Supporting Information

Hydroxylation of Organoborons *via* Uranyl-Photocatalysis

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I. General information

All the solvents were dried using standard procedure (*Purification of Laboratory Chemicals*) and distilled before use. Reactions were monitored by thin layer chromatography (TLC) supplied by Yantai Jiangyou Silicon Material Company (China) and visualization was accomplished with a combination of UV-light (254 nm or 365 nm). Flash column chromatography was achieved using 300 – 400 mesh silica gel supplied by Yantai Jiangyou Silicon Material Company (China). ^{1}H and ^{13}C spectra were recorded on 400 MHz or 500 MHz NMR spectrometers (Bruker AVANCE). Chemical shifts for protons are reported in parts per million (ppm) downfield and are referenced to residual protium in the NMR solvent ($CHCl_3$ = δ 7.26 ppm, DMSO = δ 2.50 ppm). Chemical shifts for carbon are reported in parts per million downfield and are referenced to the carbon resonances of solvent ($CHCl_3$ = δ 77.00 ppm, DMSO = δ 39.52 ppm). Data are represented as follows: chemical shift, multiplicity (br = broad, s = singlet, d = double, t = triplet, q = quartet, m = multiplet), coupling constants in Hertz (Hz), integration. IR spectra were recorded on a SHIMADZU IR Tracer-100 Spectrometer. Mass spectra were in general recorded on a Shimadzu GCMS-QP2010 Ultra and an HP 5989A mass selective detector.

Safety information about U-based derivatives

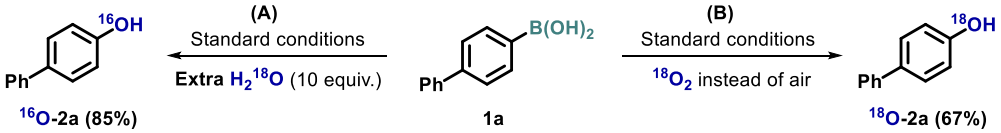
Isotope	^{234}U	^{235}U	^{238}U
Radiation (μSv)[a]	strong	strong	weak (<199)
Natural uranium	0.0057%	0.72%	99.28%
Depleted uranium	0.001%	0.20%	99.8%
Commercial uranium	0	0	100%

Note 0-199 μSv: harmless; 200-1000 μSv: slight damage; 1-1000 mSv: mild dizziness with a feeling of vomiting; 1000-4000 mSv: internal organs are damaged; > 4000 mSv: extreme danger, a threat to life. 1000 μSv = 1 mSv.

II. Mechanistic studies

1) Oxygen labeling reactions

$^{18}O_2$ and $H_2{}^{18}O$ labelling experiments of aryl borides.



To a 25 mL Schlenk tube, 4-biphenylboronic acid **1a** (0.2 mmol, 39.6 mg), $UO_2(OAc)_2{\cdot}2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and $H_2{}^{18}O$ (2 mmol, 40 mg) were stirred in acetone (2 mL) under N_2 atmosphere at room temperature for 20 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. The result of mass spectrometry (MS) indicated that there was no ^{18}O-labeled product **2a**.

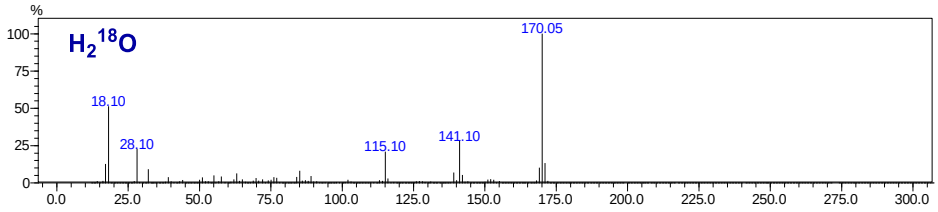


Figure S1. ^{18}O labeling experiments of 4-biphenylboronic acid **1a** with $H_2{}^{18}O$.

To a 25 mL Schlenk tube, 4-biphenylboronic acid **1a** (0.2 mmol, 39.6 mg), $UO_2(OAc)_2{\cdot}2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under $^{18}O_2$ atmosphere at room temperature for 20 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. The result of mass spectrometry (MS) indicated that most of product **2a** was labeled with $^{18}O_2$.

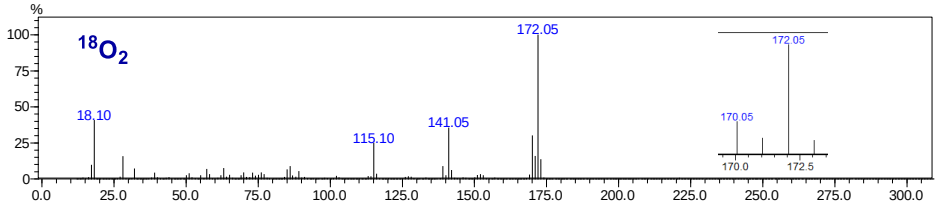


Figure S2. ^{18}O labeling experiments of 44-biphenyl boronicacid **1a** with $^{18}O_2$.

Labeling experiments with $H_2{}^{18}O$ and $^{18}O_2$ unambiguously confirmed that the oxygen atoms on the phenols originated from oxygen instead of water.

2) UV-vis absorption experiments

Ultraviolet-visible absorption experiments were performed using a Shimadzu UV-2700 UV-visible spectrophotometer. In each experiment, the varying samples were combined in acetone in screw-top 1.0 cm quartz cuvettes. The concentration of each component was under standard conditions.

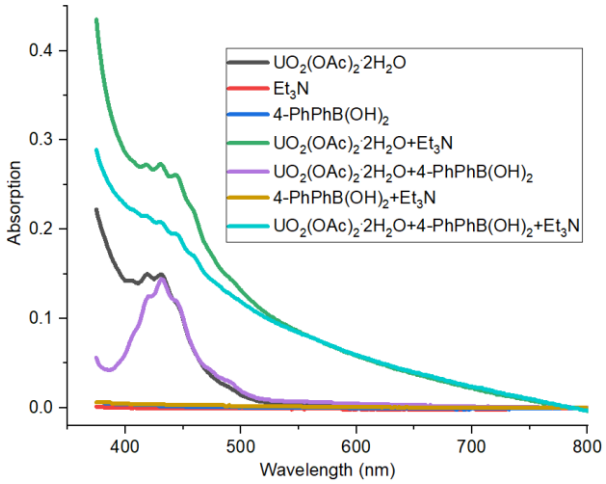


Figure S3. UV-visible absorption experiments.

Ultraviolet-visible absorption of each reaction component indicated that $UO_2(OAc)_2{\cdot}2H_2O$ served as a photoredox catalyst in this system.

3) Fluorescence quenching experiments

Stern–Volmer fluorescence quenching experiments with $UO_2(OAc)_2{\cdot}2H_2O$. Fluorescence quenching studies were performed using a Shimadzu RF-6000 fluorescence spectrophotometer. In each experiment, the photoredox catalyst and varying concentrations of quencher were combined in acetone in screw-top 1.0 cm quartz cuvettes. For the emission quenching of $UO_2(OAc)_2{\cdot}2H_2O$, the photoredox catalyst concentration was 2×10^{-4} M, the solution was irradiated at 432 nm.

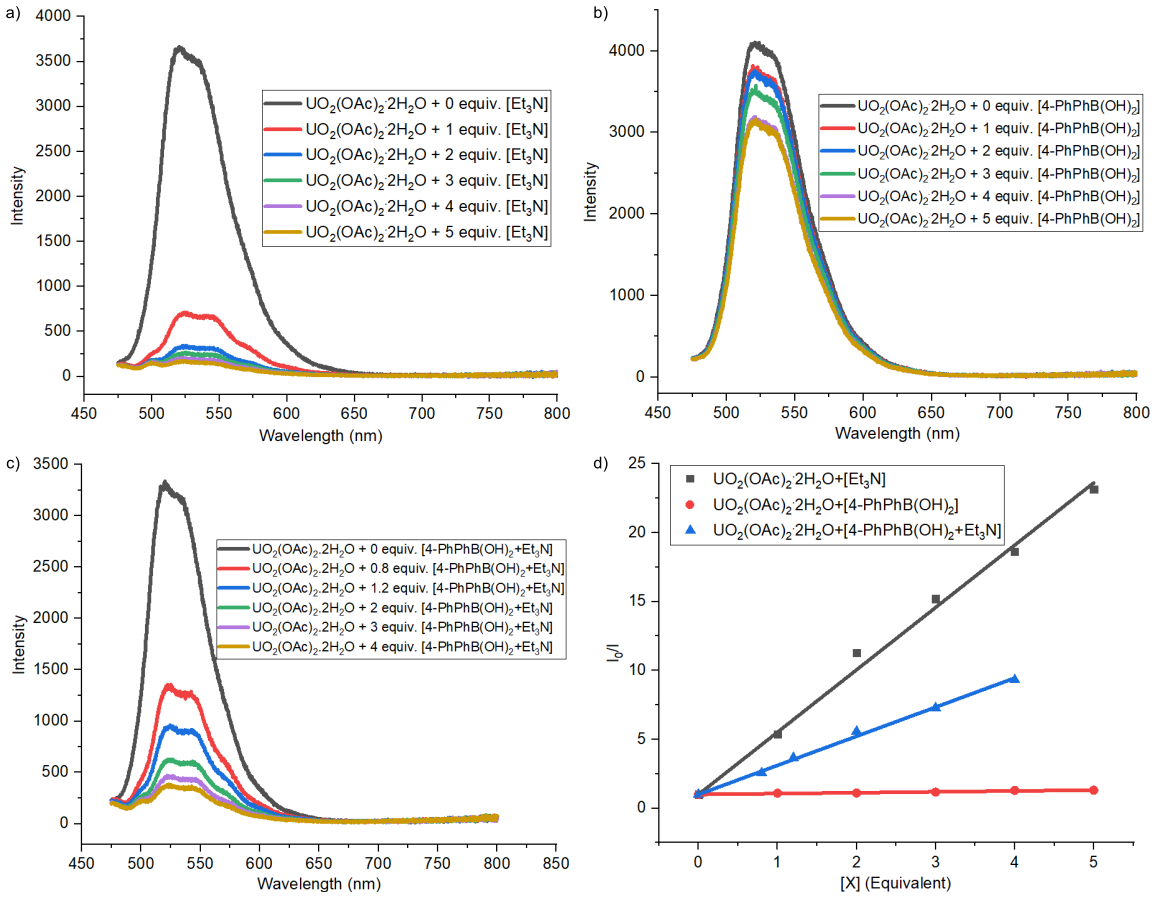


Figure S4: a) Fluorescence quenching of $UO_2(OAc)_2{\cdot}2H_2O$ with varied [Et_3N] in acetone. **b)** Fluorescence quenching of $UO_2(OAc)_2{\cdot}2H_2O$ with varied [4-PhPhB$(OH)_2$] in acetone. **c)** Fluorescence quenching of $UO_2(OAc)_2{\cdot}2H_2O$ with varied [4-PhPhB$(OH)_2$] in acetone. **d)** Stern-Volmer plots of fluorescence quenching experiments.

The Stern-Volmer analysis revealed that the excited state of $UO_2(OAc)_2{\cdot}2H_2O$ photoredox catalyst was efficiently quenched by the mixture of 4-PhPhB$(OH)_2$ and Et_3N in acetone at room temperature.

Fluorescence quenching experiments with other electron donors. Fluorescence quenching studies were performed using a Shimadzu RF-6000 Fluorescence Spectrophotometer. In each experiment, the photoredox catalyst and varying concentrations of quencher were combined in acetone in screw-top 1.0 cm quartz cuvettes. For the fluorescence quenching of $UO_2(OAc)_2{\cdot}2H_2O$ with Et_3N, Et_2NH, DIPEA, *i*-Pr_2NH and PMe_3, the photoredox catalyst concentration was 2×10^{-4} M, the solution was irradiated at 432 nm.

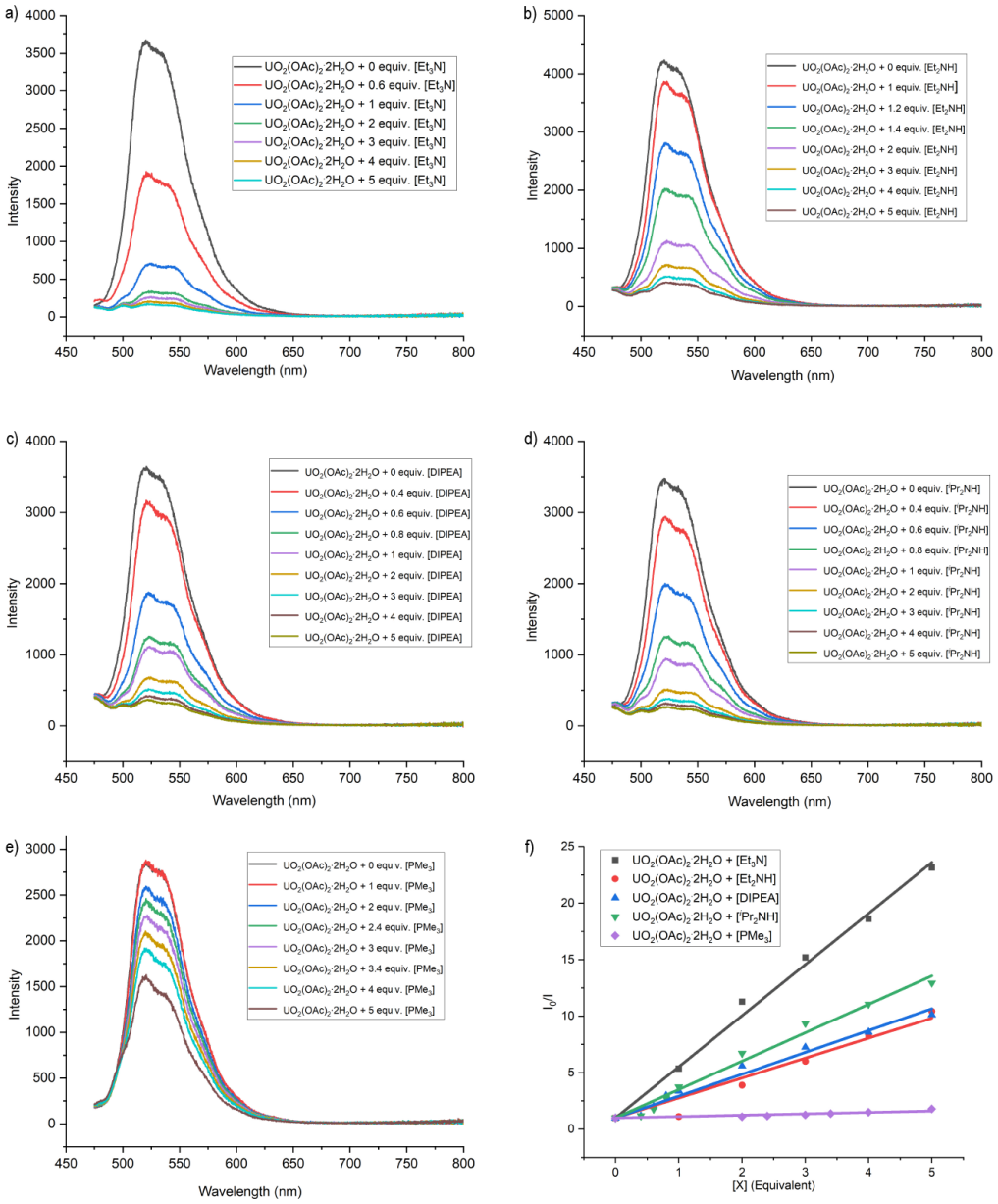


Figure S5: a) Fluorescence quenching of $UO_2(OAc)_2{\cdot}2H_2O$ with varied [Et_3N] in acetone. **b)** Fluorescence quenching of $UO_2(OAc)_2{\cdot}2H_2O$ with varied [Et_2NH] in acetone. **c)** Fluorescence quenching of $UO_2(OAc)_2{\cdot}2H_2O$ with varied [DIPEA] in acetone. **d)** Fluorescence quenching of $UO_2(OAc)_2{\cdot}2H_2O$ with varied [$^i Pr_2NH$] in acetone. **e)** Fluorescence quenching of $UO_2(OAc)_2{\cdot}2H_2O$ with varied [PMe_3] in acetone. **f)** Stern-Volmer plots of fluorescence quenching experiments.

4) Mass spectrometry (MS) experiments

To a 25 mL Schlenk tube, 4-biphenyl boronic acid **1a** (0.2 mmol, 39.6 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under an air atmosphere at room temperature for 20 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. The result of mass spectrometry (MS) indicates ted presence of diethylamine (Et_2NH) in the system.

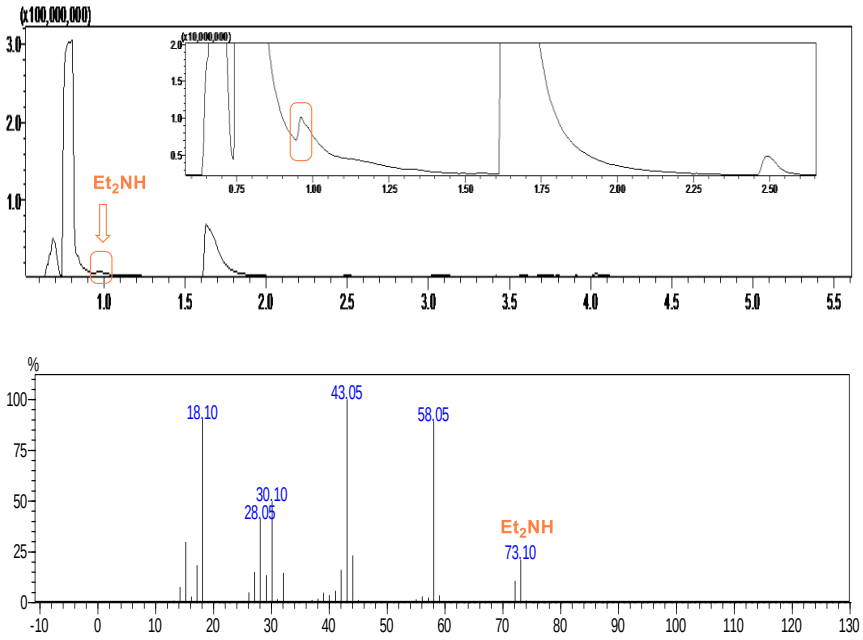


Figure S6: GC-MS data of diethylamine (Et_2NH).

III. DFT calculations

DFT calculations were performed with the Gaussian16 program suite[1]. All molecule geometry optimizations were performed by using B3LYP-D3(BJ)/6-31+G(d,p). Frequency calculations were conducted to ensure that these structures were indeed local minima. Transition state structures were verified by frequency calculations and only one imaginary frequency was found in the transition states. All molecular structures were rendered by Visual Molecular Dynamics (VMD 1.9.3) software[2].

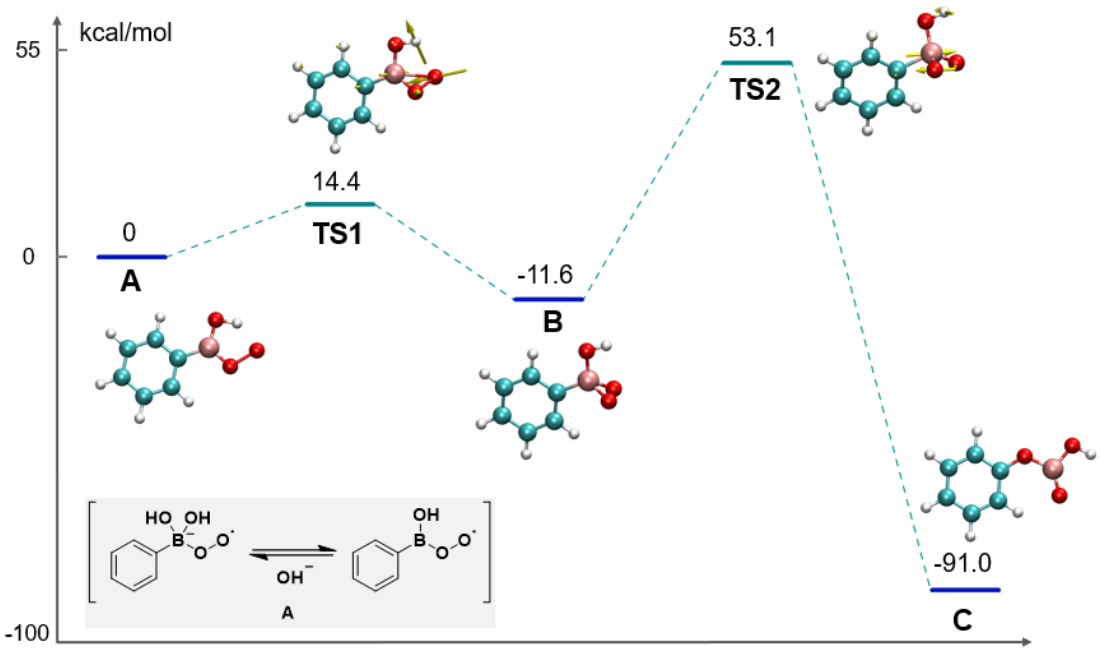
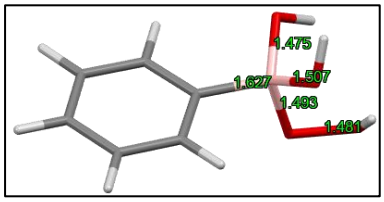


Figure S7: DFT calculated reaction pathway **b** at the level of M06-2X-D3(0)/ma-def2-TZVP. The yellow arrow was the imaginary frequency vibration vector of the transition state. All units were in kcal mol^{-1}.

Molecular cartesian coordinates and thermochemistry data

Optimized Cartesian coordinates (in Å) of all calculated molecules at the B3LYP-D3(BJ)/6-31+G(d,p) level of theory in the gas phase.

Path A: **B**

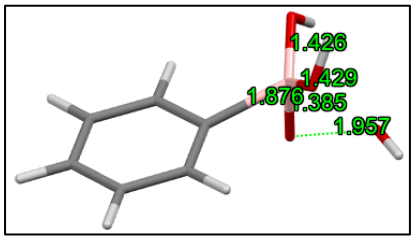


Thermal correction to Gibbs Free Energy ($G_{corr}(T)$) = 0.104063 Hartrees
Electronic energy (ε_{ele}) = -559.306896607 Hartrees
The sum of electronic and thermal Free Energies (G(T)) = -559.202833607 Hartrees
Temperature = 298.15 K, Pressure = 1 Atm.

Atom	x	y	z
B	1.17258300	0.15271300	0.23993000
O	1.72520100	-0.86667600	1.20231800
H	1.88454200	-0.39866100	2.02891500
O	1.71301900	-0.22420500	-1.10033300
C	-0.44504200	0.05840200	0.09675100

C	-1.10615500	-1.18236600	0.12890600
C	-1.24469900	1.20032100	-0.07736200
C	-2.49401400	-1.28475400	-0.00711500
H	-0.50908300	-2.07942700	0.27250800
C	-2.63572300	1.11581500	-0.21119500
H	-0.75188300	2.16878400	-0.09824700
C	-3.26840100	-0.13109000	-0.17838500
H	-2.97546400	-2.26117700	0.02081300
H	-3.22770600	2.02057300	-0.34207300
H	-4.34922000	-0.20392500	-0.28346700
O	3.18022000	-0.35036300	-0.94297700
O	1.58182900	1.50739800	0.65853600
H	2.44598500	1.66476800	0.25781100
H	3.18197400	-0.86170000	-0.10585300

Path A: **TS**

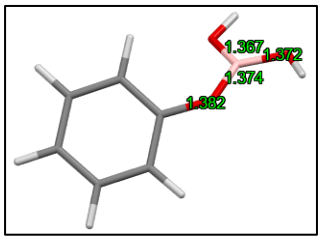


Thermal correction to Gibbs Free Energy ($G_{corr}(T)$) = 0.099193 Hartrees
Electronic energy (ε_{ele}) = -559.248311325 Hartrees
Sum of electronic and thermal Free Energies (G(T)) = -559.149118325 Hartrees
Temperature = 298.15 K, Pressure = 1 Atm.

Atom	x	y	z
B	1.37978400	-0.17011100	0.48006700
O	1.59146800	-1.50172200	0.95258200
H	2.06766400	-1.43821700	1.78763600
O	1.26018300	0.12294800	-0.86805500
C	-0.42144800	-0.01691100	-0.02414500
C	-1.16730100	-1.17062400	-0.28922900
C	-1.08078100	1.20278000	0.15704200
C	-2.56345100	-1.11290200	-0.33216700
H	-0.64470400	-2.11074900	-0.43696600
C	-2.47715900	1.26277300	0.12433800
H	-0.49226700	2.09735800	0.33952800
C	-3.22417400	0.10437100	-0.12348800
H	-3.13947600	-2.01558300	-0.52849100
H	-2.98514300	2.21198600	0.28529300
H	-4.31021700	0.15044900	-0.15716000
O	1.87193900	0.83835800	1.36123200

H	2.34755600	1.44697000	0.77719400
O	3.10229800	0.46154400	-1.43670200
H	3.25644600	-0.47760500	-1.61393600

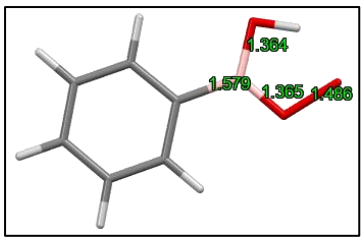
Path A: **C**



Thermal correction to Gibbs Free Energy ($G_{corr}(T)$) = 0.094315 Hartrees
Electronic energy (ε_{ele}) = -483.543731311 Hartrees
Sum of electronic and thermal Free Energies (G(T)) = -483.449416311 Hartrees
Temperature = 298.15 K, Pressure = 1 Atm.

Atom	x	y	z
B	-2.15915600	-0.00839500	-0.05167500
O	-2.07160200	-1.22061300	-0.67692700
H	-2.94060700	-1.57854900	-0.89455900
O	-1.04255600	0.70462700	0.31353000
C	0.27616800	0.32256100	0.16175000
C	0.70978000	-0.98210500	0.40737600
C	1.18534500	1.32081300	-0.19257600
C	2.06825000	-1.28189600	0.28355200
H	-0.00593000	-1.74924800	0.67572500
C	2.54036900	1.00906700	-0.31135000
H	0.81935400	2.32682800	-0.36851400
C	2.98800700	-0.29347600	-0.07506100
H	2.40595800	-2.29678600	0.47075400
H	3.24548100	1.78683400	-0.58859300
H	4.04198100	-0.53517700	-0.16800300
O	-3.39132900	0.53212200	0.21748900
H	-3.33407000	1.38919300	0.65668400

Path B: **A**

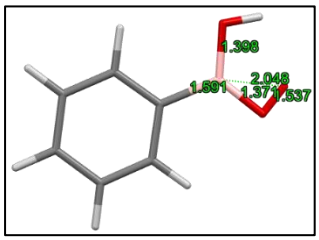


Thermal correction to Gibbs Free Energy ($G_{corr}(T)$) = 0.080288 Hartrees
Electronic energy (ε_{ele}) = -482.830706209 Hartrees

Sum of electronic and thermal Free Energies (G(T)) = -482.750418209 Hartrees
Temperature = 298.15 K, Pressure = 1 Atm.

Atom	x	y	z
B	1.33597000	0.20507400	-0.00002800
O	1.97538700	1.41011900	0.00003200
H	2.92419700	1.00136400	0.00006200
O	2.10385600	-0.92348300	0.00001400
C	-0.23667000	0.06872000	-0.00001200
C	-1.05924300	1.20997100	-0.00002300
C	-0.87117400	-1.18772300	0.00000700
C	-2.45353400	1.10646700	-0.00001500
H	-0.58441200	2.18749700	-0.00004000
C	-2.26318500	-1.30233600	0.00001400
H	-0.25005300	-2.07937400	0.00001400
C	-3.06228000	-0.15272000	0.00000400
H	-3.06674200	2.00539600	-0.00002400
H	-2.72868800	-2.28580200	0.00002700
H	-4.14663700	-0.23827100	0.00001000
O	3.52688100	-0.49544200	-0.00001500

Path B: **TS1**

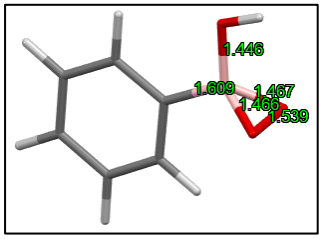


Thermal correction to Gibbs Free Energy ($G_{corr}(T)$) = 0.080326 Hartrees
Electronic energy (ε_{ele}) = -482.807829429 Hartrees
Sum of electronic and thermal Free Energies (G(T)) = -482.727503429 Hartrees
Temperature = 298.15 K, Pressure = 1 Atm.

Atom	x	y	z
B	-1.42245700	0.20974200	-0.19183500
O	-1.93551500	1.50948100	-0.14596500
H	-2.87807800	1.33413500	0.03175100
O	-2.15160600	-0.90098900	-0.52607100
C	0.16122500	0.09062400	-0.07959300
C	0.99564700	1.21859100	0.01859600
C	0.77550200	-1.17379900	-0.07995100
C	2.38526300	1.09415100	0.10626000
H	0.53400100	2.20227300	0.02798200
C	2.16384100	-1.31149900	0.01092500

H	0.13399900	-2.04809900	-0.14806300
C	2.97470000	-0.17514700	0.10288400
H	3.01051300	1.98195800	0.18002400
H	2.61581400	-2.30142600	0.01243200
H	4.05558100	-0.27719900	0.17430000
O	-3.04995600	-0.65822700	0.69778700

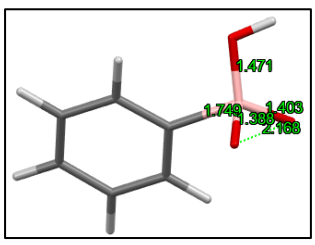
Path B: **B**



Thermal correction to Gibbs Free Energy ($G_{corr}(T)$) = 0.080306 Hartrees
Electronic energy (ε_{ele}) = -482.849240921 Hartrees
Sum of electronic and thermal Free Energies (G(T)) = -482.768934921 Hartrees
Temperature = 298.15 K, Pressure = 1 Atm.

Atom	x	y	z
B	-1.54781500	0.15816500	-0.00020700
O	-2.02959300	1.52130400	-0.00053500
H	-2.99276900	1.47039500	0.00143100
O	-2.27871000	-0.85445900	-0.76915500
C	0.05927100	0.08787100	-0.00007400
C	0.87763700	1.23040100	0.00004400
C	0.70861500	-1.16083200	-0.00020100
C	2.27530700	1.13885500	0.00017200
H	0.39797700	2.20580600	0.00023200
C	2.10139000	-1.26834700	-0.00009700
H	0.08989000	-2.05449600	-0.00032200
C	2.89557900	-0.11402000	0.00012900
H	2.88112500	2.04391500	0.00030700
H	2.57333200	-2.24976700	-0.00014500
H	3.98107900	-0.19216600	0.00023800
O	-2.27899000	-0.85410500	0.76962200

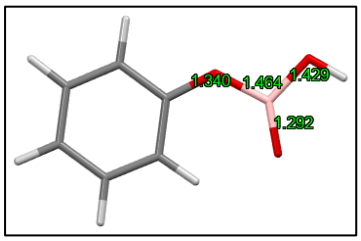
Path B: **TS2**



Thermal correction to Gibbs Free Energy ($G_{corr}(T)$) = 0.076322 Hartrees
Electronic energy (ε_{ele}) = -482.742070253 Hartrees
Sum of electronic and thermal Free Energies (G(T)) = -482.665748253 Hartrees
Temperature = 298.15 K, Pressure = 1 Atm.

Atom	x	y	z
B	1.72018000	-0.00620400	-0.00783700
O	2.08521200	1.27929300	-0.62476800
H	2.95473300	1.14370500	-1.01932800
O	1.71088800	-0.22916300	1.36164200
C	-0.02765000	0.01362400	0.03513400
C	-0.76368700	1.20030000	0.15968700
C	-0.71246300	-1.20485700	-0.09274200
C	-2.15973700	1.18152600	0.07122500
H	-0.23016000	2.13793800	0.28552600
C	-2.10546200	-1.22529900	-0.19353600
H	-0.13191400	-2.12114100	-0.14681100
C	-2.83550500	-0.03138300	-0.10875400
H	-2.72299200	2.11062800	0.14380700
H	-2.62751800	-2.17092600	-0.33096000
H	-3.92124600	-0.04832800	-0.17419400
O	2.41705200	-1.12817100	-0.47999200

Path B: **C**



Thermal correction to Gibbs Free Energy ($G_{corr}(T)$) = 0.082227 Hartrees
Electronic energy (ε_{ele}) = -482.977629552 Hartrees
Sum of electronic and thermal Free Energies (G(T)) = -482.895402552 Hartrees
Temperature = 298.15 K, Pressure = 1 Atm.

Atom	x	y	z
B	-2.21944000	0.11458500	-0.00001700
O	-3.35024000	-0.75919600	0.00009000
H	-4.10829500	-0.16311200	-0.00014900
O	-1.04215800	-0.75574700	0.00000500
C	0.23330400	-0.34261600	0.00001300
C	1.22802400	-1.34288200	-0.00001900
C	0.63436400	1.01119600	0.00005700
C	2.58129000	-1.00637000	-0.00003000

H	0.90629700	-2.38041800	-0.00010200
C	1.99341000	1.32870300	0.00003200
H	-0.15322100	1.75843600	0.00009600
C	2.98072200	0.33533400	-0.00000800
H	3.32855300	-1.79769300	-0.00008900
H	2.28648100	2.37716500	0.00010700
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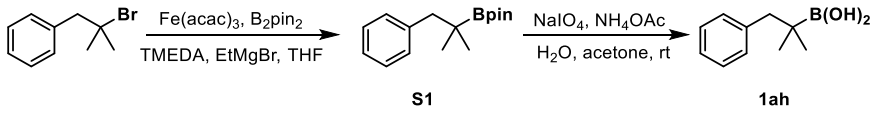
O	-2.24567900	1.40655000	-0.00010400

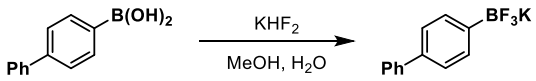
IV. Procedures for starting materials



S1 **1ah**

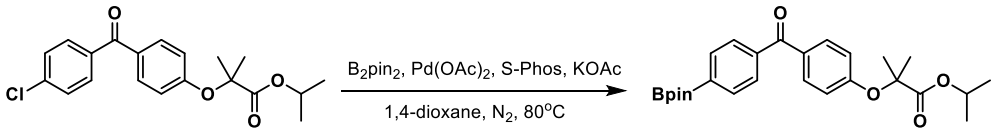
4,4,5,5-Tetramethyl-2-(2-methyl-1-phenylpropan-2-yl)-1,3,2-dioxaborolane (**S1**): $Fe(acac)_3$ (2.0 mmol, 706 mg) and bis(pinacolato)diboron (35.0 mmol, 8.9 g) were added to a 100 mL round-bottomed flask. The vessel was evacuated and filled with nitrogen gas three times. THF (50 mL) was added, followed by TMEDA (8 mL of a 4% (v/v) solution of TMEDA (0.32 mL) in THF, 2.0 mmol). Next, EtMgBr (2 M in THF, 22.5 mL, 45.0 mmol) was added dropwise to the reaction mixture at a rate of one drop/sec. Finally, (2-bromo-2-methylpropyl) benzene (10.0 mmol, 2.13g) was added to the reaction mixture at once. The reaction mixture was stirred at room temperature for 12 h. The mixture was filtered (washed with Et_2O) and concentrated under reduced pressure. The resulting crude mixture was chromatographed on silica gel using petroleum ether as eluent to give **S1** as a colorless liquid.

(2-Methyl-1-phenylpropanoid-2-yl) boronic acid (**1ah**): To a solution of **S1** (1.5 mmol, 1 g) in acetone (20 mL) and water (10 mL) were added NH_4OAc (9.0 mmol, 693 g) and $NaIO_4$ (9.0 mmol, 1.9 g). The resulting reaction mixture was stirred at room temperature for 48 h. The reaction mixture was diluted with Et_2O, and filtered through a pad of elite. The filtrate was concentrated under reduced pressure to give **1ah** as a colorless liquid. **^{1}H NMR** (400 MHz, CD_3Cl) δ 7.33-7.20 (m, 5H), 2.76 (s, 2H), 1.22 (s, 6H). **^{13}C NMR** (100 MHz, CD_3Cl) δ 137.7, 130.4, 128.1, 126.4, 70.7, 49.7, 29.1. **IR** (neat) 3321, 1458, 1419, 1257, 1111, 852, 754 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 178.



1a 1ao

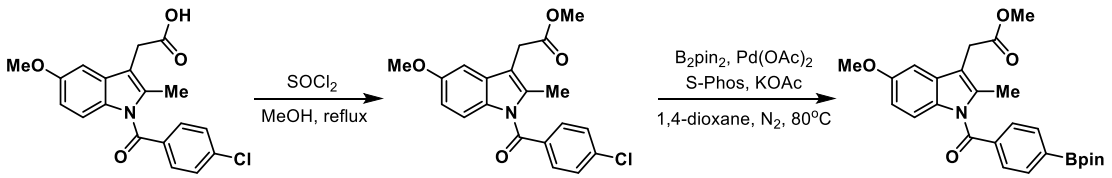
Potassium [1,1'-biphenyl]-4-yltrifluoroborate (**1ao**): KHF_2 (40 mmol, 3.12 g) in H_2O (10 mL) was added to a stirring solution of [1,1'-biphenyl]-4-ylboronic acid **1a** (10 mmol, 1.98 g) dissolved in MeOH (45 mL) in one portion. A white precipitate usually formed rapidly, and the reaction mixture was stirred at room temperature overnight. After the reaction by TLC, all solvents were removed in vacuo and the solid residue was then dissolved in refluxing acetone and filtered over a pad of celite. This process was usually repeated three times and the combined filtrates were then concentrated under reduced pressure to give **1ao** as a white solid. **^{1}H NMR** (400 MHz, DMSO-d_6) δ 7.50 (d, J = 6.8 Hz, 2H), 7.43-7.37 (m, 6H), 7.29 (t, J = 7.2 Hz, 1H). **^{13}C NMR** (100 MHz, DMSO-d_6) δ 141.5, 136.7, 131.92, 131.90, 128.7, 126.4, 126.3, 124.6. **^{19}F NMR** (376 MHz, DMSO-d_6) δ -139.1. **IR** (neat) 3446, 1628, 1506, 1265, 1082, 745 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 260.



Fenofibrate 1au

Isopropyl 2-methyl-2-(4-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl) benzoyl) phenoxy) propanoate (**1au**): A mixture of Fenofibrate (1 mmol, 361 mg), bis(pinacolato)diboron (B_2pin_2, 3 mmol, 762 mg), $Pd(OAc)_2$ (4 mol%, 9 mg), 2-dicyclohexylphosphino-2',6'-dimethoxybiphenyl (S-Phos, 10 mol%, 41 mg), KOAc (3 mol, 295 mg) was stirred in 1,4-dioxane (4 mL) under N_2 atmosphere at 80°C. After the reaction by TLC, the reaction mixture was diluted with H_2O and extracted with ethyl acetate (×3). The combined organics were washed with brine, dried over Na_2SO_4, and concentrated under reduced pressure. The resulting crude mixture was chromatographed on silica gel using petroleum ether/ethyl acetate (PE/EA = 10/1) as eluent to give **1au** as a white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.90 (d, J = 8.4 Hz,

2H), 7.74 (d, *J* = 8.8 Hz, 2H), 7.71 (d, *J* = 8.0 Hz, 2H), 6.85 (d, *J* = 8.8 Hz, 2H), 5.11-5.05 (m, 1H), 1.66 (s, 6H), 1.37 (s, 12H), 1.20 (d, *J* = 6.4 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 195.7, 173.1, 159.6, 140.4, 134.5, 132.1, 130.6, 128.7, 117.2, 84.2, 79.4, 69.3, 25.4, 24.9, 21.5. **IR** (neat) 3734, 3055, 2986, 1734, 1647, 1506, 1361 cm^{-1}. **MS** (ESI) Calculated for $C_{26}H_{34}BO_6$ $(M+H)^+$: 453.2; Found 453.4.

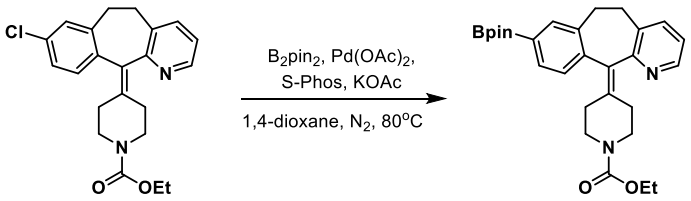


Indometacin **S2** **1av**

Methyl 2-(1-(4-chlorobenzene)-5-methoxy-2-methyl-1H-indol-3-yl)acetate (**S2**): $SOCl_2$ (7.7 mmol, 1.5 mL) was dropwise added to a solution of Indometacin (4 mmol, 1.43 g) and MeOH (10 mL) at 0℃. Subsequently, the temperature is from 0℃ to 80℃. After the reaction by TLC, the reaction mixture was concentrated under reduced pressure to give **S2** as a white solid.

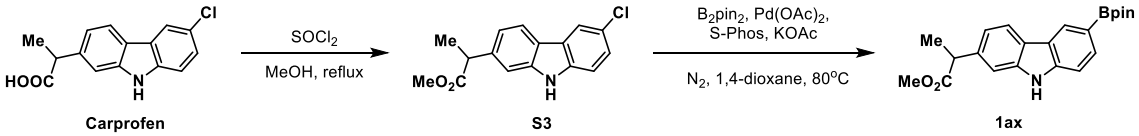
Methyl 2-(5-methoxy-2-methyl-1-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzoyl)-1H-indol-3-yl)acetate (**1av**): A mixture of **S2** (1 mmol, 372 mg), bis(pinacolato)diboron (B_2pin_2, 3 mmol, 762 mg), $Pd(OAc)_2$ (4 mol%, 9 mg), 2-dicyclohexylphosphino-2',6'-dimethoxybiphenyl (S-Phos, 10 mol%, 41 mg), KOAc (3 mol, 295 mg) was stirred in 1, 4-dioxane (4 mL) under N_2 atmosphere at 80℃. After the reaction by TLC, the reaction mixture was diluted with H_2O and extracted with ethyl acetate (×3). The combined organics were washed with brine, dried over Na_2SO_4, and concentrated under reduced pressure. The resulting crude mixture was chromatographed on silica gel using petroleum ether/ethyl acetate (PE/EA = 10/1) as eluent to give **1av** as a white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.4 Hz, 2H), 7.68 (d, *J* = 8.0 Hz, 2H), 7.95 (d, *J* = 2.4 Hz, 1H), 6.88 (d, *J* = 9.2 Hz, 1H), 6.64 (dd, *J* = 8.8, 2.4 Hz, 1H), 3.83 (s, 3H), 3.70 (s, 3H), 3.66 (s, 2H), 2.36 (s, 3H), 1.38 (s, 12H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 171.4, 169.5, 156.0, 137.9, 136.1, 134.9, 130.9, 130.6, 128.6, 115.2, 112.3, 111.5, 101.2, 84.3, 55.7, 52.1, 30.2, 24.9, 13.4. **IR** (neat)

3055, 2986, 1717, 1684, 1362, 1144, 856 cm^{-1}. **MS** (ESI) Calculated for $C_{26}H_{31}BNO_6$ $(M+H)^+$: 464.2; Found 464.4.



Loratadine **1aw**

Ethyl 4-(8-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-5,6-dihydro-11H-benzo[5,6]cyclohepta[1,2-b]pyridin-11-ylidene)piperidine-1-carboxylate (**1aw**): A mixture of Loratadine (1 mmol, 383 mg), bis(pinacolato)diboron (B_2pin_2, 3 mmol, 762 mg), $Pd(OAc)_2$ (4 mol%, 9 mg), 2-dicyclohexylphosphino-2',6'-dimethoxybiphenyl (S-Phos, 10 mol%, 41 mg), KOAc (3 mol, 295 mg) was stirred in 1,4-dioxane (4 mL) under N_2 atmosphere at 80°C. After the reaction by TLC, the reaction mixture was diluted with H_2O and extracted with ethyl acetate (×3). The combined organics were washed with brine, dried over Na_2SO_4, and concentrated under reduced pressure. The resulting crude mixture was chromatographed on silica gel using petroleum ether/ethyl acetate (PE/EA = 1/1) as eluent to give **1aw** as a white solid. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.38 (d, *J* = 3.0 Hz, 1H), 7.63-7.61 (m, 2H), 7.43 (d, *J* = 9.5 Hz, 1H), 7.21 (d, *J* = 7.5 Hz, 1H), 7.08 (dd, *J* = 7.8, 4.8 Hz, 1H), 4.13 (q, *J* = 7.0 Hz, 2H), 3.83-3.77 (m, 2H), 3.48-3.31 (m, 2H), 3.16-3.09 (m, 2H), 2.89-2.82 (m, 2H), 2.52-2.29 (m, 4H), 1.32 (s, 12H), 1.24 (t, *J* = 7.0 Hz, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.9, 155.5, 146.3, 142.4, 137.7, 137.0, 135.3, 135.1, 133.8, 132.5, 128.6, 122.2, 83.8, 61.3, 44.82, 44.80, 31.8, 31.5, 30.7, 30.5, 24.83, 24.77, 14.7. **IR** (neat) 3649, 2986, 2909, 1740, 1373, 1240, 1045 cm^{-1}. **MS** (ESI) Calculated for $C_{28}H_{36}BN_2O_4$ $(M+H)^+$: 475.3; Found 475.4.

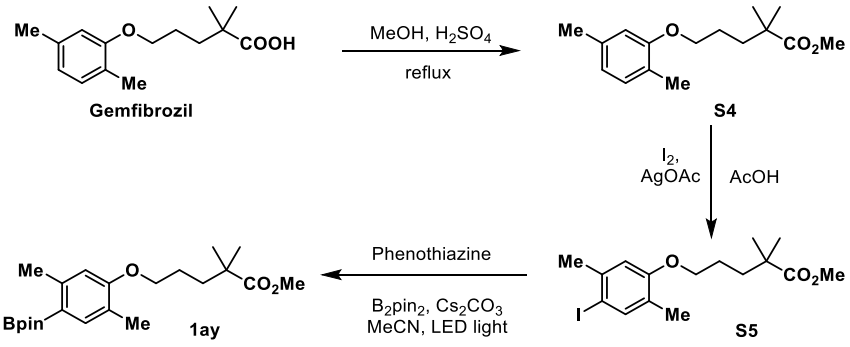


Carprofen **S3** **1ax**

Methyl 2-(6-chloro-9H-carbazol-2-yl)propanoate (**S3**): $SOCl_2$ (7.7 mmol, 1.5 mL) was added to a solution of Carprofen (4 mmol, 1.09 g) and MeOH (10 mL) at 0 °C.

Subsequently, the temperature is from 0℃ to 80℃. After the reaction by TLC, the reaction mixture was concentrated under reduced pressure to give **S3** as a yellow solid.

Methyl 2-(6-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-9H-carbazol-2-yl)propanoate (**1ax**)**:** A mixture of **S3** (1 mmol, 383 mg), bis(pinacolato)diboron (B_2pin_2, 3 mmol, 762 mg), $Pd(OAc)_2$ (4 mol%, 9 mg), 2-dicyclohexylphosphino-2',6'-dimethoxybiphenyl (S-Phos, 10 mol%, 41 mg), KOAc (3 mol, 295 mg) was stirred in 1,4-dioxane (4 mL) under N_2 atmosphere at 80℃. After the reaction by TLC, the reaction mixture was diluted with H_2O and extracted with ethyl acetate (×3). The combined organics were washed with brine, dried over Na_2SO_4, and concentrated under reduced pressure. The resulting crude mixture was chromatographed on silica gel using petroleum ether/ethyl acetate (PE/EA = 10/1) as eluent to give **1ax** as a white solid. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.54 (s, 1H), 8.20 (s, 1H), 8.03 (d, *J* = 8.0 Hz, 1H), 7.86 (d, *J* = 8.5 Hz, 1H), 7.39 (d, *J* = 8.0 Hz, 1H), 3.51 (s, 1H), 7.17 (d, *J* = 8.0 Hz, 1H), 3.68 (s, 3H), 1.58 (d, *J* = 7.5 Hz, 3H), 1.40 (s, 12H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 175.3, 142.0, 139.7, 138.5, 132.2, 127.6, 122.8, 122.6, 120.6, 119.6, 110.0, 109.3, 83.6, 52.1, 45.8, 24.9, 19.0. **IR** (neat) 3055, 2986, 1734, 1717, 1541, 1361, 858 cm^{-1}. **MS** (ESI) Calculated for $C_{22}H_{27}BNO_4$ $(M+H)^+$: 380.2; Found 380.3.

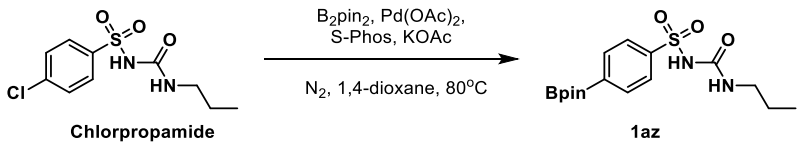


Methyl 5-(2,5-dimethylphenoxy)-2,2-dimethylpentanoate (**S4**): Gemfibrozil (5 mmol, 1.25 g) was dissolved in 50 mL of dry methanol. 6 drops of concentrated sulfuric acid were added to the mixture and the reaction was allowed to stir at reflux overnight. The reaction mixture was cooled to room temperature and quenched with $NaHCO_3$ (aq.) until a basic pH was reached. The organic layer was extracted using ethyl acetate (3 ×

50 mL) and the organic layer was washed with brine (50 mL) and dried using $MgSO_4$. The organic layer was concentrated to give **S4** as a colorless oil.

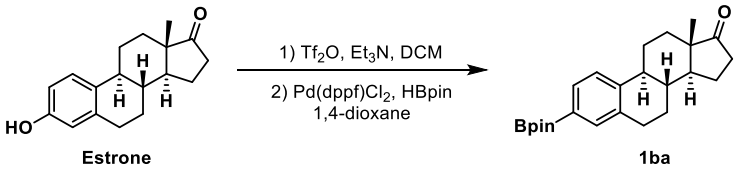
Methyl 5-(4-iodo-2,5-dimethylphenoxy)-2,2-dimethylpentanoate (**S5**): Methyl 5-(2,5-dimethylphenoxy)-2,2-dimethylpentanoate **S4** (5 mmol, 1.32 g) was added to a mixture of iodine (5 mmol, 1.27 g) and silver acetate (5 mmol, 0.83 g) in acetic acid (15 mL) at room temperature. The reaction mixture was stirred at room temperature overnight. The reaction mixture was quenched with 0.1 M NaOH and tpartitionedtion between diethyl ether and H_2O. The combined organic layers were washed with brine, dried, filtered, and concentrated. The resulting crude mixture was chromatographed on silica gel using petroleum ether as eluent to give **S5** as a brown liquid.

Methyl 5-(2,5-dimethyl-4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenoxy)-2,2-dimethylpentanoate (**1ay**): A mixture of **S5** (1 mmol, 390 mg), B_2pin_2 (2 mmol, 505 mg), Cs_2CO_3 (2 mmol, 660 mg), a phenothiazine (0.02 mmol, 4 mg) and CH_3CN (10 mL) was irradiated with a 400 nm LED light for 48 h. The reaction mixture was concentrated under reduced pressure and the resulting crude mixture was chromatographed on silica gel using petroleum ether/ethyl acetate (PE/EA = 20/1) as eluent to give **1ay** as a colorless liquid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.53 (s, 1H), 6.59 (s, 1H), 3.94 (t, J = 5.6 Hz, 2H), 3.66 (s, 3H), 2.50 (s, 3H), 2.17 (s, 3H), 1.72-1.71 (m, 4H), 1.32 (s, 12H), 1.22 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 178.3, 159.2, 144.7, 138.3, 122.8, 112.5, 83.0, 67.7, 51.7, 42.1, 37.0, 25.2, 25.1, 24.9, 22.2, 15.4. **IR** (neat) 2986, 1740, 1647, 1506, 1373, 1240, 1047 cm^{-1}. **MS** (ESI) Calculated for $C_{23}H_{37}BO_7$ $(M+HCOOH)^+$: 436.3; Found 436.4.



N-(propyl carbamoyl)-4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzenesulfonamide (**1az**): A mixture of Chlorpropamide (1 mmol, 277 mg), bis(pinacolato)diboron (B_2pin_2, 3 mmol, 762 mg), $Pd(OAc)_2$ (4 mol%, 9 mg), 2-dicyclohexylphosphino-2',6'-dimethoxybiphenyl (S-Phos, 10 mol%, 41 mg), KOAc (3

mol, 295 mg) was stirred in 1,4-dioxane (4 mL) under N_2 atmosphere at 80℃. After the reaction by TLC, the reaction mixture was diluted with H_2O and extracted with ethyl acetate (×3). The combined organics were washed with brine, dried over Na_2SO_4, and concentrated under reduced pressure. The resulting crude mixture was chromatographed on silica gel using petroleum ether/ethyl acetate (PE/EA = 3/1) as eluent to give **1az** as a yellow solid. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.23 (s, 1H), 7.96 (d, *J* = 7.0 Hz, 2H), 7.87 (d, *J* = 6.5 Hz, 2H), 6.58 (t, *J* = 6.0 Hz, 1H), 3.17 (dd, *J* = 13.0, 6.0 Hz, 2H), 1.54-1.46 (m, 2H), 1.35 (s, 12H), 0.88 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 151.2, 141.6, 135.6, 125.7, 84.5, 42.1, 24.8, 22.7, 11.2. **IR** (neat) 3340, 2986, 1734, 1541, 1362, 1267, 745 cm^{-1}. **MS** (ESI) Calculated for $C_{16}H_{26}BN_2O_5S$ $(M+H)^+$: 369.2; Found 369.3.

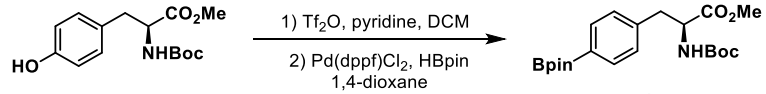


Estrone 1ba

3-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl) extra-1,3,5 (10)-trien-17-one (**1ba**): To a solution of Estrone (3.7 mmol, 1 g) and Et_3N (7.4 mmol, 1.03 mL) in DCM (20 mL) was added Tf_2O (4.1 mmol, 684 μL) at 0℃. The reaction mixture was stirred at room temperature. After the reaction by TLC, saturated aqueous $NaHCO_3$ was added. The phases were separated and the aqueous phase was extracted with DCM, washed with brine, dried over Na_2SO_4, and concentrated. The resulting crude mixture was chromatographed on silica gel using petroleum ether/ethyl acetate (PE/EA = 10/1) as eluent to give 3-(trifluoromethanesulfonyl)estrone as a white solid.

A mixture of 3-(trifluoromethanesulfonyl)estrone (1 mmol, 402 mg), pinacolborane (HBpin, 1.5 mmol, 0.22 mL), $Pd(dppf)Cl_2$ (3 mol%, 22 mg), Et_3N (0.3 mol, 0.42 mL) was stirred in 1,4-dioxane (5 mL) under N_2 atmosphere at 110℃. After the reaction by TLC, the reaction mixture was diluted with H_2O and extracted with DCM (×3). The combined organics were washed with brine, dried over Na_2SO_4, and concentrated under reduced pressure. The resulting crude mixture was

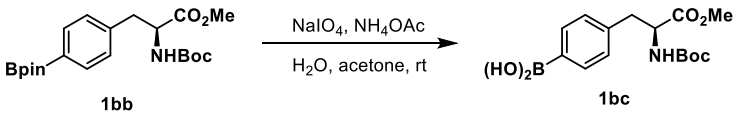
chromatographed on silica gel using petroleum ether/ethyl acetate (PE/EA = 10/1) as eluent to give **1ba** as a white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.60 (d, *J* = 8.0 Hz, 1H), 7.57 (s, 1H), 7.32 (d, *J* = 8.0 Hz, 1H), 2.95-2.91 (m, 2H), 2.54-2.43 (m, 2H), 2.33 (td, *J* = 10.8, 4.0 Hz, 1H), 2.19-1.95 (m, 4H), 1.66-1.44 (m, 6H), 1.34 (s, 12H), 0.91 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 220.8, 143.1, 135.8, 135.6, 132.2, 124.8, 83.7, 50.6, 48.0, 44.7, 38.0, 35.8, 31.6, 29.1, 26.5, 25.6, 24.83, 24.79, 21.6, 13.8. **IR** (neat) 3055, 2986, 1734, 1506, 1265, 1049, 745 cm^{-1}. **MS** (ESI) Calculated for $C_{24}H_{34}BO_3$ $(M+H)^+$: 381.3; Found 381.4.



