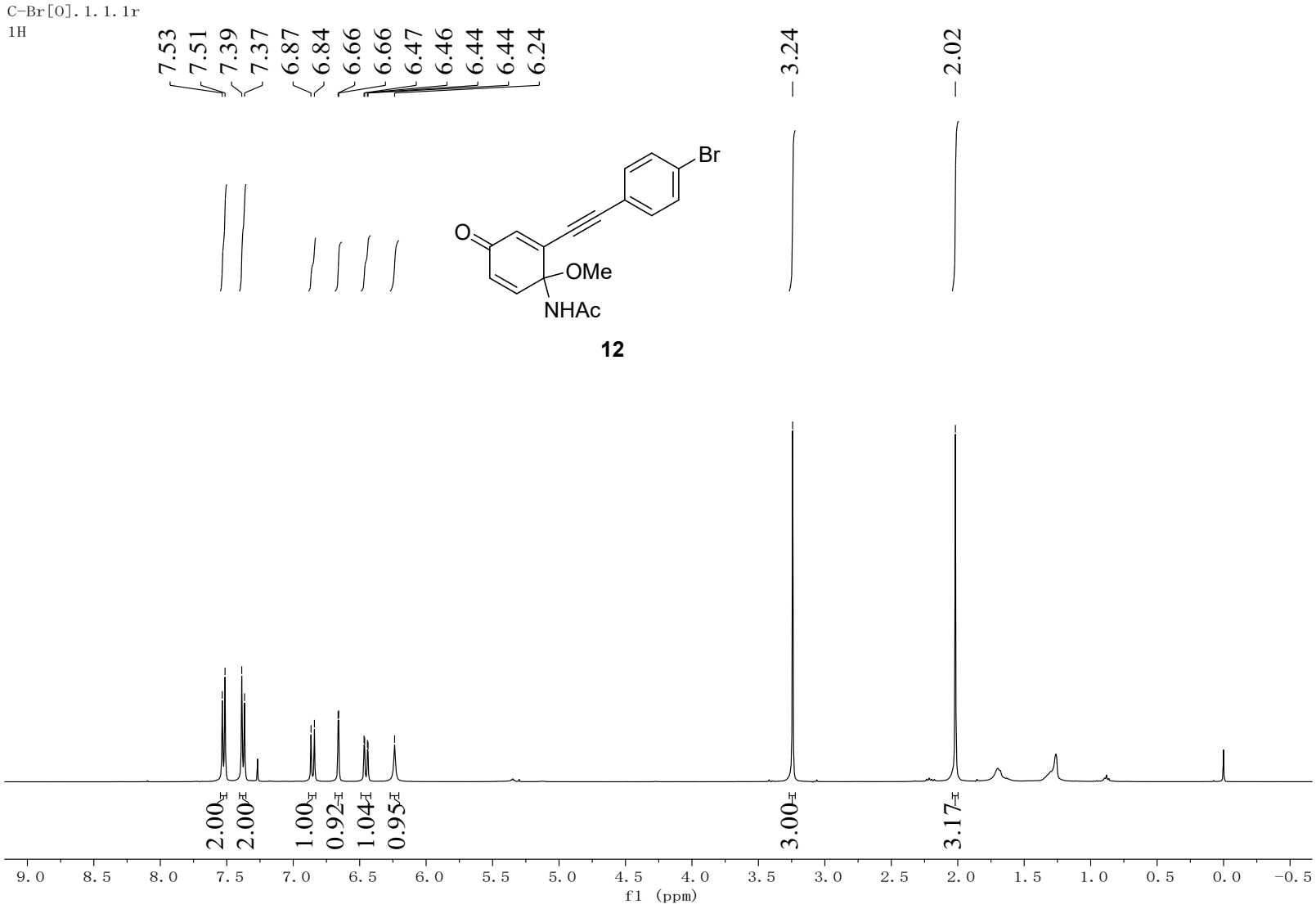
9

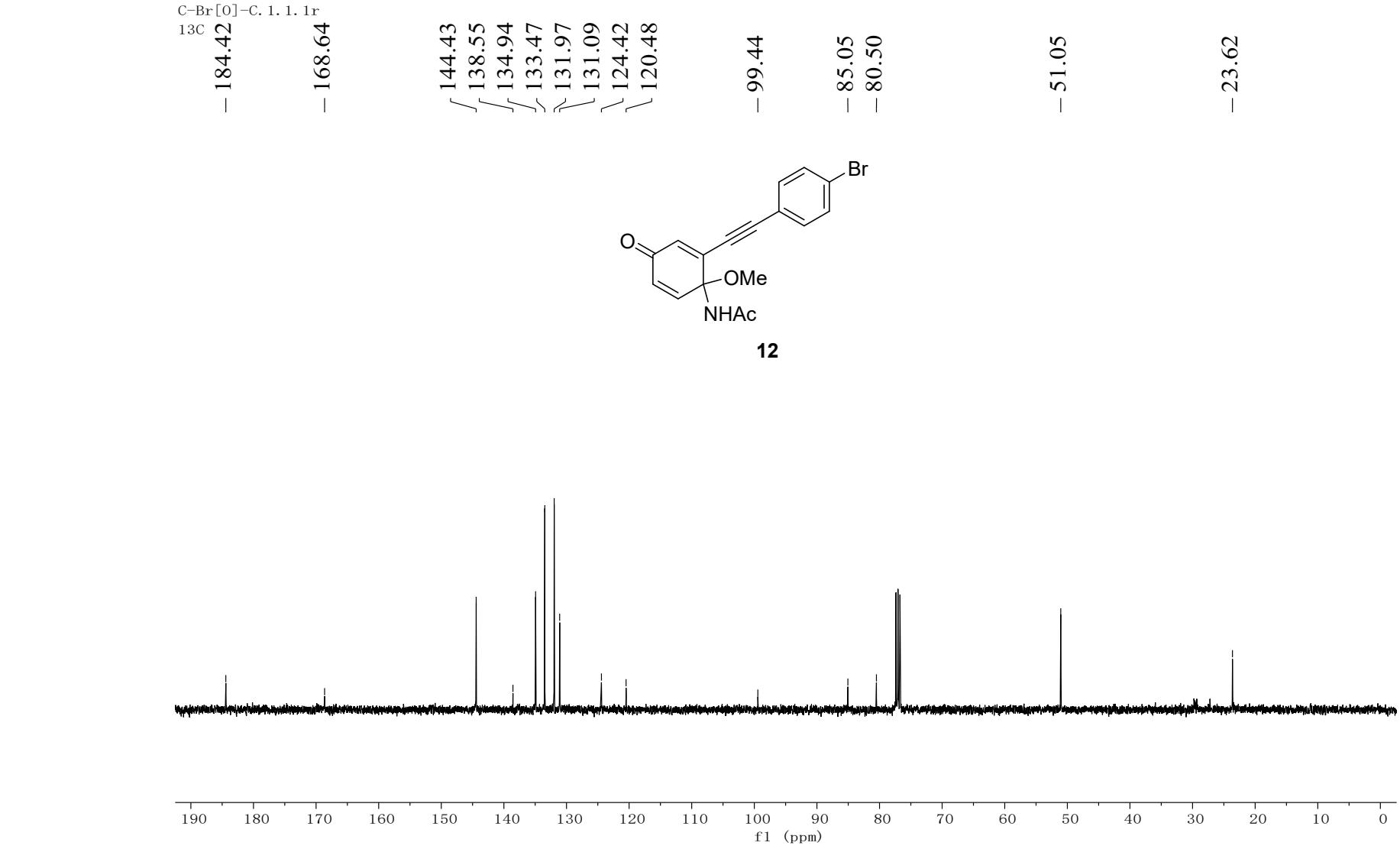

C-4Cl[O]-NEW.1.fid
1H
7.46
7.44
7.37
7.35
6.87
6.84
6.66
6.65
6.47
6.46
6.44
6.44
6.26
3.24
2.02
Cl
O
OMe
NHAc
10
2.10
2.09
1.08
1.00
1.05
0.88
3.00
3.01
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


C-4Cl[O]-C-NEW.1.fid
13C
184.41
168.66
144.42
138.56
136.10
134.89
133.32
131.07
129.02
120.01
99.41
84.91
80.51
51.04
23.62
Cl
O
OMe
NHAc
10
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

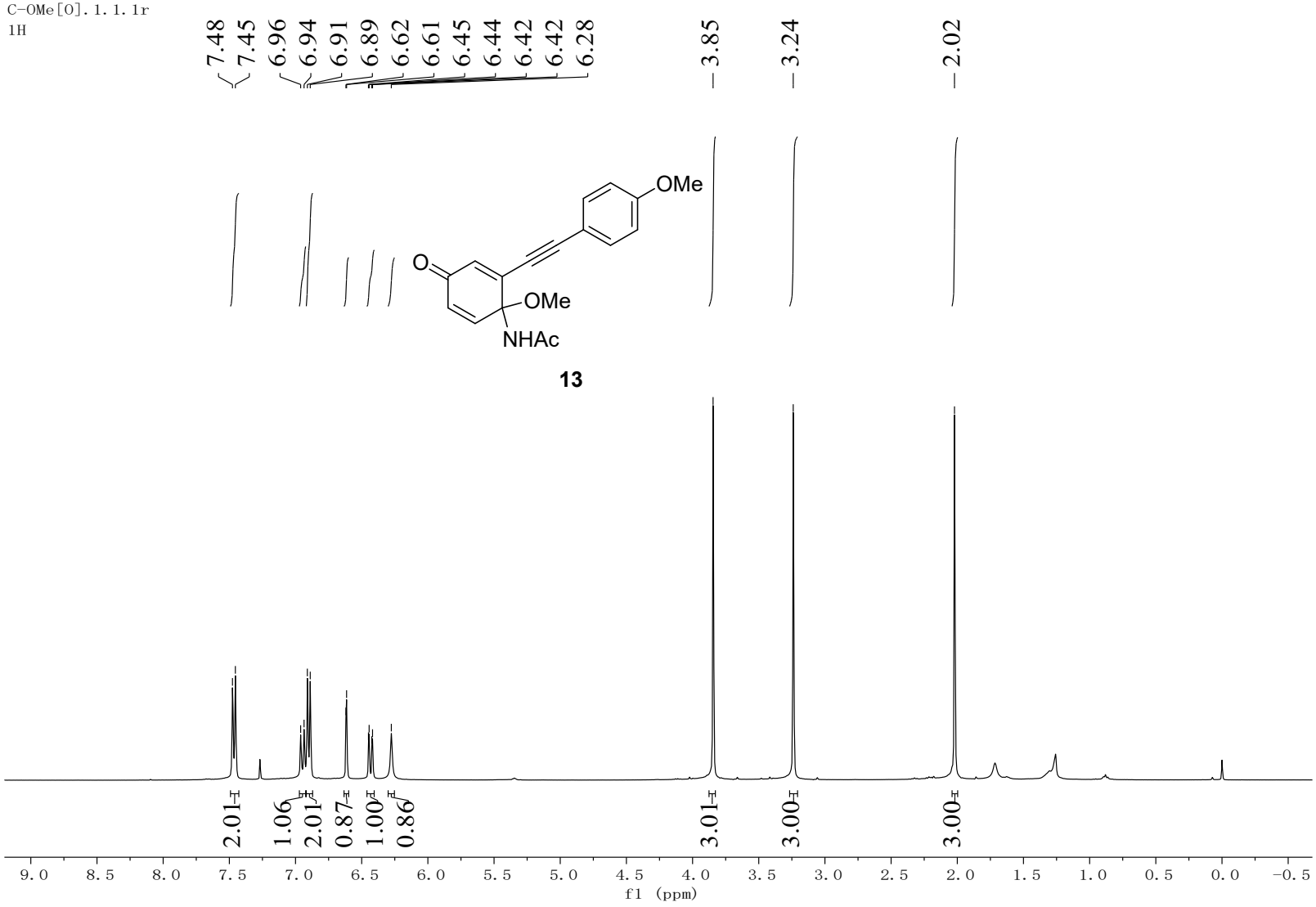

C-F[O].1.1.1r
1H
7.53
7.52
7.51
7.49
7.10
7.08
7.06
6.87
6.84
6.65
6.65
6.46
6.46
6.44
6.43
6.43
3.24
2.02
F
O
OMe
NHAc
11
2.03
2.02
1.00
0.95
1.97
3.00
3.05
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

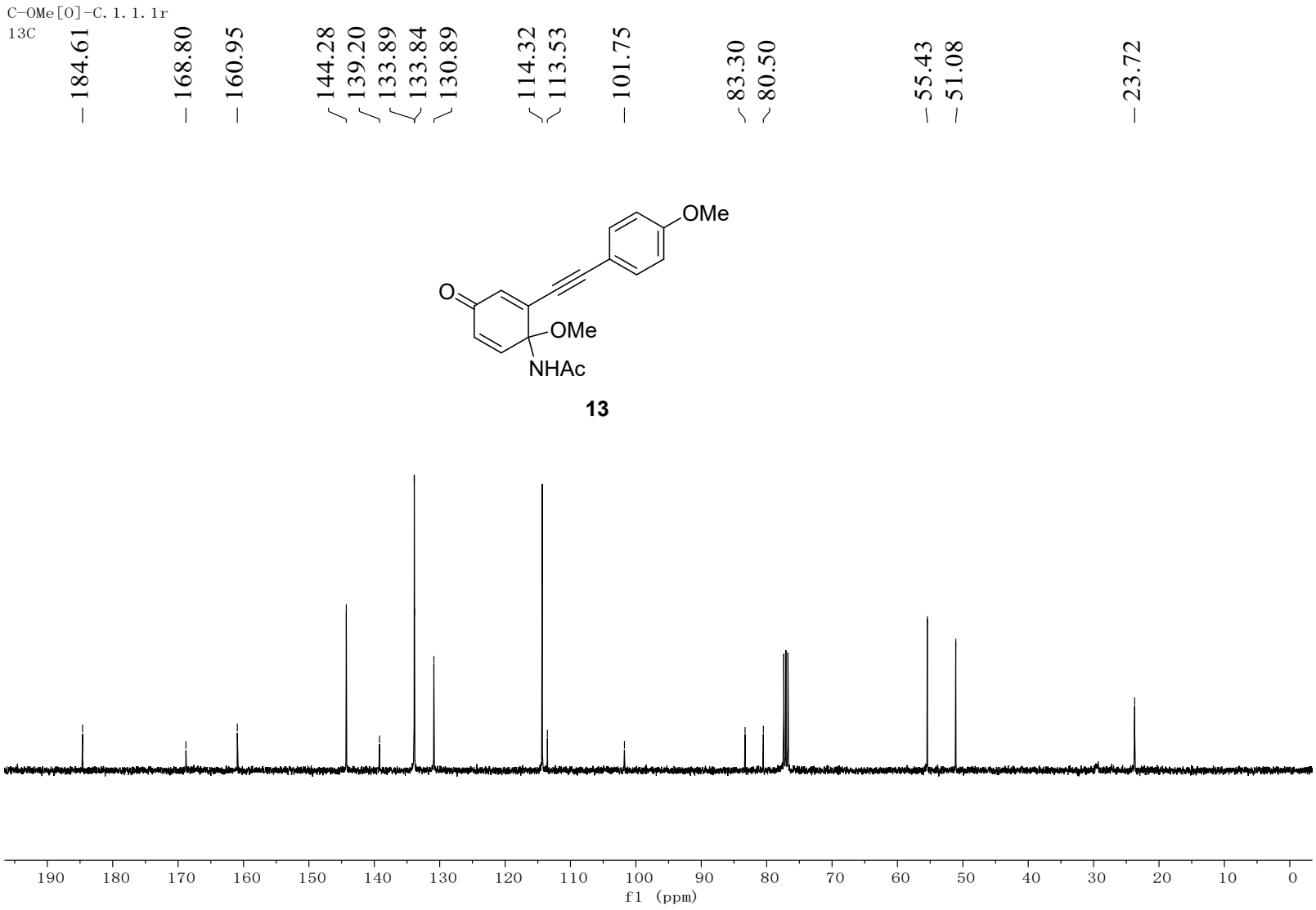

C-F[O]-C. 1. 1. 1r
13C
184.54
168.80
164.65
162.13
144.55
138.85
134.62
134.29
134.21
131.00
117.73
117.70
116.17
115.95
99.69
83.91
80.54
51.00
23.56
F
O
OMe
NHAc
11
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


C-Br[O].1.1.1r
1H
7.53
7.51
7.39
7.37
6.87
6.84
6.66
6.66
6.47
6.46
6.44
6.44
6.24
3.24
2.02
Br
O
OMe
NHAc
12
2.00
2.00
1.00
0.92
1.04
0.95
3.00
3.17
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 -0.5
f1 (ppm)

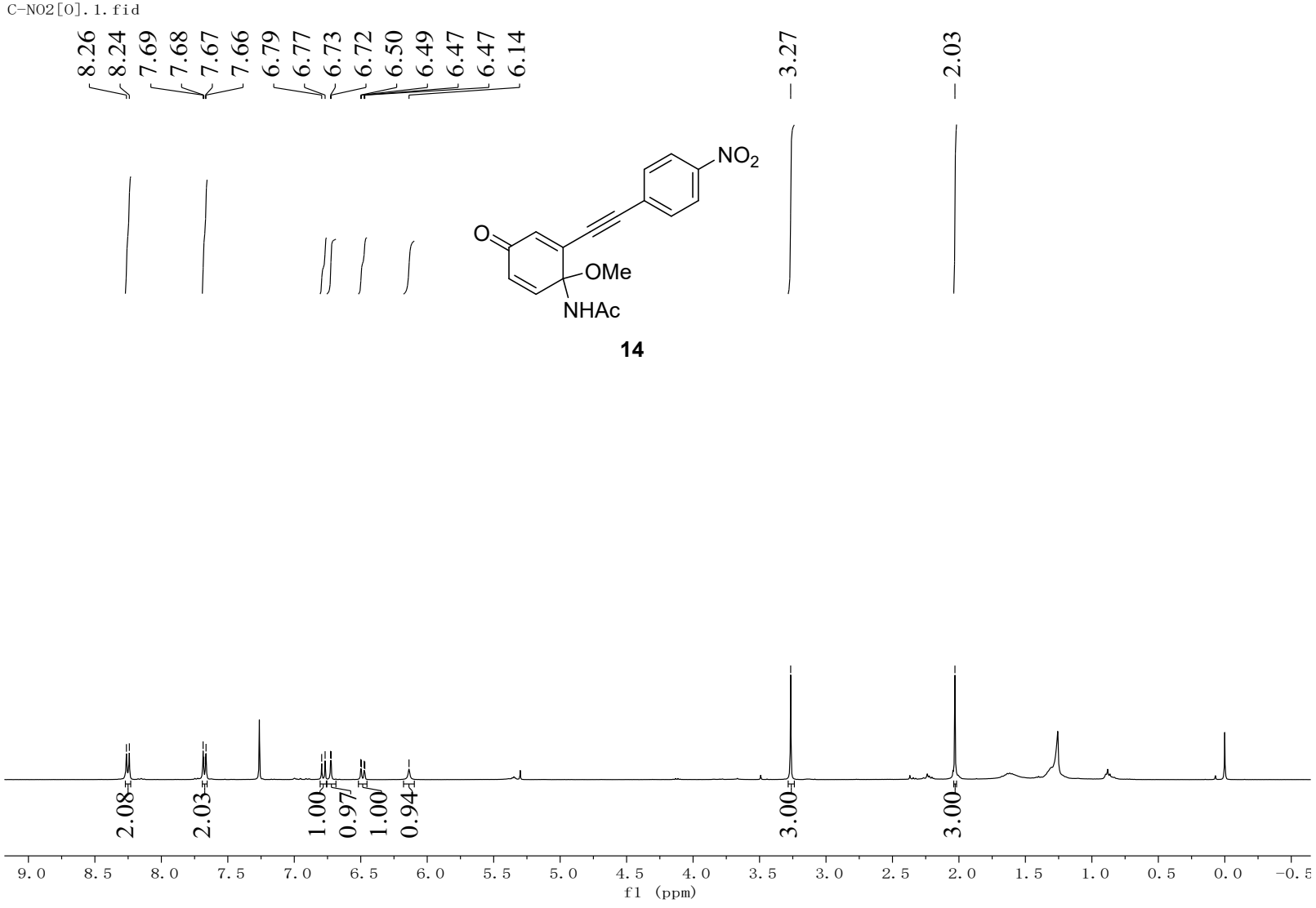

C-Br[O]-C.1.1.1r
13C
184.42
168.64
144.43
138.55
134.94
133.47
131.97
131.09
124.42
120.48
99.44
85.05
80.50
51.05
23.62
Br
O
OMe
NHAc
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

12


C-OMe[O].1.1.1r
1H
7.48
7.45
6.96
6.94
6.91
6.89
6.62
6.61
6.45
6.44
6.42
6.42
6.28
3.85
3.24
2.02
OMe
O
OMe
NHAc
13
2.01
1.06
2.01
0.87
1.00
0.86
3.01
3.00
3.00
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 -0.5
f1 (ppm)


C-OMe[O]-C.1.1.1r
13C
184.61
168.80
160.95
144.28
139.20
133.89
133.84
130.89
114.32
113.53
101.75
83.30
80.50
55.43
51.08
23.72
OMe
O
OMe
NHAc
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

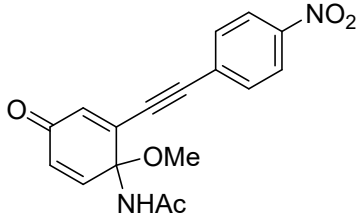
13


C-NO2[O].1.fid
8.26
8.24
7.69
7.68
7.67
7.66
6.79
6.77
6.73
6.72
6.50
6.49
6.47
6.47
6.14
3.27
2.03
NO2
O
OMe
NHAc
2.08
2.03
1.00
0.97
1.00
0.94
3.00
3.00
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
−0.5
f1 (ppm)

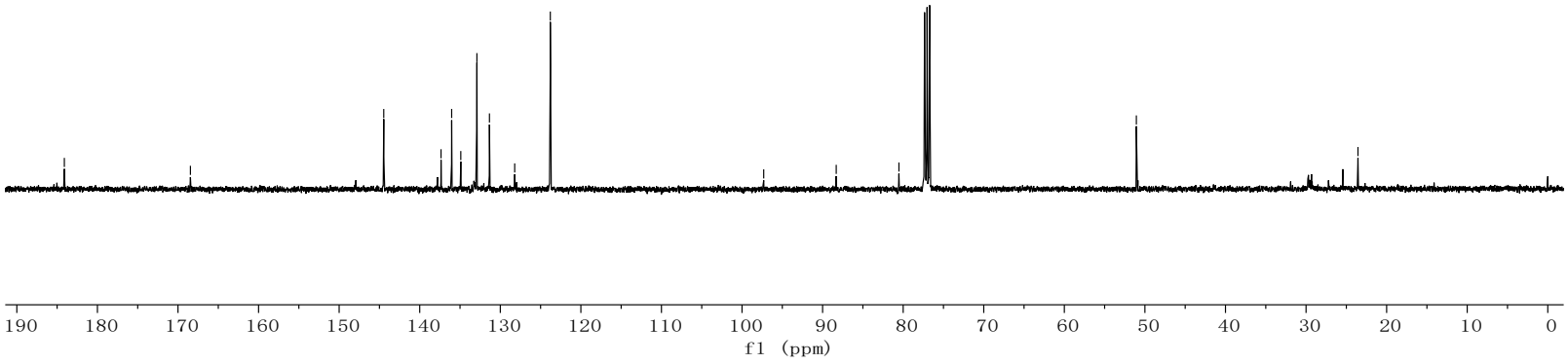
14

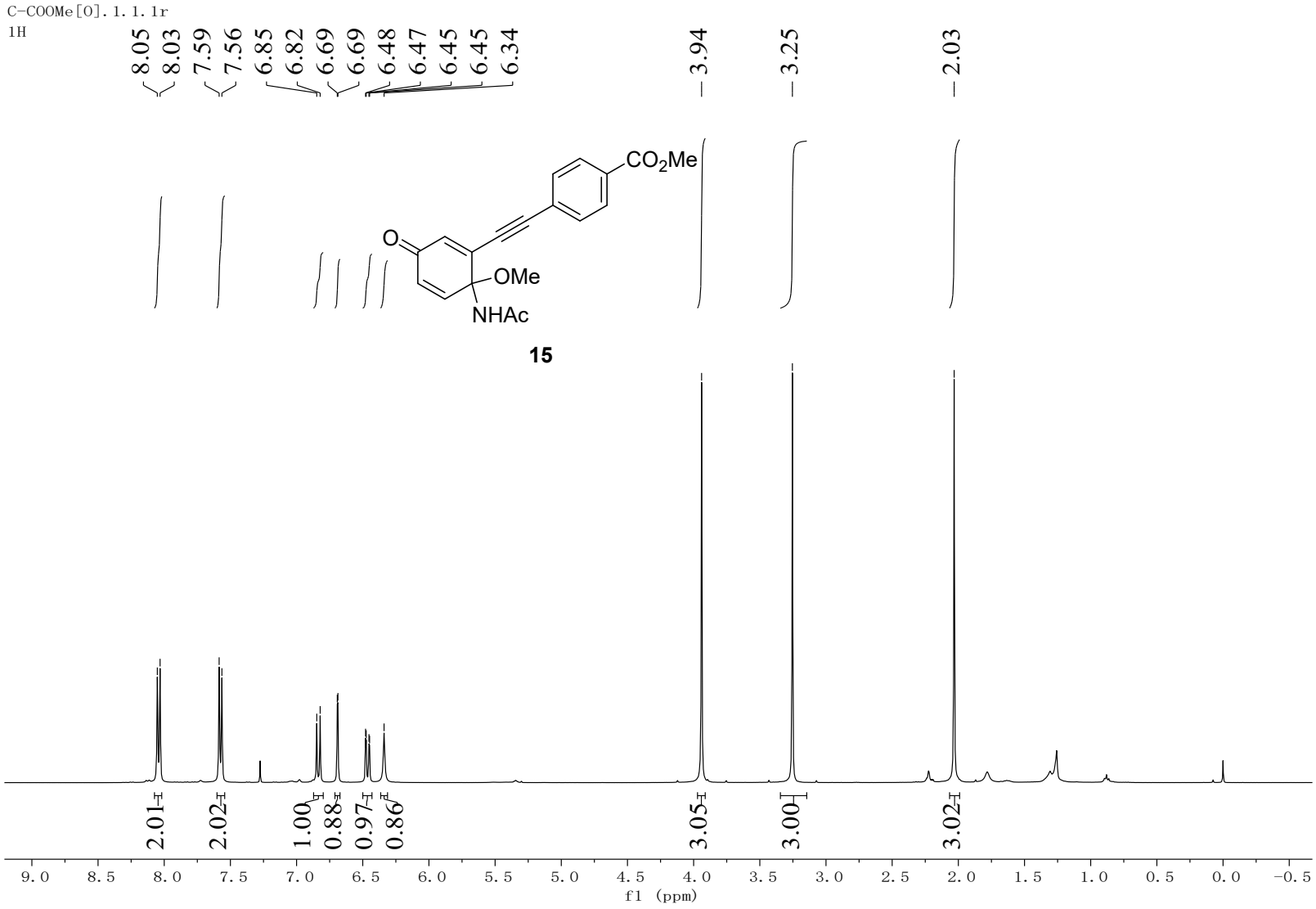
C-NO2[O]-FIN-C.1.fid

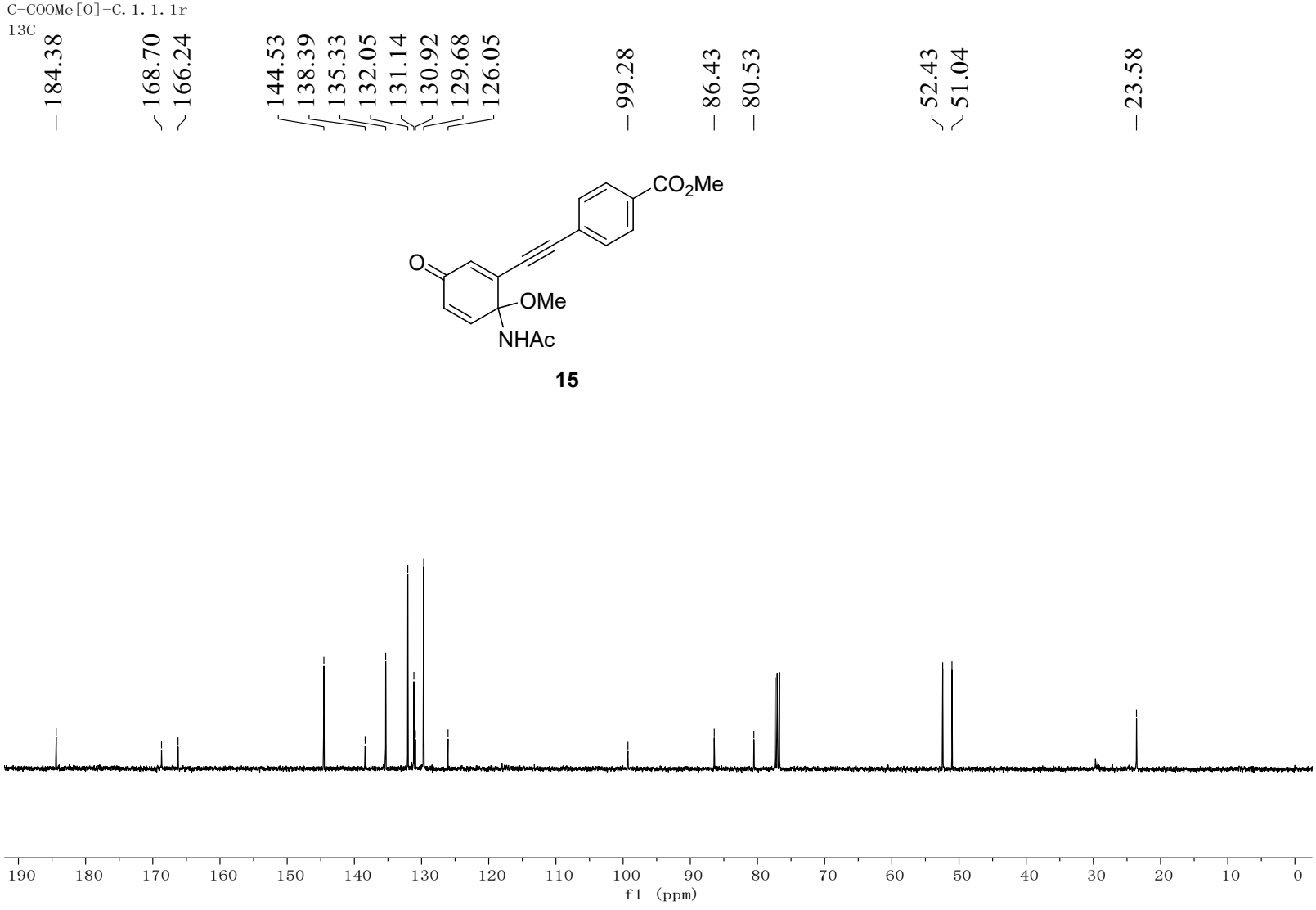
184.10
168.46
144.46
137.35
136.04
134.90
132.90
131.35
128.21
123.79
97.31
88.31
80.53
51.06
23.56

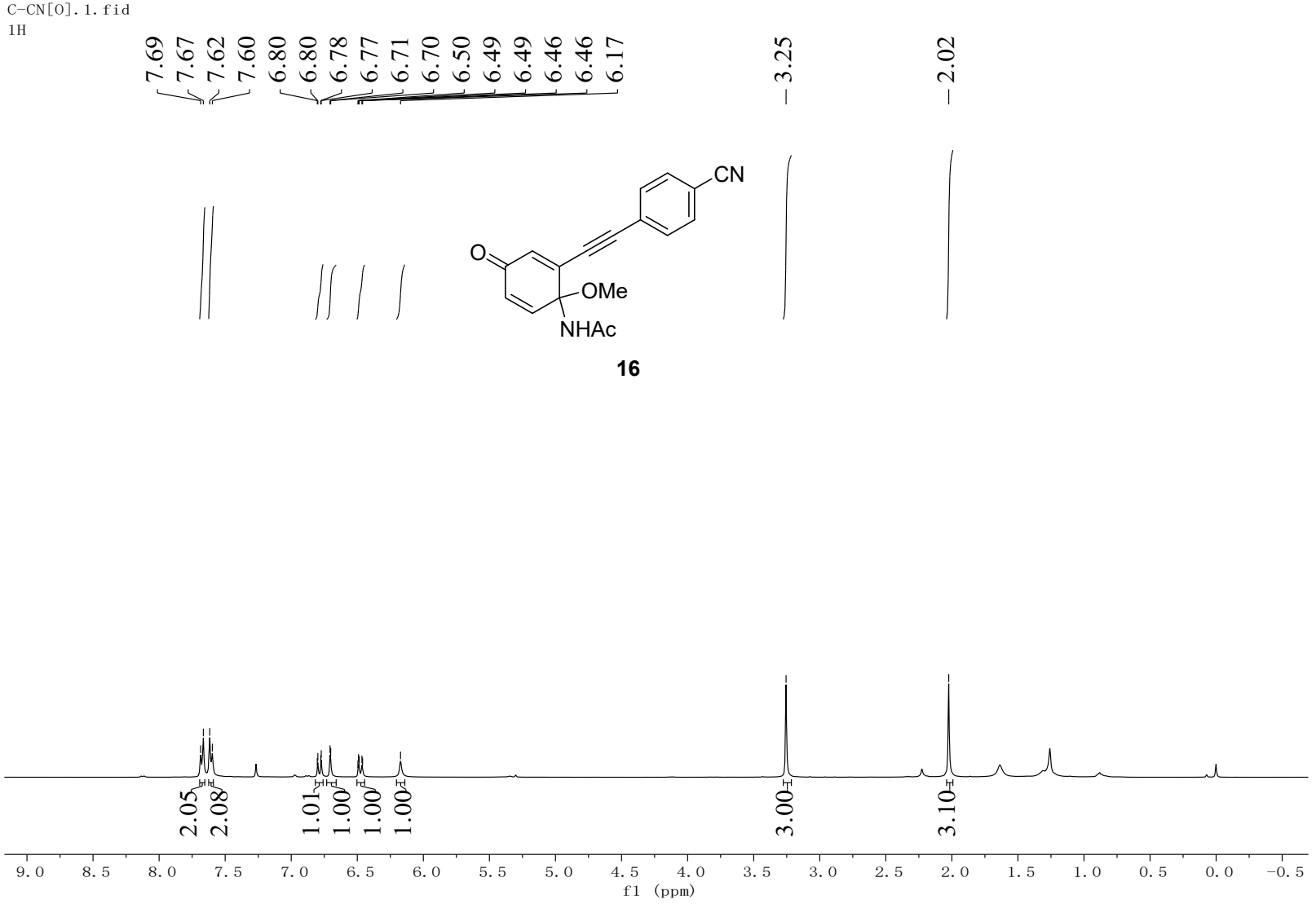


14




C-COOMe[O].1.1.1r
1H
8.05
8.03
7.59
7.56
6.85
6.82
6.69
6.69
6.48
6.47
6.45
6.45
6.34
3.94
3.25
2.03
CO2Me
O
OMe
NHAc
15
2.01
2.02
1.00
0.88
0.97
0.86
3.05
3.00
3.02
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
-0.5
f1 (ppm)

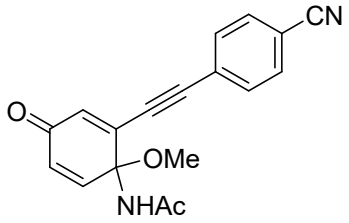

C-COOMe[O]-C.1.1.1r
13C
184.38
168.70
166.24
144.53
138.39
135.33
132.05
131.14
130.92
129.68
126.05
99.28
86.43
80.53
52.43
51.04
23.58
CO2Me
O
OMe
NHAc
15
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


C-CN[O].1.fid
1H
7.69
7.67
7.62
7.60
6.80
6.80
6.78
6.77
6.71
6.70
6.50
6.49
6.49
6.46
6.46
6.17
3.25
2.02
CN
O
OMe
NHAc
2.05
2.08
1.01
1.00
1.00
1.00
3.00
3.10
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
-0.5
f1 (ppm)

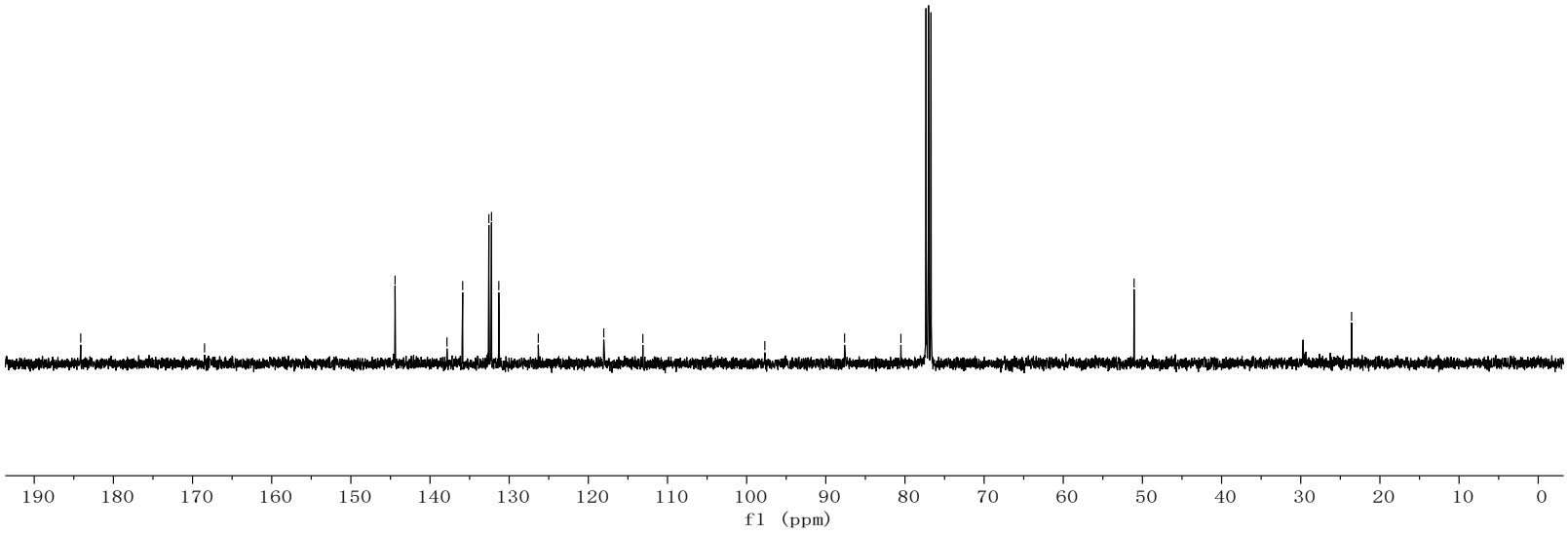
16

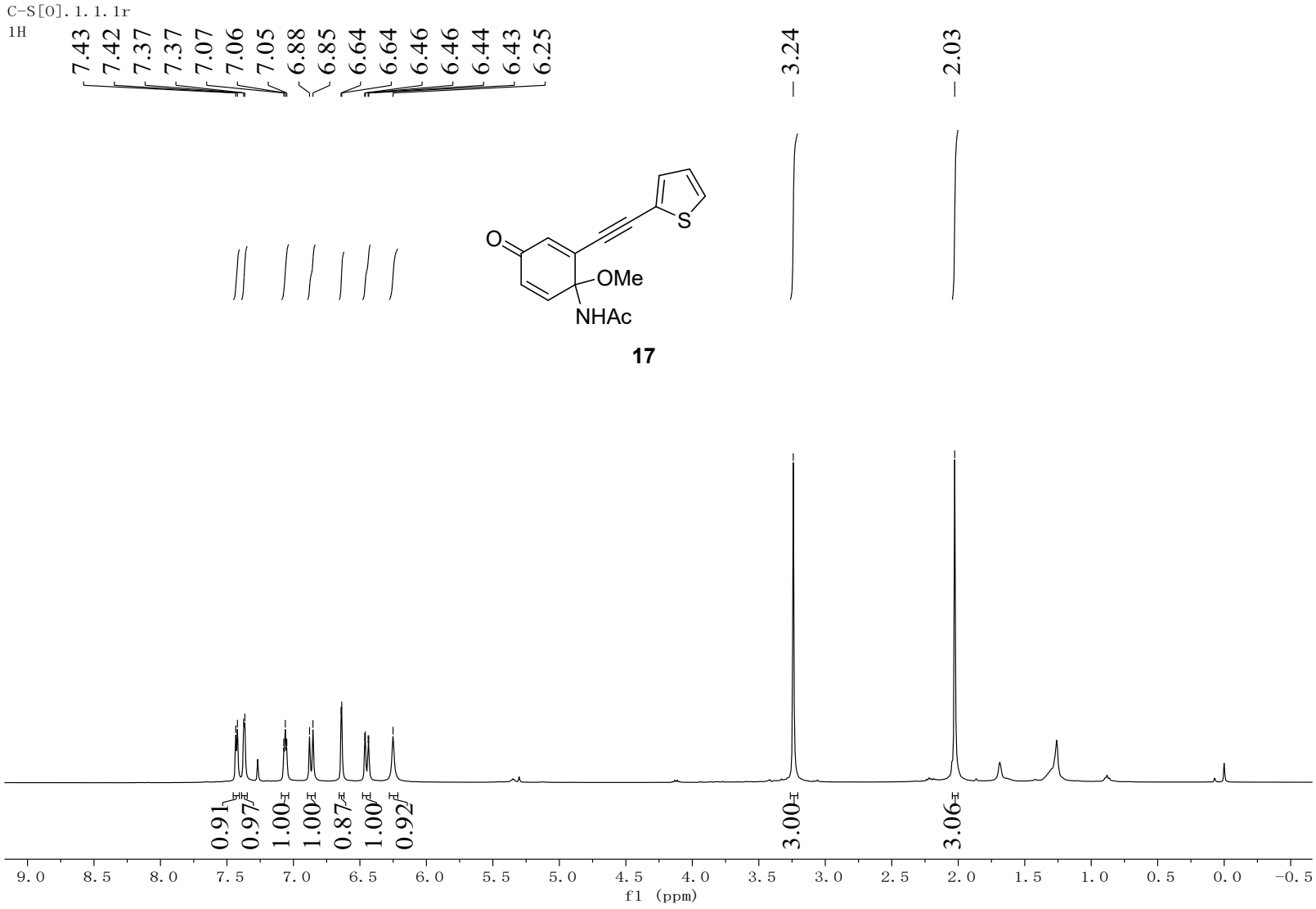
C-CN[O]-C.1.fid

184.13
168.49
144.42
137.86
135.87
132.57
132.23
131.31
126.32
118.05
113.11
97.70
87.63
80.51
51.05
23.58



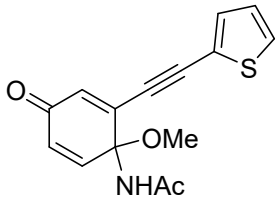
16



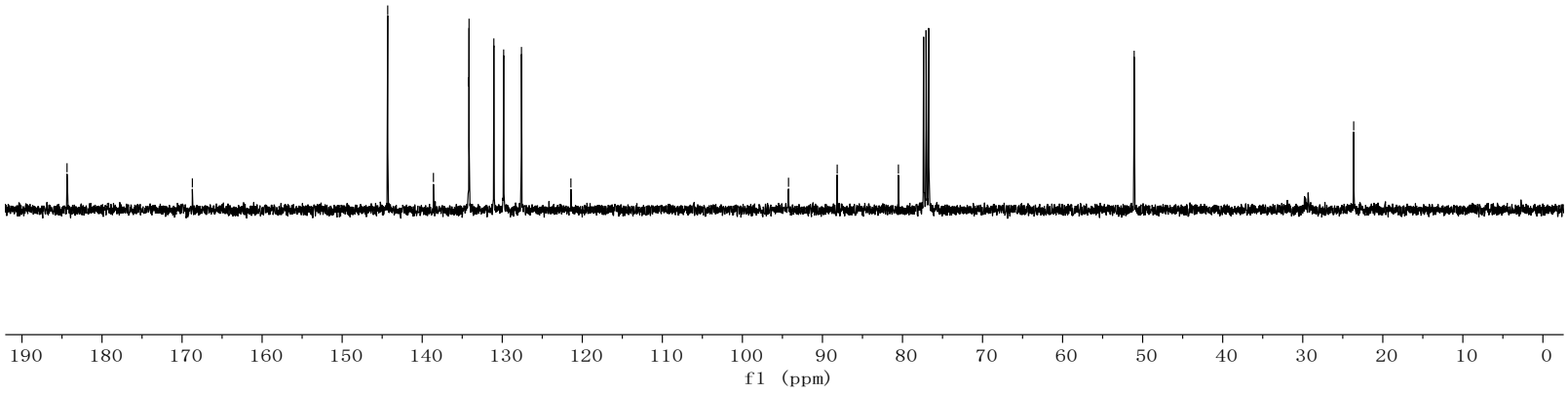

C-S[O].1.1.1r
1H
7.43
7.42
7.37
7.37
7.07
7.06
7.05
6.88
6.85
6.64
6.64
6.46
6.46
6.44
6.43
6.25
3.24
2.03
O
S
OMe
NHAc
17
0.91
0.97
1.00
1.00
0.87
1.00
0.92
3.00
3.06
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