N-Boc-L-tyrosine methyl ester 1bb

Methyl (S)-2-((tert-butoxycarbonyl)amino)-3-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)propanoa (**1bb**): To a solution of N-Boc-L-tyrosine methyl ester (16.94 mmol, 5 g) and pyridine (84.7 mmol, 6.8 mL) in DCM (50 mL) was added Tf_2O (19.48 mmol, 3.5 mL) at 0℃. The reaction mixture was stirred at the same temperature for 30 min. The reaction was diluted with DCM (100 mL), washed with water (50 mL), NaOH (0.5 M), brine, dried over Na_2SO_4, and concentrated. The resulting crude mixture was chromatographed on silica gel using petroleum ether/ethyl acetate (PE/EA = 5/1) as eluent to give trifluoromethanesulfonic ester as a white solid. A mixture of trifluoromethanesulfonic ester (10 mmol, 4.27 g), pinacolborane (HBpin, 15 mmol, 2.2 mL), $Pd(dppf)Cl_2$ (3 mol%, 220 mg), Et_3N (3 mol, 4.2 mL) was stirred in 1,4-dioxane (50 mL) under N_2 atmosphere at 110℃. After the reaction by TLC, the reaction mixture was diluted with H_2O and extracted with DCM (×3). The combined organics were washed with brine, dried over Na_2SO_4, and concentrated under reduced pressure. The resulting crude mixture was chromatographed on silica gel using petroleum ether/ethyl acetate (PE/EA = 5/1) as eluent to give **1bb** as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 7.6 Hz, 2H), 7.12 (d, *J* = 7.6 Hz, 2H), 4.97-4.95 (m, 1H), 4.60-4.54 (m, 1H), 3.69 (s, 3H), 3.11-3.07 (m, 2H), 1.41 (s, 9H), 1.33 (s, 12H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 172.2, 155.0, 139.2, 135.0, 129.2, 128.7, 83.7,

79.9, 54.3, 52.1, 38.4, 28.2, 24.8. **IR** (neat) 3447, 3366, 2980, 1744, 1356, 1146, 1090 cm^{-1}. **MS** (ESI) Calculated for $C_{21}H_{33}BNO_6$ $(M+H)^+$: 406.2; Found 406.3.



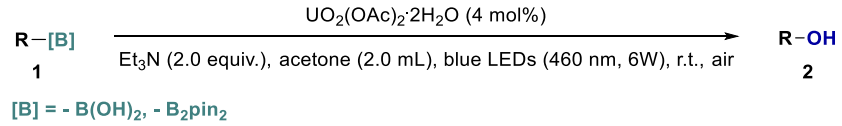
(S)-(4-(2-((Tert-butoxycarbonyl)amino)-3-methoxy-3-oxopropyl)phenyl)boronic acid (**1bc**): To a solution of **1bb** (2.46 mmol, 1 g) in acetone (30 mL) and water (15 mL) was added NH_4OAc (14.8 mmol, 1.14 g) and $NaIO_4$ (14.8 mmol, 3.17 g). The resulting reaction mixture was stirred at room temperature for 48 h. The reaction mixture was diluted with Et_2O, and filtered through a pad of celite. The filtrate was concentrated under reduced pressure to give **1bc** as a white solid. **^{1}H NMR** (400 MHz, DMSO-d_6) δ 7.93 (s, 2H), 7.69 (d, J = 7.6 Hz, 2H), 7.27 (d, J = 8.0 Hz, 1H), 7.18 (d, J = 7.6 Hz, 2H), 4.20-4.14 (m, 1H), 3.60 (s, 3H), 3.01-2.82 (m, 2H), 1.32 (s, 9H). **^{13}C NMR** (100 MHz, DMSO-d_6) δ 172.6, 155.3, 139.4, 134.0, 128.1, 78.2, 55.1, 51.7, 36.5, 28.1. **IR** (neat) 3055, 2986, 1734, 1647, 1506, 1362, 1267 cm^{-1}. **MS** (ESI) Calculated for $C_{15}H_{23}BNO_6$ $(M+H)^+$: 324.2; Found 324.3.

V. General procedures

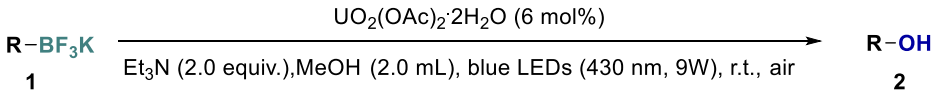
A. Paralleled photoreactor

Figure S8: Paralleled photoreactor for research and development.

B. General procedures for phenols and alcohols.

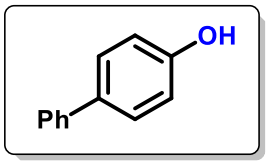


To a 25 mL Schlenk tube, Et_3N (0.4 mmol, 56 μL) was added to the mixture of **1** (0.2 mmol), $UO_2(OAc)_2·2H_2O$ (4 mol%, 3.4 mg), and acetone (2 mL). Subsequently, the reaction was irradiated by blue light (460 nm) under air atmosphere at room temperature in the paralleled reactor. The reaction mixture was quenched with ethyl acetate (EA) and then concentrated under reduced pressure. Purify the crude residue by column chromatography on silica gel using PE/EA to afford **2**. For detailed modification, please see the corresponding procedure.

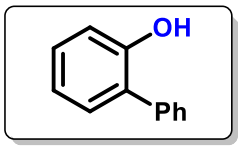


To a 25 mL Schlenk tube, Et_3N (0.4 mmol, 56 μL) was added to the mixture of **1** (0.2 mmol), $UO_2(OAc)_2·2H_2O$ (6 mol%, 5.1 mg), and MeOH (2 mL). Subsequently, the reaction was irradiated by blue light (430 nm) under air atmosphere at room temperature in the paralleled reactor. The reaction mixture was quenched with ethyl acetate (EA) and then concentrated under reduced pressure. Purify the crude residue by column chromatography on silica gel using PE/EA to afford **2**. For detailed modification, please see the corresponding procedure.

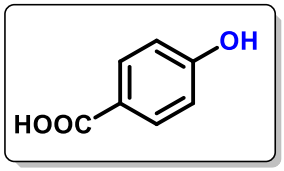
VI. Procedures and data



4-Phenylphenol (2a)[3]: 4-Phenylbenzeneboronic acid **1a** (0.2 mmol, 39.6 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 20 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2a** (30.6 mg, 90%) was obtained through column chromatography (PE/EA = 5/1) as a white solid, R_f = 0.5 (PE/EA = 3/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.54 (d, *J* = 7.6 Hz, 2H), 7.49 (d, *J* = 8.4 Hz, 2H), 7.42 (t, *J* = 7.6 Hz, 2H), 7.31 (t, *J* = 7.4 Hz, 1H), 6.91 (d, *J* = 8.4 Hz, 2H), 4.81 (s, 1H).**^{13}C NMR** (100 MHz, $CDCl_3$) δ 155.1, 140.8, 134.1, 128.7, 128.4, 126.7, 126.7, 115.6. **IR** (film) 3321, 1458, 1419, 1257, 1111, 852, 754 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 170.

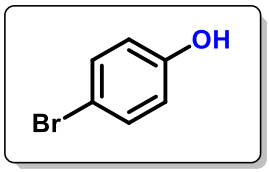


2-Phenylphenol (2b)[4]: 2-Phenylbenzeneboronic acid **1b** (0.2 mmol, 39.6 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 20 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2b** (32.3 mg, 95%) was obtained through column chromatography (PE/EA = 5/1) as a white solid, R_f = 0.5 (PE/EA = 5/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.52-7.47 (m, 4H), 7.43-7.38 (m, 1H), 7.29-7.24 (m, 2H), 7.02-6.98 (m, 2H), 5.21 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 152.4, 137.1, 130.2, 129.3, 129.2, 129.1, 128.1, 127.9, 120.8, 115.8. **IR** (film) 3535, 3062, 1583, 1479, 1435, 1182, 795 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 170.



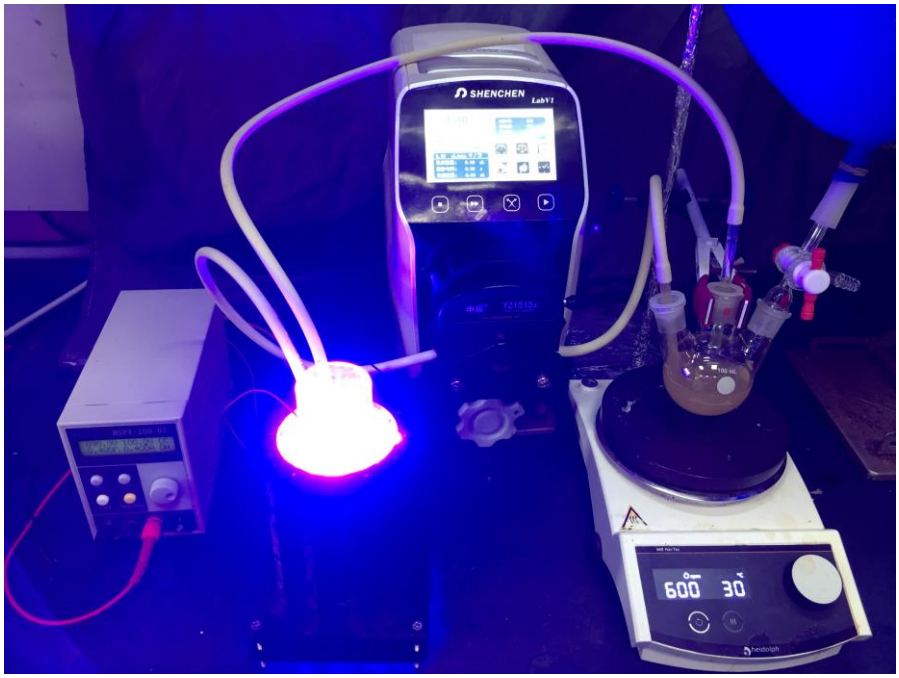
4-Hydroxybenzoic acid (2c)[5]: 4-Boronobenzoic acid **1c** (0.2 mmol, 33.2 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 20 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2c** (26.2 mg, 95%) was obtained through column chromatography (PE/EA = 1/1) as a white solid, R_f = 0.4

(PE/EA = 1/1); **^{1}H NMR** (400 MHz, DMSO-d_6) δ 12.39 (s, 1H), 10.21 (s, 1H), 7.79 (d, J = 8.4 Hz, 2H), 6.82 (d, J = 8.4 Hz, 2H),.**^{13}C NMR** (100 MHz, DMSO-d_6) δ 167.2, 161.6, 131.6, 121.4, 115.1. **IR** (film) 3408, 1651, 1595, 1338, 1024, 991, 825 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 138.



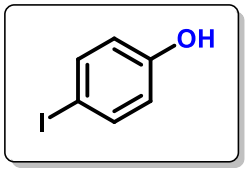
4-Bromophenol (2d)[4]: 4-Bromophenylboronic acid **1d** (0.2 mmol, 40.2 mg), $UO_2(OAc)_2{\cdot}2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 16 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2d** (31.0 mg, 89%) was obtained through column chromatography (PE/EA = 5/1) as a brown liquid, R_f = 0.4 (PE/EA = 5/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.33 (d, J = 8.8 Hz, 2H), 6.73 (d, J = 8.8 Hz, 2H), 5.53 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 154.6, 132.5, 117.2, 112.9. **IR** (film) 3414, 1587, 1495, 1433, 1244, 1070, 823 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 173.

Flow Photoreactor

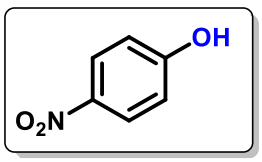


To a 100 mL bottle, a solution of 4-bromophenylboronic acid **1d** (20 mmol, 4.02 g), $UO_2(OAc)_2{\cdot}2H_2O$ (0.4 mmol, 170 mg), Et_3N (2 equiv., 5.6 mL) and acetone (80 mL)

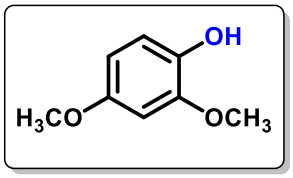
were stirred under air atmosphere at room temperature. The solution was pumped into a flow micro tube by a pump (0.5 mL/min), which was made of PTFE tubing (O.D. = 2 mm, I.D. = 1 mm, length = 5.68 m, volume = 4.45 mL), and returned to Schlenk tube with the same pump. This circulatory system was irradiated by blue light (460 nm, 36 W) for about 22 hours (the temperature was below 30°C). After the reaction, DCM was pumped into a flow micro tube to quench it and MeOH was added to wash the tube. The solvent was concentrated under reduced pressure and the resulting crude mixture was chromatographed on silica gel using petroleum ether/ethyl acetate (PE/EA = 5/1) as eluent to give **2d** (78%, 2.71 g) as a brown liquid.



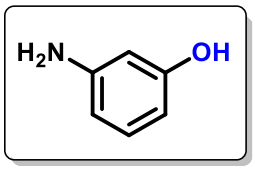
4-Iodophenol (2e)[3]: 4-Iodophenylboronic acid **1e** (0.2 mmol, 49.6 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 22 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2e** (39.2 mg, 89%) was obtained through column chromatography (PE/EA = 5/1) as a brown liquid, R_f = 0.6 (PE/EA = 3/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.51 (d, J = 8.8 Hz, 2H), 6.63 (d, J = 8.8 Hz, 2H), 5.26 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 155.4, 138.4, 117.8, 82.6. **IR** (film) 3346, 3017, 1580, 1485, 1217, 1005, 735 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 220.



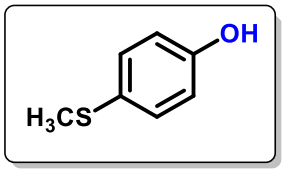
4-Nitrophenol (2f)[4]: 4-Nitrophenylboronic acid **1f** (0.2 mmol, 33.4 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 16 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2f** (26.7 mg, 96%) was obtained through column chromatography (PE/EA = 3/1) as a brown solid, R_f = 0.4 (PE/EA = 3/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.16 (d, J = 9.2 Hz, 2H), 6.94 (d, J = 8.8 Hz, 2H), 6.94 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 161.9, 141.3, 126.3, 115.8. **IR** (film) 3294, 1593, 1498, 1338, 1290, 1111, 850 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 139.



2,4-Dimethoxyphenol (2g)[6]: 2,4-Dimethoxyphenylboronic acid **1g** (0.2 mmol, 36.4 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 19 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2g** (29.0 mg, 94%) was obtained through column chromatography (PE/EA = 5/1) as a white solid, R_f = 0.6 (PE/EA = 3/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.83 (d, *J* = 8.8 Hz, 1H), 6.50 (d, *J* = 2.8 Hz, 1H), 6.39 (dd, *J* = 8.8, 2.8 Hz, 1H), 5.26 (s, 1H), 3.86 (s, 3H), 3.76 (s, 3H).**^{13}C NMR** (100 MHz, $CDCl_3$) δ 153.5, 147.0, 139.8, 114.1, 104.2, 99.4, 55.9, 55.8. **IR** (film) 3456, 2941, 1610, 1512, 1232, 1207, 1029 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 154.

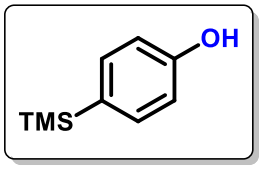


3-Aminophenol (2h)[7]: 3-Aminophenylboronic acid **1h** (0.2 mmol, 27.4 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 20 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2h** (17.4 mg, 80%) was obtained through column chromatography (PE/EA = 3/1) as a brown solid, R_f = 0.4 (PE/EA = 3/1); **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.81 (s, 1H), 6.76 (t, *J* = 8.0 Hz, 1H), 6.00-5.98 (m, 2H), 5.93-5.91 (m, 1H), 4.87 (s, 1H). **^{13}C NMR** (100 MHz, DMSO-d_6) δ 158.1, 149.8, 129.4, 105.5, 103.3, 101.0. **IR** (film) 3607, 3391, 1651, 1269, 1047, 1024, 991 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 109.

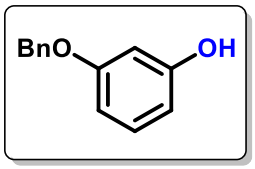


4-(Methylthio)phenol (2i)[3]: 4-(Methylthio)phenylboronic acid **1i** (0.2 mmol, 33.6 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 20 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2i** (26.6 mg, 95%) was obtained through column chromatography (PE/EA = 5/1) as a white solid,

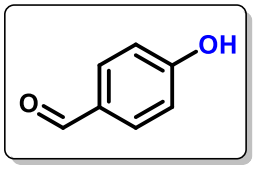
R_f = 0.5 (PE/EA = 5/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.22 (d, *J* = 8.4 Hz, 2H), 6.79 (d, *J* = 8.8 Hz, 2H), 5.13 (s, 1H), 2.44 (s, 3H).**^{13}C NMR** (100 MHz, $CDCl_3$) δ 154.1, 130.4, 128.8, 116.1, 18.0. **IR** (film) 3498, 3053, 1601, 1495, 1265, 825, 704 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 140.



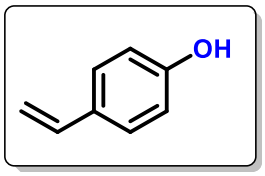
4-(Trimethylsilyl)phenol (2j)[8]: 4-(Trimethylsilyl)phenylboronic acid **1j** (0.2 mmol, 38.8 mg), $UO_2(OAc)_2{\cdot}2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 20 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2j** (31.8 mg, 95%) was obtained through column chromatography (PE/EA = 5/1) as a white solid, R_f = 0.6 (PE/EA = 5/1); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.44 (d, *J* = 8.4 Hz, 2H), 6.88 (d, *J* = 8.4 Hz, 2H), 5.49 (s, 1H), 0.27 (s, 9H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.1, 134.9, 131.6, 114.9, -0.9. **IR** (film) 3354, 3055, 2956, 1597, 1502, 1265, 839 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 166.



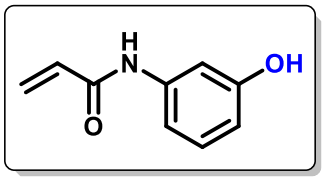
3-(Benzyloxy)phenol (2k)[9]: 3-(Benzyloxy)phenylboronic acid **1k** (0.2 mmol, 45.6 mg), $UO_2(OAc)_2{\cdot}2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 20 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2k** (38.8 mg, 97%) was obtained through column chromatography (PE/EA = 5/1) as a white solid, R_f = 0.5 (PE/EA = 5/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.45-7.34 (m, 5H), 7.15 (t, *J* = 8.0 Hz, 1H), 6.60 (dd, *J* = 8.4, 2.4 Hz, 1H), 6.51 (t, *J* = 2.4 Hz, 1H), 6.46 (dd, *J* = 8.0, 2.4 Hz, 1H), 5.03 (s, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 160.1, 156.7, 136.8, 130.2, 128.6, 128.0, 127.5, 108.1, 107.3, 102.5, 70.0. **IR** (film) 3408, 1593, 1489, 1282, 1148, 1026, 736 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 200.



4-Hydroxybenzaldehyde (2l)[4]**:** 4-Formylphenylboronic acid **1l** (0.2 mmol, 30.0 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 24 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2l** (23.2 mg, 95%) was obtained through column chromatography (PE/EA = 1/1) as a white solid, R_f = 0.5 (PE/EA = 1/1); **^{1}H NMR** (400 MHz, DMSO-d_6) δ 10.57 (s, 1H), 9.79 (s, 1H), 7.76 (d, J = 8.8 Hz, 2H), 6.93 (d, J = 8.4 Hz, 2H). **^{13}C NMR** (100 MHz, DMSO-d_6) δ 191.0, 163.3, 132.1, 128.5, 115.9. **IR** (film) 3448, 3055, 2927, 1647, 1265, 1026, 745 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 122.

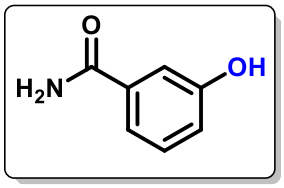


4-Vinylphenol (2m)[10]**:** 4-Vinylphenylboronic acid **1m** (0.2 mmol, 29.6 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 20 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2m** (12.1 mg, 50%) was obtained through column chromatography (PE/EA = 5/1) as a white solid, R_f = 0.5 (PE/EA = 5/1); **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.50 (s, 1H), 7.28 (d, J = 8.8 Hz, 2H), 6.73 (d, J = 8.8 Hz, 2H), 6.61 (dd, J = 17.6, 10.8 Hz, 1H), 5.57 (dd, J = 17.6, 1.2 Hz, 1H), 5.03 (dd, J = 11.2, 1.2 Hz, 1H). **^{13}C NMR** (100 MHz, DMSO-d_6) δ 157.3, 136.4, 128.2, 127.4, 115.3, 110.6. **IR** (film) 3448, 2357, 1653, 1558, 1065, 1006, 825 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 120.

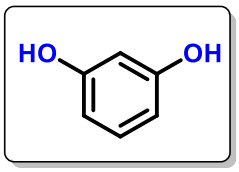


N-(3-hydroxyphenyl)acrylamide (2n)[11]**:** 3-Acrylamidophenylboronic acid **1n** (0.2 mmol, 38.2 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 18 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2n** (25.0 mg, 77%) was obtained through column

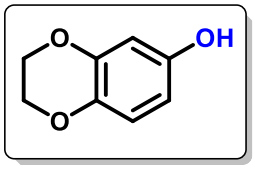
chromatography (PE/EA = 3/1) as a white solid, R_f = 0.4 (PE/EA = 3/1); **^{1}H NMR** (400 MHz, DMSO-d_6) δ 10.0 (s, 1H), 9.42 (s, 1H), 7.26-7.25 (m, 1H),7.10-7.01(m, 2H), 6.48-6.39 (m, 2H), 6.23 (dd, J = 16.8, 2.0 Hz, 1H), 5.72 (dd, J = 10.0, 2.0 Hz, 1H). **^{13}C NMR** (100 MHz, DMSO-d_6) δ 163.1, 157.6, 140.1, 132.0, 129.4, 126.7, 110.7, 110.2, 106.6. **IR** (film) 3419, 2260, 1653, 1558, 1047, 993, 765 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 163.



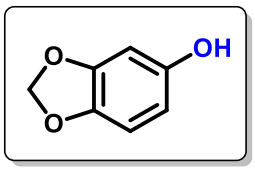
3-Hydroxybenzamide (2o)[12]**:** 3-Carbamoylphenylboronic acid **1o** (0.2 mmol, 33.0 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 40 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2o** (17.1 mg, 62%) was obtained through column chromatography (CH_2Cl_2/MeOH = 10/1) as a yellow solid, R_f = 0.4 (CH_2Cl_2/MeOH = 10/1); **^{1}H NMR** (500 MHz, DMSO-d_6) δ 9.60 (s, 1H), 7.85 (s, 1H), 7.29-7.20 (m, 4H), 6.90-6.88 (m, 1H). **^{13}C NMR** (125 MHz, DMSO-d_6) δ 168.0, 157.3, 135.8, 129.2, 118.1, 118.0, 114.5. **IR** (film) 3543, 3373, 1653, 1506, 1288, 1024, 993 cm^{-1}. **MS** (ESI) Calculated for $C_7H_8NO_2$ $(M+H)^+$: 138.1; Found 138.1.



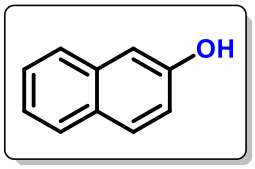
Resorcinol (2p)[13]**:** 1,3-Phenylenediboronic acid **1p** (0.2 mmol, 33.2 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 30 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2p** (18.8 mg, 68%) was obtained through column chromatography (PE/EA = 3/1) as a white solid, R_f = 0.5 (PE/EA = 3/1); **^{1}H NMR** (500 MHz, DMSO-d_6) δ 9.16 (s, 2H), 6.91 (t, J = 8.3 Hz, 1H), 6.19-6.16 (m, 3H). **^{13}C NMR** (125 MHz, DMSO-d_6) δ 158.5, 129.8, 106.2, 102.5. **IR** (film) 3408, 1653, 1506, 1265, 1026, 825, 680 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 110.



2,3-Dihydro-1,4-benzodioxin-6-ol (2q)[14]**:** (1,4-Benzodioxan-6-yl)boronic acid **1q** (0.2 mmol, 36.0 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 16 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2q** (28.9 mg, 95%) was obtained through column chromatography (PE/EA = 5/1) as a yellow solid, R_f = 0.5 (PE/EA = 5/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.72 (d, *J* = 8.4 Hz, 1H), 6.39 (d, *J* = 2.8 Hz, 1H), 6.33 (dd, *J* = 8.4, 2.8 Hz, 1H), 4.68 (s, 1H), 4.25-4.18 (m, 4H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 149.9, 143.9, 137.7, 117.5, 108.2, 104.3, 64.6, 64.1. **IR** (film) 3399, 2985, 1608, 1509, 1473, 1312, 1067 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 152.

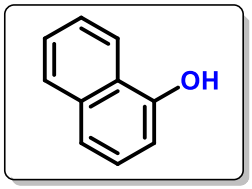


Benzo[*d*][1,3]dioxol-5-ol (2r)[3]**:** Benzo[*d*][1,3]dioxol-5-ylboronic acid **1r** (0.2 mmol, 33.2 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 19 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2r** (21.0 mg, 76%) was obtained through column chromatography (PE/EA = 5/1) as a colorless liquid, R_f = 0.5 (PE/EA = 5/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.65 (d, *J* = 8.0 Hz, 1H), 6.43 (d, *J* = 2.8 Hz, 1H), 6.26 (dd, *J* = 8.4, 2.4 Hz, 1H), 5.90 (s, 2H), 4.98 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 150.6, 148.3, 141.6, 108.1, 106.6, 101.2, 98.3. **IR** (film) 3406, 2893, 1647, 1506, 1489, 1188, 1030 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 138.

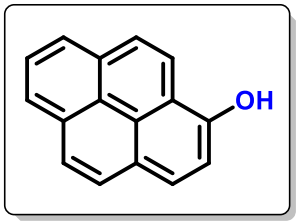


2-Naphthol (2s)[4]**:** 2-Naphthylboronic acid **1s** (0.2 mmol, 34.4 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 18 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2s** (27.0 mg, 94%) was obtained through column chromatography (PE/EA = 5/1) as a white solid, R_f = 0.5 (PE/EA = 5/1); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.80-7.76 (m, 2H), 7.69 (d, *J* = 8.5 Hz, 1H), 7.47-7.43 (m,

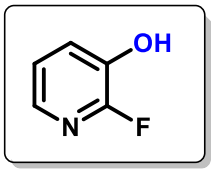
1H), 7.37-7.35 (m, 1H), 7.16 (d, J = 2.5 Hz, 1H), 7.13 (dd, J = 8.5, 2.5 Hz, 1H), 5.40 (s, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 153.2, 134.5, 129.8, 127.7, 126.5, 126.4, 123.6, 117.7, 109.5. **IR** (film) 3275, 3253, 1627, 1508, 1265, 745, 704 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 144.



1-Naphthol (2t)[3]: 1-Naphthylboronic acid **1t** (0.2 mmol, 34.4 mg), $UO_2(OAc)_2{\cdot}2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 16 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2t** (22.0 mg, 76%) was obtained through column chromatography (PE/EA = 5/1) as a white solid, R_f = 0.5 (PE/EA = 5/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.20-8.17 (m, 1H), 7.84-7.81 (m, 1H), 7.51-7.44 (m, 3H), 7.32 (t, J = 7.8 Hz, 1H), 6.83 (d, J = 7.6 Hz, 1H), 5.24 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 151.3, 134.8, 127.7, 126.4, 125.8, 125.3, 124.3, 121.5, 120.7, 108.6. **IR** (film) 3327, 3055, 1599, 1508, 1386, 1269, 781 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 144.



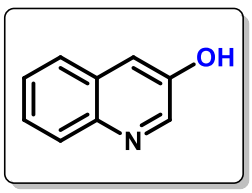
1-Hydroxypyrene (2u)[8]: 1-Pyreneboronic acid **1u** (0.2 mmol, 49.2 mg), $UO_2(OAc)_2{\cdot}2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 16 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2u** (41.8 mg, 96%) was obtained through column chromatography (PE/EA = 3/1) as a white solid, R_f = 0.5 (PE/EA = 3/1); **^{1}H NMR** (300 MHz, DMSO-d_6) δ 10.7 (s, 1H), 8.33 (d, J = 9.3 Hz, 1H), 8.14-8.10 (m, 3H), 8.05-7.88 (m, 4H), 7.60 (d, J = 8.1 Hz, 1H). **^{13}C NMR** (100 MHz, DMSO-d_6) δ 152.2, 131.4, 131.3, 127.4, 126.2, 126.1, 125.5, 125.4, 124.5, 123.9, 123.8, 123.63, 123.61, 121.4, 118.1, 113.3. **IR** (film) 3566, 1508, 1263, 1028, 831, 750, 706 cm^{-1}. **MS** (ESI) Calculated for $C_{16}H_{11}O$ $(M+H)^+$: 219.1; Found 219.1.



2-Fluoro-3-pyridinol (2v)[15]**:** 2-Fluoro-3-pyridineboronic acid **1v** (0.2 mmol, 28.2 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 16 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2v** (22.0 mg, 97%) was obtained through column chromatography (CH_2Cl_2/MeOH = 20/1) as a white solid, R_f = 0.5 (CH_2Cl_2/MeOH = 20/1); **^{1}H NMR** (400 MHz, DMSO-d_6) δ 10.38 (s, 1H), 7.61-7.59 (m, 1H), 7.40-7.35 (m, 1H), 7.16-7.13 (m, 1H), **^{13}C NMR** (100 MHz, DMSO-d_6) δ 152.7 (d, J = 231.7 Hz), 140.2 (d, J = 27.3 Hz), 135.6 (d, J = 13.0 Hz), 126.2 (d, J = 5.5 Hz), 122.6 (d, J = 3.9 Hz). **^{19}F NMR** (376 MHz, DMSO-d_6) δ -88.9. **IR** (film) 3383, 1653, 1558, 1508, 1267, 1026, 994 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 113.

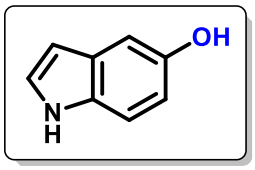
Flow Photoreactor

To a 100 mL bottle, a solution of 2-fluoro-3-pyridineboronic acid **1v** (20 mmol, 2.82 g), $UO_2(OAc)_2 \cdot 2H_2O$ (0.4 mmol, 170 mg), Et_3N (2 equiv., 5.6 mL) and acetone (80 mL) were stirred under air atmosphere at room temperature. The solution was pumped into a flow micro tube by a pump (0.5 mL/min), which was made of PTFE tubing (O.D. = 2 mm, I.D. = 1 mm, length = 5.68 m, volume = 4.45 mL), and returned to Schlenk tube with the same pump. This circulatory system was irradiated by blue light (460 nm, 36 W) for about 18 hours (the temperature was below 30°C). After the reaction, DCM was pumped into a flow micro tube to quench it and MeOH was added to wash the tube. The solvent was concentrated under reduced pressure and the resulting crude mixture was chromatographed on silica gel using petroleum ether/ethyl acetate (CH_2Cl_2/MeOH = 20/1) as eluent to give **2v** (63%, 1.42 g) as a white solid.

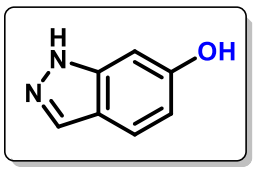


3-Hydroxyquinoline (2w)[8]**:** Quinolin-3-ylboronic acid **1w** (0.2 mmol, 34.6 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 24 h under the irradiation of

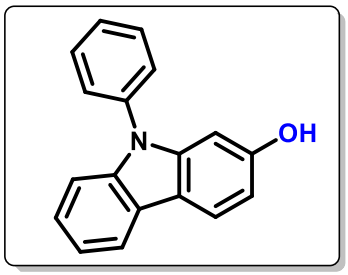
blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2w** (26.0 mg, 90%) was obtained through column chromatography (PE/EA = 3/1) as a white solid, R_f = 0.4 (PE/EA = 3/1); **^{1}H NMR** (400 MHz, DMSO-d_6) δ 10.48 (s, 1H), 8.60 (d, J = 2.8 Hz, 1H), 7.91-7.89 (m, 1H), 7.79-7.76 (m, 1H), 7.51-7.47 (m, 3H), **^{13}C NMR** (125 MHz, DMSO-d_6) δ 150.9, 143.9, 142.4, 129.1, 128.6, 126.8, 126.6, 125.9, 115.4. **IR** (film) 3391, 1653, 1541, 1265, 1026, 995, 747 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 145.



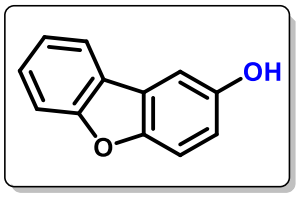
5-Hydroxyindole (2x)[16]**:** 5-Indolylboronic acid **1x** (0.2 mmol, 32.2 mg), $UO_2(OAc)_2{\cdot}2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 40 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2x** (23.0 mg, 86%) was obtained through column chromatography (PE/EA = 5/1) as a white solid, R_f = 0.5 (PE/EA = 5/1); **^{1}H NMR** (500 MHz, DMSO-d_6) δ 10.73 (s, 1H), 8.57 (s, 1H), 7.20 (t, J = 2.8 Hz, 1H), 7.17 (d, J = 8.5 Hz, 1H), 6.84 (d, J = 2.0 Hz, 1H), 6.61-6.59 (m, 1H), 6.22-6.21 (m, 1H). **^{13}C NMR** (125 MHz, DMSO-d_6) δ 150.5, 130.4, 128.4, 125.5, 111.6, 111.3, 103.8, 100.2. **IR** (film) 3417, 1653, 1541, 1265, 1026, 995, 747 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 133.



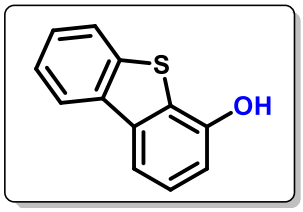
6-Hydroxyindazole (2y)[17]**:** 1H-Indazol-6-ylboronic acid **1y** (0.2 mmol, 32.4 mg), $UO_2(OAc)_2{\cdot}2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 40 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2y** (20.1 mg, 75%) was obtained through column chromatography (PE/EA = 1/1) as a white solid, R_f = 0.4 (PE/EA = 1/1); **^{1}H NMR** (400 MHz, DMSO-d_6) δ 12.55 (s, 1H), 9.53 (s, 1H), 7.86 (s, 1H), 7.52 (d, J = 8.4 Hz, 1H), 6.78 (s, 1H), 6.64 (dd, J = 8.4, 2.0 Hz, 1H). **^{13}C NMR** (100 MHz, DMSO-d_6) δ 156.5, 141.4, 133.3, 121.1, 117.0, 112.3, 93.2. **IR** (film) 3410, 3055, 1653, 1265, 1026, 995, 705 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 134.



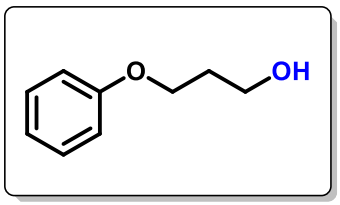
9-Phenyl-9H-carbazol-2-ol (2z)[3]**:** 9-Phenylcarbazole-2-boronic acid **1z** (0.2 mmol, 57.4 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 16 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2z** (46.0 mg, 89%) was obtained through column chromatography (PE/EA = 5/1) as a white solid, R_f = 0.5 (PE/EA = 5/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.01-7.99 (m, 1H), 7.93 (d, *J* = 8.4 Hz, 1H), 7.58-7.49 (m, 4H), 7.44-7.40 (m, 1H), 7.31-7.30 (m, 2H), 7.25-7.21 (m, 1H), 6.80 (d, *J* = 2.0 Hz, 1H), 6.76 (dd, *J* = 8.4, 2.4 Hz, 1H), 4.90 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 154.8, 142.3, 141.1, 137.6, 129.9, 127.5, 127.1, 124.7, 123.5, 121.2, 120.0, 119.4, 117.4, 109.6, 109.0, 96.1. **IR** (film) 3649, 3545, 3053, 2988, 1506, 1265, 745 cm^{-1}. **HRMS** (ESI) Calculated for $C_{18}H_{14}NO$ $(M+H)^+$: 260.1075; Found 260.1065.



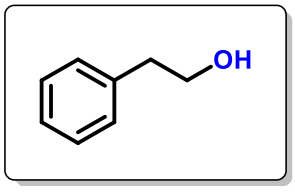
2-Dibenzofuranol (2aa)[18]**:** Dibenzo[b,d]furan-2-ylboronic acid **1aa** (0.2 mmol, 42.4 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 20 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2aa** (33.1 mg, 90%) was obtained through column chromatography (PE/EA = 5/1) as a white solid, R_f = 0.5 (PE/EA = 5/1); **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.44 (s, 1H), 8.04 (d, *J* = 9.2 Hz, 1H), 7.61 (d, *J* = 8.4 Hz, 1H), 7.50-7.43 (m, 3H), 7.35-7.31 (m, 1H), 6.96 (dd, *J* = 8.8, 2.4 Hz, 1H). **^{13}C NMR** (100 MHz, DMSO-d_6) δ 156.1, 153.5., 149.3, 127.3, 124.2, 123.9, 122.6, 121.1, 115.7, 111.9, 111.5, 106.0. **IR** (film) 3401, 3055, 1653, 1265, 1026, 995, 745 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 184.



2-Hydroxydibenzothiophene (2ab)[4]: Dibenzo[b,d]thiophen-4-ylboronic acid **1ab** (0.2 mmol, 45.6 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 17 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2ab** (35.0 mg, 88%) was obtained through column chromatography (PE/EA = 5/1) as a white solid, R_f = 0.6 (PE/EA = 3/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.15-8.13 (m, 1H), 7.90-7.88 (m, 1H), 7.79 (d, *J* = 7.6 Hz, 1H), 7.48-7.46 (m, 2H), 7.35 (t, *J* = 7.8 Hz, 1H), 6.89 (d, *J* = 8.4 Hz, 1H), 5.20 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 150.4, 139.5, 137.9, 135.9, 126.9, 126.5, 125.7, 124.4, 123.1, 122.0, 114.5, 111.7. **IR** (film) 3501, 1647, 1541, 1267, 1028, 750, 705 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 200.



3-Phenoxy-1-propanol (2ac)[19]: 3-Phenoxypropylboronic acid **1ac** (0.2 mmol, 36.0 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 16 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2ac** (29.2 mg, 96%) was obtained through column chromatography (PE/EA = 5/1) as a white solid, R_f = 0.5 (PE/EA = 5/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.25-7.20 (m, 2H), 6.92-6.85 (m, 3H), 4.07 (t, *J* = 6.0 Hz, 2H), 3.81 (t, *J* = 5.8 Hz, 2H), 2.02 (s, 1H), 2.02-1.97 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.7, 129.4, 120.8, 114.4, 65.5, 60.4, 31.9. **IR** (film) 3402, 3055, 2951, 1599, 1497, 1244, 745 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 152.

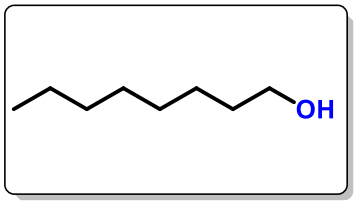


2-Phenylethanol (2ad)[4]: Phenethylboronic acid **1ad** (0.2 mmol, 30.0 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 17 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2ad** (23.9 mg,

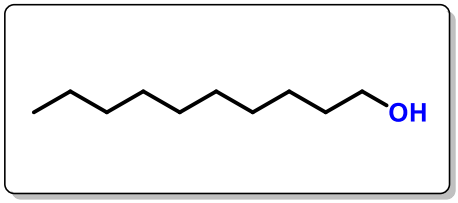
98%) was obtained through column chromatography (PE/EA = 3/1) as a colorless liquid, R_f = 0.4 (PE/EA = 3/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.26-7.22 (m, 2H), 7.18-7.14 (m, 3H), 3.78 (t, *J* = 6.6 Hz, 2H), 2.79 (t, *J* = 6.6 Hz, 2H), 1.53 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 138.5, 129.0, 128.5, 126.4, 63.6, 39.2. **IR** (film) 3420, 3055, 2988, 1647, 1506, 1028, 745 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 122.

Flow Photoreactor

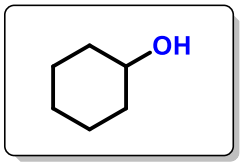
To a 100 mL bottle, a solution of phenethylboronic acid **1ad** (20 mmol, 3.0 g), $UO_2(OAc)_2{\cdot}2H_2O$ (0.4 mmol, 170 mg), Et_3N (2 equiv., 5.6 mL) and acetone (80 mL) were stirred under air atmosphere at room temperature. The solution was pumped into a flow micro tube by a pump (0.5 mL/min), which was made of PTFE tubing (O.D. = 2 mm, I.D. = 1 mm, length = 5.68 m, volume = 4.45 mL), and returned to Schlenk tube with the same pump. This circulatory system was irradiated by blue light (460 nm, 36 W) for about 12 hours (the temperature was below 30°C). After the reaction, DCM was pumped into a flow micro tube to quench it and MeOH was added to wash the tube. The solvent was concentrated under reduced pressure and the resulting crude mixture was chromatographed on silica gel using petroleum ether/ethyl acetate (PE/EA = 3/1) as eluent to give **2ad** (84%, 2.05 g) as a colorless liquid.



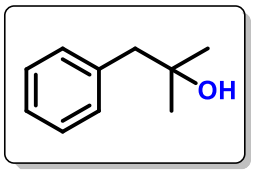
1-Octanol (2ae)[20]**:** Octylboronic acid **1ae** (0.2 mmol, 31.6 mg), $UO_2(OAc)_2{\cdot}2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 12 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2ae** (24.0 mg, 95%) was obtained through column chromatography (PE/EA = 5/1) as a yellow liquid, R_f = 0.5 (PE/EA = 5/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.63 (t, *J* = 6.6 Hz, 2H), 1.59-1.52 (m, 3H), 1.32-1.26 (m, 10H), 0.88 (t, *J* = 6.8 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 63.1, 32.8, 31.8, 29.4, 29.3, 25.7, 22.6, 14.1. **IR** (film) 3566, 3055, 2988, 1508, 1265, 1208, 736 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 130.



1-Decanol (2af)[3]**:** Decylboronic acid **1af** (0.2 mmol, 37.2 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 12 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2af** (28.0 mg, 89%) was obtained through column chromatography (PE/EA = 5/1) as a colorless liquid, R_f = 0.5 (PE/EA = 5/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.62 (t, *J* = 6.6 Hz, 2H), 1.71-1.69 (m, 1H), 1.56-1.53 (m, 2H), 1.35-1.25 (m, 14H), 0.87 (t, *J* = 6.8 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 63.0, 32.8, 31.9, 29.6, 29.5, 29.4, 29.3, 25.7, 22.6, 14.0. **IR** (film) 3331, 2925, 2854, 1466, 1377, 1057, 707 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 158.

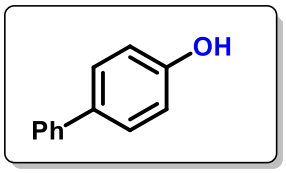


Cyclohexanol (2ag)[21]**:** Cyclohexylboronic acid **1ag** (0.2 mmol, 25.6 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 12 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2ag** (16.1 mg, 80%) was obtained through column chromatography (PE/EA = 5/1) as a yellow liquid, R_f = 0.5 (PE/EA = 5/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.64-3.57 (m, 1H), 1.91-1.86 (m, 2H), 1.74-1.71 (m, 2H), 1.59-1.52 (m, 2H), 1.30-1.21 (m, 5H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 70.3, 35.6, 25.5, 24.1. **IR** (film) 3354, 2925, 2855, 2666, 1452, 1363, 1066 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 100.

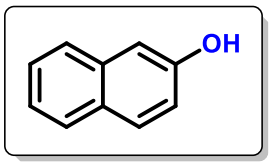


2-Methyl-1-phenyl-2-propanol (2ah)[22]**:** (2-Methyl-1-phenylpropan-2-yl)boronic acid **1ah** (0.2 mmol, 35.6 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 12 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2ah** (16.3 mg, 54%) was obtained through column chromatography

(PE/EA = 5/1) as a colorless oil, R_f = 0.6 (PE/EA =3/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.35-7.22 (m, 5H), 2.79 (s, 2H), 1.25 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 137.8, 130.5, 128.2, 126.5, 70.7, 49.7, 29.2. **IR** (film) 3397, 2961, 2922, 2363, 1153, 727, 702 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 150.



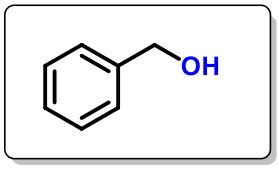
4-Phenylphenol (2ai)[3]: 2-([1,1'-Biphenyl]-4-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane **1ai** (0.2 mmol, 56.0 mg), $UO_2(OAc)_2{\cdot}2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 40 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2ai** (20.4 mg, 60%) was obtained through column chromatography (PE/EA = 5/1) as a white solid, R_f = 0.5 (PE/EA = 3/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.54 (d, *J* = 7.3 Hz, 2H), 7.49 (d, *J* = 8.4 Hz, 2H), 7.42 (t, *J* = 7.6 Hz, 2H), 7.31 (t, *J* = 7.4 Hz, 1H), 6.91 (d, *J* = 8.8 Hz, 2H), 4.83 (s, 1H).**^{13}C NMR** (100 MHz, $CDCl_3$) δ 155.1, 140.8, 134.1, 128.7, 128.4, 126.7, 126.7, 115.6. **IR** (film) 3321, 1458, 1419, 1257, 1111, 852, 754 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 170.



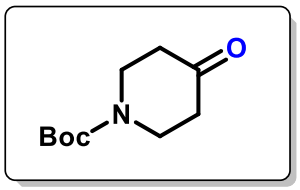
2-Naphthol (2aj)[4]: 4,4,5,5-Tetramethyl-2-(naphthalen-2-yl)-1,3,2-dioxaborolane **1aj** (0.2 mmol, 50.8 mg), $UO_2(OAc)_2{\cdot}2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 22 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2aj** (23.9 mg, 83%) was obtained through column chromatography (PE/EA = 5/1) as a white solid, R_f = 0.5 (PE/EA = 5/1); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.80-7.76 (m, 2H), 7.69 (d, *J* = 8.5 Hz, 1H), 7.47-7.43 (m, 1H), 7.37-7.35 (m, 1H), 7.16 (d, *J* = 2.5 Hz, 1H), 7.13 (dd, *J* = 8.5, 2.5 Hz, 1H), 5.40 (s, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 153.2, 134.5, 129.8, 127.7, 126.5, 126.4, 123.6, 117.7, 109.5. **IR** (film) 3275, 3253, 1627, 1508, 1265, 745, 704 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 144.

Flow Photoreactor

To a 100 mL bottle, a solution of 4,4,5,5-tetramethyl-2-(naphthalen-2-yl)-1,3,2-dioxaborolane **1aj** (10 mmol, 2.5 g), $UO_2(OAc)_2 \cdot 2H_2O$ (0.2 mmol, 85 mg), Et_3N (2 equiv., 2.8 ml) and acetone (50 mL) were stirred under air atmosphere at room temperature. The solution was pumped into a flow micro tube by a pump (0.5 mL/min), which was made of PTFE tubing (O.D. = 2 mm, I.D. = 1 mm, length = 5.68 m, volume = 4.45 mL), and returned to Schlenk tube with the same pump. This circulatory system was irradiated by blue light (460 nm, 36 W) for about 28 hours (the temperature was below 30°C). After the reaction, DCM was pumped into a flow micro tube to quench it and MeOH was added to wash the tube. The solvent was concentrated under reduced pressure and the resulting crude mixture was chromatographed on silica gel using petroleum ether/ethyl acetate (PE/EA = 5/1) as eluent to give **2aj** (85%, 1.22 g) as a white solid.

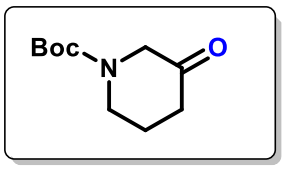


Phenylmethanol (2ak)[4]: 2-Benzyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane **1ak** (0.2 mmol, 43.6 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 12 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2ak** (20.0 mg, 93%) was obtained through column chromatography (PE/EA = 5/1) as a yellow oil, R_f = 0.4 (PE/EA = 5/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.38-7.29 (m, 5H), 4.68 (s, 2H), 1.86 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 140.9, 128.5, 127.6, 127.0, 65.3. **IR** (film) 3566, 2930, 2857, 1456, 1267, 1028, 748 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 108.

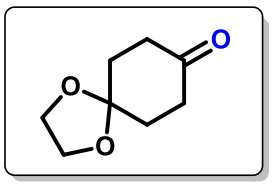


***tert*-Butyl 4-oxopiperidine-1-carboxylate (2al)[23]:** *tert*-Butyl 4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-3,6-dihydropyridine-1(2H)-carboxylate **1al** (0.2 mmol, 61.8 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56

μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 22 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2al** (30.2 mg, 76%) was obtained through column chromatography (PE/EA = 5/1) as a white solid, R_f = 0.6 (PE/EA = 3/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.69 (t, *J* = 6.2 Hz, 4H), 2.41 (t, *J* = 6.2 Hz, 4H), 1.46 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 207.7, 154.4, 80.4, 43.0, 41.1, 28.3. **IR** (film) 2986, 1710, 1697, 1420, 1267, 746, 706 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 199.

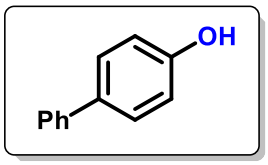


***tert*-Butyl 3-oxopiperidine-1-carboxylate (2am)[24]:** *tert*-Butyl 5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-3,6-dihydropyridine-1(2H)-carboxylate **1am** (0.2 mmol, 61.8 mg), $UO_2(OAc)_2{\cdot}2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 19 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2am** (22.3 mg, 56%) was obtained through column chromatography (PE/EA = 5/1) as a yellow oil, R_f = 0.5 (PE/EA = 3/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.98 (s, 2H), 3.57 (t, *J* = 6.2 Hz, 2H), 2.45 (t, *J* = 6.8 Hz, 2H), 2.00-1.93 (m, 2H), 1.45 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 206.0, 154.5, 80.4, 53.8, 41.9, 38.4, 28.3, 22.3. **IR** (film) 2959, 2926, 2359, 1740, 1736, 1373, 741 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 199.

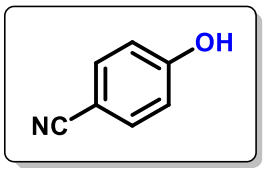


1,4-Dioxaspiro[4.5]decan-8-one (2an)[25]: 4,4,5,5-Tetramethyl-2-(1,4-dioxaspiro[4.5]dec-7-en-8-yl)-1,3,2-dioxaborolane **1an** (0.2 mmol, 53.2 mg), $UO_2(OAc)_2{\cdot}2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 30 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2an** (28.1 mg, 90%) was obtained through column chromatography (PE/EA = 3/1) as a colorless oil, R_f = 0.6 (PE/EA = 1/1); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 4.00 (s, 4H), 2.48 (t, *J* = 7.0 Hz, 4H), 1.98 (t, *J* = 7.3 Hz,

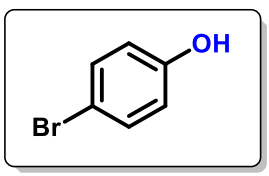
4H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 210.3, 107.1, 64.6, 38.1, 33.8. **IR** (film) 2963, 1717, 1267, 1130, 1090, 741, 704 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 156.



4-Phenylphenol (2ao)[3]: Potassium [1,1'-biphenyl]-4-yltrifluoroborate **1ao** (0.2 mmol, 52.0 mg), $UO_2(OAc)_2{\cdot}2H_2O$ (0.012 mmol, 5.1 mg), Et_3N (2 equiv., 56 μL) and MeOH (2 mL) were stirred under air atmosphere at room temperature for 26 h under the irradiation of blue LEDs (430 nm, 9 W) in a paralleled photoreactor. **2ao** (25.2 mg, 74%) was obtained through column chromatography (PE/EA = 5/1) as a white solid, R_f = 0.5 (PE/EA = 3/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.54 (d, *J* = 7.3 Hz, 2H), 7.49 (d, *J* = 8.4 Hz, 2H), 7.42 (t, *J* = 7.6 Hz, 2H), 7.31 (t, *J* = 7.4 Hz, 1H), 6.91 (d, *J* = 8.8 Hz, 2H), 4.83 (s, 1H).**^{13}C NMR** (100 MHz, $CDCl_3$) δ 155.1, 140.8, 134.1, 128.7, 128.4, 126.7, 126.7, 115.6. **IR** (film) 3321, 1458, 1419, 1257, 1111, 852, 754 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 170.

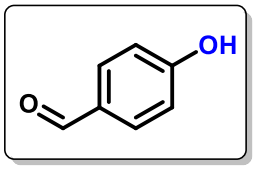


4-Hydroxybenzonitrile (2ap)[4]: Potassium (4-cyanophenyl)trifluoroborate **1ap** (0.2 mmol, 41.8 mg), $UO_2(OAc)_2{\cdot}2H_2O$ (0.012 mmol, 5.1 mg), Et_3N (2 equiv., 56 μL) and MeOH (2 mL) were stirred under air atmosphere at room temperature for 26 h under the irradiation of blue LEDs (430 nm, 9 W) in a paralleled photoreactor. **2ap** (19.0 mg, 80%) was obtained through column chromatography (PE/EA = 5/1) as a yellow solid, R_f = 0.4 (PE/EA = 5/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.55 (d, *J* = 8.8 Hz, 2H), 6.93 (d, *J* = 8.8 Hz, 2H), 6.74 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 160.2, 134.3, 119.2, 116.4, 103.1. **IR** (film) 3283, 2234, 1611, 1508, 1285, 1167, 834 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 119.

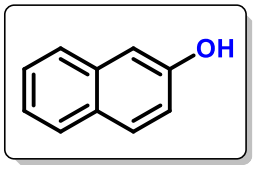


4-Bromophenol (2aq)[4]: Potassium (4-bromophenyl)trifluoroborate **1aq** (0.2 mmol, 52.6 mg), $UO_2(OAc)_2{\cdot}2H_2O$ (0.012 mmol, 5.1 mg), Et_3N (2 equiv., 56 μL)

and MeOH (2 mL) were stirred under air atmosphere at room temperature for 26 h under the irradiation of blue LEDs (430 nm, 9 W) in a paralleled photoreactor. **2aq** (22.1 mg, 64%) was obtained through column chromatography (PE/EA = 5/1) as a brown liquid, R_f = 0.4 (PE/EA = 5/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.33 (d, *J* = 8.8 Hz, 2H), 6.73 (d, *J* = 8.8 Hz, 2H), 5.53 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 154.6, 132.5, 117.2, 112.9. **IR** (film) 3414, 1587, 1495, 1433, 1244, 1070, 823 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 173.

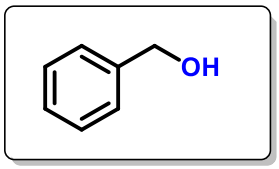


4-Hydroxybenzaldehyde (2ar)[4]: Potassium 4-formylphenyltrifluoroborate **1ar** (0.2 mmol, 42.4 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.012 mmol, 5.1 mg), Et_3N (2 equiv., 56 μL) and MeOH (2 mL) were stirred under air atmosphere at room temperature for 26 h under the irradiation of blue LEDs (430 nm, 9 W) in a paralleled photoreactor. **2ar** (17.3 mg, 71%) was obtained through column chromatography (PE/EA = 1/1) as a white solid, R_f = 0.5 (PE/EA = 1/1); **^{1}H NMR** (400 MHz, DMSO-d_6) δ 10.57 (s, 1H), 9.79 (s, 1H), 7.76 (d, *J* = 8.8 Hz, 2H), 6.93 (d, *J* = 8.4 Hz, 2H). **^{13}C NMR** (100 MHz, DMSO-d_6) δ 191.0, 163.3, 132.1, 128.5, 115.9. **IR** (film) 3448, 3055, 2927, 1647, 1265, 1026, 745 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 122.

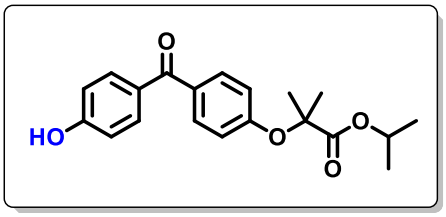


2-Naphthol (2as)[4]: Potassium 2-naphthalenetrifluoroborate **1as** (0.2 mmol, 46.8 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.012 mmol, 5.1 mg), Et_3N (2 equiv., 56 μL) and MeOH (2 mL) were stirred under air atmosphere at room temperature for 26 h under the irradiation of blue LEDs (430 nm, 9 W) in a paralleled photoreactor. **2as** (15.1 mg, 52%) was obtained through column chromatography (PE/EA = 5/1) as a white solid, R_f = 0.5 (PE/EA = 5/1); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.80-7.76 (m, 2H), 7.69 (d, *J* = 8.5 Hz, 1H), 7.47-7.43 (m, 1H), 7.37-7.35 (m, 1H), 7.16 (d, *J* = 2.5 Hz, 1H), 7.13 (dd, *J* = 8.5, 2.5 Hz, 1H), 5.40 (s, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 153.2, 134.5, 129.8, 127.7, 126.5, 126.4, 123.6, 117.7, 109.5. **IR** (film) 3275, 3253, 1627, 1508, 1265, 745, 704 cm^{-1}. GCMS (EI)

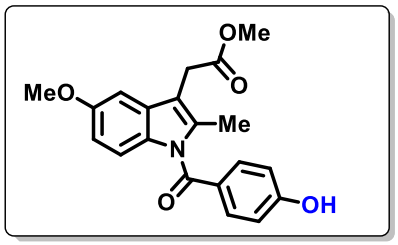
m/z, $[M]^+$ = 144.



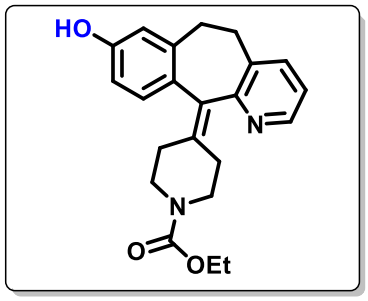
Phenylmethanol (2at)[4]: Potassium benzyltrifluoroborate **1at** (0.2 mmol, 39.6 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.012 mmol, 5.1 mg), Et_3N (2 equiv., 56 μL) and MeOH (2 mL) were stirred under air atmosphere at room temperature for 26 h under the irradiation of blue LEDs (430 nm, 9 W) in a paralleled photoreactor. **2at** (14.9 mg, 69%) was obtained through column chromatography (PE/EA = 5/1) as a yellow oil, R_f = 0.4 (PE/EA = 5/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.38-7.29 (m, 5H), 4.68 (s, 2H), 1.86 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 140.9, 128.5, 127.6, 127.0, 65.3. **IR** (film) 3566, 2930, 2857, 1456, 1267, 1028, 748 cm^{-1}. GCMS (EI) m/z, $[M]^+$ = 108.



Isopropyl 2-(4-(4-hydroxybenzoyl)phenoxy)-2-methylpropanoate (2au)[26]: Isopropyl 2-methyl-2-(4-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzoyl)phenoxy)propanoate **1au** (0.2 mmol, 90.4 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 48 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2au** (53.5 mg, 78%) was obtained through column chromatography (PE/EA = 3/1) as a white solid, R_f = 0.3 (PE/EA = 3/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.80 (s, 1H), 7.71 (dd, J = 8.8, 6.8 Hz, 4H), 6.89 (dd, J = 20.0, 8.8 Hz, 4H), 5.09 (m, 1H), 1.65 (s, 6H), 1.21 (d, J = 6.4 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 195.6, 173.5, 160.7, 159.3, 132.7, 131.9, 131.1, 129.7, 117.3, 115.3, 79.4, 69.5, 25.3, 21.5. **IR** (film) 3264, 1730, 1599, 1560, 1280, 1167, 1099 cm^{-1}. **HRMS** (ESI) Calculated for $C_{20}H_{23}O_5$ $(M+H)^+$: 343.1545; Found 343.1532.

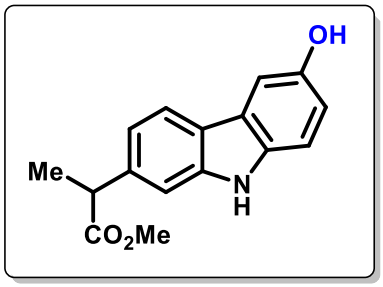


Methyl 2-(1-(4-hydroxybenzoyl)-5-methoxy-2-methyl-1H-indol-3-yl)acetate (2av): Methyl 2-(5-methoxy-2-methyl-1-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzoyl)-1H-indol-3-yl)acetate **1av** (0.2 mmol, 92.6 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 48 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2av** (61.4 mg, 87%) was obtained through column chromatography (PE/EA = 3/1) as a light brown solid, R_f = 0.3 (PE/EA = 3/1); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.62 (d, *J* = 8.7 Hz, 2H), 6.95 (d, *J* = 2.4 Hz, 1H), 6.90-6.84 (m, 3H), 6.65 (dd, *J* = 9.0, 2.7 Hz, 1H), 3.83 (s, 3H), 3.72 (s, 3H), 3.69 (s, 2H), 2.39 (s, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 172.0, 169.1, 160.4, 155.7, 136.2, 132.6, 131.1, 130.3, 127.3, 115.6, 114.8, 111.5, 111.4, 101.0, 55.7, 52.3, 30.2, 13.0. **IR** (film) 3325, 3055, 1717, 1670, 1373, 1265, 745 cm^{-1}. **HRMS** (ESI) Calculated for $C_{20}H_{20}NO_5$ $(M+H)^+$: 354.1341; Found 354.1327.

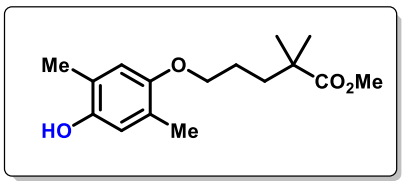


Ethyl 4-(8-hydroxy-5,6-dihydro-11H-benzo[5,6]cyclohepta[1,2-b]pyridin-11-ylidene)piperidine-1-carboxylate (2aw): Ethyl 4-(8-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-5,6-dihydro-11H-benzo[5,6]cyclohepta[1,2-b]pyridin-11-ylidene)piperidine-1-carboxylate **1aw** (0.2 mmol, 94.8 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 48 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2aw** (67.2 mg, 92%) was obtained through column chromatography (PE/EA = 1/1) as a white solid, R_f = 0.3 (PE/EA = 1/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.60 (dd, *J* = 4.8, 1.6 Hz, 1H), 7.75 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.52 (s, 1H), 7.38 (dd, *J* = 7.6, 5.2 Hz, 1H), 7.16 (d, *J* = 8.0 Hz, 1H), 6.93 (d, *J* = 2.4 Hz, 1H), 6.86 (dd, *J* = 8.2, 2.6 Hz, 1H), 4.37 (q, *J* = 7.1 Hz, 2H), 4.06-4.01 (m, 1H), 3.94-3.90

(m, 1H), 3.62-3.55 (m, 2H), 3.43-3.35 (m, 2H), 3.12-2.97 (m, 2H), 2.72-2.46 (m, 4H), 1.48 (t, J = 7.0 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 157.9, 156.8, 155.5, 145.4, 138.7, 138.0, 136.6, 134.4, 133.9, 130.2, 129.6, 122.3, 116.4, 113.3, 77.2, 61.3, 44.6, 44.5, 31.9, 31.4, 30.4, 14.6. **IR** (film) 3690, 3055, 2988, 1697, 1429, 1265, 745 cm^{-1}. **HRMS** (ESI) Calculated for $C_{22}H_{25}N_2O_3$ $(M+H)^+$: 365.1865; Found 365.1850.

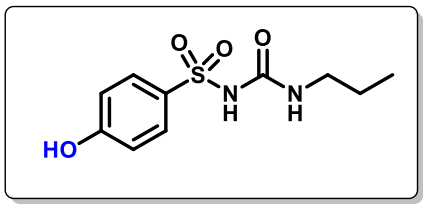


Methyl 2-(6-hydroxy-9H-carbazol-2-yl)propanoate (2ax): Methyl 2-(6-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-9H-carbazol-2-yl)propanoate **1ax** (0.2 mmol, 75.8 mg), $UO_2(OAc)_2{\cdot}2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 42 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2ax** (44.1 mg, 82%) was obtained through column chromatography (PE/EA = 3/1) as a white solid, R_f = 0.3 (PE/EA = 3/1); **^{1}H NMR** (400 MHz, DMSO-d_6) δ 10.84 (s, 1H), 8.93 (s, 1H), 7.90 (d, J = 8.0 Hz, 1H), 7.38 (d, J = 2.4 Hz, 1H), 7.29-7.26 (m, 2H), 6.97 (dd, J = 8.2, 1.4 Hz, 1H), 6.87 (dd, J = 8.8, 2.4 Hz, 1H), 3.91 (q, J = 7.1 Hz, 1H), 3.58 (s, 3H), 1.45 (d, J = 6.8 Hz, 3H). **^{13}C NMR** (100 MHz, DMSO-d_6) δ 174.7, 150.5, 140.7, 137.9, 134.0, 122.9, 121.4, 120.2, 117.4, 114.9, 111.3, 109.4, 104.8, 51.7, 44.9, 19.0. **IR** (film) 3649, 3055, 1717, 1697, 1506, 1265, 745 cm^{-1}. **HRMS** (ESI) Calculated for $C_{16}H_{15}NO_3Na$ $(M+Na)^+$: 292.0950; Found 292.0940.

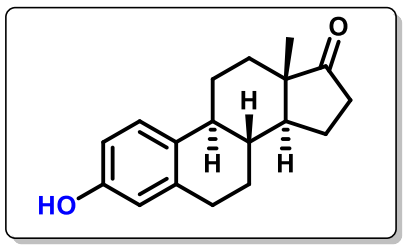


Methyl 5-(4-hydroxy-2,5-dimethylphenoxy)-2,2-dimethylpentanoate (2ay): Methyl 5-(2,5-dimethyl-4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenoxy)-2,2-dimethylpentanoate **1ay** (0.2 mmol, 78.0 mg), $UO_2(OAc)_2{\cdot}2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 48 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2ay** (44.8 mg, 80%)

was obtained through column chromatography (PE/EA = 5/1) as a brown solid, R_f = 0.5 (PE/EA = 3/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.58 (d, *J* = 4.4 Hz, 2H), 4.96 (s, 1H), 3.87-3.84 (m, 2H), 3.67 (s, 3H), 2.20 (s, 3H), 2.14 (s, 3H), 1.71-1.69 (m, 4H), 1.22 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 178.6, 150.9, 147.3, 125.3, 121.2, 117.5, 114.8, 69.1, 51.8, 42.1, 37.1, 25.3, 25.1, 15.73, 15.65. **IR** (film) 3445, 2951, 1730, 1717, 1518, 1198, 1150 cm^{-1}. **HRMS** (ESI) Calculated for $C_{16}H_{25}O_4$ $(M+H)^+$: 281.1753; Found 281.1742.

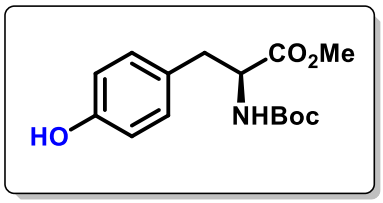


4-Hydroxy-N-(propylcarbamoyl)benzenesulfonamide (2az): N-(propylcarbamoyl)-4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzenesulfonamide **1az** (0.2 mmol, 73.6 mg), $UO_2(OAc)_2{\cdot}2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 24 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2az** (45.4 mg, 88%) was obtained through column chromatography (PE/EA = 1/1) as a yellow solid, R_f = 0.3 (PE/EA = 1/1); **^{1}H NMR** (500 MHz, acetone-d_6) δ 9.45 (s, 1H), 9.38 (s, 1H), 7.85 (d, *J* = 9.0 Hz, 2H), 6.99 (d, *J* = 9.0 Hz, 2H), 6.53 (t, *J* = 5.3 Hz, 1H), 3.10 (dd, *J* = 12.5, 7.0 Hz, 2H), 1.48-1.41 (m, 2H), 0.81 (t, *J* = 7.5 Hz, 3H). **^{13}C NMR** (125 MHz, acetone-d_6) δ 162.6, 152.6, 131.8, 130.7, 116.3, 42.2, 23.5, 11.4. **IR** (film) 3566, 3055, 2986, 1717, 1506, 1265, 745 cm^{-1}. **HRMS** (ESI) Calculated for $C_{10}H_{15}N_2O_4S$ $(M+H)^+$: 259.0753; Found 259.0744.



Estrone (2ba)[27]: 3-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)estra-1,3,5(10)-trien-17-one **1ba** (0.2 mmol, 76.0 mg), $UO_2(OAc)_2{\cdot}2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 24 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled

photoreactor. **2ba** (51.3 mg, 95%) was obtained through column chromatography (PE/EA = 3/1) as a white solid, R_f = 0.4 (PE/EA = 3/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.15 (d, *J* = 8.4 Hz, 1H), 6.64 (dd, *J* = 8.4, 2.8 Hz, 1H), 6.59 (d, *J* = 2.8 Hz, 1H), 4.8 (s, 1H), 2.88-2.85 (m, 2H), 2.51 (dd, *J* = 18.6, 8.6 Hz, 1H), 2.41-2.36 (m, 1H), 2.26-2.21 (m, 1H), 2.17-1.94 (m, 4H), 1.68-1.38 (m, 6H), 0.91 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 221.1, 153.5, 138.0, 132.1, 126.5, 115.3, 112.8, 50.4, 48.0, 44.0, 38.4, 35.9, 31.6, 29.5, 26.5, 25.9, 21.6, 13.9. **IR** (film) 3649, 3055, 2988, 1717, 1506, 1420, 895 cm^{-1}. **HRMS** (ESI) Calculated for $C_{18}H_{23}O_2$ $(M+H)^+$: 271.1698; Found 271.1685.

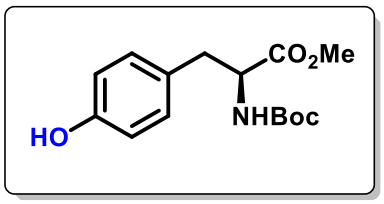


N-Boc-L-tyrosine methyl ester (2bb)[28]**:** Methyl (S)-2-((tert-butoxycarbonyl)amino)-3-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)propanoa **1bb** (0.2 mmol, 81.0 mg), $UO_2(OAc)_2{\cdot}2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 42 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2bb** (53.7 mg, 91%) was obtained through column chromatography (PE/EA = 3/1) as a white solid, R_f = 0.3 (PE/EA = 3/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.96 (d, *J* = 8.4 Hz, 2H), 6.73 (d, *J* = 8.0 Hz, 2H), 6.11 (s, 1H), 5.03 (d, *J* = 8.4 Hz, 1H), 4.56-4.51 (m, 1H), 3.71 (s, 3H), 3.05-2.93 (m, 2H), 1.42 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 172.6, 155.3, 155.1, 130.3, 127.5, 115.5, 80.2, 54.6, 52.2, 37.6, 28.3. **IR** (film) 3398, 2985, 1717, 1684, 1508, 1362, 1265 cm^{-1}. **HRMS** (ESI) Calculated for $C_{15}H_{21}NO_5Na$ $(M+Na)^+$: 318.1317; Found 318.1307.

Flow Photoreactor

To a 100 mL bottle, a solution of methyl (S)-2-((tert-butoxycarbonyl)amino)-3-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)propanoa **1bb** (6.9 mmol, 2.81 g), $UO_2(OAc)_2{\cdot}2H_2O$ (0.14 mmol, 58.5 mg), Et_3N (2 equiv., 2.0 mL) and acetone (50 mL) were stirred under air atmosphere at room temperature. The solution was pumped into a flow micro tube by a pump (0.5 mL/min), which was made of PTFE tubing (O.D. =

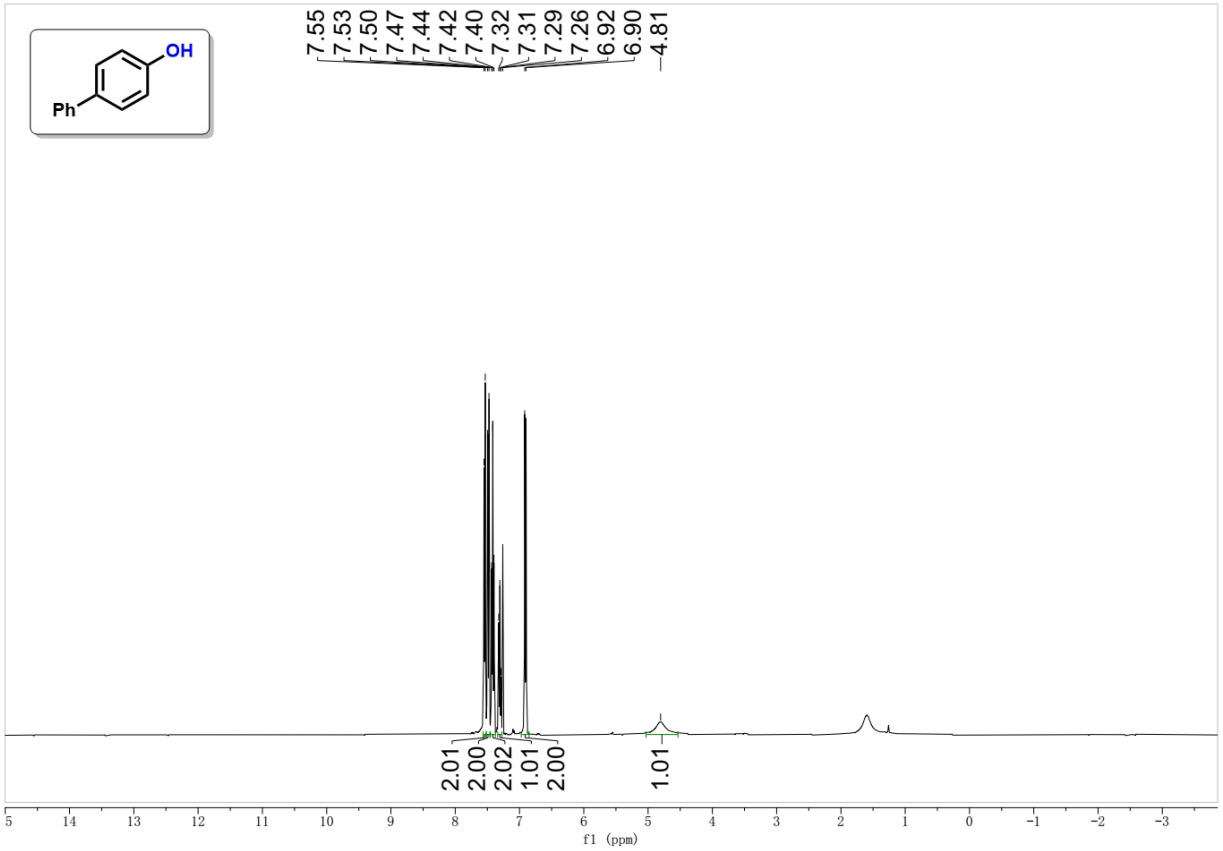
2 mm, I.D. = 1 mm, length = 5.68 m, volume = 4.45 mL), and returned to Schlenk tube with the same pump. This circulatory system was irradiated by blue light (460 nm, 36 W) for about 44 hours (the inner temperature was below 30℃). After the reaction, DCM was pumped into a flow micro tube to quench it and MeOH was added to wash the tube. The solvent was concentrated under reduced pressure and the resulting crude mixture was chromatographed on silica gel using petroleum ether/ethyl acetate (PE/EA = 3/1) as eluent to give **2bb** (66%, 1.34 g) as a white solid.



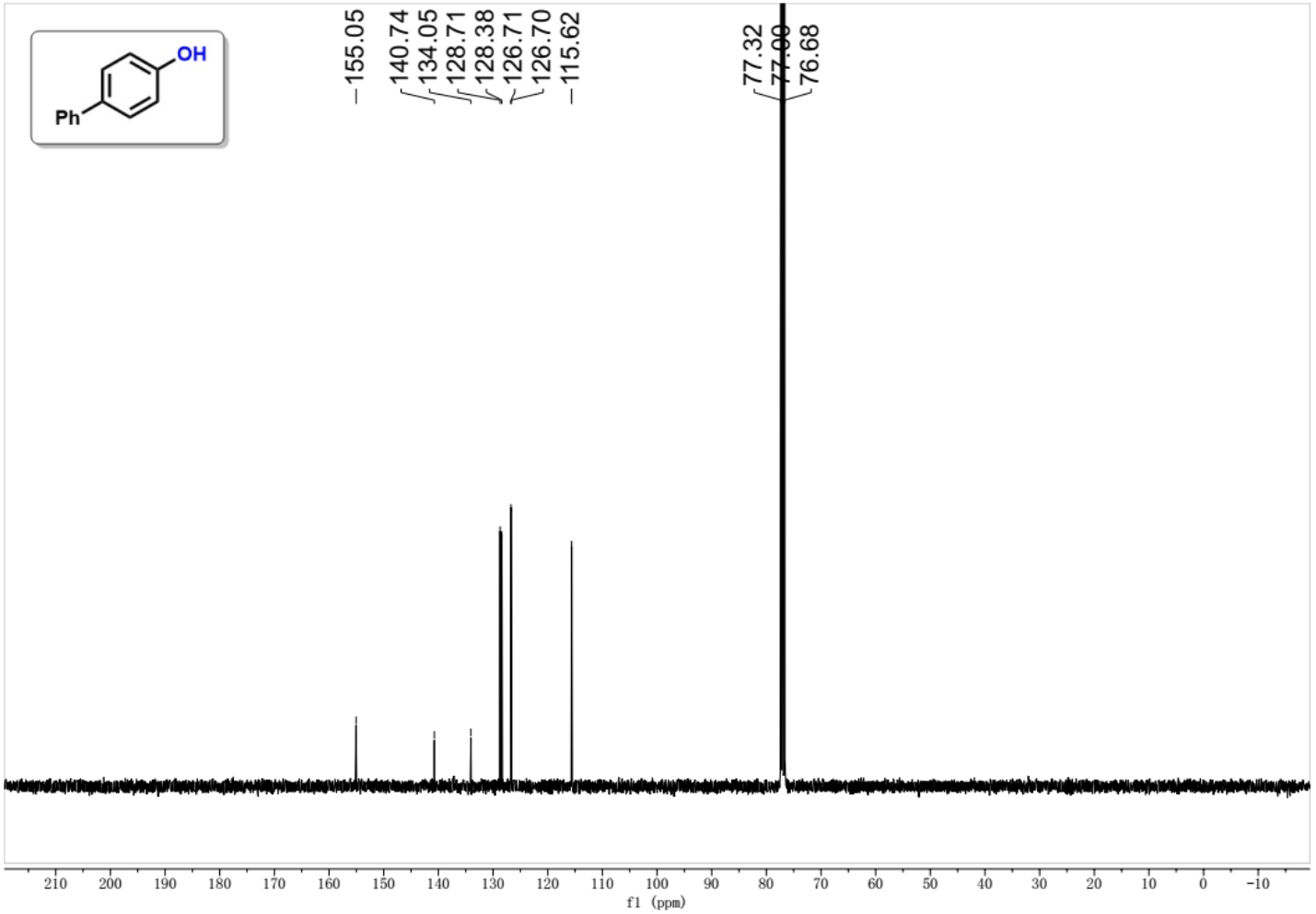
N-Boc-L-tyrosine methyl ester (2bc)[28]**:** (S)-(4-(2-((tert-Butoxycarbonyl)amino)-3-methoxy-3-oxopropyl)phenyl)boronic acid **1bc** (0.2 mmol, 64.6 mg), $UO_2(OAc)_2 \cdot 2H_2O$ (0.008 mmol, 3.4 mg), Et_3N (2 equiv., 56 μL) and acetone (2 mL) were stirred under air atmosphere at room temperature for 20 h under the irradiation of blue LEDs (460 nm, 6 W) in a paralleled photoreactor. **2bc** (53.1 mg, 90%) was obtained through column chromatography (PE/EA = 3/1) as a white solid, R_f = 0.3 (PE/EA = 3/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.96 (d, *J* = 8.4 Hz, 2H), 6.73 (d, *J* = 8.0 Hz, 2H), 6.11 (s, 1H), 5.03 (d, *J* = 8.4 Hz, 1H), 4.56-4.51 (m, 1H), 3.71 (s, 3H), 3.05-2.93 (m, 2H), 1.42 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 172.6, 155.3, 155.1, 130.3, 127.5, 115.5, 80.2, 54.6, 52.2, 37.6, 28.3. **IR** (film) 3398, 2985, 1717, 1684, 1508, 1362, 1265 cm^{-1}. **HRMS** (ESI) Calculated for $C_{15}H_{21}NO_5Na$ $(M+Na)^+$: 318.1317; Found 318.1307.

VII. NMR spectra

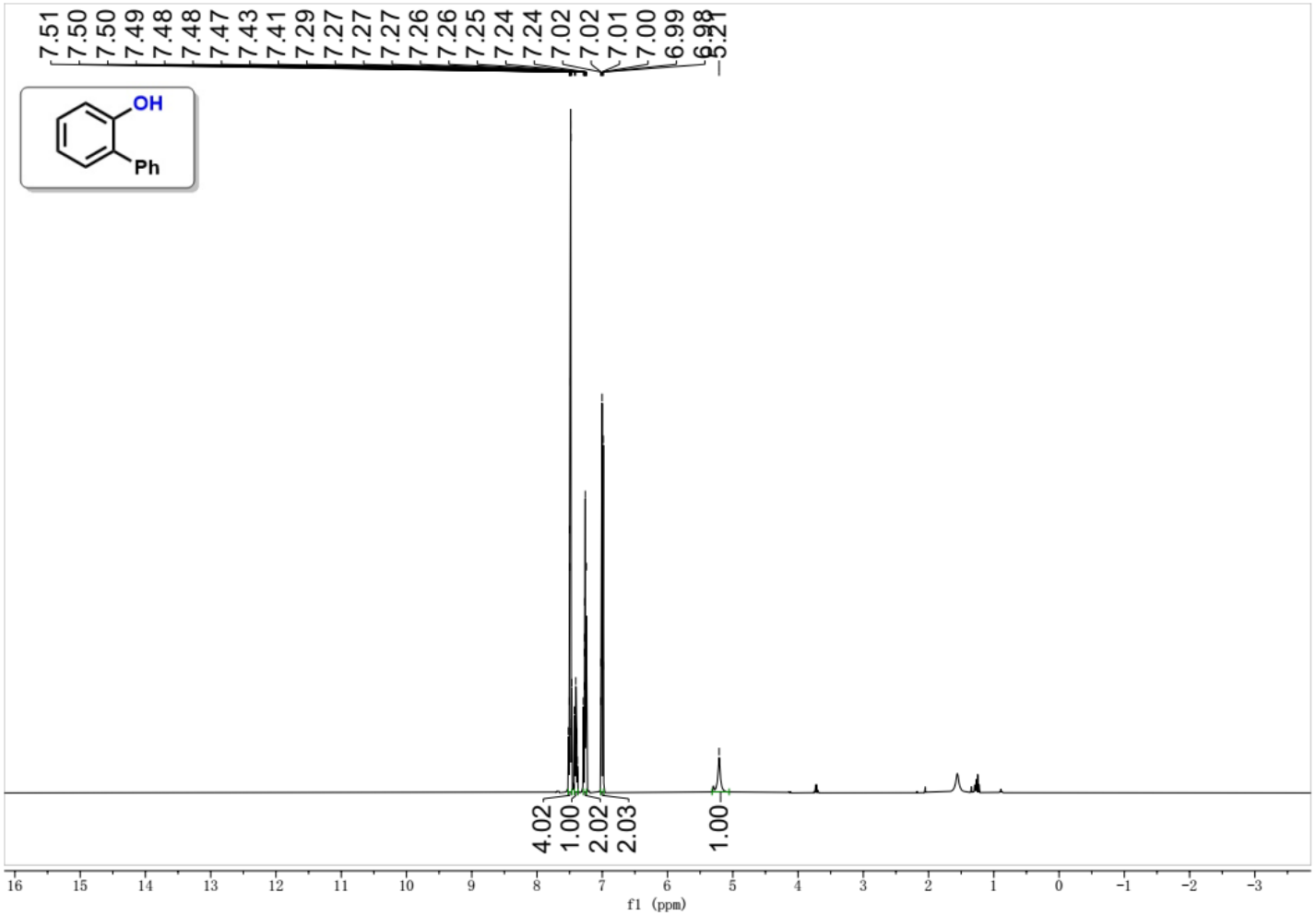
^{1}H NMR of 2a



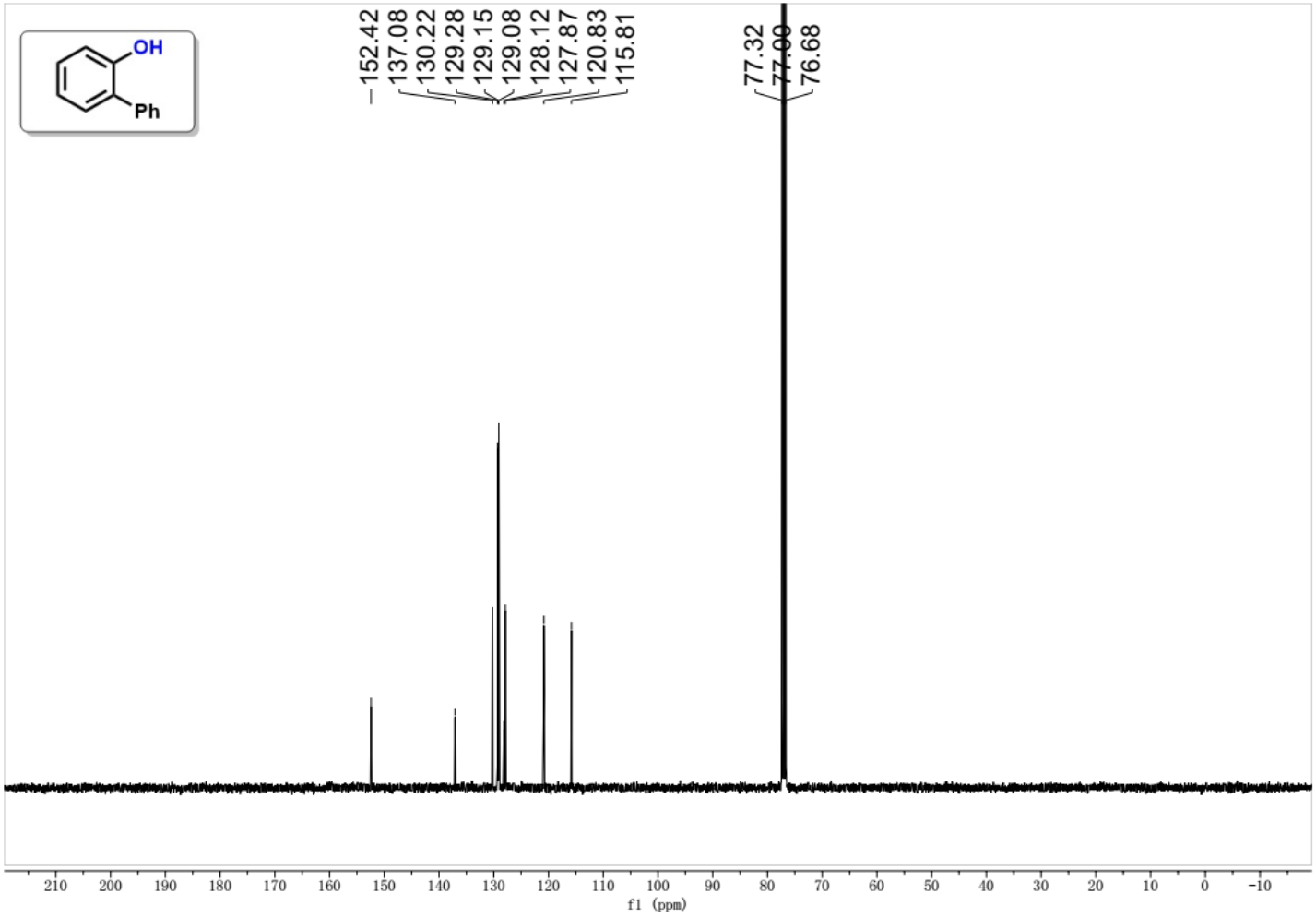
^{13}C NMR of 2a


OH
Ph
155.05
140.74
134.05
128.71
128.38
126.71
126.70
115.62
77.32
77.00
76.68
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

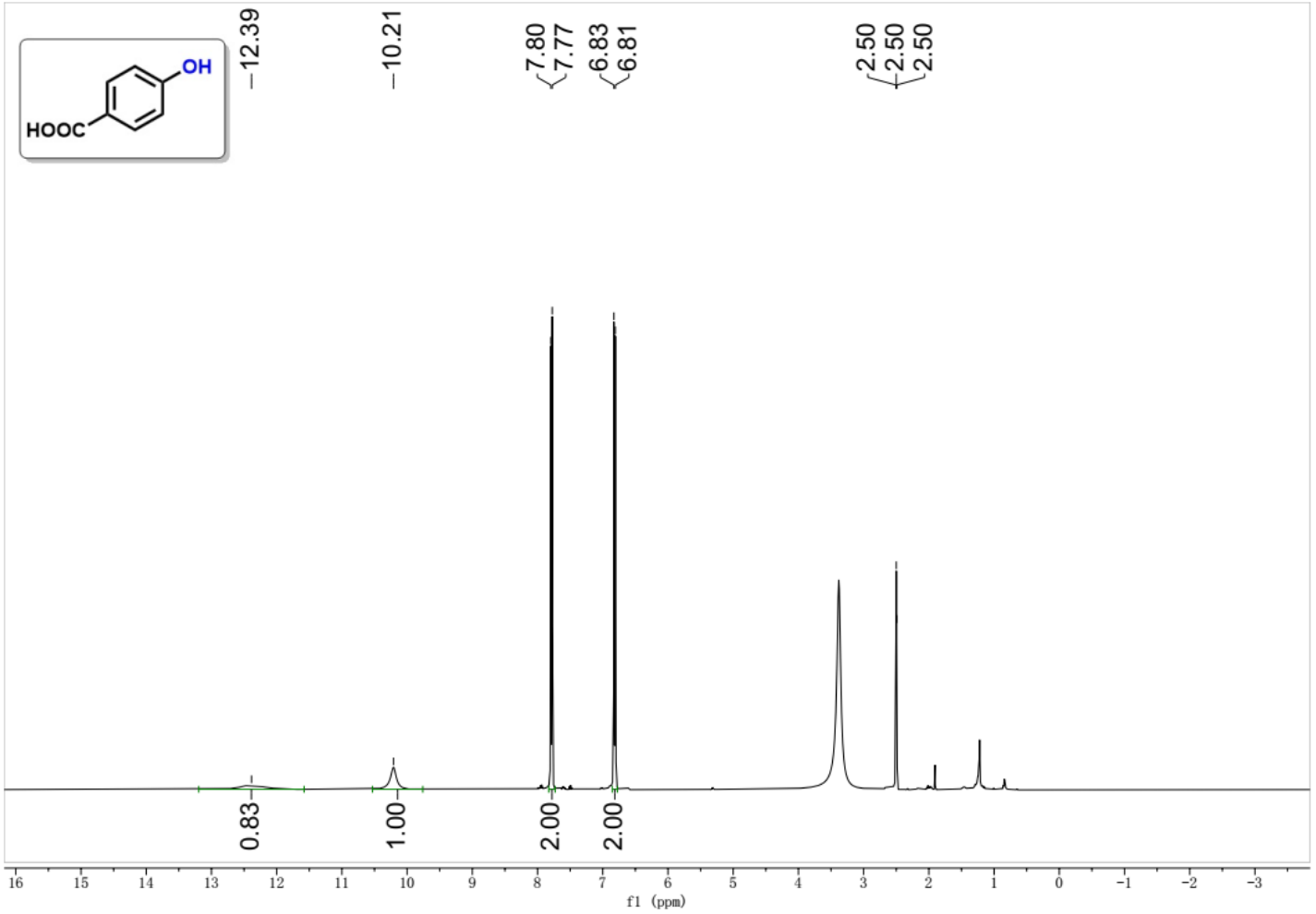
^{1}H NMR of 2b


7.51
7.50
7.50
7.49
7.48
7.48
7.47
7.43
7.41
7.29
7.27
7.27
7.27
7.26
7.26
7.25
7.24
7.24
7.02
7.02
7.01
7.00
6.99
6.98
5.21
OH
Ph
4.02
1.00
2.02
2.03
1.00
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
−1
−2
−3
f1 (ppm)

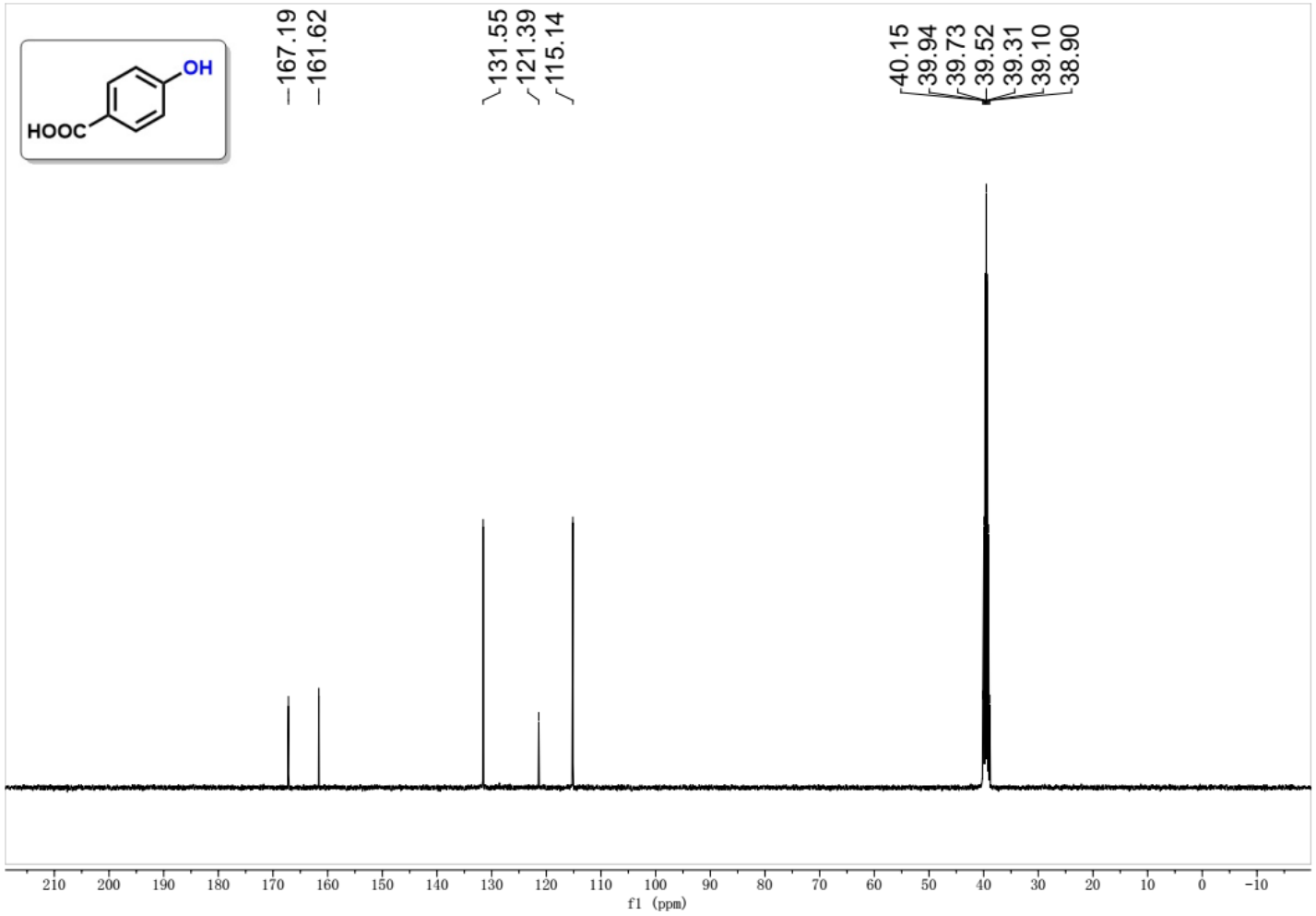
^{13}C NMR of 2b


OH
Ph
152.42
137.08
130.22
129.28
129.15
129.08
128.12
127.87
120.83
115.81
77.32
77.00
76.68
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

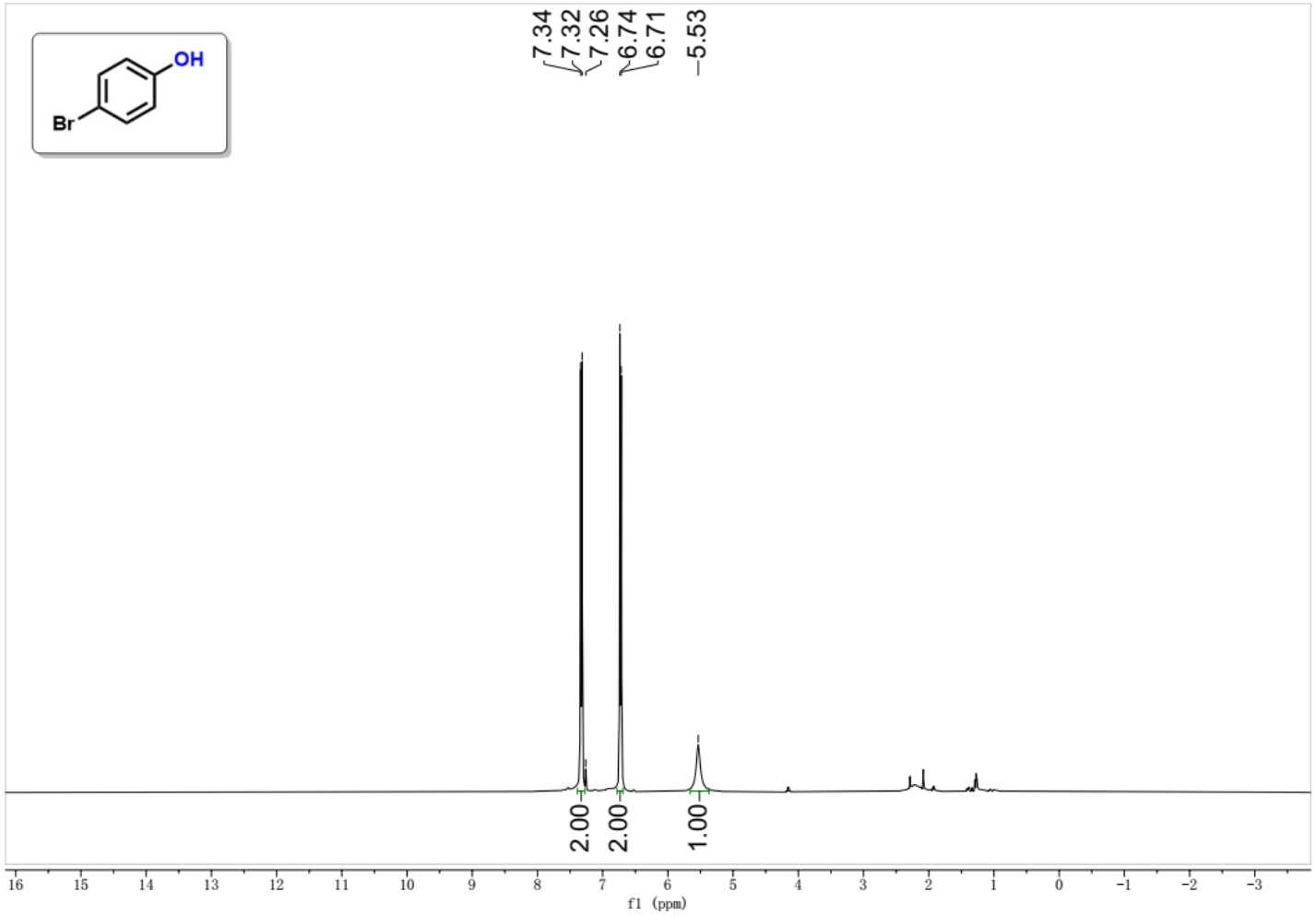
^{1}H NMR of 2c


OH
HOOC
12.39
10.21
7.80
7.77
6.83
6.81
2.50
2.50
2.50
0.83
1.00
2.00
2.00
f1 (ppm)

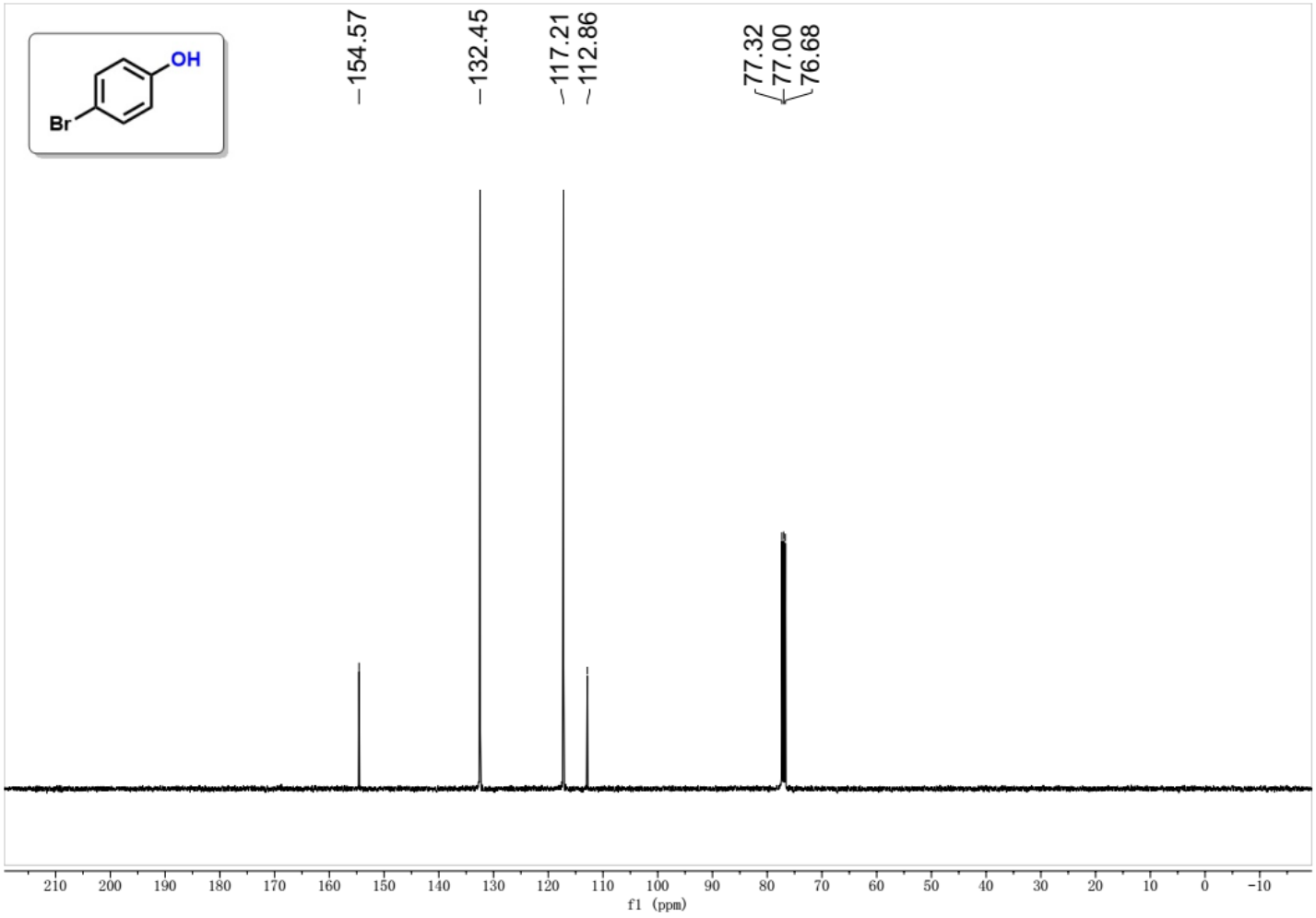
^{13}C NMR of 2c


OH
HOOC
167.19
161.62
131.55
121.39
115.14
40.15
39.94
39.73
39.52
39.31
39.10
38.90
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

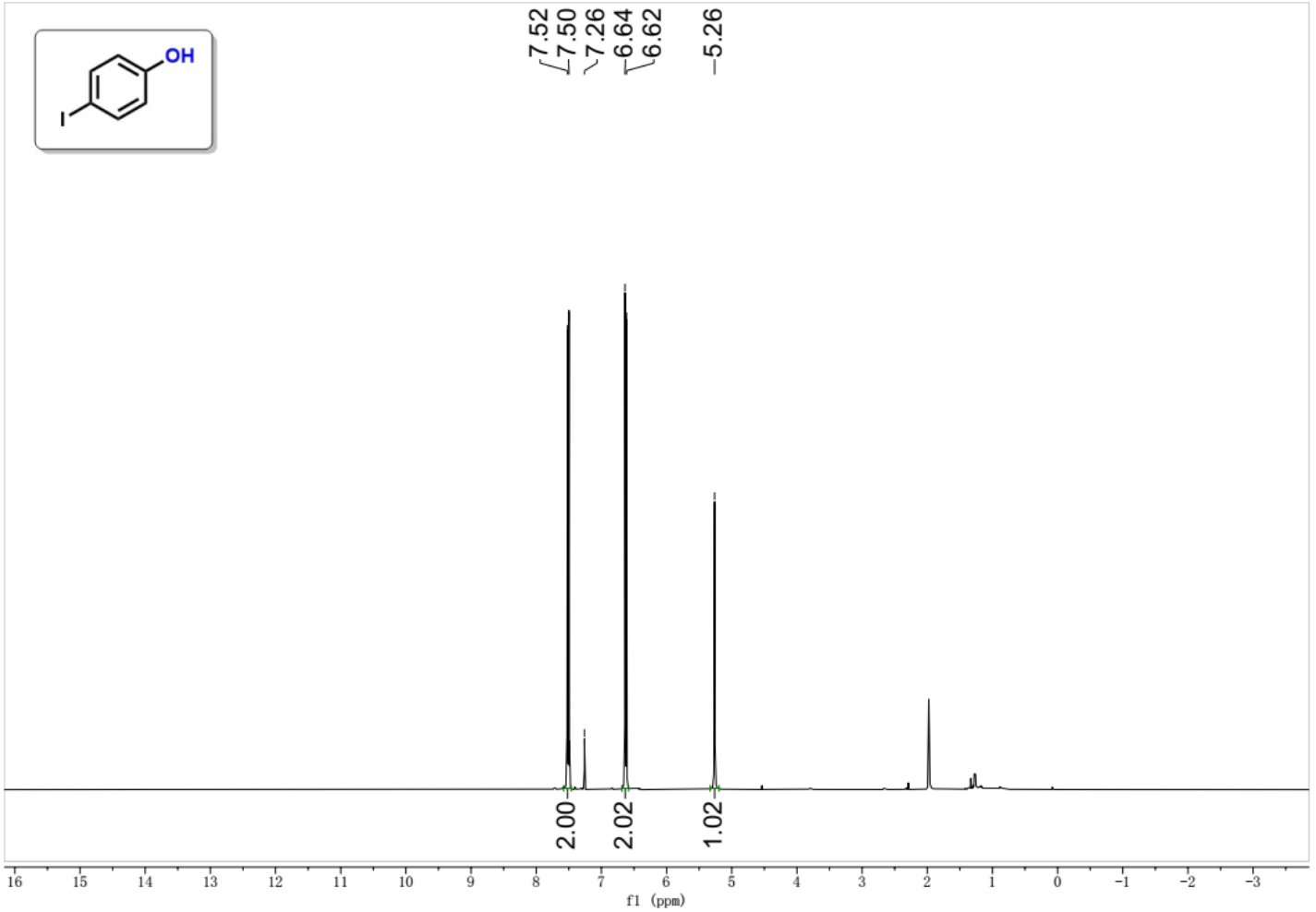
^{1}H NMR of 2d


OH
Br
7.34
7.32
7.26
6.74
6.71
5.53
2.00
2.00
1.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 −3
f1 (ppm)

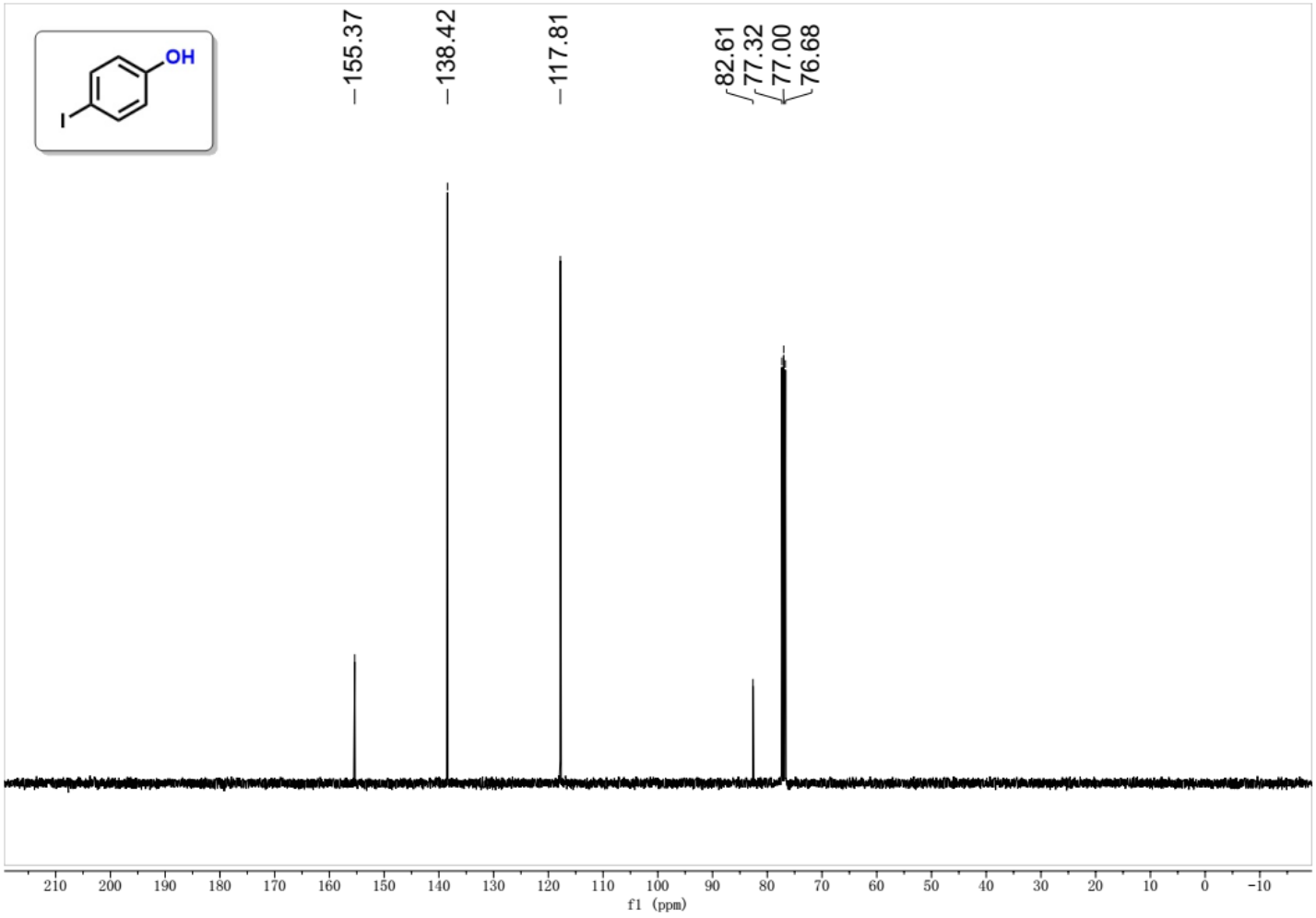
^{13}C NMR of 2d


OH
Br
154.57
132.45
117.21
112.86
77.32
77.00
76.68
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