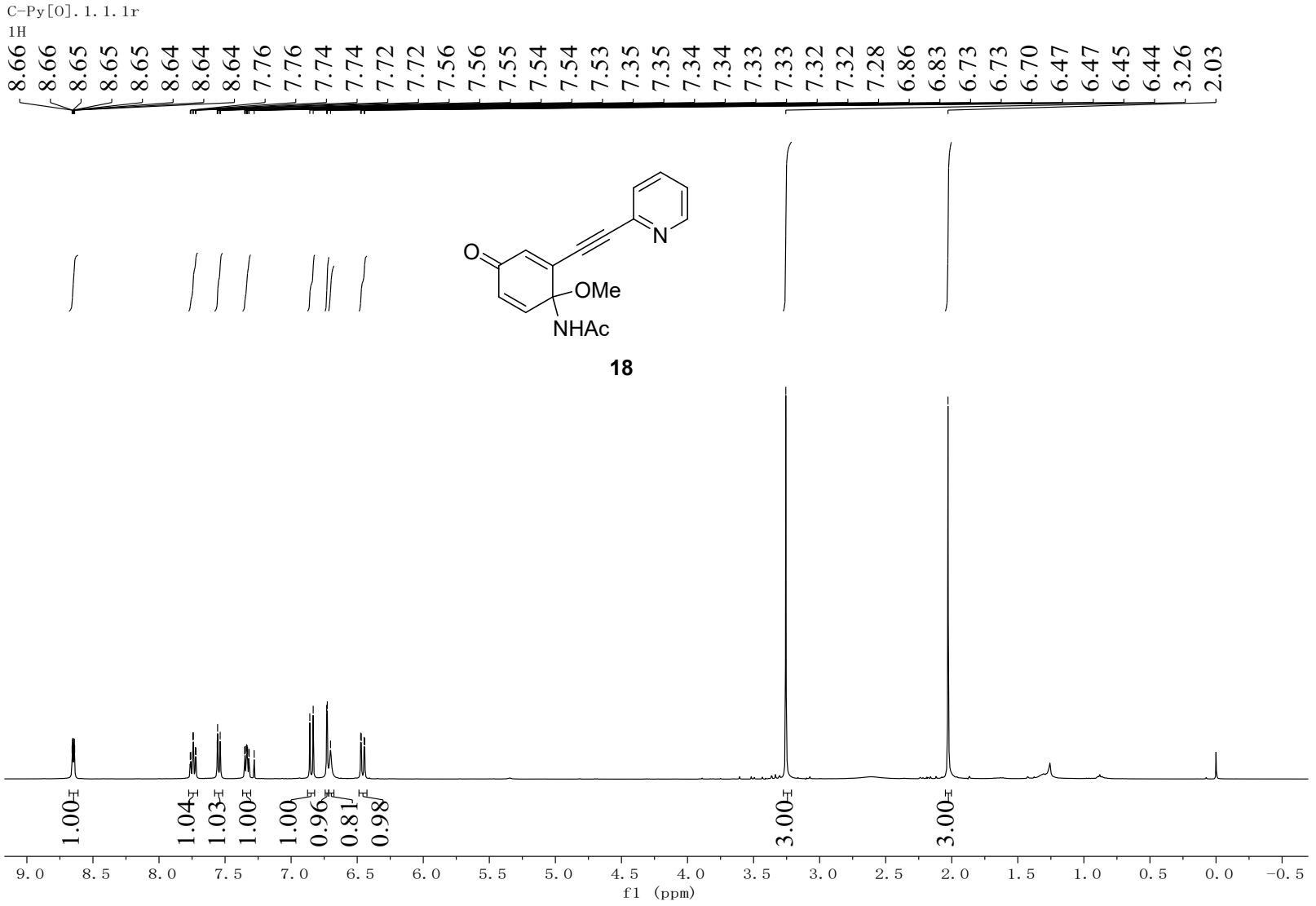
C-S[O]-C.2.1.1r
C-S[O]-C

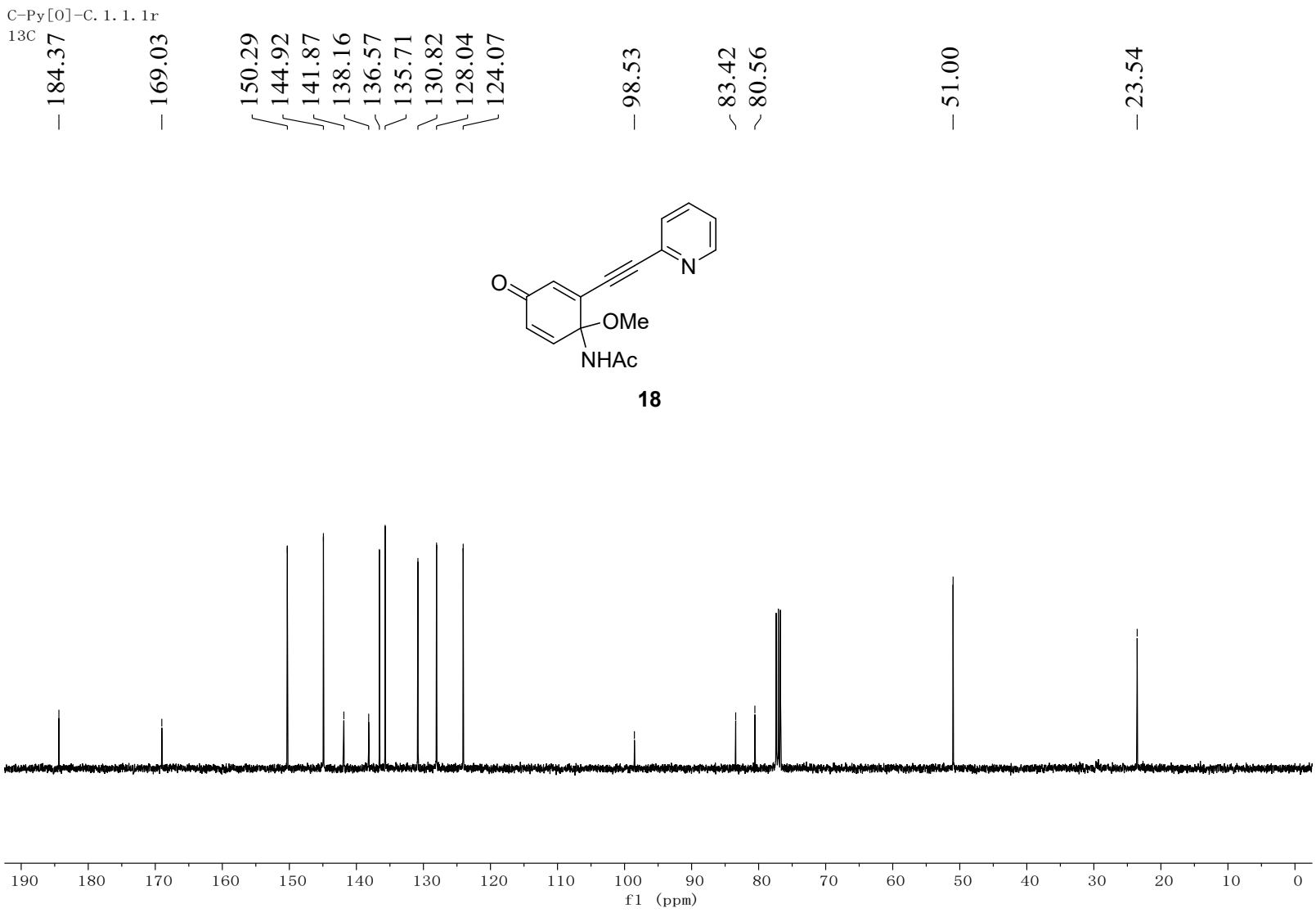
184.37
168.71
144.31
138.60
134.23
134.15
131.05
129.82
127.60
121.42
94.24
88.16
80.51
51.06
23.64

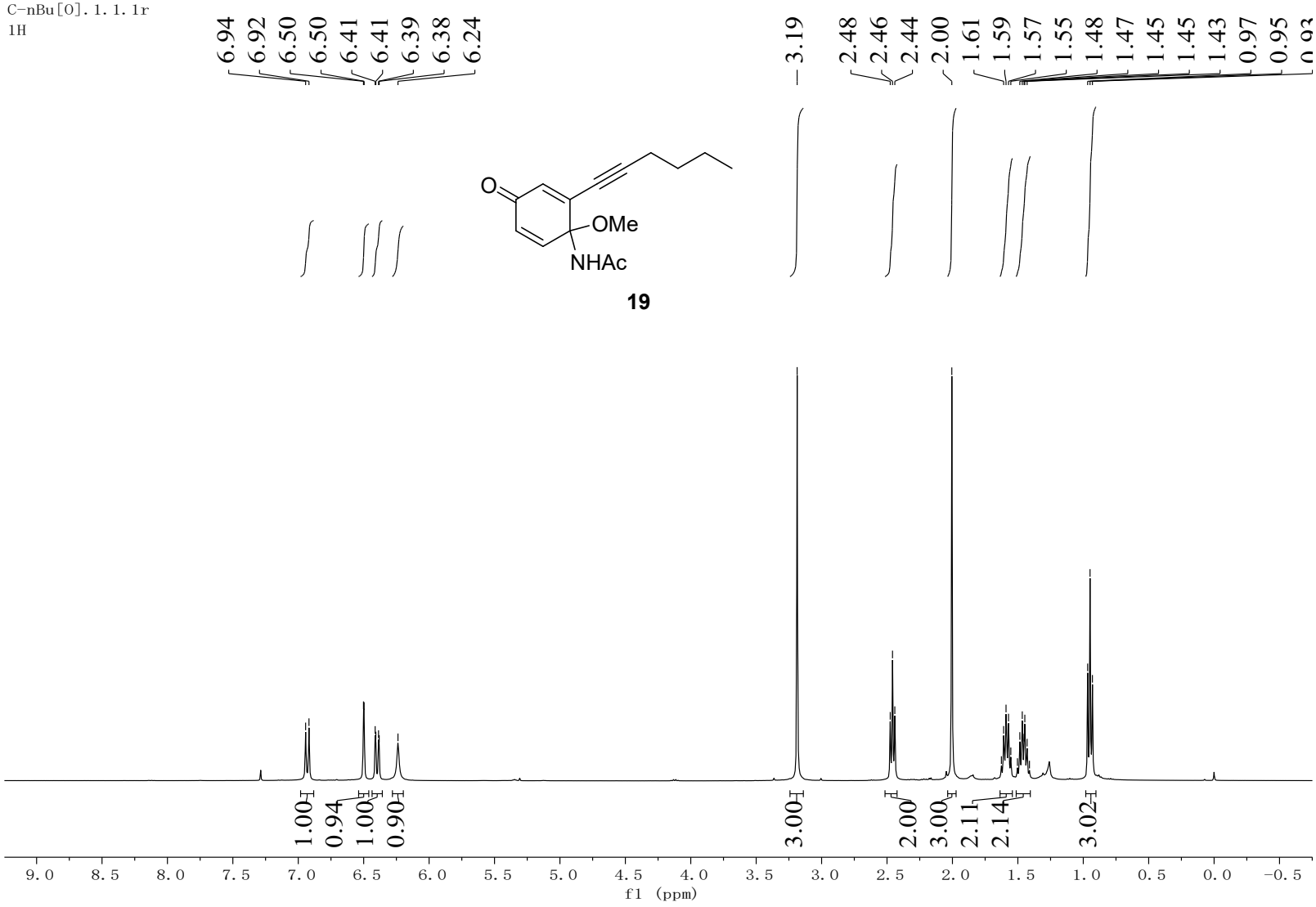


17



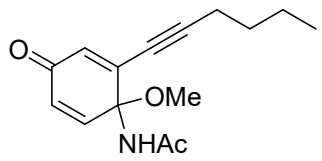

C-Py[O].1.1.1r
1H
8.66 8.66 8.65 8.65 8.65 8.64 8.64 8.64 7.76 7.76 7.74 7.74 7.72 7.72 7.56 7.56 7.55 7.54 7.54 7.53 7.35 7.35 7.34 7.34 7.33 7.33 7.32 7.32 7.28 6.86 6.83 6.73 6.73 6.70 6.47 6.47 6.45 6.44 3.26 2.03
O
N
OMe
NHAc
18
1.00 1.04 1.03 1.00 1.00 0.96 0.81 0.98 3.00 3.00
9.0 8.5 8.0 7.5 7.0 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 -0.5
f1 (ppm)


C-Py[O]-C.1.1.1r
13C
184.37
169.03
150.29
144.92
141.87
138.16
136.57
135.71
130.82
128.04
124.07
98.53
83.42
80.56
51.00
23.54
O
N
OMe
NHAc
18
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

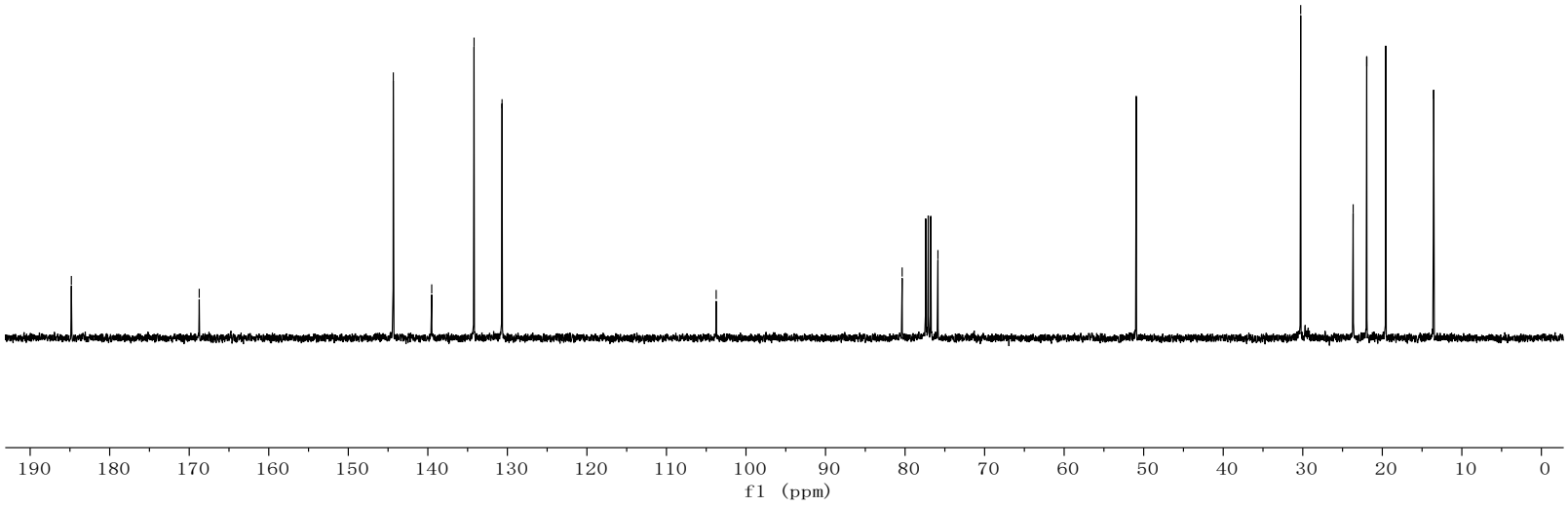

C-nBu[O].1.1.1r
1H
6.94
6.92
6.50
6.50
6.41
6.41
6.39
6.38
6.24
3.19
2.48
2.46
2.44
2.00
1.61
1.59
1.57
1.55
1.48
1.47
1.45
1.45
1.43
0.97
0.95
O
OMe
NHAc
19
1.00
0.94
1.00
0.90
3.00
2.00
3.00
2.11
2.14
3.02
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

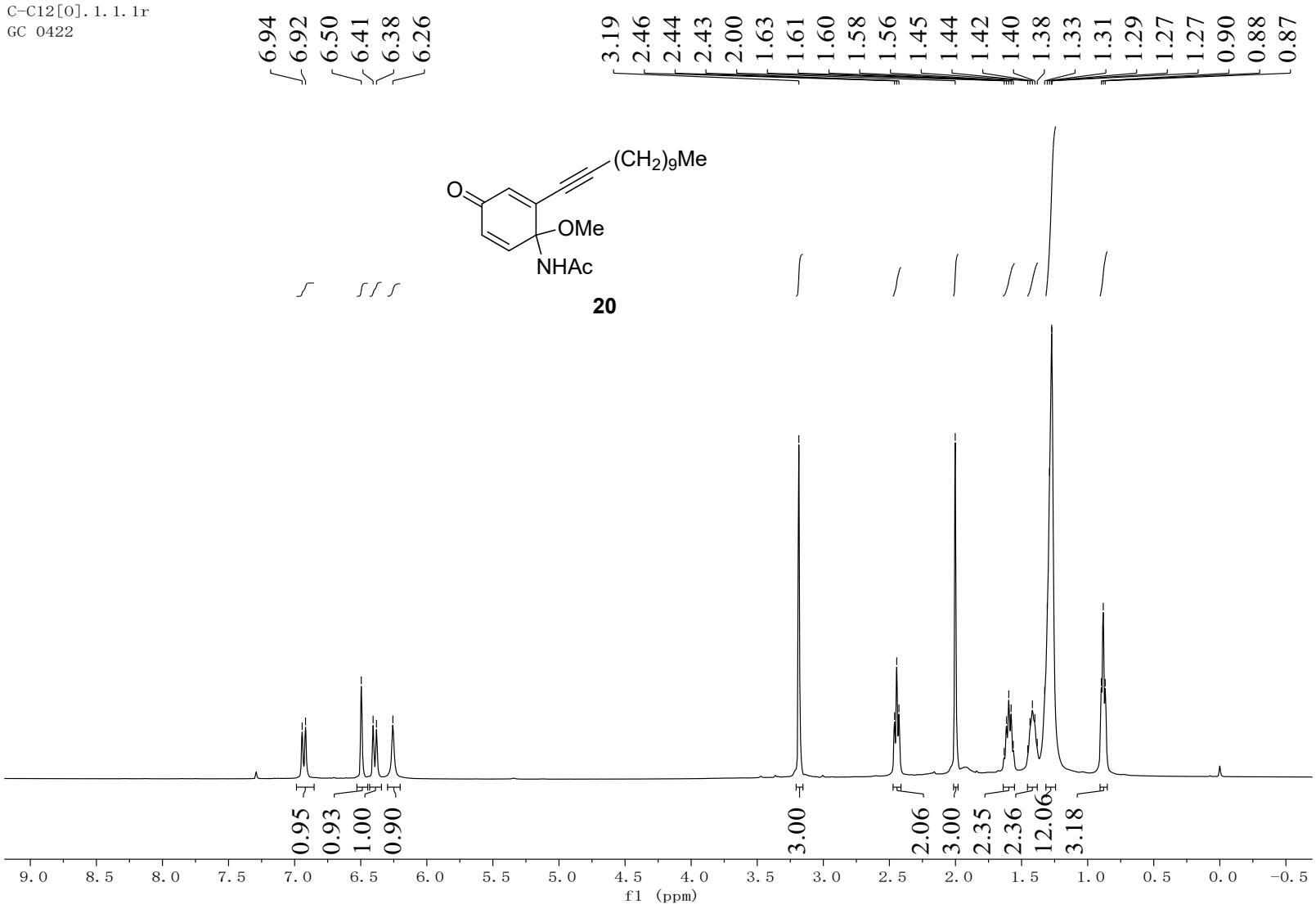
C-nBu[O]-C.1.1.1r
13C

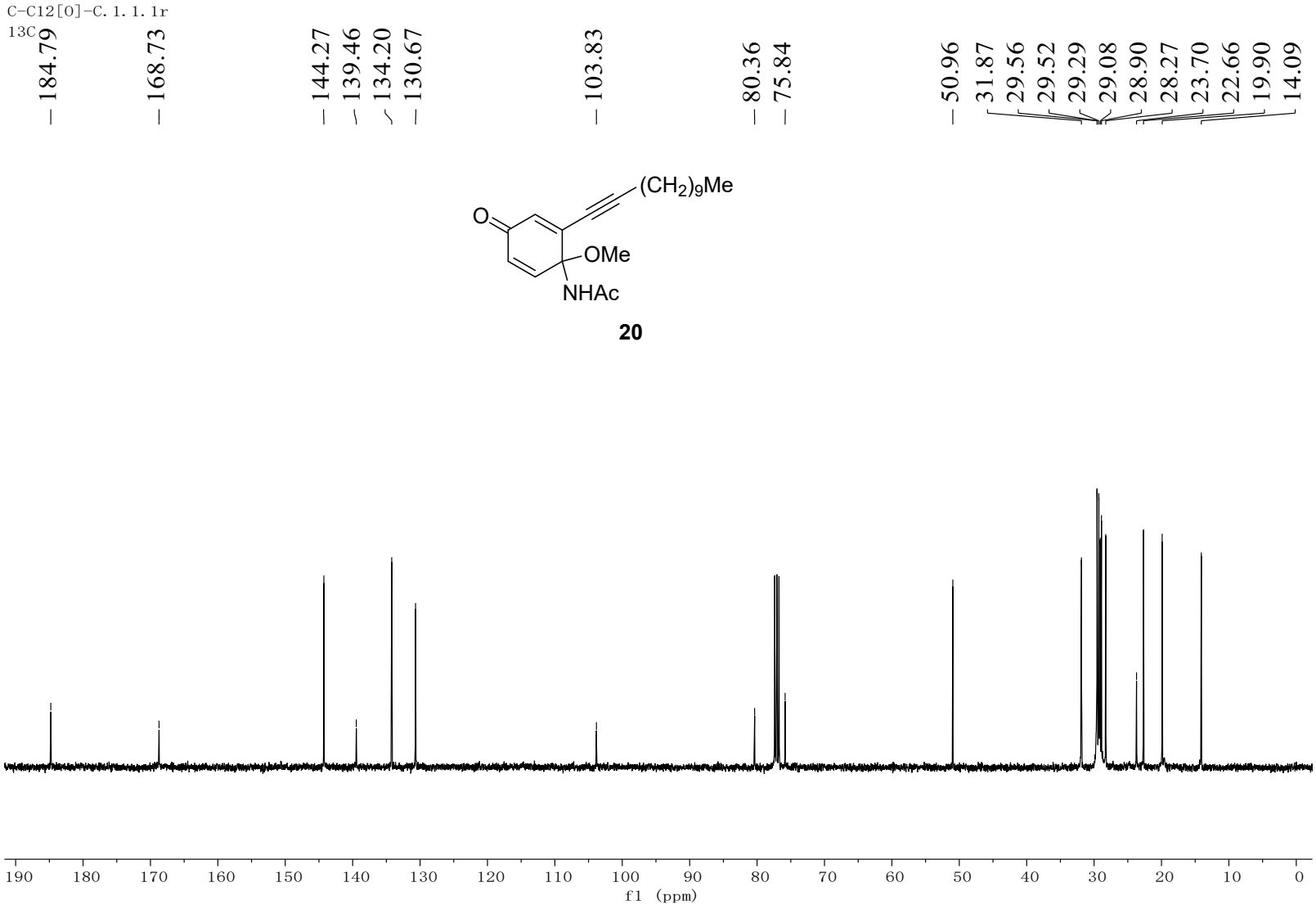
184.82
168.74
144.34
139.51
134.19
130.67
103.75
80.37
75.87
50.94
30.26
23.66
21.95
19.57
13.54

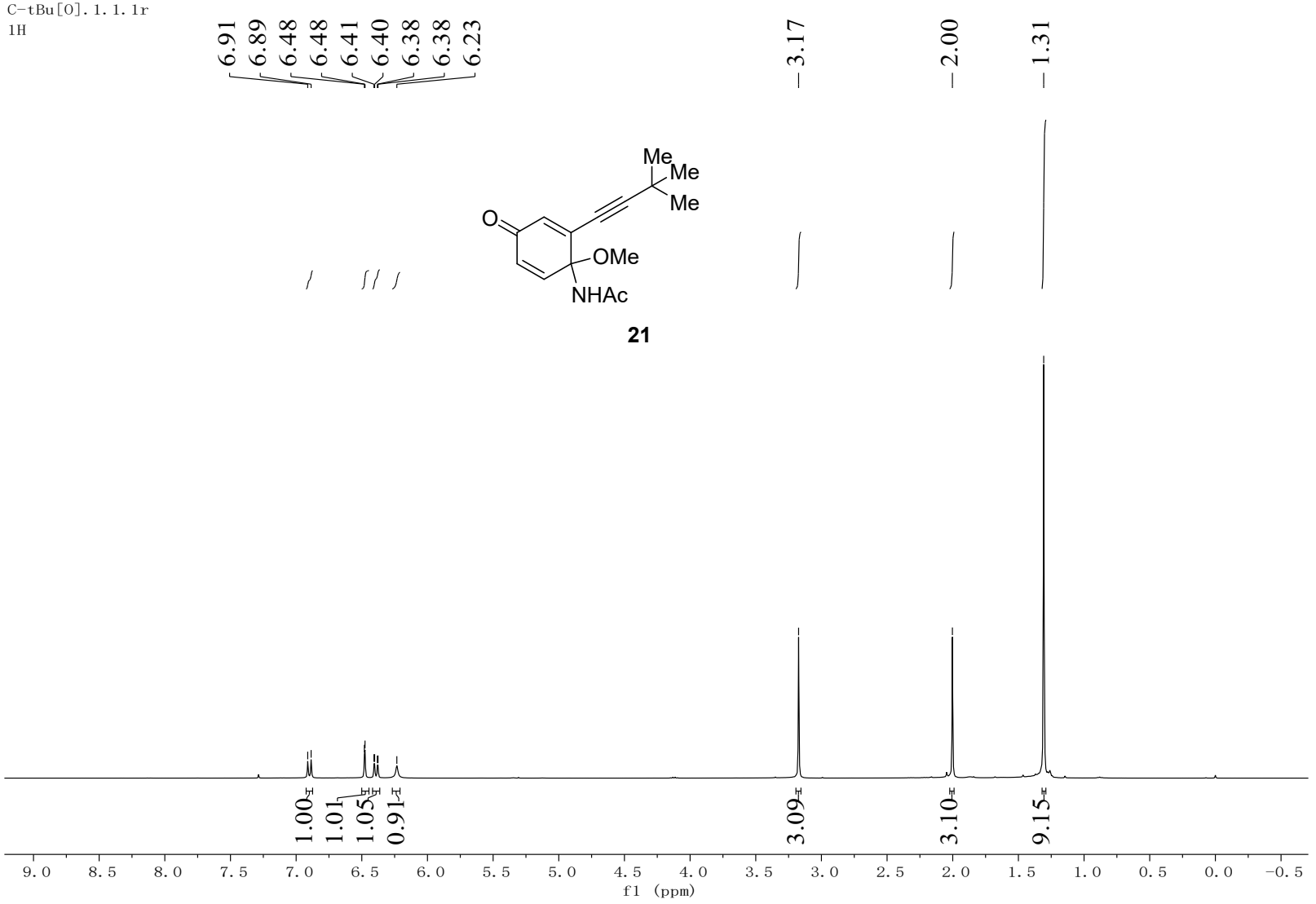


19



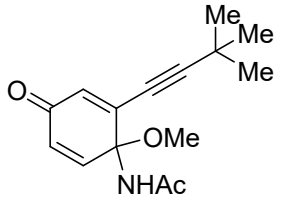

C-C12[O].1.1.1r
GC 0422
6.94
6.92
6.50
6.41
6.38
6.26
3.19
2.46
2.44
2.43
2.00
1.63
1.61
1.60
1.58
1.56
1.45
1.44
1.42
1.40
1.38
1.33
1.31
1.29
1.27
1.27
0.90
0.88
0.87
O
(CH2)9Me
OMe
NHAc
20
0.95
0.93
1.00
0.90
3.00
2.06
3.00
2.35
2.36
12.06
3.18
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


C-C12[O]-C.1.1.1r
13C
184.79
168.73
144.27
139.46
134.20
130.67
103.83
80.36
75.84
50.96
31.87
29.56
29.52
29.29
29.08
28.90
28.27
23.70
22.66
19.90
14.09
O
(CH2)9Me
OMe
NHAc
20
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

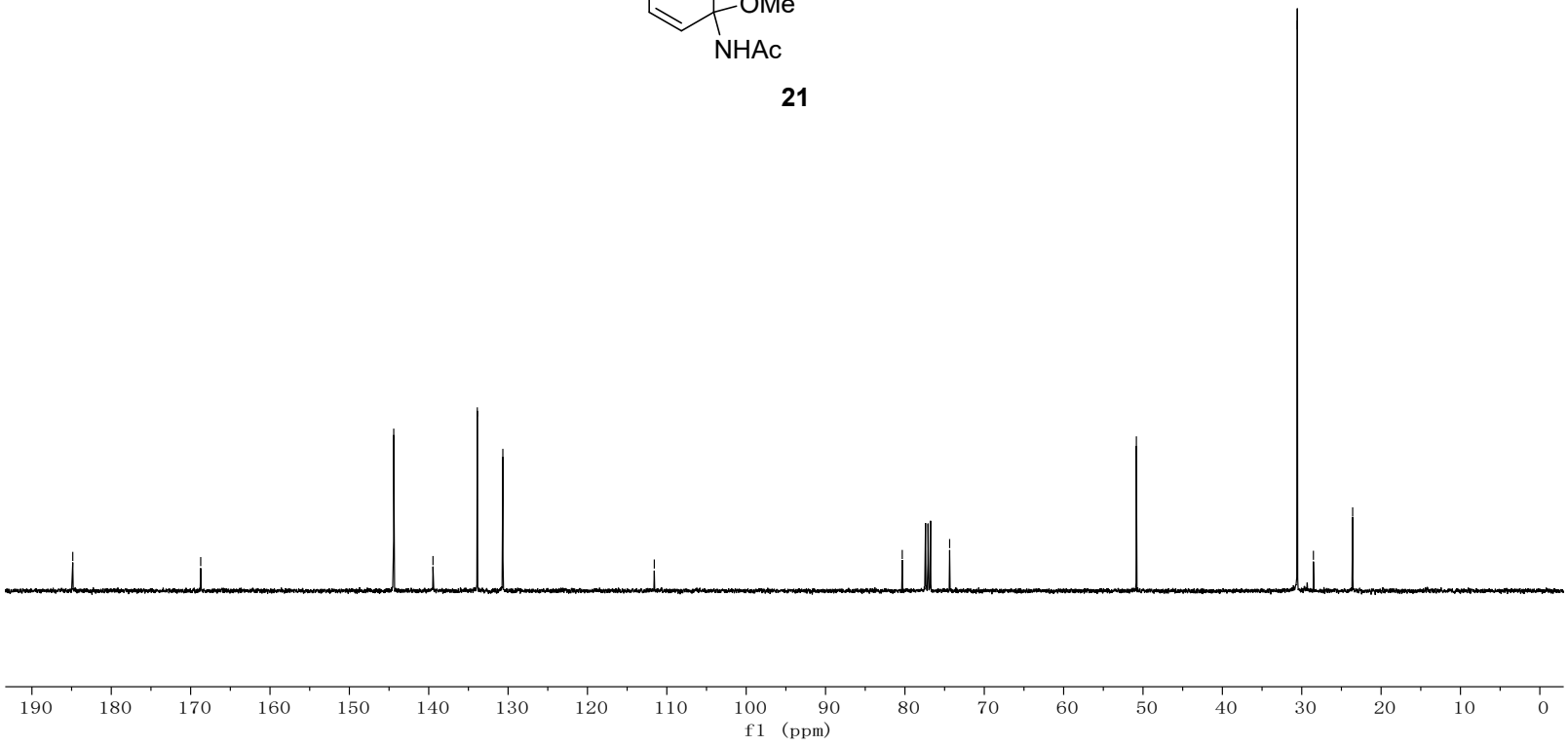

C-tBu[O].1.1.1r
1H
6.91
6.89
6.48
6.48
6.41
6.40
6.38
6.38
6.23
3.17
2.00
1.31
Me
Me
Me
O
OMe
NHAc
21
1.00
1.01
1.05
0.91
3.09
3.10
9.15
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

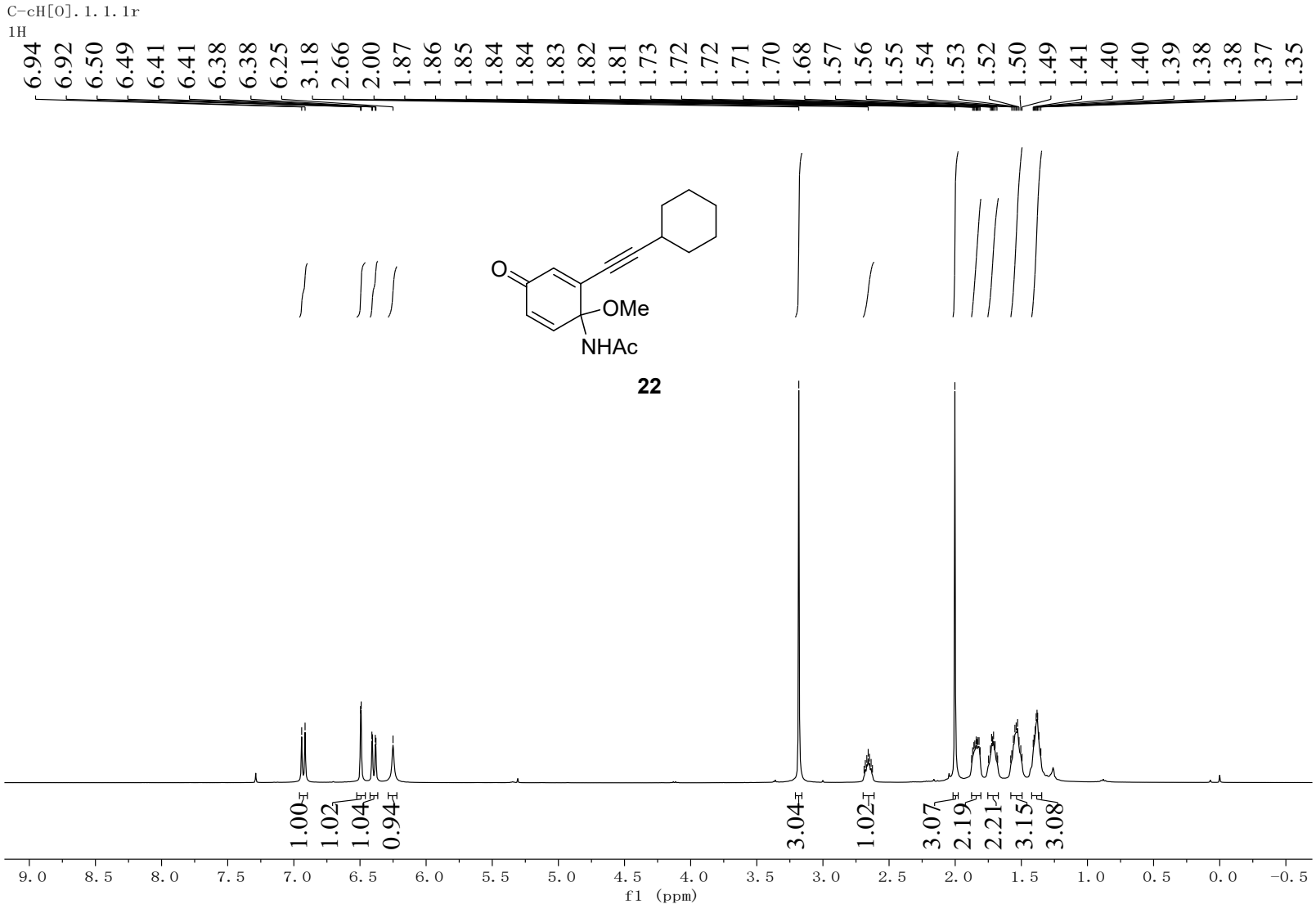
C-tBu[O]-C.1.1.1r
13C

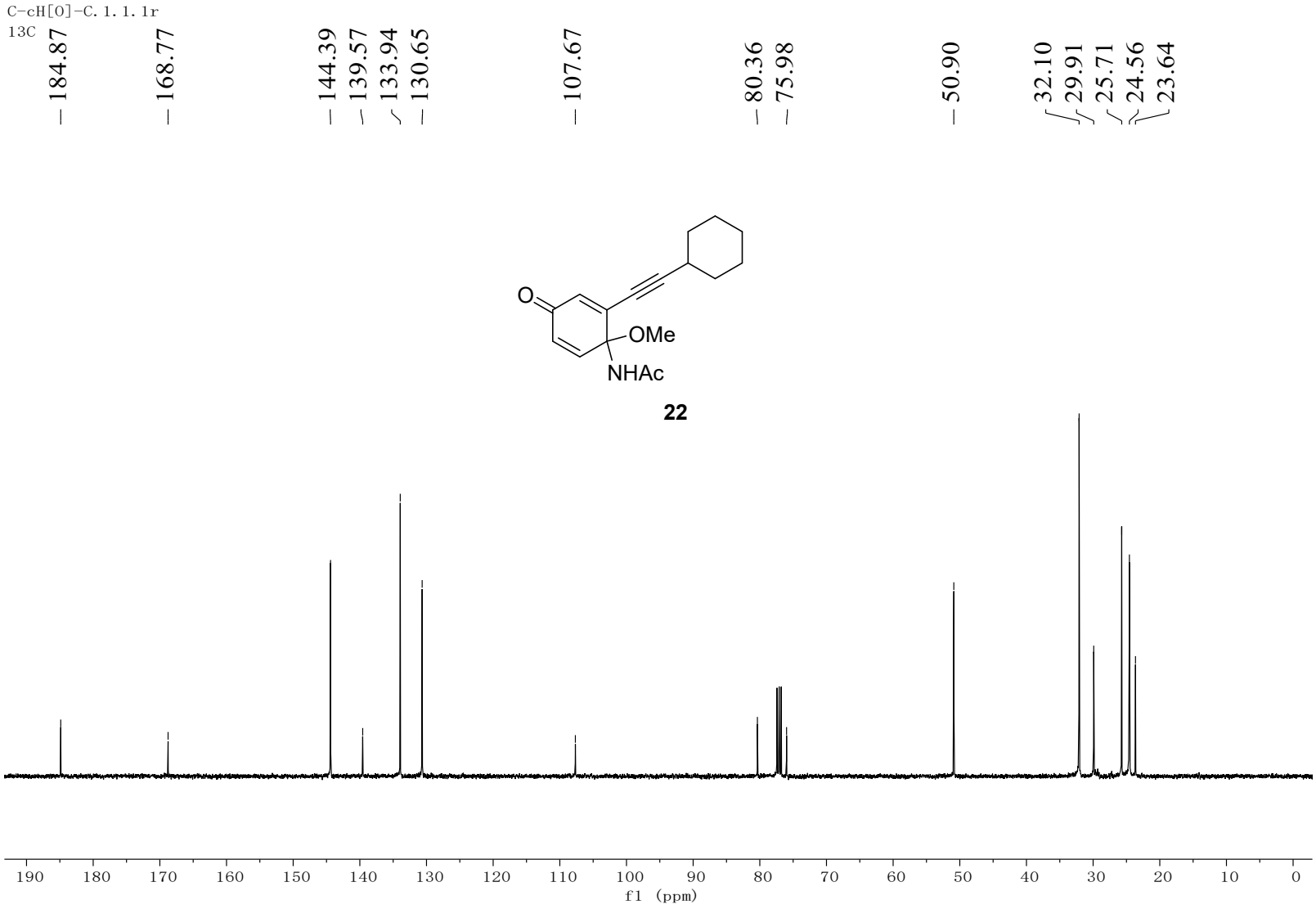
184.85
168.73
144.41
139.46
133.88
130.66
111.58
80.34
74.36
50.84
30.56
28.53
23.58

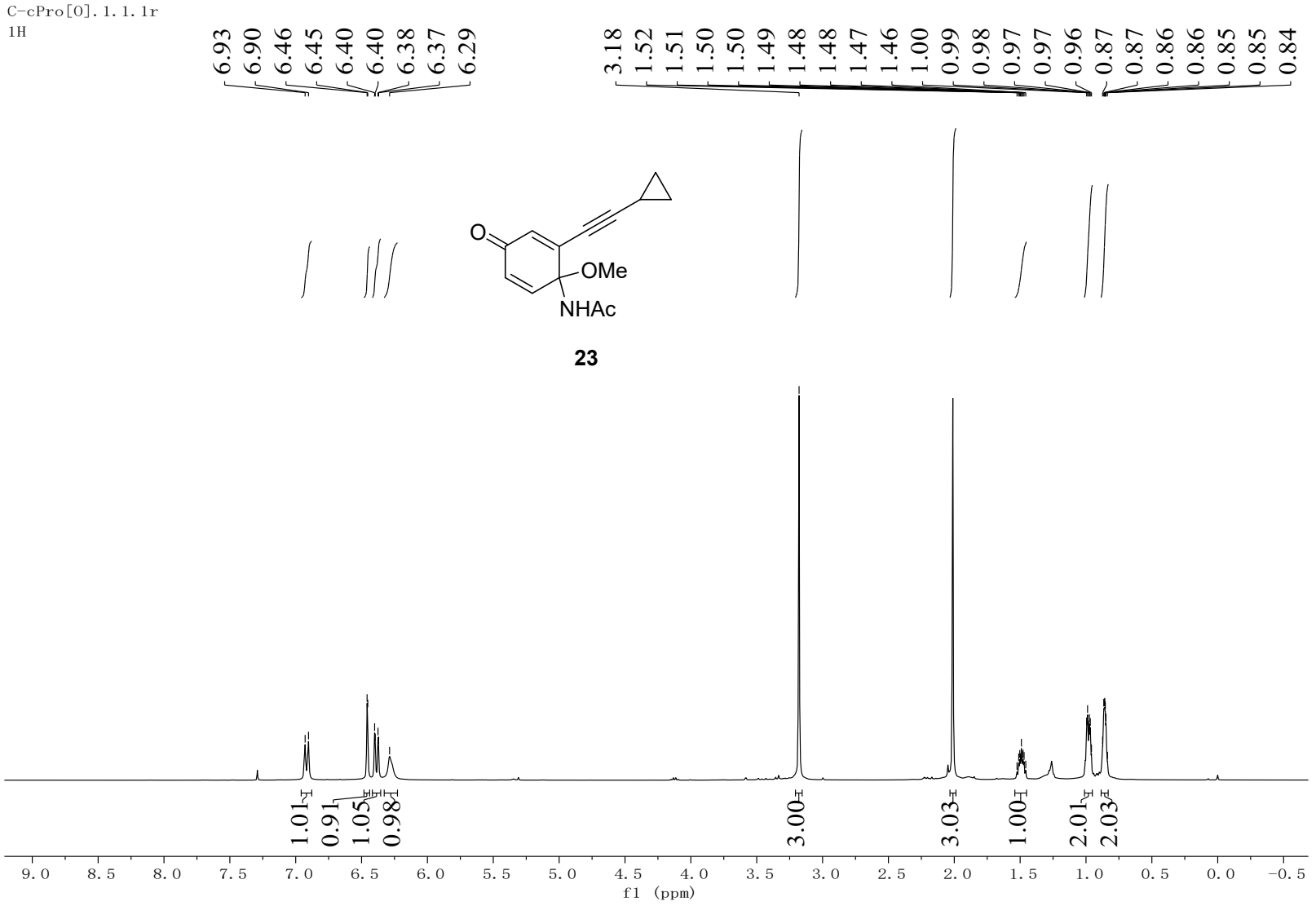


21



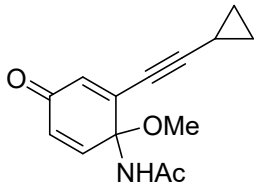

C-cH[O].1.1.1r
1H
6.94
6.92
6.50
6.49
6.41
6.41
6.38
6.38
6.25
3.18
2.66
2.00
1.87
1.86
1.85
1.84
1.84
1.83
1.82
1.81
1.73
1.72
1.72
1.71
1.70
1.68
1.57
1.56
1.55
1.54
1.53
1.52
1.50
1.49
1.41
1.40
1.40
1.39
1.38
1.38
1.37
1.35
O
OMe
NHAc
22
1.00
1.02
1.04
0.94
3.04
1.02
3.07
2.19
2.21
3.15
3.08
9.0
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
-0.5
f1 (ppm)


C-cH[O]-C.1.1.1r
13C
184.87
168.77
144.39
139.57
133.94
130.65
107.67
80.36
75.98
50.90
32.10
29.91
25.71
24.56
23.64
O
OMe
NHAc
22
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

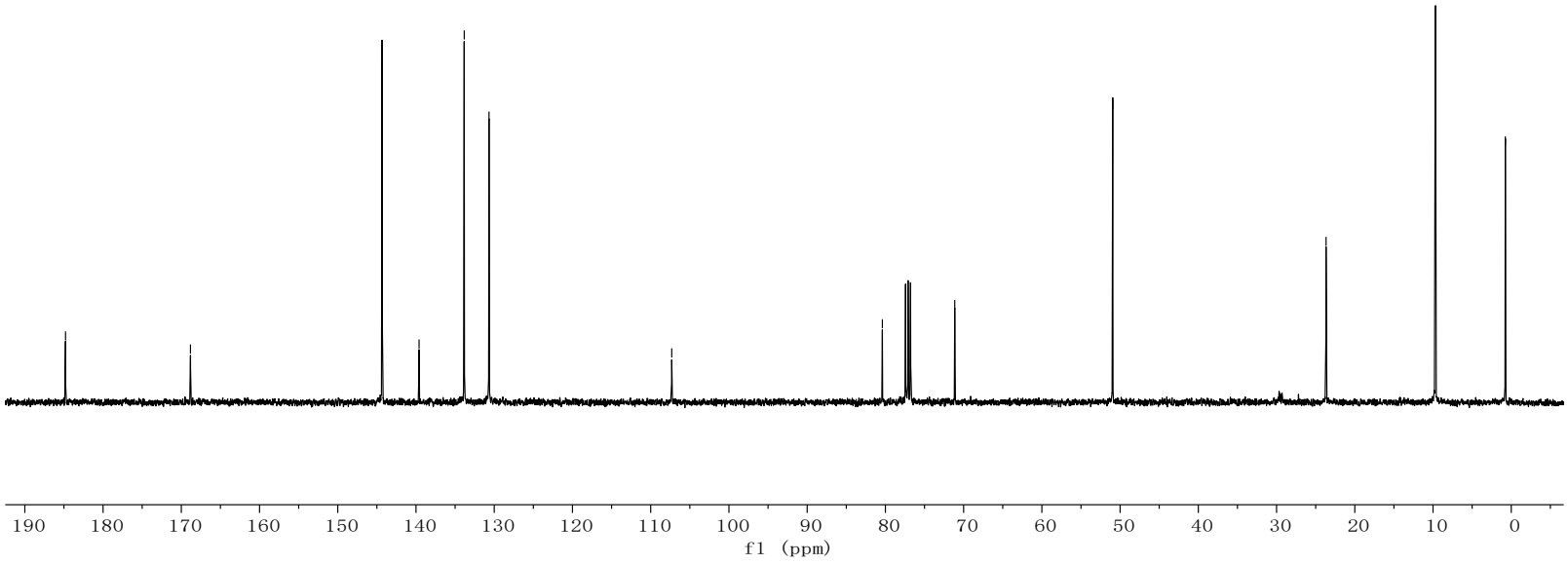

C-cPro[O].1.1.1r
1H
6.93
6.90
6.46
6.45
6.40
6.40
6.38
6.37
6.29
3.18
1.52
1.51
1.50
1.50
1.49
1.48
1.48
1.47
1.46
1.00
0.99
0.98
0.97
0.97
0.96
0.87
0.87
0.86
0.86
0.85
0.85
0.84
O
OMe
NHAc
23
1.01
0.91
1.05
0.98
3.00
3.03
1.00
2.01
2.03
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