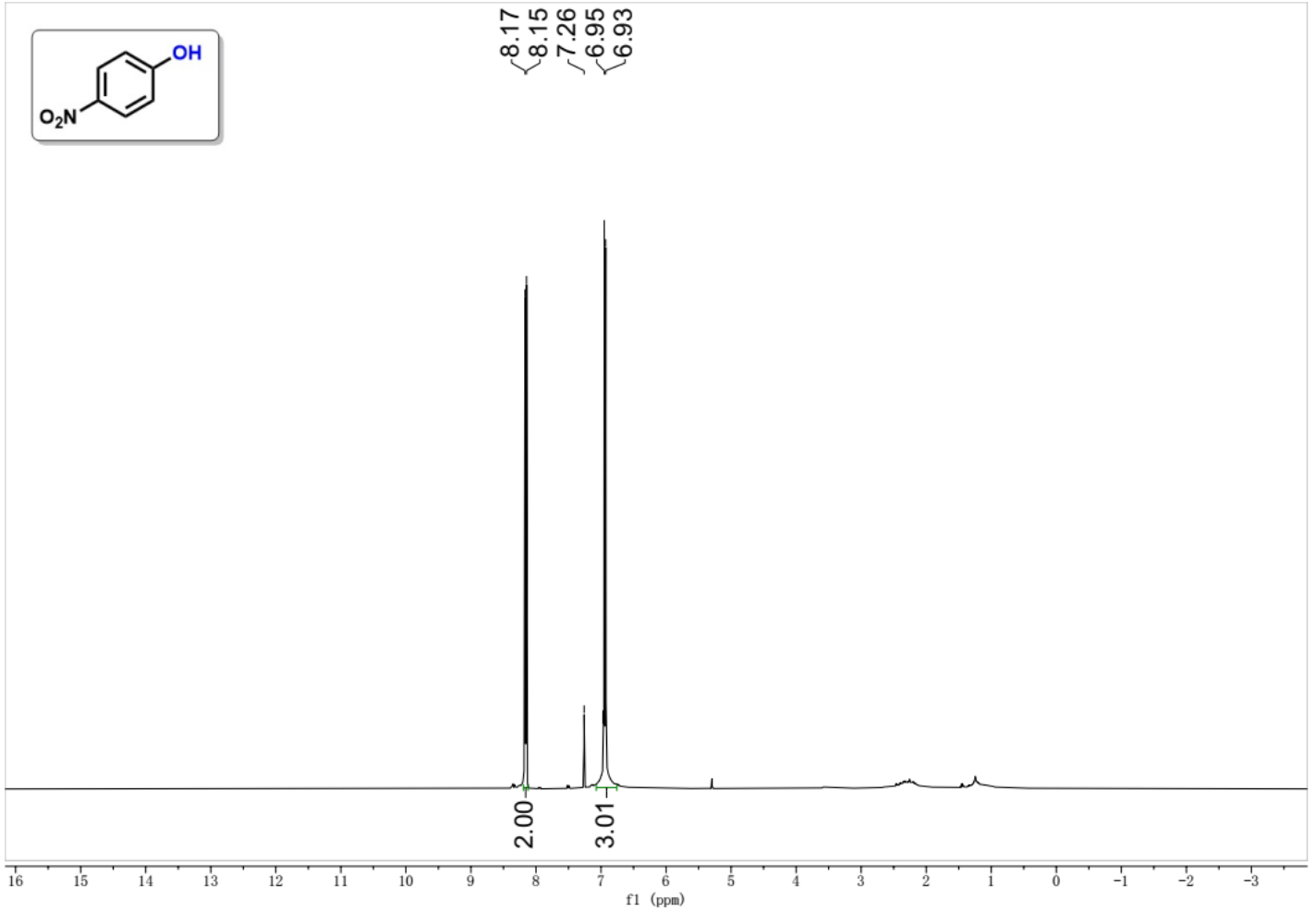
^{1}H NMR of 2e


OH
I
7.52
7.50
7.26
6.64
6.62
5.26
2.00
2.02
1.02
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 −3
f1 (ppm)

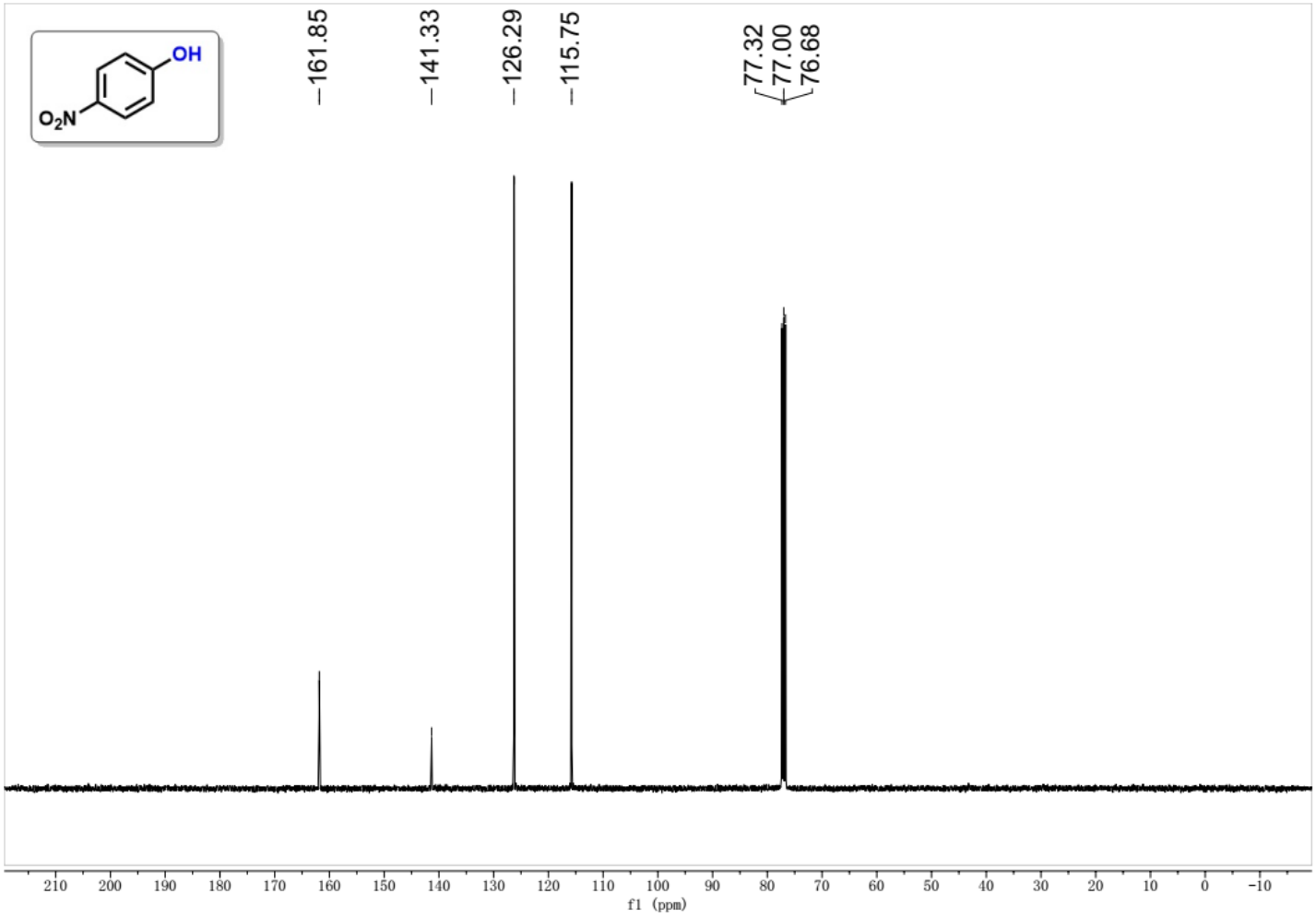
^{13}C NMR of 2e


OH
I
155.37
138.42
117.81
82.61
77.32
77.00
76.68
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

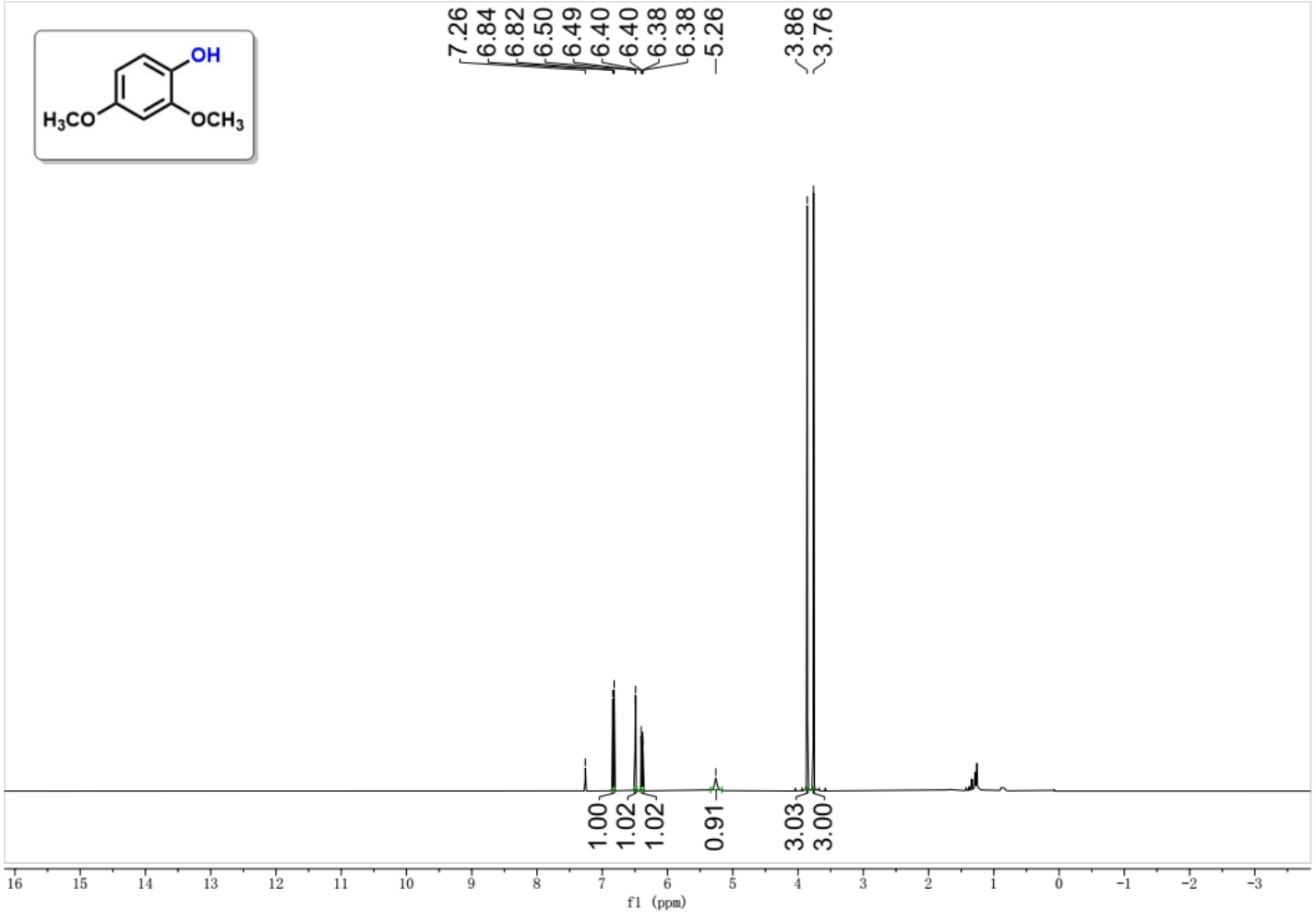
^{1}H NMR of 2f


OH
O_2N
8.17
8.15
7.26
6.95
6.93
2.00
3.01
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 −3
f1 (ppm)

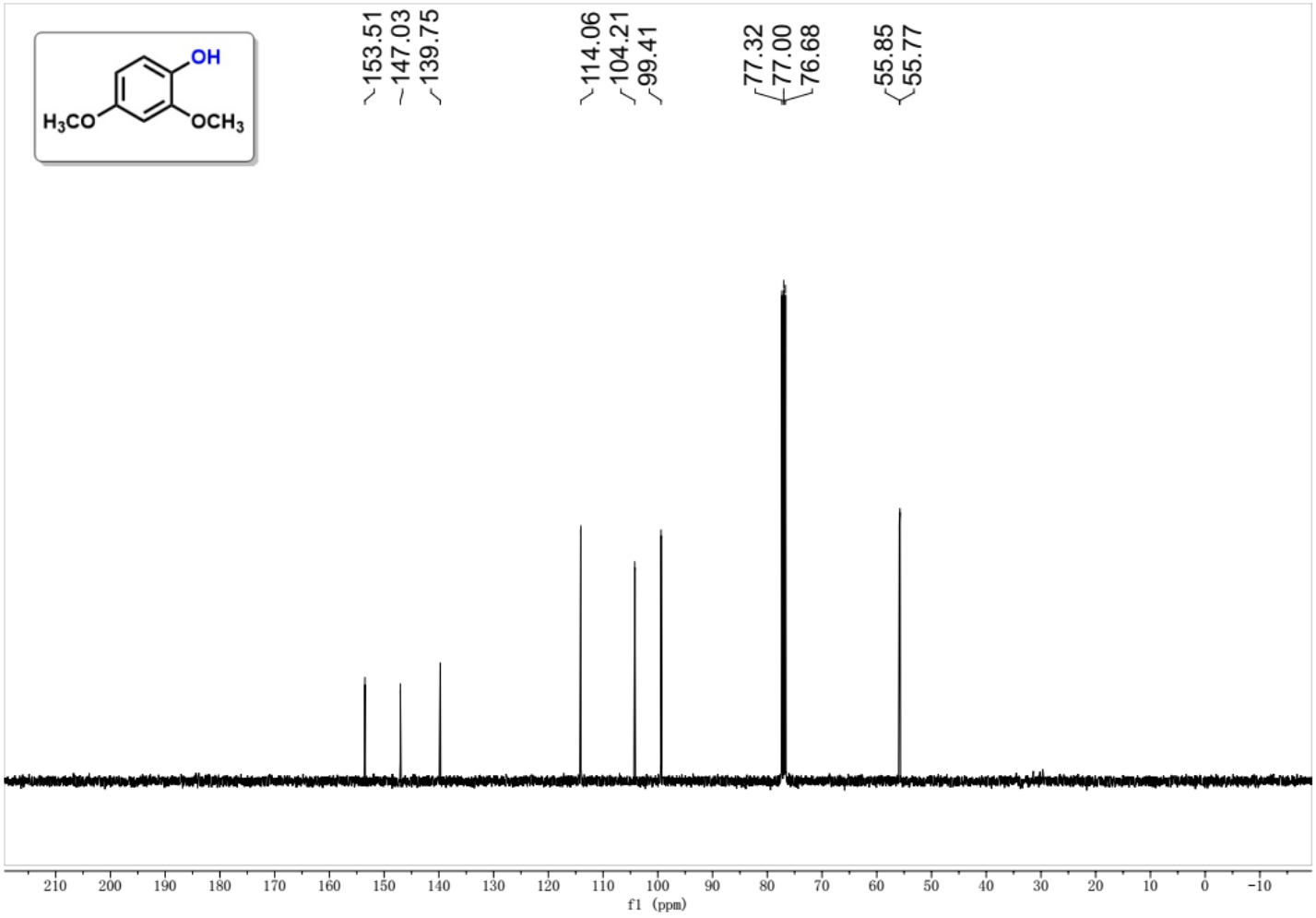
^{13}C NMR of 2f


OH
O2N
161.85
141.33
126.29
115.75
77.32
77.00
76.68
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

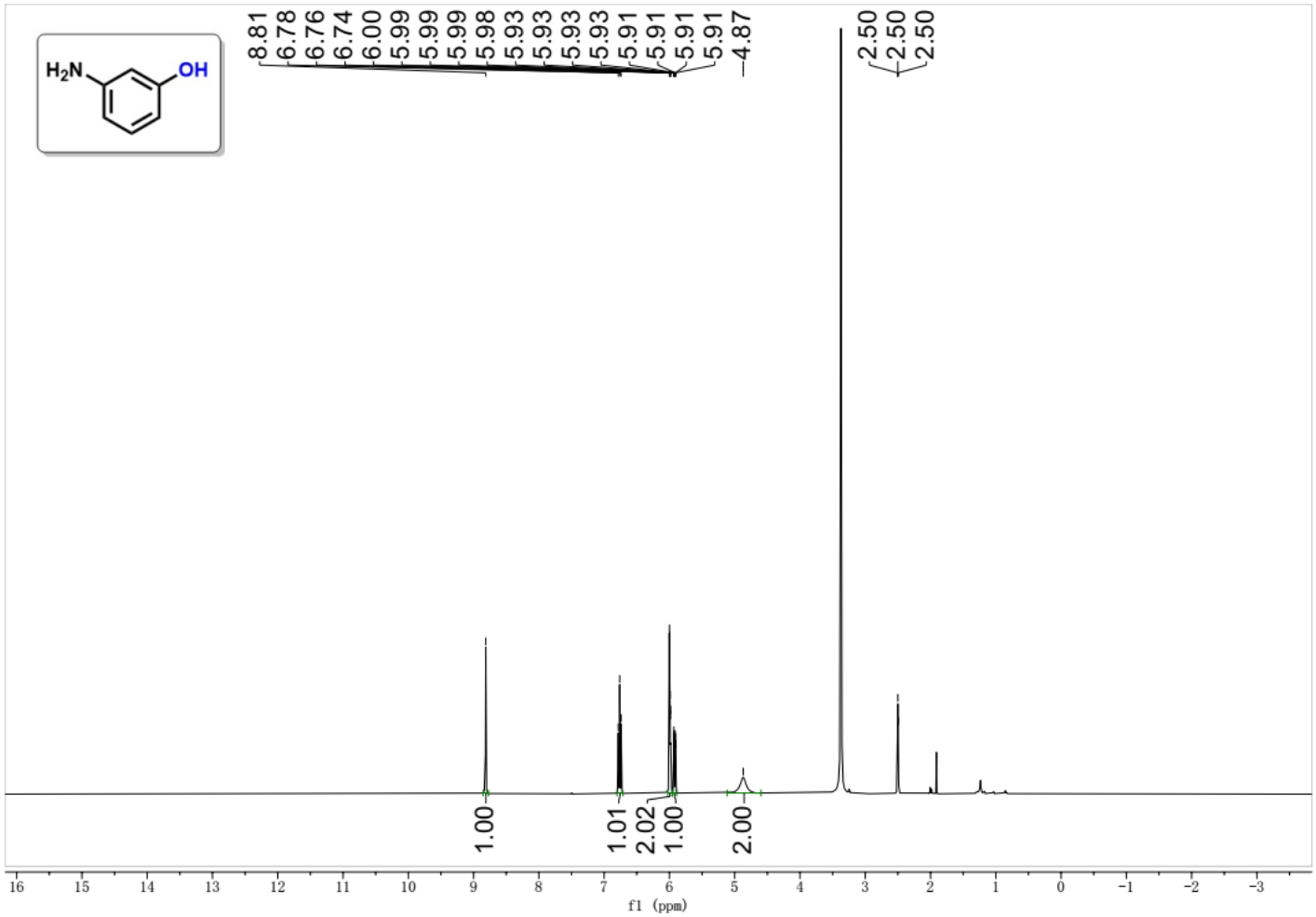
^{1}H NMR of 2g


OH
H3CO
OCH3
7.26
6.84
6.82
6.50
6.49
6.40
6.40
6.38
6.38
5.26
3.86
3.76
1.00
1.02
1.02
0.91
3.03
3.00
f1 (ppm)

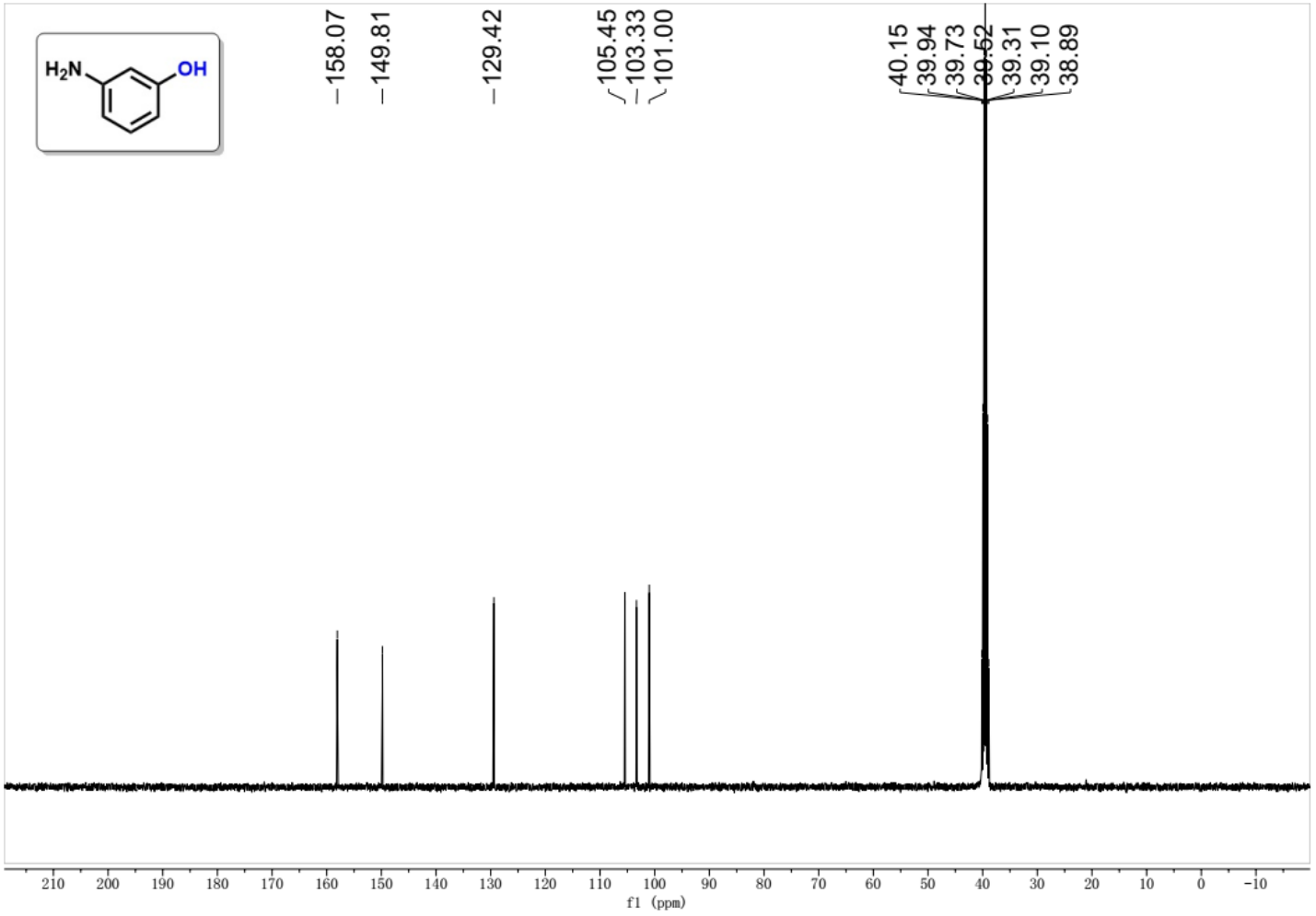
^{13}C NMR of 2g


OH
H3CO
OCH3
153.51
147.03
139.75
114.06
104.21
99.41
77.32
77.00
76.68
55.85
55.77
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

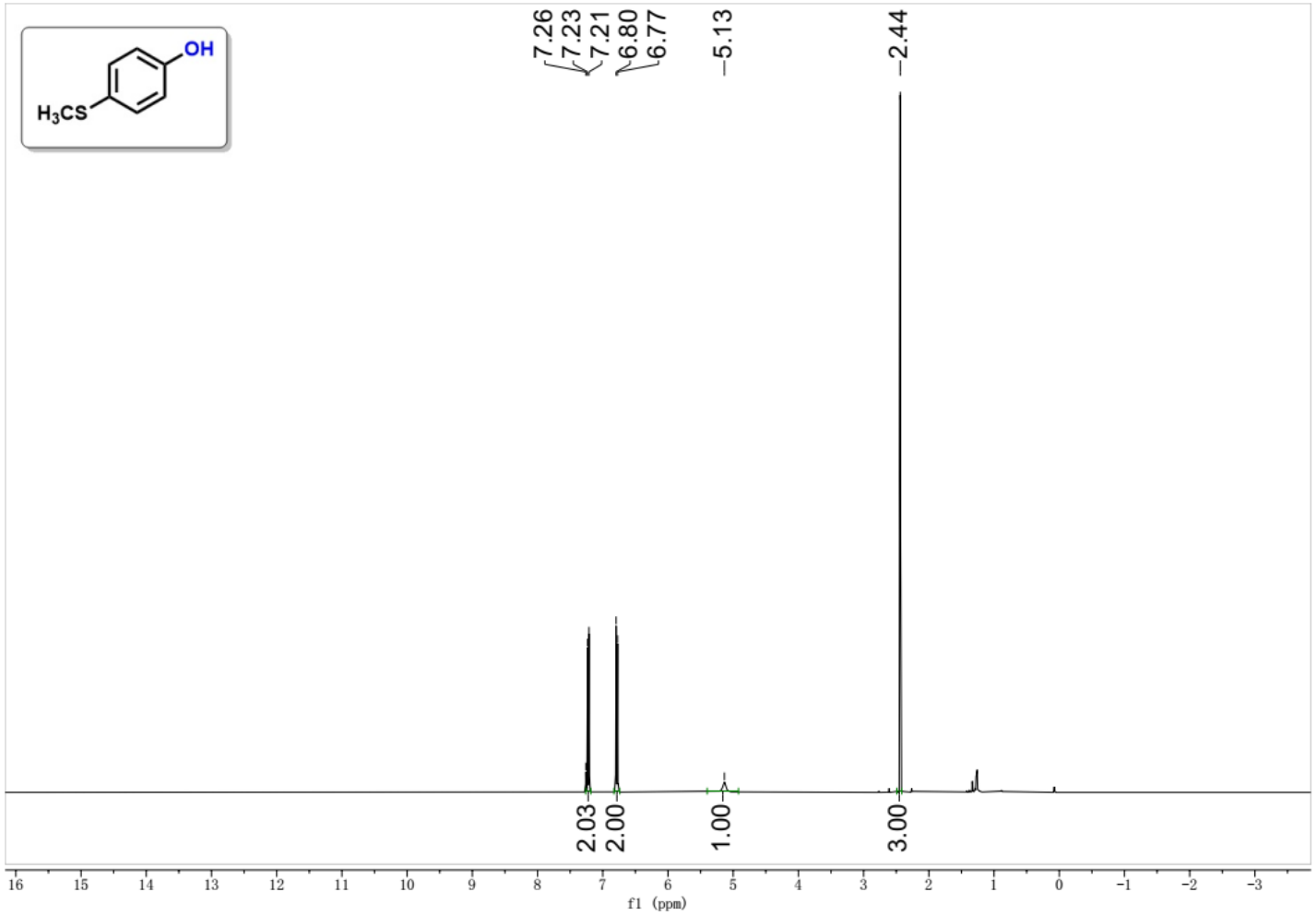
^{1}H NMR of 2h


H2N
OH
8.81
6.78
6.76
6.74
6.00
5.99
5.99
5.99
5.98
5.93
5.93
5.93
5.93
5.91
5.91
5.91
5.91
4.87
2.50
2.50
2.50
1.00
1.01
2.02
1.00
2.00
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
−1
−2
−3
f1 (ppm)

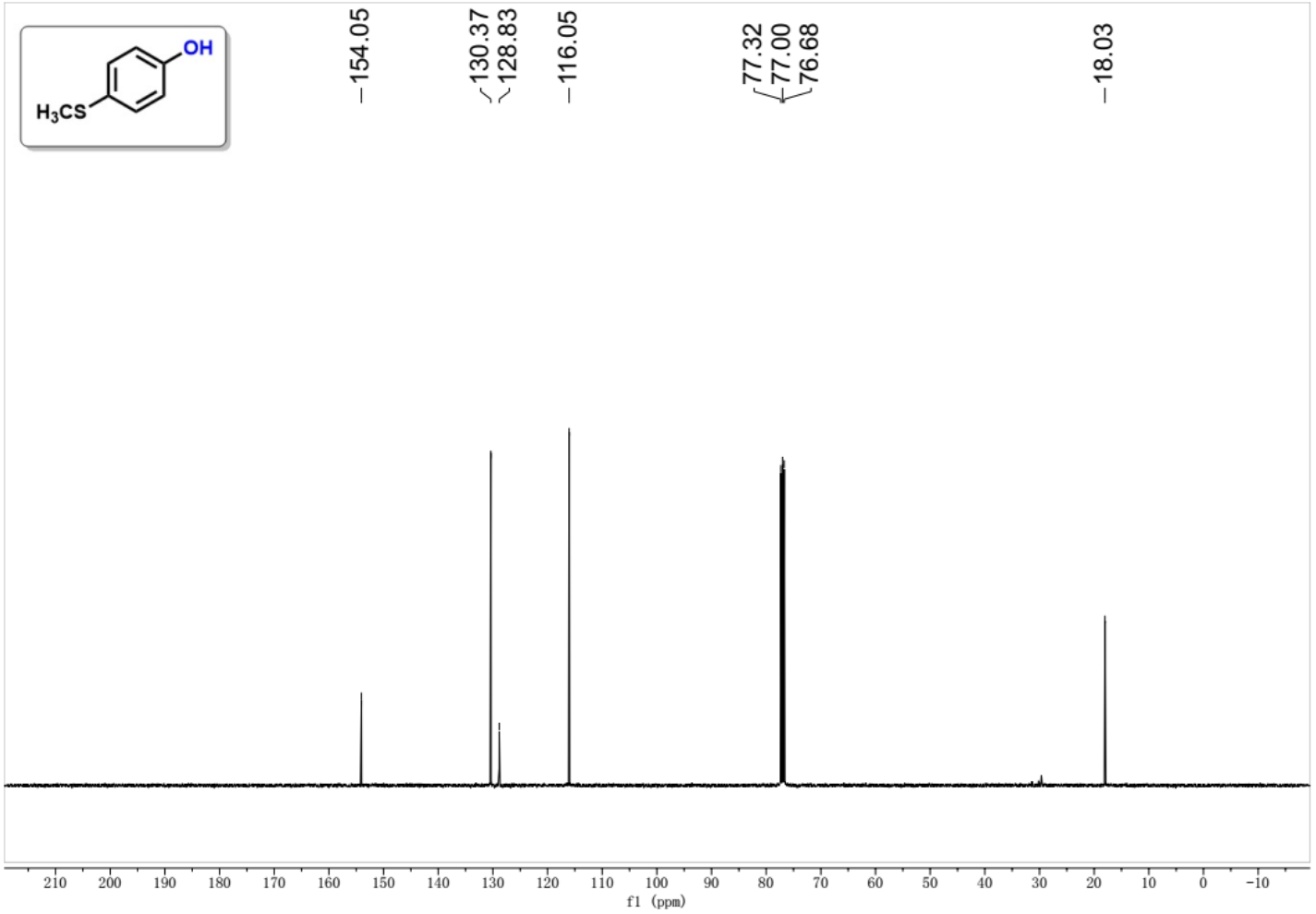
^{13}C NMR of 2h


H2N
OH
158.07
149.81
129.42
105.45
103.33
101.00
40.15
39.94
39.73
39.52
39.31
39.10
38.89
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

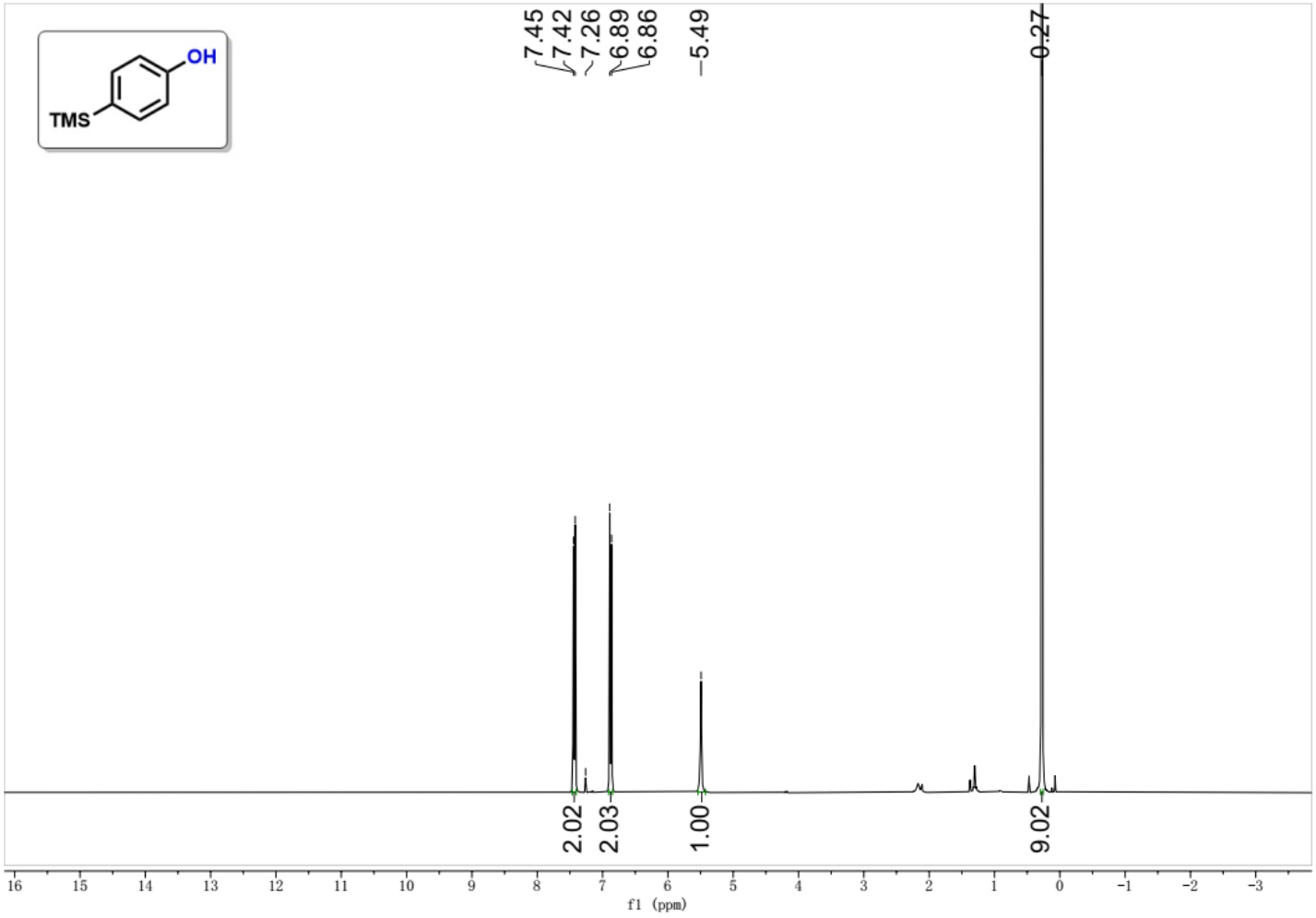
^{1}H NMR of 2i


OH
H3CS
7.26
7.23
7.21
6.80
6.77
5.13
2.44
2.03
2.00
1.00
3.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

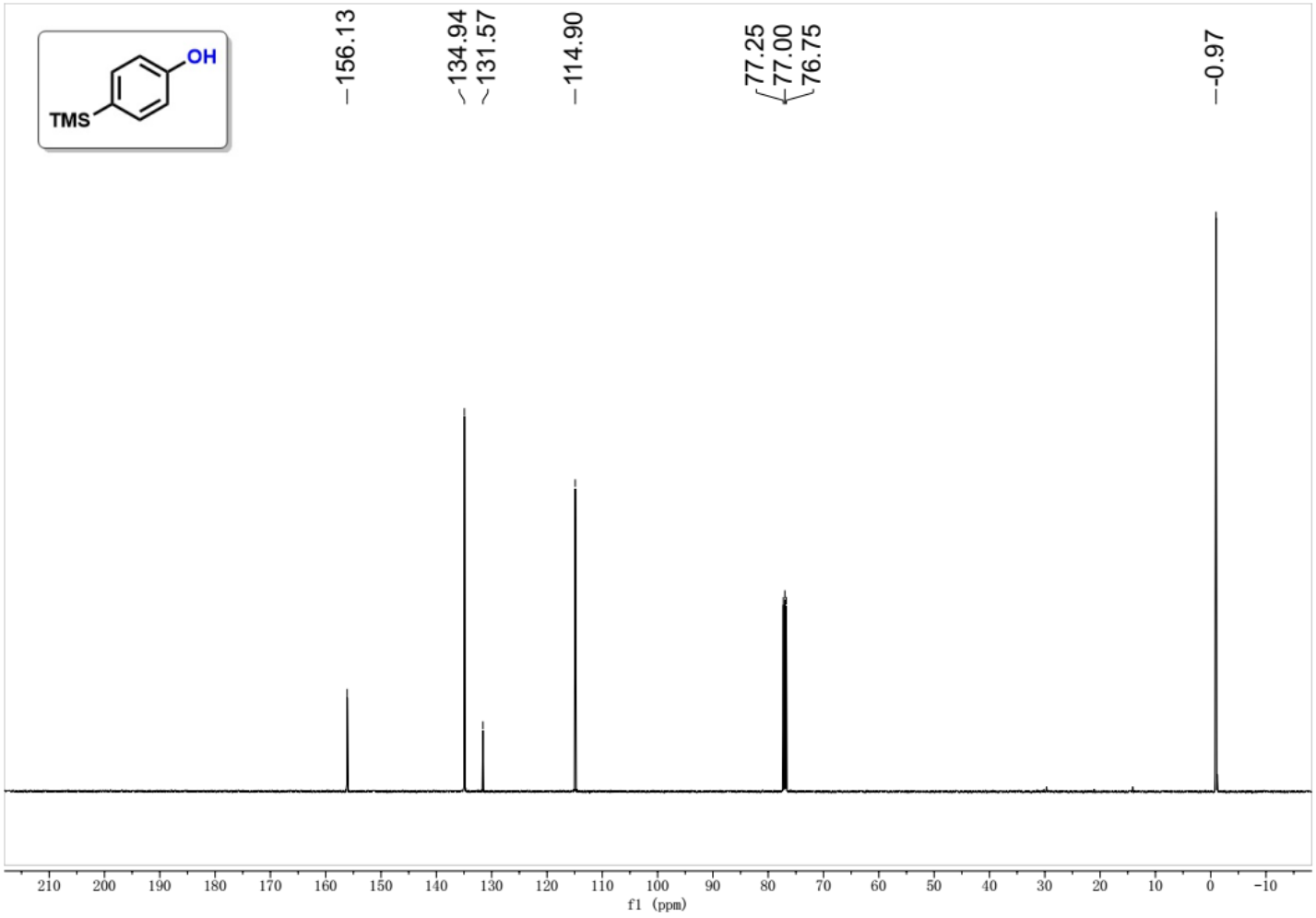
^{13}C NMR of 2i


OH
H3CS
154.05
130.37
128.83
116.05
77.32
77.00
76.68
18.03
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

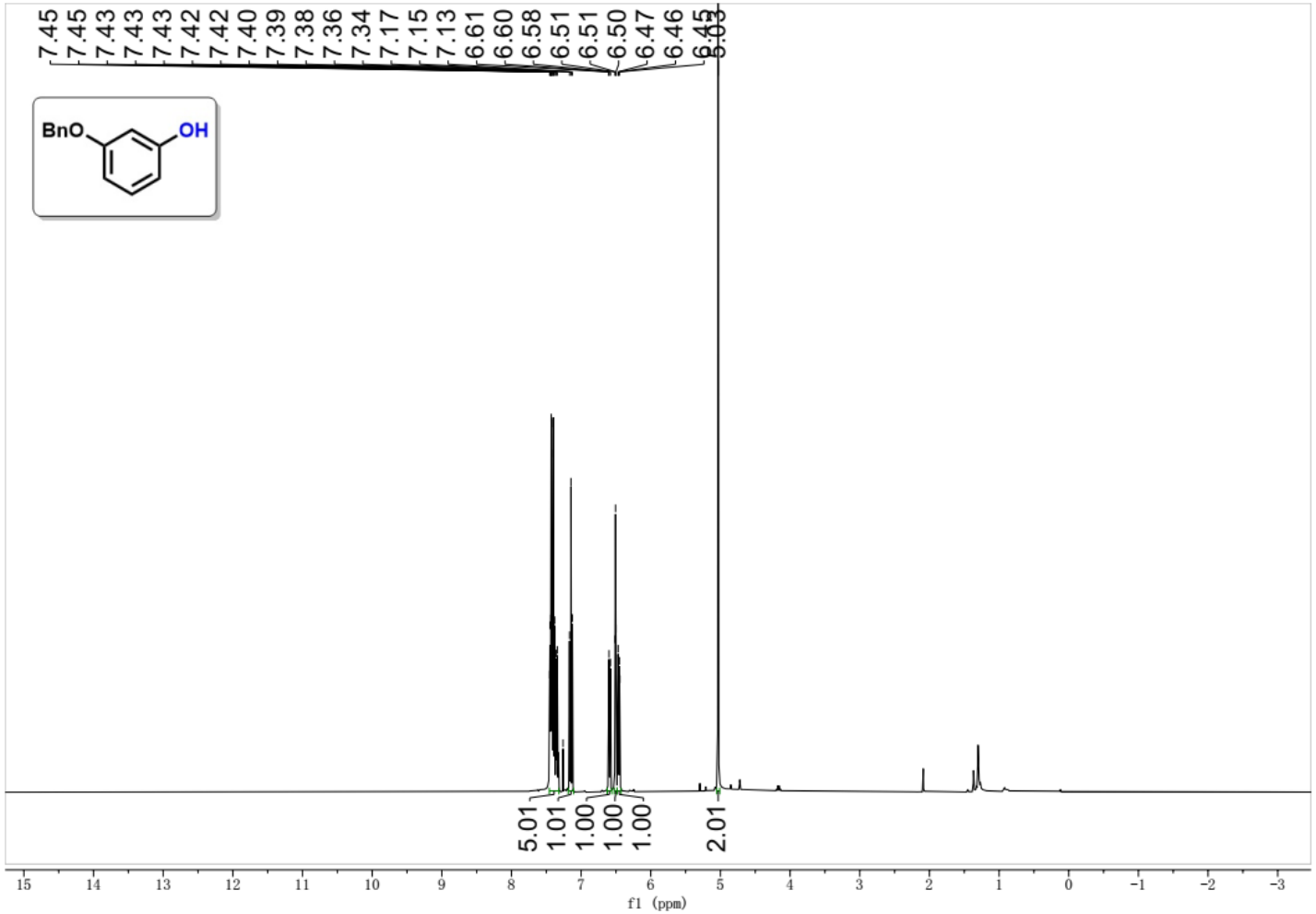
^{1}H NMR of 2j


OH
TMS
7.45
7.42
7.26
6.89
6.86
5.49
0.27
2.02
2.03
1.00
9.02
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 −3
f1 (ppm)

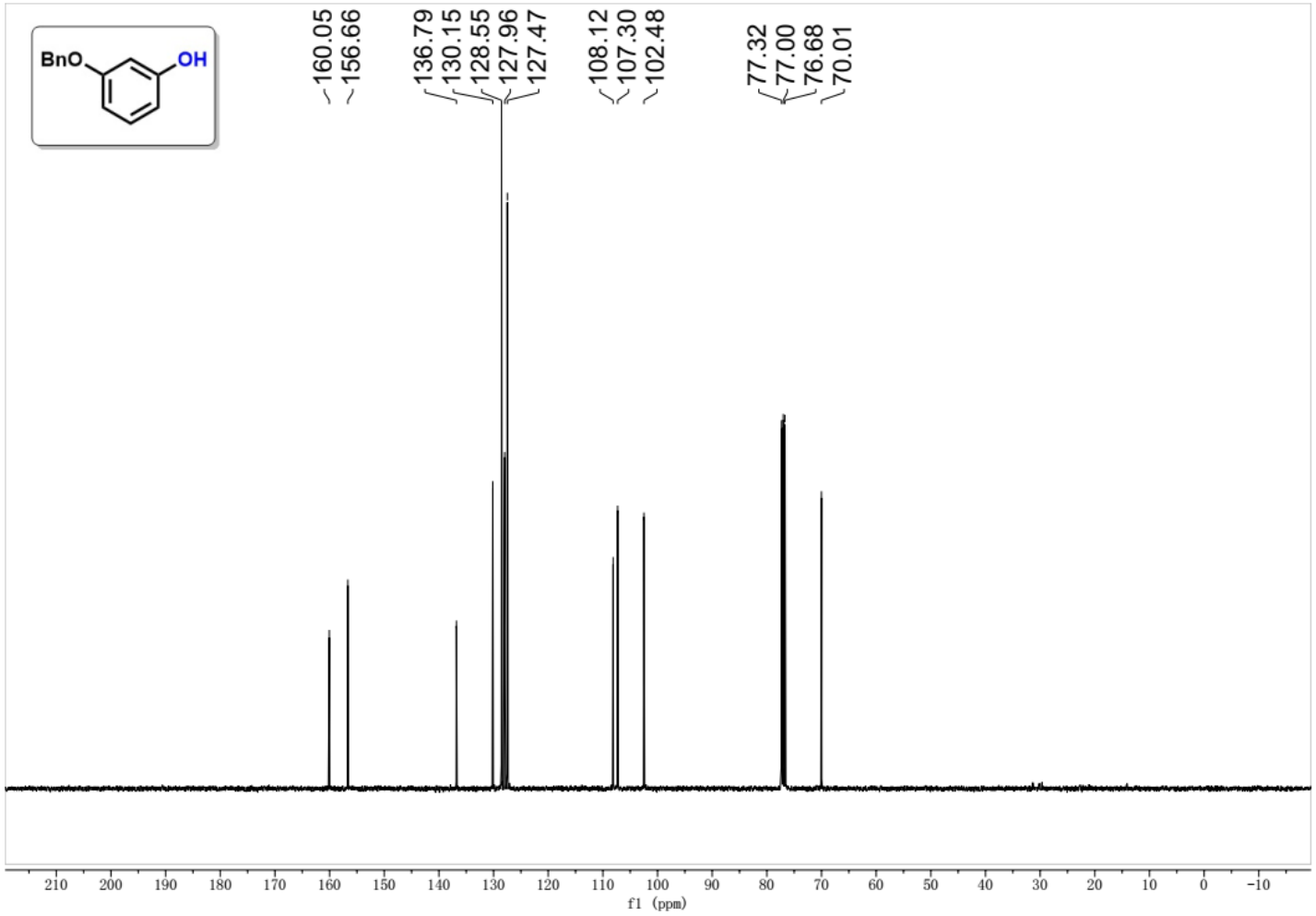
^{13}C NMR of 2j


OH
TMS
156.13
134.94
131.57
114.90
77.25
77.00
76.75
-0.97
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

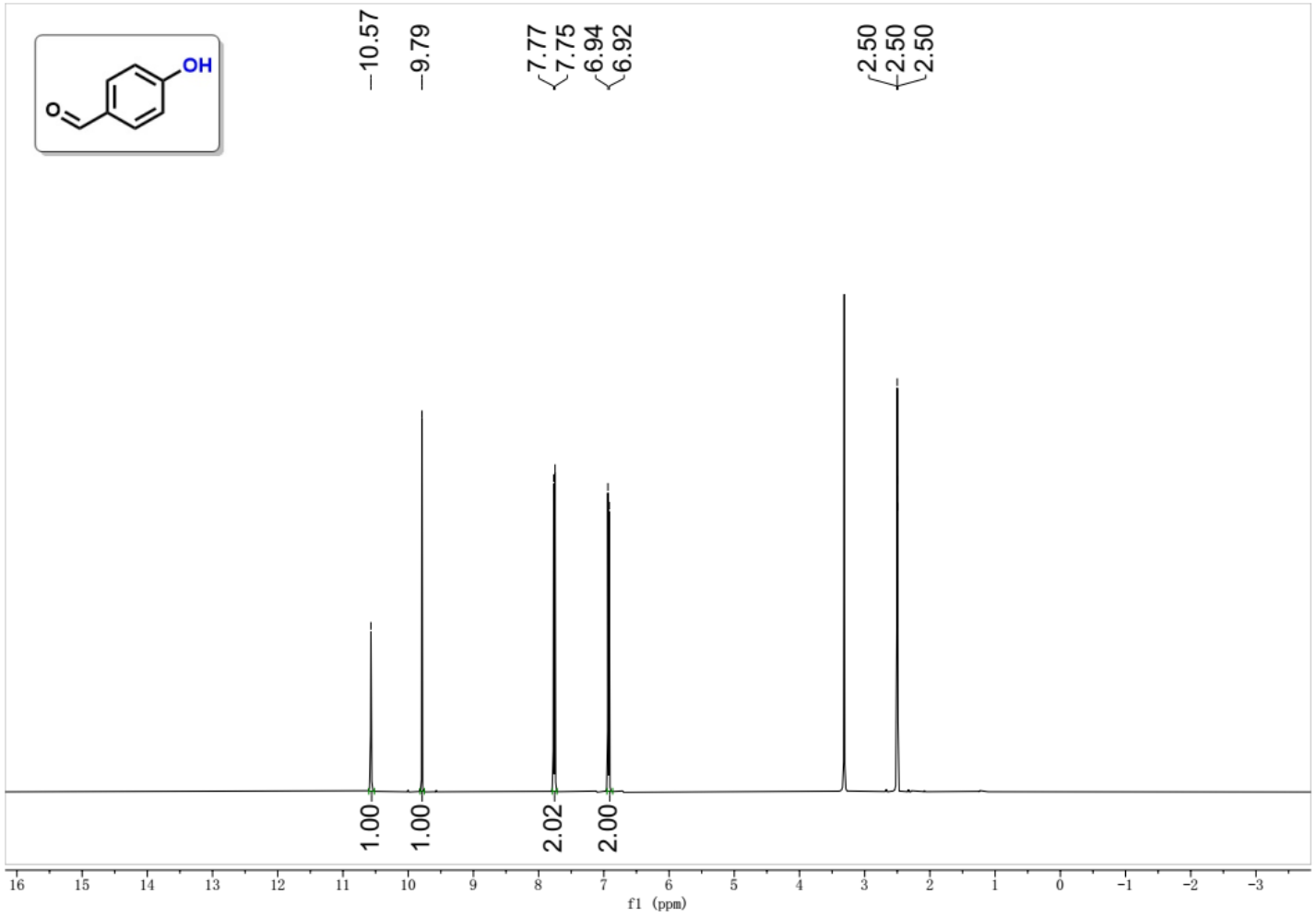
^{1}H NMR of 2k


7.45
7.45
7.43
7.43
7.43
7.42
7.42
7.40
7.39
7.38
7.36
7.34
7.17
7.15
7.13
6.61
6.60
6.58
6.51
6.51
6.50
6.47
6.46
6.45
BnO
OH
5.01
1.01
1.00
1.00
1.00
2.01
f1 (ppm)

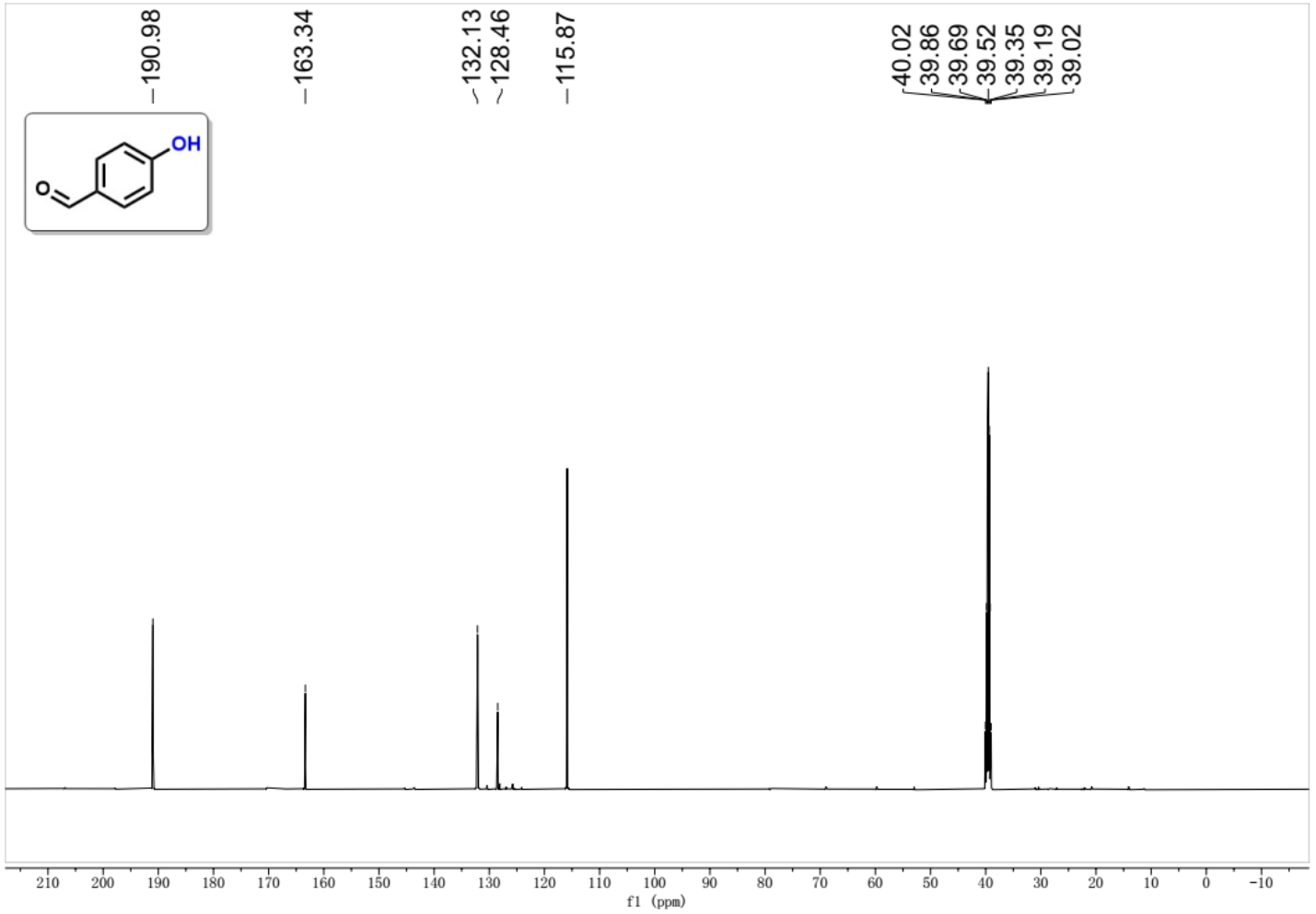
^{13}C NMR of 2k


BnO
OH
160.05
156.66
136.79
130.15
128.55
127.96
127.47
108.12
107.30
102.48
77.32
77.00
76.68
70.01
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

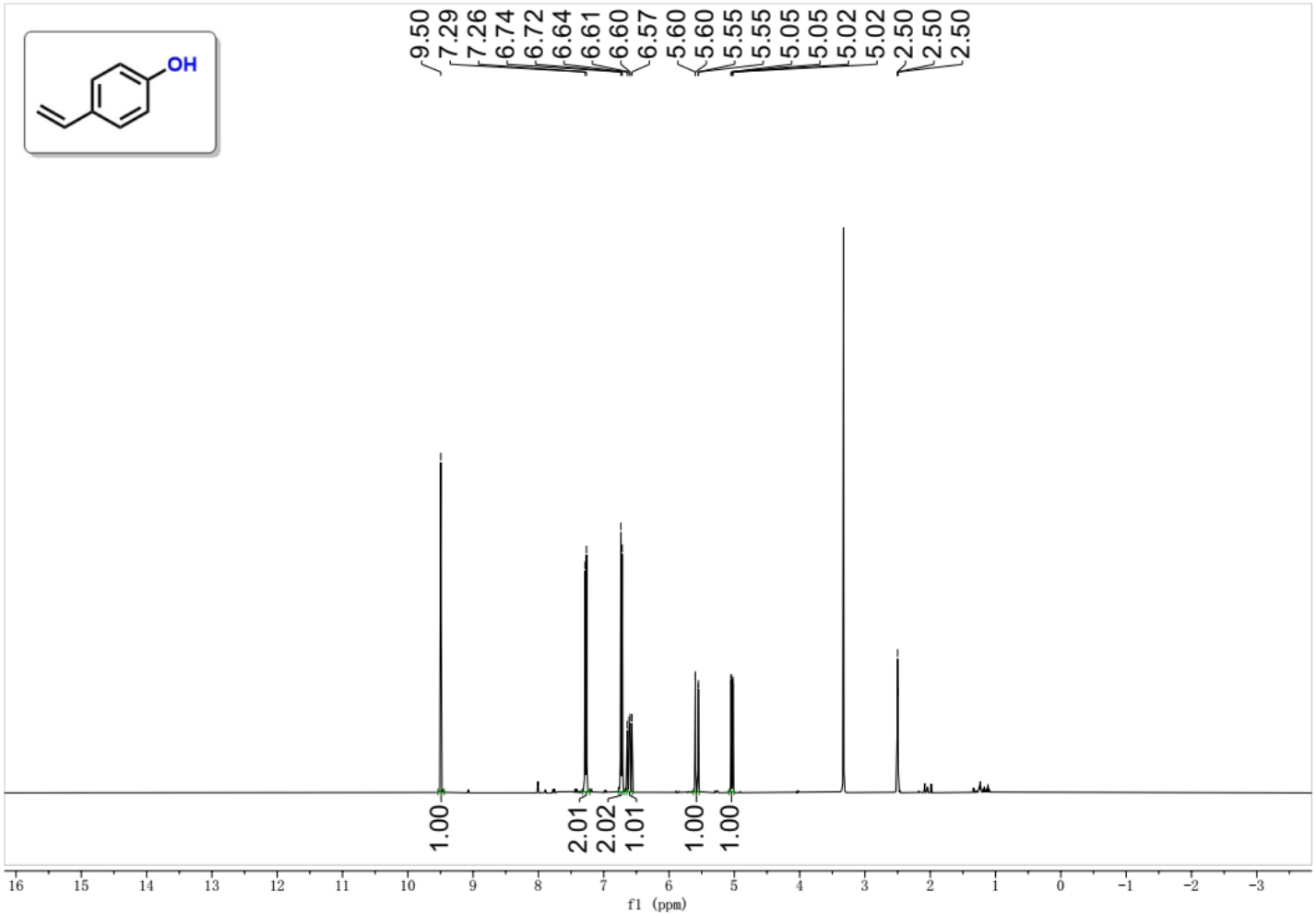
^{1}H NMR of 2l


OH
O
10.57
9.79
7.77
7.75
6.94
6.92
2.50
2.50
2.50
1.00
1.00
2.02
2.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 −3
f1 (ppm)

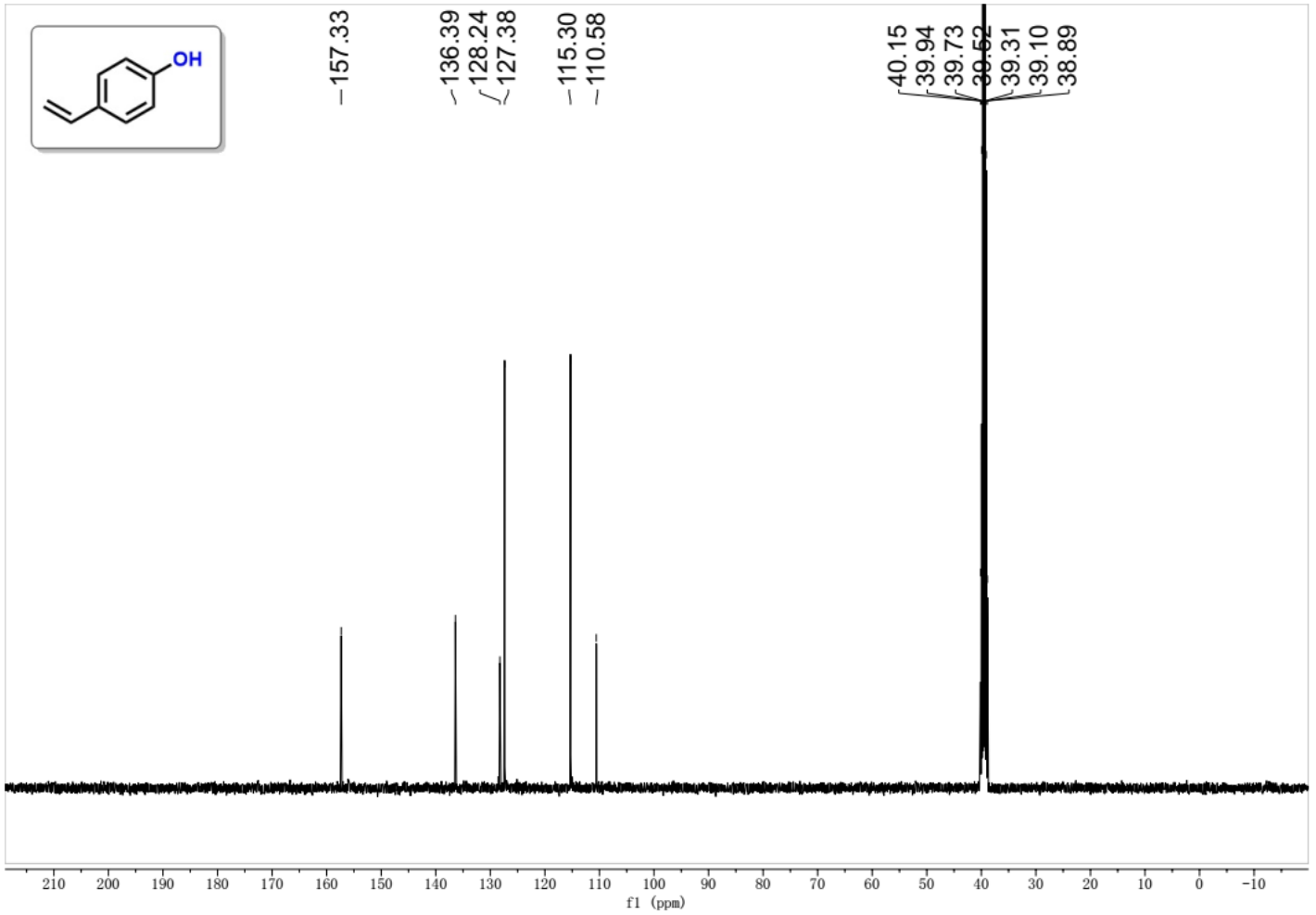
^{13}C NMR of 2l


190.98
163.34
132.13
128.46
115.87
40.02
39.86
39.69
39.52
39.35
39.19
39.02
OH
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

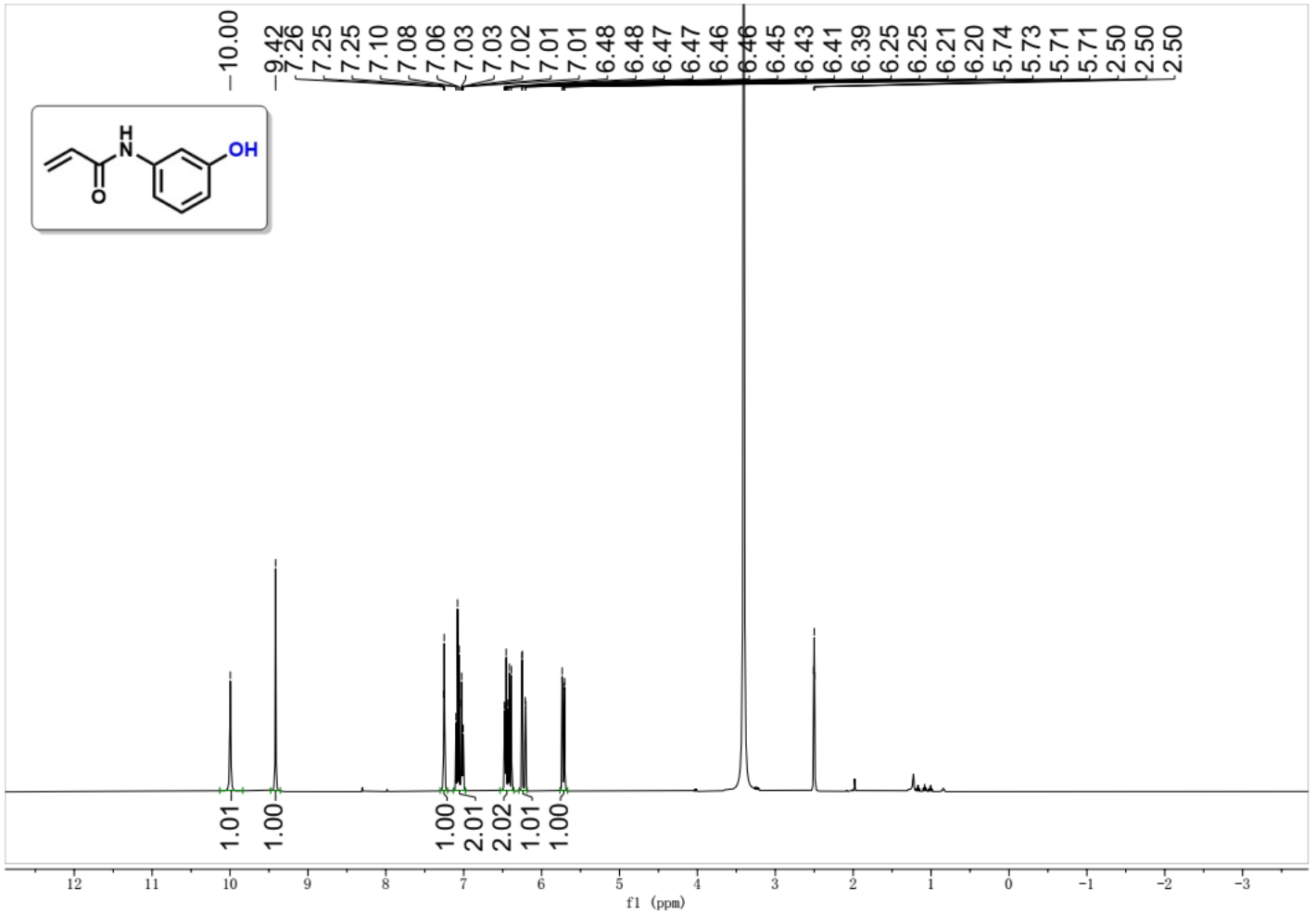
^{1}H NMR of 2m


OH
9.50
7.29
7.26
6.74
6.72
6.64
6.61
6.60
6.57
5.60
5.60
5.55
5.55
5.05
5.05
5.02
5.02
2.50
2.50
2.50
1.00
2.01
2.02
1.01
1.00
1.00
f1 (ppm)

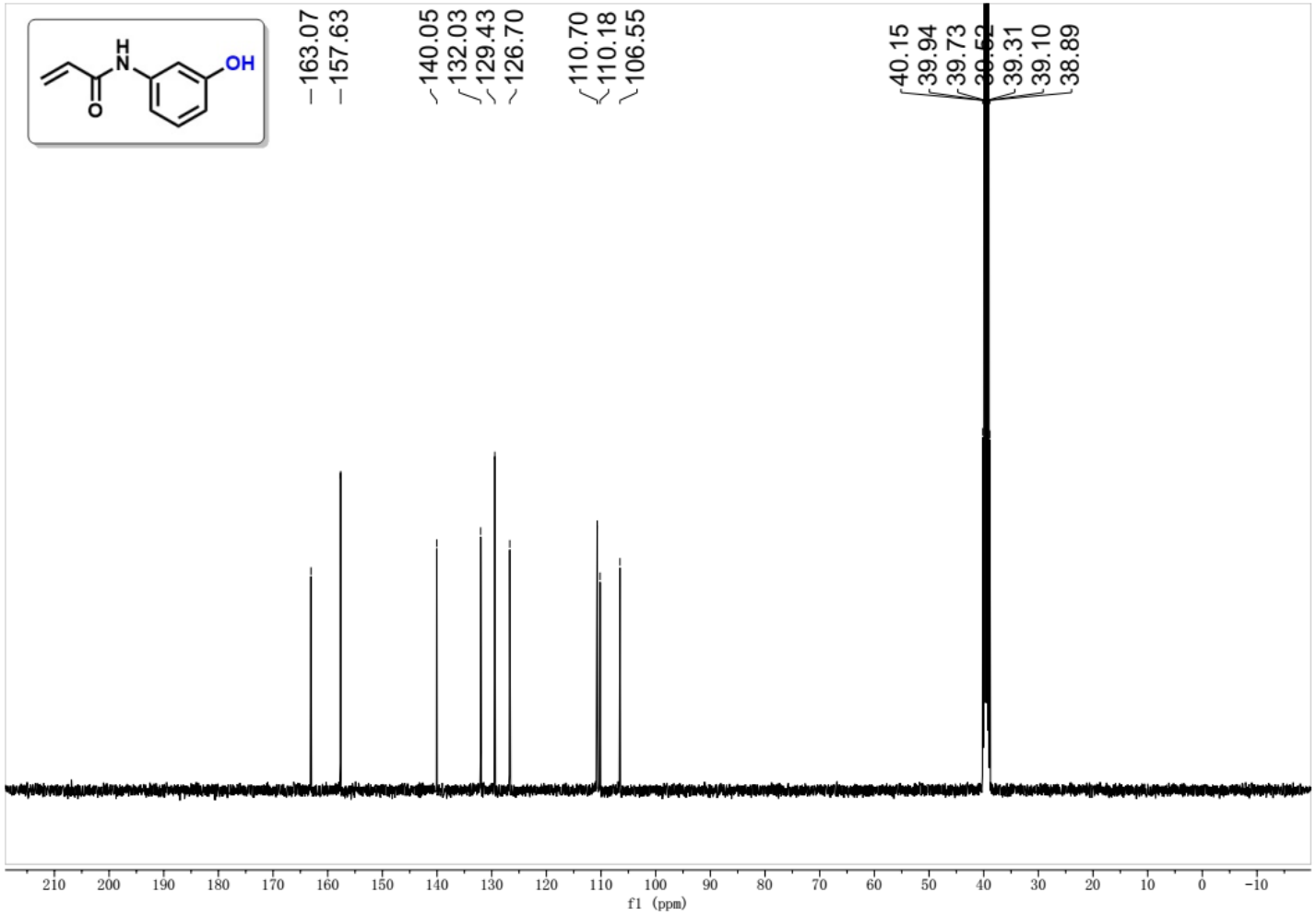
^{13}C NMR of 2m


OH
157.33
136.39
128.24
127.38
115.30
110.58
40.15
39.94
39.73
39.52
39.31
39.10
38.89
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

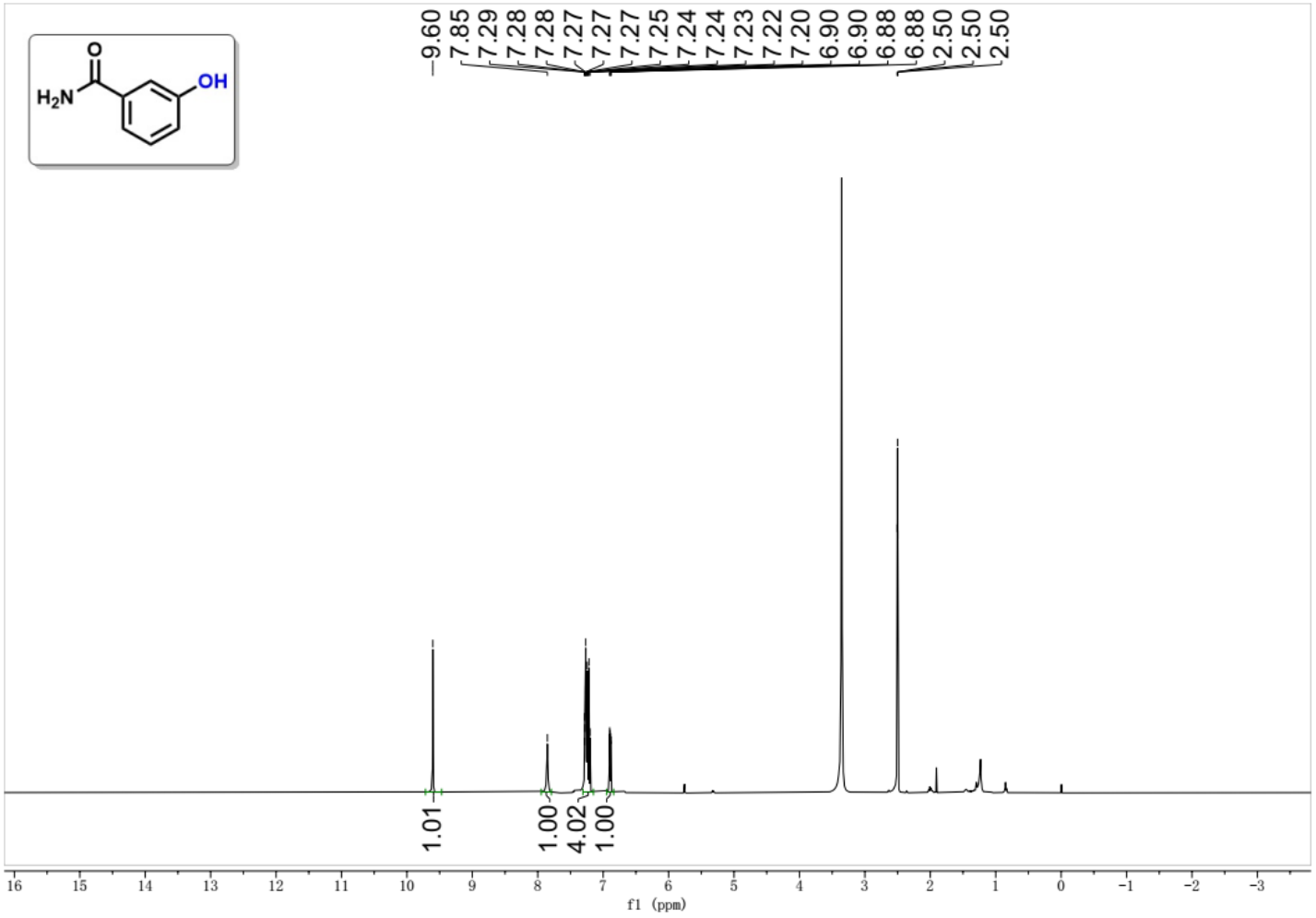
^{1}H NMR of 2n


10.00
9.42
7.26
7.25
7.25
7.10
7.08
7.06
7.03
7.03
7.02
7.01
7.01
6.48
6.48
6.47
6.47
6.46
6.46
6.45
6.43
6.41
6.39
6.25
6.25
6.21
6.20
5.74
5.73
5.71
5.71
2.50
2.50
2.50
OH
1.01
1.00
1.00
2.01
2.02
1.01
1.00
f1 (ppm)

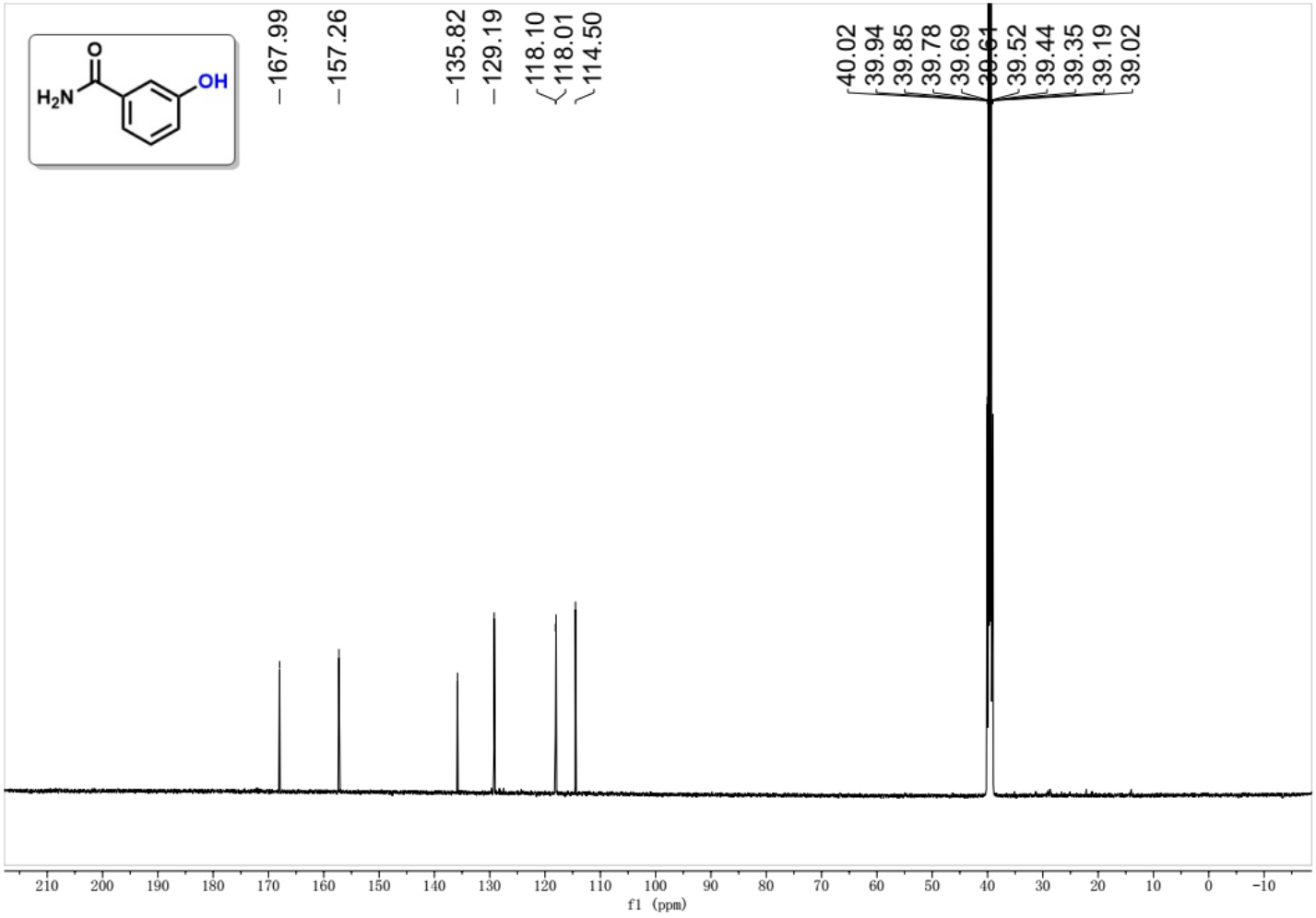
^{13}C NMR of 2n


163.07
157.63
140.05
132.03
129.43
126.70
110.70
110.18
106.55
40.15
39.94
39.73
39.52
39.31
39.10
38.89
OH
H
N
O
f1 (ppm)

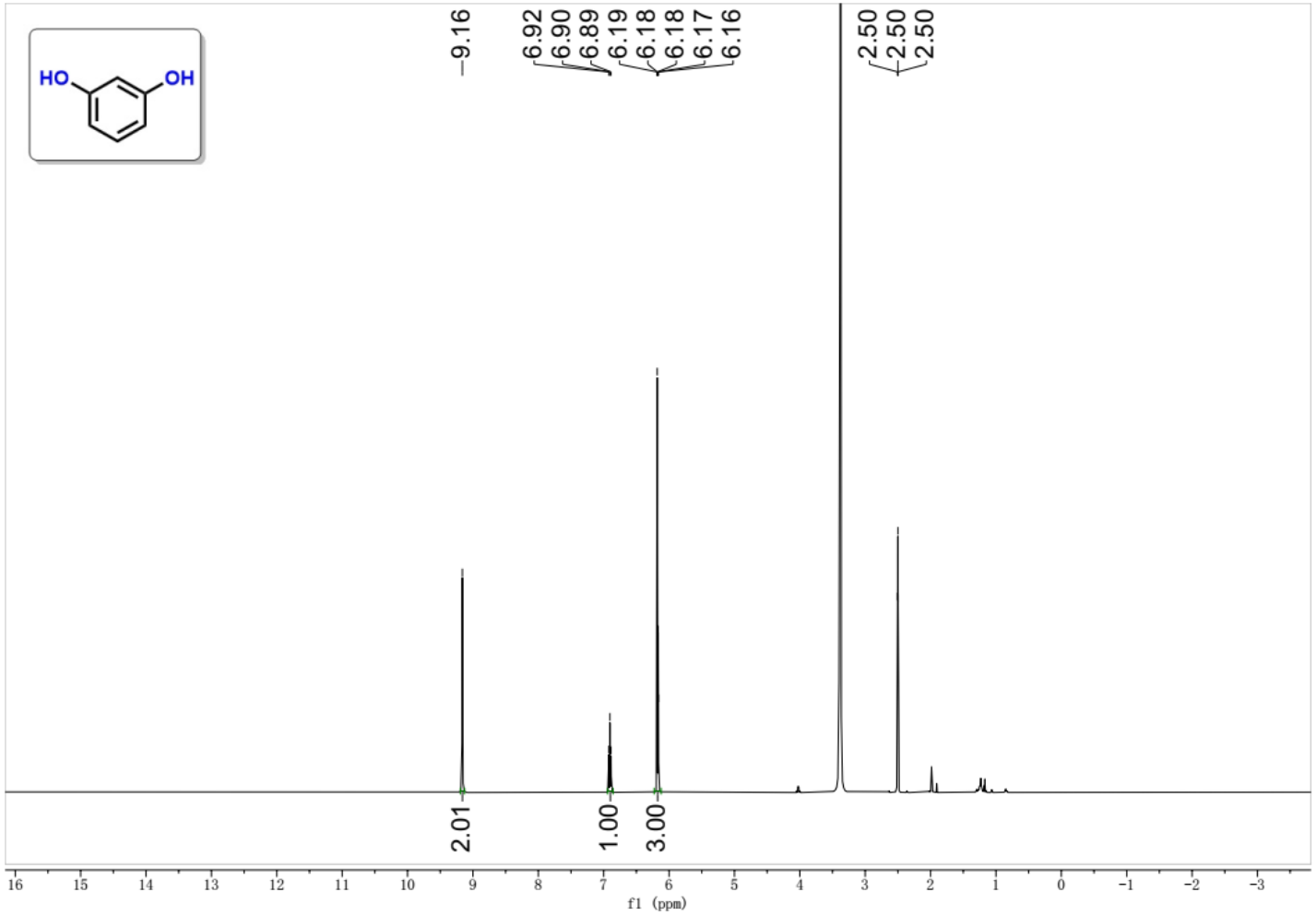
^{1}H NMR of 2o


9.60
7.85
7.29
7.28
7.28
7.27
7.27
7.27
7.25
7.24
7.24
7.23
7.22
7.20
6.90
6.90
6.88
6.88
2.50
2.50
2.50
1.01
1.00
4.02
1.00
f1 (ppm)

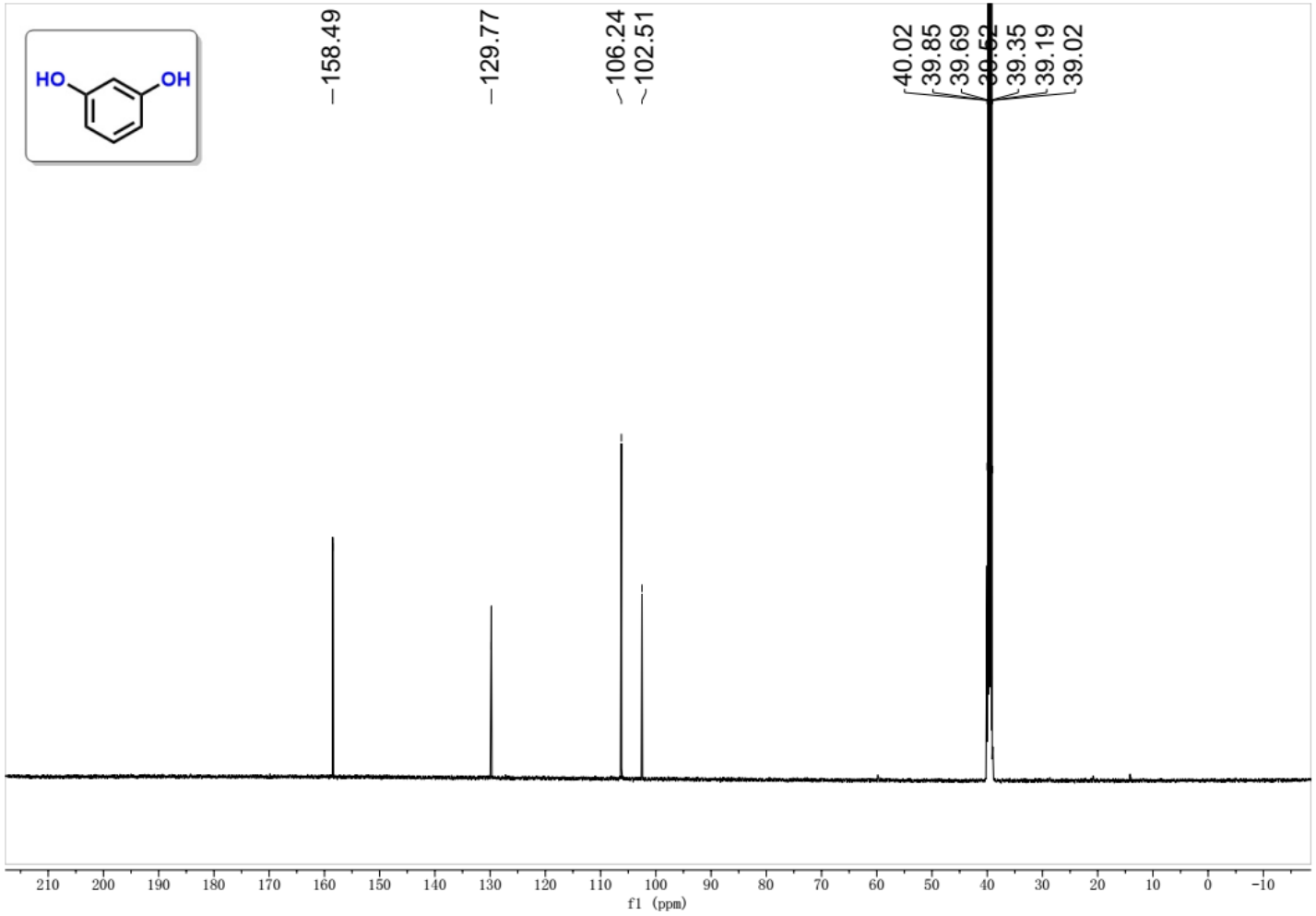
^{13}C NMR of 2o


167.99
157.26
135.82
129.19
118.10
118.01
114.50
40.02
39.94
39.85
39.78
39.69
39.61
39.52
39.44
39.35
39.19
39.02
H2N
O
OH
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

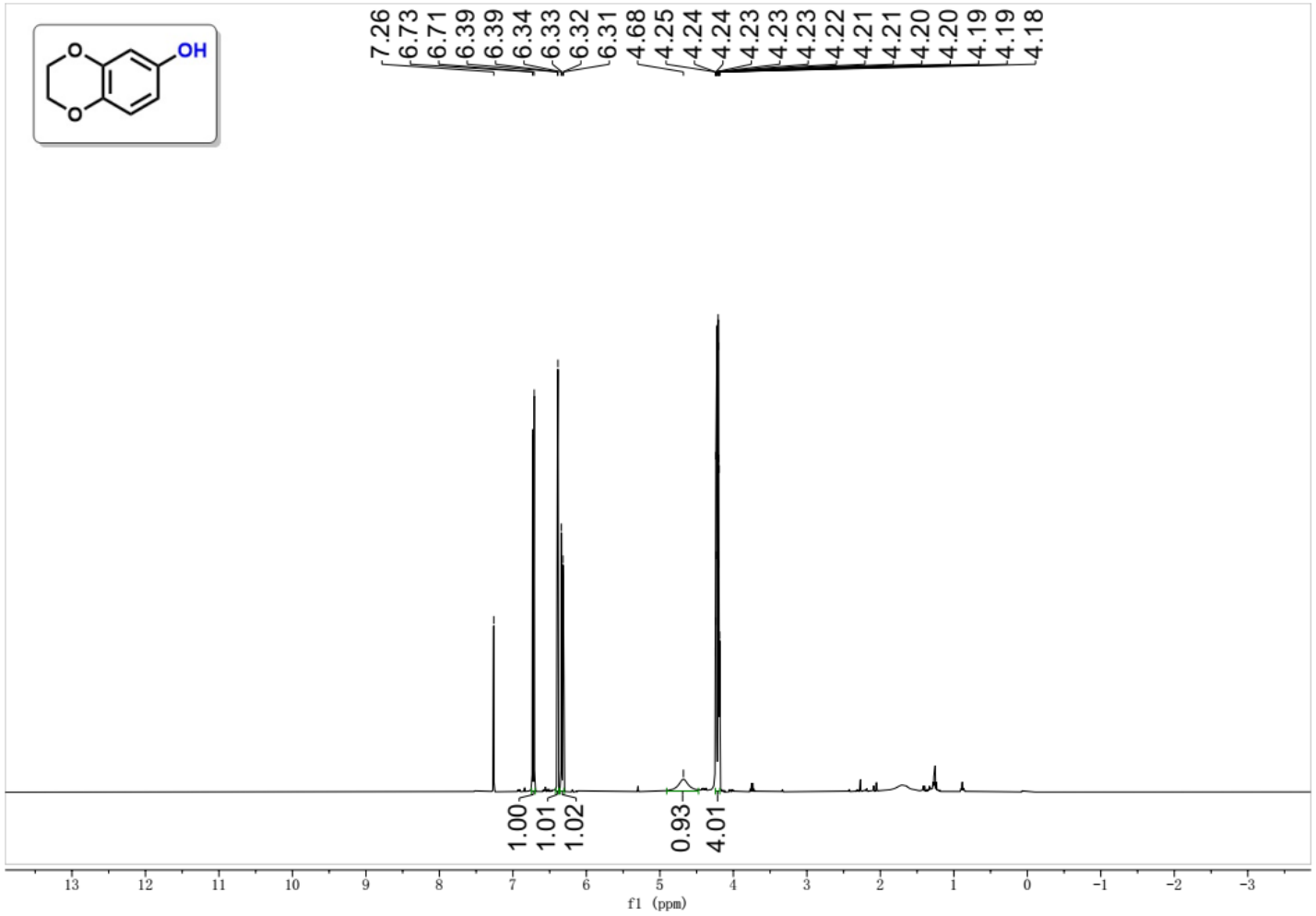
^{1}H NMR of 2p


HO
OH
9.16
6.92
6.90
6.89
6.19
6.18
6.18
6.17
6.16
2.50
2.50
2.50
2.01
1.00
3.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 −3
f1 (ppm)

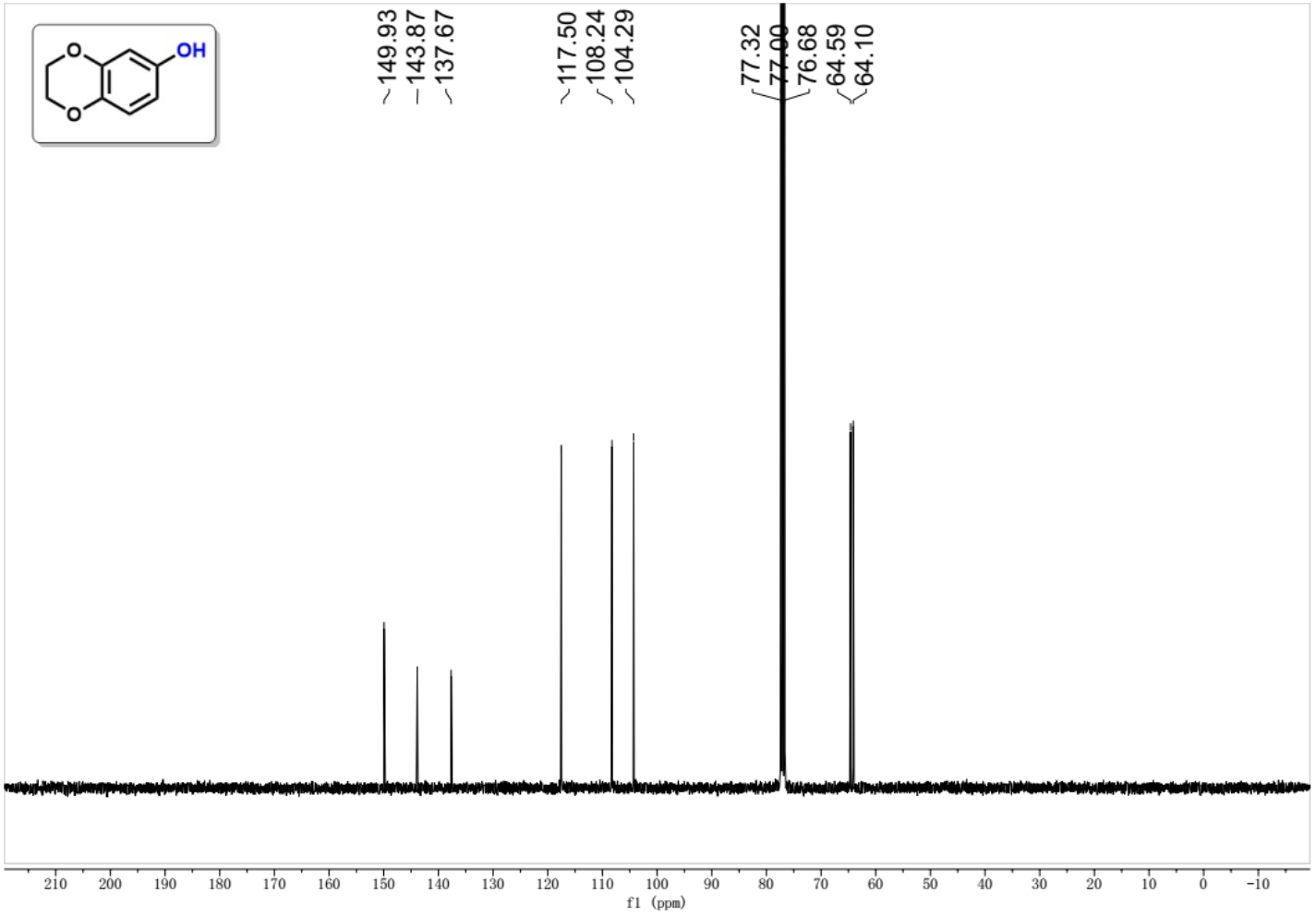
^{13}C NMR of 2p


HO
OH
158.49
129.77
106.24
102.51
40.02
39.85
39.69
39.52
39.35
39.19
39.02
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

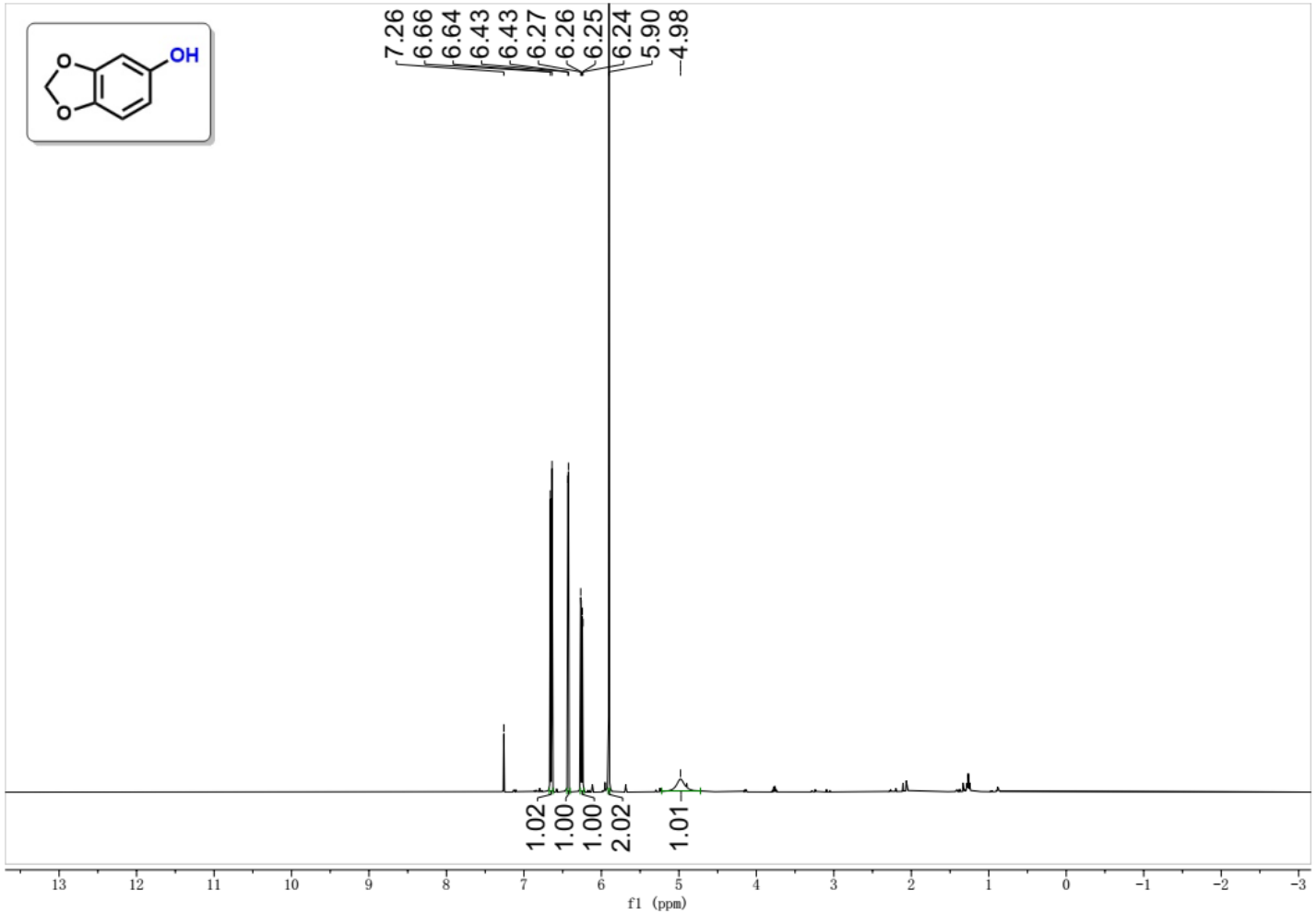
^{1}H NMR of 2q


OH
7.26
6.73
6.71
6.39
6.39
6.34
6.33
6.32
6.31
4.68
4.25
4.24
4.24
4.23
4.23
4.23
4.22
4.21
4.21
4.20
4.20
4.19
4.19
4.18
1.00
1.01
1.02
0.93
4.01
13
12
11
10
9
8
7
6
5
4
3
2
1
0
−1
−2
−3
f1 (ppm)

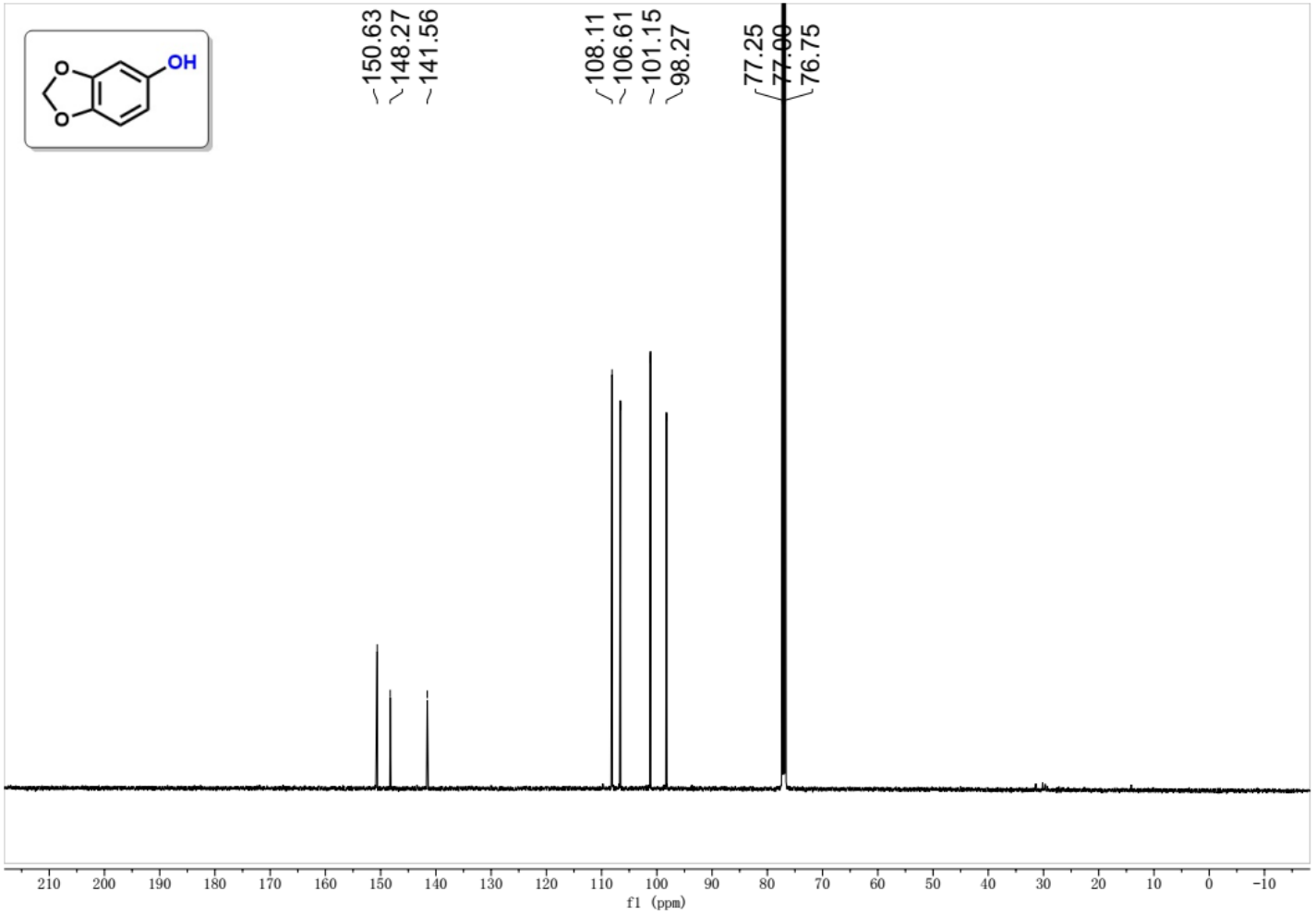
^{13}C NMR of 2q


OH
149.93
143.87
137.67
117.50
108.24
104.29
77.32
77.00
76.68
64.59
64.10
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

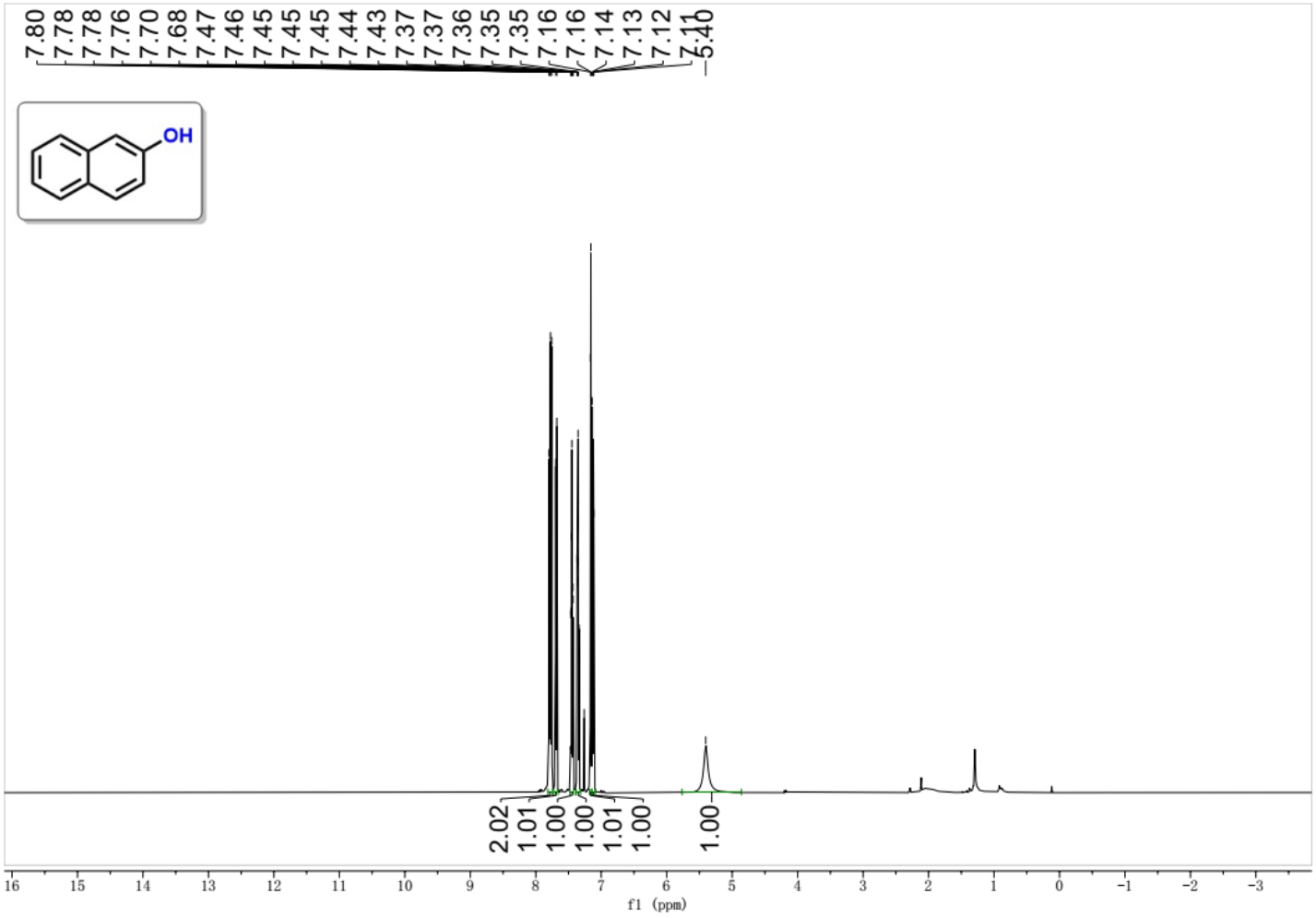
^{1}H NMR of 2r


OH
7.26
6.66
6.64
6.43
6.43
6.27
6.26
6.25
6.24
5.90
4.98
1.02
1.00
1.00
2.02
1.01
13
12
11
10
9
8
7
6
5
4
3
2
1
0
−1
−2
−3
f1 (ppm)

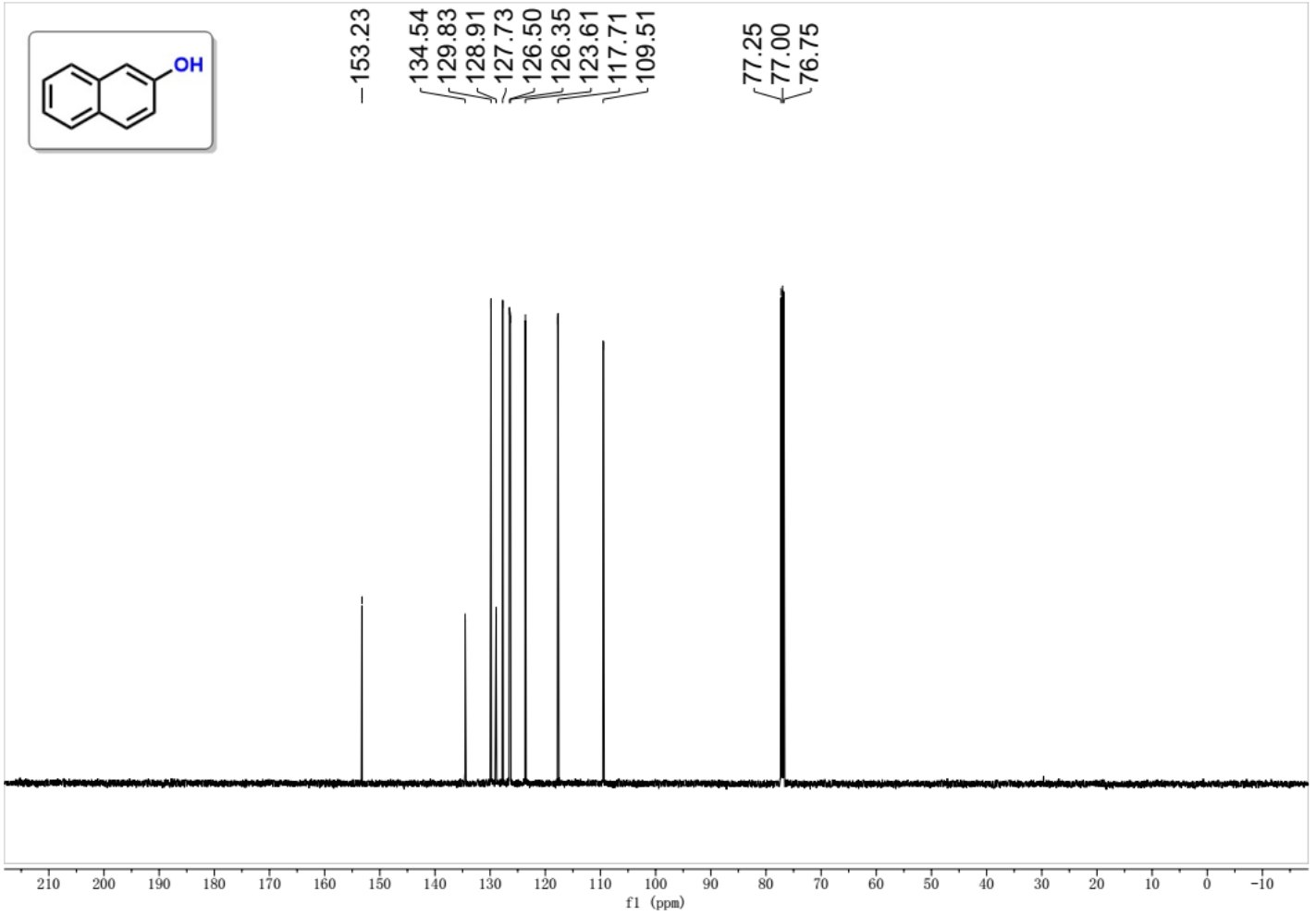
^{13}C NMR of 2r


OH
150.63
148.27
141.56
108.11
106.61
101.15
98.27
77.25
77.00
76.75
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

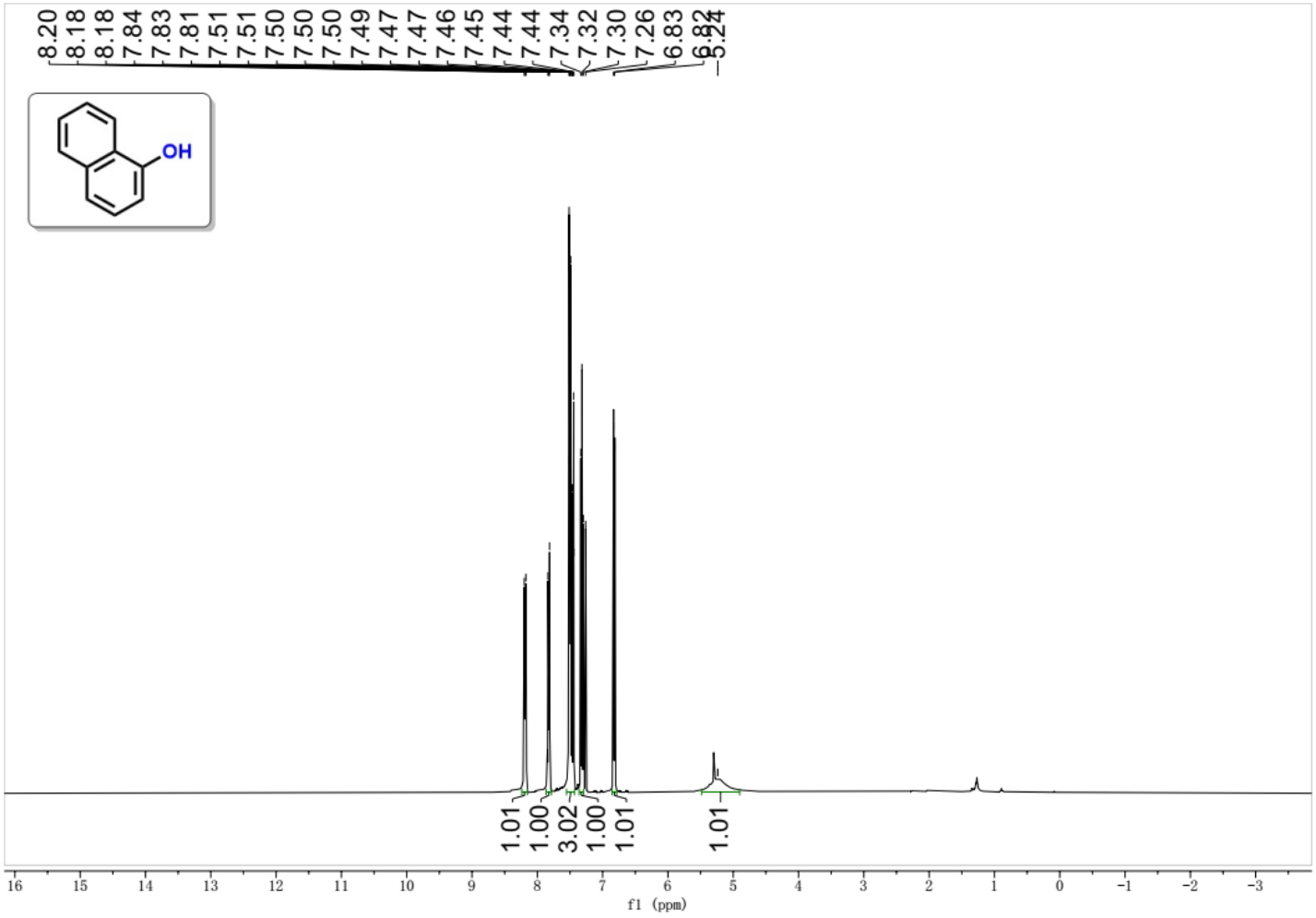
^{1}H NMR of 2s


7.80
7.78
7.78
7.76
7.70
7.68
7.47
7.46
7.45
7.45
7.45
7.44
7.43
7.37
7.37
7.36
7.35
7.35
7.16
7.16
7.14
7.13
7.12
7.11
5.40
OH
2.02
1.01
1.00
1.00
1.01
1.00
1.00
f1 (ppm)