C-cPro[O]-C.1.1.1r
13C

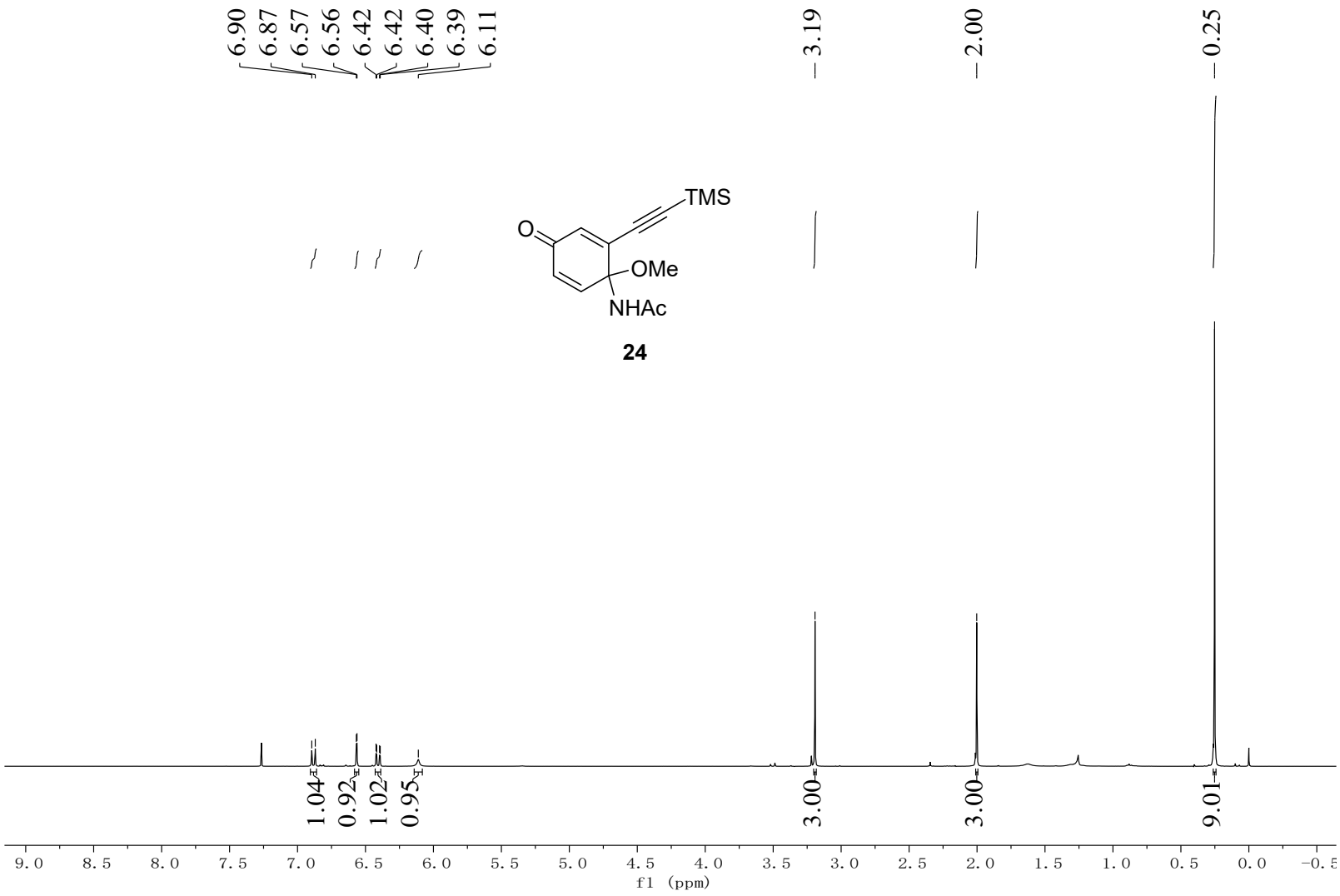
184.80
168.83
144.33
139.61
133.83
130.67
107.31
80.39
71.14
50.95
23.67
9.71
0.76



23

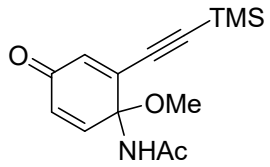


C-TMS[O]-NEW.1.1.1r

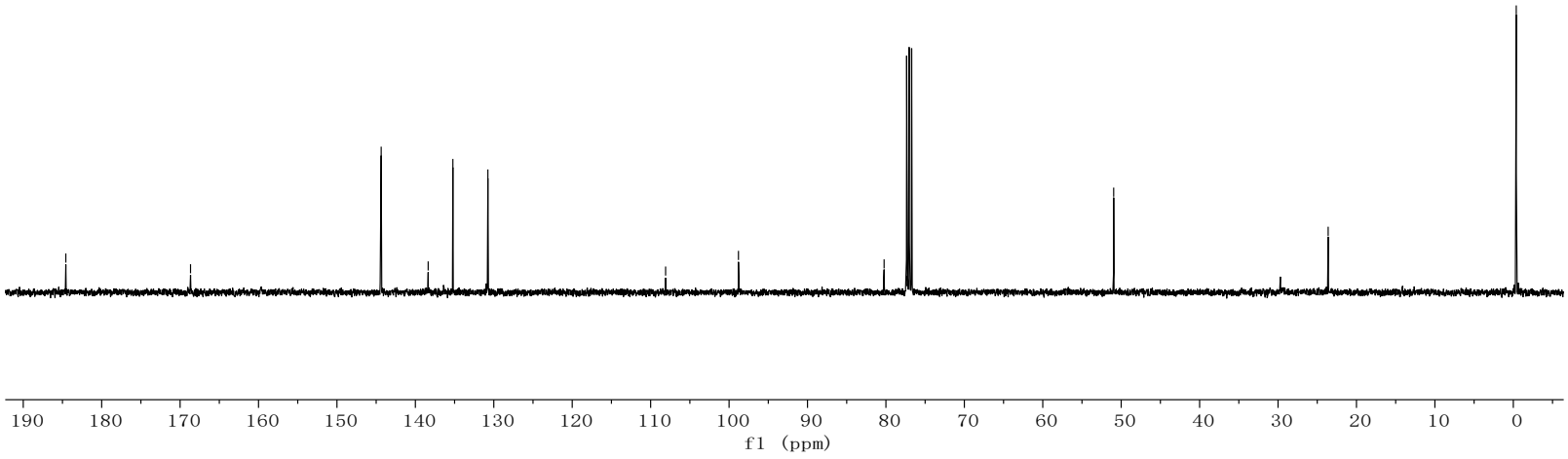


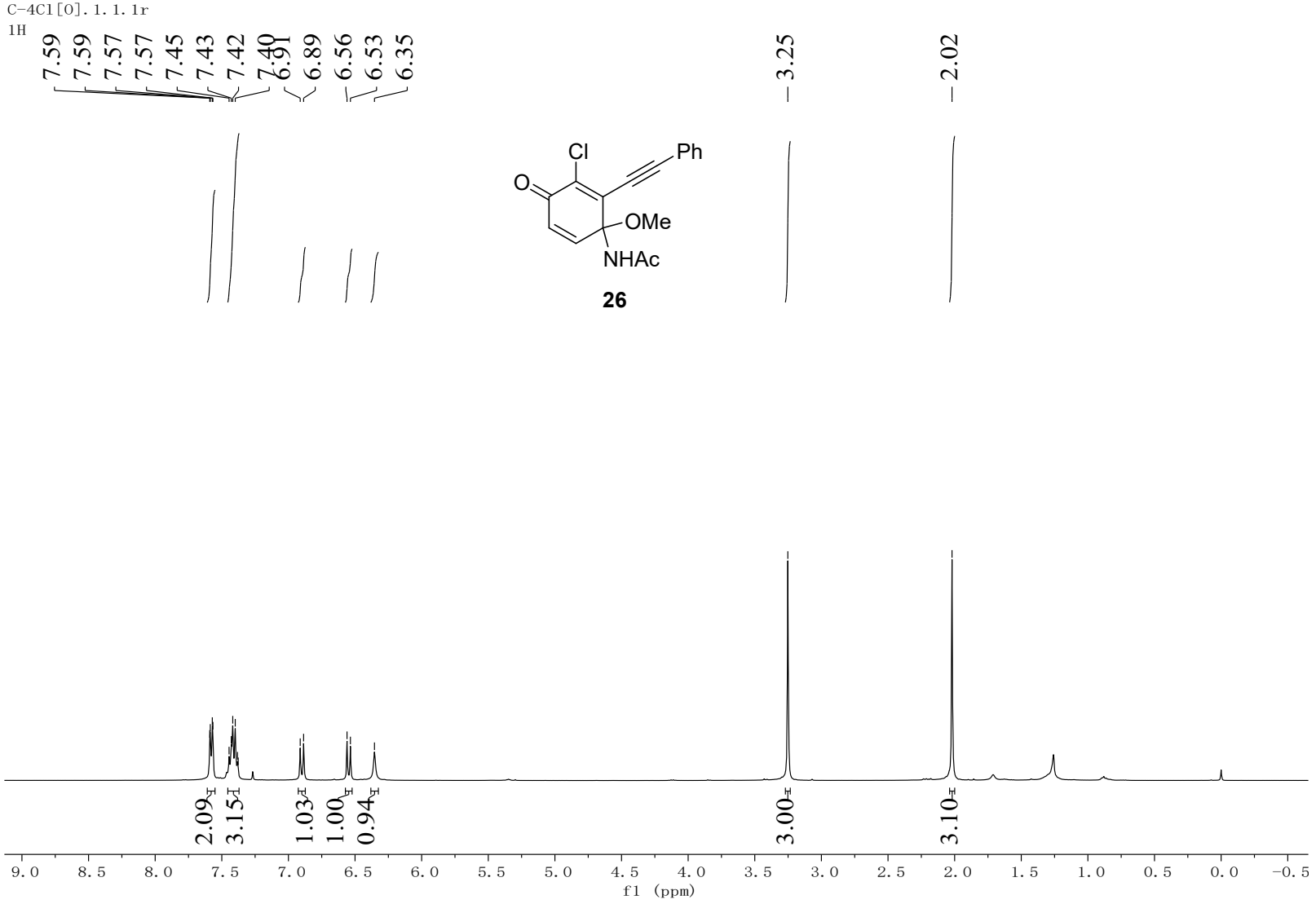
C-TMS[O]-C.1.1.1r

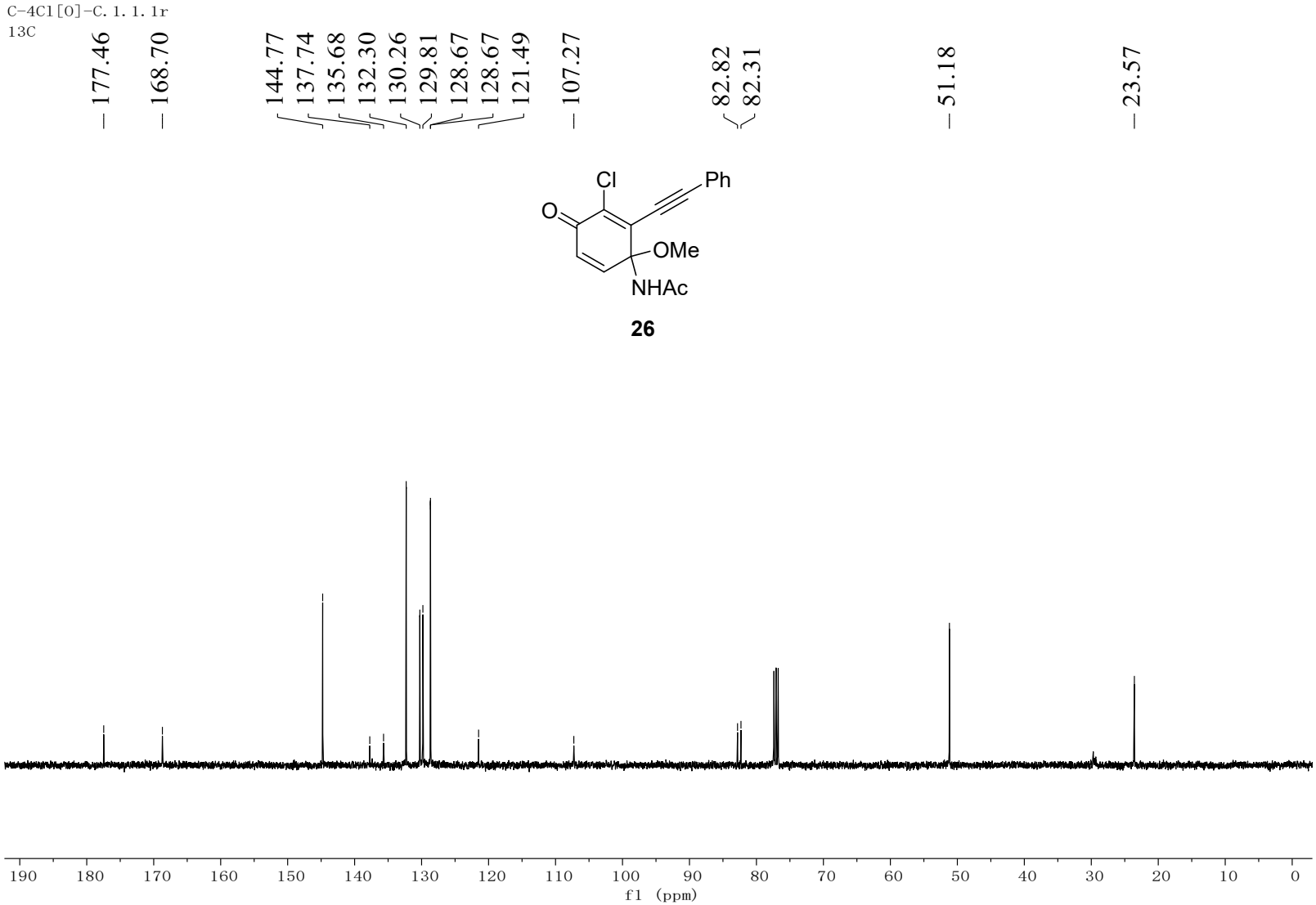
184.57
168.67
144.36
138.37
135.22
130.75
108.09
98.80
80.23
50.96
23.61
-0.37



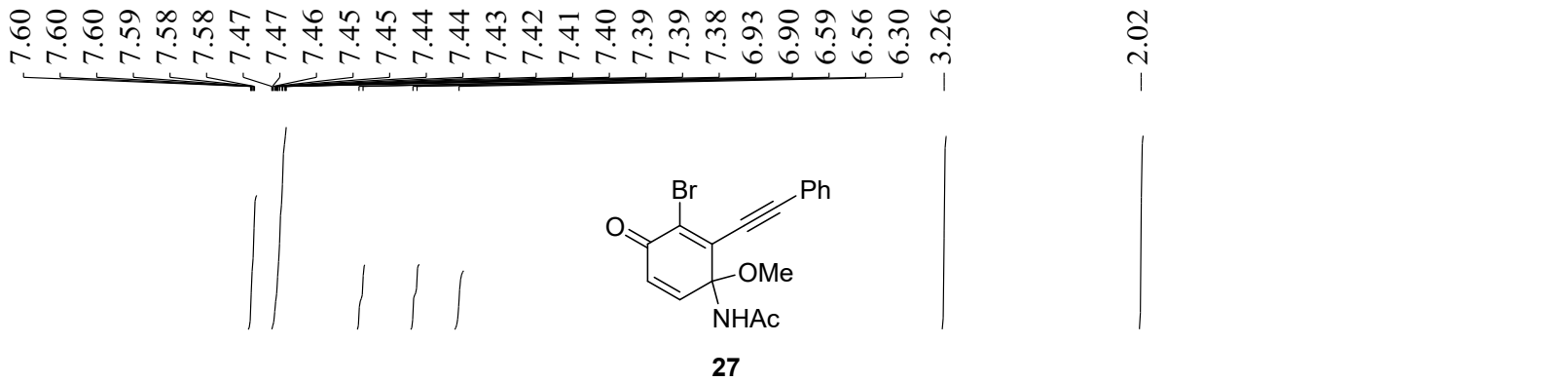
24



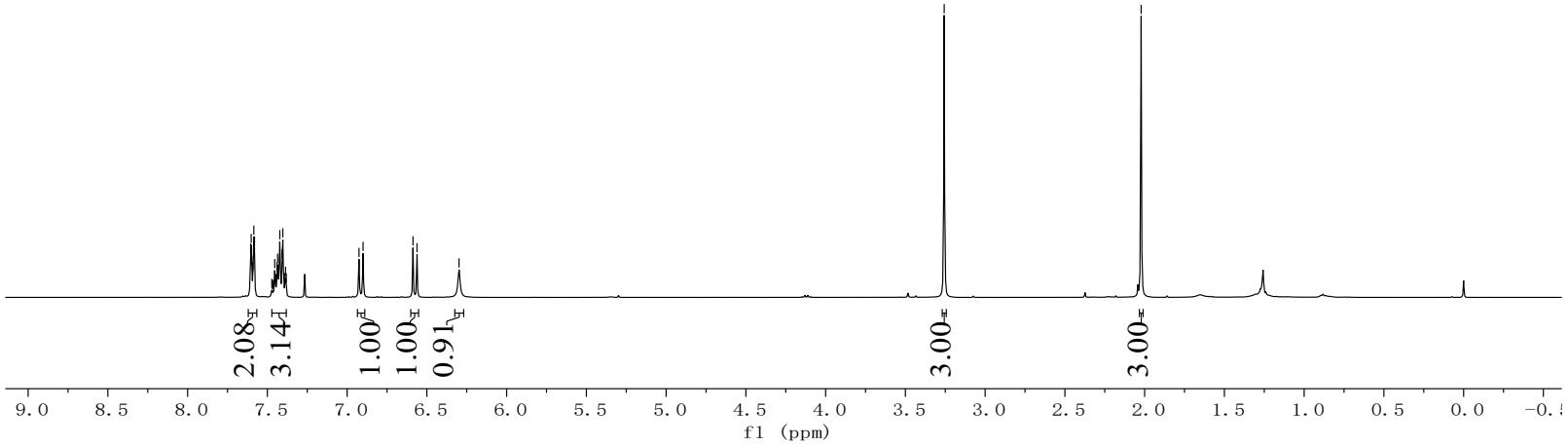

C-4Cl[O].1.1.1r
1H
7.59
7.59
7.57
7.57
7.45
7.43
7.42
7.40
6.91
6.89
6.56
6.53
6.35
3.25
2.02
Cl
Ph
O
OMe
NHAc
26
2.09
3.15
1.03
1.00
0.94
3.00
3.10
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


C-4Cl[O]-C.1.1.1r
13C
177.46
168.70
144.77
137.74
135.68
132.30
130.26
129.81
128.67
128.67
121.49
107.27
82.82
82.31
51.18
23.57
Cl
Ph
O
OMe
NHAc
26
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

C-4Br[O]-NEW.1.1.1r
1H

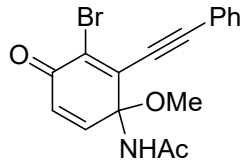


27

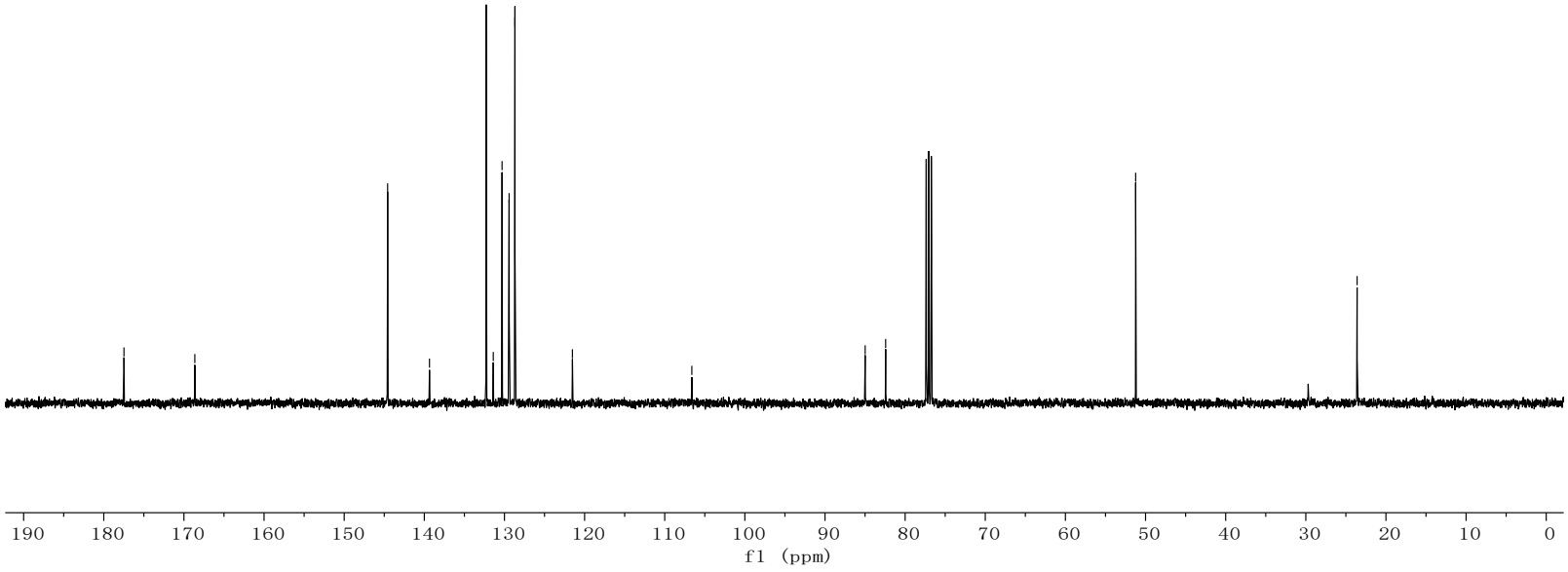


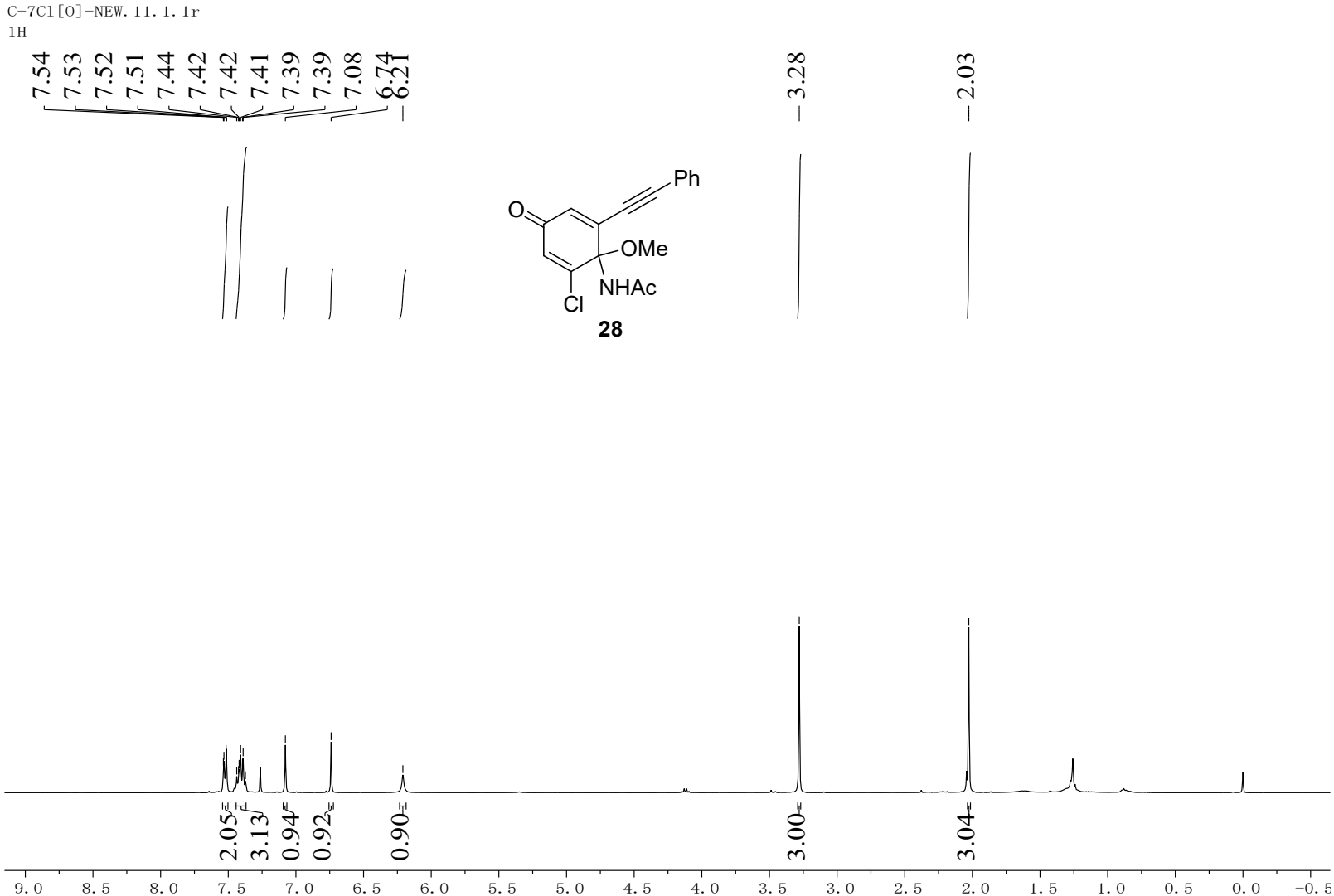
C-4Br[O]-C-NEW.1.1.1r
13C

177.48
168.64
144.58
139.35
132.28
131.40
130.30
129.42
128.69
121.52
106.62
84.99
82.43
51.23
23.60



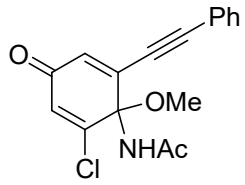
27



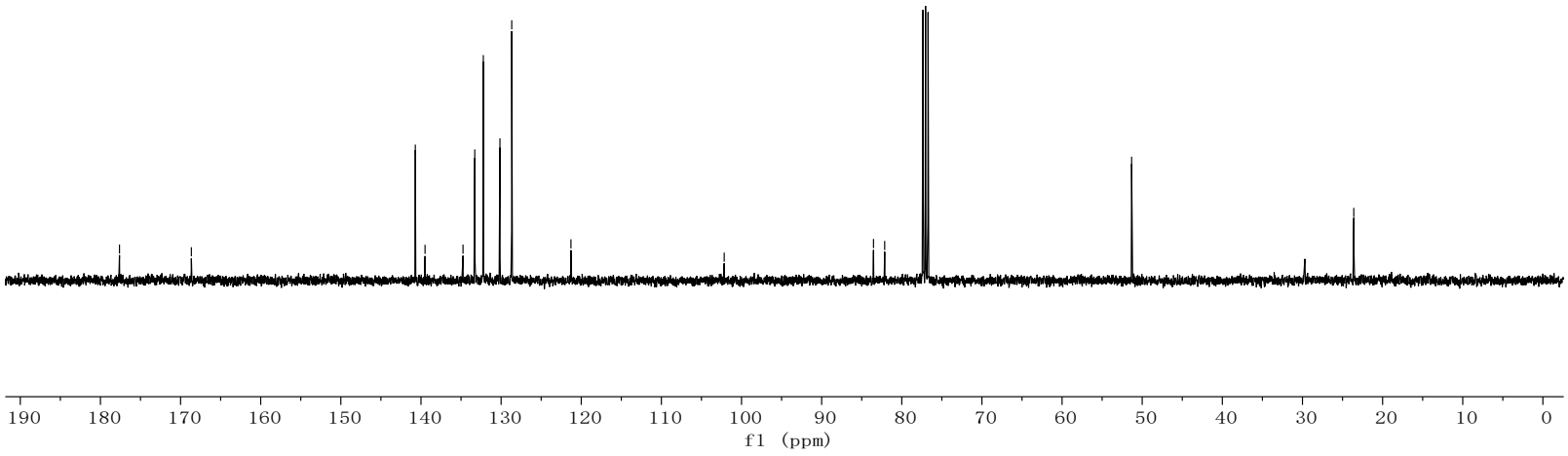

C-7Cl[O]-NEW.11.1.1r
1H
7.54
7.53
7.52
7.51
7.44
7.42
7.42
7.41
7.39
7.39
7.08
6.74
6.21
3.28
2.03
Ph
O
OMe
NHAc
Cl
28
2.05
3.13
0.94
0.92
0.90
3.00
3.04
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
f1 (ppm)

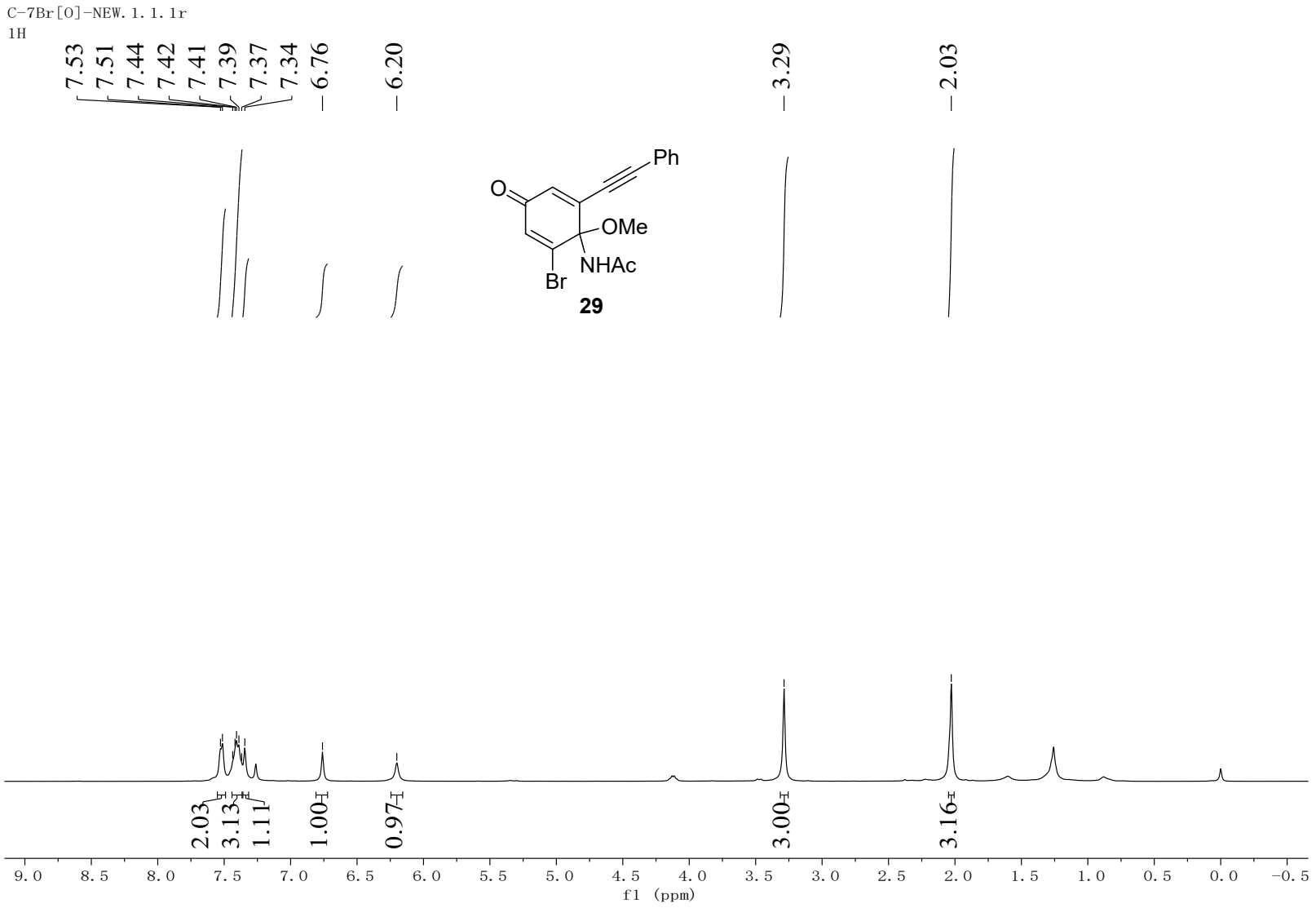
C-7Cl[O]-C-NEW.1.1.1r
13C

177.62
168.65
140.71
139.49
134.75
133.29
132.22
130.15
128.67
121.29
102.16
83.53
82.14
51.32
23.60



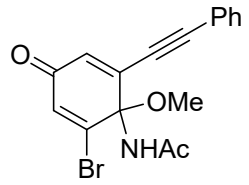
28



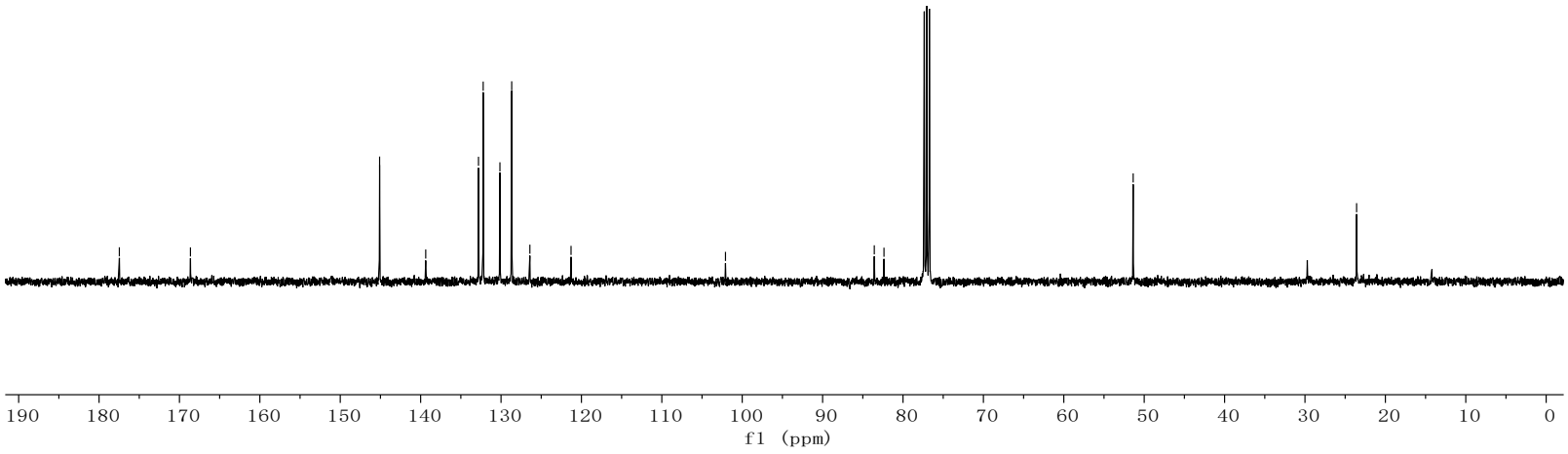

C-7Br[O]-NEW.1.1.1r
1H
7.53
7.51
7.44
7.42
7.41
7.39
7.37
7.34
6.76
6.20
3.29
2.03
O
Ph
OMe
NHAc
Br
29
2.03
3.13
1.11
1.00
0.97
3.00
3.16
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

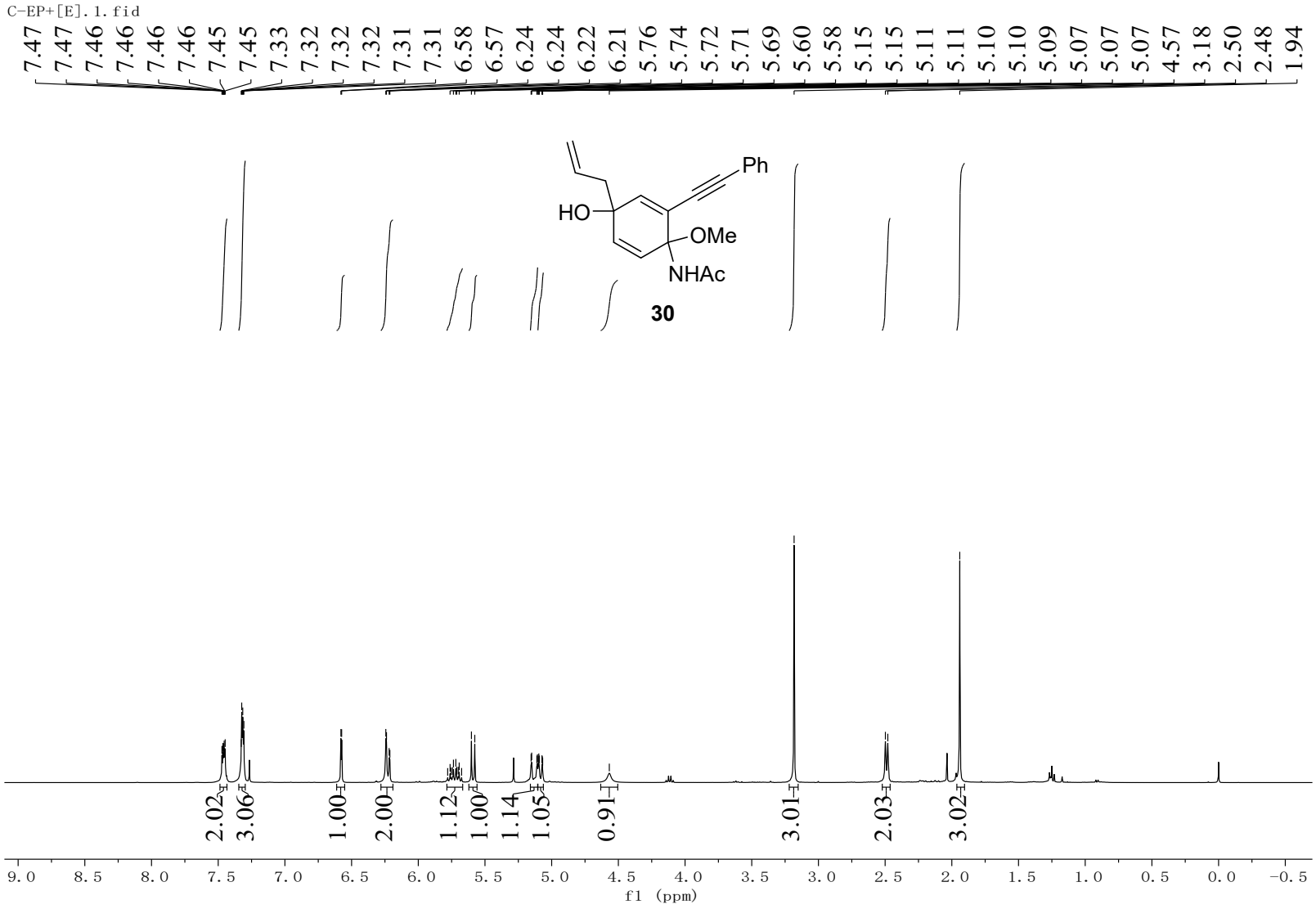
C-7Br[O]-C-NEW.1.1.1r
13C

177.48
168.63
145.10
139.37
132.80
132.22
130.14
128.67
126.41
121.29
102.08
83.57
82.37
51.37
23.58



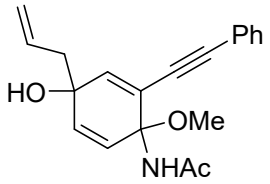
29



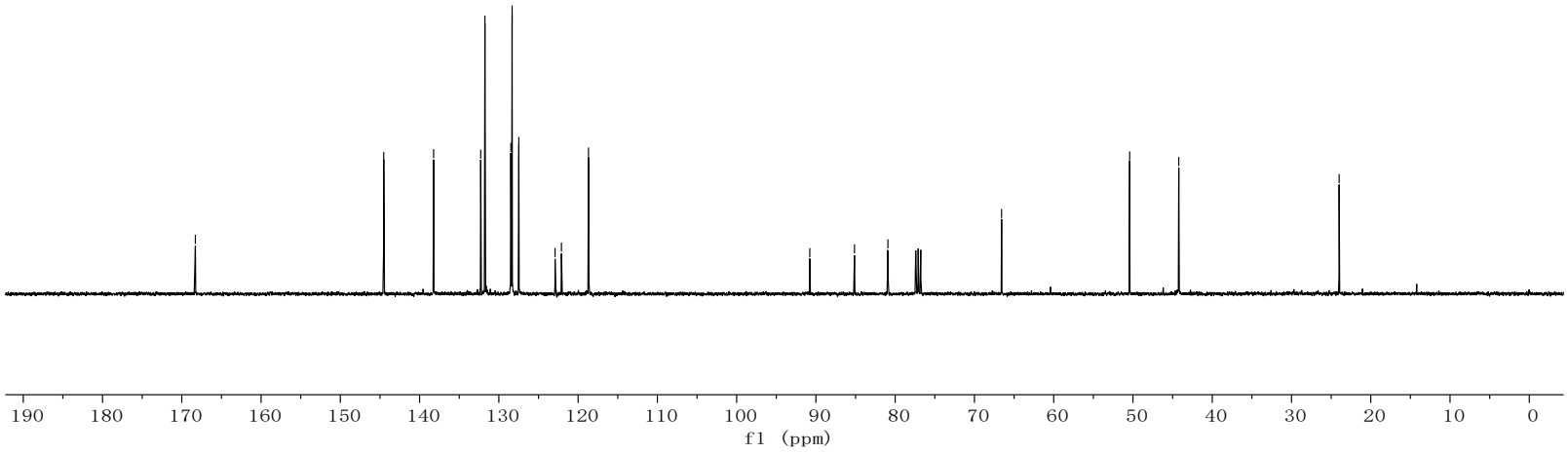

C-EP+[E].1.fid
7.47 7.47 7.46 7.46 7.46 7.46 7.45 7.45 7.33 7.32 7.32 7.32 7.31 7.31 6.58 6.57 6.24 6.24 6.22 6.21 5.76 5.74 5.72 5.71 5.69 5.60 5.58 5.15 5.15 5.11 5.11 5.10 5.10 5.09 5.07 5.07 5.07 4.57 3.18 2.50 2.48 1.94
Ph
HO
OMe
NHAc
30
2.02 3.06 1.00 2.00 1.12 1.00 1.14 1.05 0.91 3.01 2.03 3.02
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 -0.5
f1 (ppm)

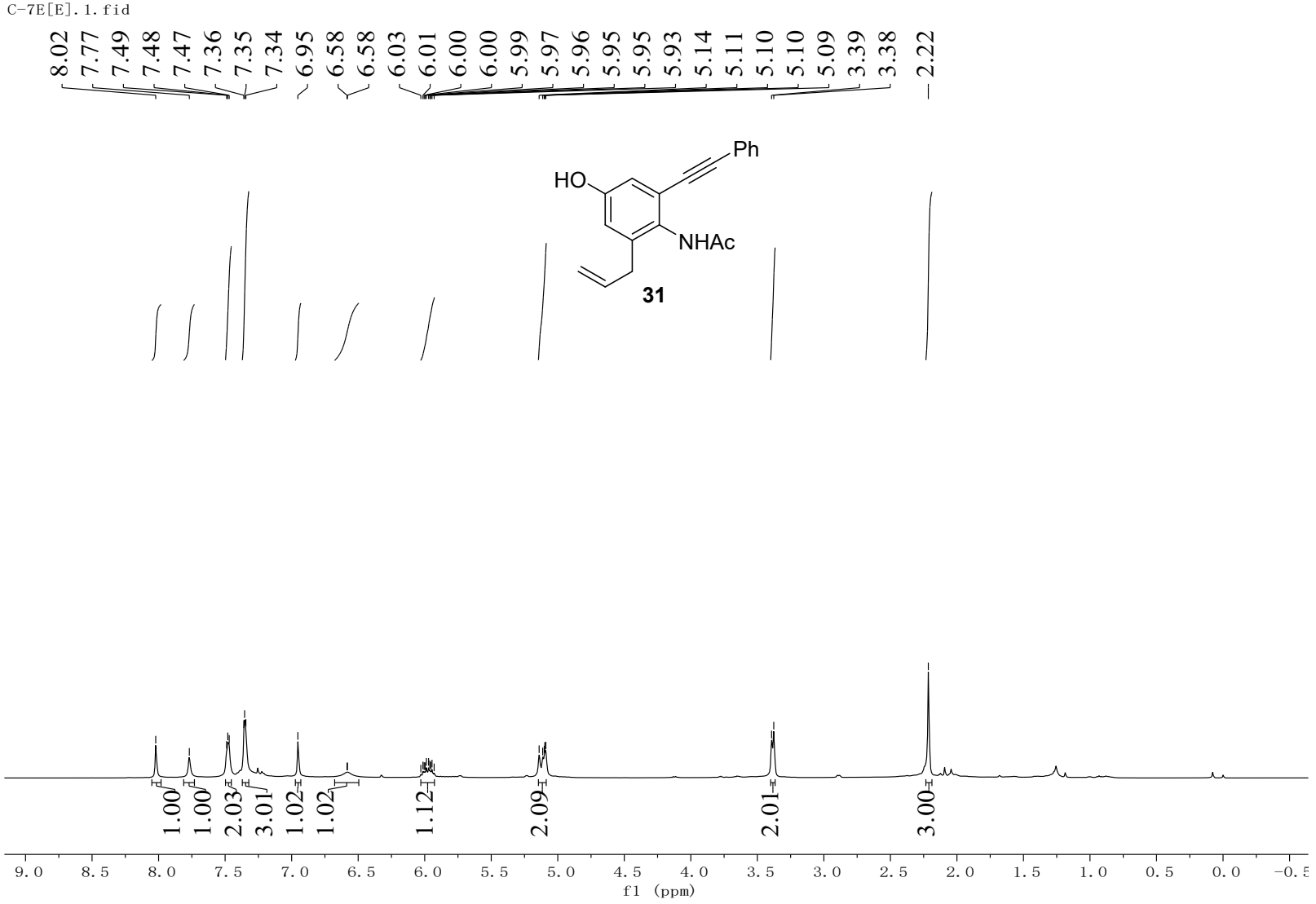
C-EP+[E]-C.1.fid

168.29
144.53
138.23
132.29
131.76
128.49
128.33
127.48
122.91
122.10
118.69
90.77
85.13
80.92
66.58
50.42
44.22
23.99



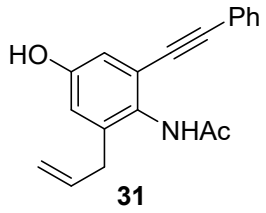
30



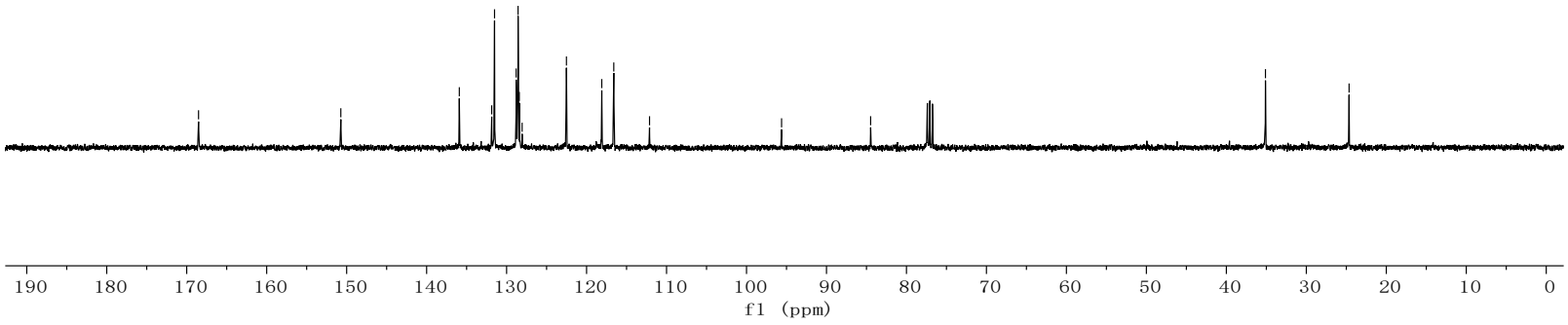

C-7E[E].1.fid
8.02
7.77
7.49
7.48
7.47
7.36
7.35
7.34
6.95
6.58
6.58
6.03
6.01
6.00
6.00
5.99
5.97
5.96
5.95
5.95
5.93
5.14
5.11
5.10
5.10
5.09
3.39
3.38
2.22
HO
Ph
NHAc
31
1.00
1.00
2.03
3.01
1.02
1.02
1.12
2.09
2.01
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