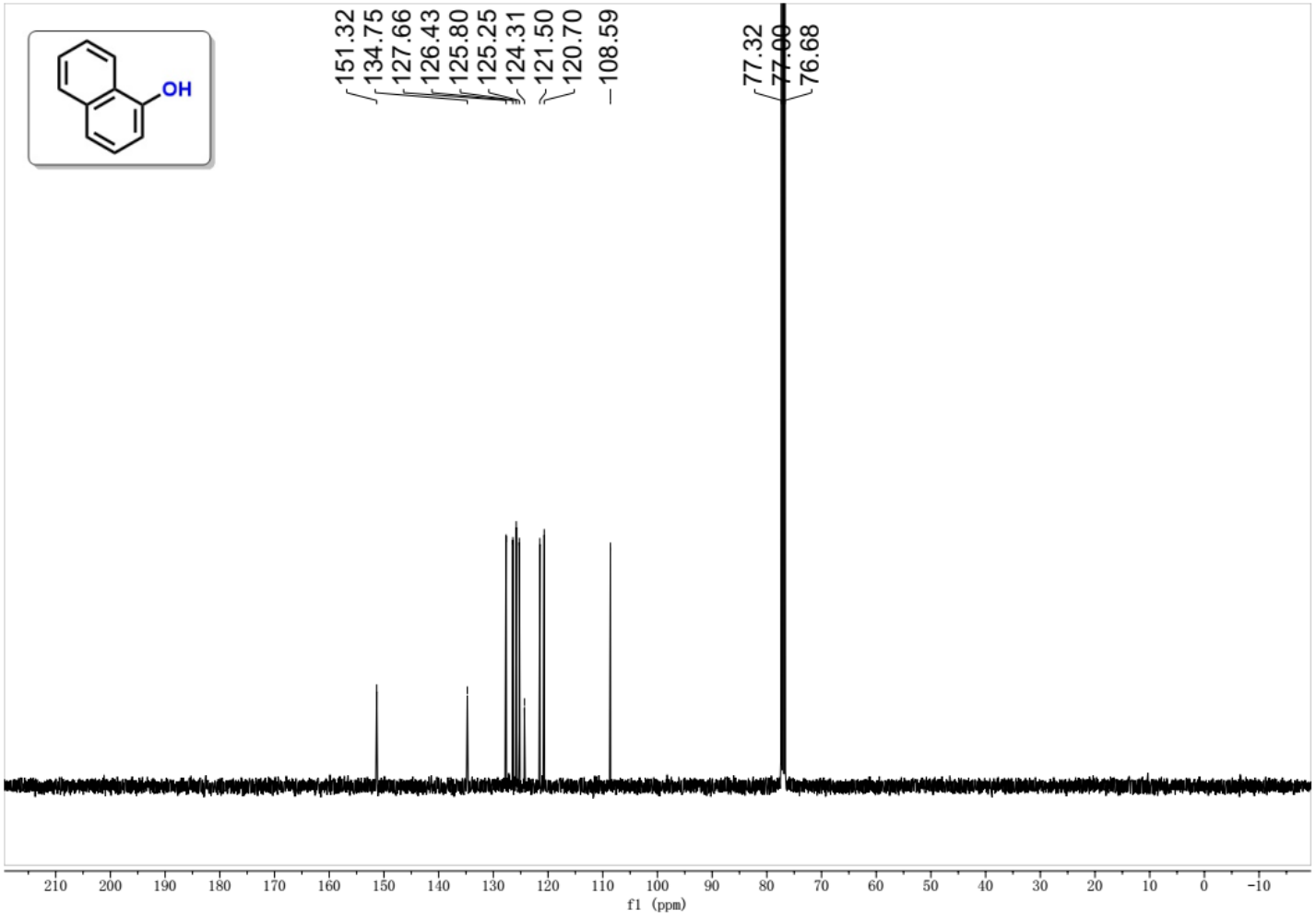
^{13}C NMR of 2s


OH
153.23
134.54
129.83
128.91
127.73
126.50
126.35
123.61
117.71
109.51
77.25
77.00
76.75
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

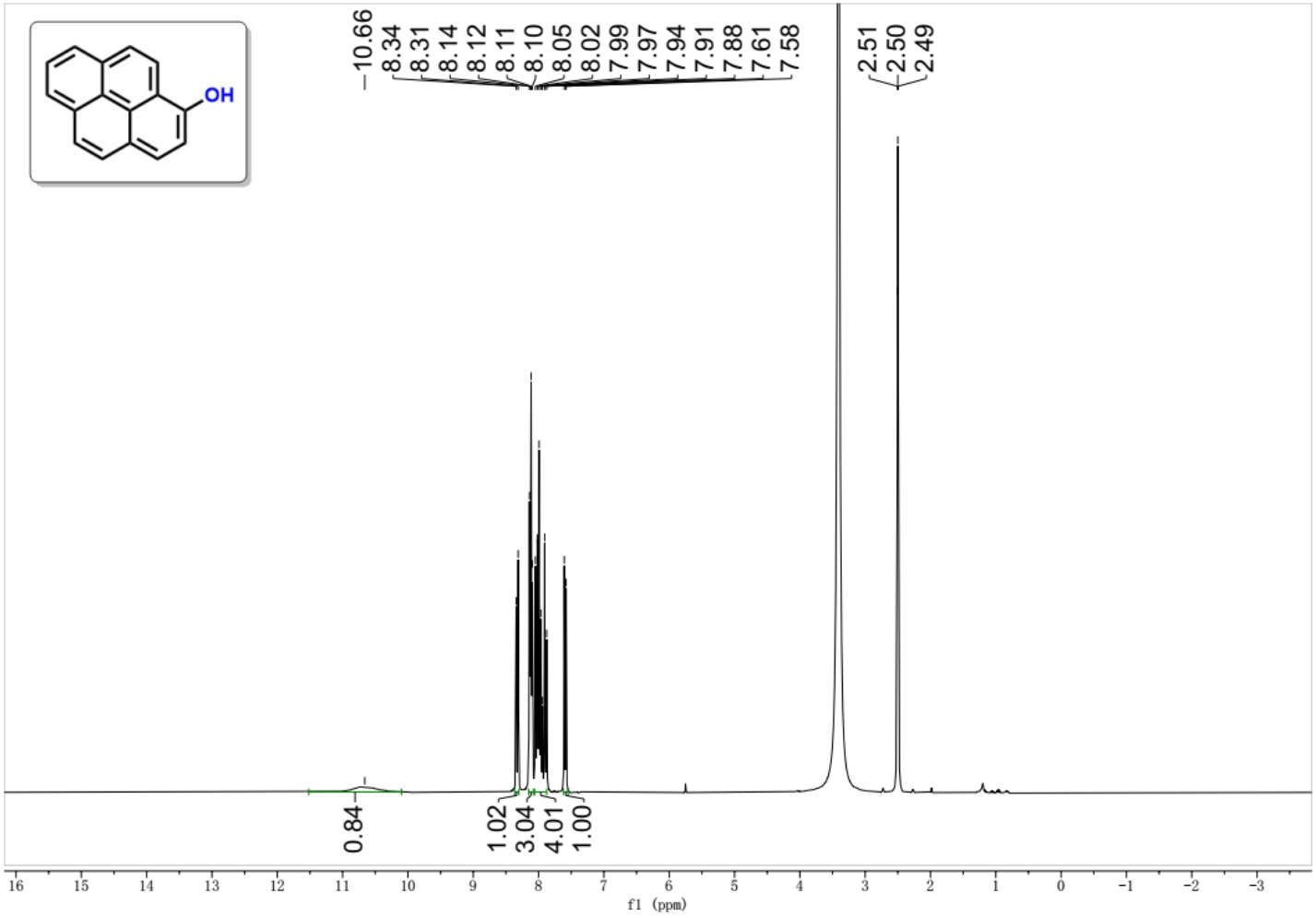
^{1}H NMR of 2t


8.20
8.18
8.18
7.84
7.83
7.81
7.51
7.51
7.50
7.50
7.50
7.49
7.47
7.47
7.46
7.45
7.44
7.44
7.34
7.32
7.30
7.26
6.83
6.82
5.24
OH
1.01
1.00
3.02
1.00
1.01
1.01
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
−1
−2
−3
f1 (ppm)

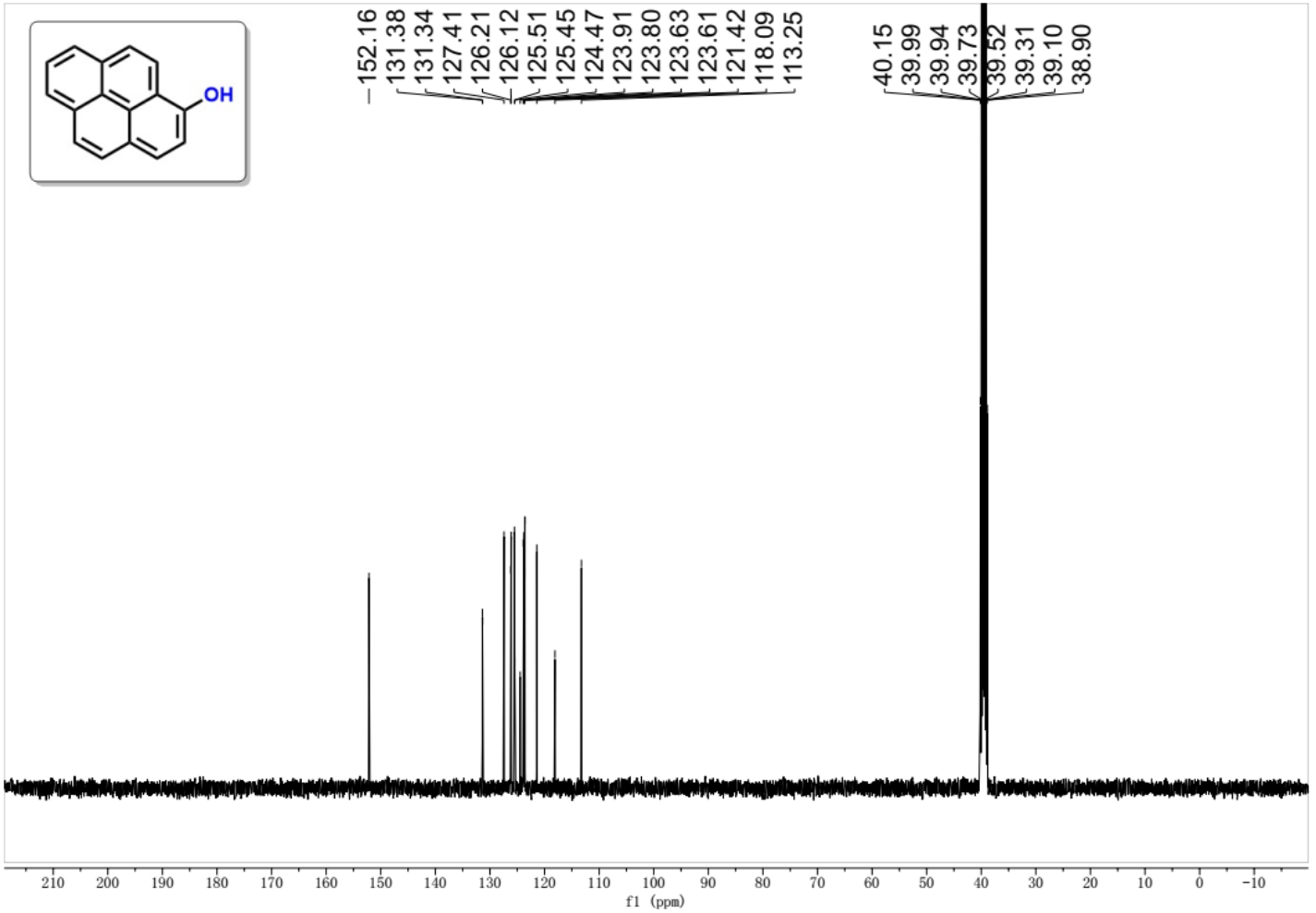
^{13}C NMR of 2t


OH
151.32
134.75
127.66
126.43
125.80
125.25
124.31
121.50
120.70
108.59
77.32
77.00
76.68
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

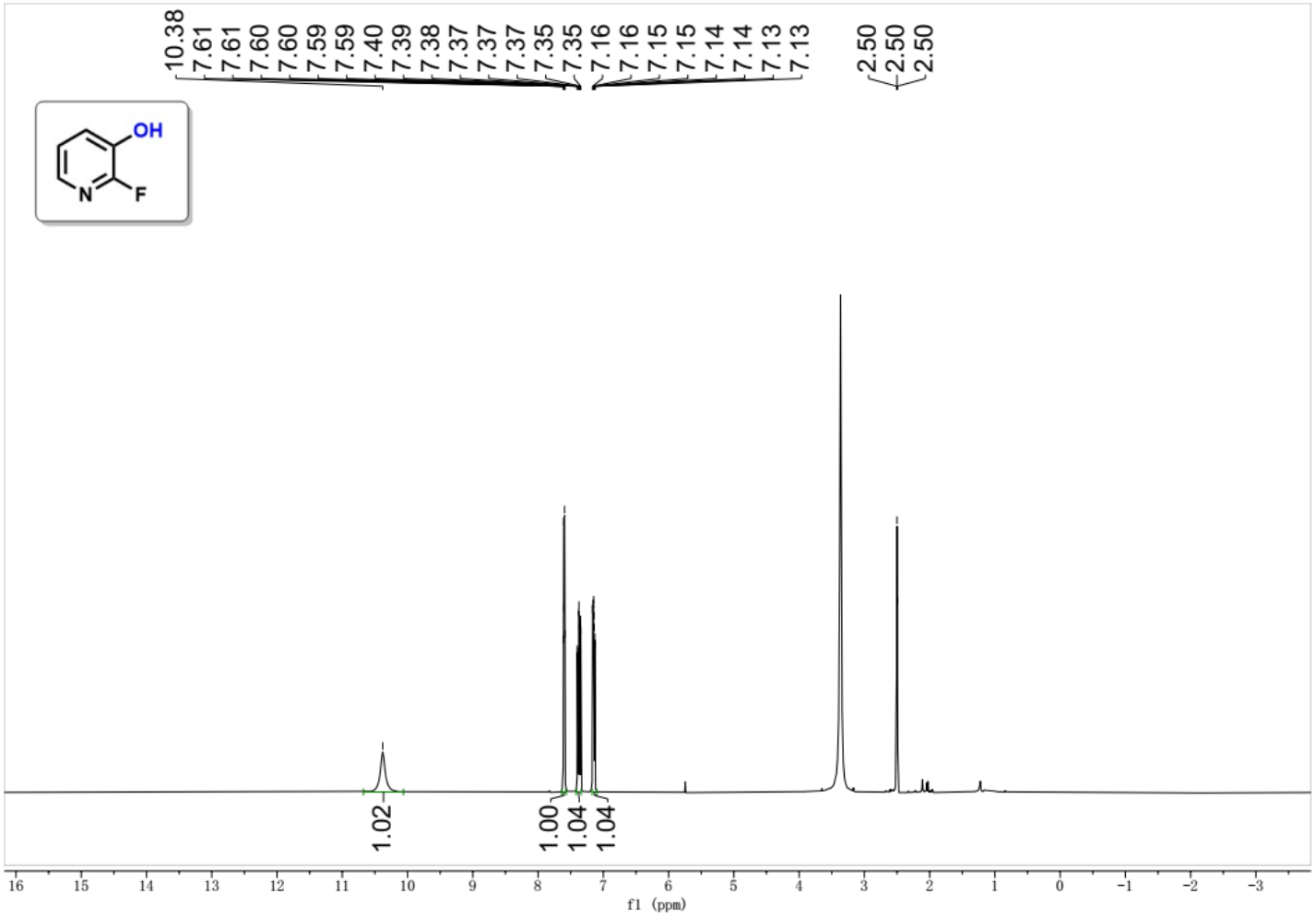
^{1}H NMR of 2u


OH
10.66
8.34
8.31
8.14
8.12
8.11
8.10
8.05
8.02
7.99
7.97
7.94
7.91
7.88
7.61
7.58
2.51
2.50
2.49
0.84
1.02
3.04
4.01
1.00
f1 (ppm)

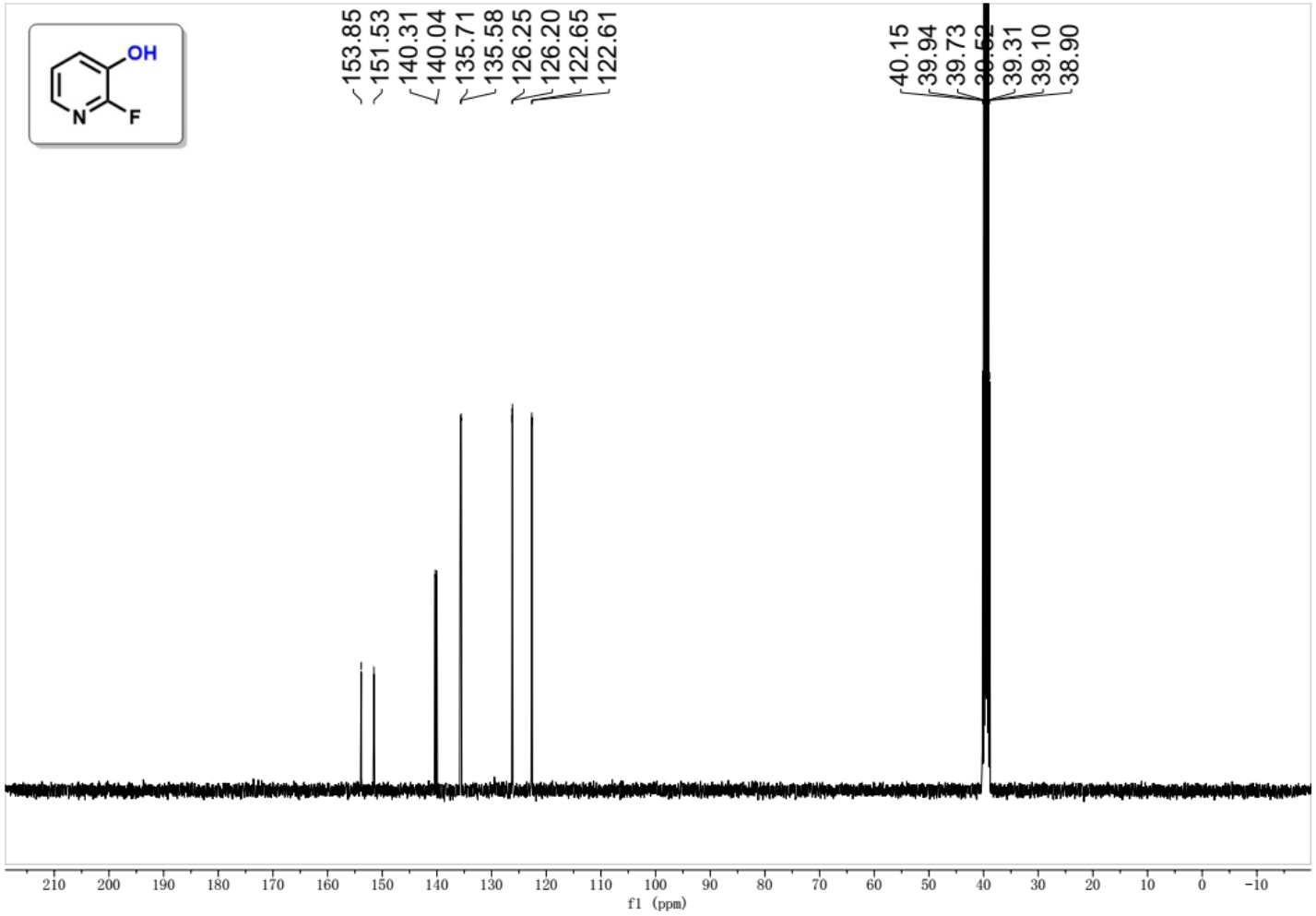
^{13}C NMR of 2u


OH
152.16
131.38
131.34
127.41
126.21
126.12
125.51
125.45
124.47
123.91
123.80
123.63
123.61
121.42
118.09
113.25
40.15
39.99
39.94
39.73
39.52
39.31
39.10
38.90
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

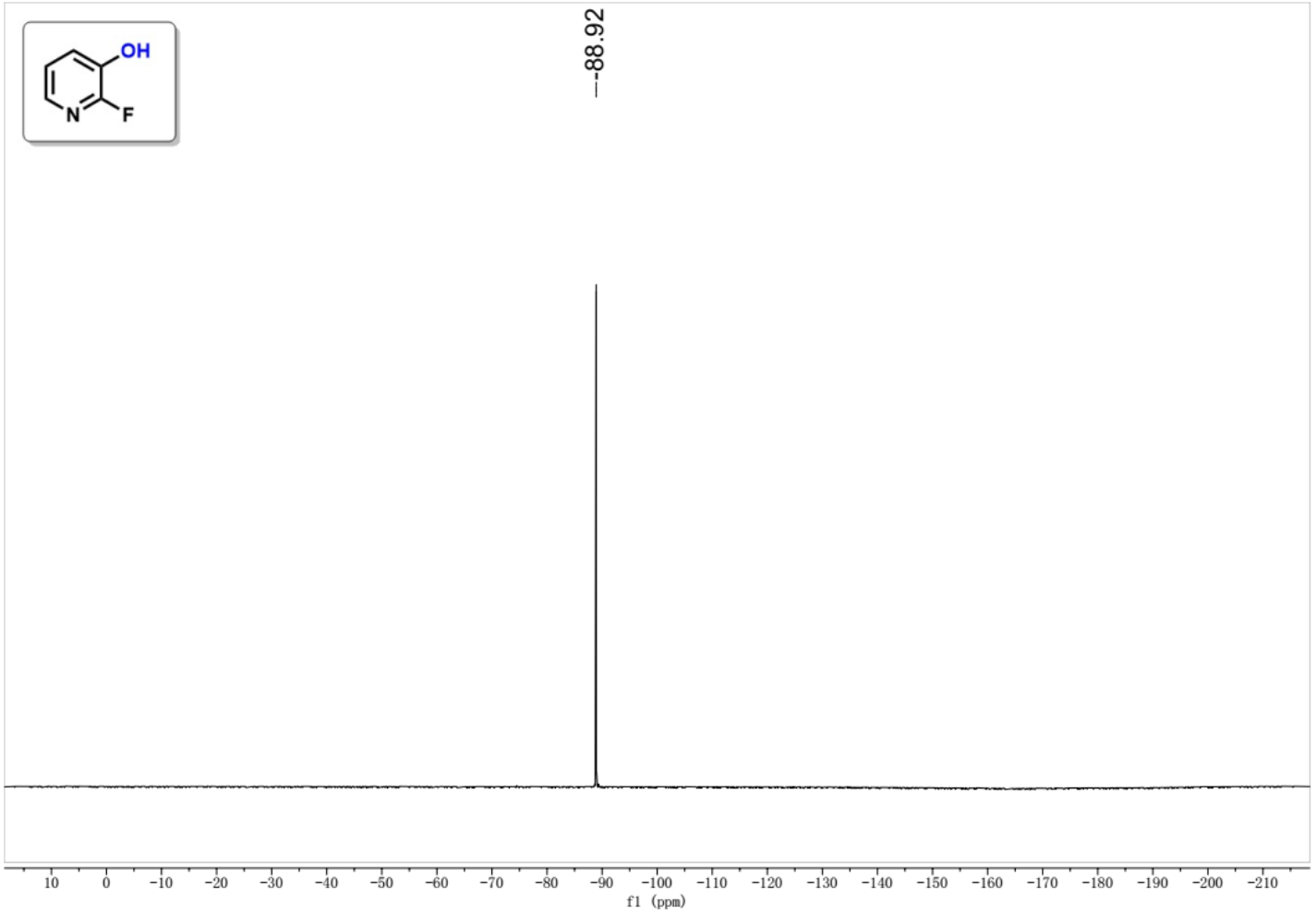
^{1}H NMR of 2v


10.38
7.61
7.61
7.60
7.60
7.59
7.59
7.40
7.39
7.38
7.37
7.37
7.37
7.35
7.35
7.16
7.16
7.15
7.15
7.14
7.14
7.13
7.13
2.50
2.50
2.50
OH
N
F
1.02
1.00
1.04
1.04
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
−1
−2
−3
f1 (ppm)

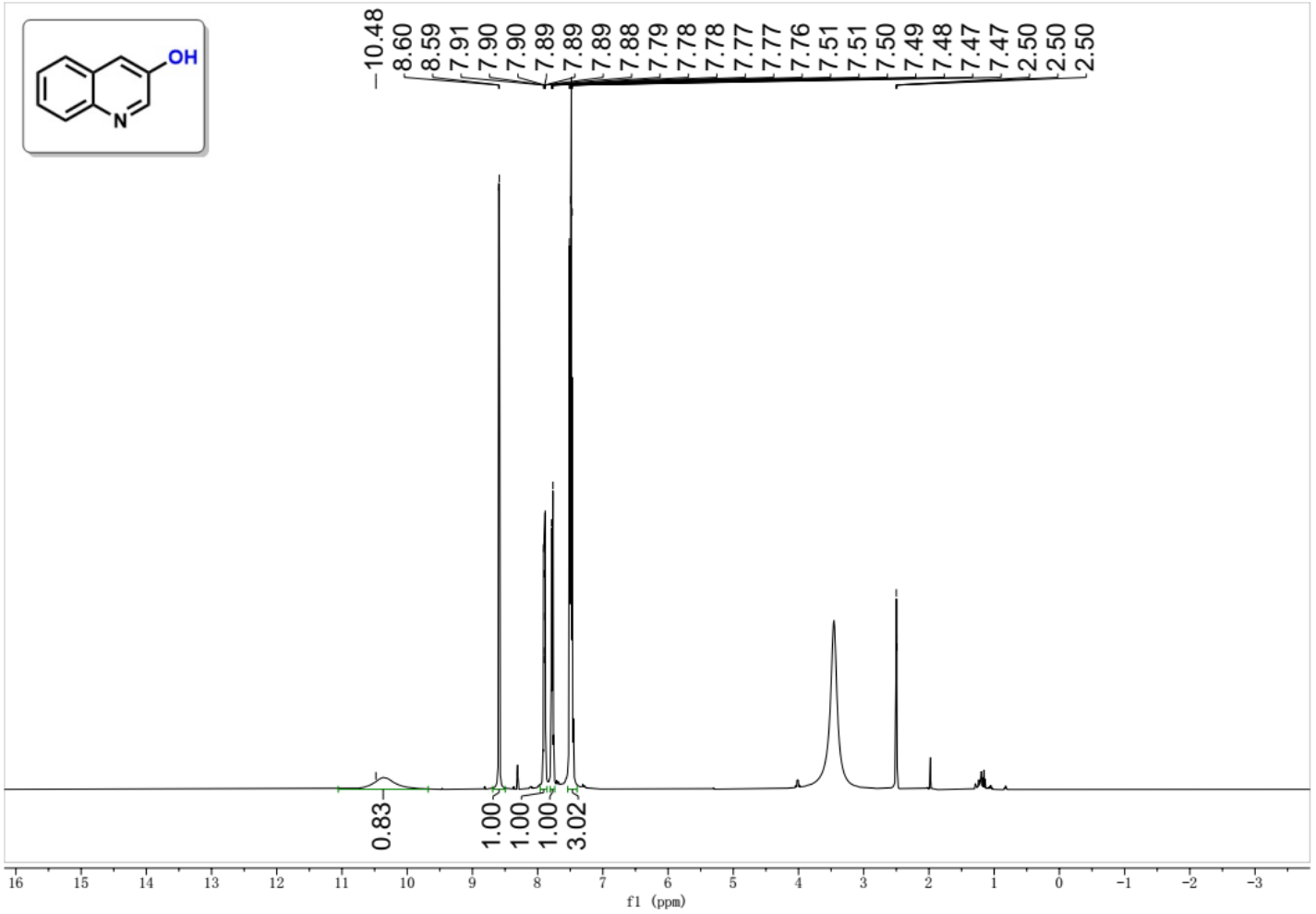
^{13}C NMR of 2v


OH
N
F
153.85
151.53
140.31
140.04
135.71
135.58
126.25
126.20
122.65
122.61
40.15
39.94
39.73
39.52
39.31
39.10
38.90
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

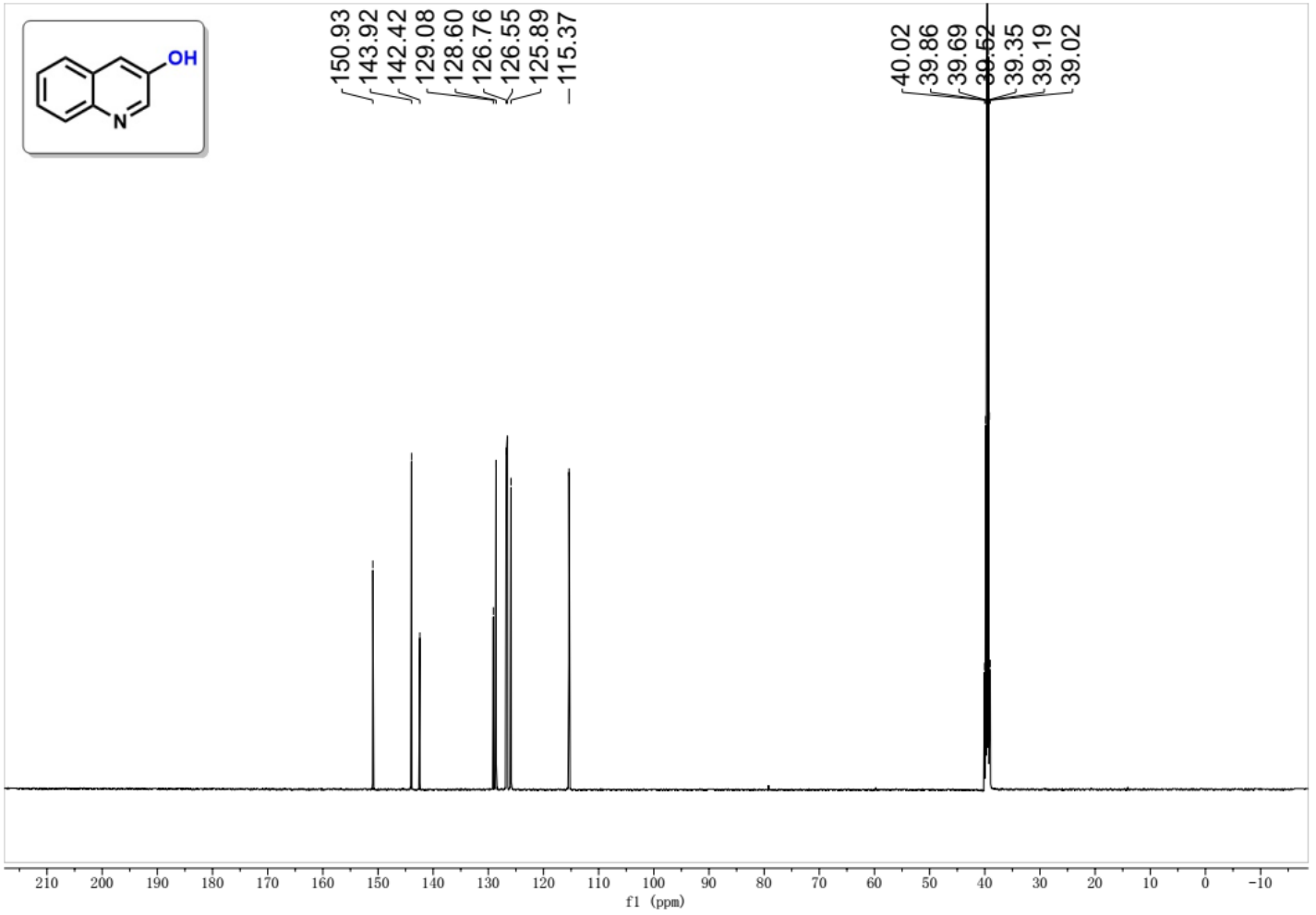
^{19}F NMR of 2v


OH
N
F
-88.92
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

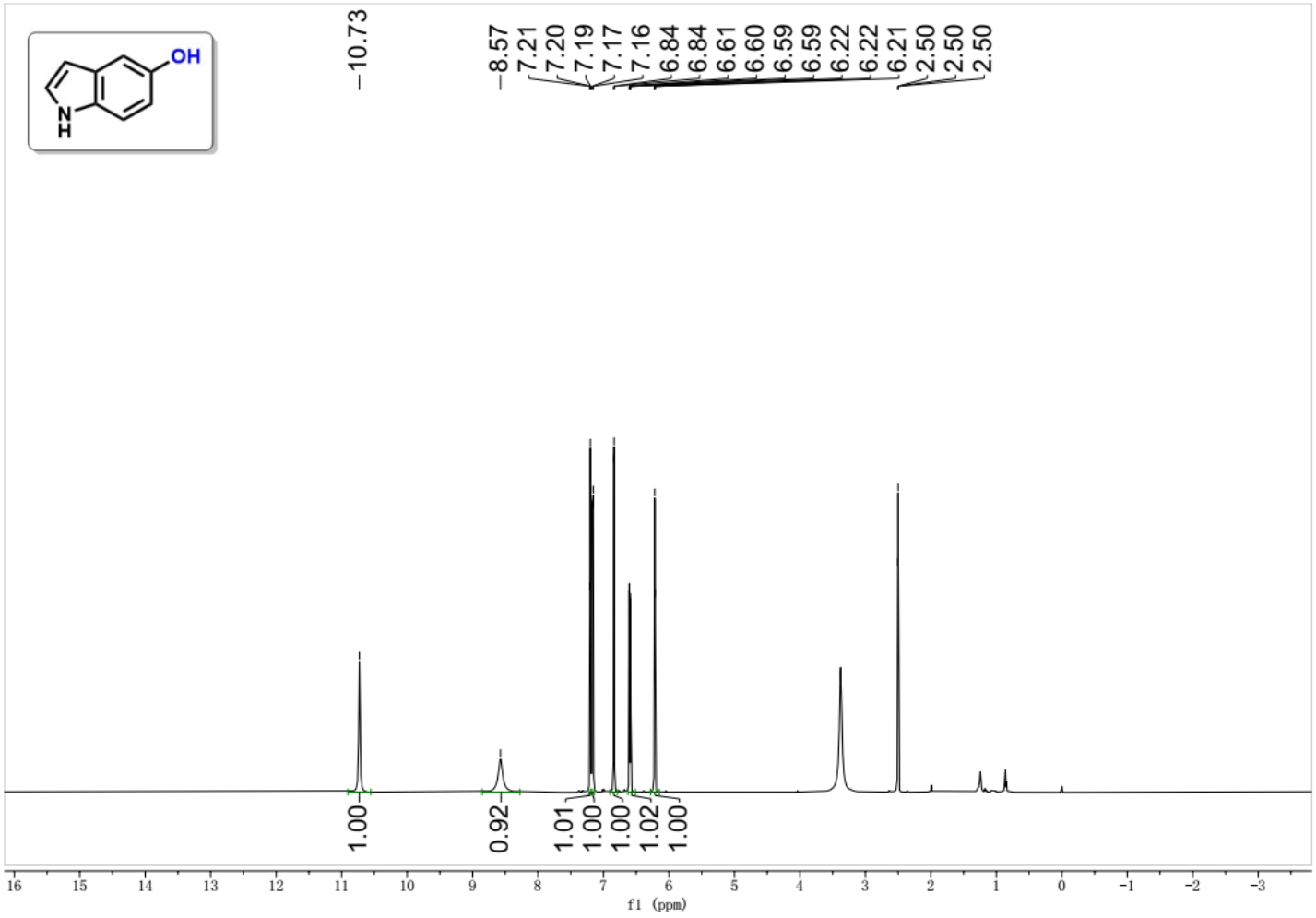
^{1}H NMR of 2w


OH
N
10.48
8.60
8.59
7.91
7.90
7.90
7.89
7.89
7.89
7.88
7.79
7.78
7.78
7.77
7.77
7.76
7.51
7.51
7.50
7.49
7.48
7.47
7.47
2.50
2.50
2.50
0.83
1.00
1.00
1.00
3.02
f1 (ppm)

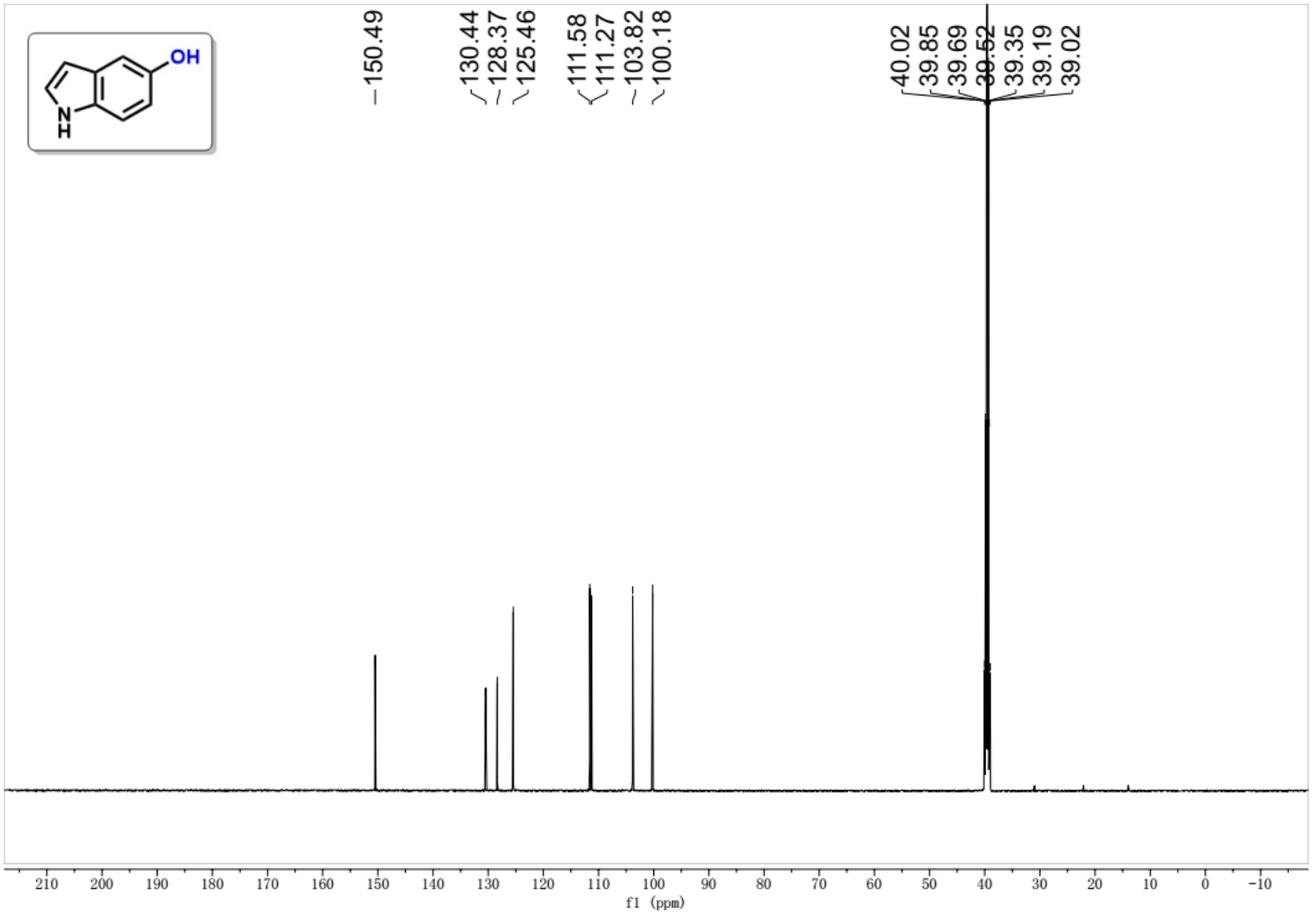
^{13}C NMR of 2w


OH
N
150.93
143.92
142.42
129.08
128.60
126.76
126.55
125.89
115.37
40.02
39.86
39.69
39.52
39.35
39.19
39.02
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

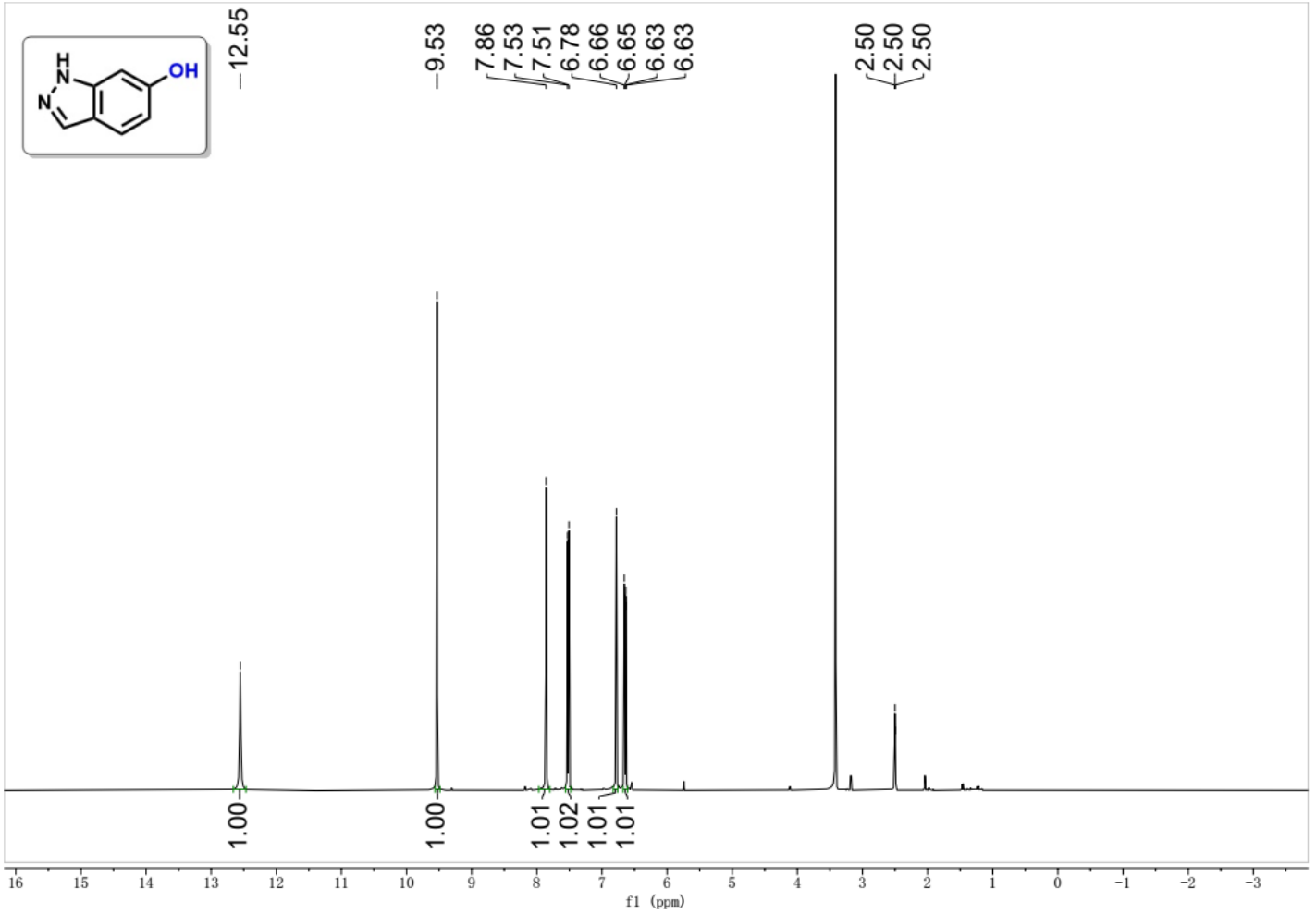
^{1}H NMR of 2x


OH
N
H
10.73
8.57
7.21
7.20
7.19
7.17
7.16
6.84
6.84
6.61
6.60
6.59
6.59
6.22
6.22
6.21
2.50
2.50
2.50
1.00
0.92
1.01
1.00
1.00
1.02
1.00
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
−1
−2
−3
f1 (ppm)

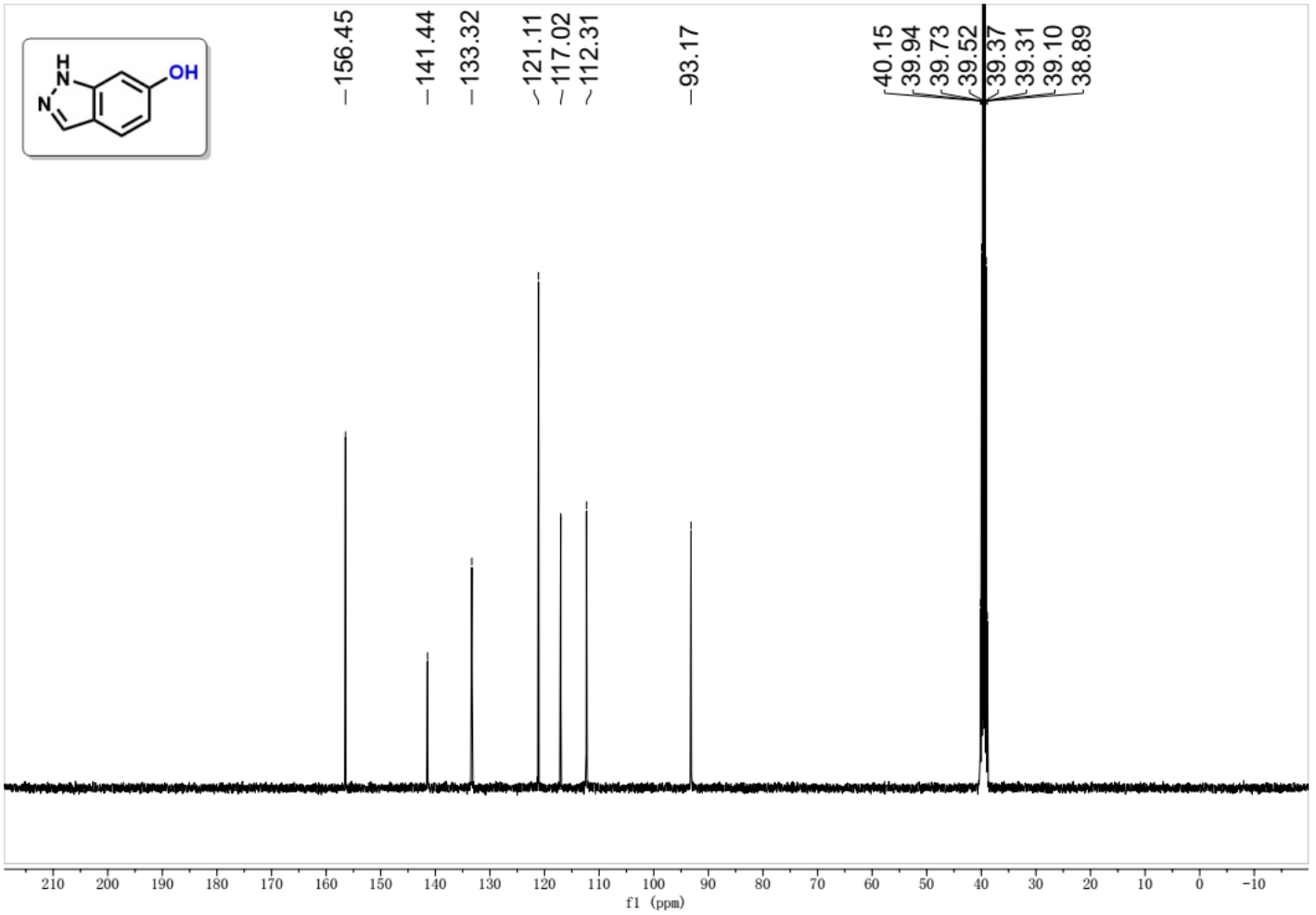
^{13}C NMR of 2x


OH
N
H
150.49
130.44
128.37
125.46
111.58
111.27
103.82
100.18
40.02
39.85
39.69
39.52
39.35
39.19
39.02
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

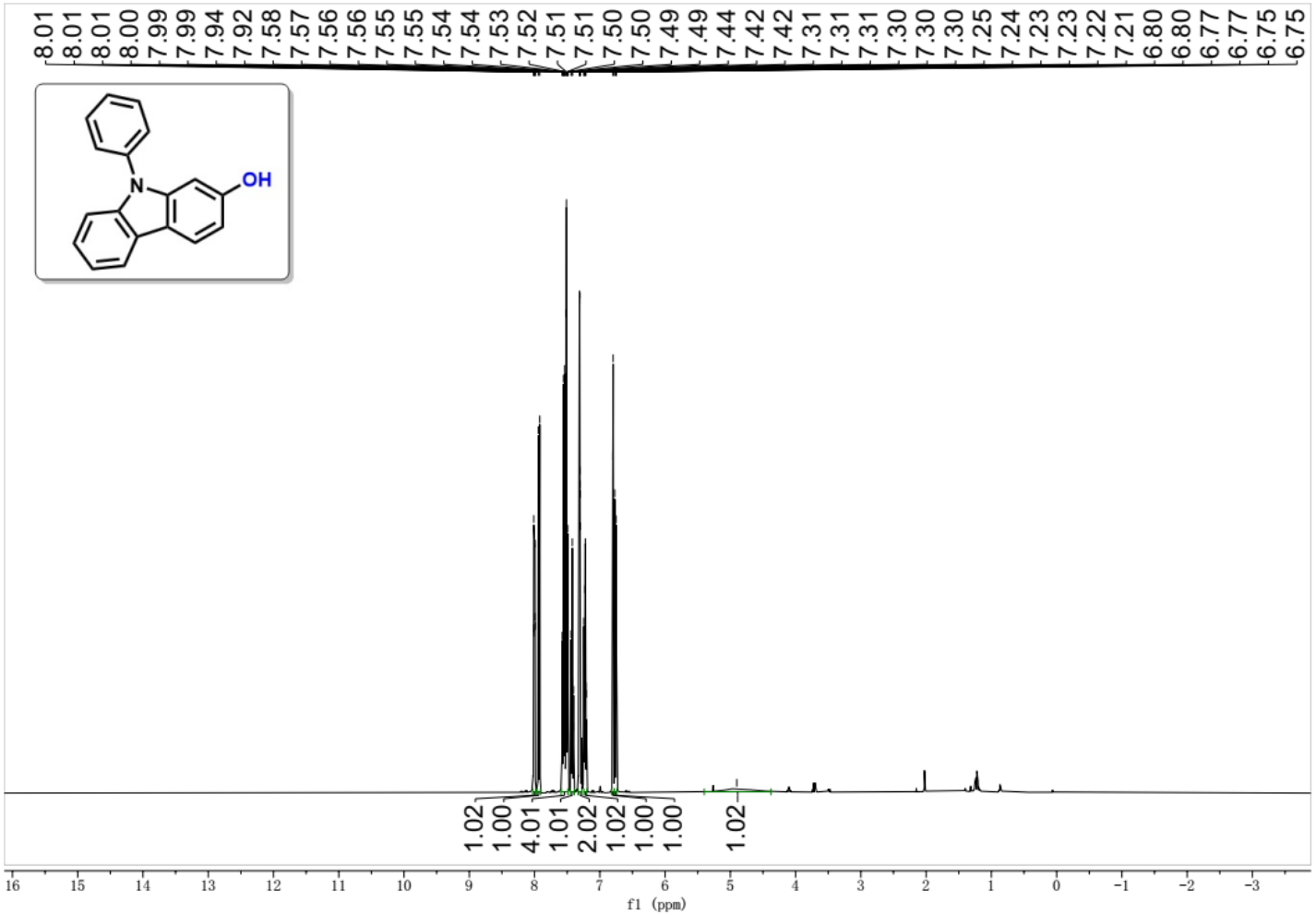
^{1}H NMR of 2y


12.55
9.53
7.86
7.53
7.51
6.78
6.66
6.65
6.63
6.63
2.50
2.50
2.50
OH
1.00
1.00
1.01
1.02
1.01
1.01
f1 (ppm)

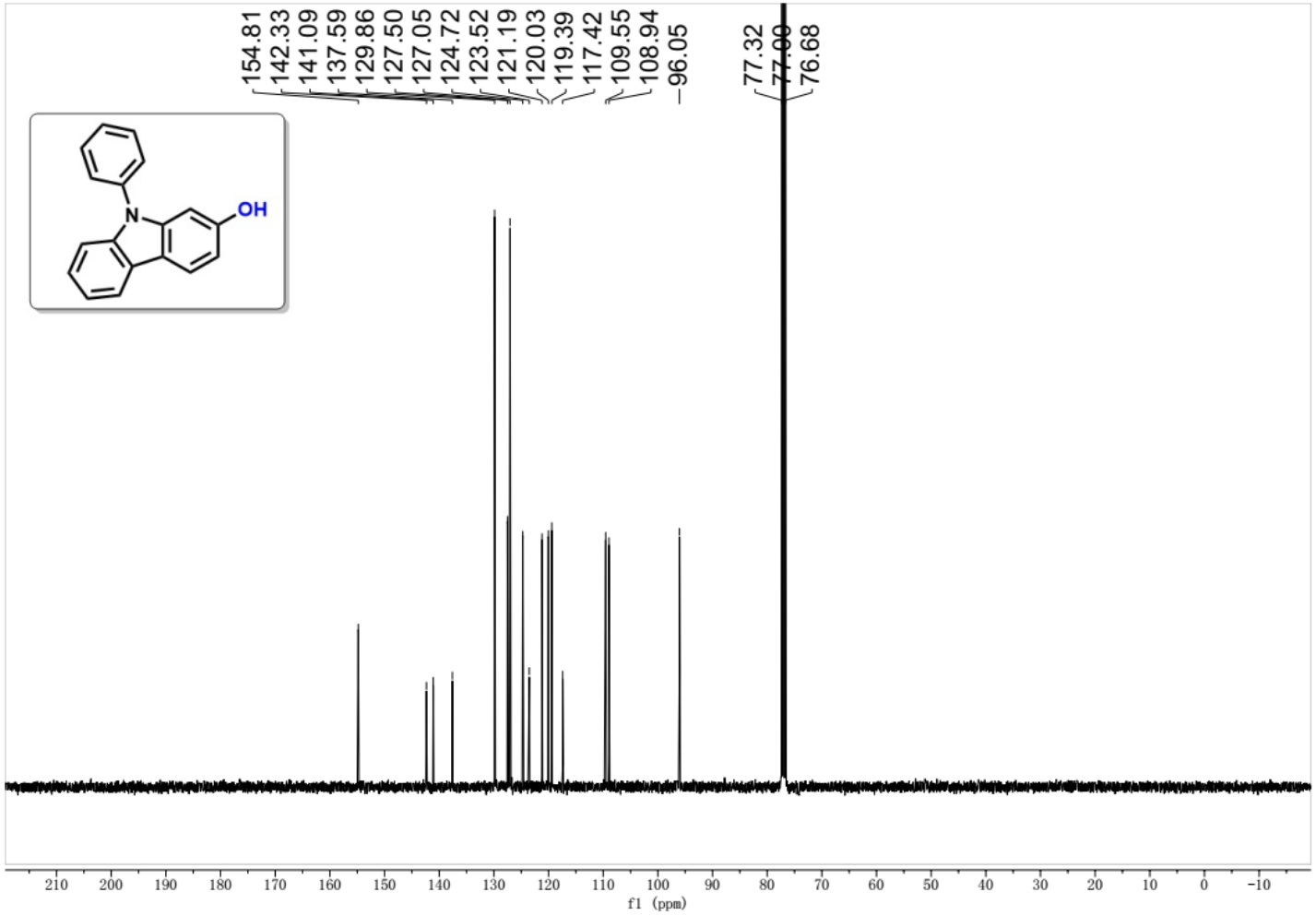
^{13}C NMR of 2y


OH
156.45
141.44
133.32
121.11
117.02
112.31
93.17
40.15
39.94
39.73
39.52
39.37
39.31
39.10
38.89
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