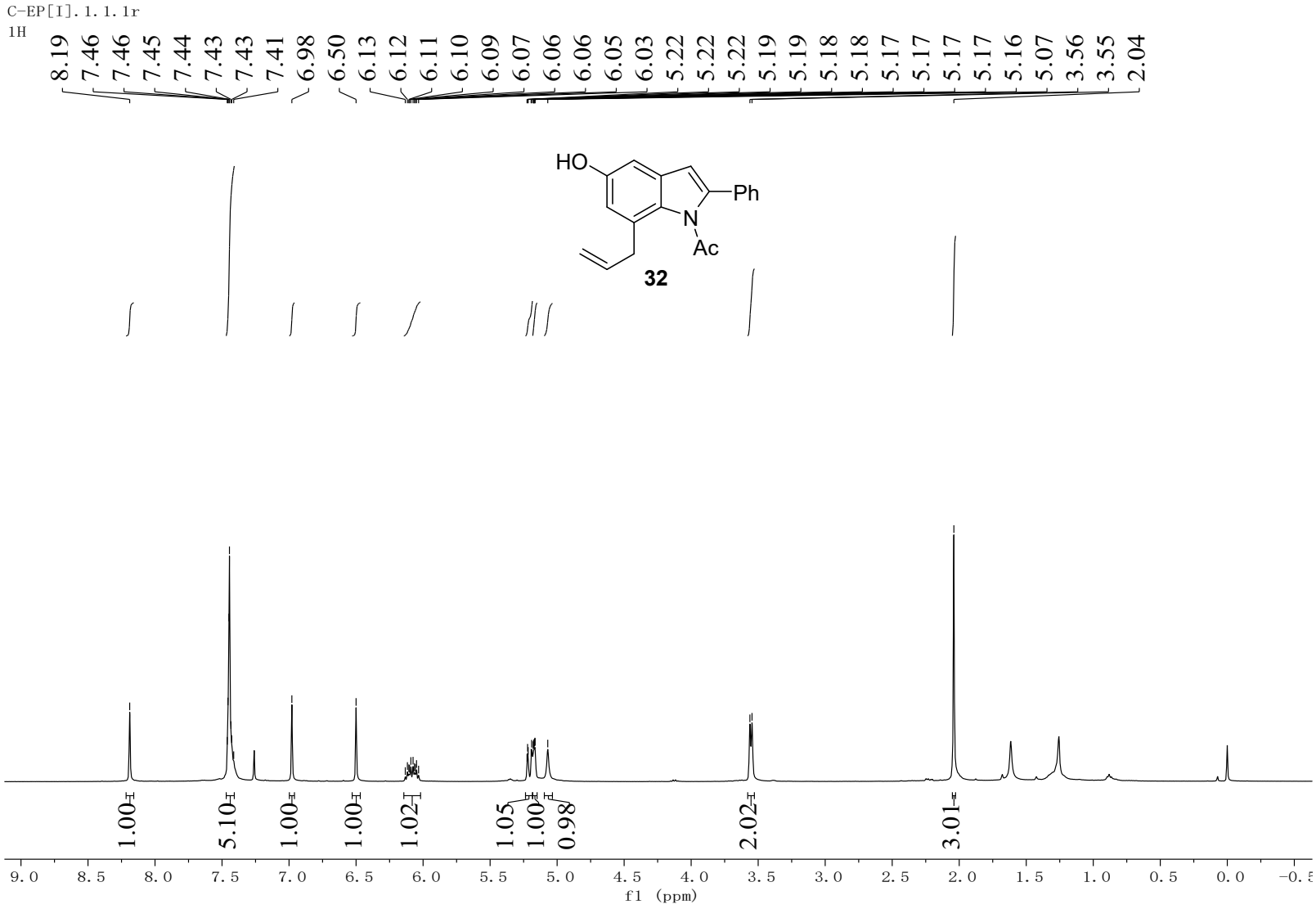
C-7E[E]-C.1.fid

168.52
150.73
135.91
131.86
131.51
128.80
128.57
128.41
128.06
122.52
118.10
116.60
112.13
95.62
84.48
35.11
24.65



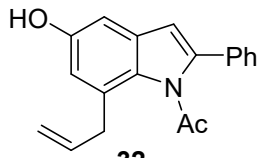
31



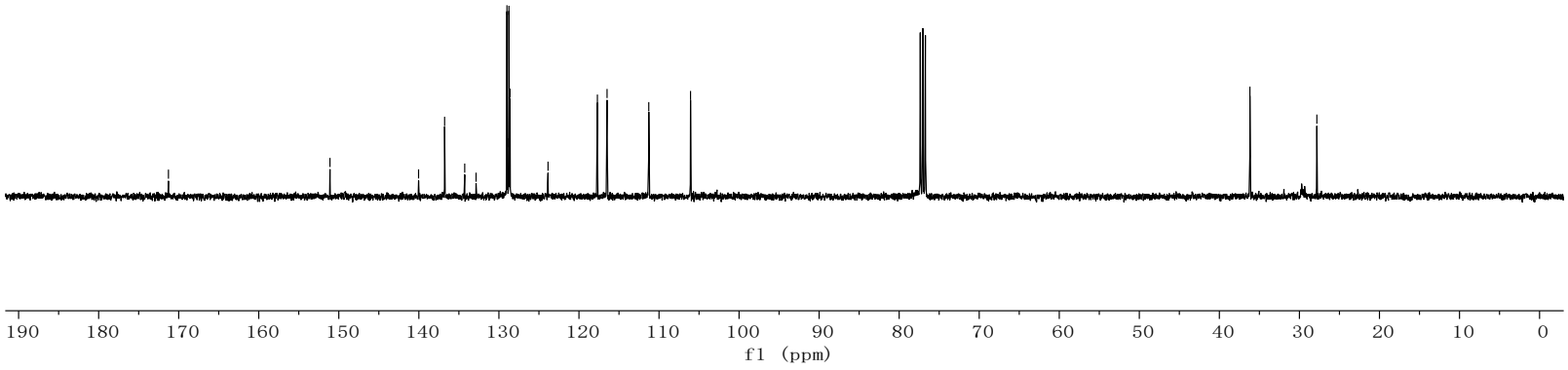

C-EP[I].1.1.1r
1H
8.19
7.46
7.46
7.45
7.44
7.43
7.43
7.41
6.98
6.50
6.13
6.12
6.11
6.10
6.09
6.07
6.06
6.06
6.05
6.03
5.22
5.22
5.22
5.19
5.19
5.18
5.18
5.17
5.17
5.17
5.17
5.16
5.07
3.56
3.55
2.04
HO
Ph
N
Ac
32
1.00
5.10
1.00
1.00
1.02
1.05
1.00
0.98
2.02
3.01
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

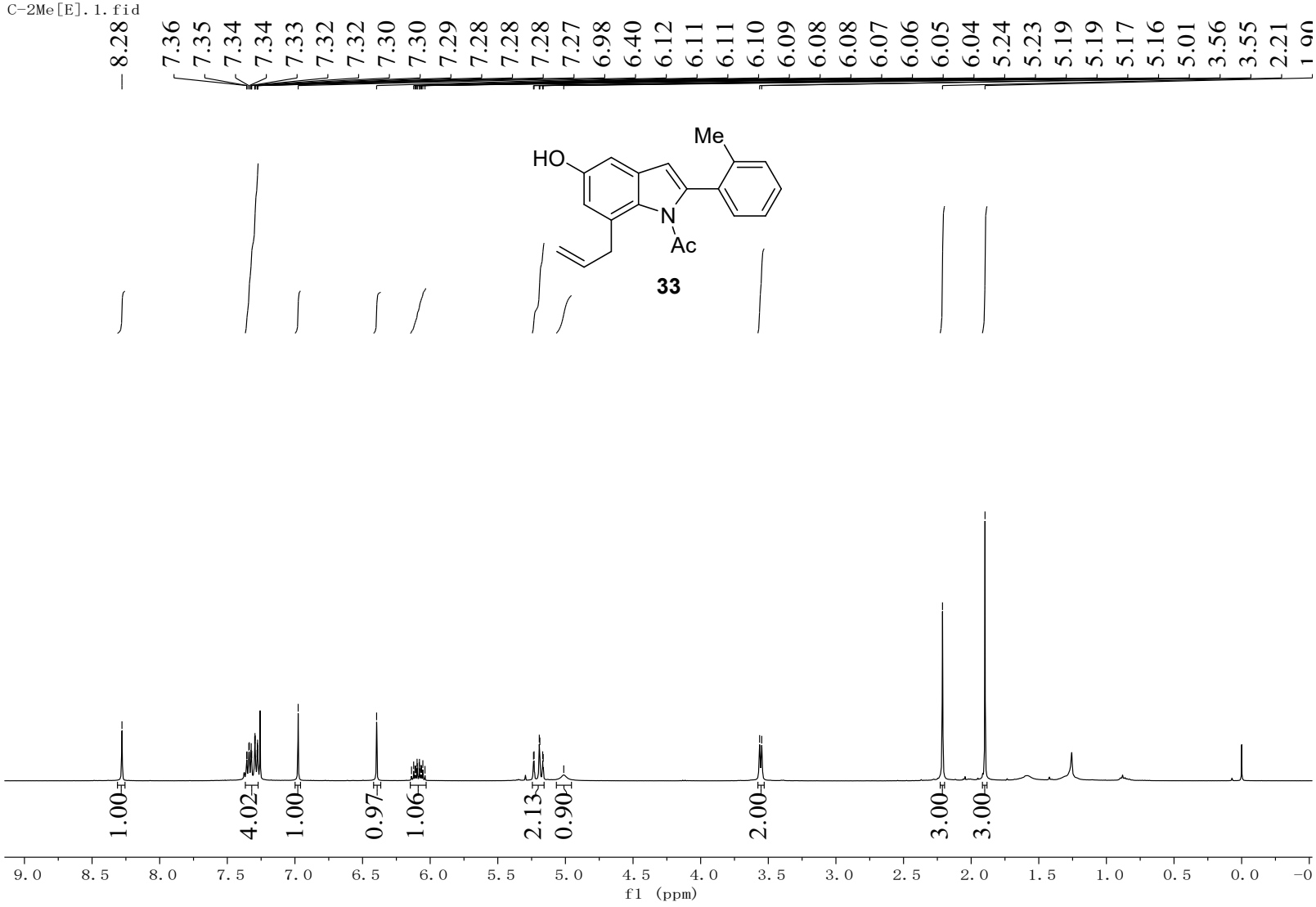
C-EP[I]-C.1.1.1r
13C

171.28
151.10
140.04
136.78
134.27
132.85
129.00
128.77
128.75
128.61
123.86
117.70
116.49
111.28
106.06
36.18
27.82



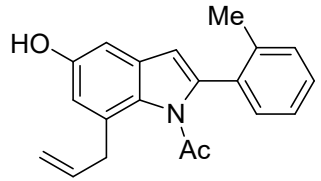
32



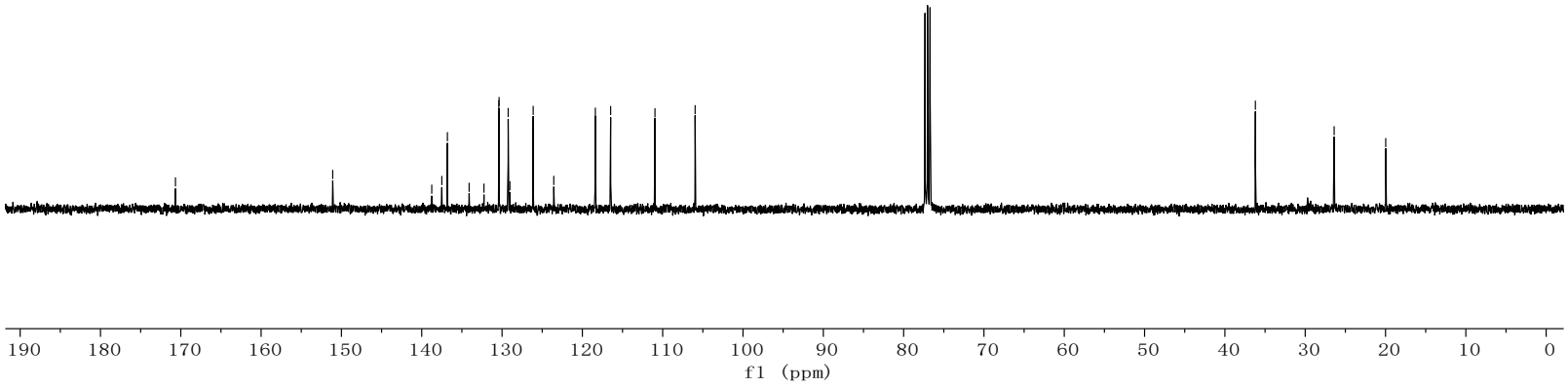

C-2Me[E].1.fid
8.28
7.36
7.35
7.34
7.34
7.33
7.32
7.32
7.30
7.30
7.29
7.28
7.28
7.28
7.27
6.98
6.40
6.12
6.11
6.11
6.10
6.09
6.08
6.08
6.07
6.06
6.05
6.04
5.24
5.23
5.19
5.19
5.17
5.16
5.01
3.56
3.55
2.21
HO
Me
N
Ac
33
1.00
4.02
1.00
0.97
1.06
2.13
0.90
2.00
3.00
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

C-2Me[E]-C.1.fid

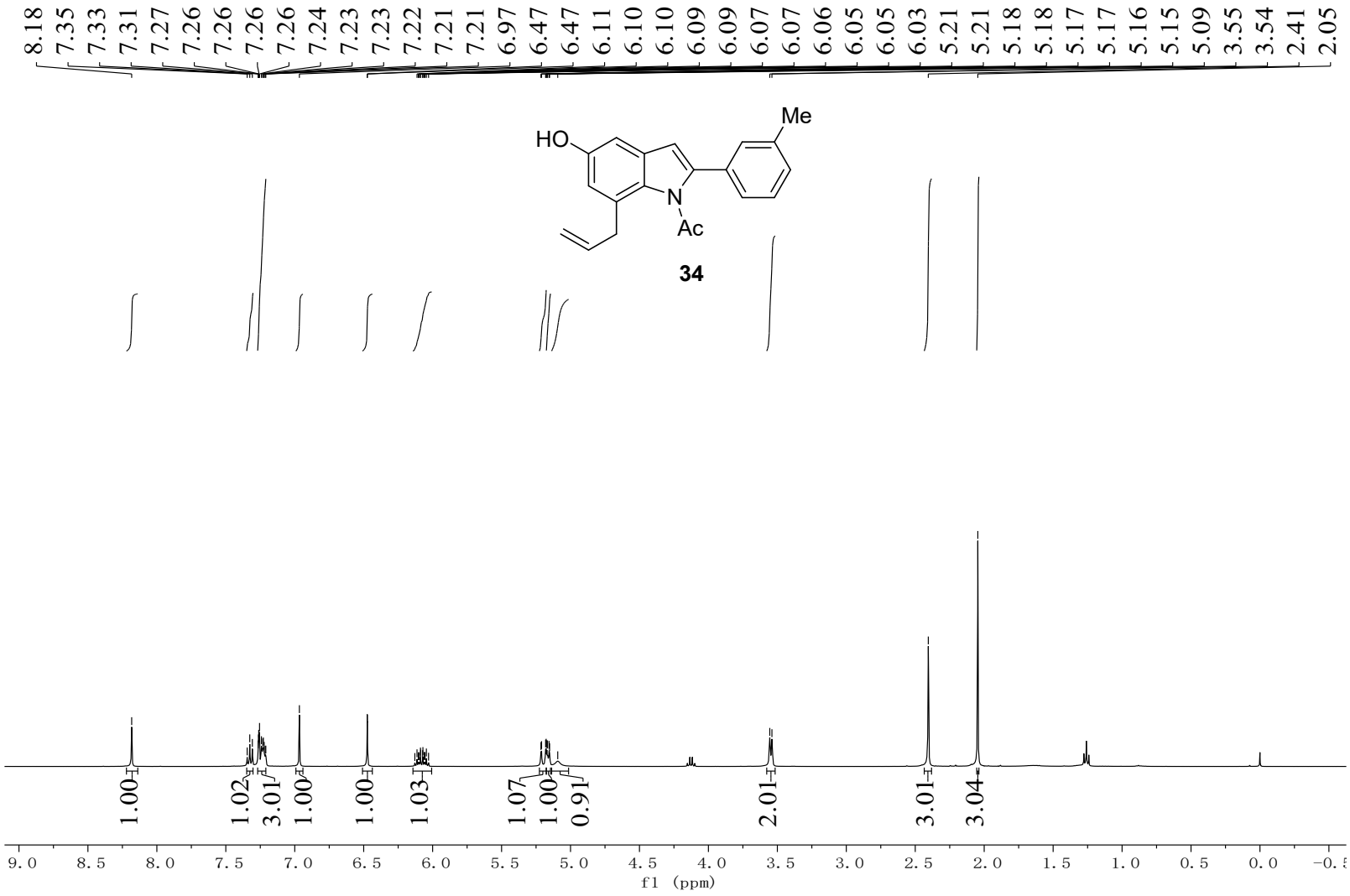
170.66
151.08
138.75
137.50
136.80
134.10
132.25
130.40
130.37
129.24
129.02
126.12
123.56
118.38
116.48
110.96
105.96
36.20
26.40
19.98



33

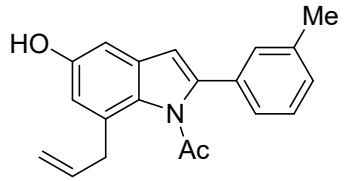


C-3Me[E].1.fid

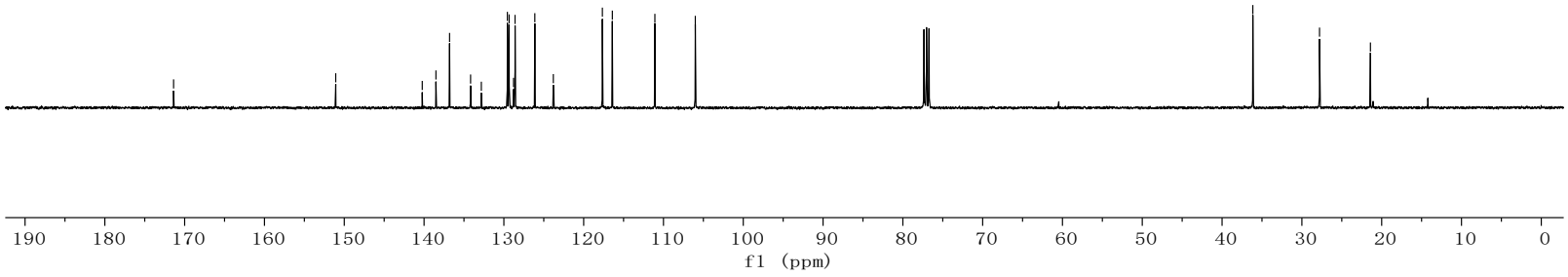

8.18
7.35
7.33
7.31
7.27
7.26
7.26
7.26
7.26
7.24
7.23
7.23
7.22
7.21
7.21
6.97
6.47
6.47
6.11
6.10
6.10
6.09
6.09
6.07
6.07
6.06
6.05
6.05
6.03
5.21
5.21
5.18
5.18
5.17
5.17
5.16
5.15
5.09
3.55
3.54
2.41
2.05
HO
Me
N
Ac
34
1.00
1.02
3.01
1.00
1.00
1.03
1.07
1.00
0.91
2.01
3.01
3.04
9.0
8.5
8.0
7.5
7.0
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)

C-3Me[E]-C.1.fid

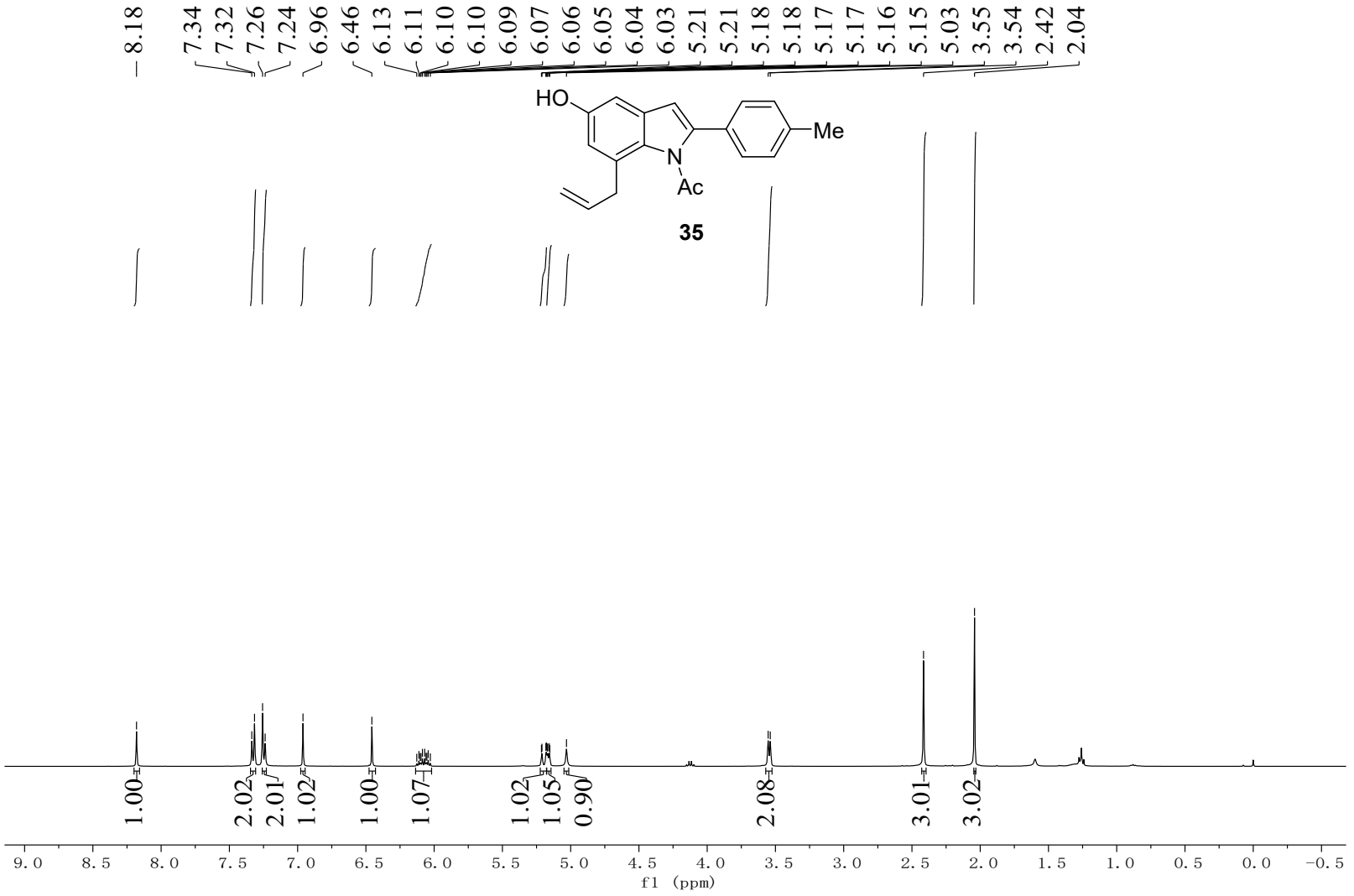
171.39
151.07
140.23
138.51
136.80
134.17
132.82
129.56
129.34
128.78
128.60
126.12
123.80
117.65
116.41
111.08
106.01
36.14
27.79
21.41



34

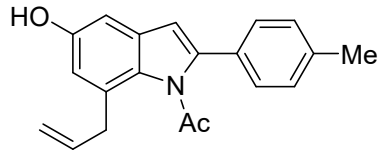


C-4Me[E].1.fid

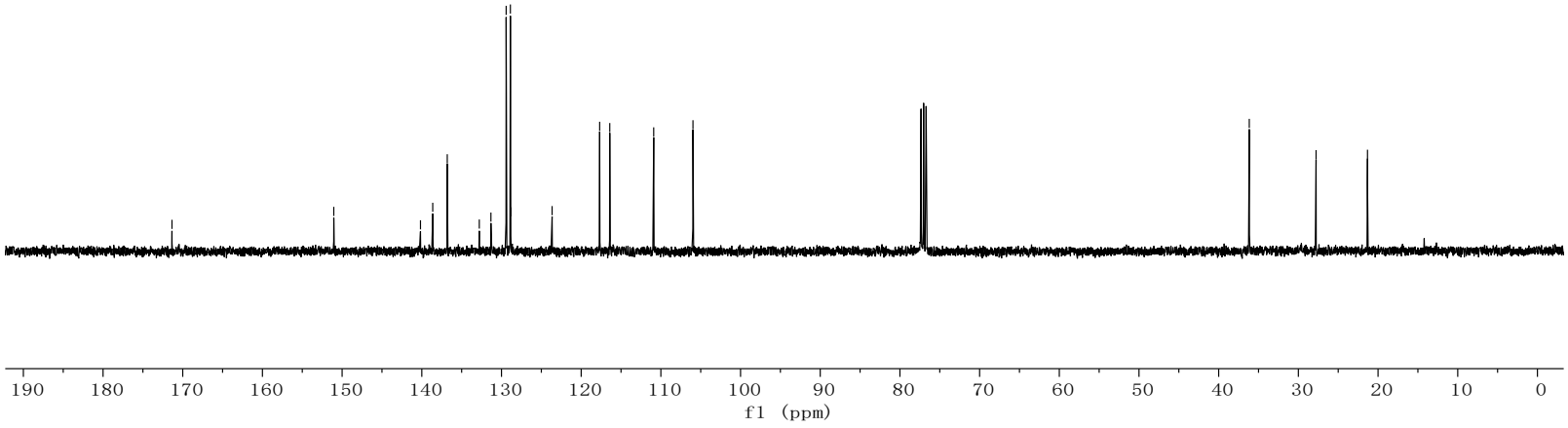

8.18
7.34
7.32
7.26
7.24
6.96
6.46
6.13
6.11
6.10
6.10
6.09
6.07
6.06
6.05
6.04
6.03
5.21
5.21
5.18
5.18
5.17
5.17
5.16
5.15
5.03
3.55
3.54
2.42
2.04
HO
Me
N
Ac
35
1.00
2.02
2.01
1.02
1.00
1.07
1.02
1.05
0.90
2.08
3.01
3.02
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

C-4Me[E]-C.1.fid

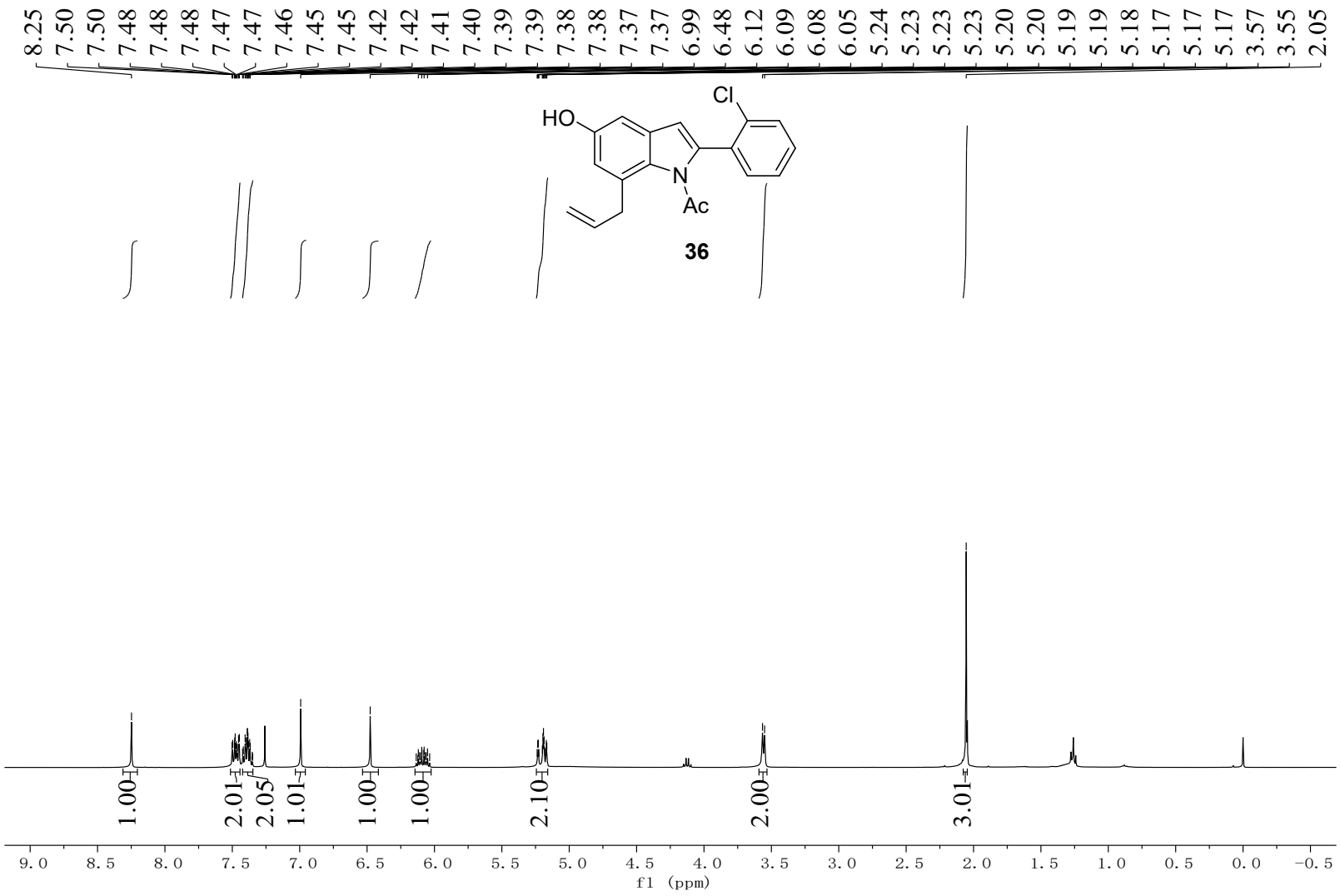
171.35
151.05
140.17
138.63
136.81
132.79
131.33
129.41
128.88
128.83
123.65
117.68
116.41
110.90
105.97
36.16
27.78
21.33



35

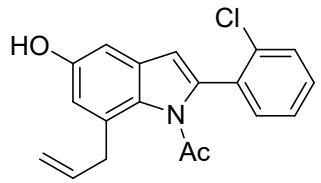


C-2Cl[E].1.fid

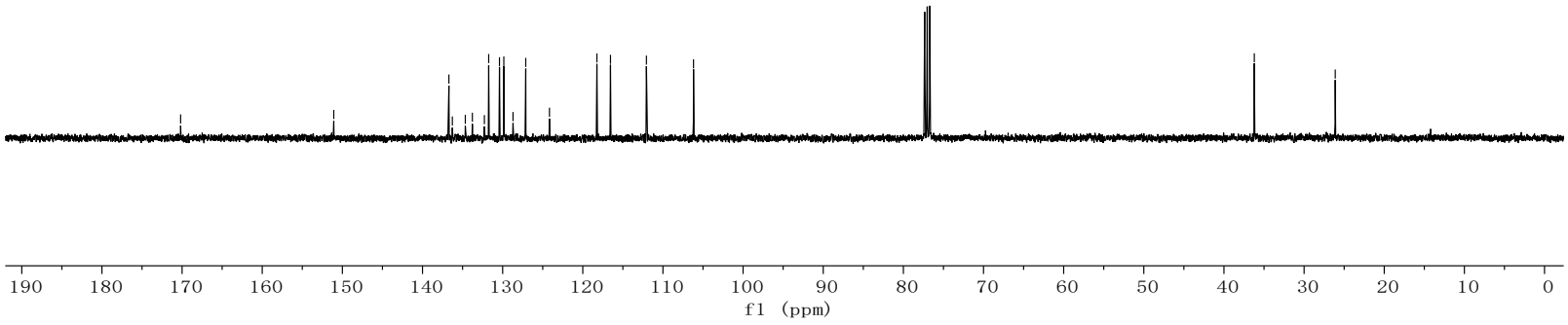

8.25
7.50
7.50
7.48
7.48
7.48
7.47
7.47
7.46
7.45
7.45
7.42
7.42
7.41
7.40
7.39
7.39
7.38
7.38
7.37
7.37
6.99
6.48
6.12
6.09
6.08
6.05
5.24
5.23
5.23
5.23
5.20
5.20
5.19
5.19
5.18
5.17
5.17
5.17
3.57
3.55
2.05
HO
Cl
N
Ac
36
1.00
2.01
2.05
1.01
1.00
1.00
2.10
2.00
3.01
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

C-2Cl[E]-C.1.fid

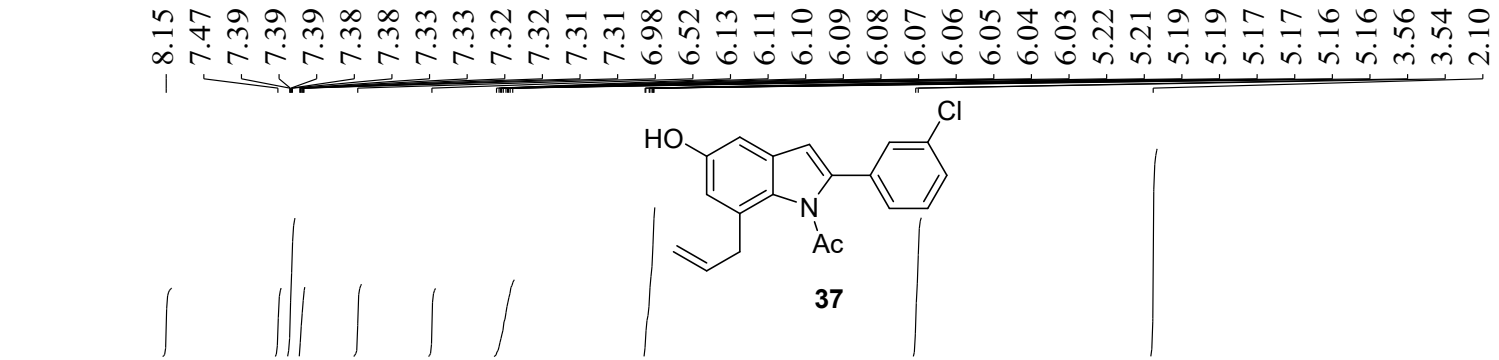
170.18
151.07
136.71
136.28
134.66
133.78
132.28
131.74
130.40
129.85
128.71
127.12
124.16
118.24
116.55
112.06
106.18
36.21
26.11

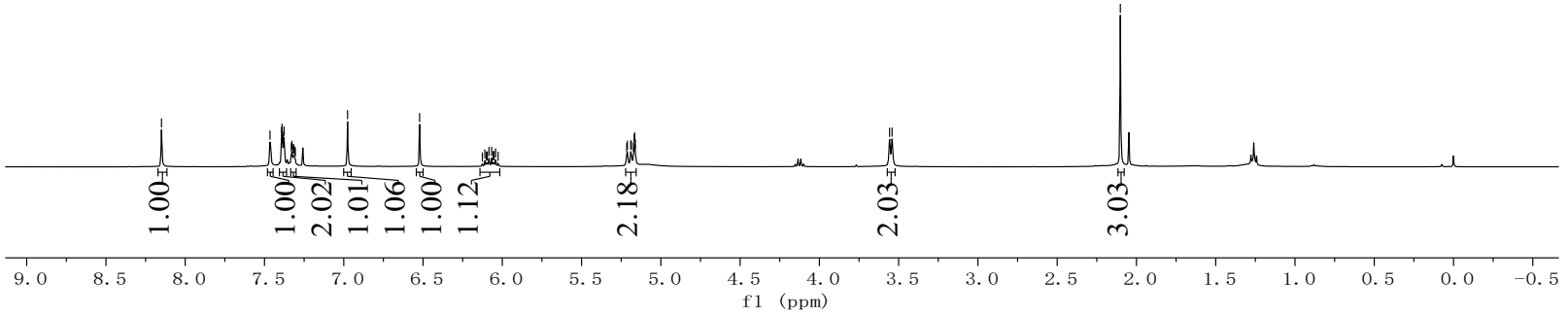


36



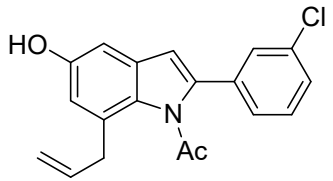
C-3Cl[E].1.fid


8.15
7.47
7.39
7.39
7.39
7.38
7.38
7.33
7.33
7.32
7.32
7.31
7.31
6.98
6.52
6.13
6.11
6.10
6.09
6.08
6.07
6.06
6.05
6.04
6.03
5.22
5.21
5.19
5.19
5.17
5.17
5.16
5.16
3.56
3.54
2.10
HO
Cl
N
Ac
37

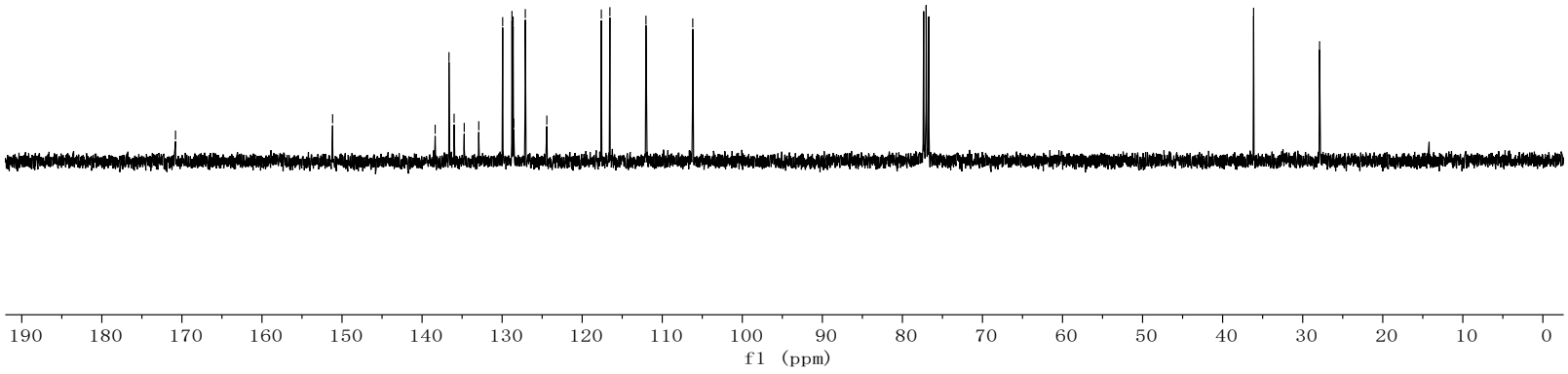

1.00
1.00
2.02
1.01
1.06
1.00
1.12
2.18
2.03
3.03
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

C-3Cl[E]-C.1.fid

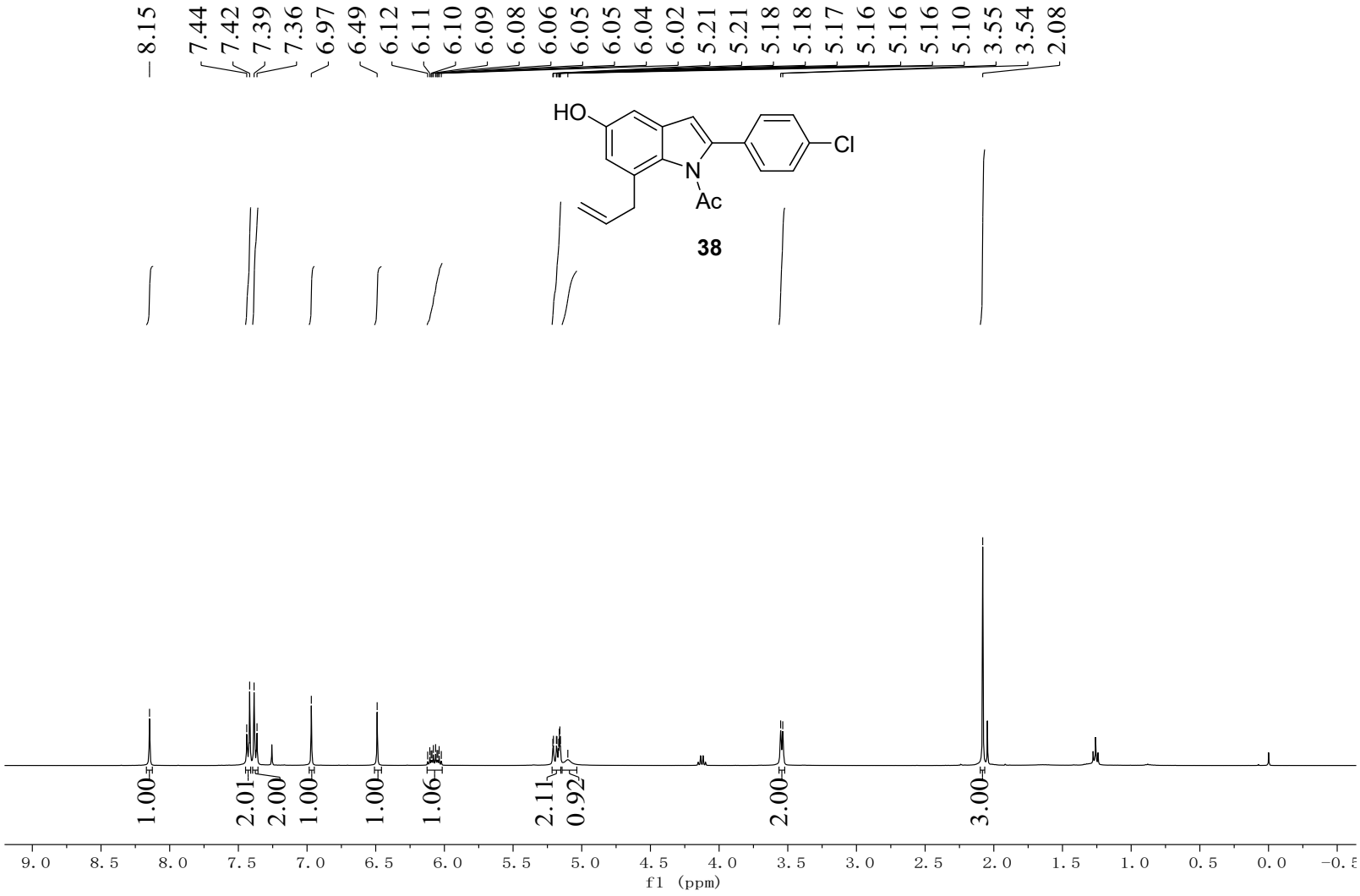
170.80
151.17
138.36
136.64
136.00
134.73
132.92
129.93
128.77
128.63
128.54
127.10
124.42
117.61
116.54
112.03
106.19
36.13
27.89



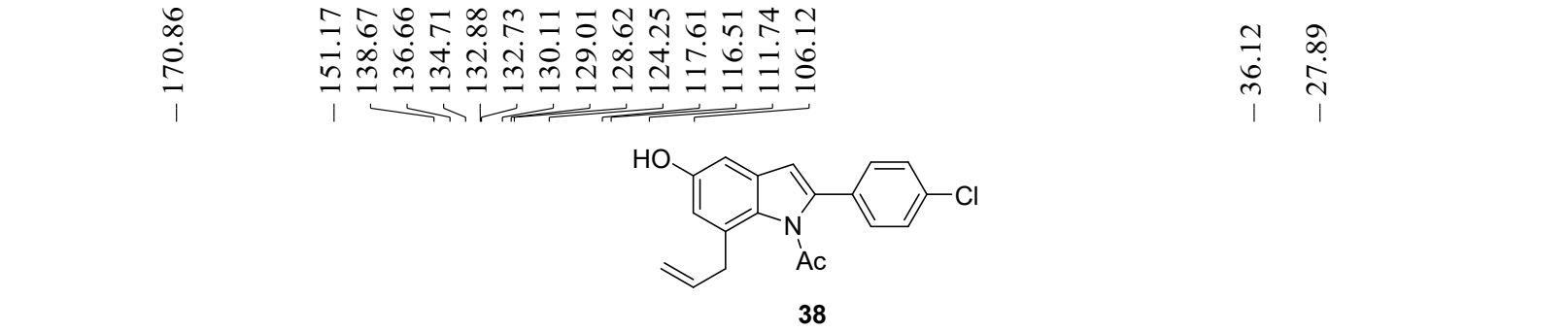
37



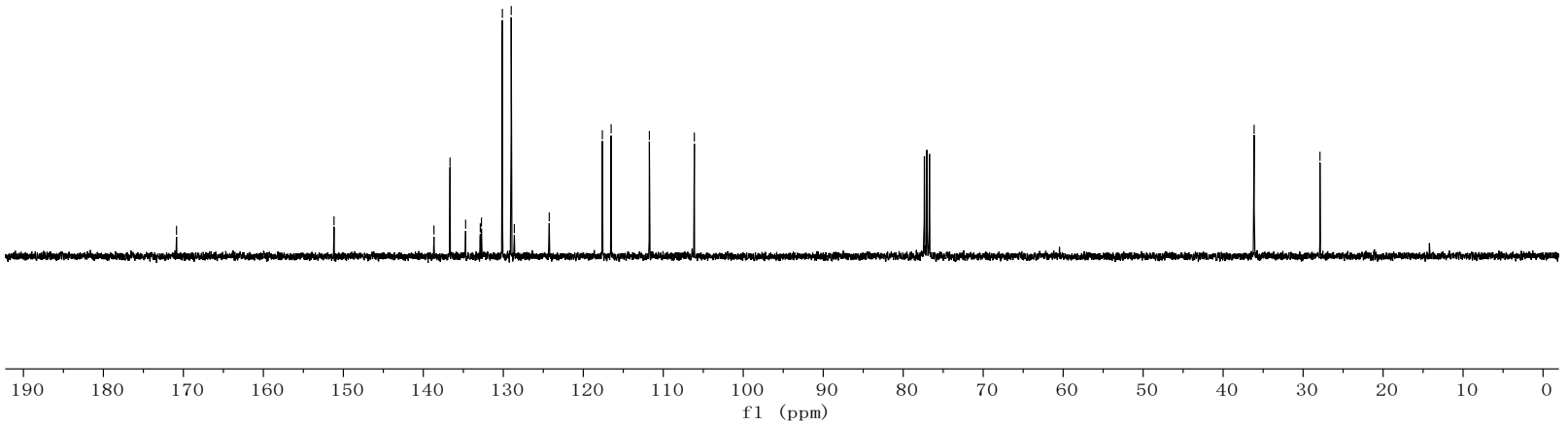
C-4Cl[E].1.fid


8.15
7.44
7.42
7.39
7.36
6.97
6.49
6.12
6.11
6.10
6.09
6.08
6.06
6.05
6.05
6.04
6.02
5.21
5.21
5.18
5.18
5.17
5.16
5.16
5.16
5.10
3.55
3.54
2.08
HO
Cl
N
Ac
38
1.00
2.01
2.00
1.00
1.00
1.06
2.11
0.92
2.00
3.00
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)

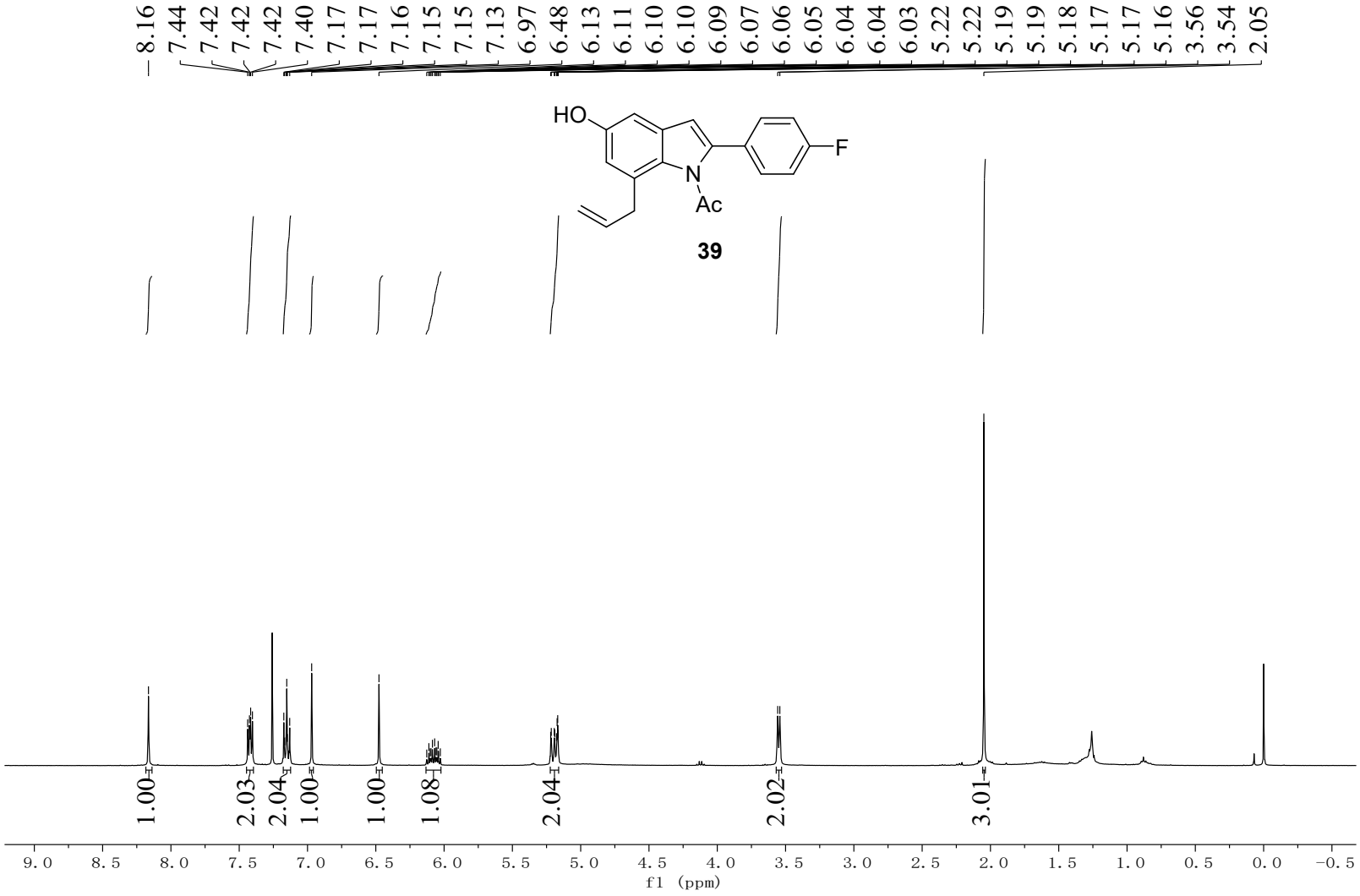
C-4Cl[E]-C.1.fid

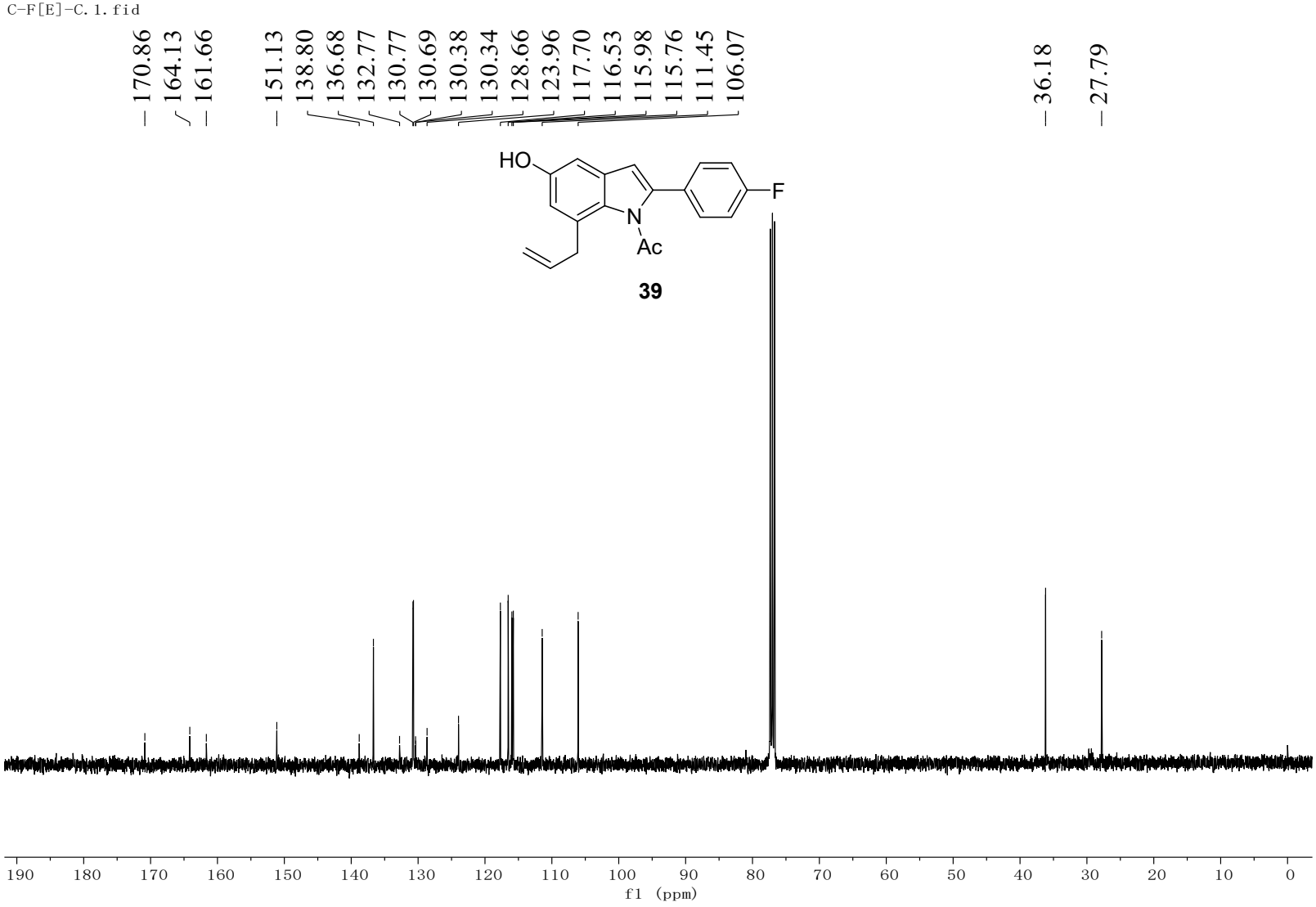


38

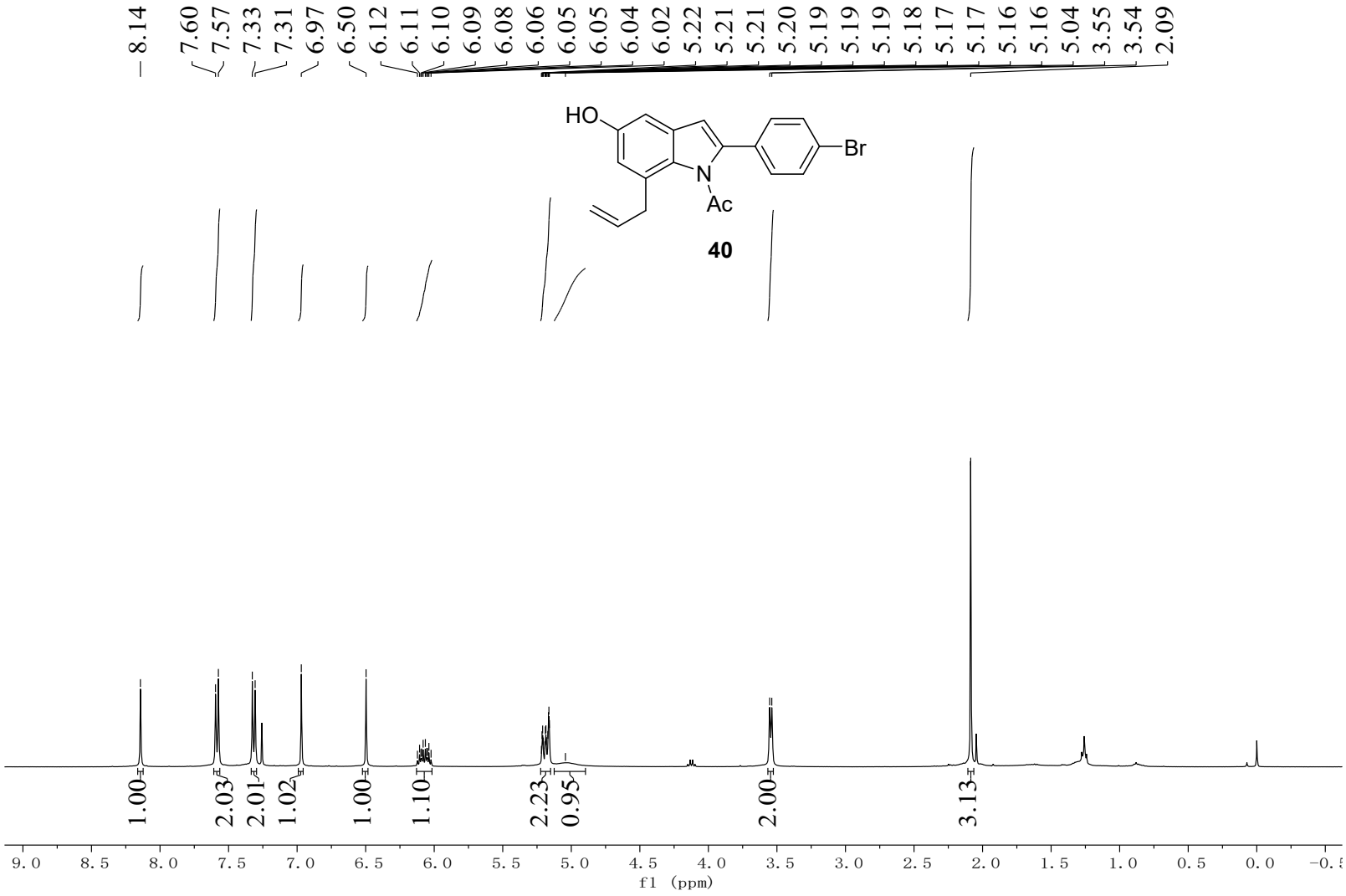


C-F[E].1.fid


8.16
7.44
7.42
7.42
7.42
7.40
7.17
7.17
7.16
7.15
7.15
7.13
6.97
6.48
6.13
6.11
6.10
6.10
6.09
6.07
6.06
6.05
6.04
6.04
6.03
5.22
5.22
5.19
5.19
5.18
5.17
5.17
5.16
3.56
3.54
2.05
HO
F
N
Ac
39
1.00
2.03
2.04
1.00
1.00
1.08
2.04
2.02
3.01
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
-0.5
f1 (ppm)

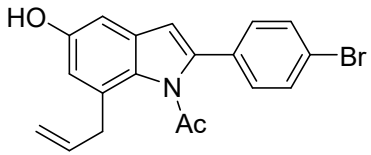

C-F[E]-C.1.fid
170.86
164.13
161.66
151.13
138.80
136.68
132.77
130.77
130.69
130.38
130.34
128.66
123.96
117.70
116.53
115.98
115.76
111.45
106.07
36.18
27.79
HO
F
N
Ac
39
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

C-Br[E].1.fid

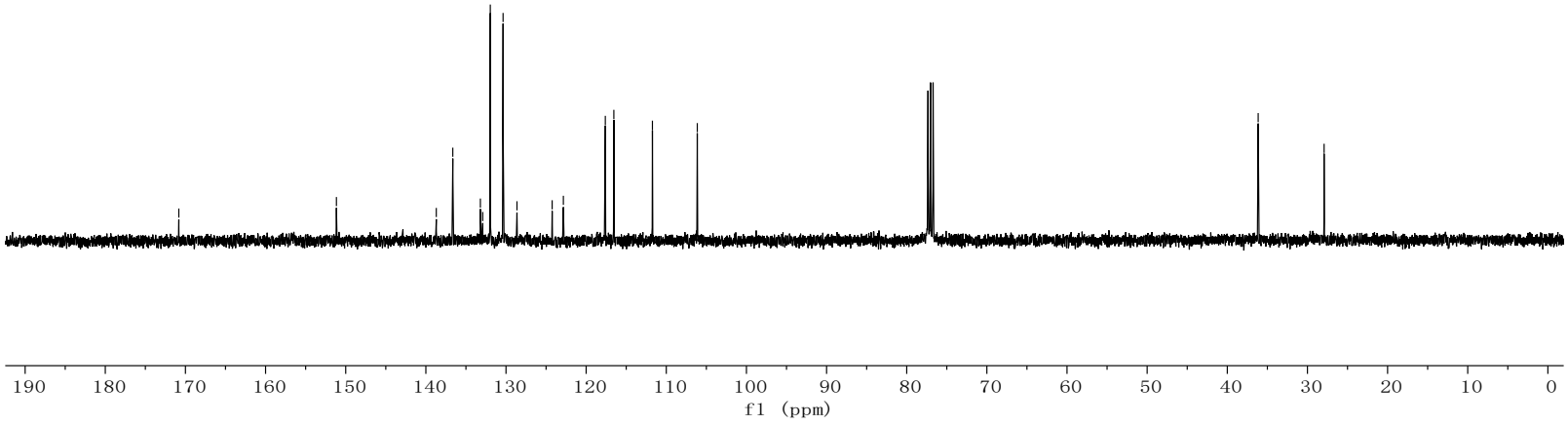

8.14
7.60
7.57
7.33
7.31
6.97
6.50
6.12
6.11
6.10
6.09
6.08
6.06
6.05
6.05
6.04
6.02
5.22
5.21
5.21
5.20
5.19
5.19
5.19
5.18
5.17
5.17
5.16
5.16
5.04
3.55
3.54
2.09
HO
Br
N
Ac
40
1.00
2.03
2.01
1.02
1.00
1.10
2.23
0.95
2.00
3.13
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

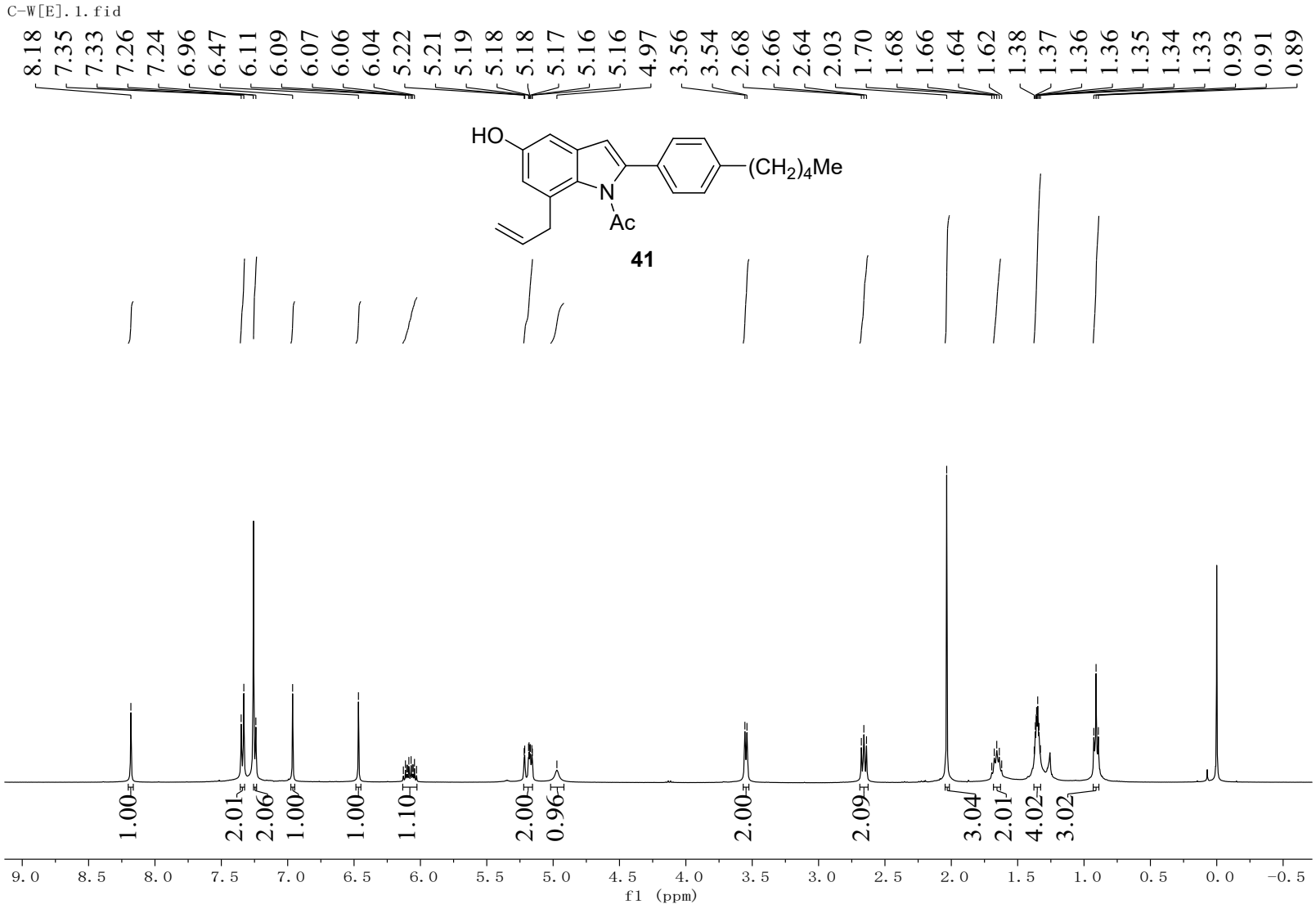
C-Br[E]-C.1.fid

170.82
151.16
138.70
136.64
133.20
132.90
131.96
130.37
128.63
124.24
122.84
117.61
116.54
111.73
106.13
36.15
27.92



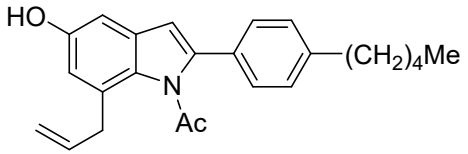
40



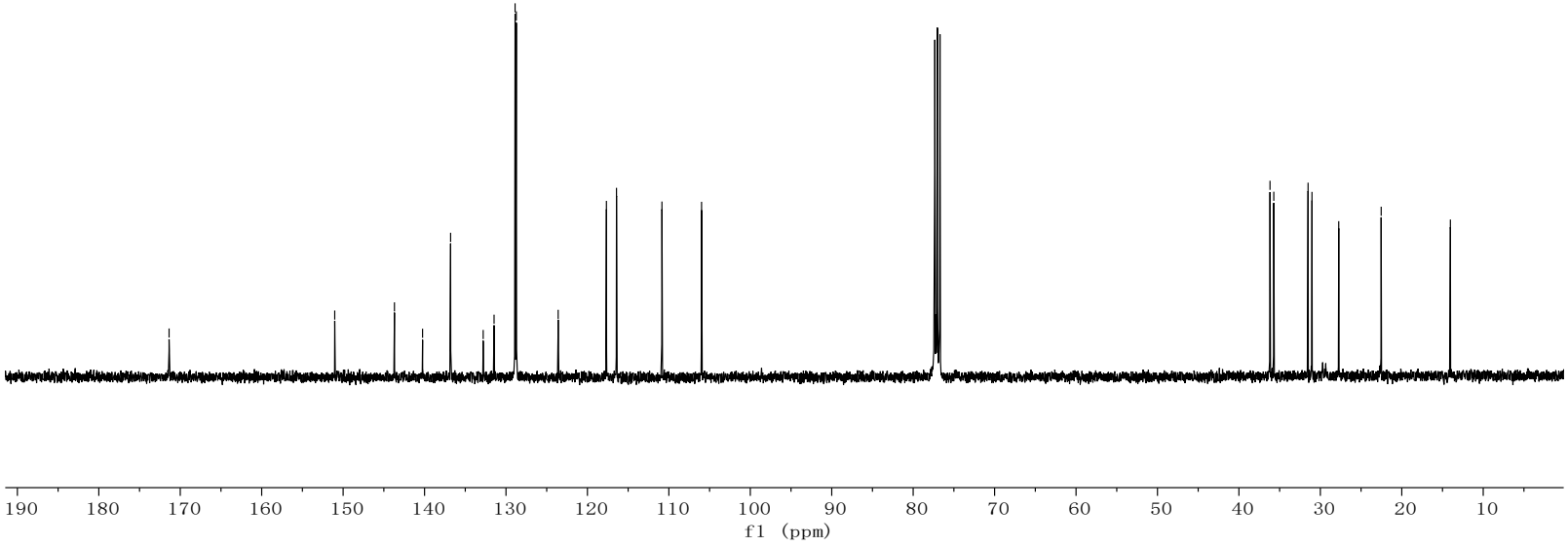

C-W[E].1.fid
8.18
7.35
7.33
7.26
7.24
6.96
6.47
6.11
6.09
6.07
6.06
6.04
5.22
5.21
5.19
5.18
5.18
5.17
5.16
5.16
4.97
3.56
3.54
2.68
2.66
2.64
2.03
1.70
1.68
1.66
1.64
1.62
1.38
1.37
1.36
1.36
1.35
1.34
1.33
0.93
0.91
0.89
HO
(CH2)4Me
N
Ac
41
1.00
2.01
2.06
1.00
1.00
1.10
2.00
0.96
2.00
2.09
3.04
2.01
4.02
3.02
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
-0.5
f1 (ppm)

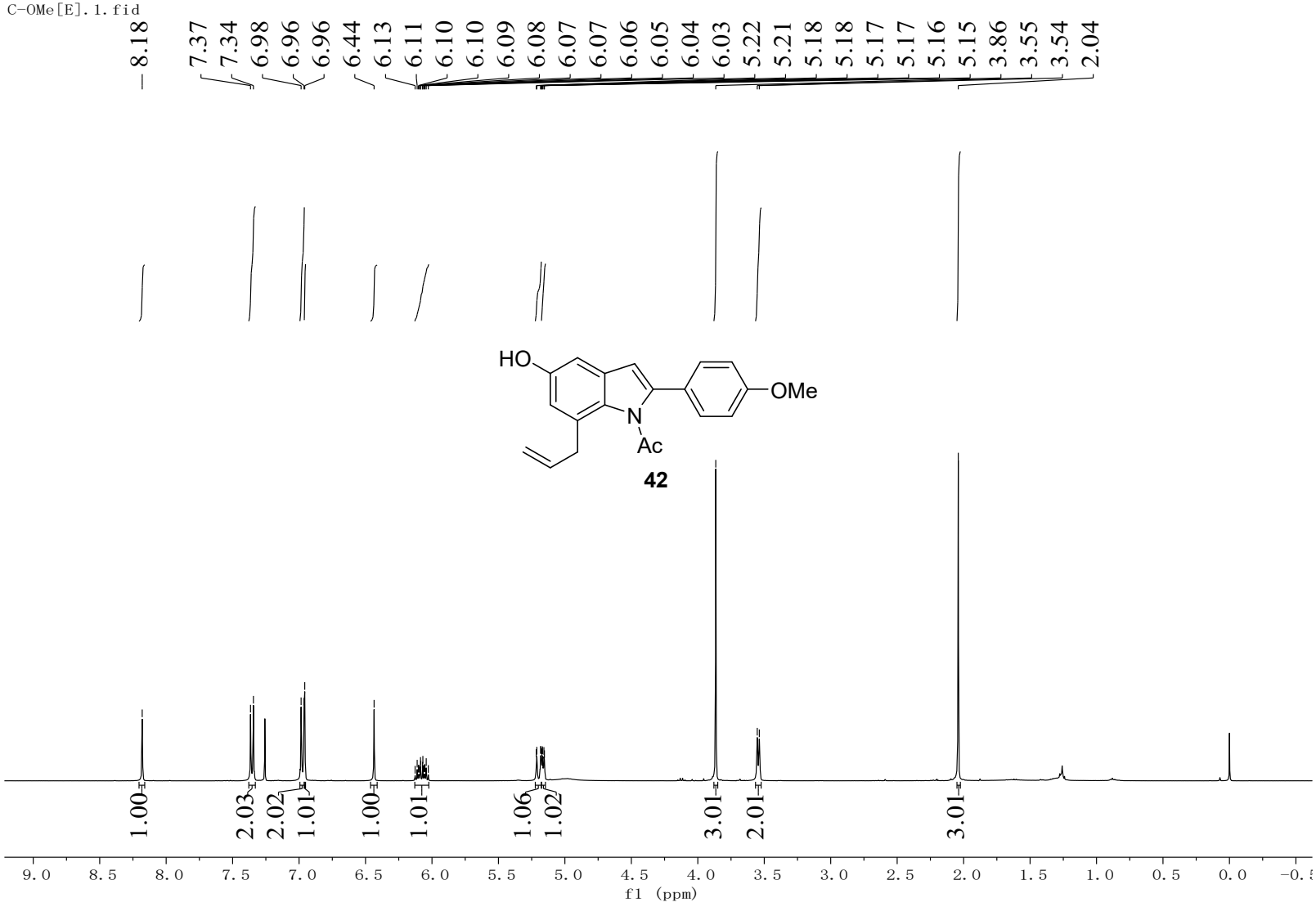
C-W[E]-C.1.fid

171.37
151.03
143.70
140.24
136.80
132.80
131.46
128.88
128.85
128.73
123.59
117.67
116.43
110.85
105.98
36.18
35.72
31.51
31.03
27.74
22.53
14.03



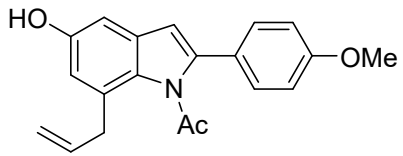
41



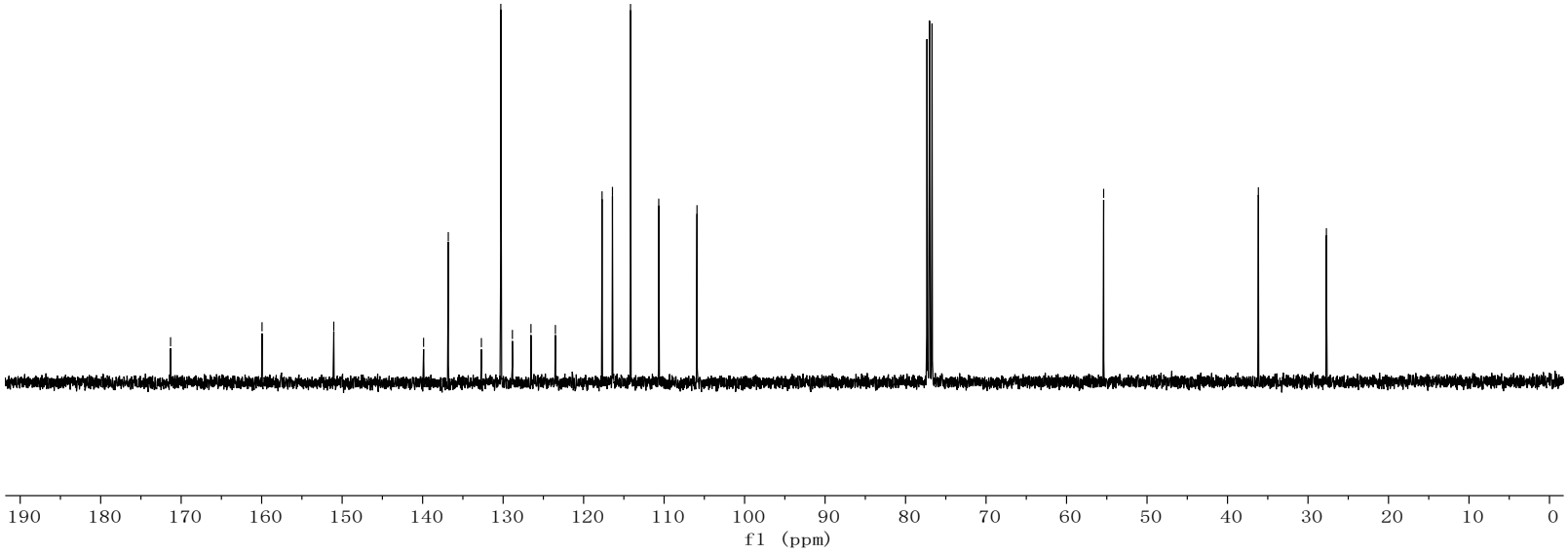

C-OMe[E].1.fid
8.18
7.37
7.34
6.98
6.96
6.96
6.44
6.13
6.11
6.10
6.10
6.09
6.08
6.07
6.07
6.06
6.05
6.04
6.03
5.22
5.21
5.18
5.18
5.17
5.17
5.16
5.15
3.86
3.55
3.54
2.04
HO
OMe
N
Ac
42
1.00
2.03
2.02
1.01
1.00
1.01
1.06
1.02
3.01
2.01
3.01
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

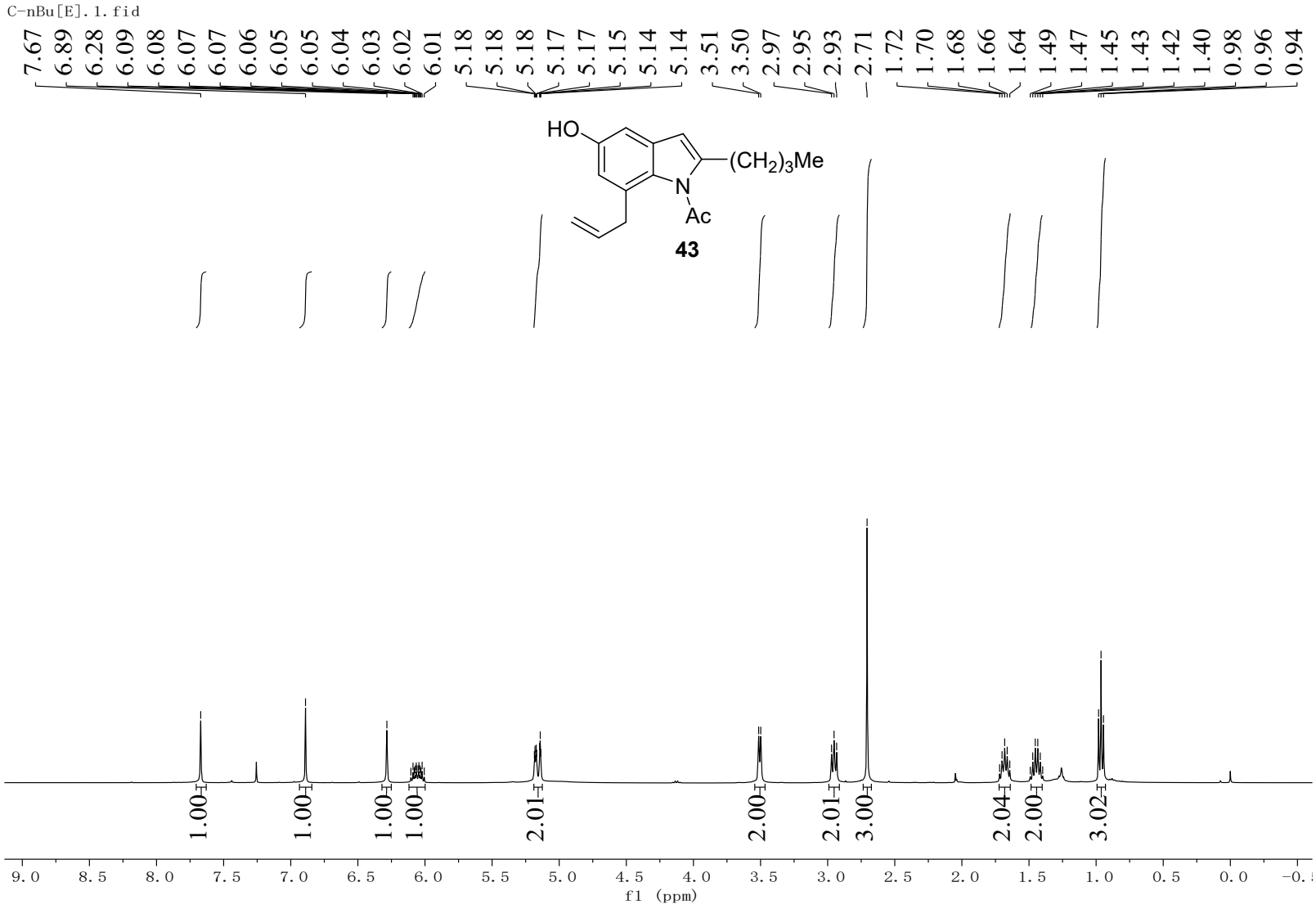
C-OMe[E]-C.1.fid

171.32
159.95
151.04
139.88
136.81
132.70
130.29
128.85
126.54
123.51
117.71
116.42
114.17
110.67
105.91
55.39
36.17
27.72



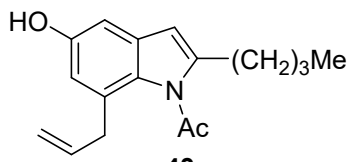
42




C-nBu[E].1.fid
7.67
6.89
6.28
6.09
6.08
6.07
6.07
6.06
6.05
6.05
6.04
6.03
6.02
6.01
5.18
5.18
5.18
5.17
5.17
5.15
5.14
5.14
3.51
3.50
2.97
2.95
2.93
2.71
1.72
1.70
1.68
1.66
1.64
1.49
1.47
1.45
1.43
1.42
1.40
0.98
0.96
0.94
HO
(CH2)3Me
N
Ac
43
1.00
1.00
1.00
1.00
2.01
2.00
2.01
3.00
2.04
2.00
3.02
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)

C-nBu[E]-C.1.fid

170.11
150.53
143.02
136.81
131.56
129.69
121.97
116.66
116.39
107.95
105.90
35.97
31.05
30.37
27.47
22.56
13.97



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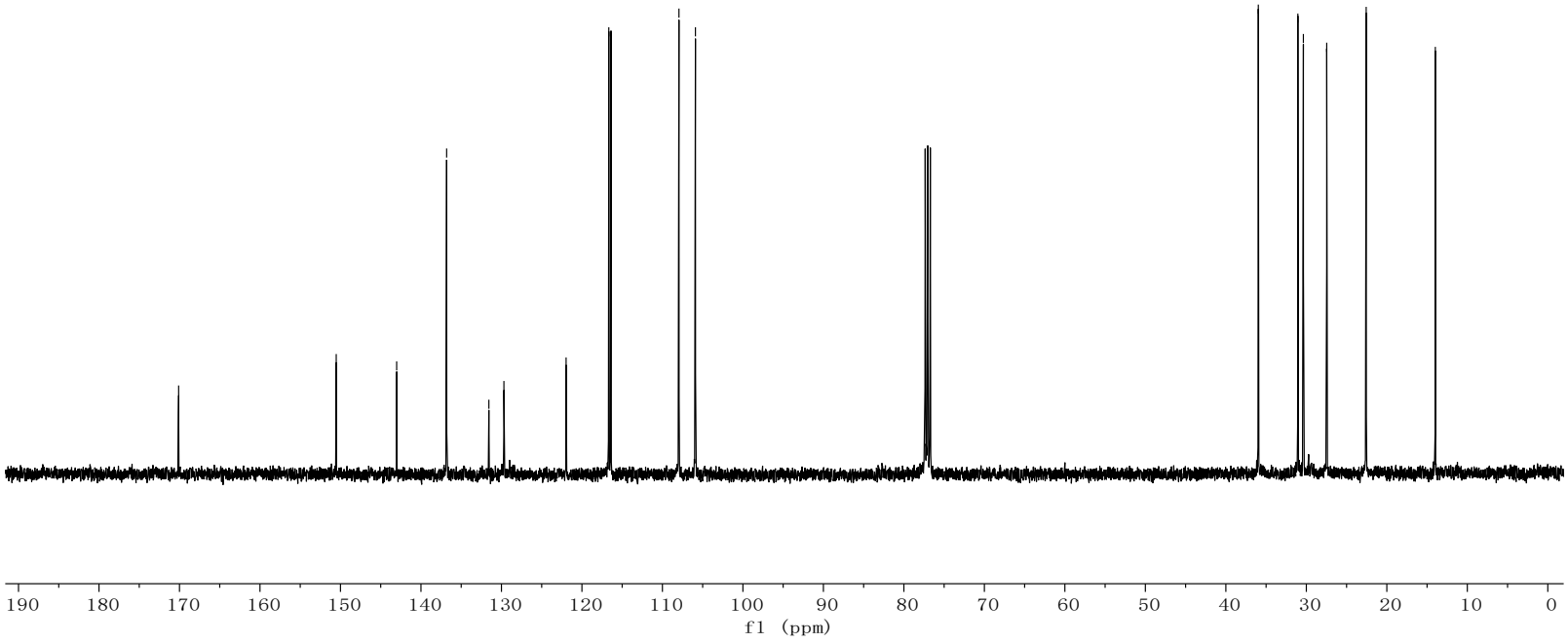
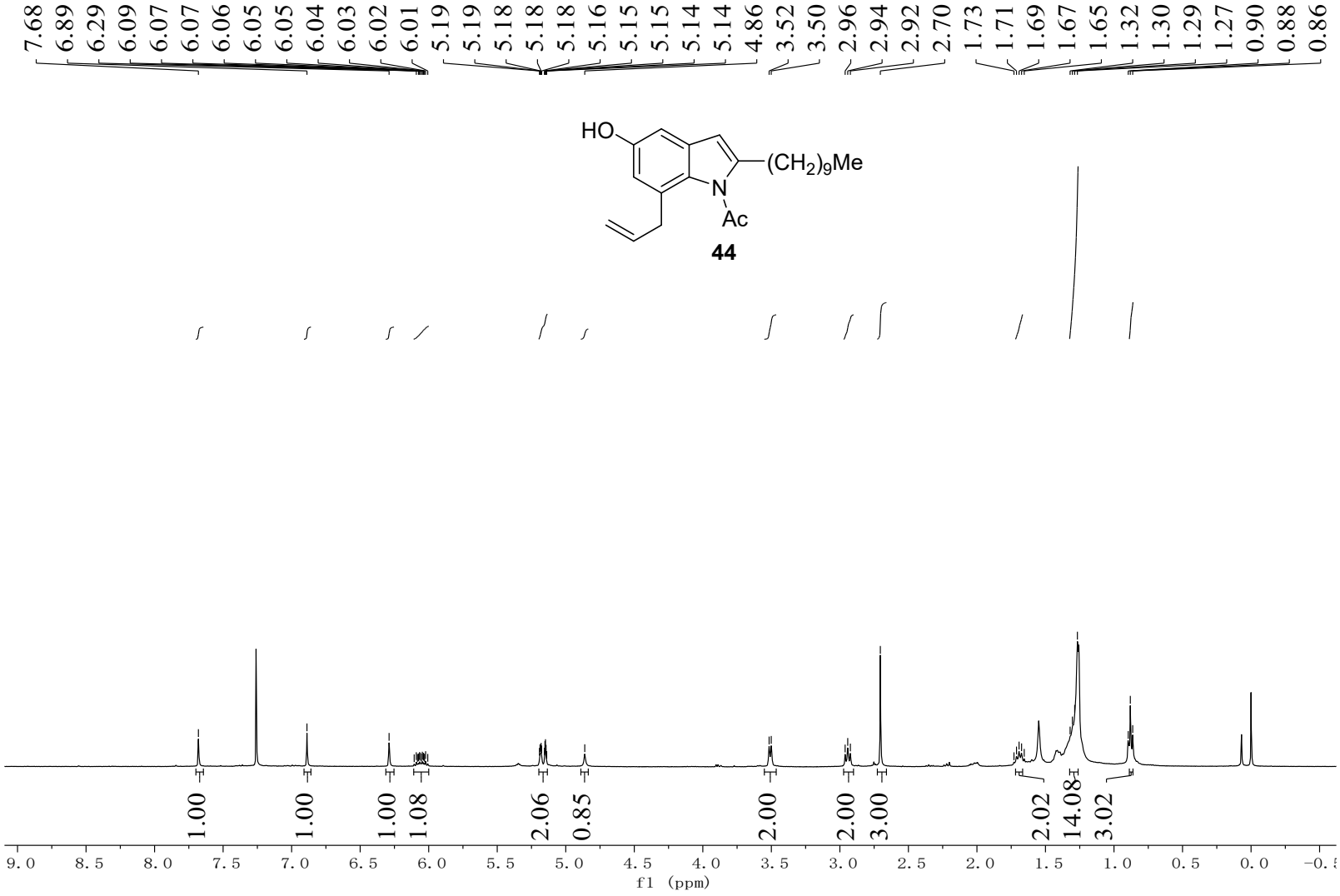
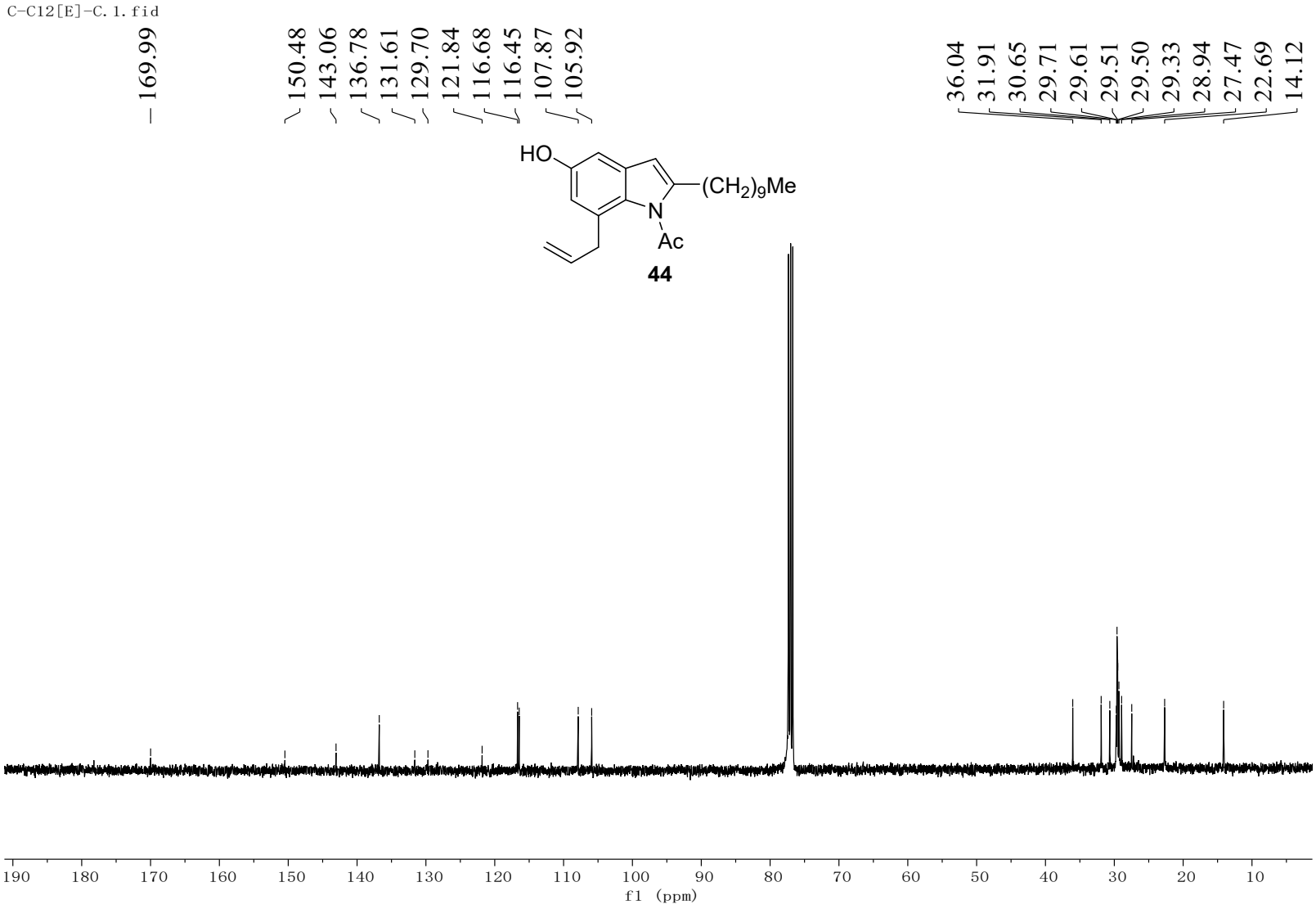
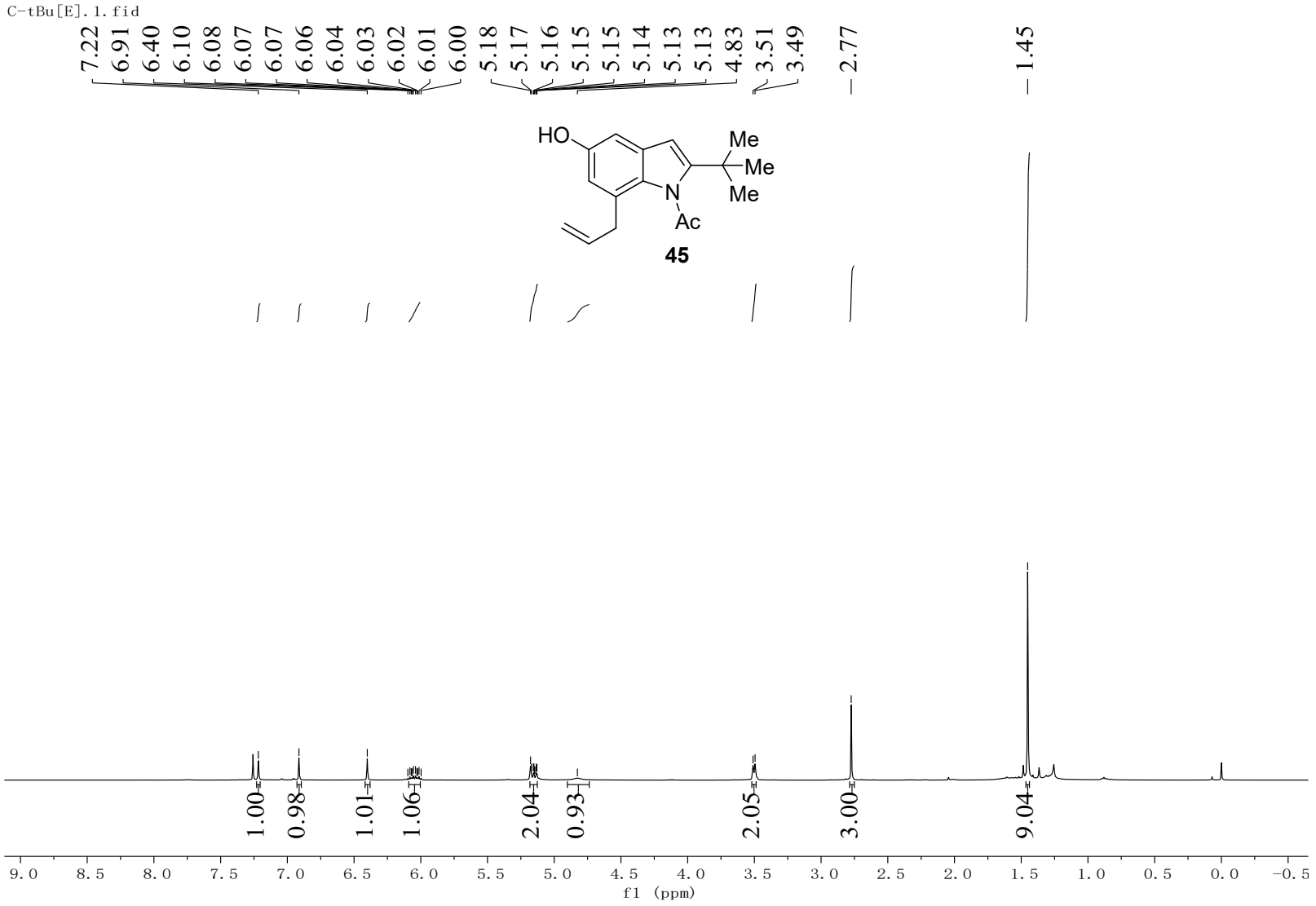