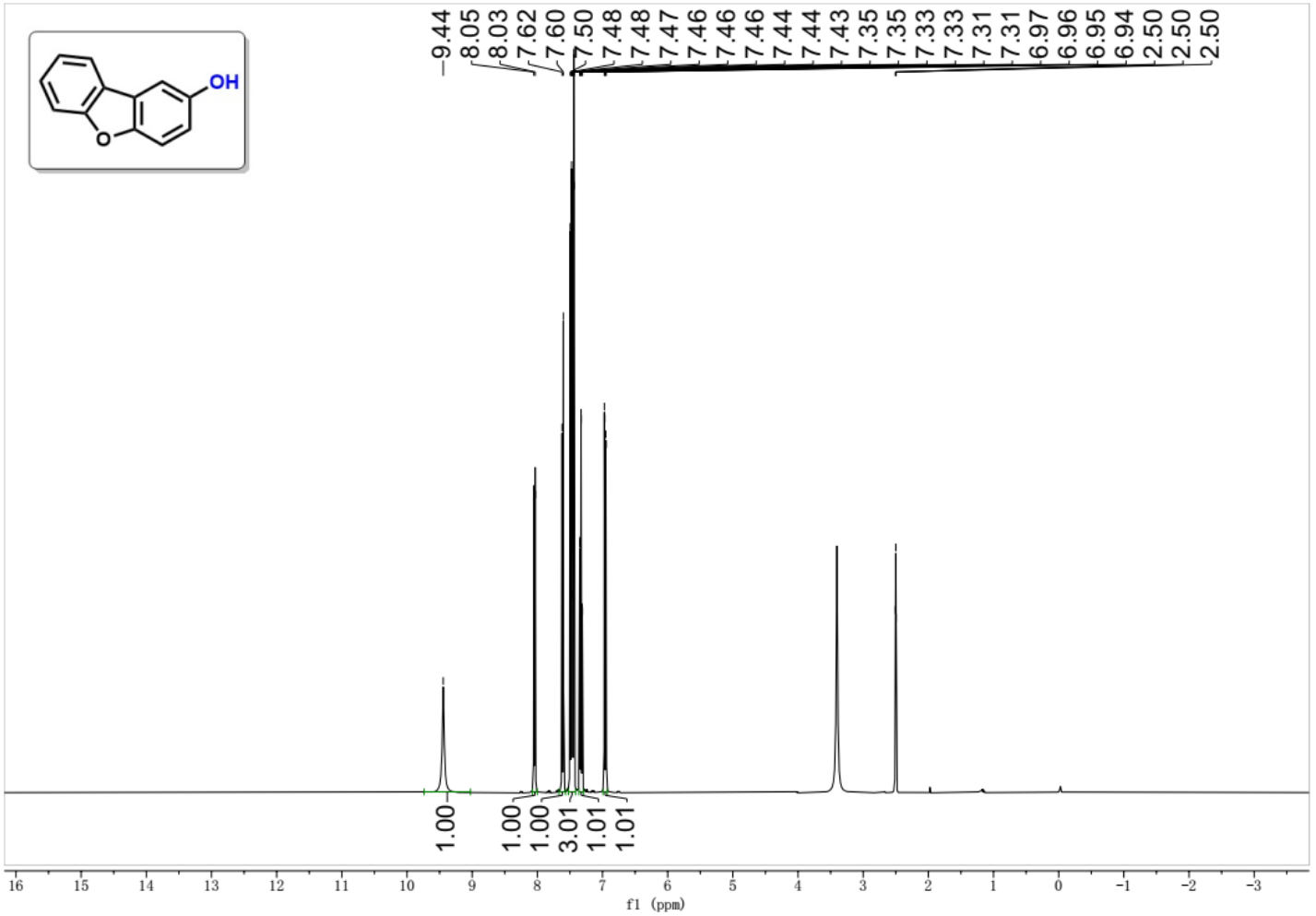
^{1}H NMR of 2z


8.01 8.01 8.01 8.00 7.99 7.99 7.94 7.92 7.58 7.57 7.56 7.56 7.55 7.55 7.54 7.54 7.53 7.52 7.51 7.51 7.50 7.50 7.49 7.49 7.44 7.42 7.42 7.31 7.31 7.31 7.30 7.30 7.30 7.25 7.24 7.23 7.23 7.22 7.21 6.80 6.80 6.77 6.77 6.75 6.75
OH
N
1.02 1.00 4.01 1.01 2.02 1.02 1.00 1.00 1.02
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 −3
f1 (ppm)

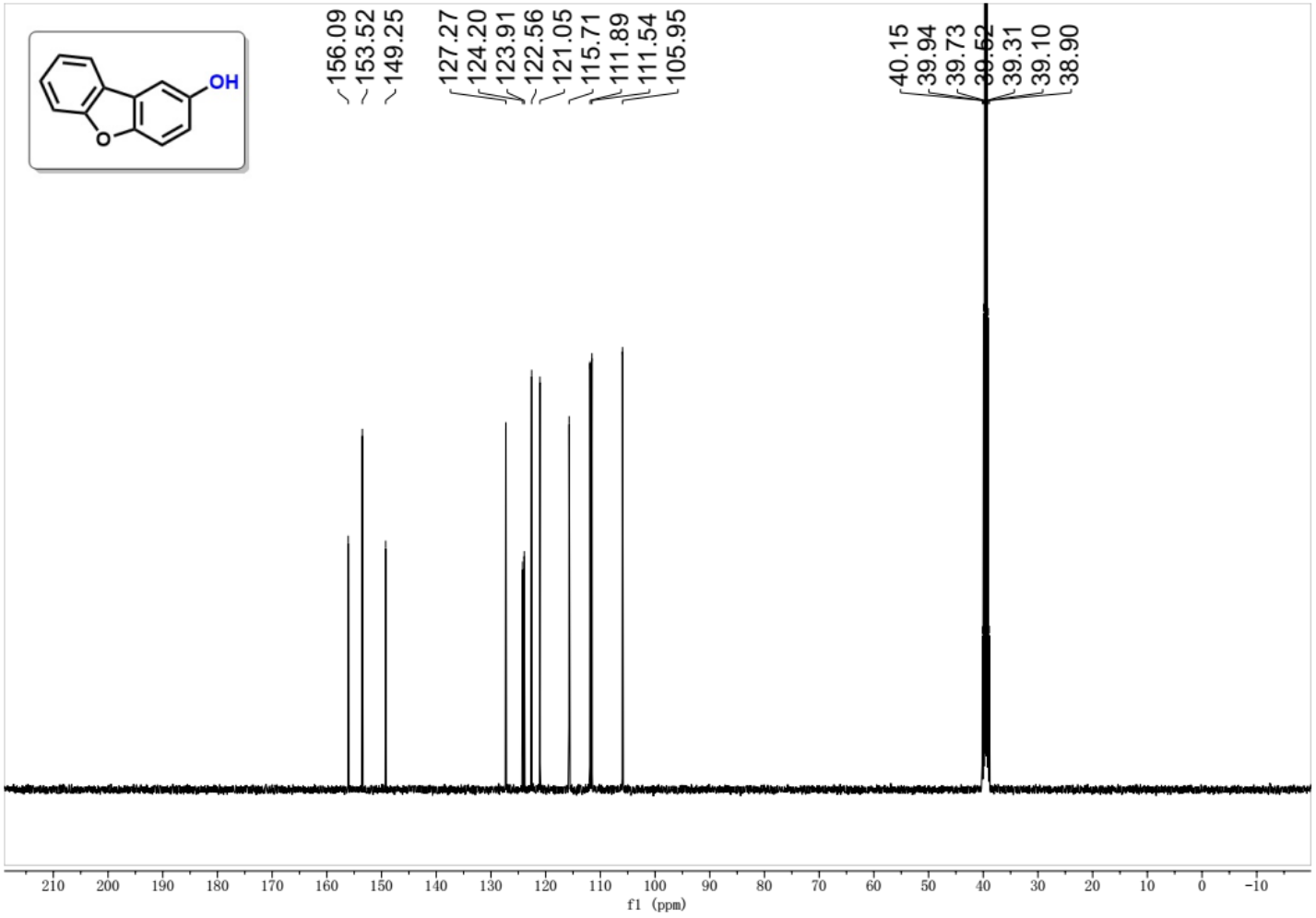
^{13}C NMR of 2z


154.81
142.33
141.09
137.59
129.86
127.50
127.05
124.72
123.52
121.19
120.03
119.39
117.42
109.55
108.94
96.05
77.32
77.00
76.68
OH
N
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

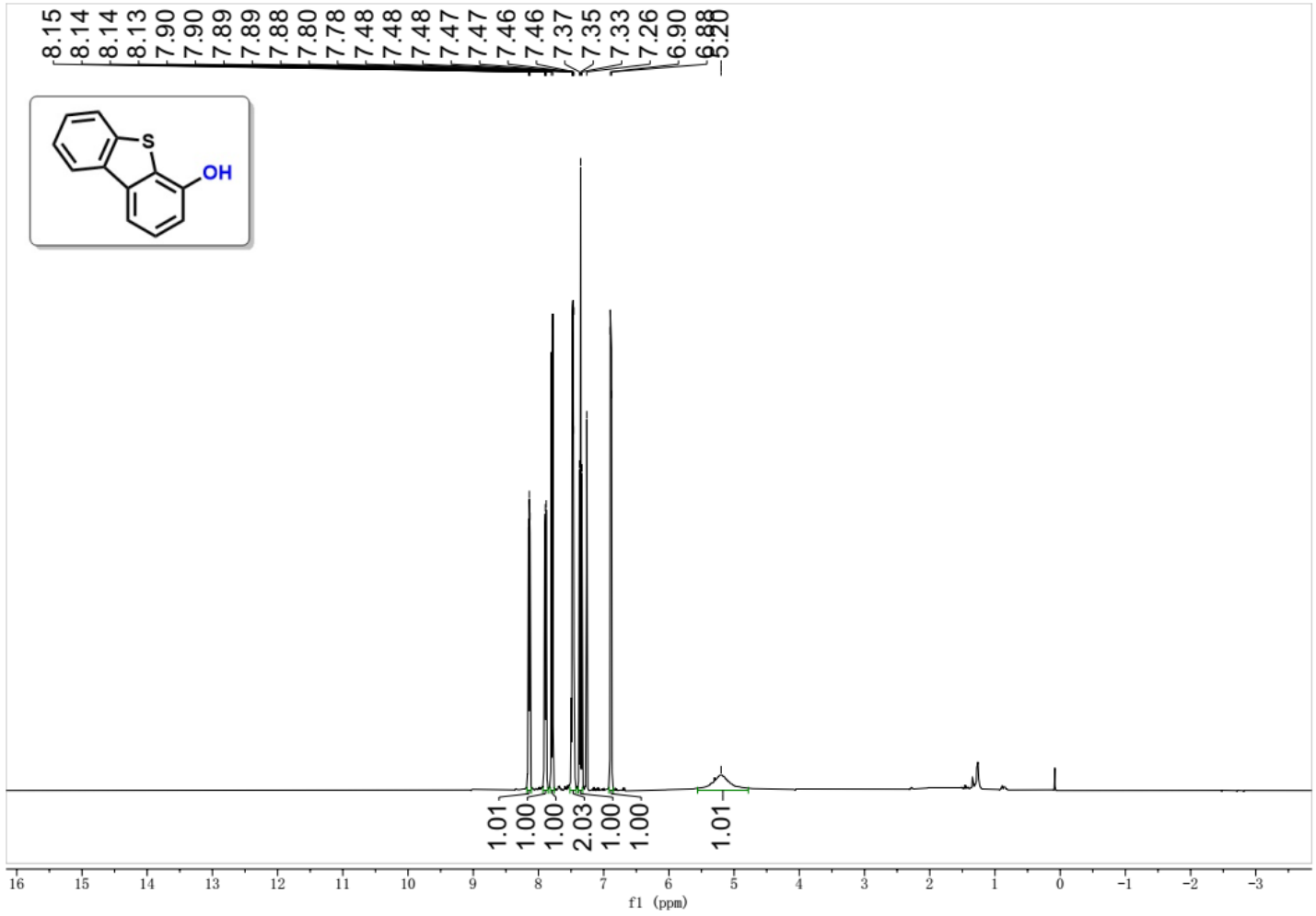
^{1}H NMR of 2aa


OH
9.44
8.05
8.03
7.62
7.60
7.50
7.48
7.48
7.47
7.46
7.46
7.46
7.44
7.44
7.43
7.35
7.35
7.33
7.33
7.31
7.31
6.97
6.96
6.95
6.94
2.50
2.50
2.50
1.00
1.00
1.00
3.01
1.01
1.01
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 −3
f1 (ppm)

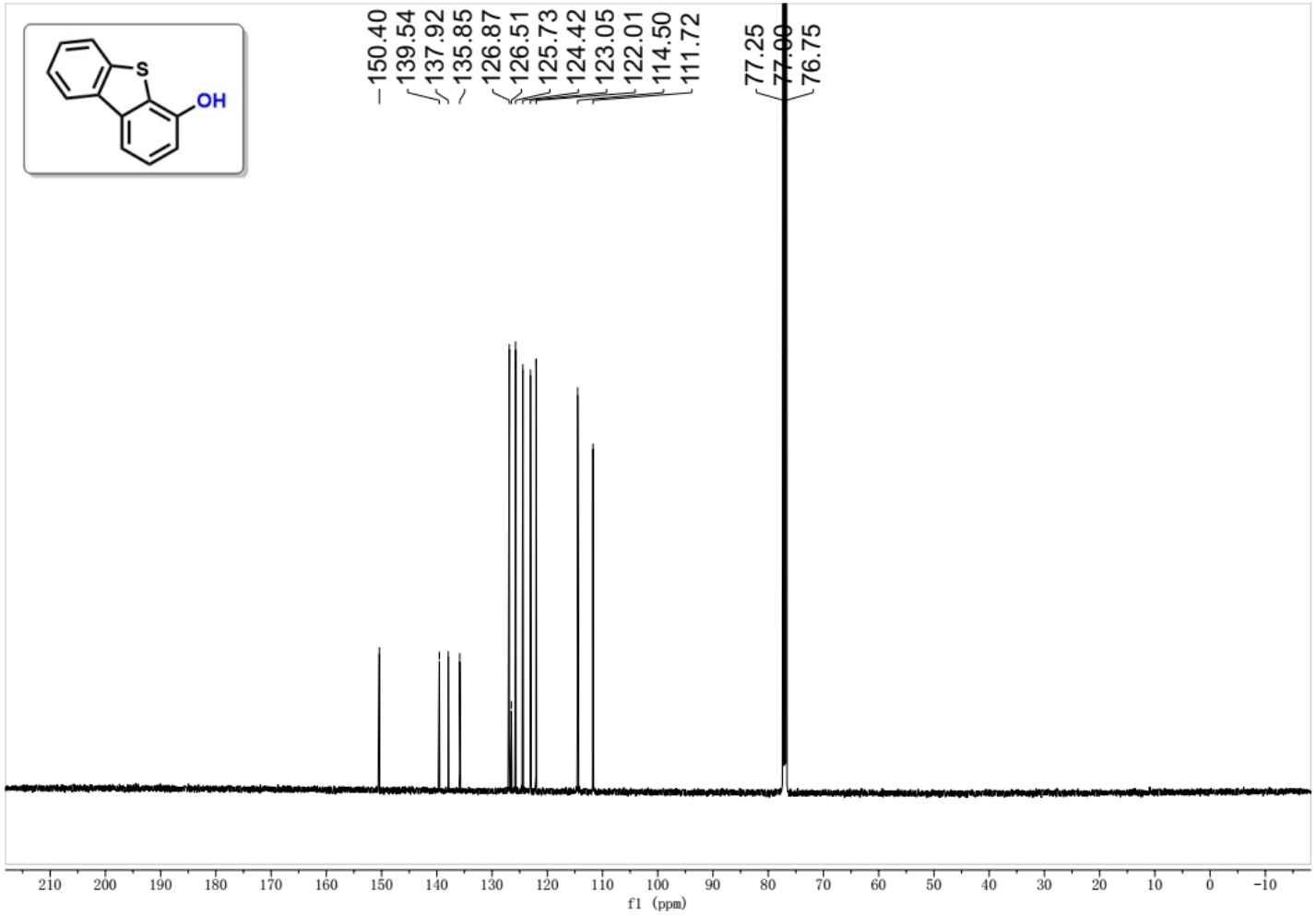
^{13}C NMR of 2aa



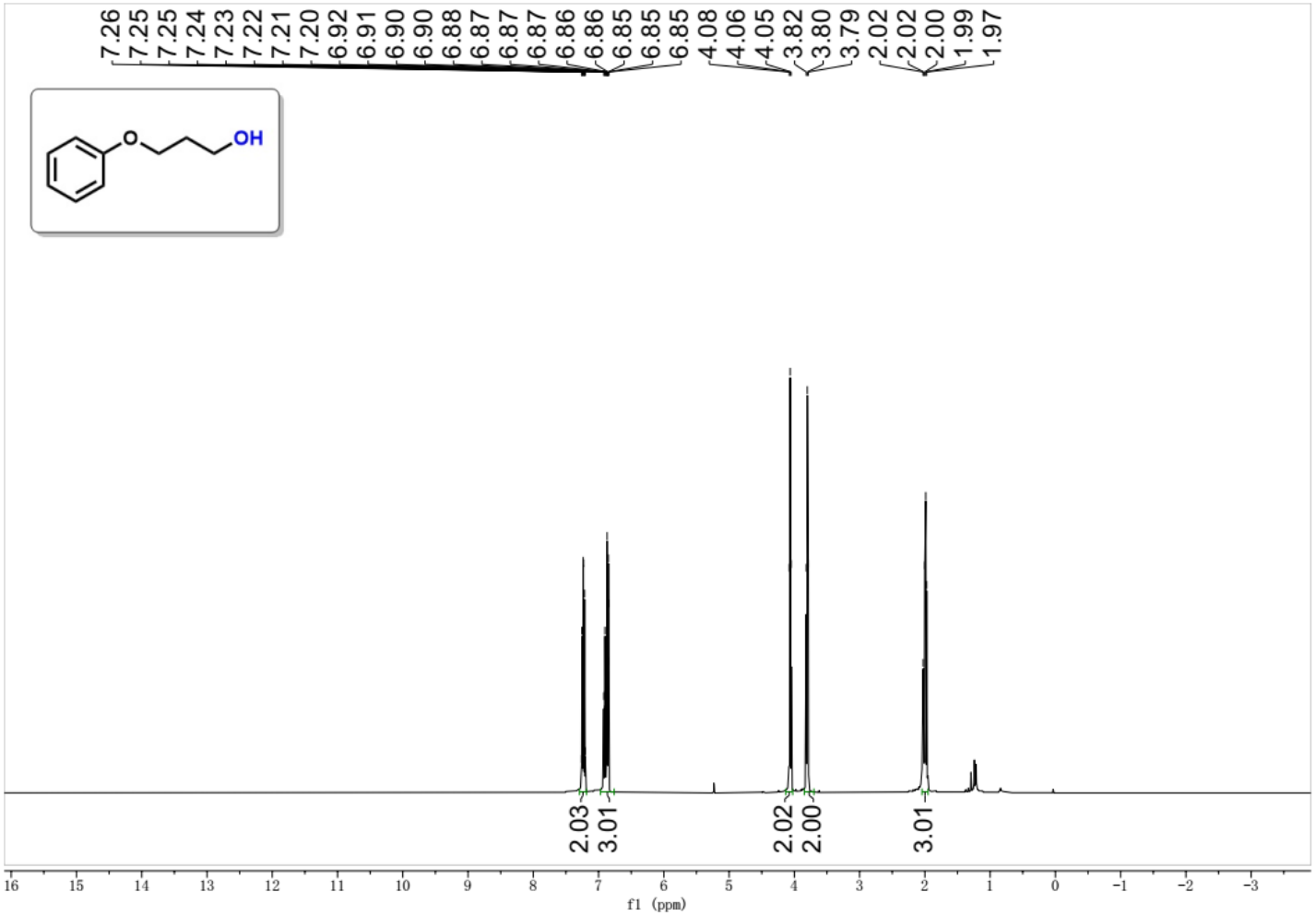
^{1}H NMR of 2ab


8.15
8.14
8.14
8.13
7.90
7.90
7.89
7.89
7.88
7.80
7.78
7.48
7.48
7.48
7.47
7.47
7.46
7.46
7.37
7.35
7.33
7.26
6.90
6.88
5.20
S
OH
1.01
1.00
1.00
2.03
1.00
1.00
1.01
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
−1
−2
−3
f1 (ppm)

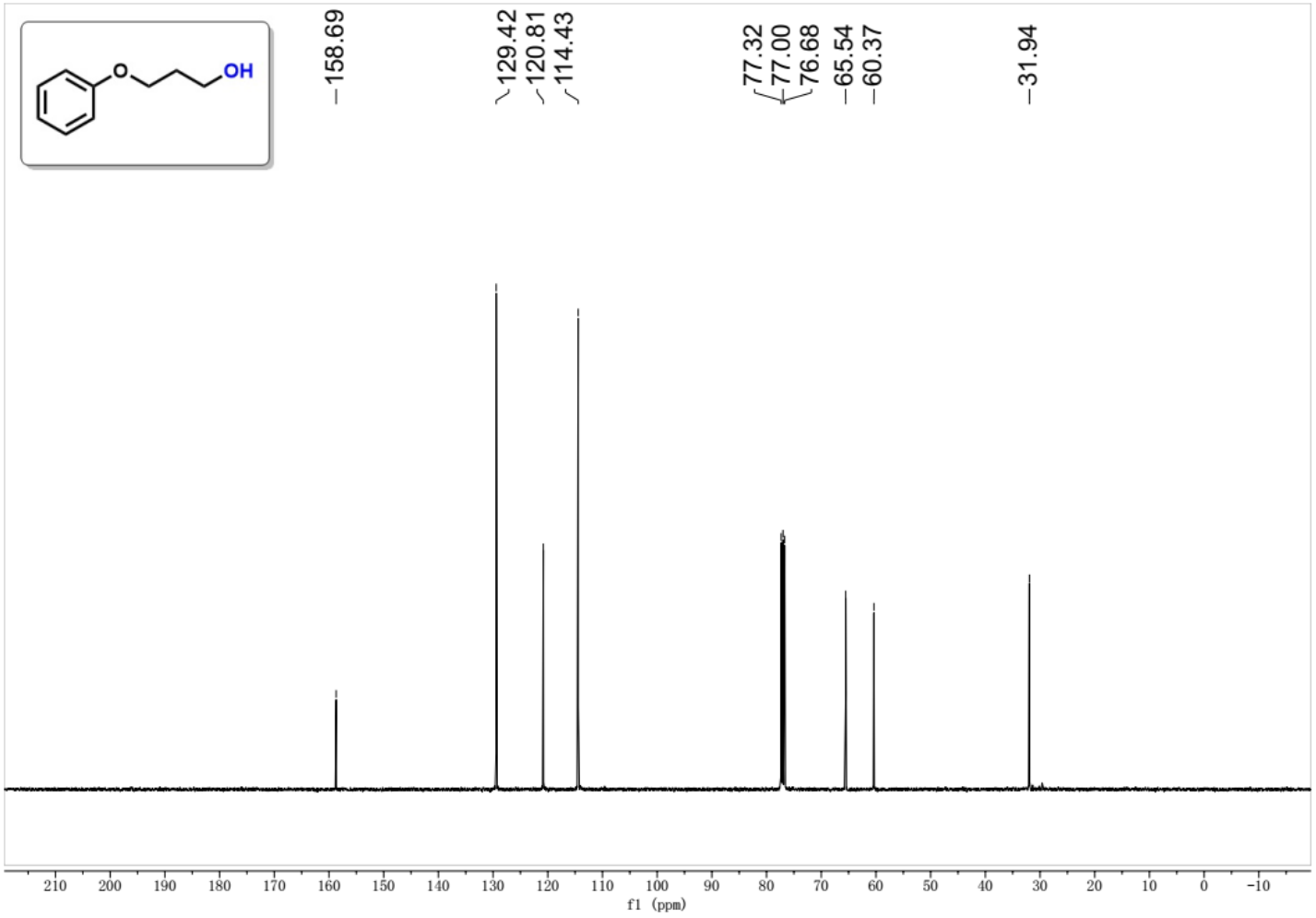
^{13}C NMR of 2ab



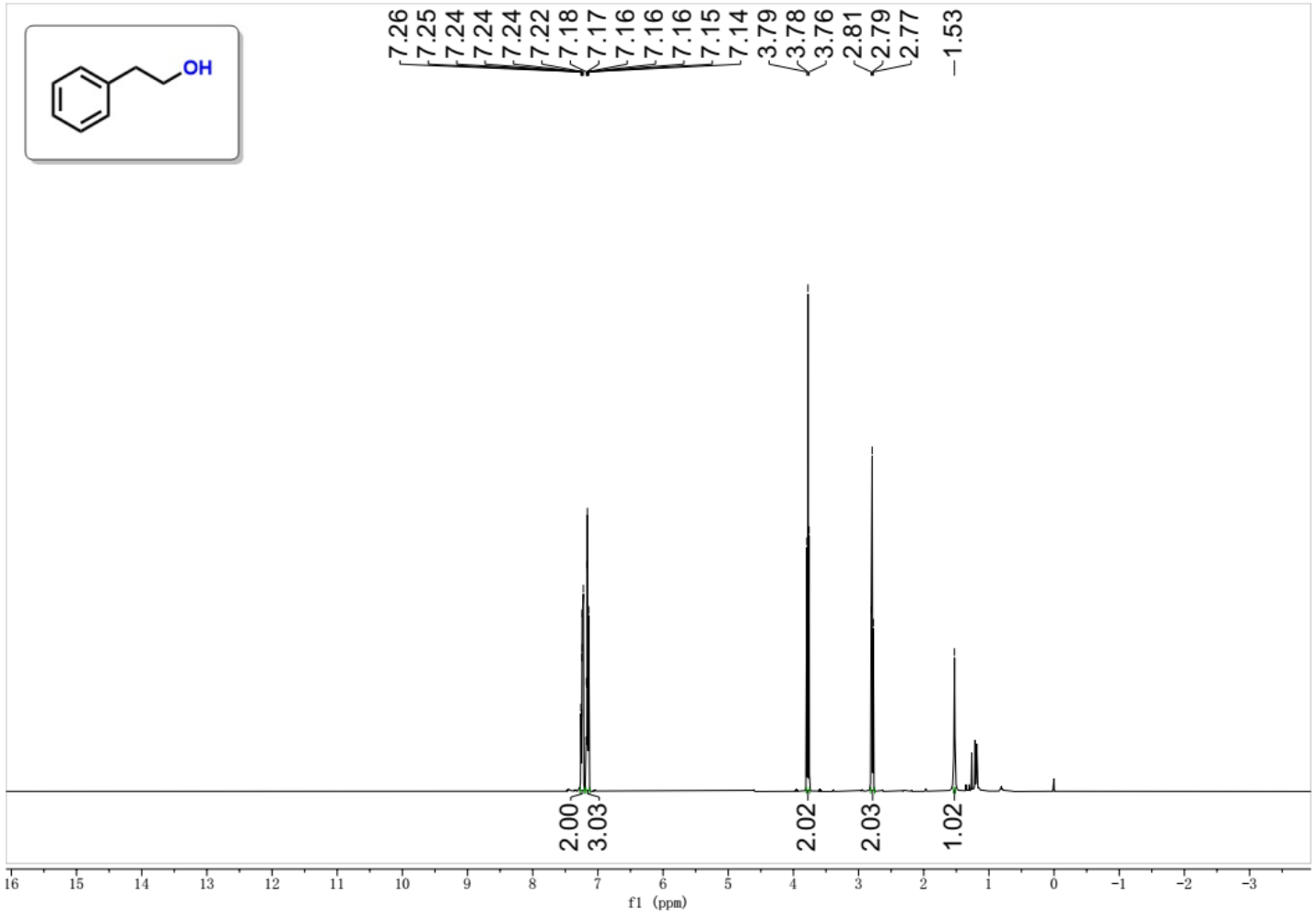
^{1}H NMR of 2ac


7.26
7.25
7.25
7.24
7.23
7.22
7.21
7.20
6.92
6.91
6.90
6.90
6.88
6.87
6.87
6.87
6.86
6.86
6.85
6.85
6.85
4.08
4.06
4.05
3.82
3.80
3.79
2.02
2.02
2.00
1.99
1.97
O
OH
2.03
3.01
2.02
2.00
3.01
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
−1
−2
−3
f1 (ppm)

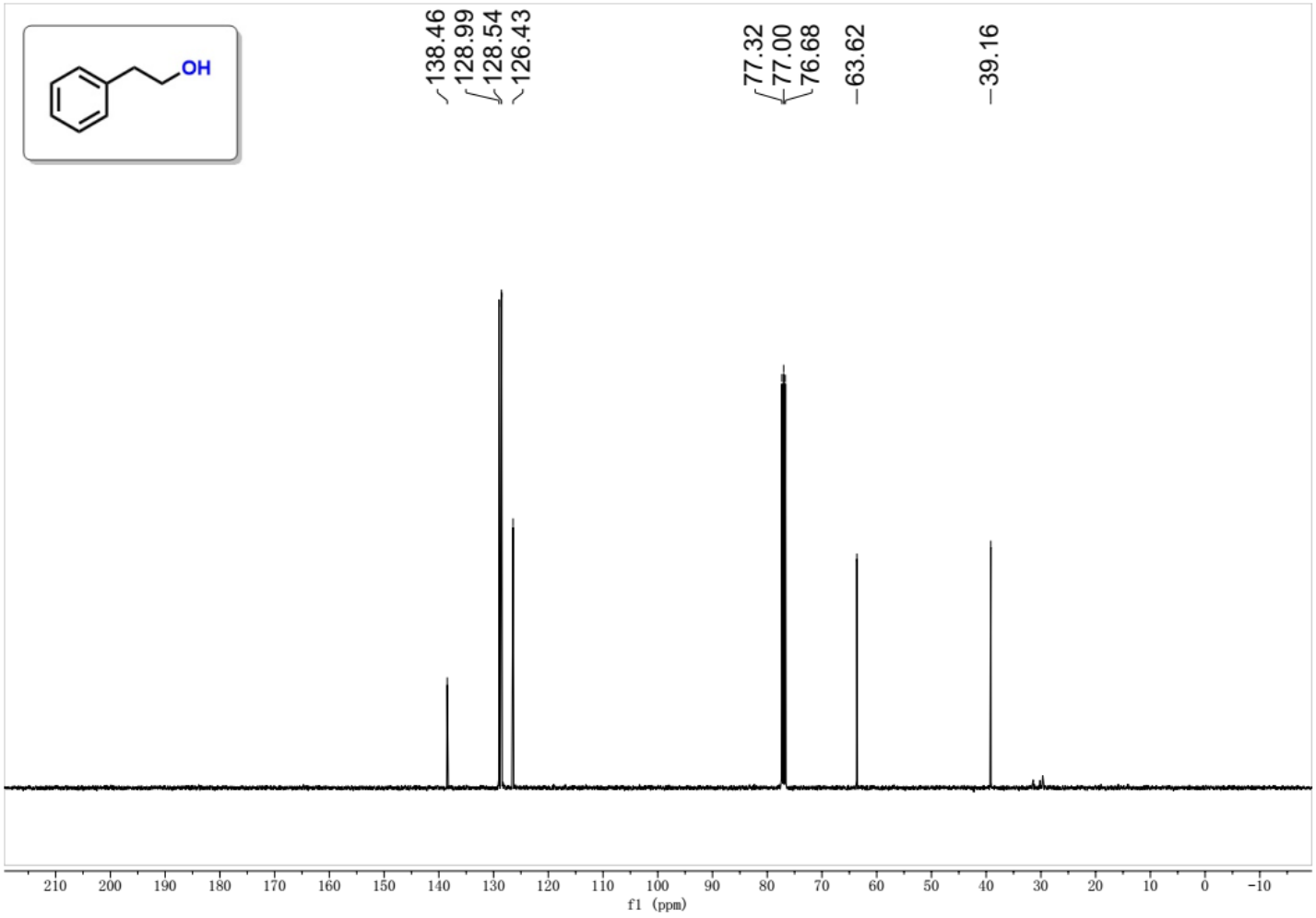
^{13}C NMR of 2ac



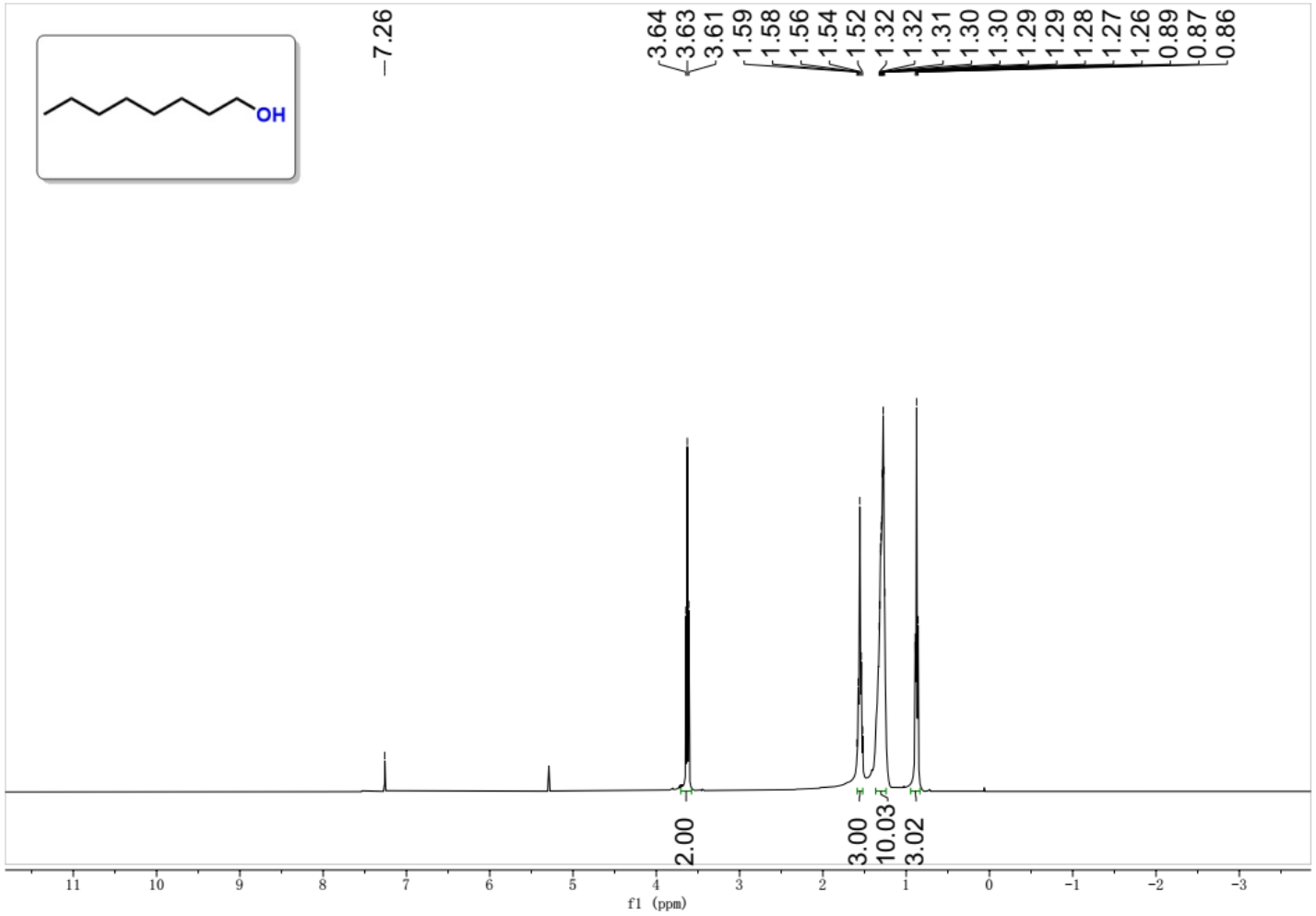
^{1}H NMR of 2ad


OH
7.26
7.25
7.24
7.24
7.24
7.22
7.18
7.17
7.16
7.16
7.16
7.15
7.14
3.79
3.78
3.76
2.81
2.79
2.77
1.53
2.00
3.03
2.02
2.03
1.02
f1 (ppm)

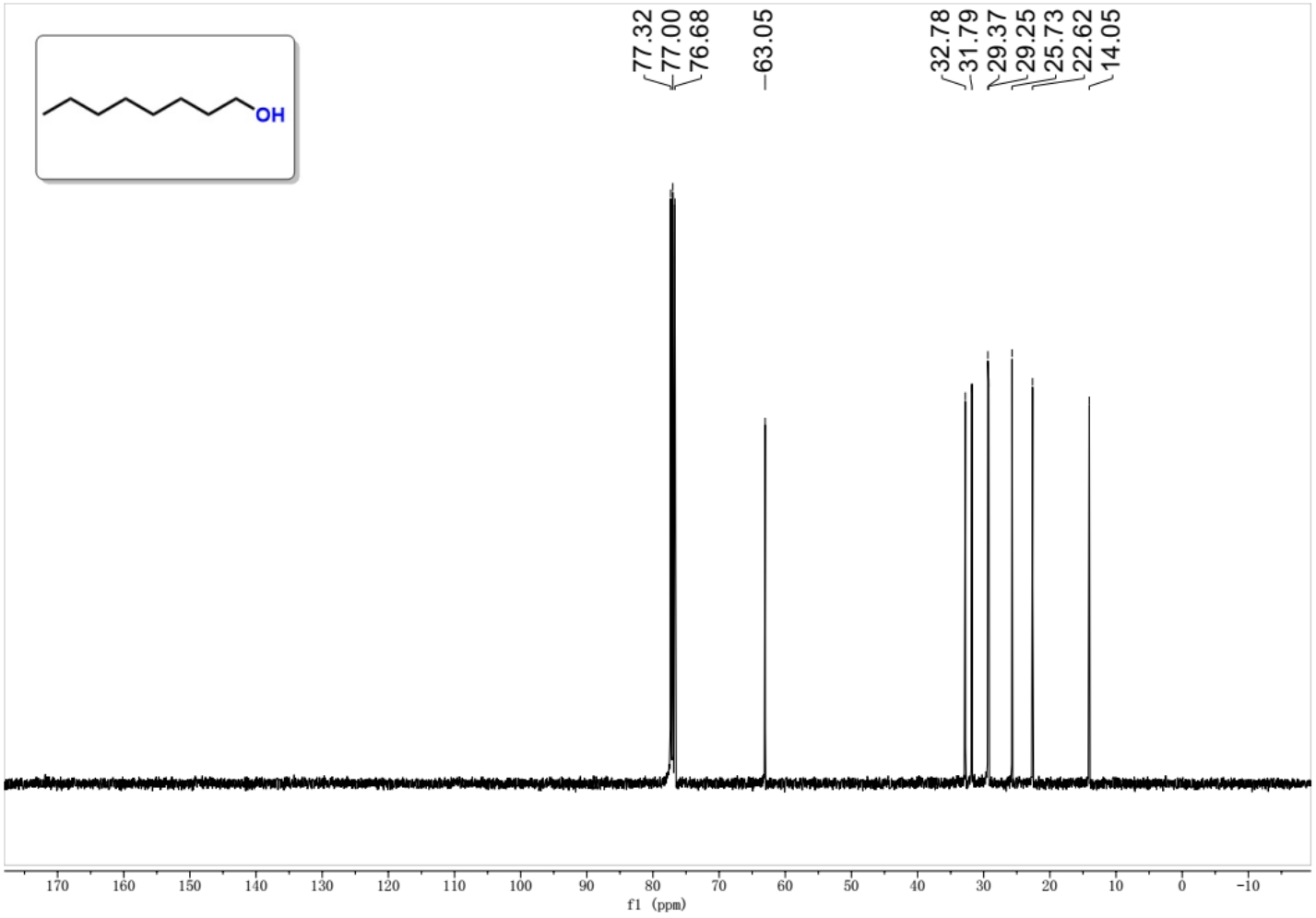
^{13}C NMR of 2ad



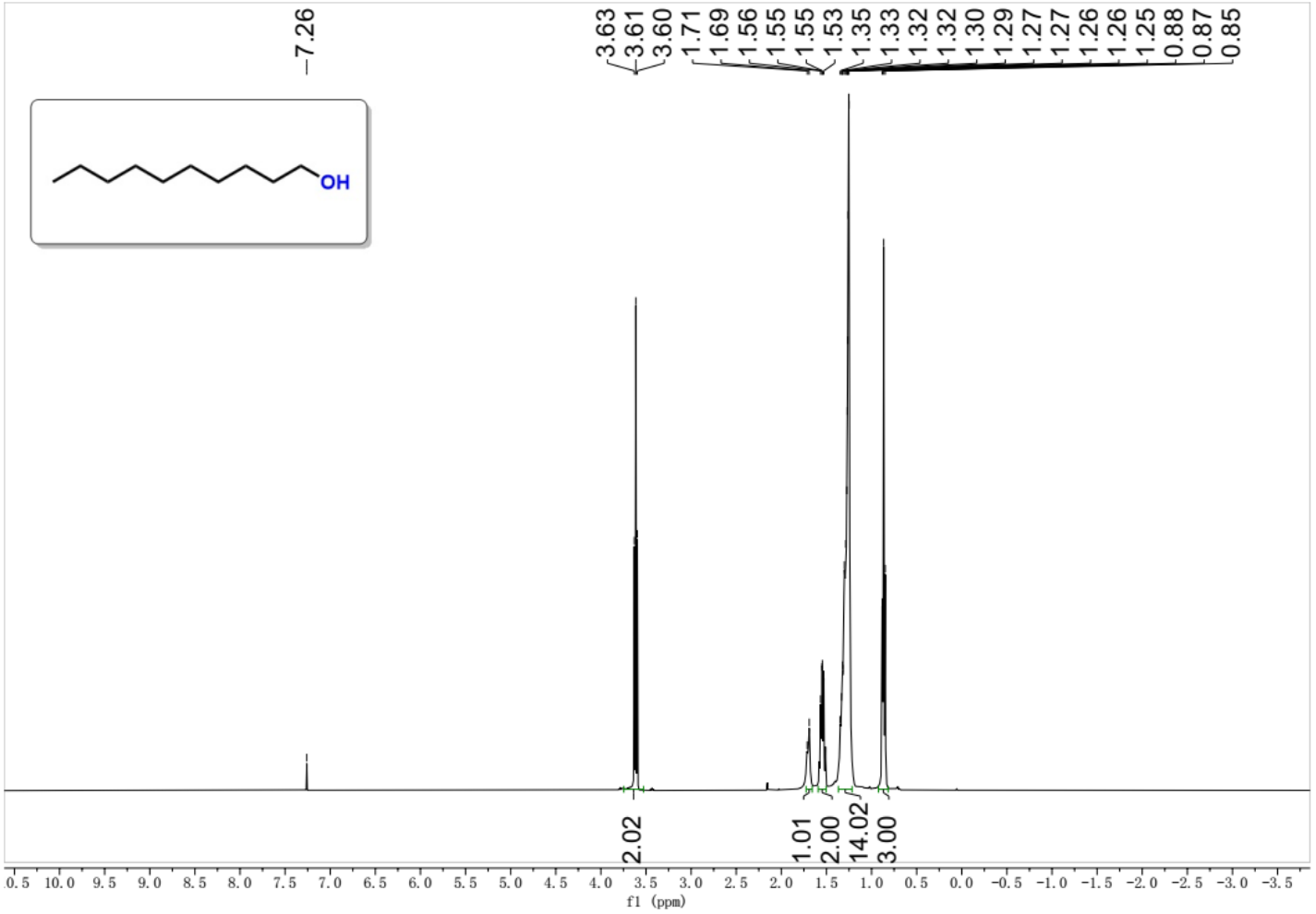
^{1}H NMR of 2ae


OH
7.26
3.64
3.63
3.61
1.59
1.58
1.56
1.54
1.52
1.32
1.32
1.31
1.30
1.30
1.29
1.29
1.28
1.27
1.26
0.89
0.87
0.86
2.00
3.00
10.03
3.02
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)

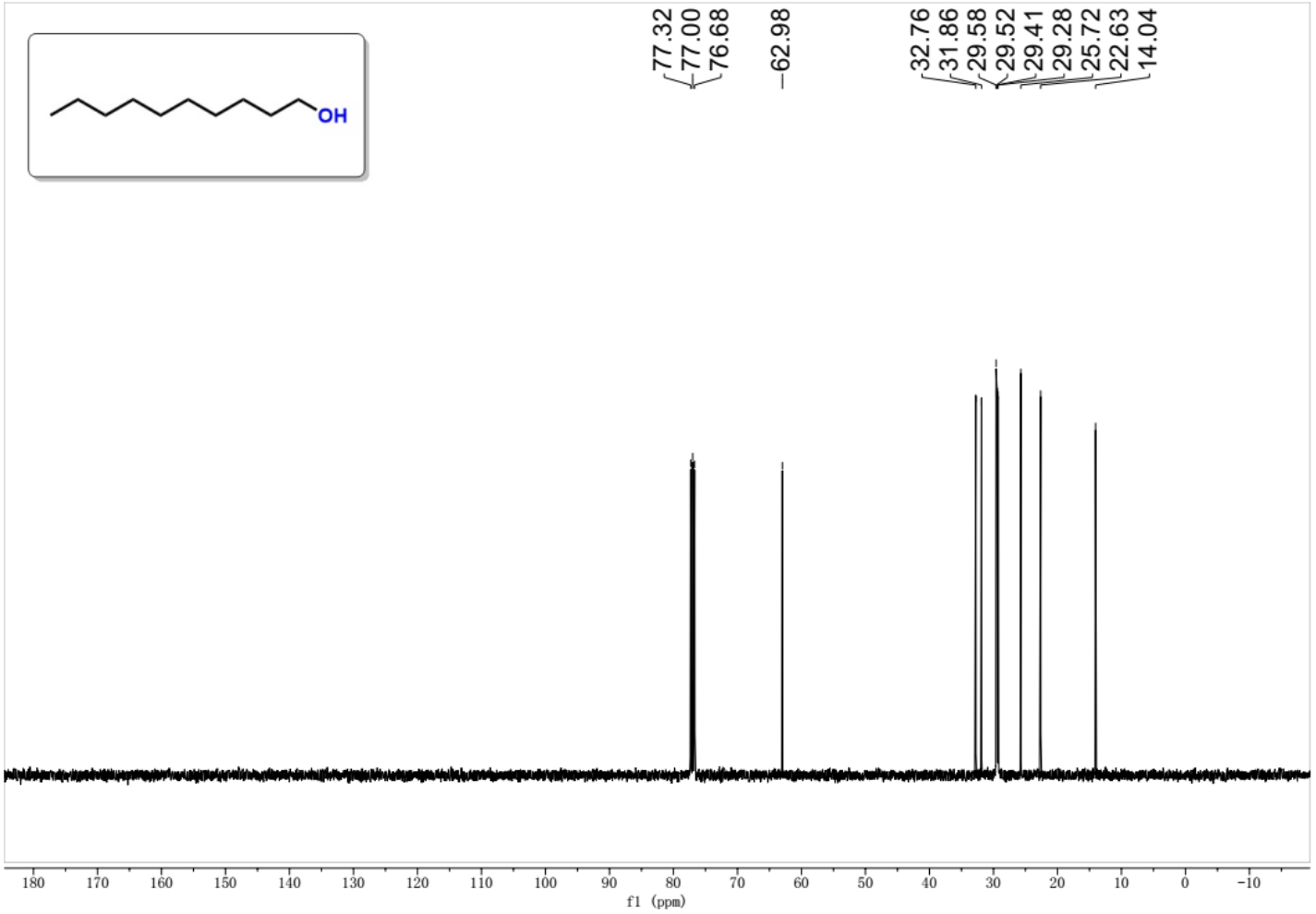
^{13}C NMR of 2ae



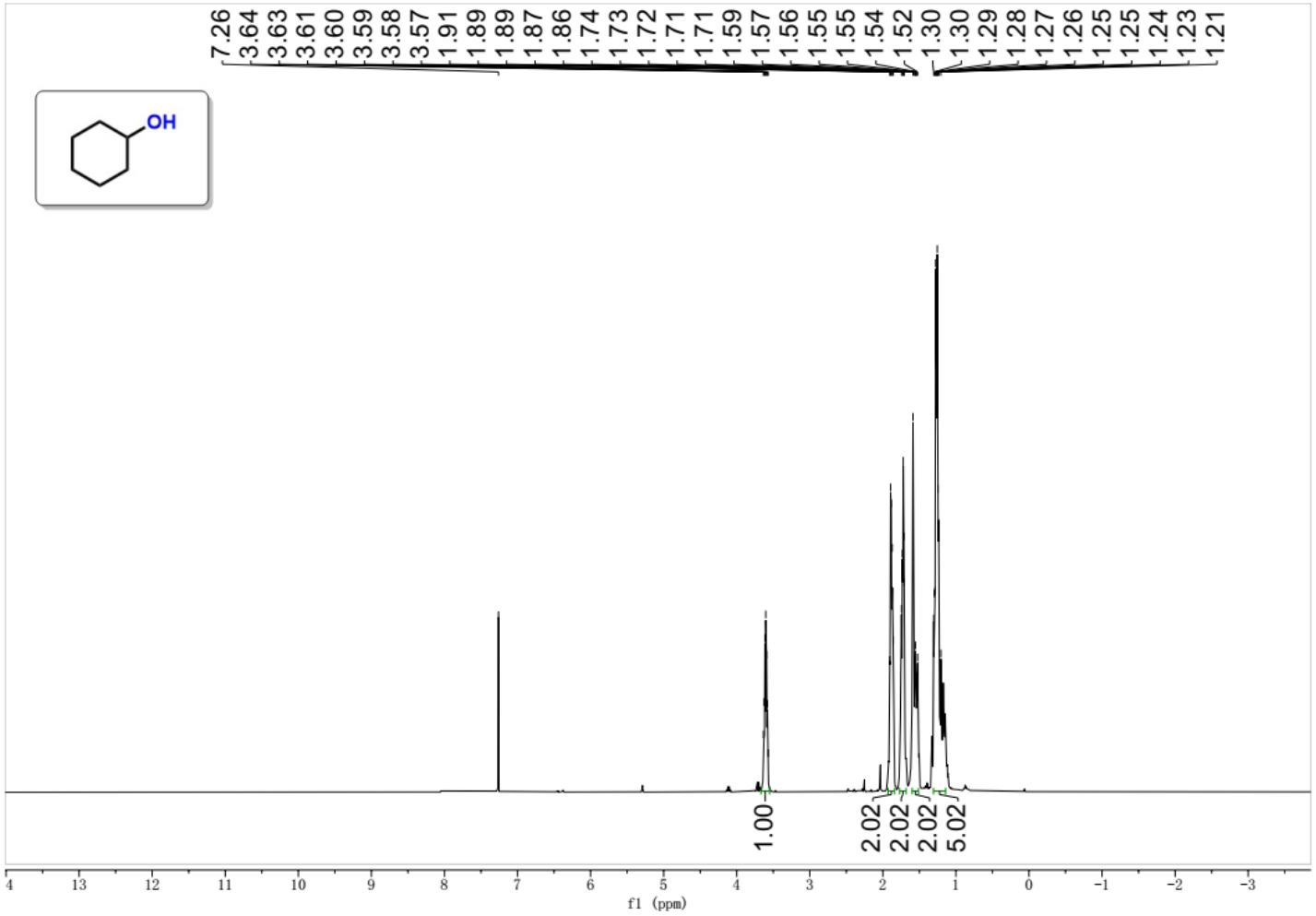
^{1}H NMR of 2af


7.26
3.63
3.61
3.60
1.71
1.69
1.56
1.55
1.55
1.53
1.35
1.33
1.32
1.32
1.30
1.29
1.27
1.27
1.26
1.26
1.25
0.88
0.87
0.85
OH
2.02
1.01
2.00
14.02
3.00
f1 (ppm)

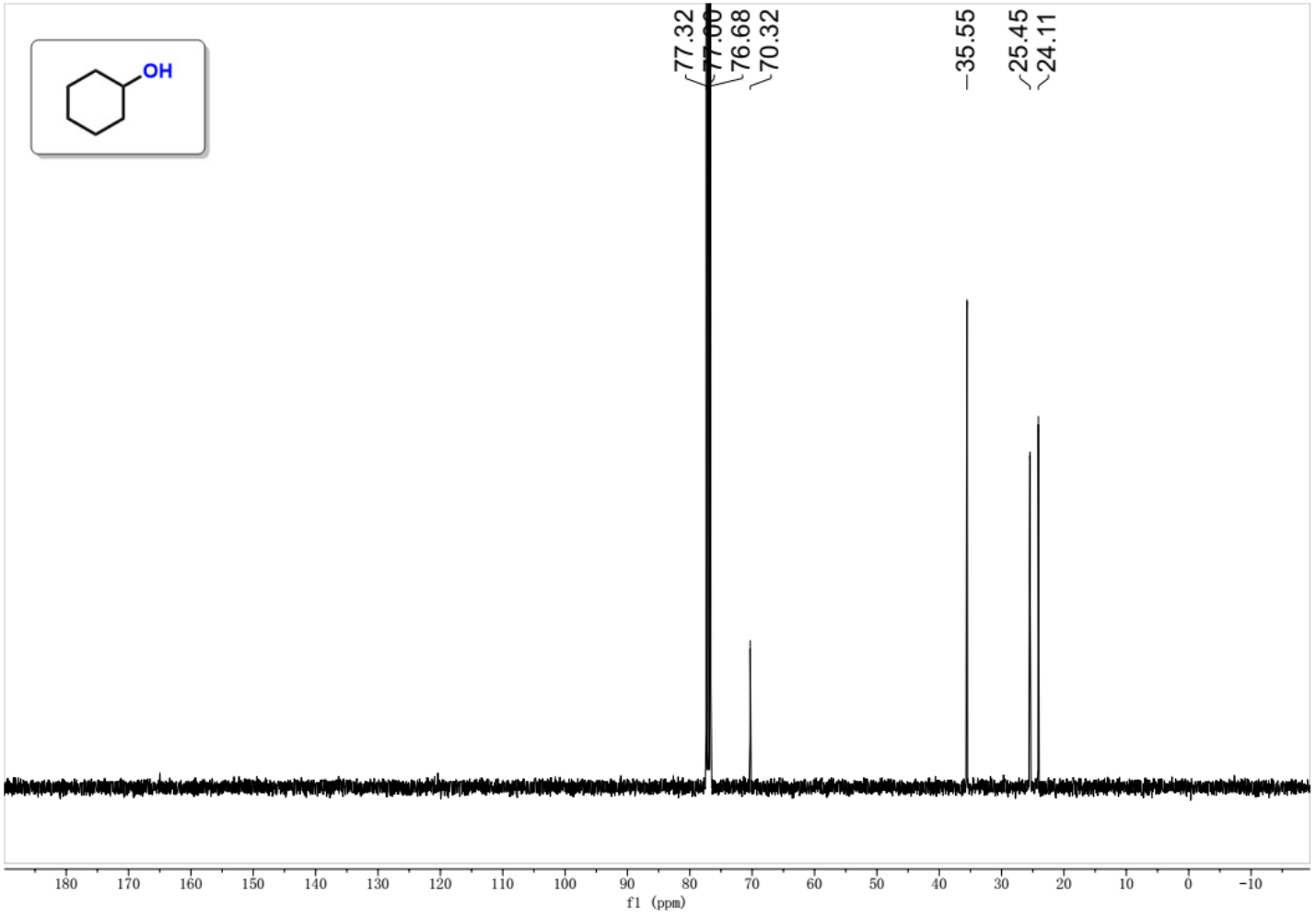
^{13}C NMR of 2af



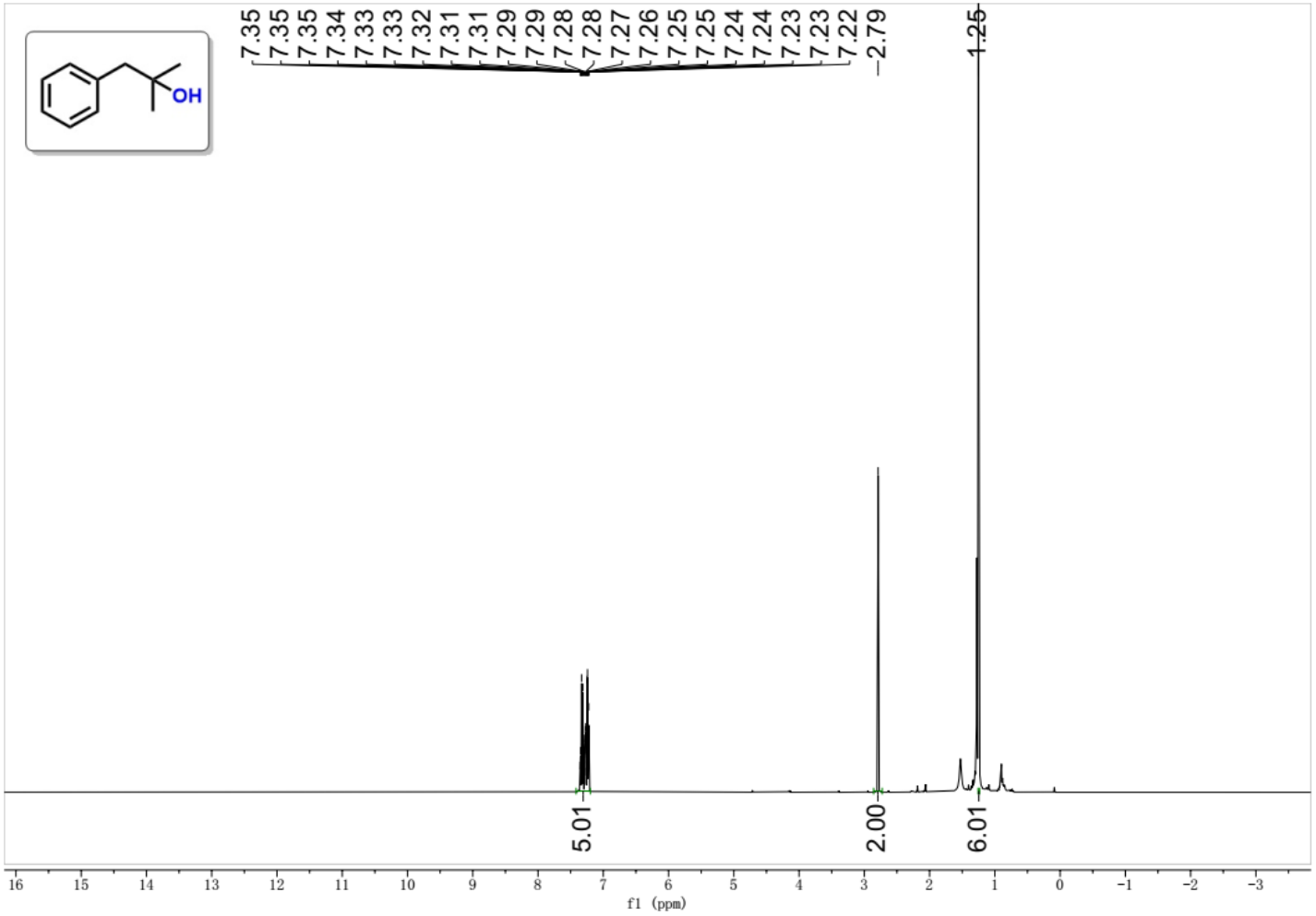
^{1}H NMR of 2ag


7.26
3.64
3.63
3.61
3.60
3.59
3.58
3.57
1.91
1.89
1.89
1.87
1.86
1.74
1.73
1.72
1.71
1.71
1.59
1.57
1.56
1.55
1.55
1.54
1.52
1.30
1.30
1.29
1.28
1.27
1.26
1.25
1.25
1.24
1.23
1.21
OH
1.00
2.02
2.02
2.02
5.02
f1 (ppm)