C-C12[E].1.fid

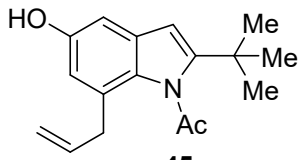

7.68
6.89
6.29
6.09
6.07
6.07
6.06
6.05
6.05
6.04
6.03
6.02
6.01
5.19
5.19
5.18
5.18
5.18
5.16
5.15
5.15
5.14
5.14
4.86
3.52
3.50
2.96
2.94
2.92
2.70
1.73
1.71
1.69
1.67
1.65
1.32
1.30
1.29
1.27
0.90
0.88
0.86
HO
(CH2)9Me
N
Ac
44
1.00
1.00
1.00
1.08
2.06
0.85
2.00
2.00
3.00
2.02
14.08
3.02
9.0
8.5
8.0
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)


C-C12[E]-C.1.fid
169.99
150.48
143.06
136.78
131.61
129.70
121.84
116.68
116.45
107.87
105.92
36.04
31.91
30.65
29.71
29.61
29.51
29.50
29.33
28.94
27.47
22.69
14.12
HO
(CH2)9Me
N
Ac
44
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

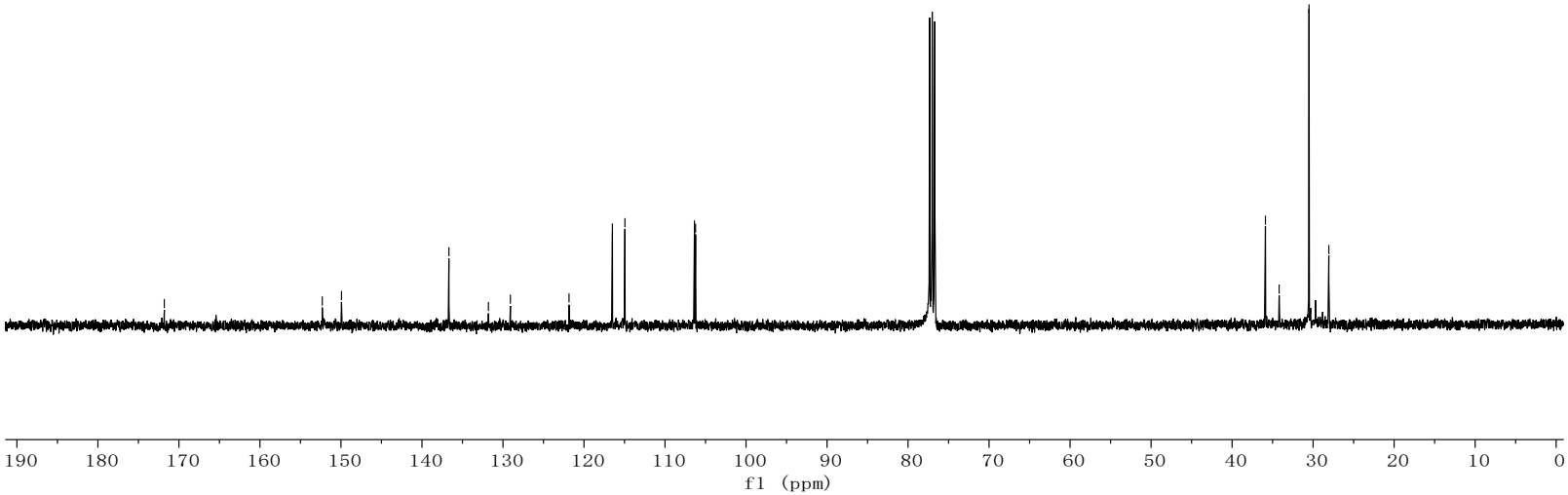

C-tBu[E].1.fid
7.22
6.91
6.40
6.10
6.08
6.07
6.07
6.06
6.04
6.03
6.02
6.01
6.00
5.18
5.17
5.16
5.15
5.15
5.14
5.13
5.13
4.83
3.51
3.49
2.77
1.45
HO
Me
Me
Me
N
Ac
45
1.00
0.98
1.01
1.06
2.04
0.93
2.05
3.00
9.04
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

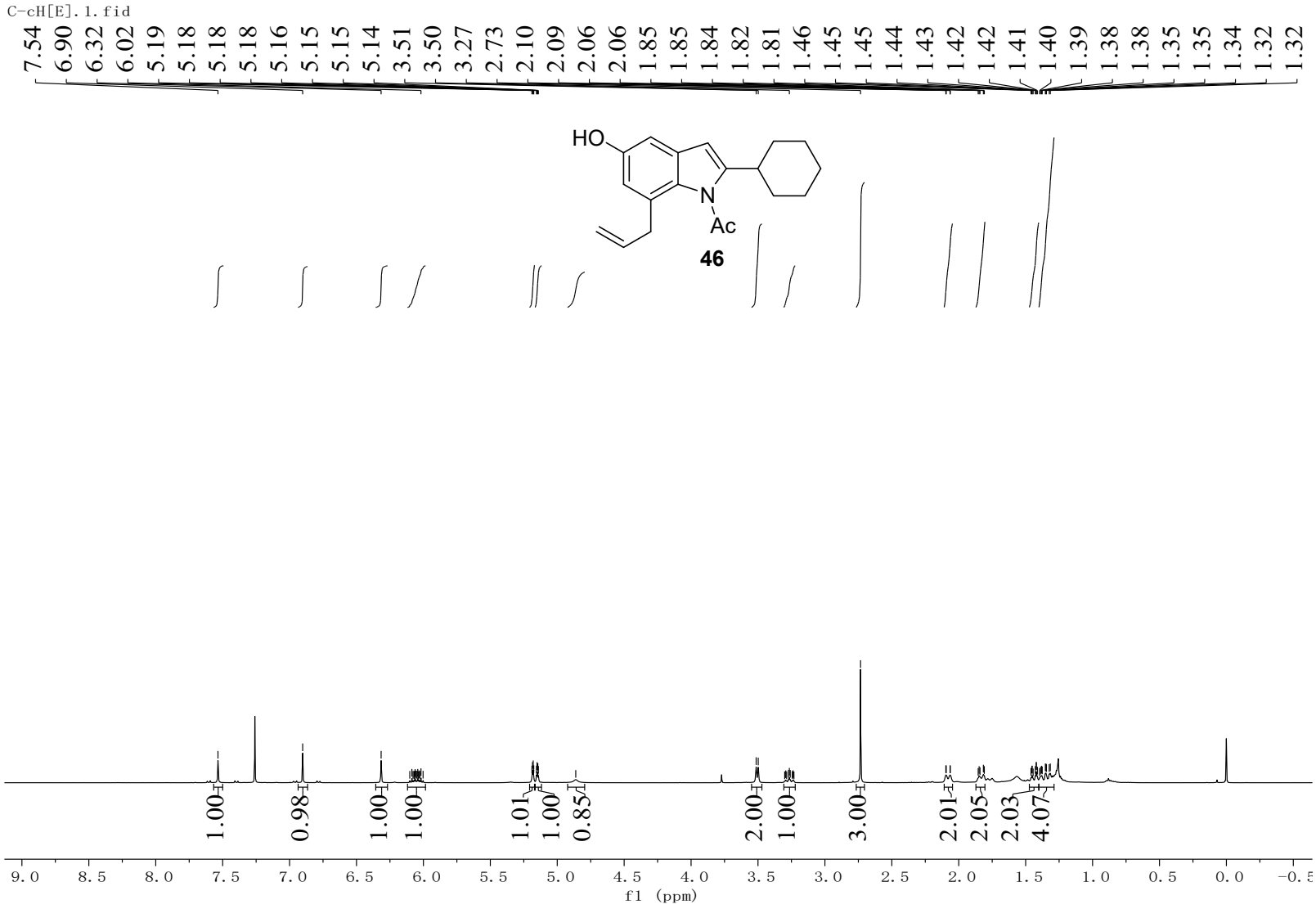
C-tBu[E]-C.1.fid

171.81
152.31
149.93
136.67
131.80
129.08
121.85
116.49
114.95
106.37
106.23
35.88
34.19
30.50
28.06



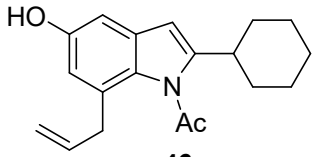
45




C-cH[E].1.fid
7.54 6.90 6.32 6.02 5.19 5.18 5.18 5.18 5.16 5.15 5.15 5.14 3.51 3.50 3.27 2.73 2.10 2.09 2.06 2.06 1.85 1.85 1.84 1.82 1.81 1.46 1.45 1.45 1.44 1.43 1.42 1.42 1.41 1.40 1.39 1.38 1.38 1.35 1.35 1.34 1.32 1.32
HO
N
Ac
46
1.00 0.98 1.00 1.00 1.01 1.00 0.85 2.00 1.00 3.00 2.01 2.05 2.03 4.07
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)

C-cH[E]-C.1.fid

170.21
150.36
149.17
136.75
131.40
129.90
121.78
116.46
116.25
106.15
105.62
38.01
36.00
33.90
27.69
26.64
26.36



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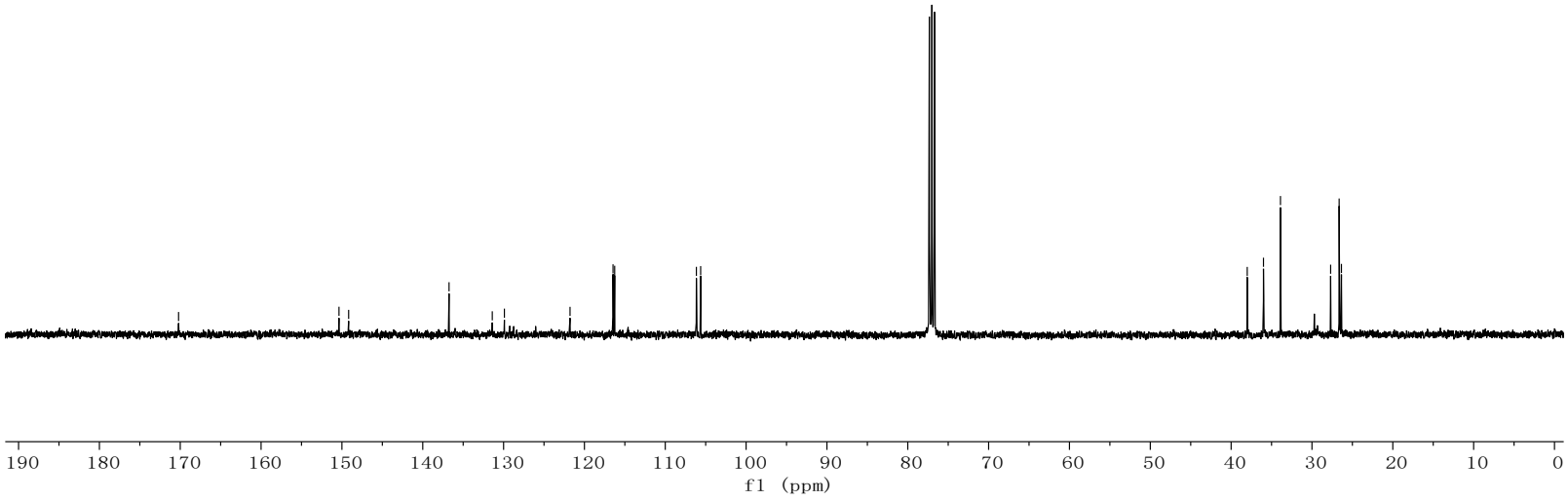
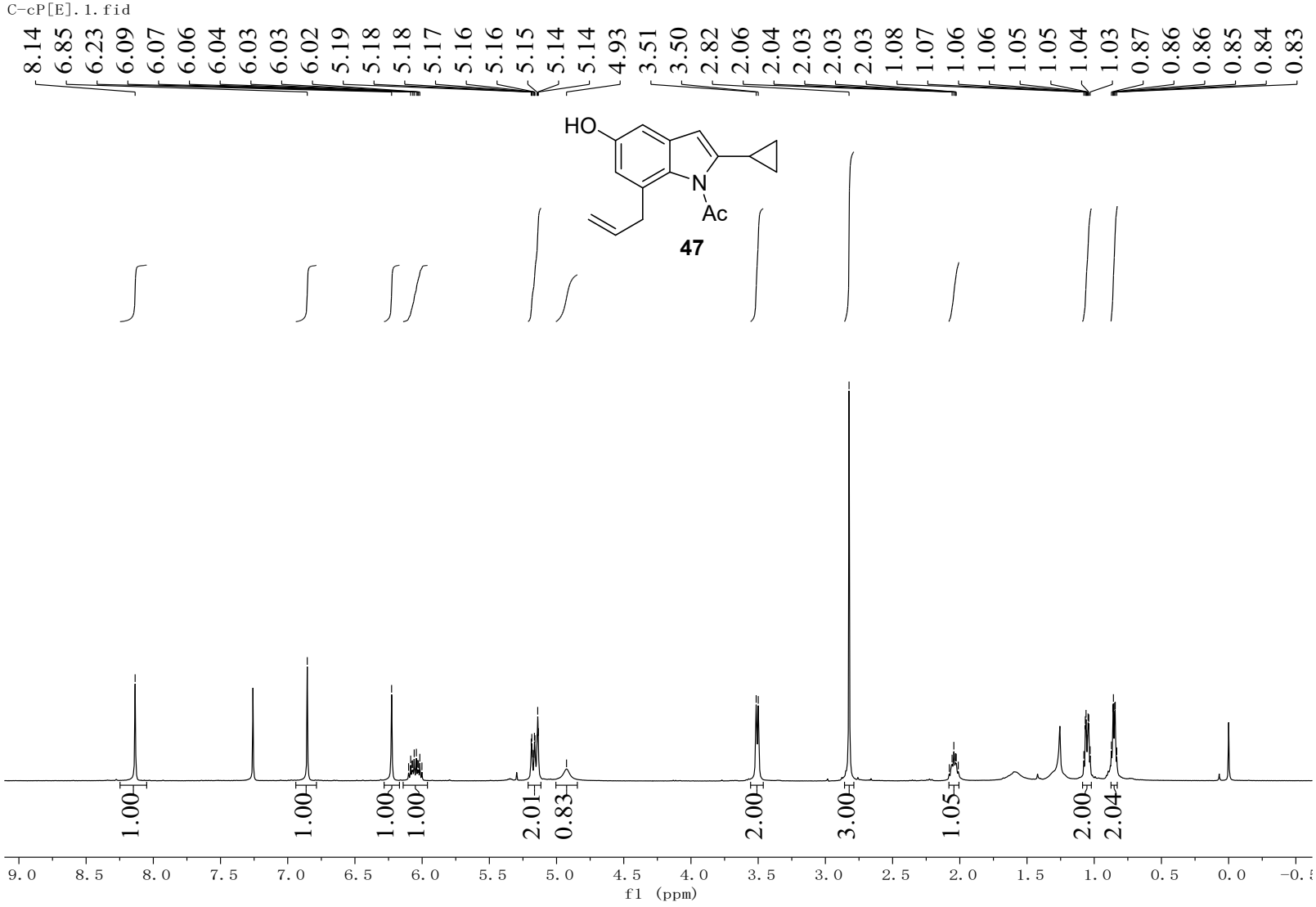
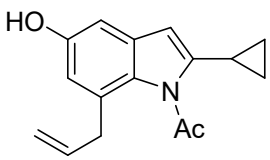



C-cP[E].1.fid
8.14 6.85 6.23 6.09 6.07 6.06 6.04 6.03 6.03 6.02 5.19 5.18 5.18 5.17 5.16 5.16 5.15 5.14 5.14 4.93 3.51 3.50 2.82 2.06 2.04 2.03 2.03 2.03 1.08 1.07 1.06 1.06 1.05 1.05 1.04 1.03 0.87 0.86 0.86 0.85 0.84 0.83
HO
N
Ac
47
1.00 1.00 1.00 1.00 2.01 0.83 2.00 3.00 1.05 2.00 2.04
9.0 8.5 8.0 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)

C-cP[E]-C.1.fid

170.39
150.84
142.42
136.87
132.45
128.77
122.73
118.11
116.35
108.46
105.71
36.13
27.03
11.79
8.54



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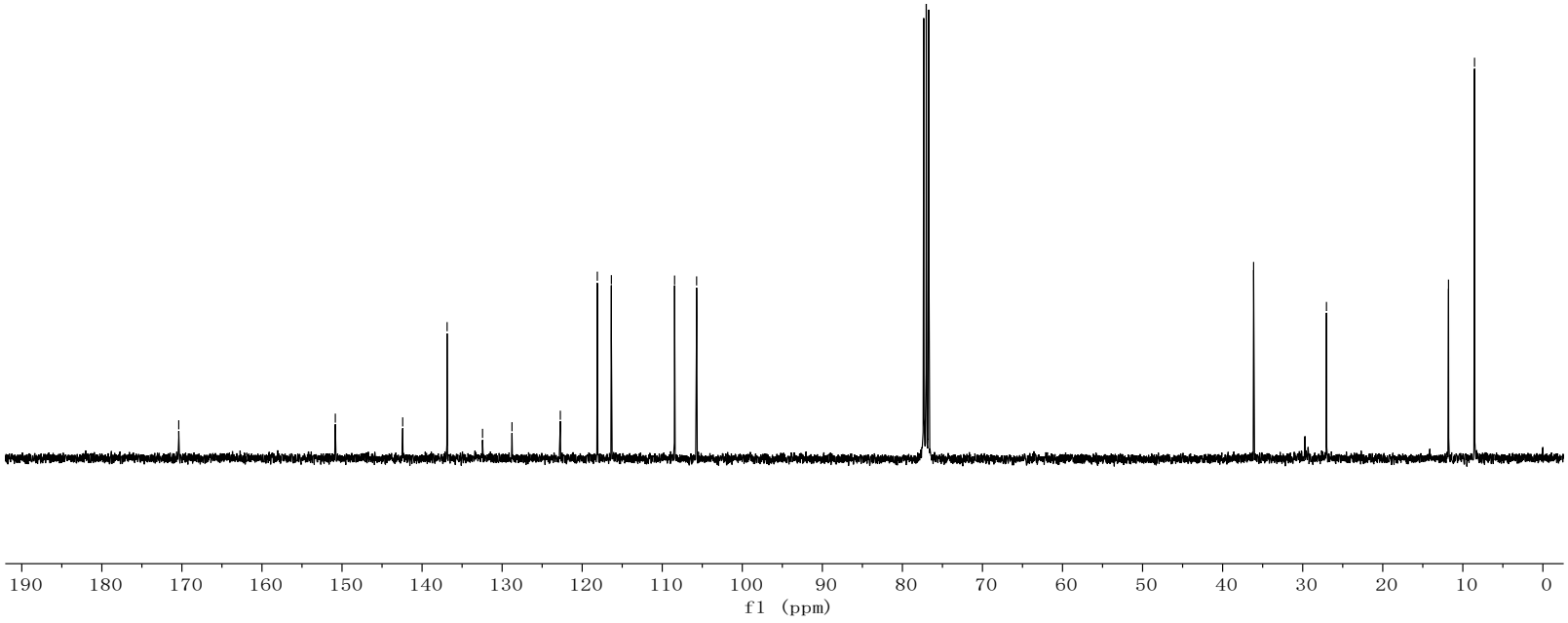
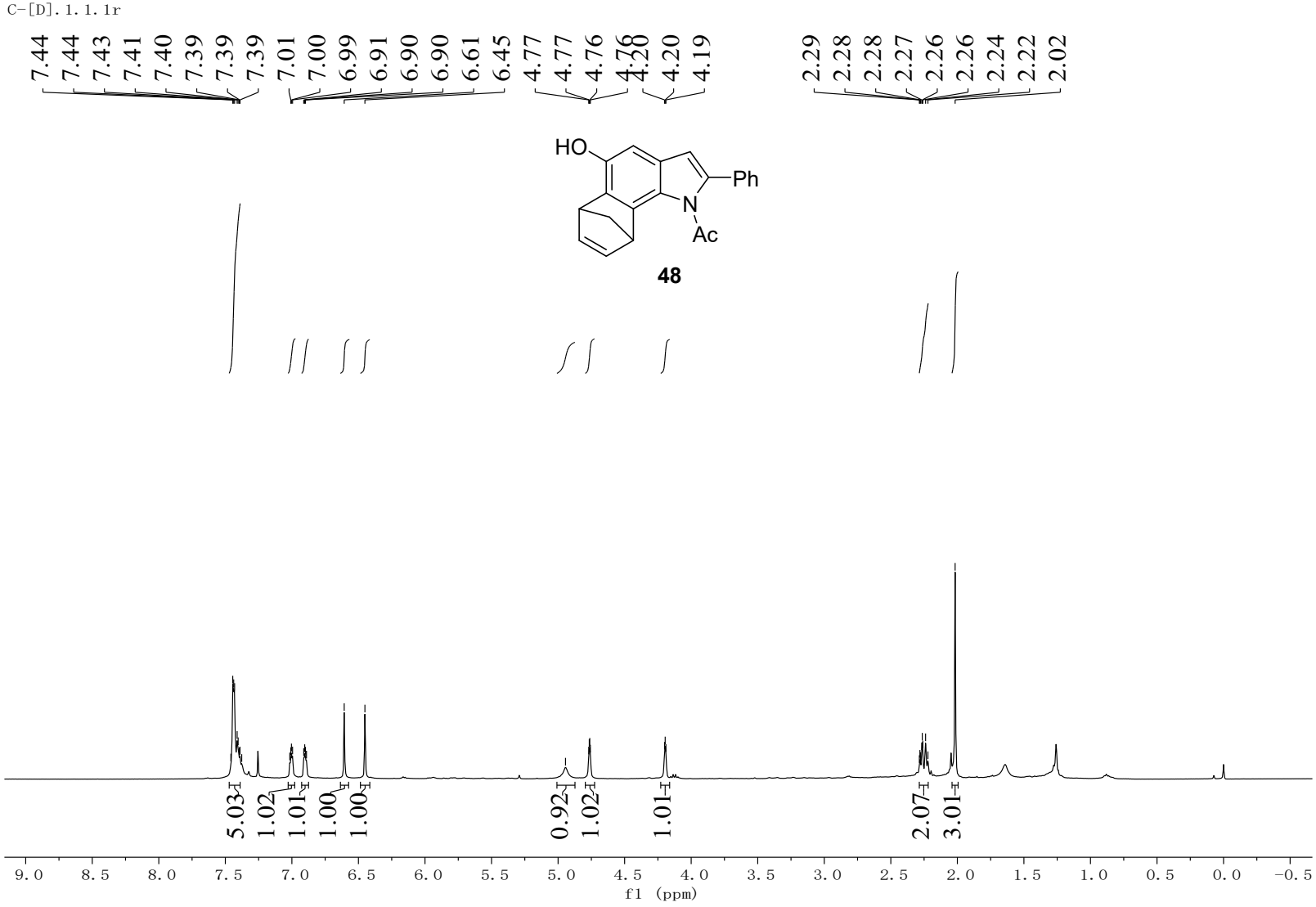
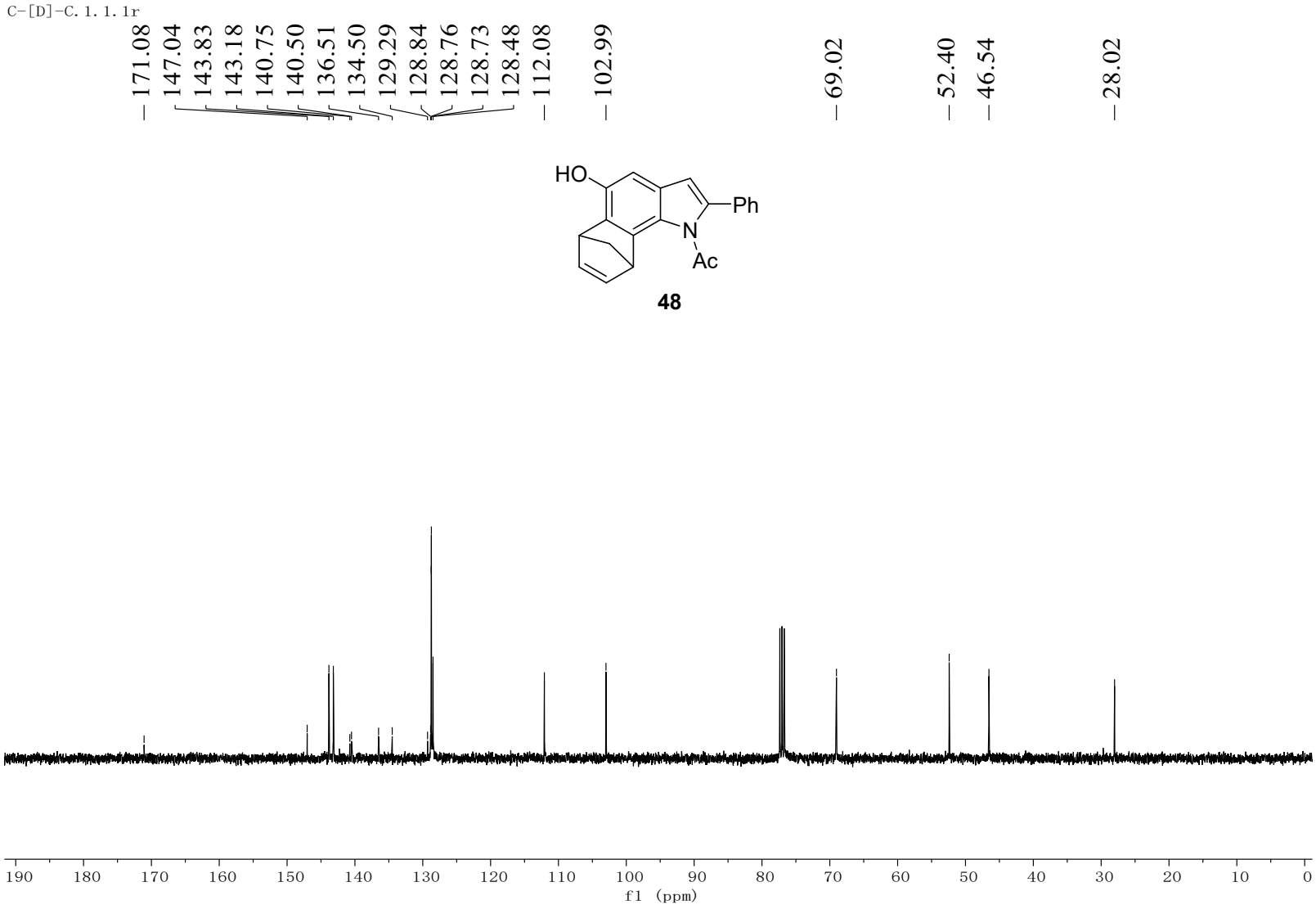
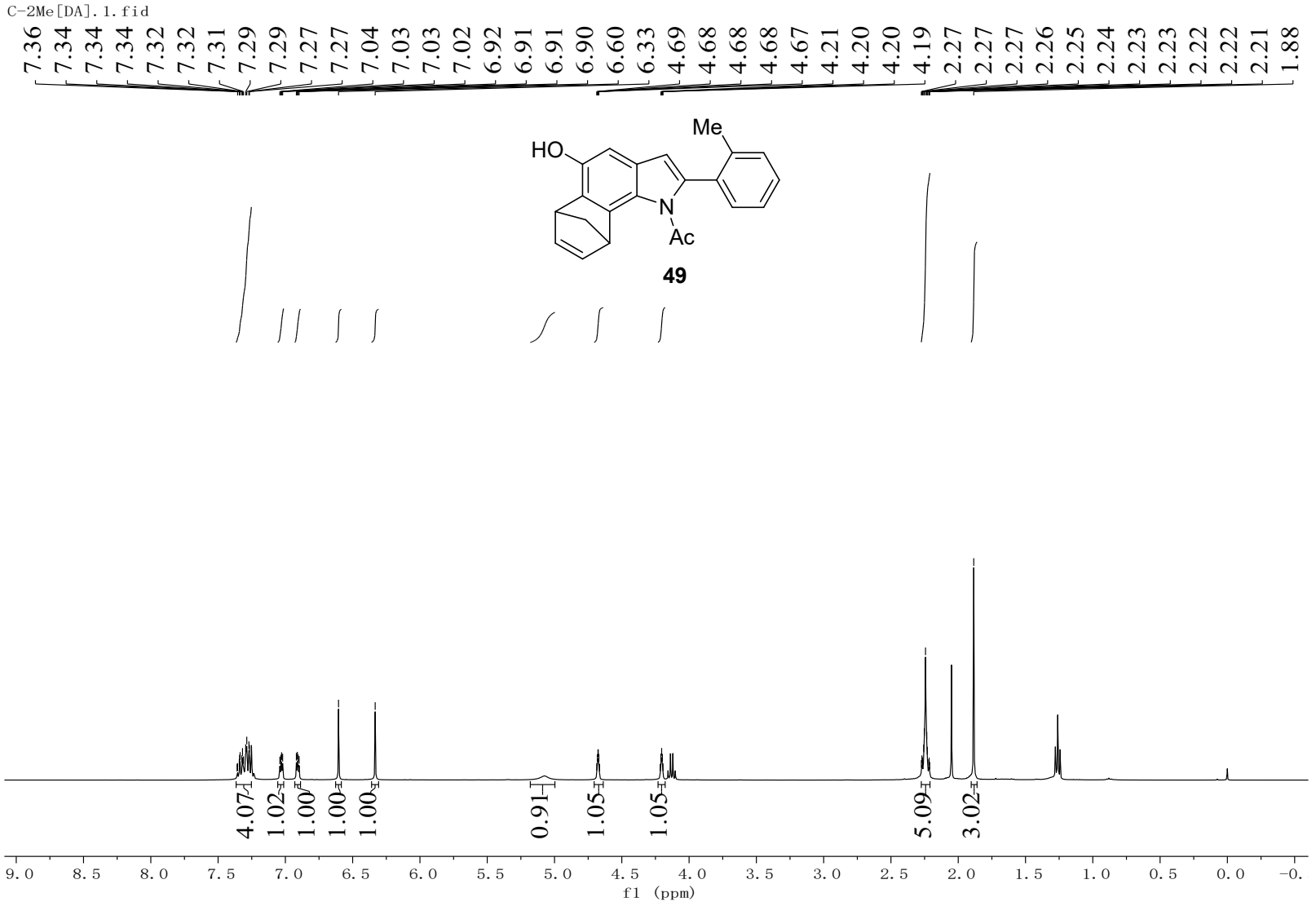
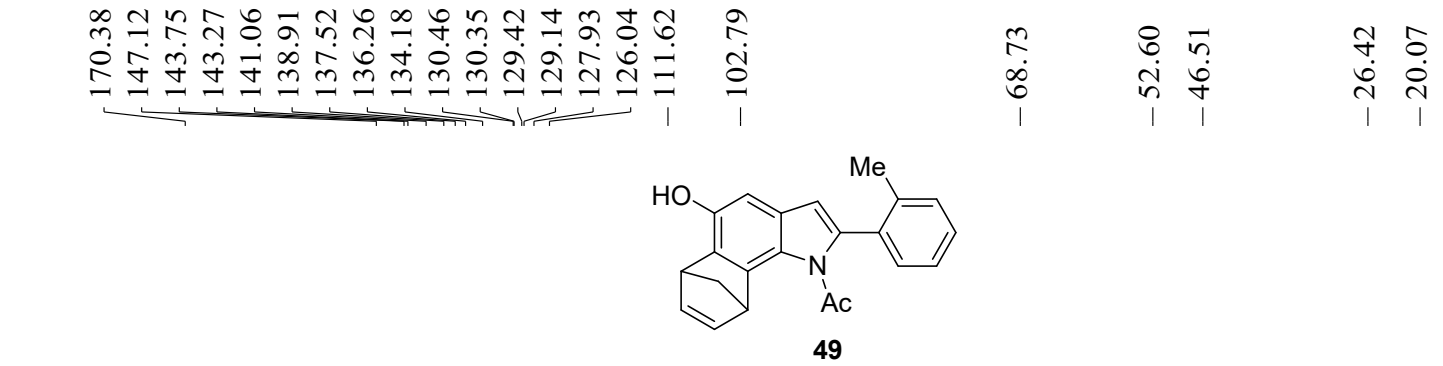



C-[D].1.1.1r
7.44
7.44
7.43
7.41
7.40
7.39
7.39
7.39
7.01
7.00
6.99
6.91
6.90
6.90
6.61
6.45
4.77
4.77
4.76
4.76
4.20
4.20
4.19
2.29
2.28
2.28
2.27
2.26
2.26
2.24
2.22
2.02
HO
Ph
N
Ac
48
5.03
1.02
1.01
1.00
1.00
0.92
1.02
1.01
2.07
3.01
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


C-[D]-C.1.1.1r
171.08
147.04
143.83
143.18
140.75
140.50
136.51
134.50
129.29
128.84
128.76
128.73
128.48
112.08
102.99
69.02
52.40
46.54
28.02
HO
Ph
N
Ac
48
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


C-2Me[DA].1.fid
7.36 7.34 7.34 7.34 7.32 7.32 7.31 7.29 7.29 7.27 7.27 7.04 7.03 7.03 7.02 6.92 6.91 6.91 6.90 6.60 6.33 4.69 4.68 4.68 4.68 4.67 4.21 4.20 4.20 4.19 2.27 2.27 2.27 2.26 2.25 2.24 2.23 2.23 2.22 2.22 2.21 1.88
HO
Me
N
Ac
49
4.07 1.02 1.00 1.00 1.00 0.91 1.05 1.05 5.09 3.02
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 -0.
f1 (ppm)

C-2Me[DA]-C.1.fid



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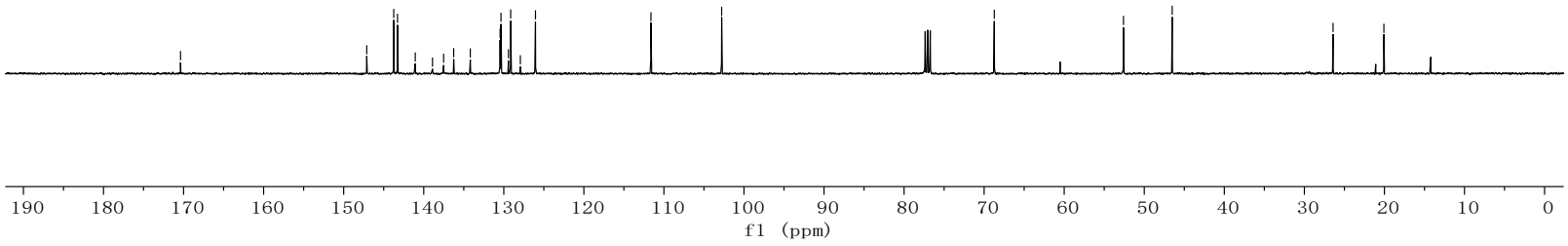
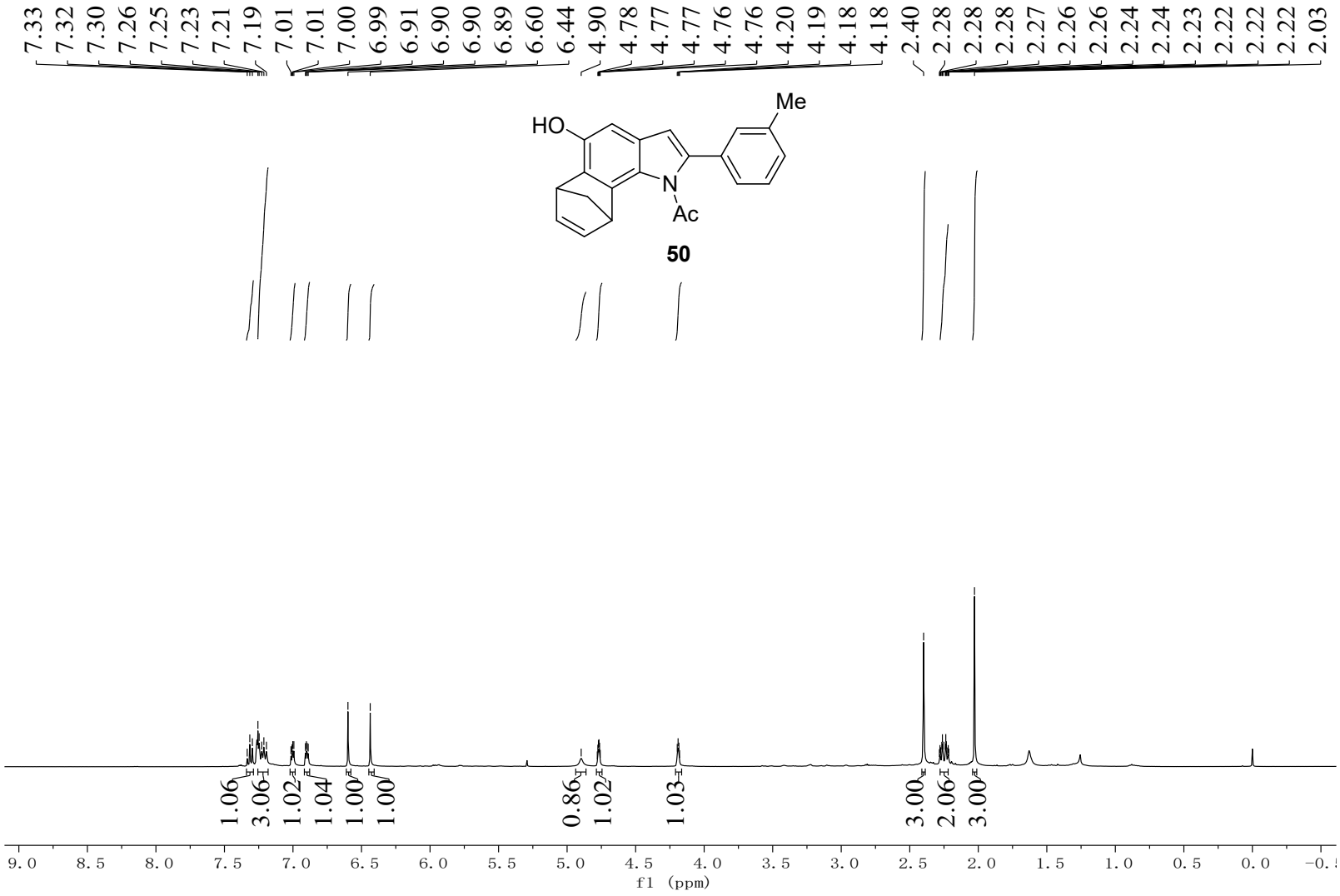
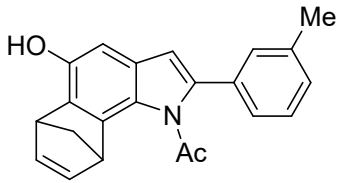


C-3Me[D].1.1.1r

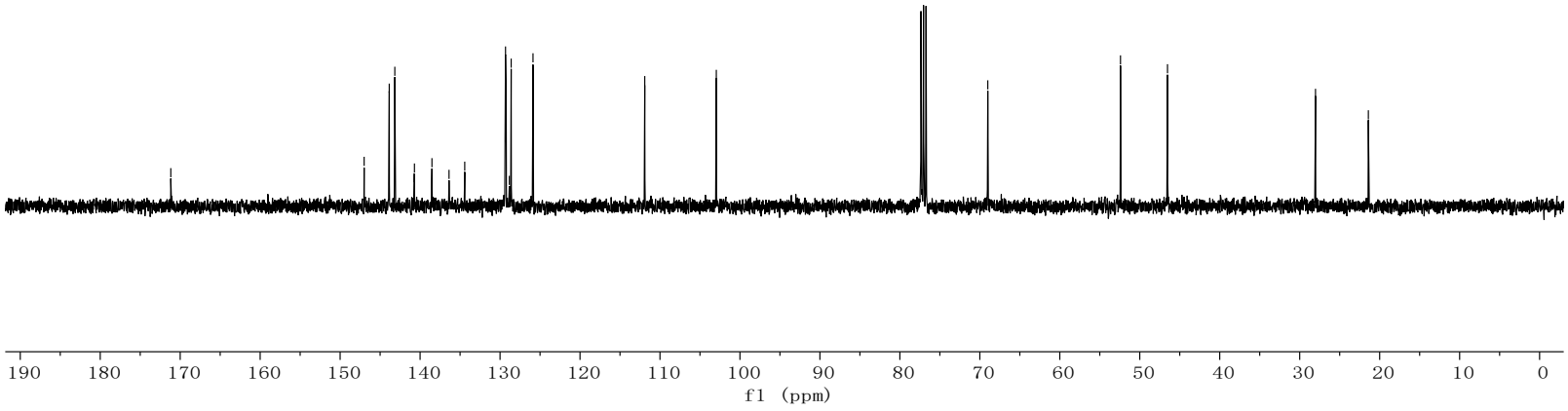

7.33
7.32
7.30
7.26
7.25
7.23
7.21
7.19
7.01
7.01
7.00
6.99
6.91
6.90
6.90
6.89
6.60
6.44
4.90
4.78
4.77
4.77
4.76
4.76
4.20
4.19
4.18
4.18
2.40
2.28
2.28
2.28
2.27
2.26
2.26
2.24
2.24
2.23
2.22
2.22
2.22
2.03
HO
Me
N
Ac
50
1.06
3.06
1.02
1.04
1.00
1.00
0.86
1.02
1.03
3.00
2.06
3.00
9.0
8.5
8.0
7.5
7.0
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)

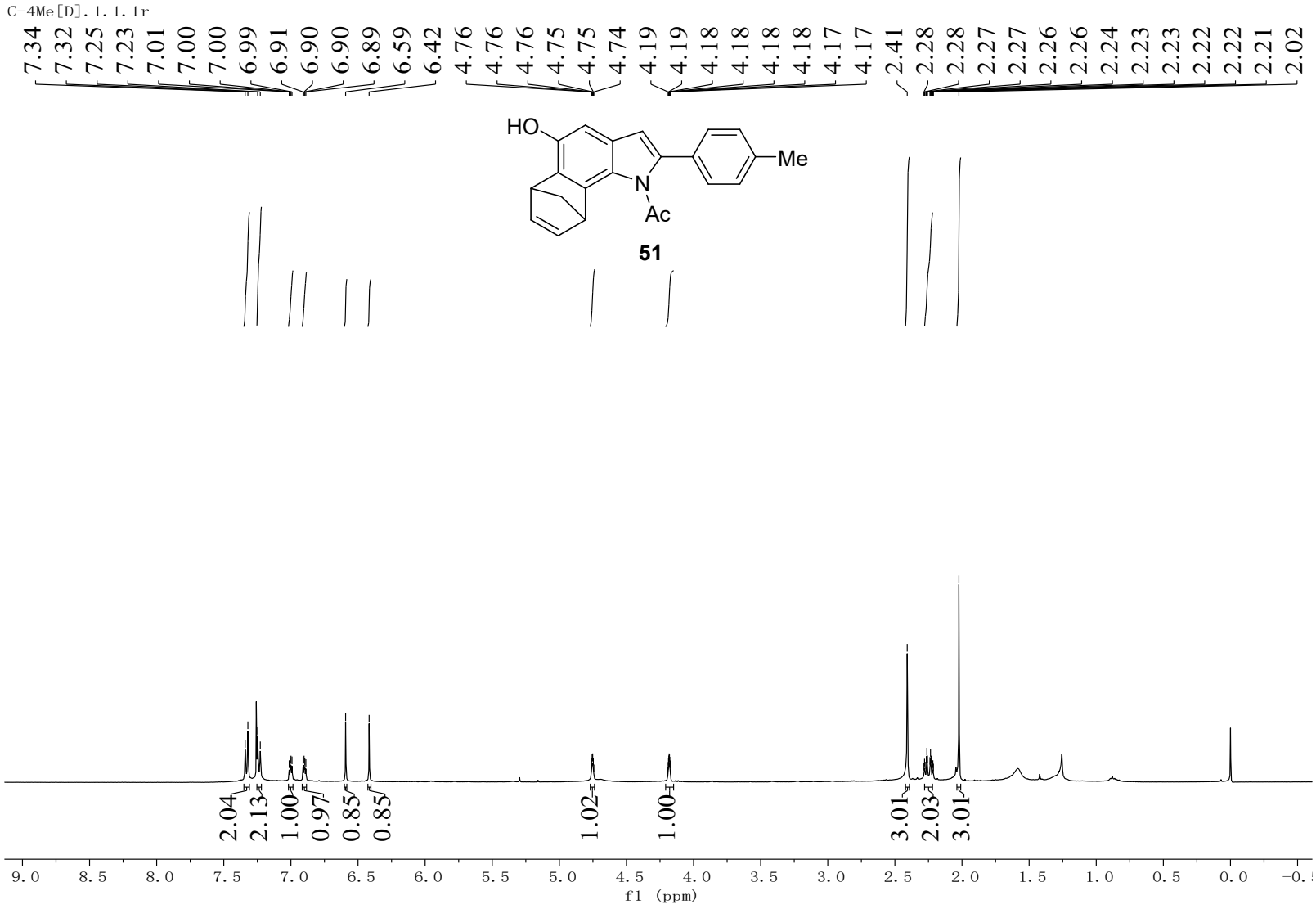
C-3Me[D]-C.1.1.1r

171.17
147.00
143.86
143.16
140.75
140.71
138.52
136.38
134.42
129.34
129.32
129.24
128.84
128.61
125.88
111.90
102.96
69.01
52.41
46.53
28.02
21.41



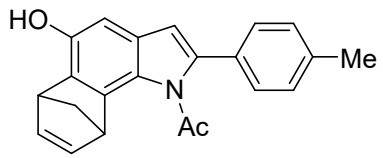
50



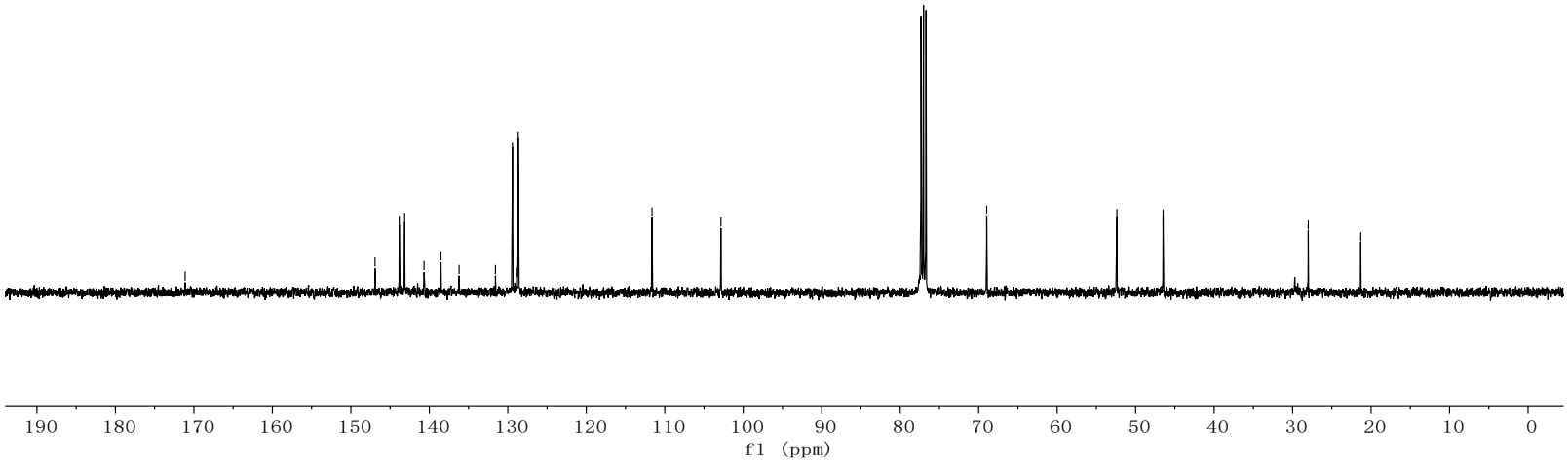

C-4Me[D].1.1.1r
7.34
7.32
7.25
7.23
7.01
7.00
7.00
6.99
6.91
6.90
6.90
6.89
6.59
6.42
4.76
4.76
4.76
4.75
4.75
4.74
4.19
4.19
4.18
4.18
4.18
4.18
4.17
4.17
2.41
2.28
2.28
2.27
2.27
2.26
2.26
2.24
2.23
2.23
2.22
2.22
2.21
2.02
HO
Me
N
Ac
51
2.04
2.13
1.00
0.97
0.85
0.85
1.02
1.00
3.01
2.03
3.01
9.0
8.5
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)

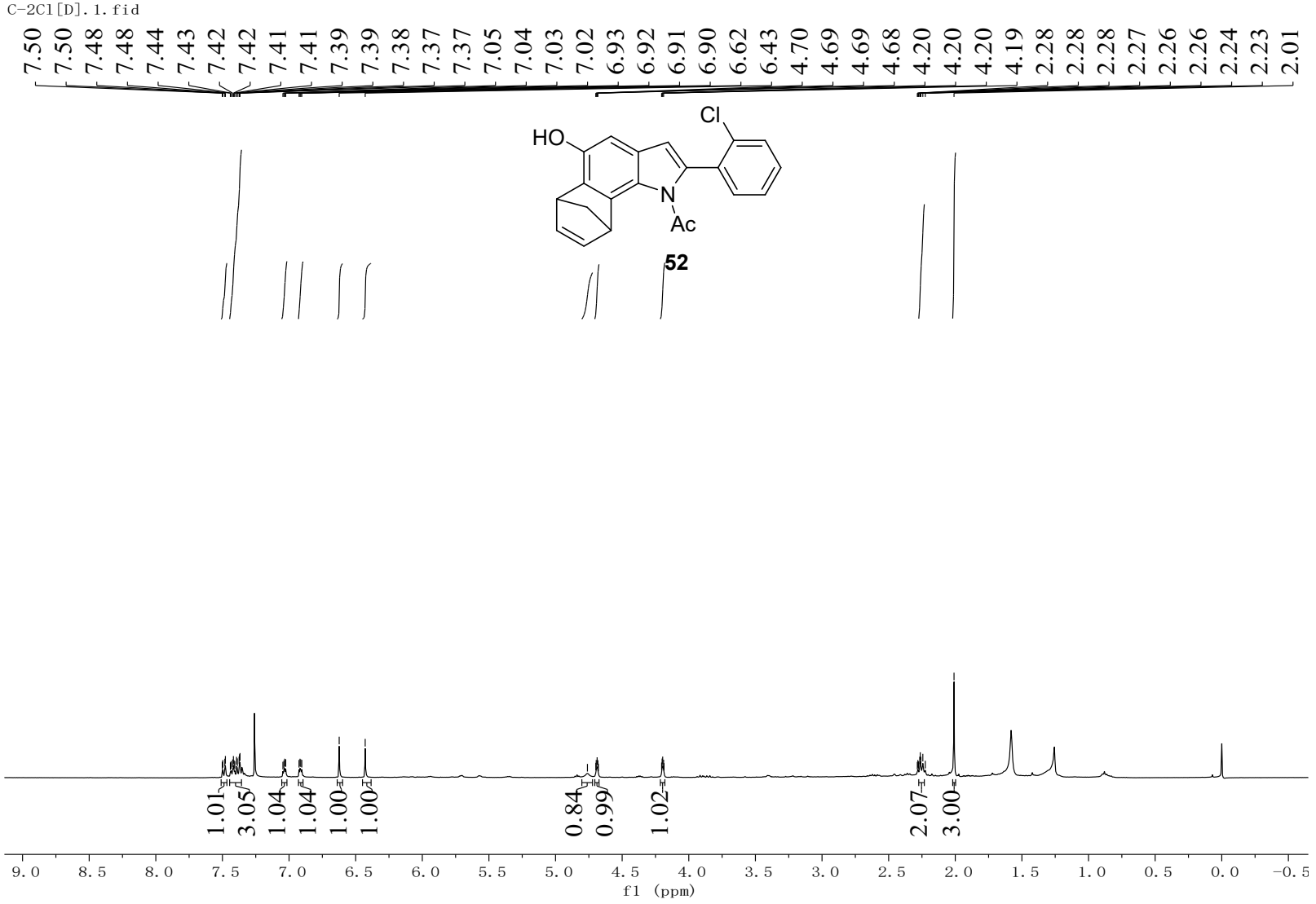
C-4Me[D]-C.1.1.1r

171.12
146.92
143.83
143.14
140.67
138.52
136.21
131.57
129.41
129.37
128.79
128.67
111.62
102.84
68.98
52.38
46.50
28.01
21.32



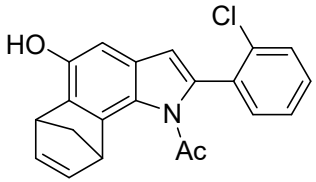
51



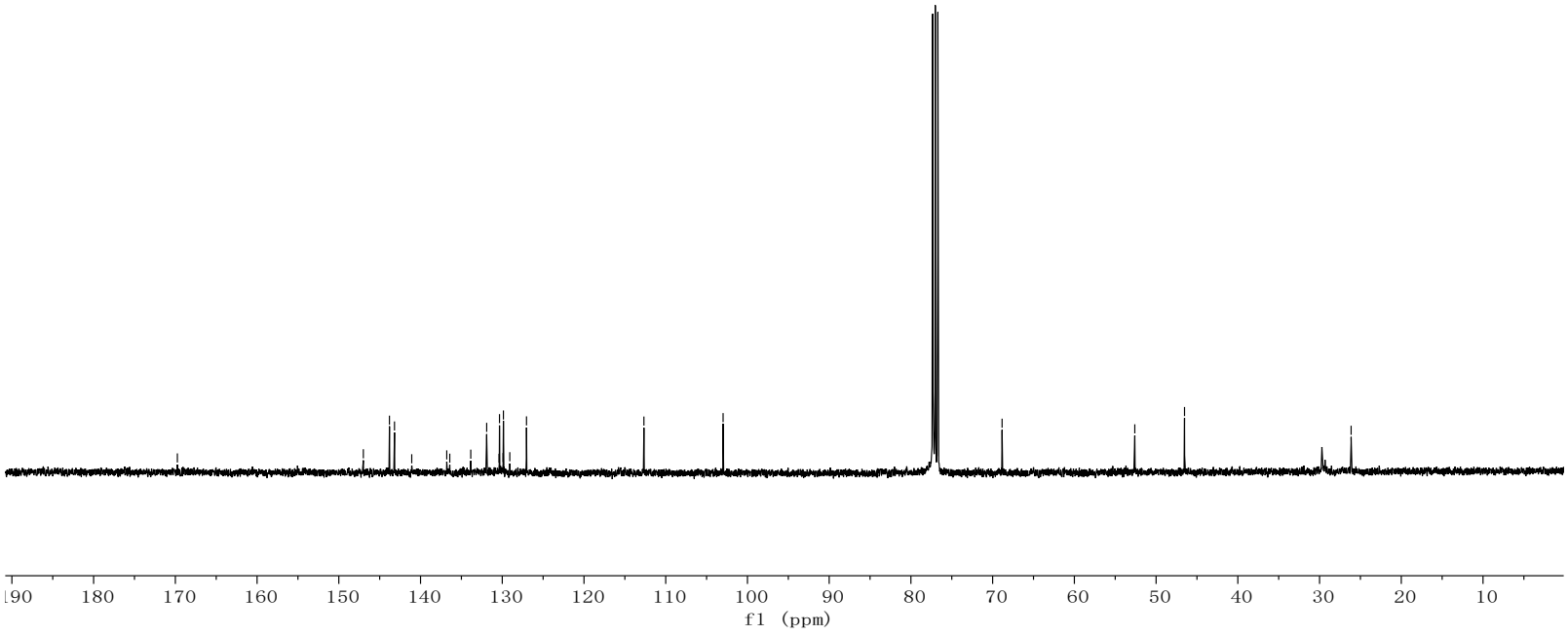

C-2Cl[D].1.fid
7.50 7.50 7.48 7.48 7.44 7.43 7.42 7.42 7.41 7.41 7.39 7.39 7.38 7.37 7.37 7.05 7.04 7.03 7.02 6.93 6.92 6.91 6.90 6.62 6.43 4.70 4.69 4.69 4.68 4.20 4.20 4.20 4.19 2.28 2.28 2.28 2.27 2.26 2.26 2.24 2.23 2.01
HO
Cl
N
Ac
52
1.01 3.05 1.04 1.04 1.00 1.00 0.84 0.99 1.02 2.07 3.00
9.0 8.5 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 −0.5
f1 (ppm)

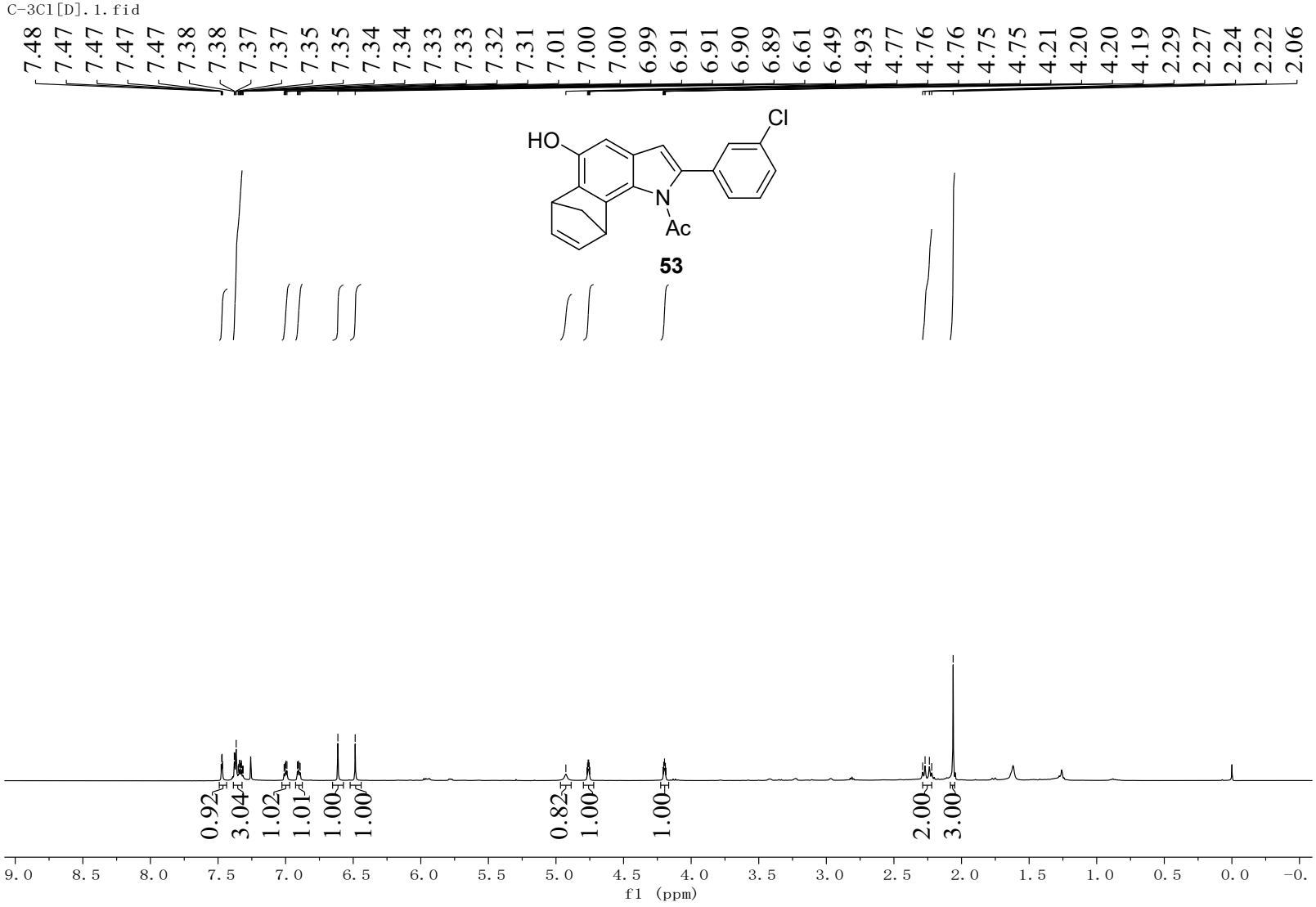
C-2Cl[D]-C-1.1.fid

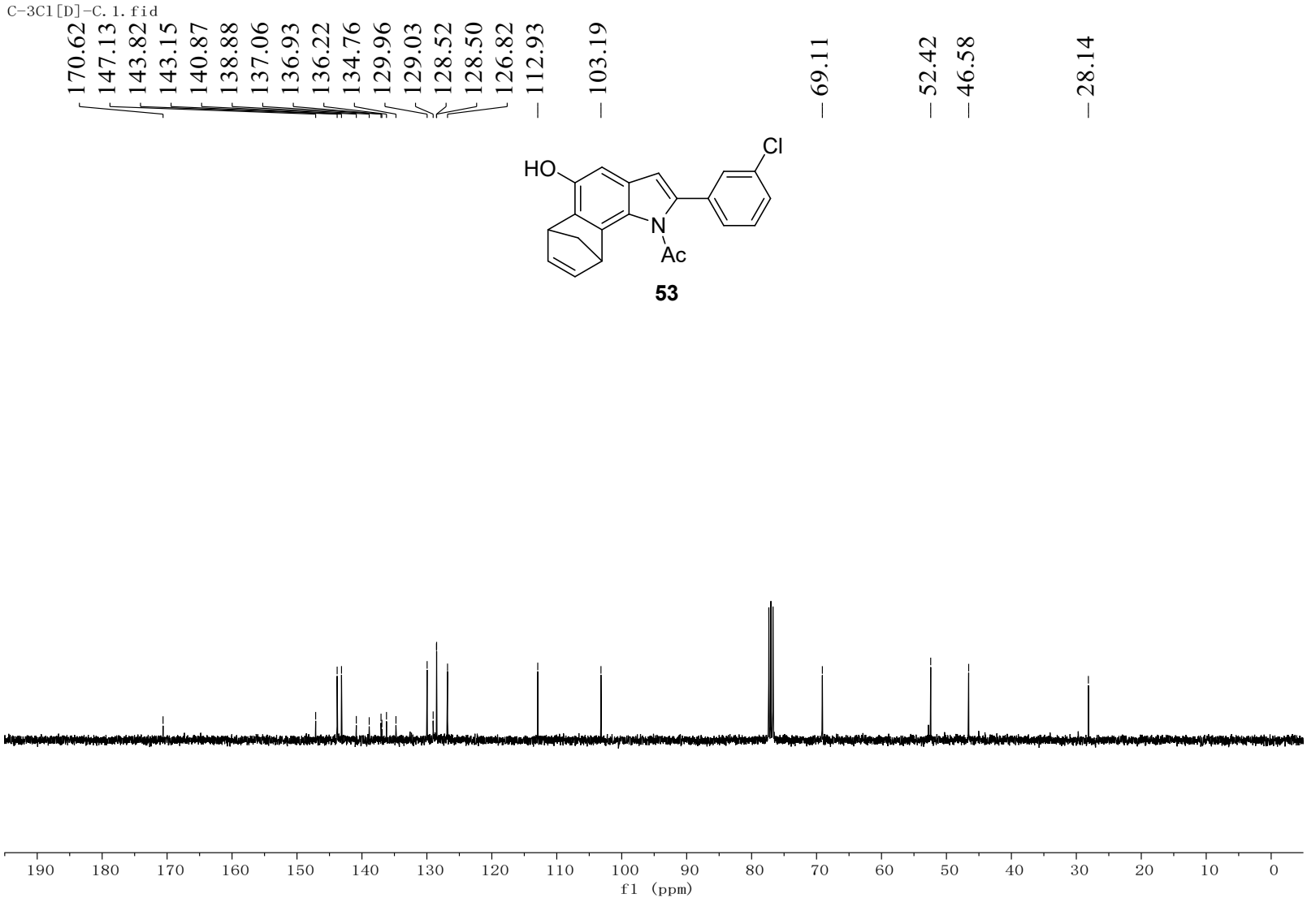
169.75
147.00
143.80
143.19
141.08
136.79
136.42
133.85
131.91
130.39
130.34
129.90
129.86
129.08
127.03
112.67
102.98
68.85
52.61
46.52
26.14

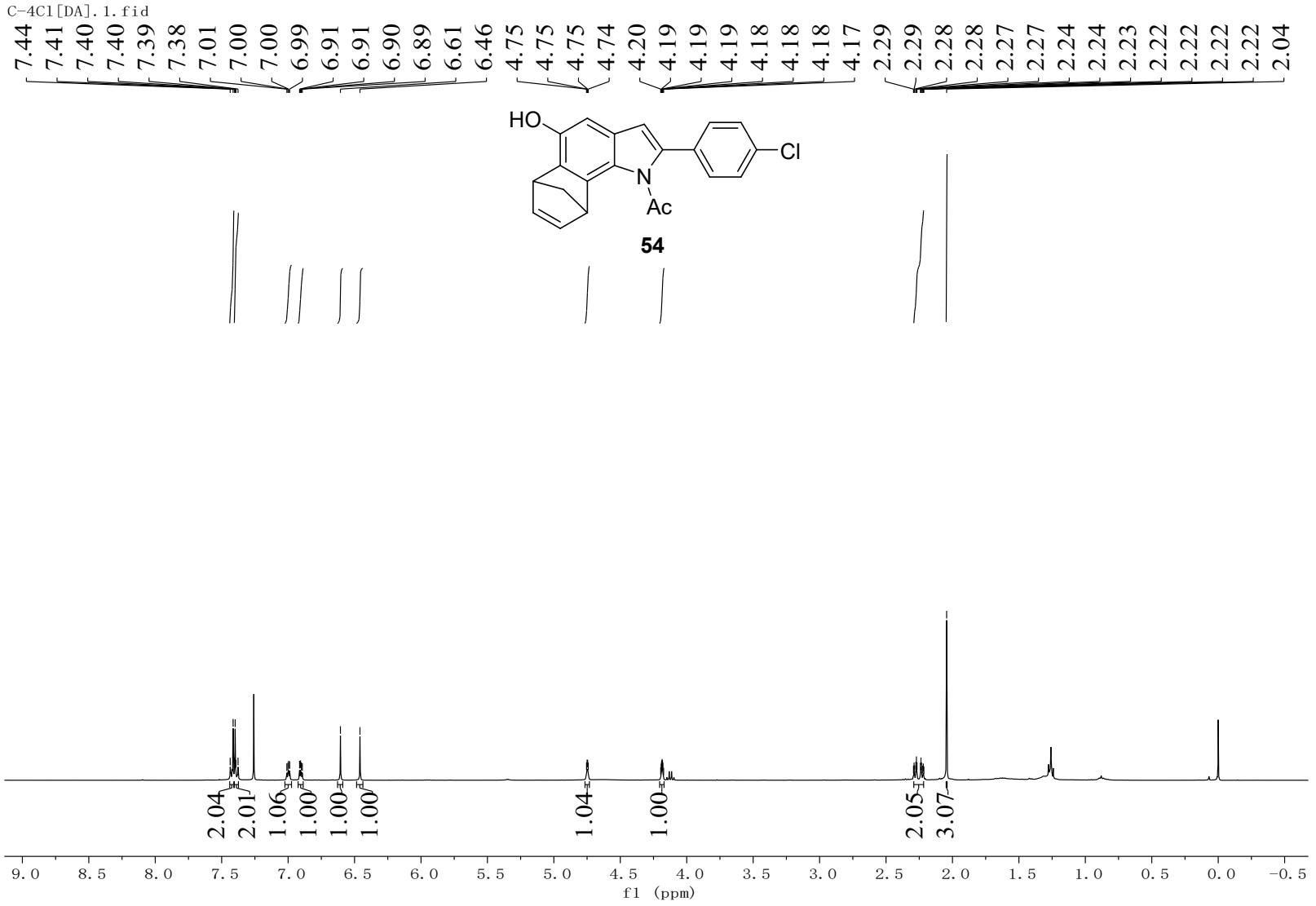


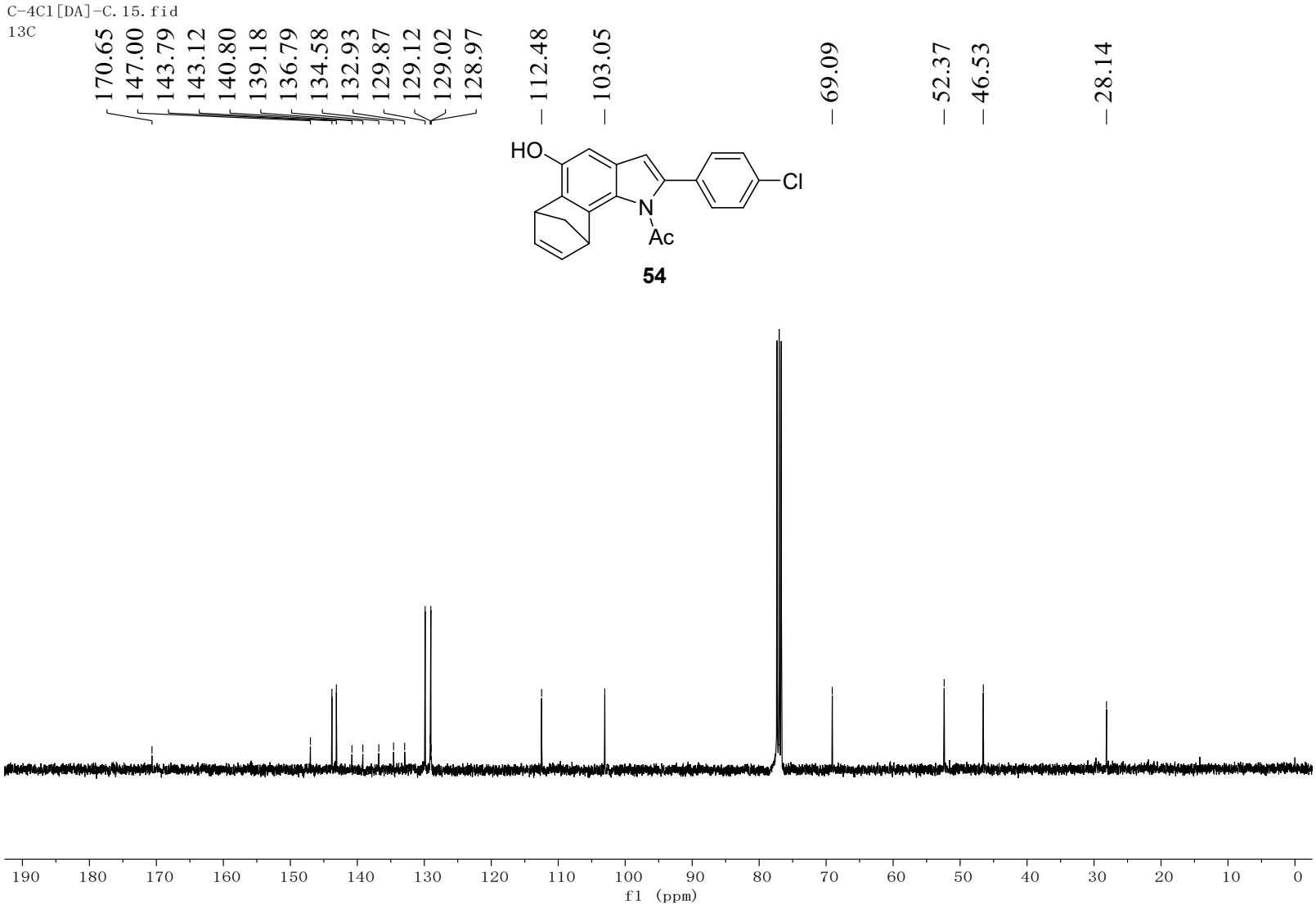
52

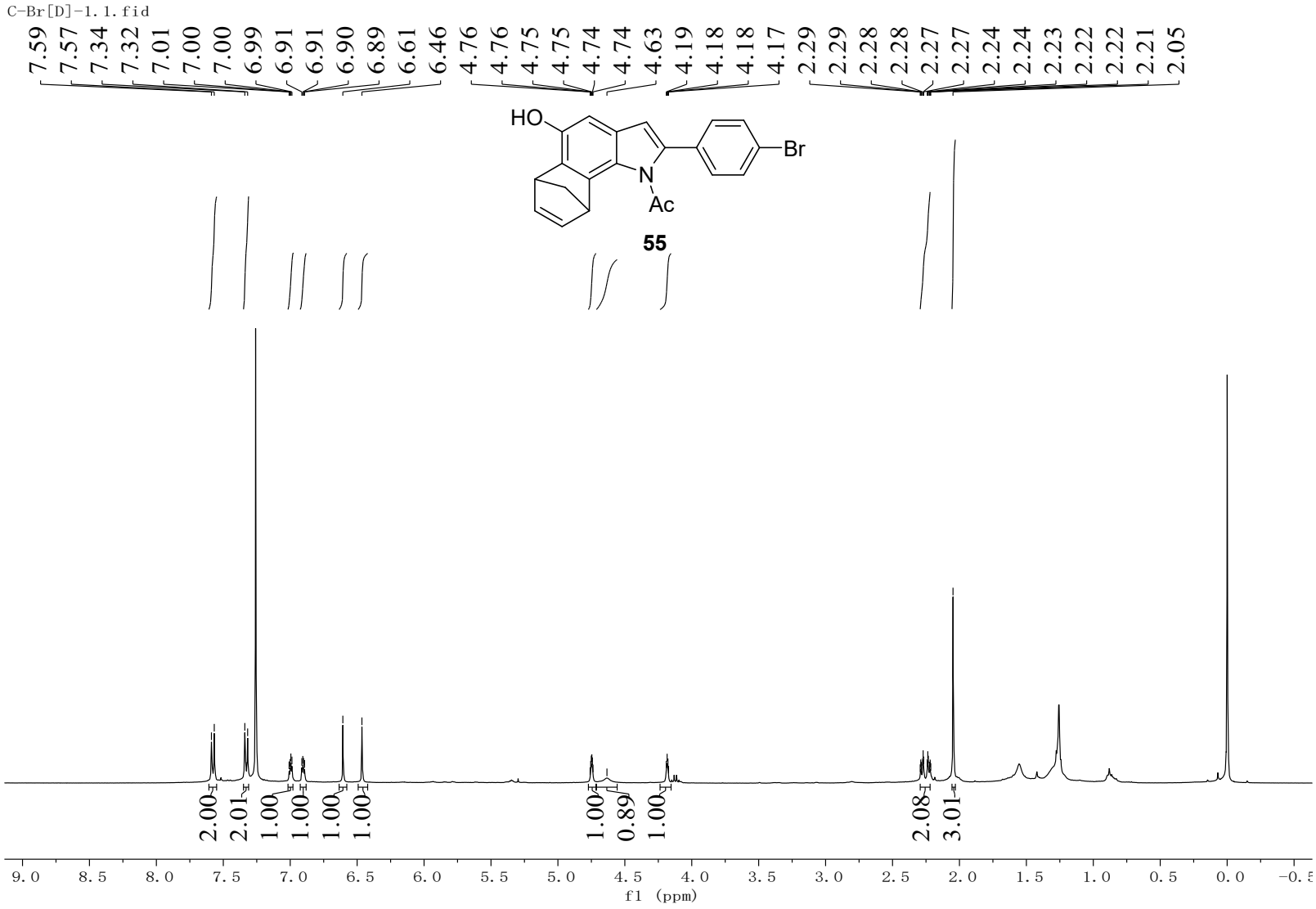


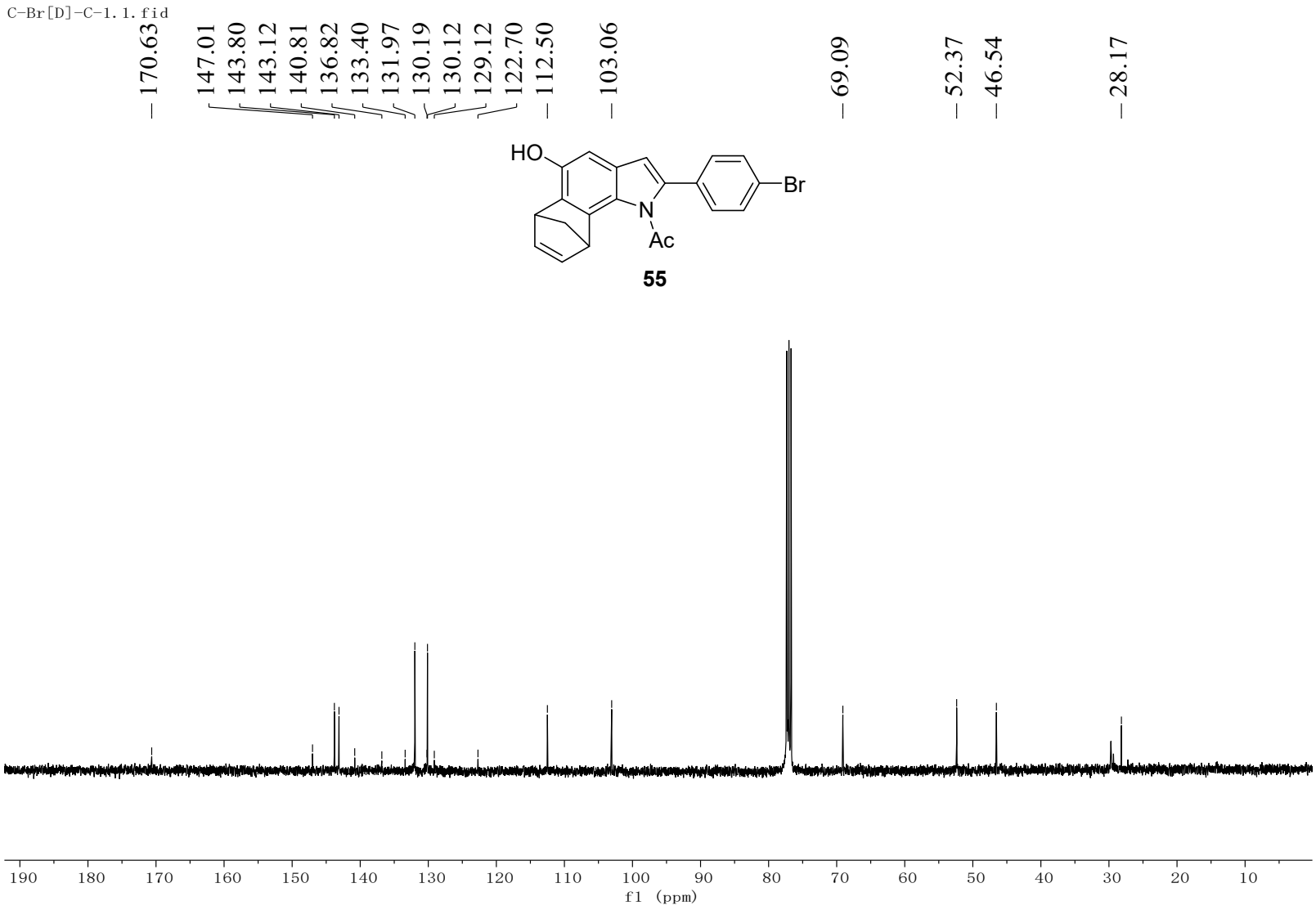

C-3Cl[D].1.fid
7.48 7.47 7.47 7.47 7.47 7.38 7.38 7.37 7.37 7.35 7.35 7.34 7.34 7.33 7.33 7.32 7.31 7.01 7.00 7.00 6.99 6.91 6.91 6.90 6.89 6.61 6.49 4.93 4.77 4.76 4.76 4.75 4.75 4.21 4.20 4.20 4.19 2.29 2.27 2.24 2.22 2.06
HO
Cl
N
Ac
53
0.92 3.04 1.02 1.01 1.00 1.00 0.82 1.00 1.00 2.00 3.00
9.0 8.5 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 -0.
f1 (ppm)

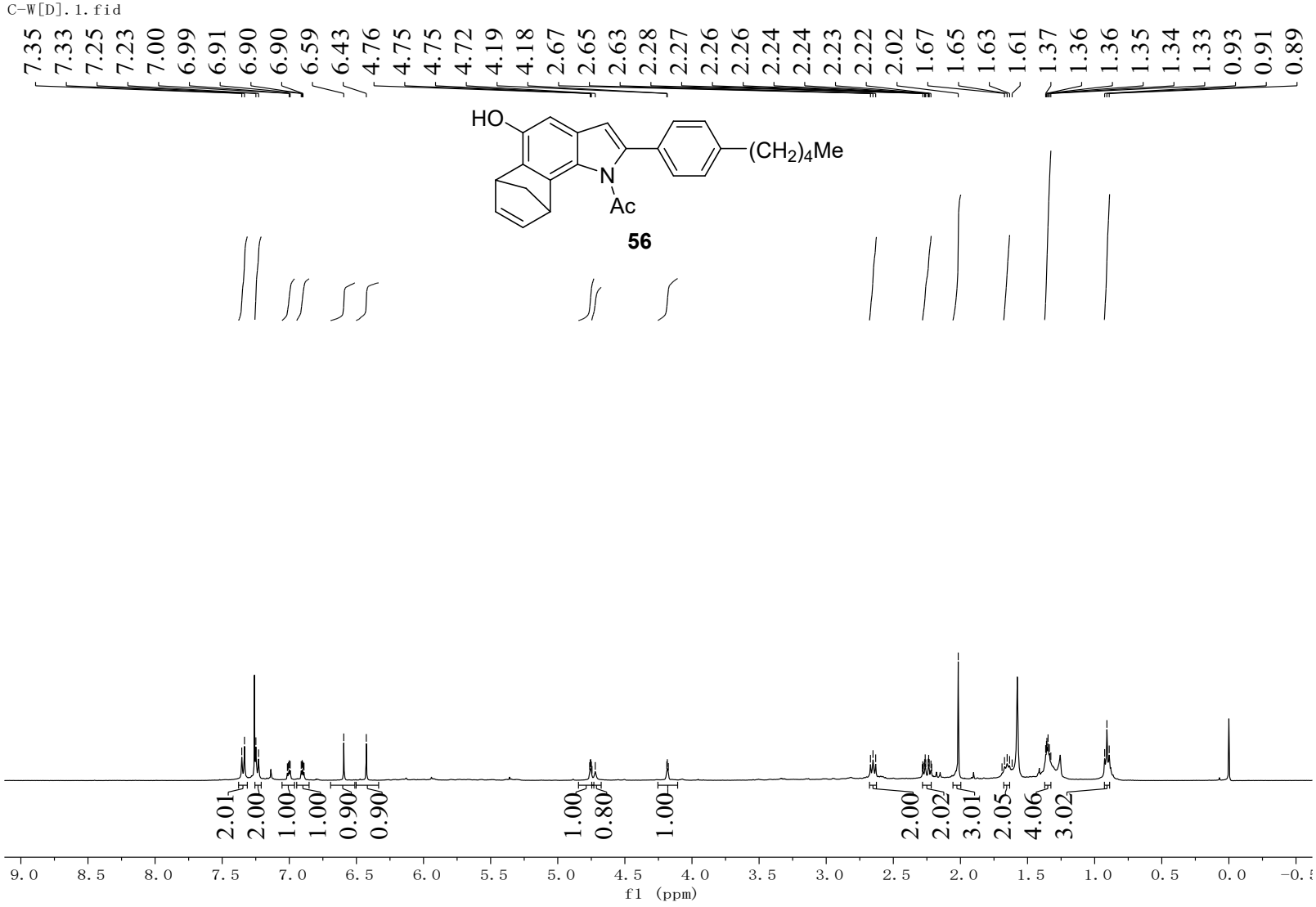

C-3Cl[D]-C.1.fid
170.62
147.13
143.82
143.15
140.87
138.88
137.06
136.93
136.22
134.76
129.96
129.03
128.52
128.50
126.82
112.93
103.19
69.11
52.42
46.58
28.14
HO
Cl
N
Ac
53
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

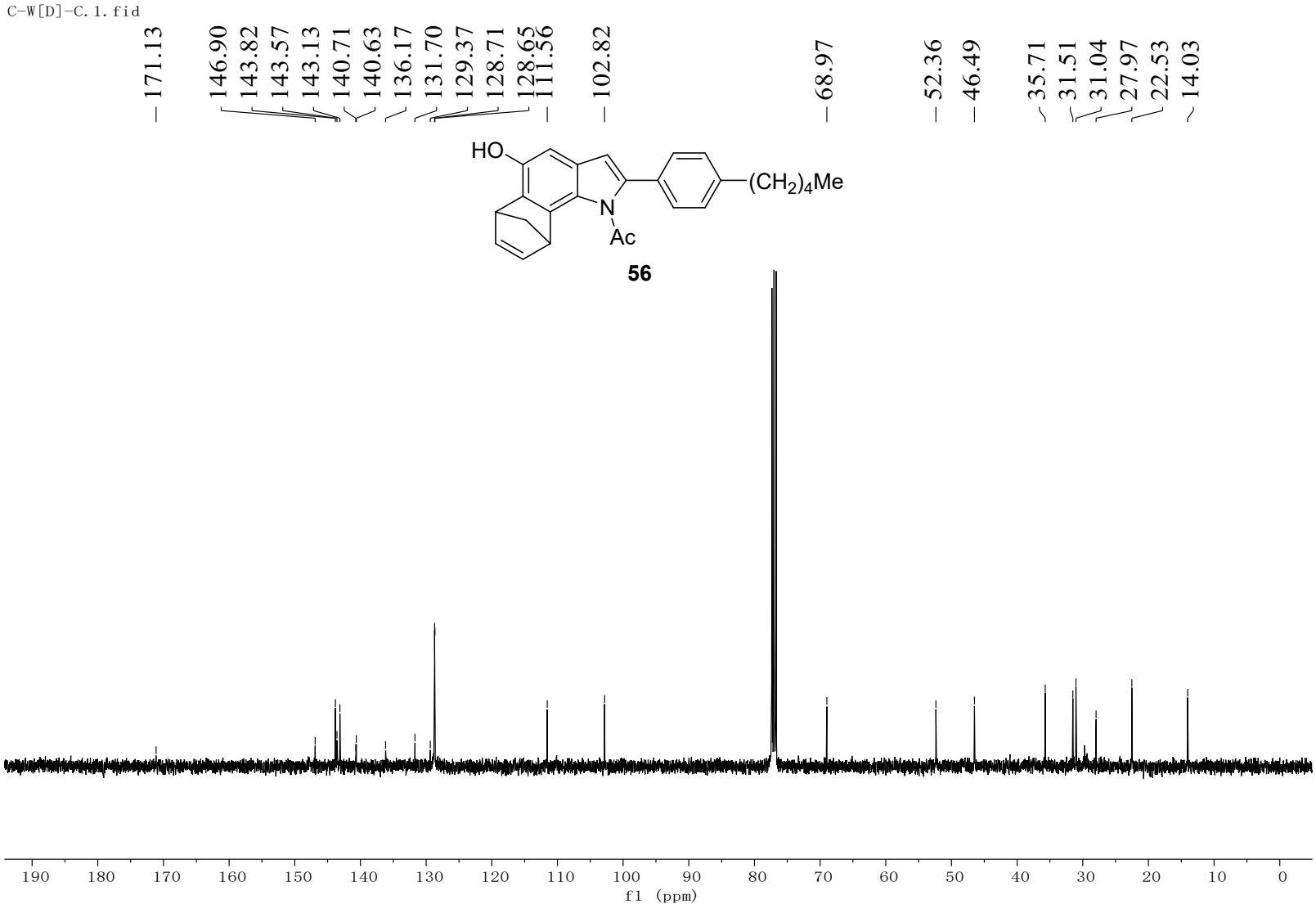

C-4Cl[DA].1.fid
7.44
7.41
7.40
7.40
7.39
7.38
7.01
7.00
7.00
6.99
6.91
6.91
6.90
6.89
6.61
6.46
4.75
4.75
4.75
4.74
4.20
4.19
4.19
4.19
4.18
4.18
4.18
4.17
2.29
2.29
2.28
2.28
2.27
2.27
2.24
2.24
2.23
2.22
2.22
2.22
2.22
2.04
HO
Cl
N
Ac
54
2.04
2.01
1.06
1.00
1.00
1.00
1.04
1.00
2.05
3.07
9.0
8.5
8.0
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
-0.5
f1 (ppm)


C-4Cl[DA]-C.15.fid
13C
170.65
147.00
143.79
143.12
140.80
139.18
136.79
134.58
132.93
129.87
129.12
129.02
128.97
112.48
103.05
69.09
52.37
46.53
28.14
HO
Cl
N
Ac
54
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

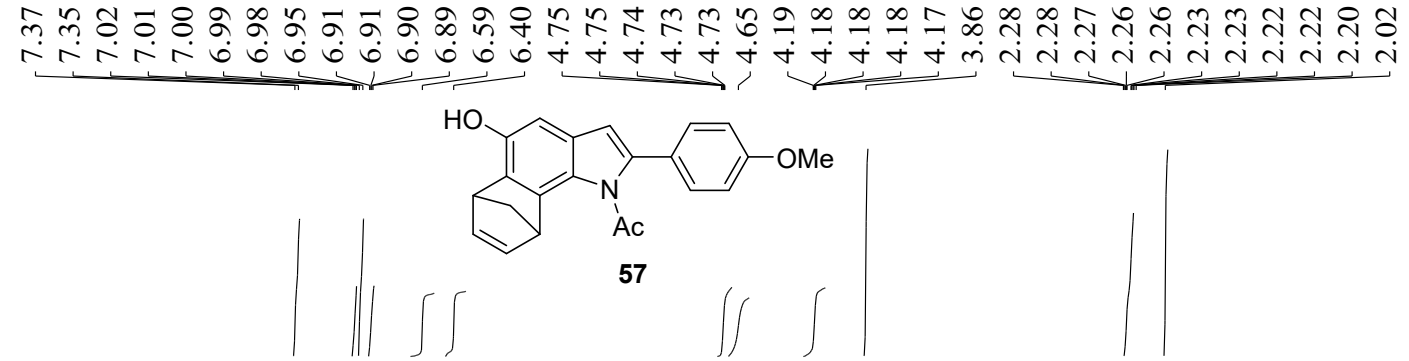

C-Br[D]-1.1.fid
7.59 7.57 7.34 7.32 7.01 7.00 7.00 6.99 6.91 6.91 6.90 6.89 6.61 6.46
4.76 4.76 4.75 4.75 4.74 4.74 4.63 4.19 4.18 4.18 4.17
2.29 2.29 2.28 2.28 2.27 2.27 2.24 2.24 2.23 2.22 2.22 2.21 2.05
HO
Br
N
Ac
55
2.00 2.01 1.00 1.00 1.00 1.00 1.00 0.89 1.00 2.08 3.01
9.0 8.5 8.0 7.5 7.0 6.5 6.0 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)


C-Br[D]-C-1.1.fid
170.63
147.01
143.80
143.12
140.81
136.82
133.40
131.97
130.19
130.12
129.12
122.70
112.50
103.06
69.09
52.37
46.54
28.17
HO
Br
N
Ac
55
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

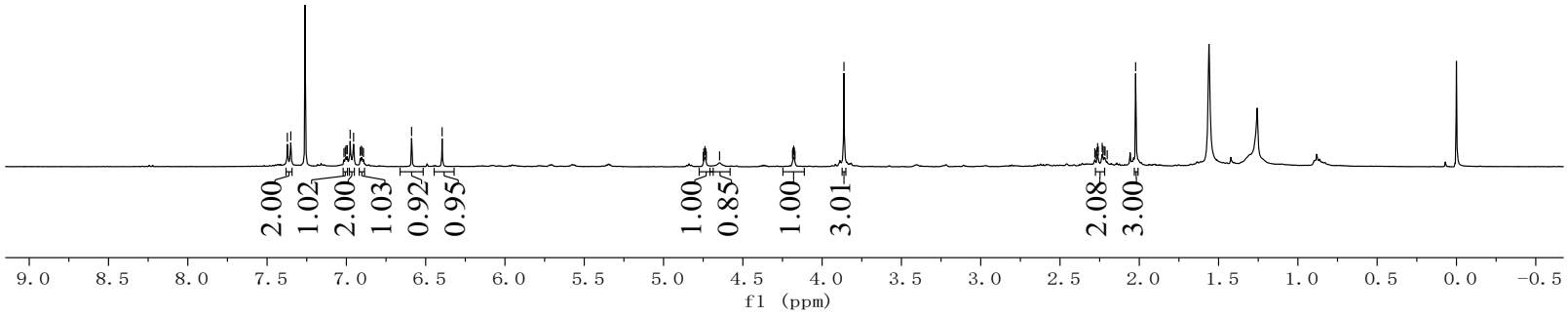

C-W[D].1.fid
7.35 7.33 7.25 7.23 7.00 6.99 6.91 6.90 6.90 6.59 6.43 4.76 4.75 4.75 4.72 4.19 4.18 2.67 2.65 2.63 2.28 2.27 2.26 2.26 2.24 2.24 2.23 2.22 2.02 1.67 1.65 1.63 1.61 1.37 1.36 1.36 1.35 1.34 1.33 0.93 0.91 0.89
HO
(CH2)4Me
N
Ac
56
2.01 2.00 1.00 1.00 0.90 0.90 1.00 0.80 1.00 2.00 2.02 3.01 2.05 4.06 3.02
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)

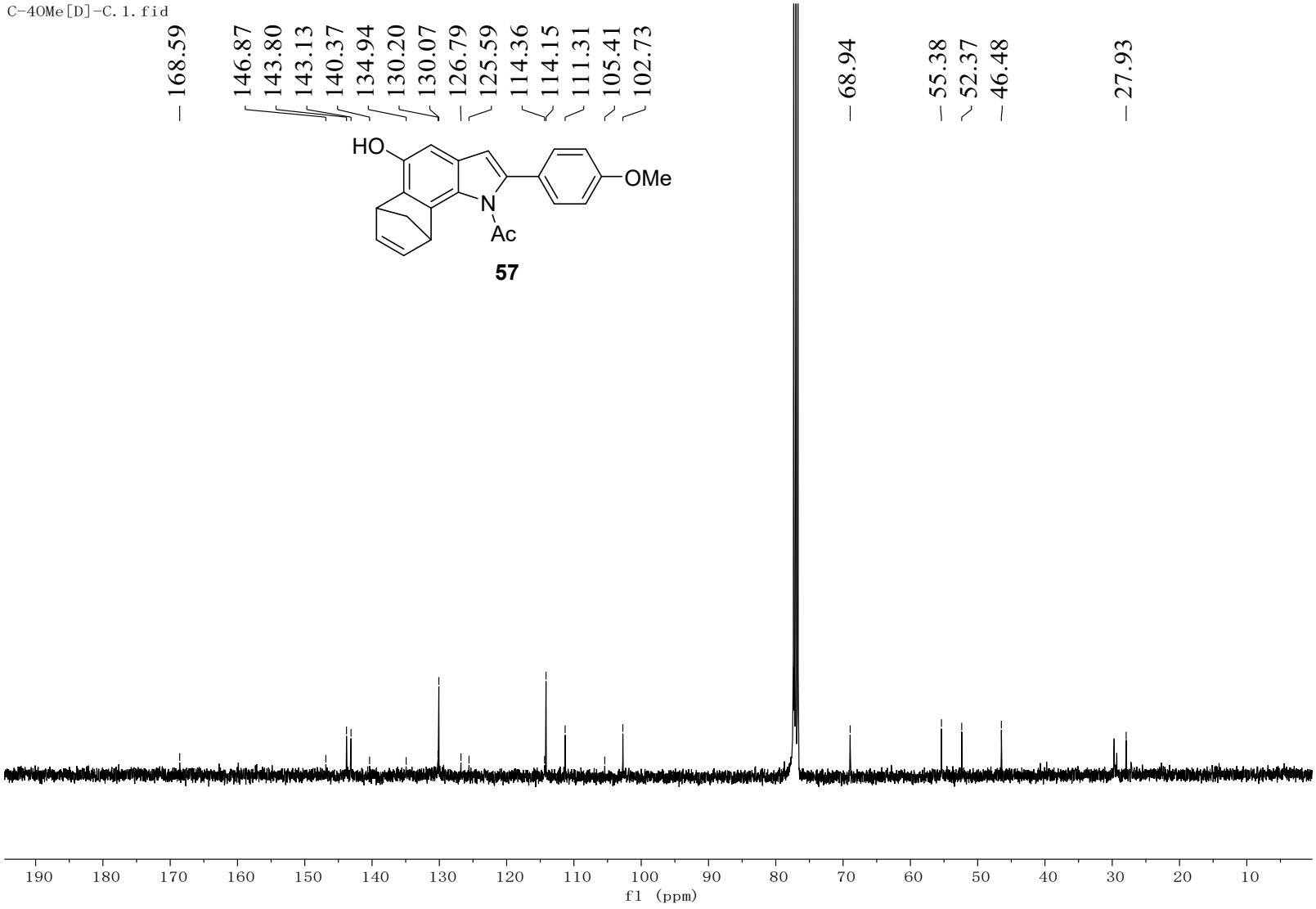

C-W[D]-C.1.fid
171.13
146.90
143.82
143.57
143.13
140.71
140.63
136.17
131.70
129.37
128.71
128.65
111.56
102.82
68.97
52.36
46.49
35.71
31.51
31.04
27.97
22.53
14.03
HO
(CH2)4Me
N
Ac
56
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

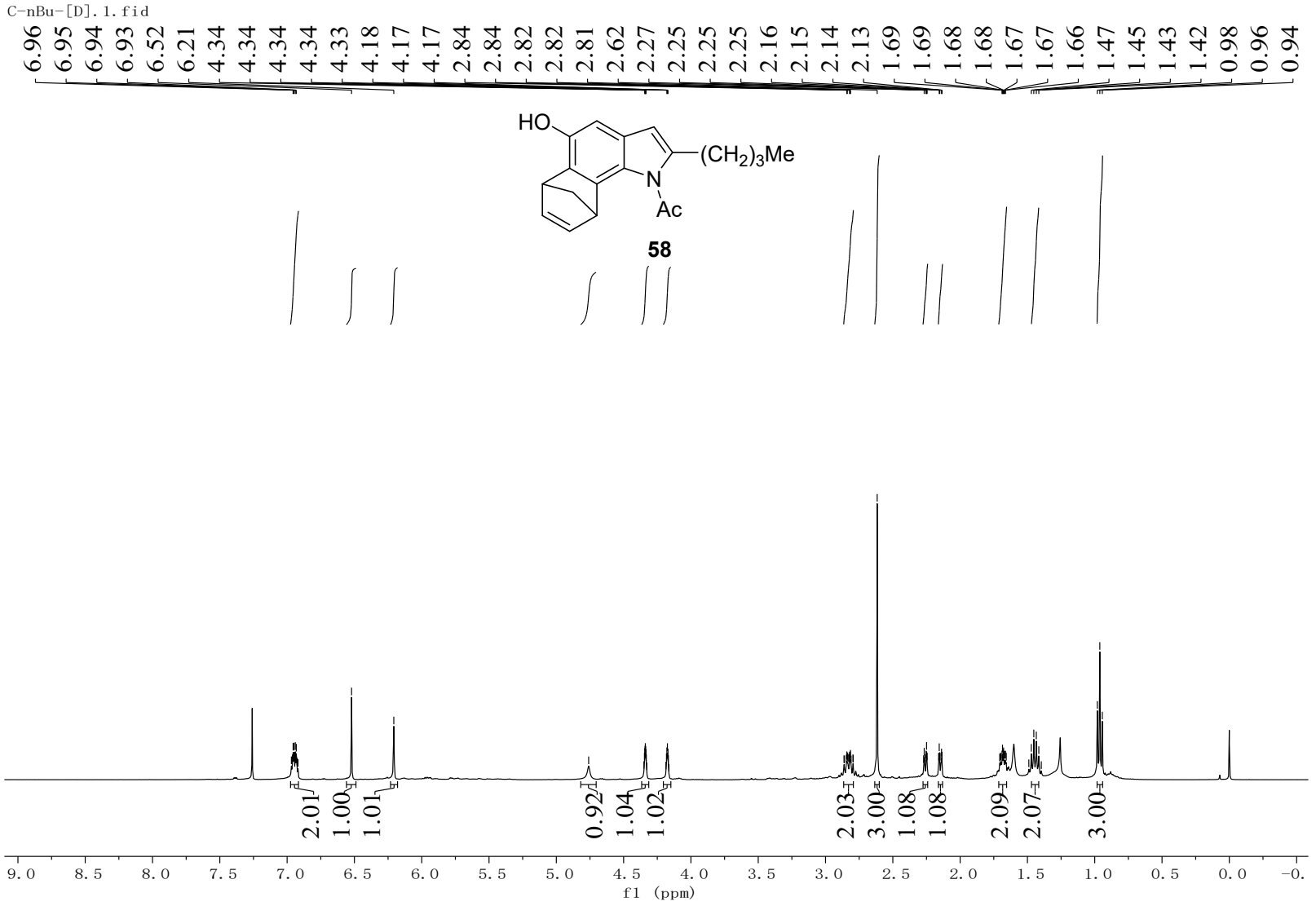
C-OMe[D].1.fid



57

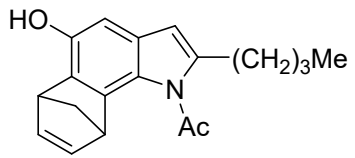



C-4OMe[D]-C.1.fid
168.59
146.87
143.80
143.13
140.37
134.94
130.20
130.07
126.79
125.59
114.36
114.15
111.31
105.41
102.73
68.94
55.38
52.37
46.48
27.93
HO
OMe
N
Ac
57
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

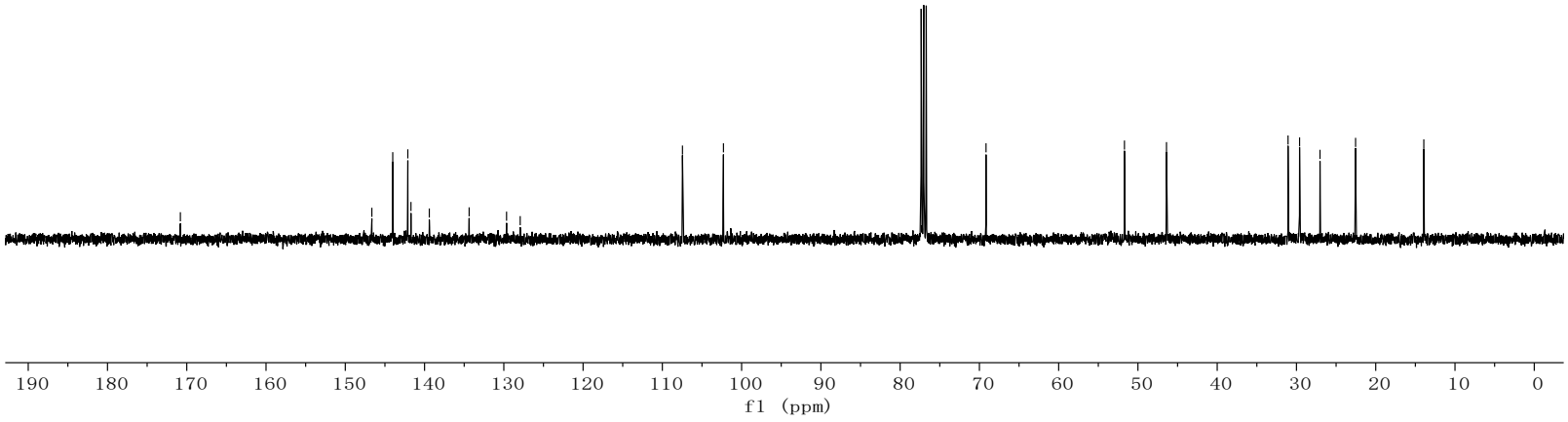

C-nBu-[D].1.fid
6.96 6.95 6.94 6.93 6.52 6.21 4.34 4.34 4.34 4.34 4.33 4.18 4.17 4.17 2.84 2.84 2.82 2.82 2.81 2.62 2.27 2.25 2.25 2.25 2.16 2.15 2.14 2.13 1.69 1.69 1.68 1.68 1.67 1.67 1.66 1.47 1.45 1.43 1.42 0.98 0.96 0.94
HO
(CH2)3Me
N
Ac
58
2.01 1.00 1.01 0.92 1.04 1.02 2.03 3.00 1.08 1.08 2.09 2.07 3.00
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 -0.
f1 (ppm)

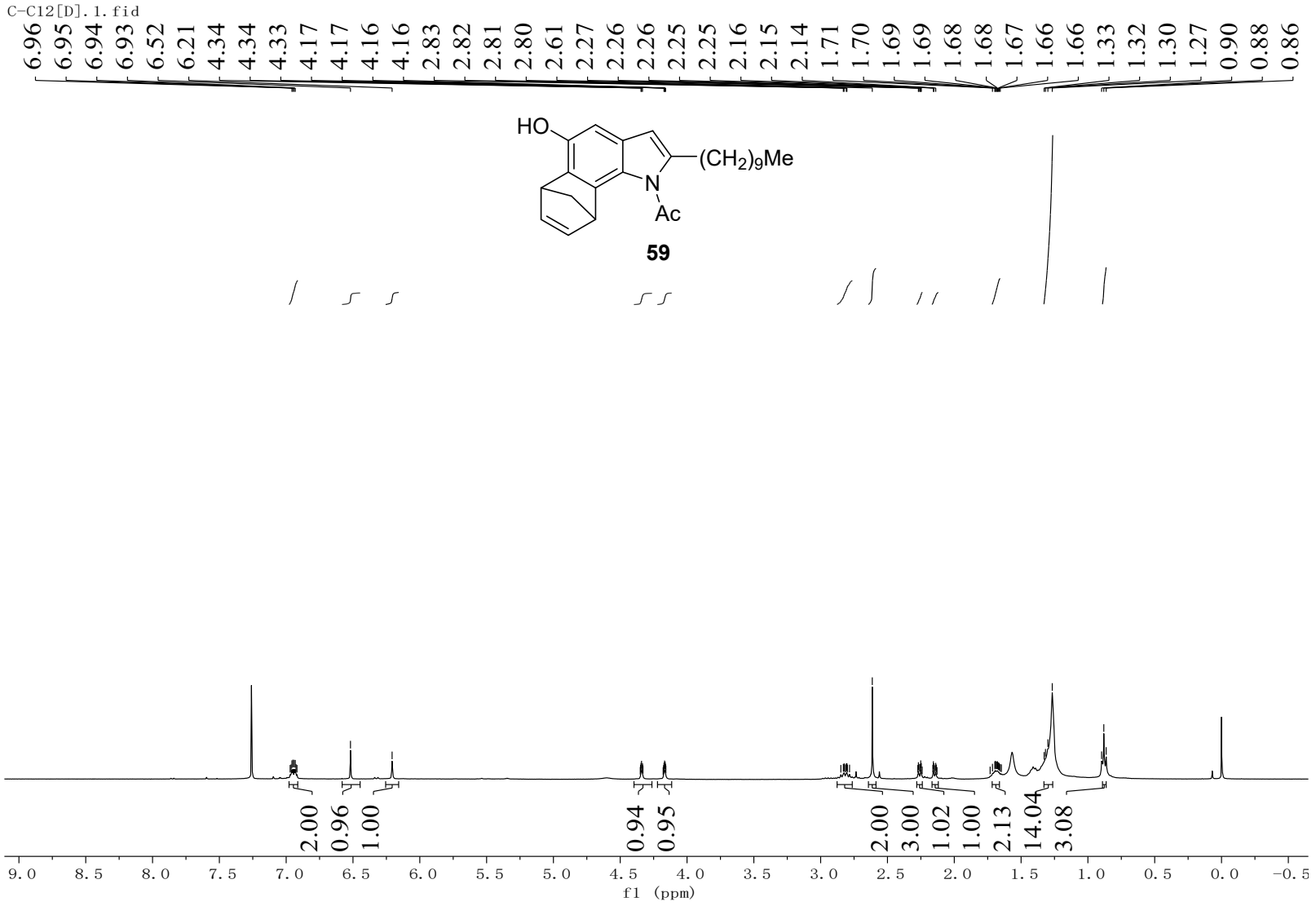
C-nBu-[D]-C.1.fid

170.81
146.65
144.01
142.11
141.71
139.40
134.37
129.65
127.93
107.46
102.29
69.16
51.70
46.40
31.06
29.61
27.03
22.54
13.93



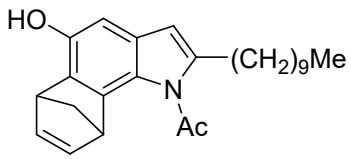
58



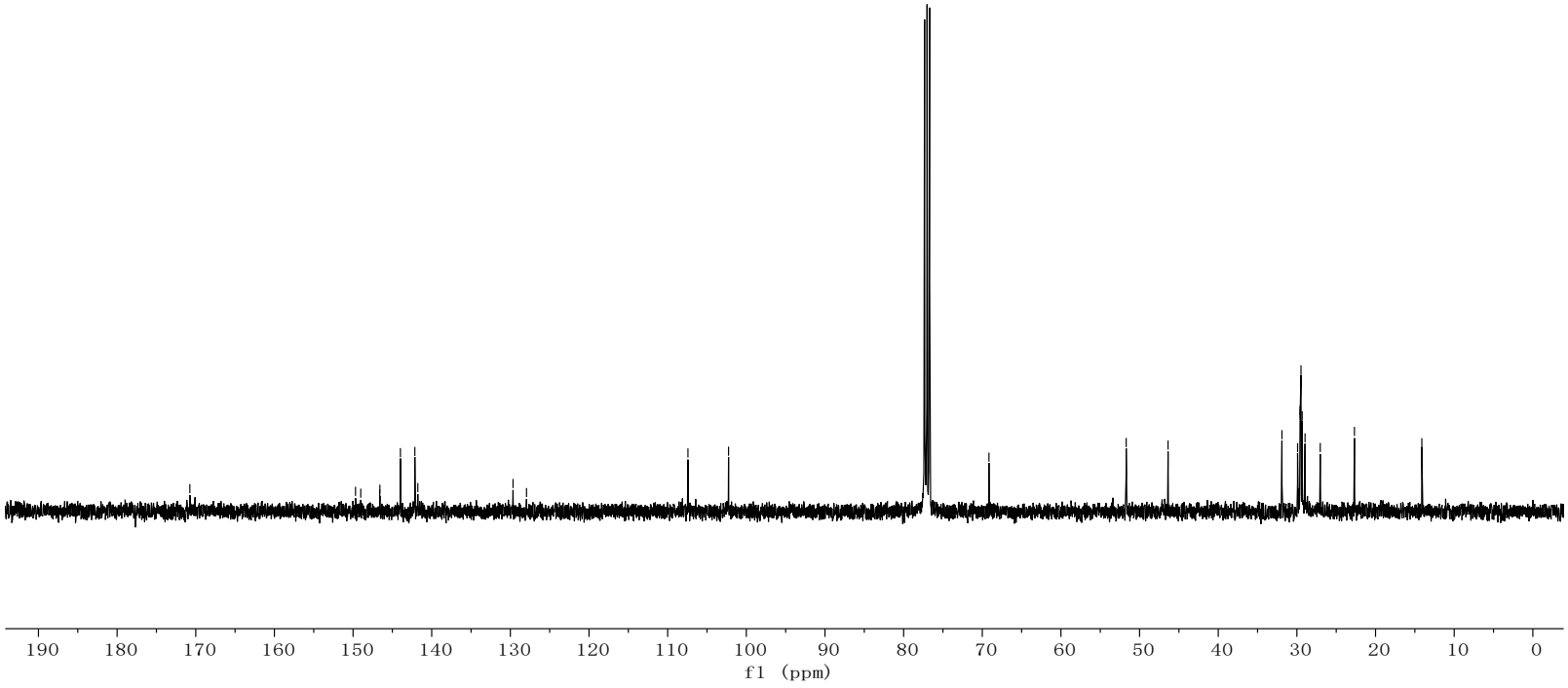

C-C12[D].1.fid
6.96 6.95 6.94 6.93 6.52 6.21 4.34 4.34 4.33 4.17 4.17 4.16 4.16 2.83 2.82 2.81 2.80 2.61 2.27 2.26 2.26 2.25 2.25 2.16 2.15 2.14 1.71 1.70 1.69 1.69 1.68 1.68 1.67 1.66 1.66 1.33 1.32 1.30 1.27 0.90 0.88 0.86
HO
(CH2)9Me
N
Ac
59
2.00 0.96 1.00 0.94 0.95 2.00 3.00 1.02 1.00 2.13 14.04 3.08
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 -0.5
f1 (ppm)

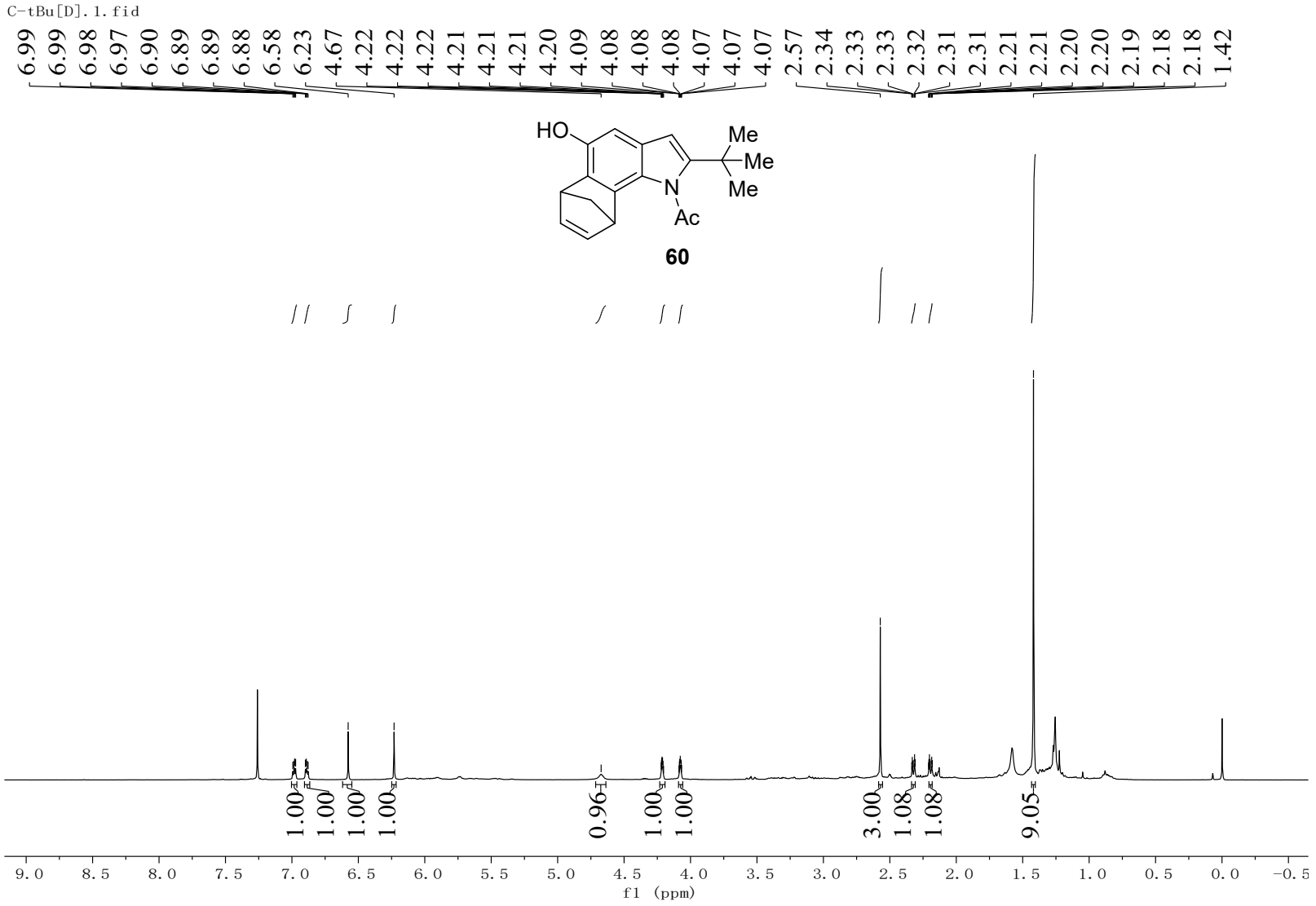
C-C12[D]-C.1.fid

170.75
149.69
149.02
146.62
146.59
143.98
142.15
141.77
129.66
127.95
107.42
102.26
69.15
51.70
46.38
31.90
29.91
29.60
29.58
29.48
29.32
28.96
27.03
22.68
14.11



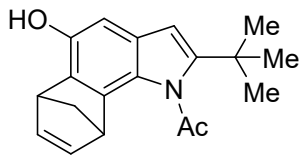
59



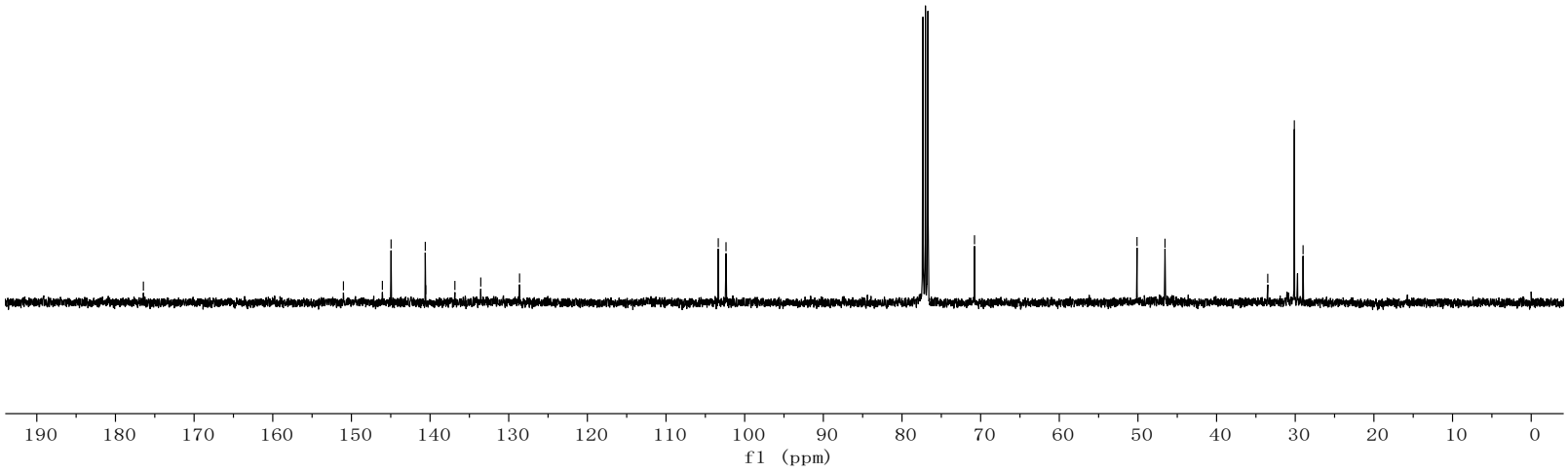

C-tBu[D].1.fid
6.99 6.99 6.98 6.97 6.90 6.89 6.89 6.88 6.58 6.23 4.67 4.22 4.22 4.22 4.21 4.21 4.21 4.20 4.09 4.08 4.08 4.08 4.07 4.07 4.07
2.57 2.34 2.33 2.33 2.32 2.31 2.31 2.21 2.21 2.20 2.20 2.19 2.18 2.18 1.42
HO
Me
Me
Me
N
Ac
60
1.00 1.00 1.00 1.00 0.96 1.00 1.00 3.00 1.08 1.08 9.05
9.0 8.5 8.0 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 -0.5
f1 (ppm)

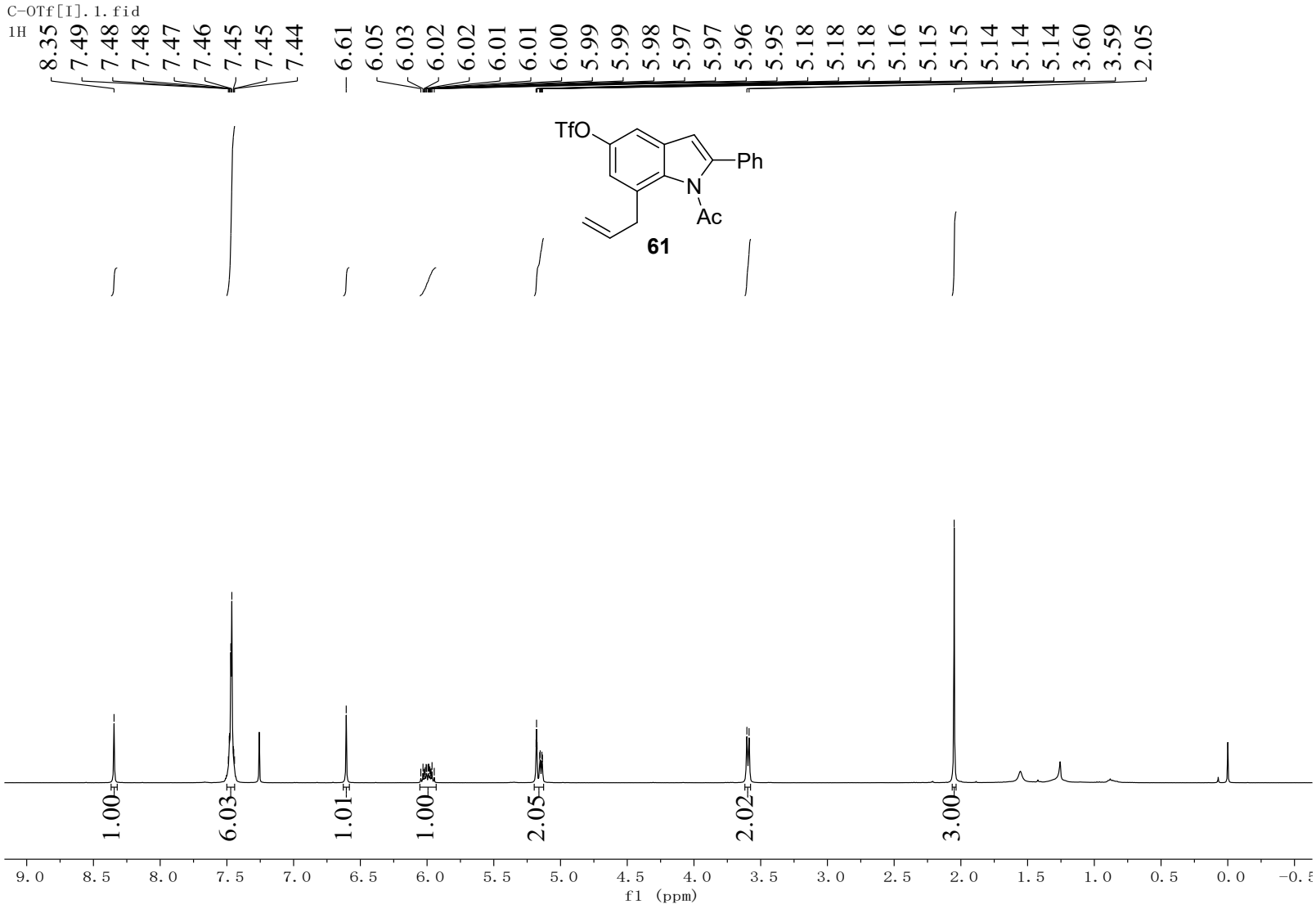
C-tBu[D]-C.1.fid

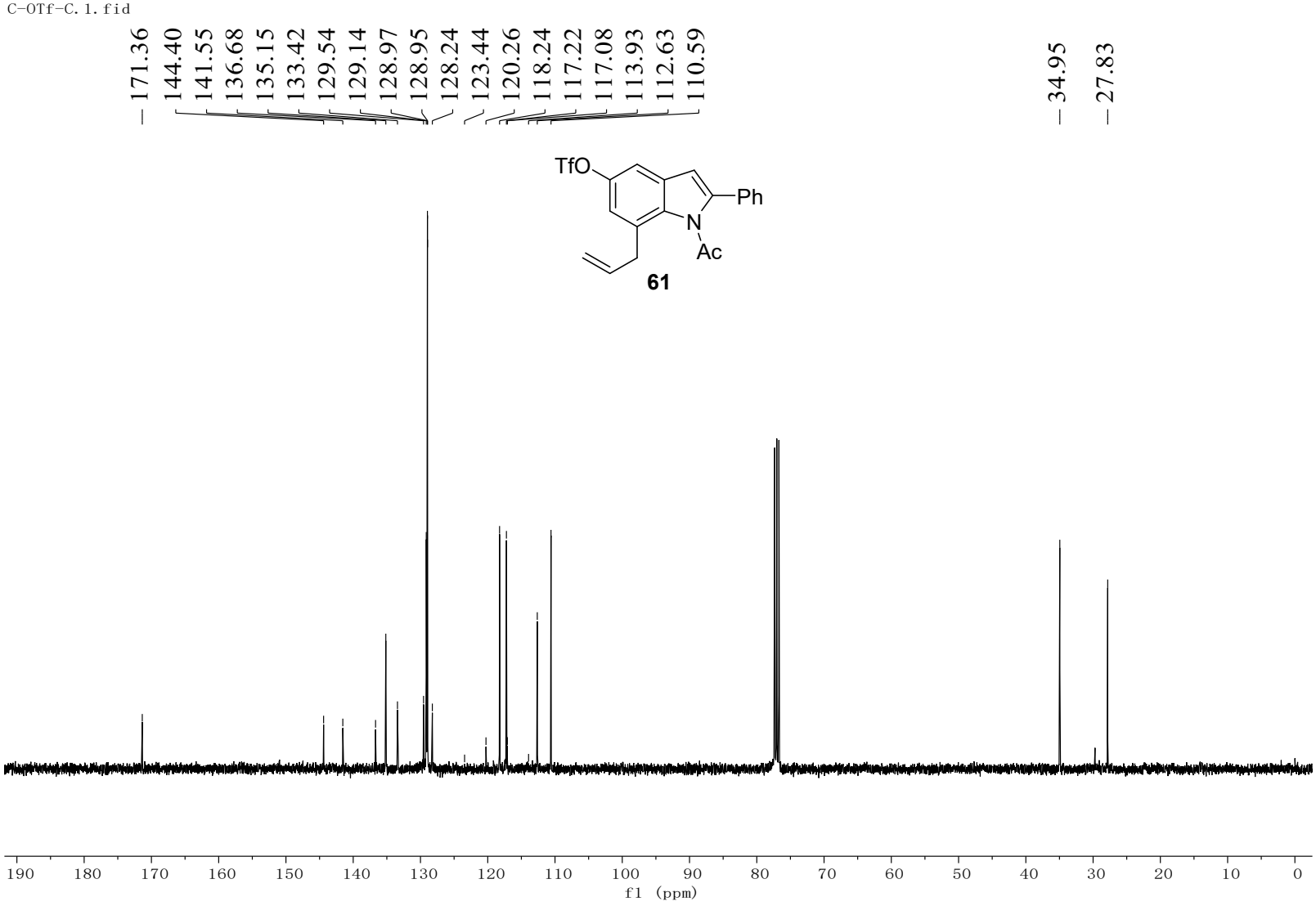
176.45
151.01
146.06
144.96
140.61
140.55
136.84
133.55
128.62
103.36
102.37
70.78
50.12
46.56
33.50
30.12
29.00



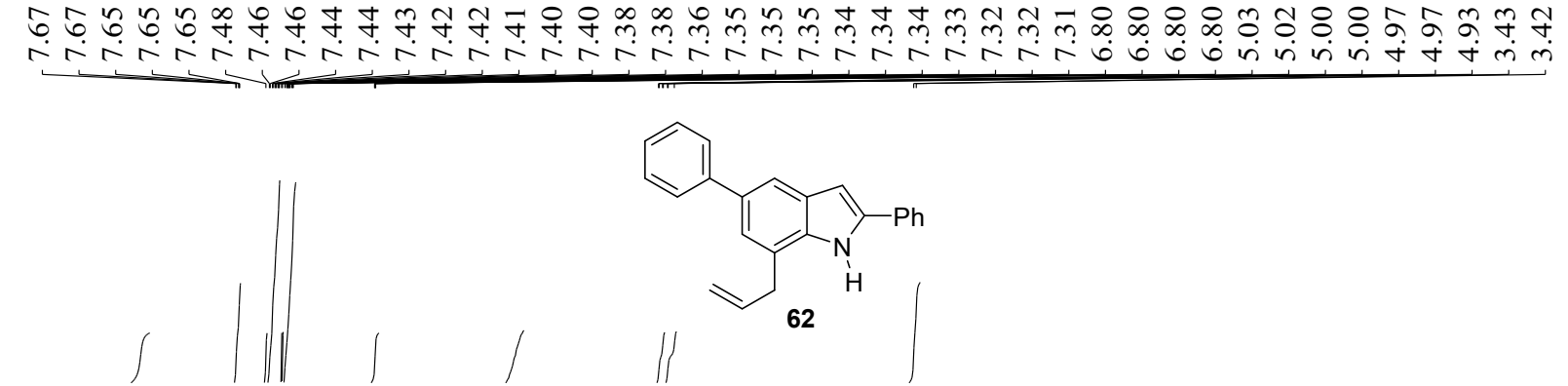
60




C-OTf[I].1.fid
1H
8.35
7.49
7.48
7.48
7.47
7.46
7.45
7.45
7.44
6.61
6.05
6.03
6.02
6.02
6.01
6.01
6.00
5.99
5.99
5.98
5.97
5.97
5.96
5.95
5.18
5.18
5.18
5.16
5.15
5.15
5.14
5.14
5.14
3.60
3.59
2.05
TfO
Ph
N
Ac
61
1.00
6.03
1.01
1.00
2.05
2.02
3.00
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)


C-OTf-C.1.fid
171.36
144.40
141.55
136.68
135.15
133.42
129.54
129.14
128.97
128.95
128.24
123.44
120.26
118.24
117.22
117.08
113.93
112.63
110.59
34.95
27.83
TfO
Ph
N
Ac
61
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

C-OTfB-2.1.fid



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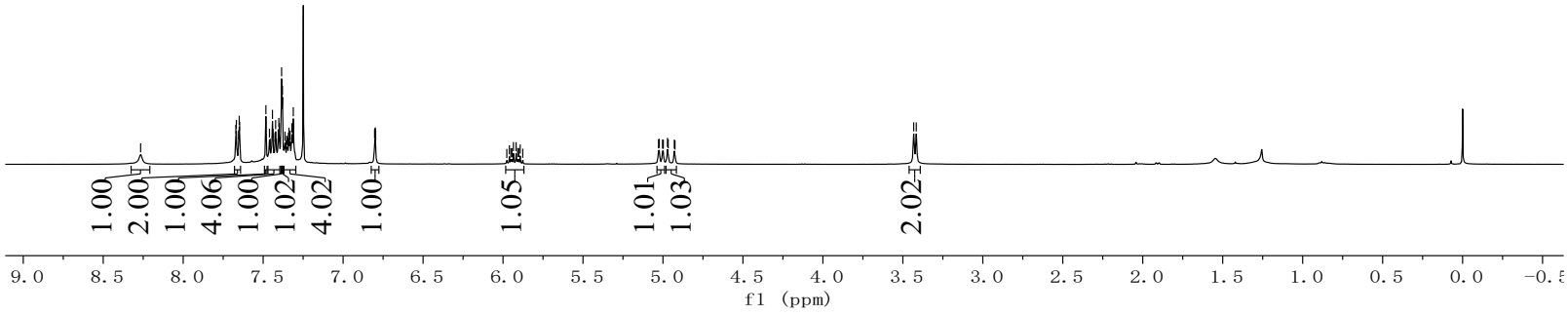
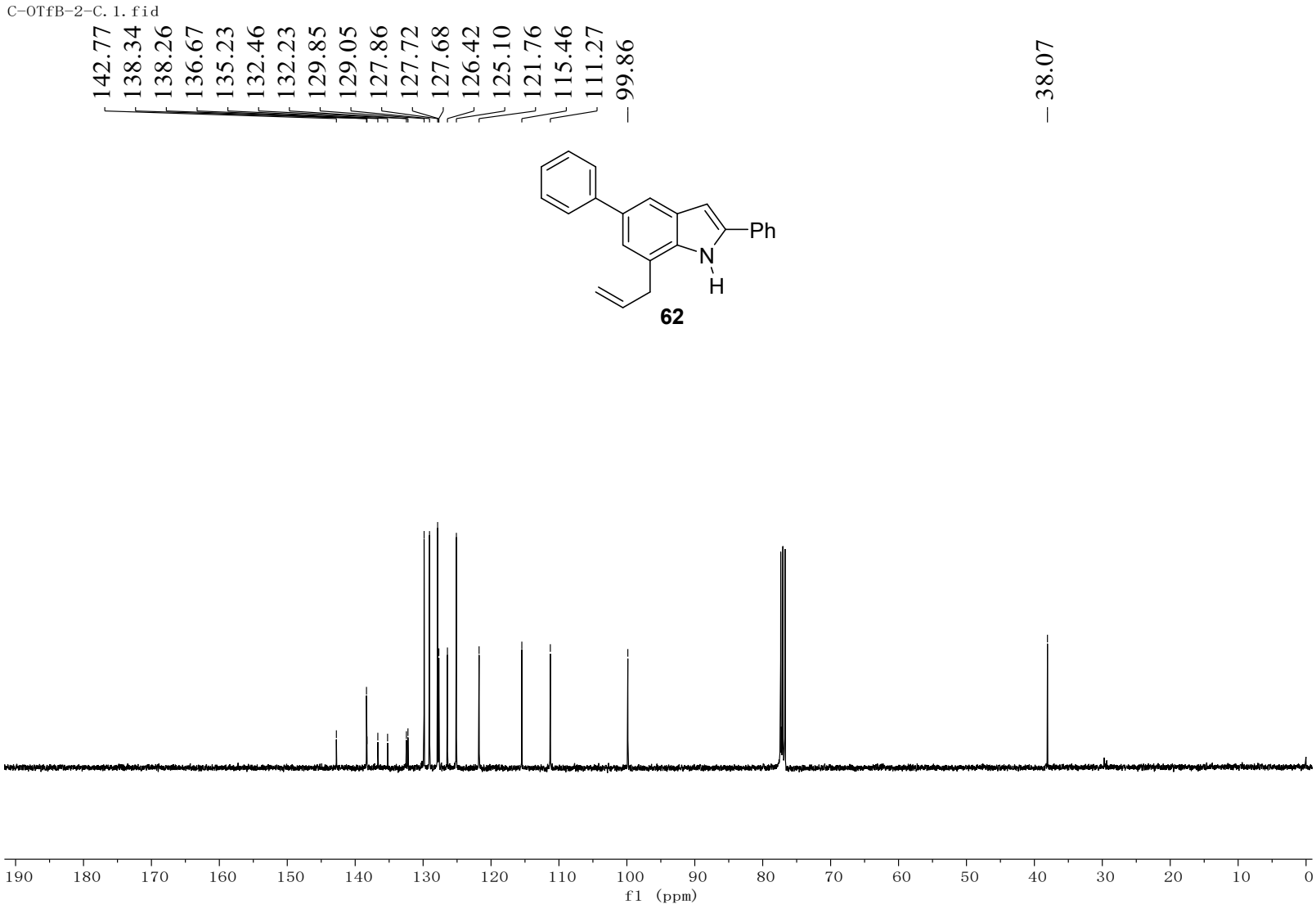
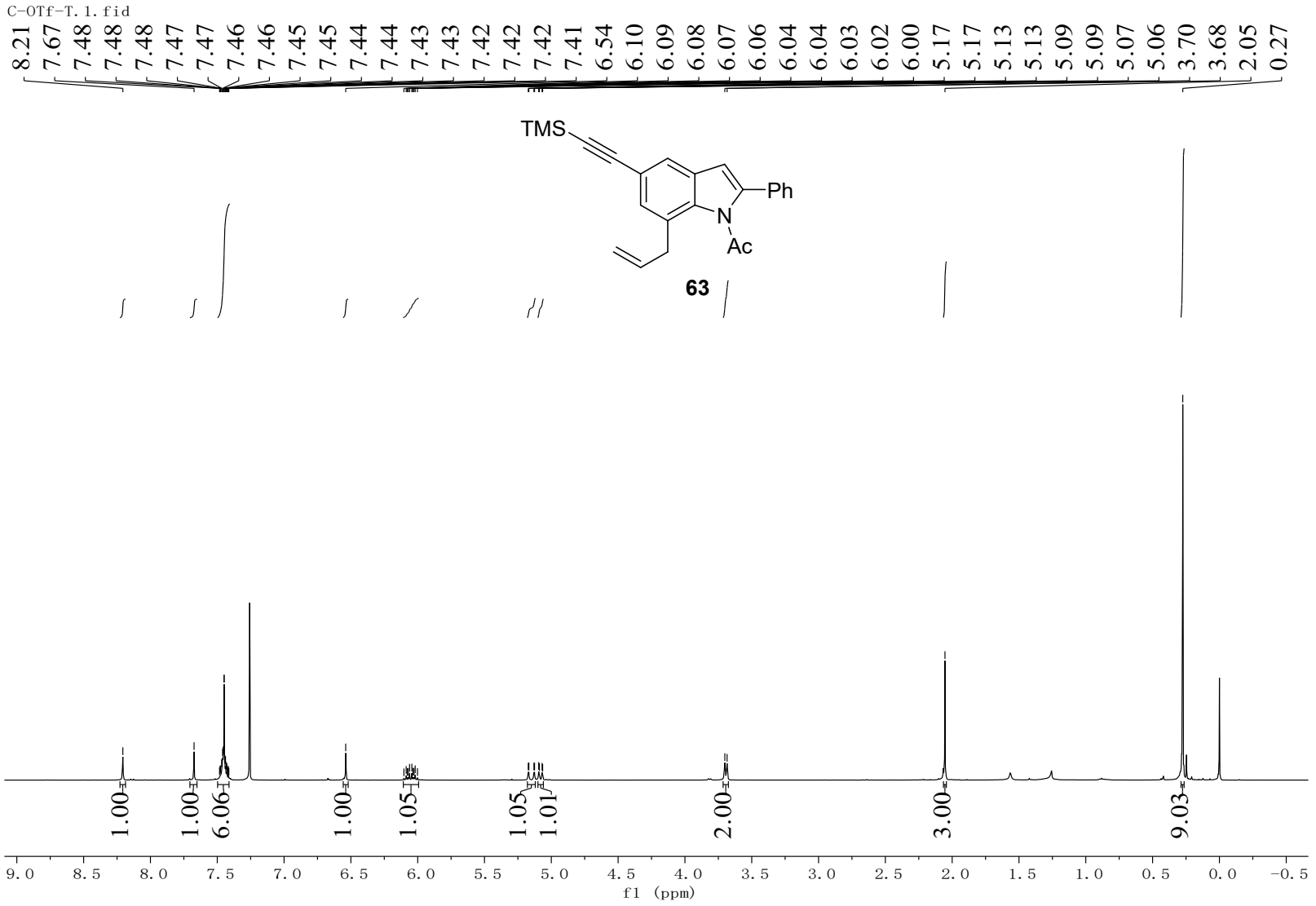
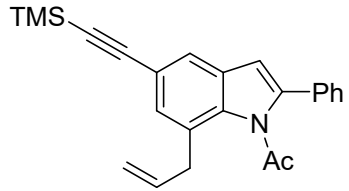



C-OTfB-2-C.1.fid
142.77
138.34
138.26
136.67
135.23
132.46
132.23
129.85
129.05
127.86
127.72
127.68
126.42
125.10
121.76
115.46
111.27
99.86
38.07
Ph
N
H
62
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


C-OTf-T.1.fid
8.21 7.67 7.48 7.48 7.48 7.47 7.47 7.46 7.46 7.45 7.45 7.44 7.44 7.43 7.43 7.42 7.42 7.42 7.41 6.54 6.10 6.09 6.08 6.07 6.06 6.04 6.04 6.03 6.02 6.00 5.17 5.17 5.13 5.13 5.09 5.09 5.07 5.06 3.70 3.68 2.05 0.27
TMS
Ph
N
Ac
63
1.00 1.00 6.06 1.00 1.05 1.05 1.01 2.00 3.00 9.03
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 -0.5
f1 (ppm)

C-OTf-T-C.1.fid

171.43
140.22
139.81
137.86
136.94
133.90
128.89
128.80
128.72
127.16
124.61
118.27
116.06
115.84
110.99
104.38
96.91
39.49
27.93
0.09



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