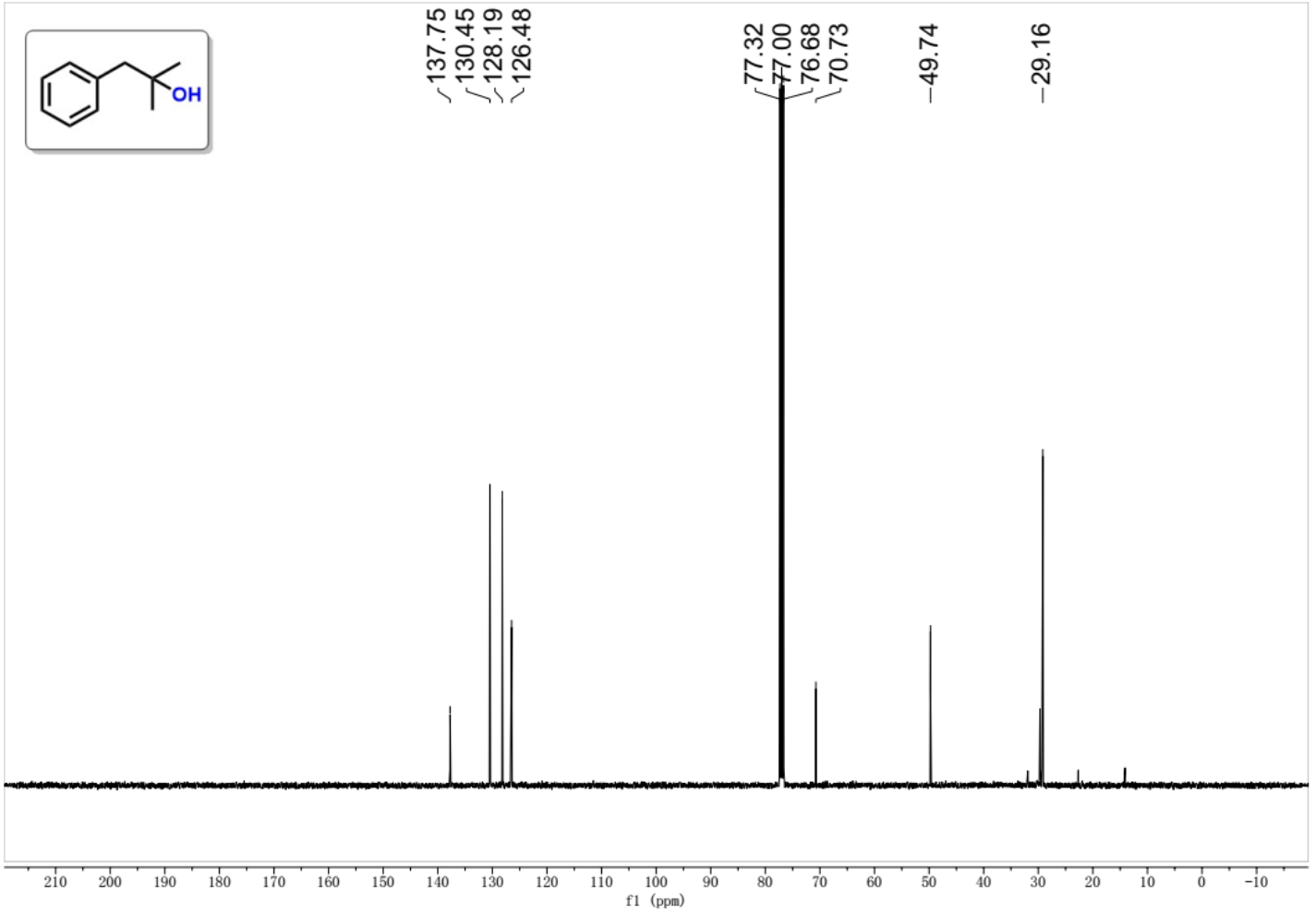
^{13}C NMR of 2ag



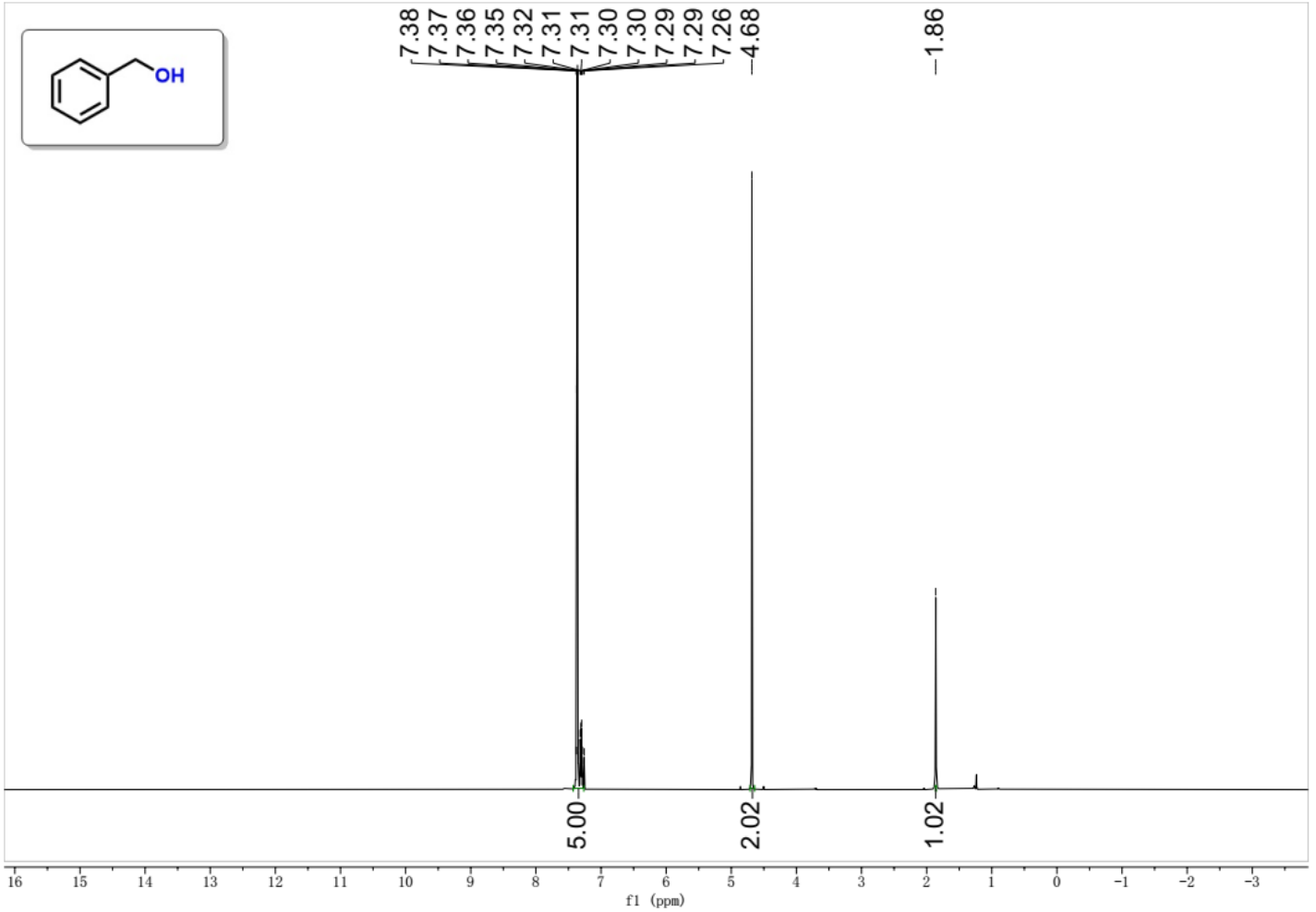
^{1}H NMR of 2ah


OH
7.35
7.35
7.35
7.34
7.33
7.33
7.32
7.31
7.31
7.29
7.29
7.28
7.28
7.27
7.26
7.25
7.25
7.24
7.24
7.23
7.23
7.22
2.79
1.25
5.01
2.00
6.01
f1 (ppm)

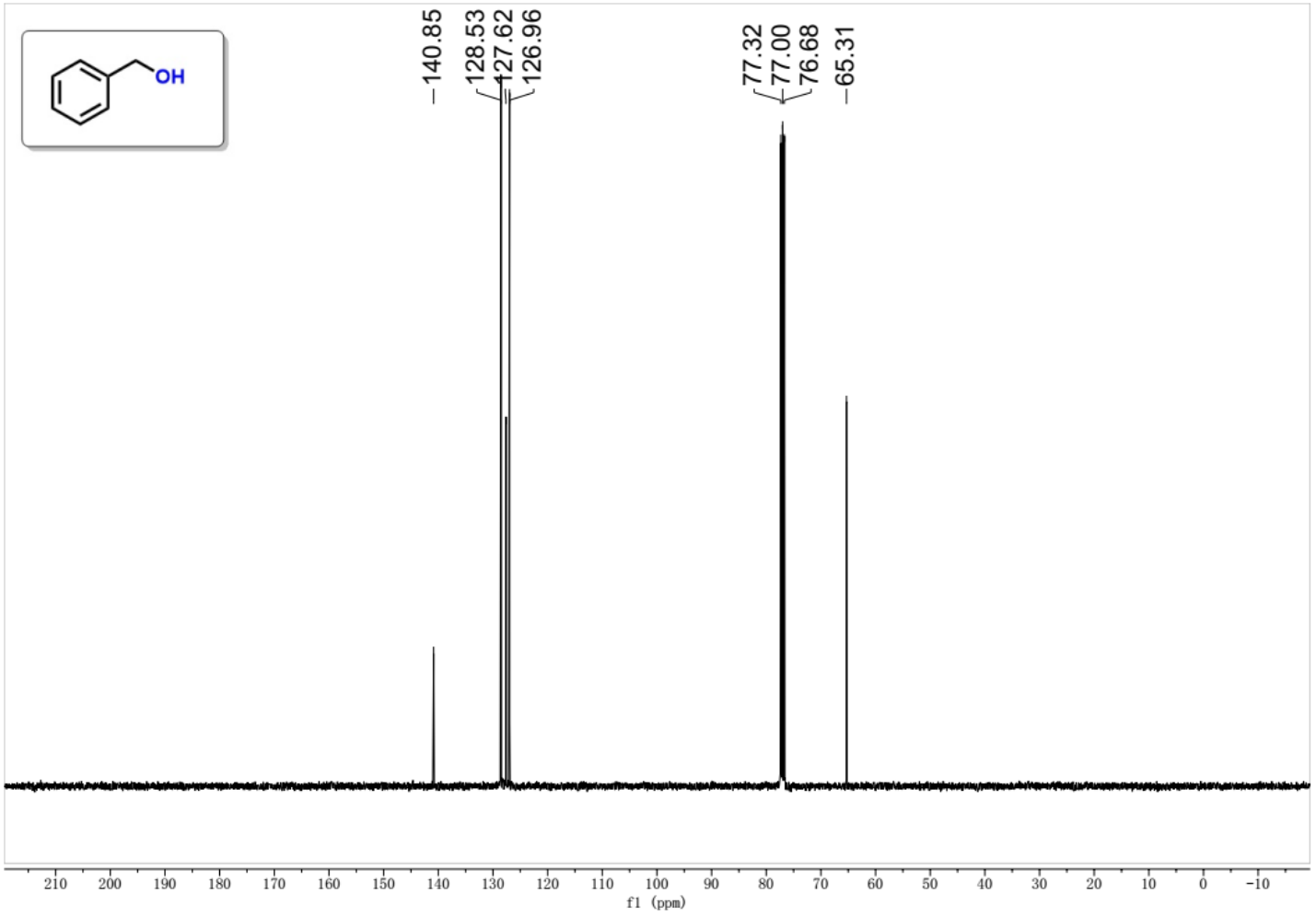
^{13}C NMR of 2ah



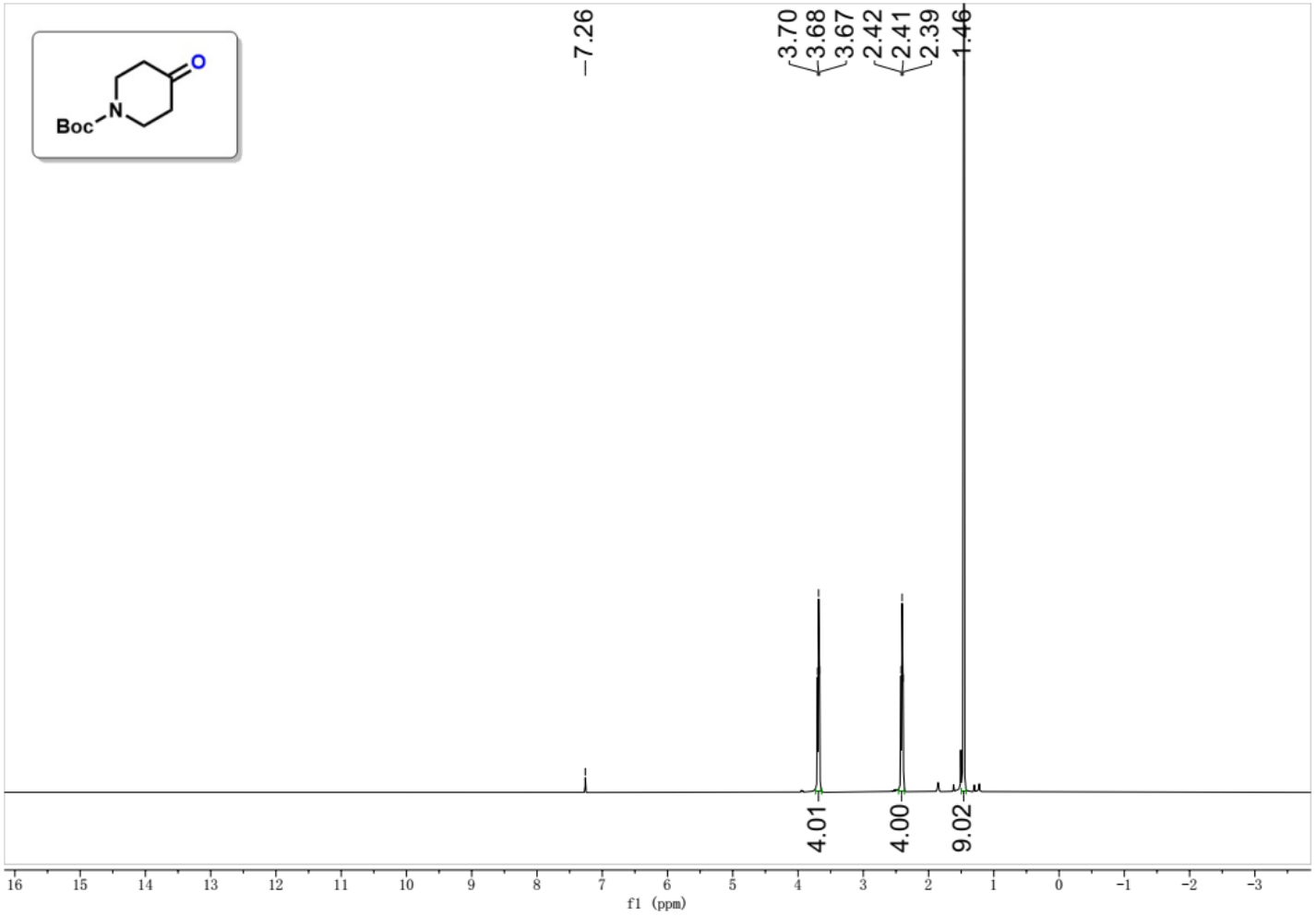
^{1}H NMR of 2ak


OH
7.38
7.37
7.36
7.35
7.32
7.31
7.31
7.30
7.30
7.29
7.29
7.26
4.68
1.86
5.00
2.02
1.02
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
−1
−2
−3
f1 (ppm)

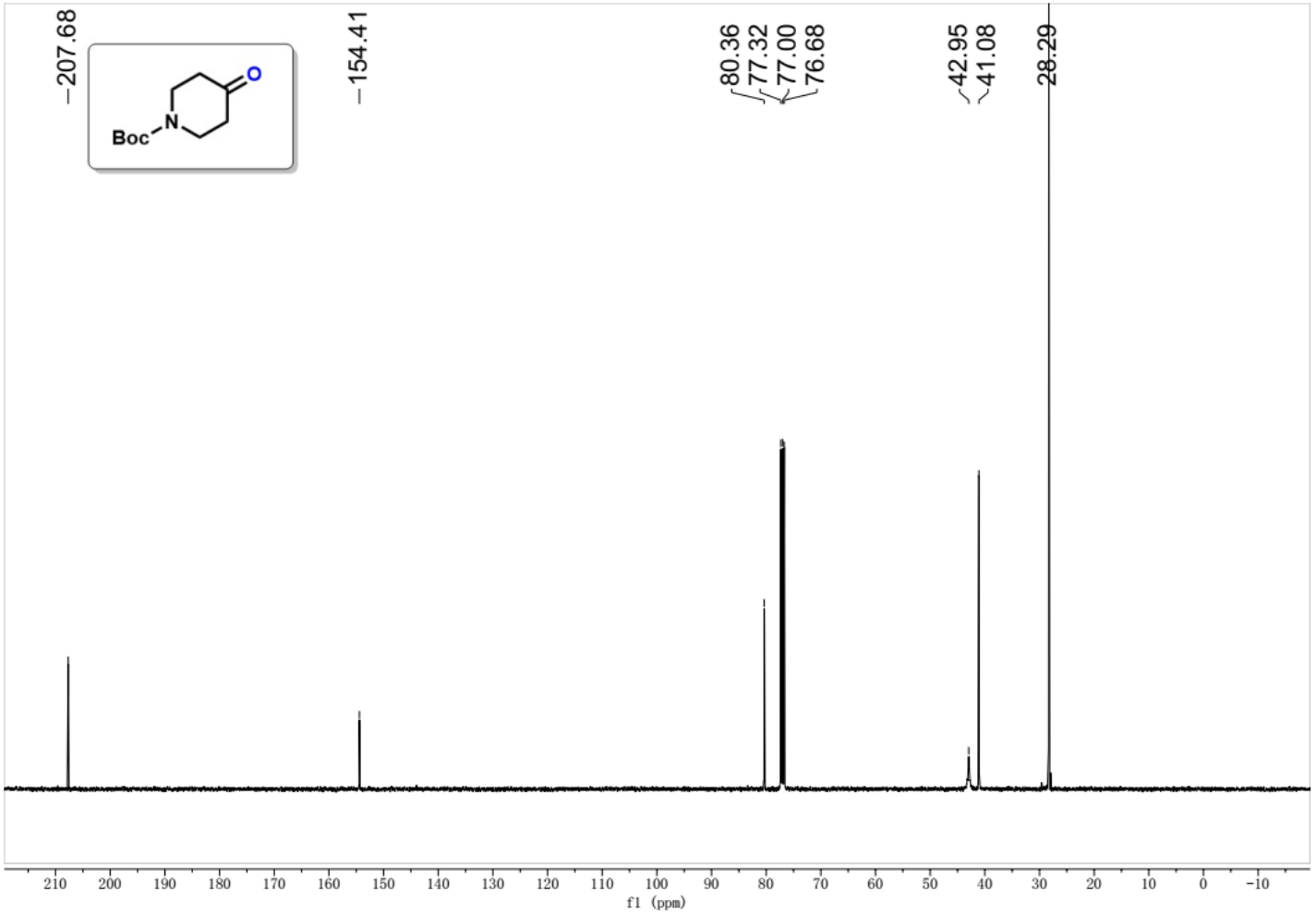
^{13}C NMR of 2ak



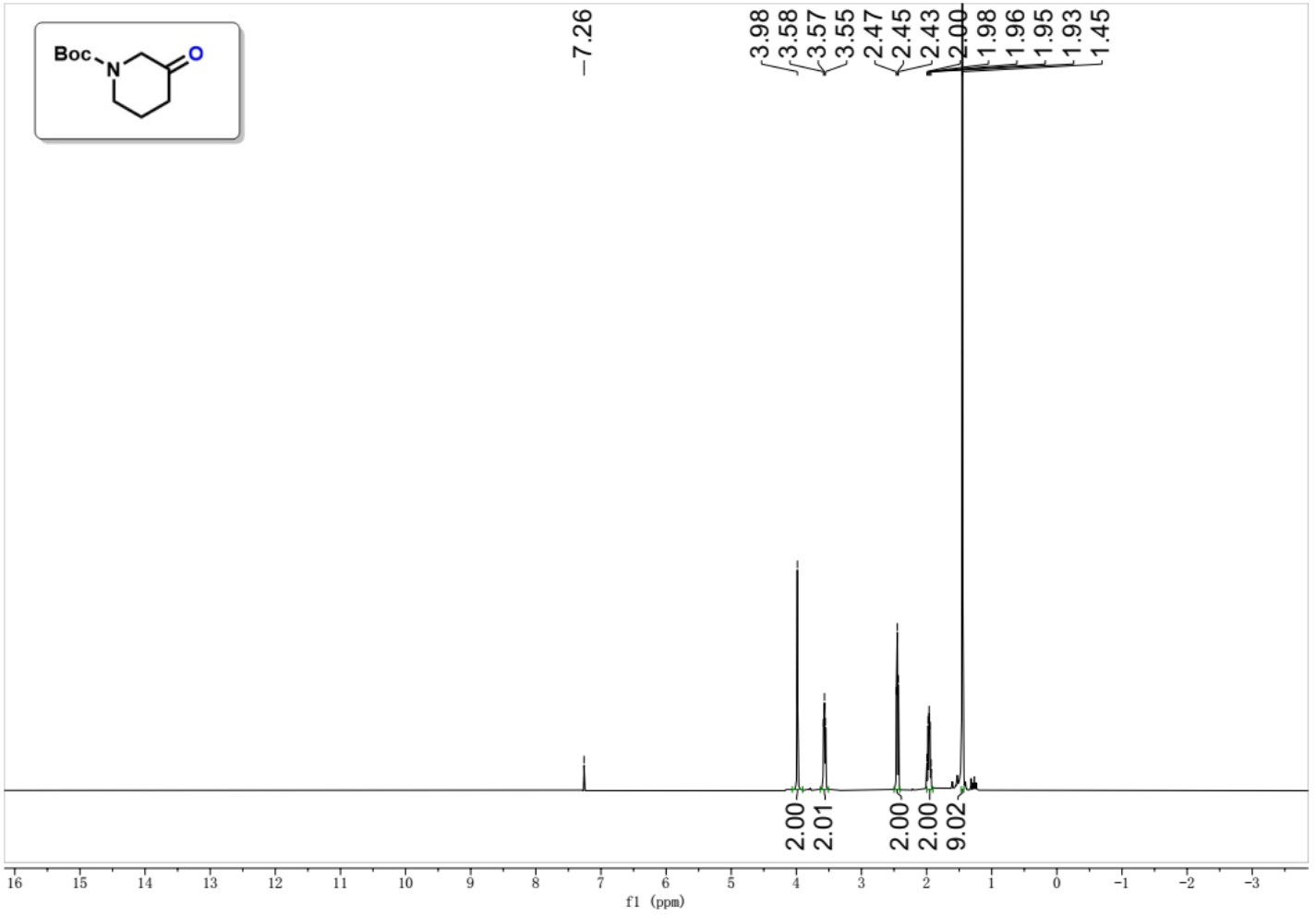
^{1}H NMR of 2al


Boc
N
O
7.26
3.70
3.68
3.67
2.42
2.41
2.39
1.46
4.01
4.00
9.02
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
−1
−2
−3
f1 (ppm)

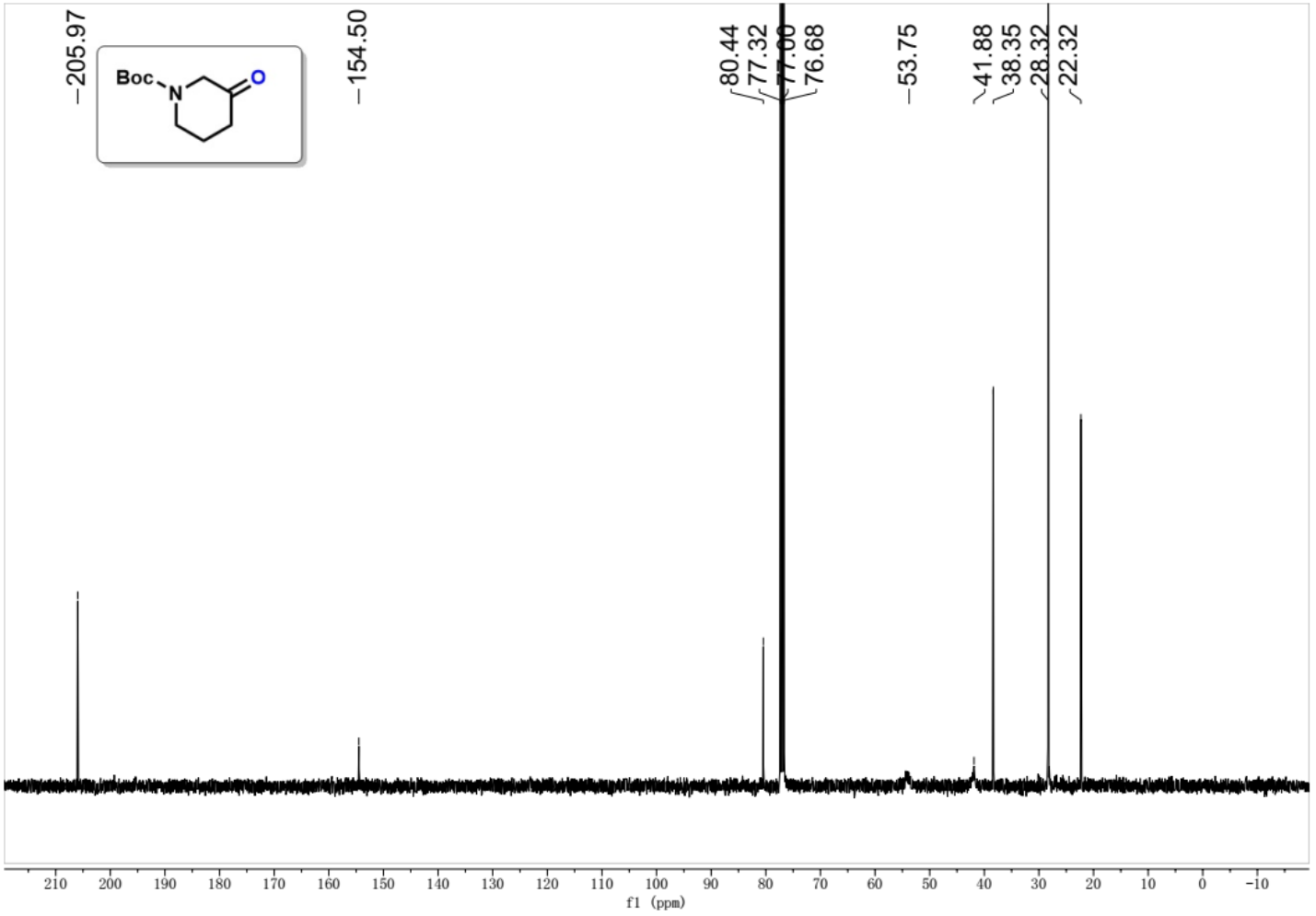
^{13}C NMR of 2al



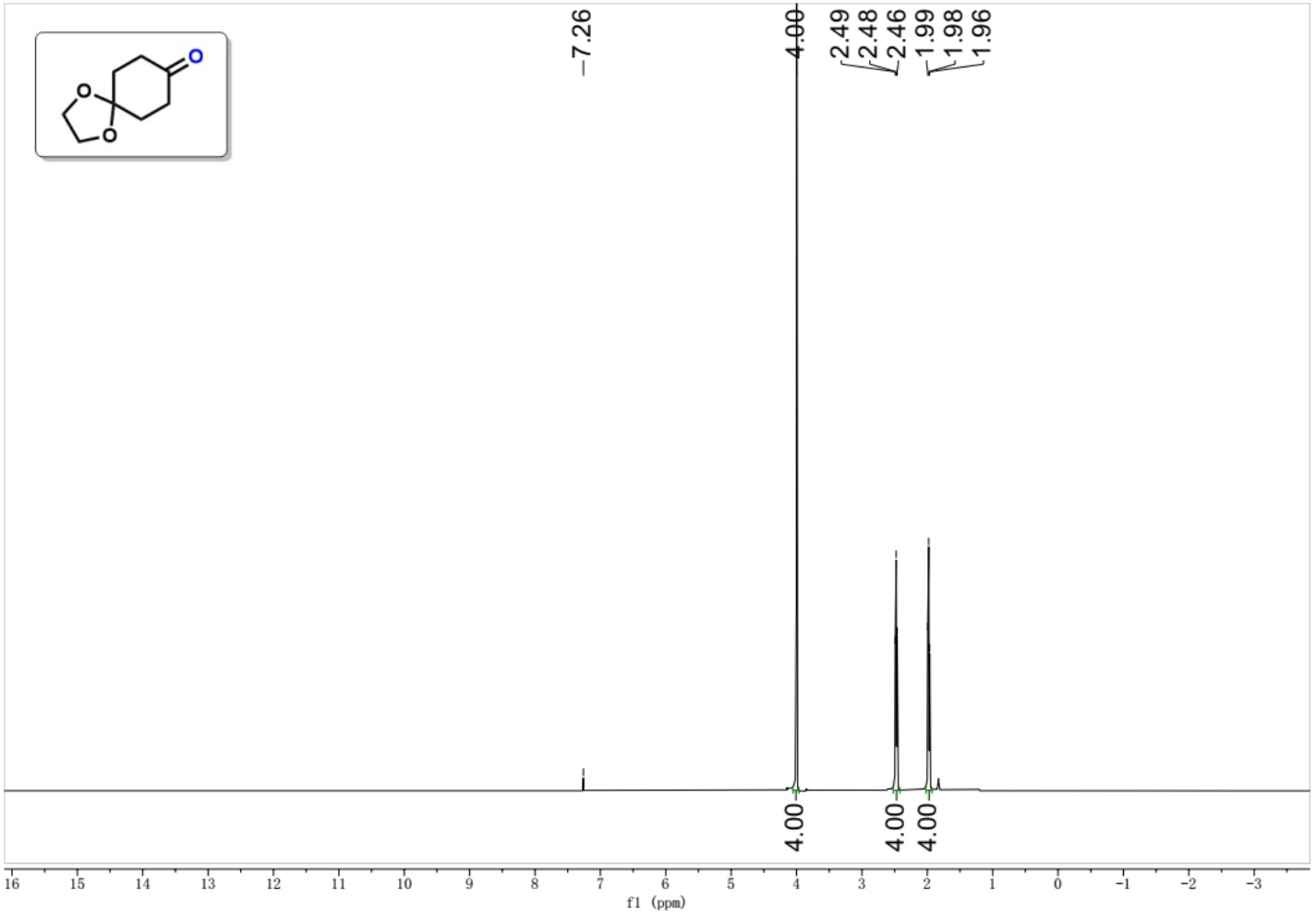
^{1}H NMR of 2am


Boc
N
O
7.26
3.98
3.58
3.57
3.55
2.47
2.45
2.43
2.00
1.98
1.96
1.95
1.93
1.45
2.00
2.01
2.00
2.00
9.02
f1 (ppm)

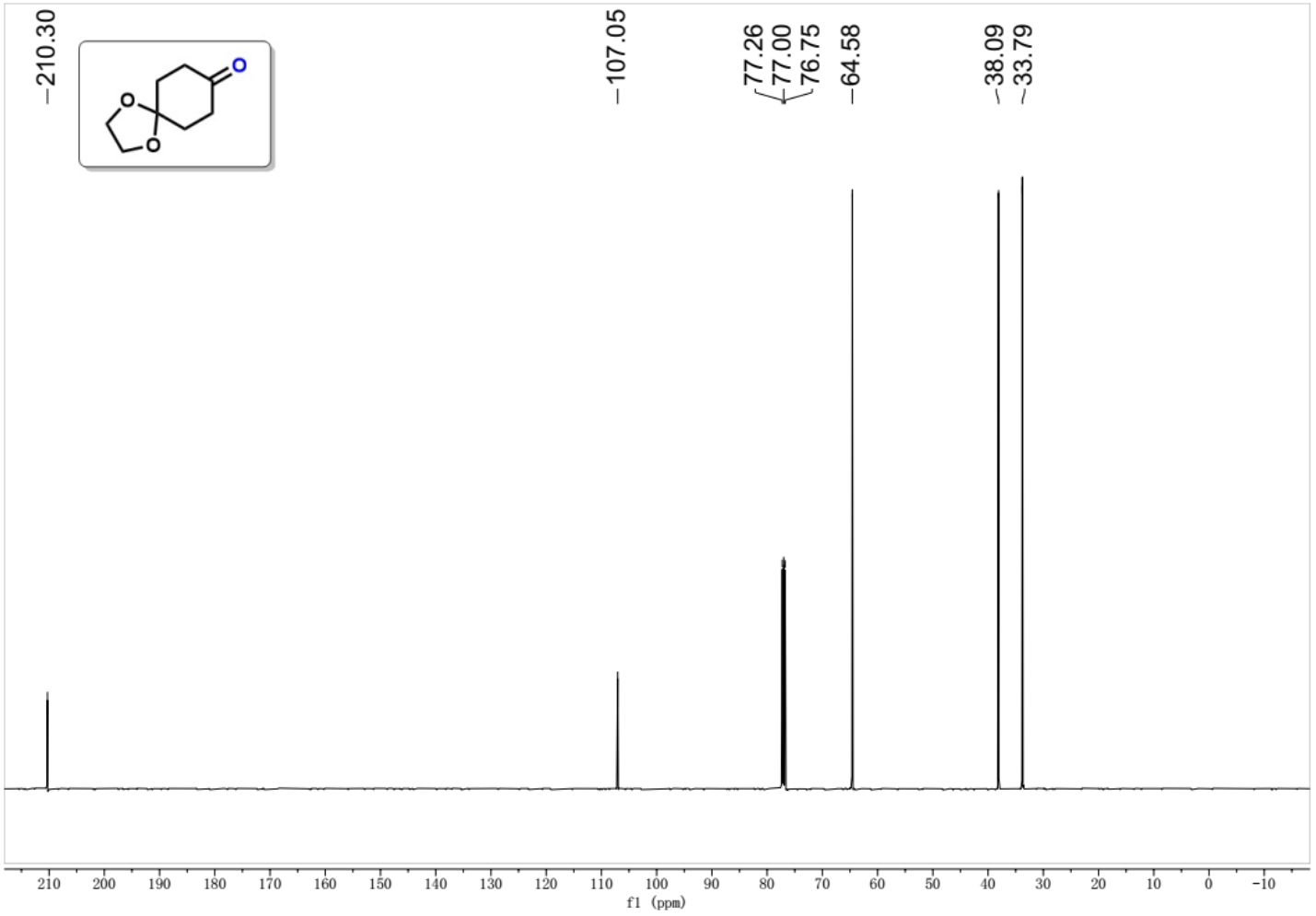
^{13}C NMR of 2am



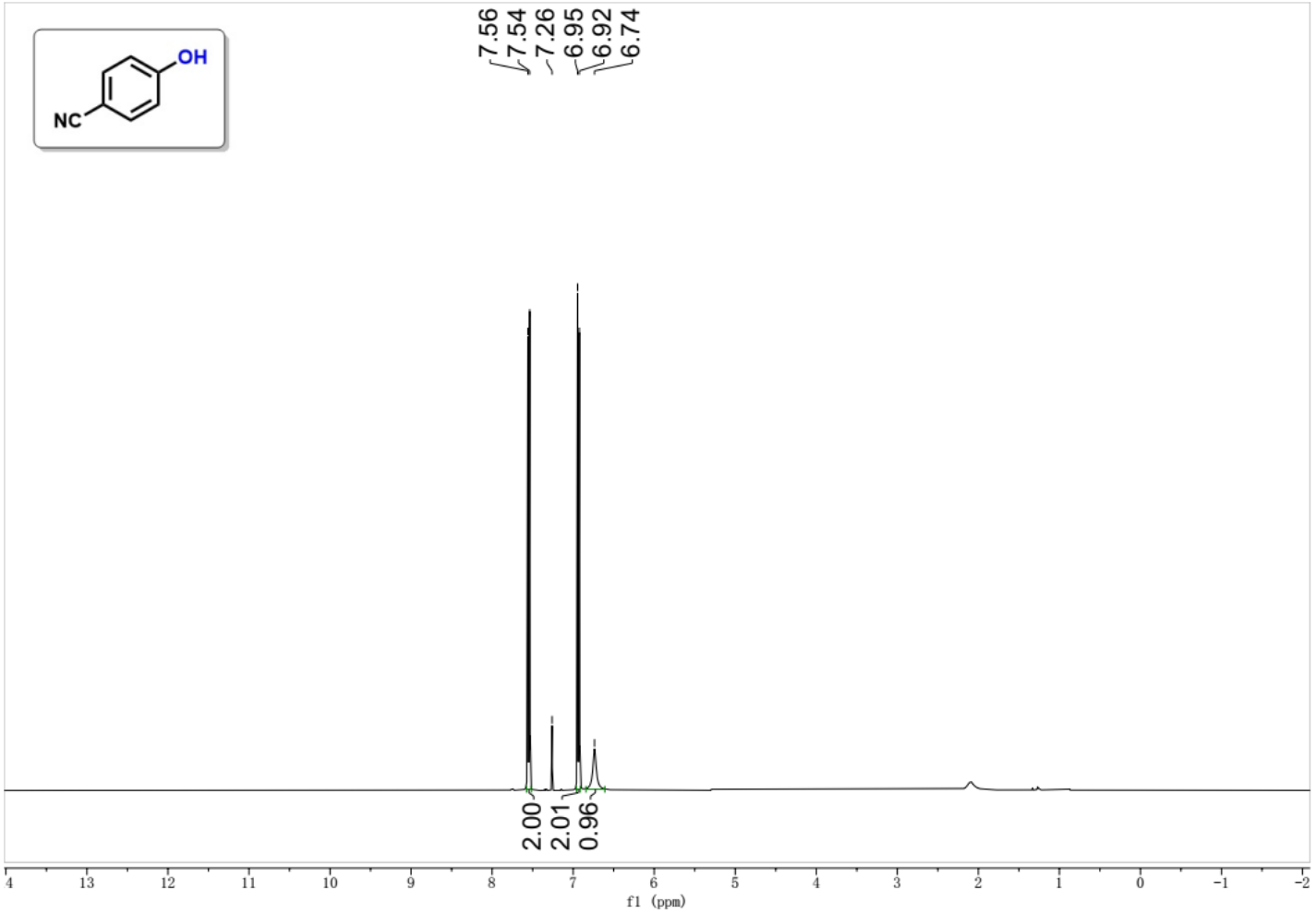
^{1}H NMR of 2an



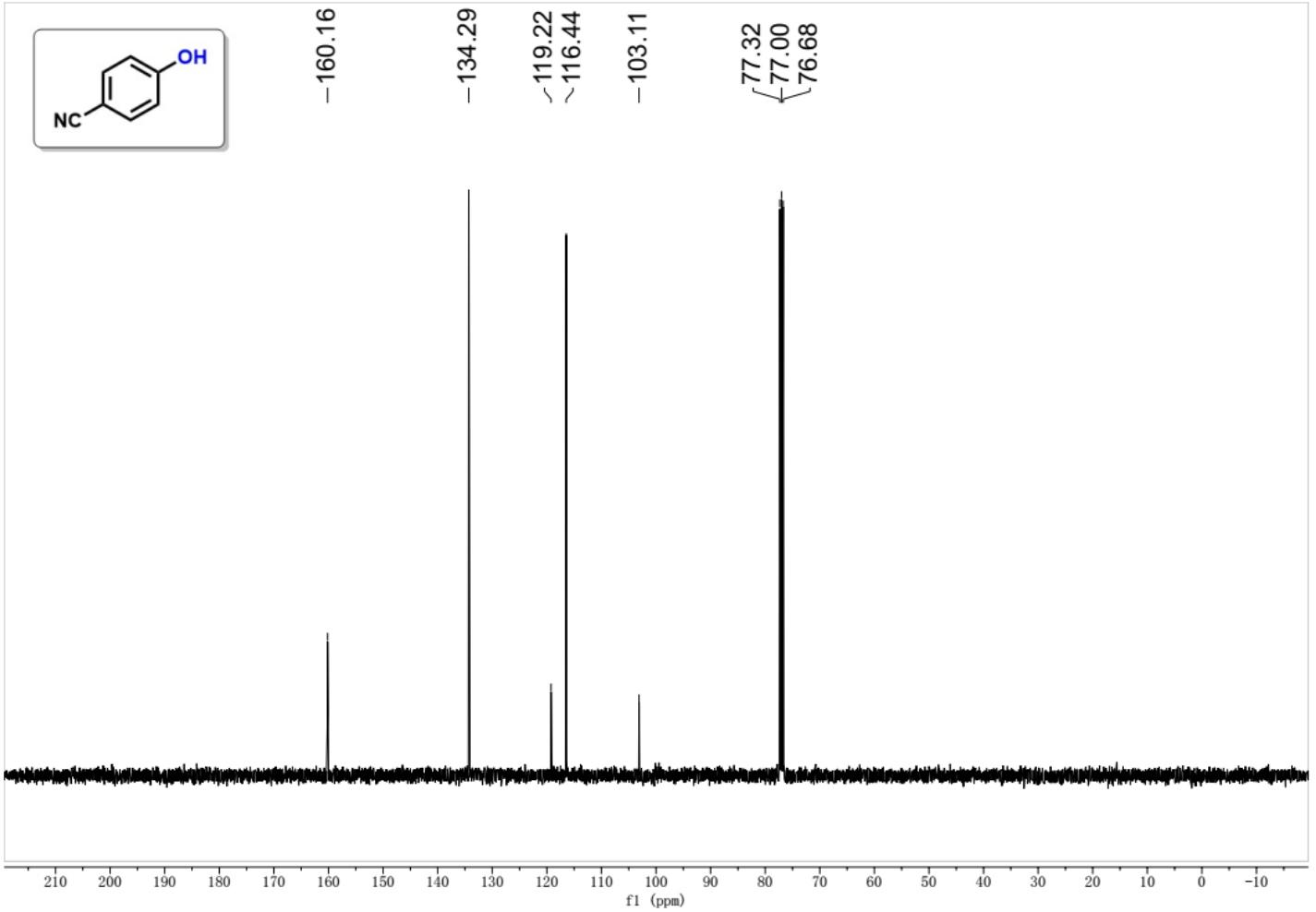
^{13}C NMR of 2an



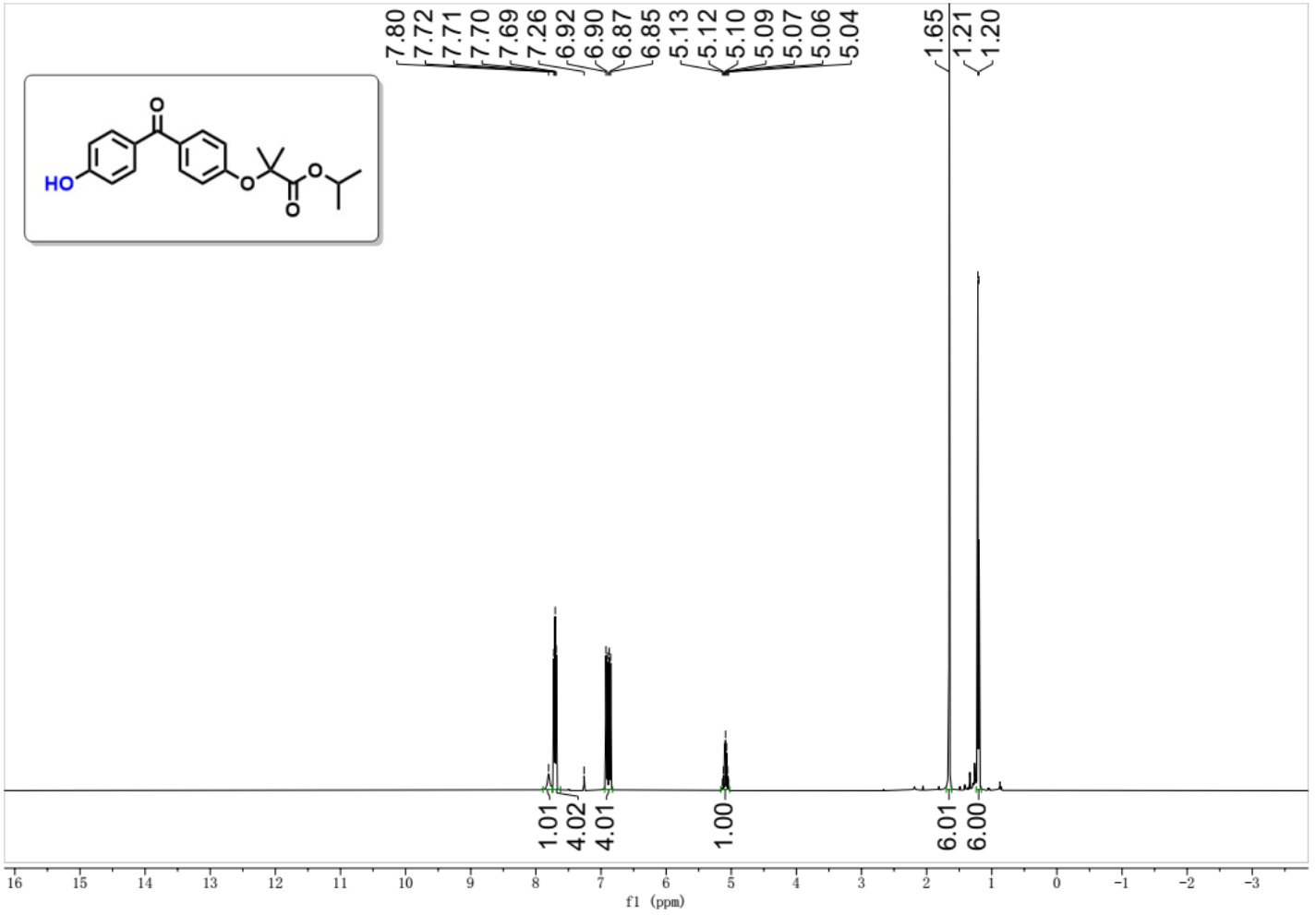
^{1}H NMR of 2ap



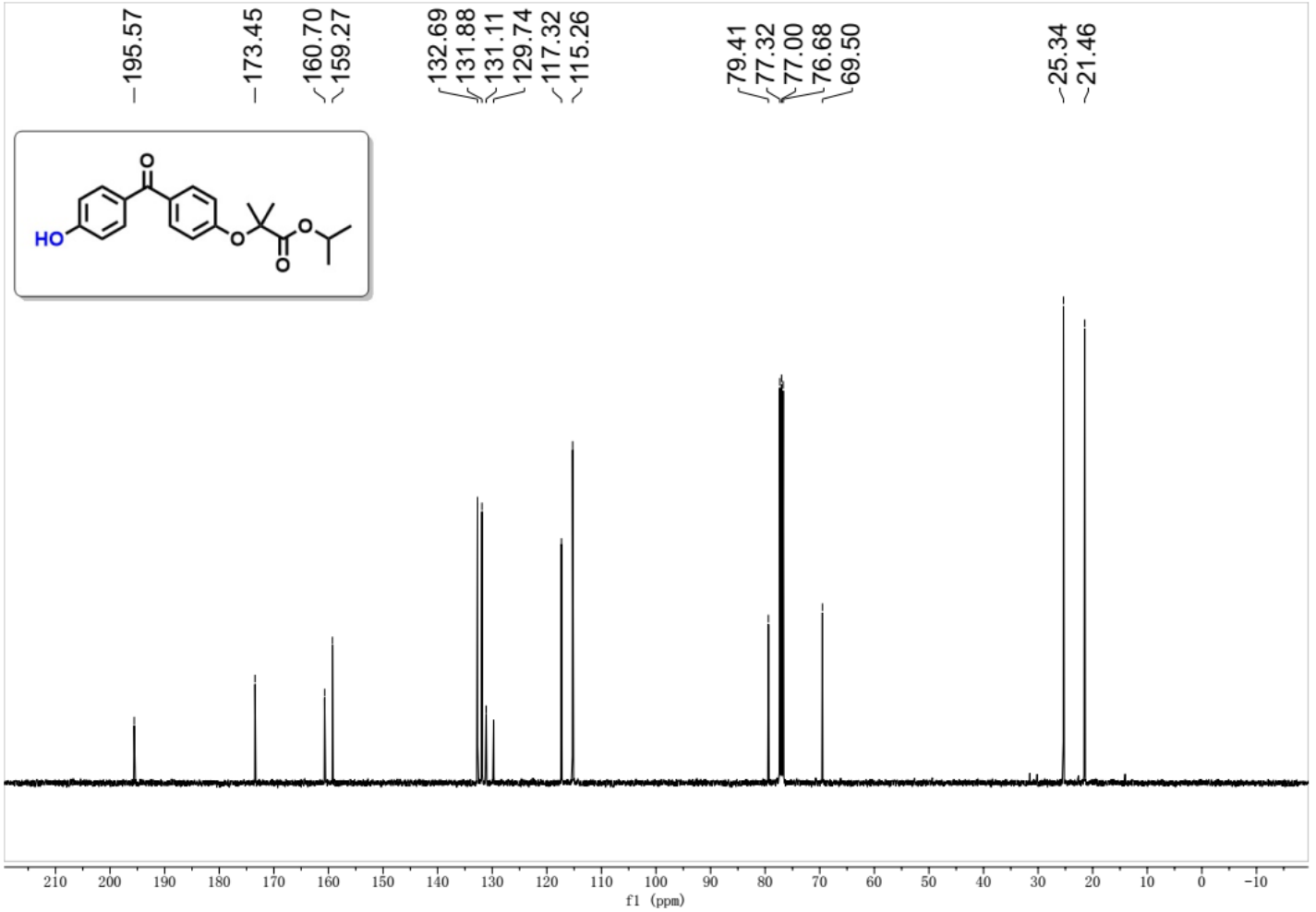
^{13}C NMR of 2ap



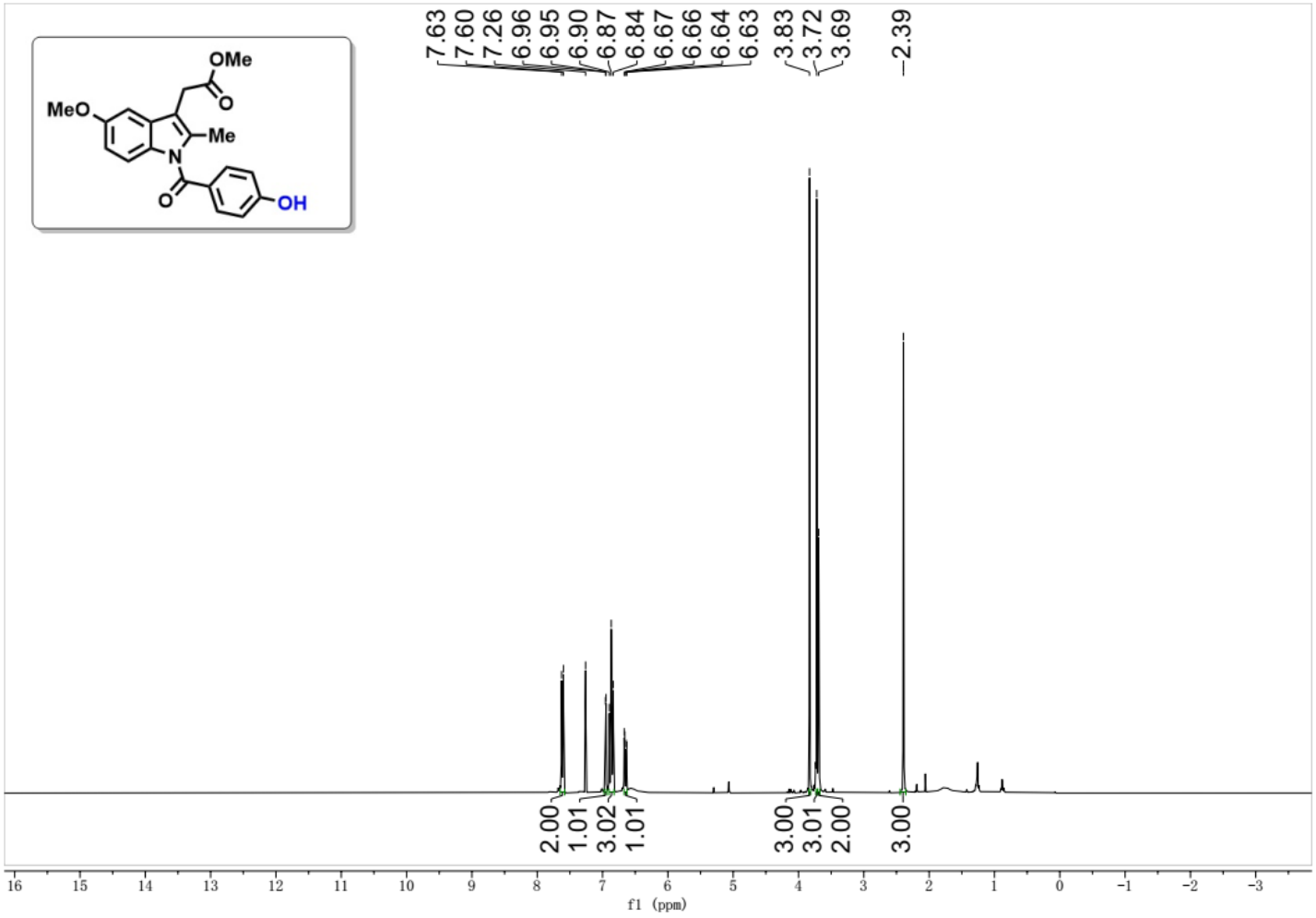
^{1}H NMR of 2au


7.80
7.72
7.71
7.70
7.69
7.26
6.92
6.90
6.87
6.85
5.13
5.12
5.10
5.09
5.07
5.06
5.04
1.65
1.21
1.20
HO
O
O
O
O
1.01
4.02
4.01
1.00
6.01
6.00
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
−1
−2
−3
f1 (ppm)

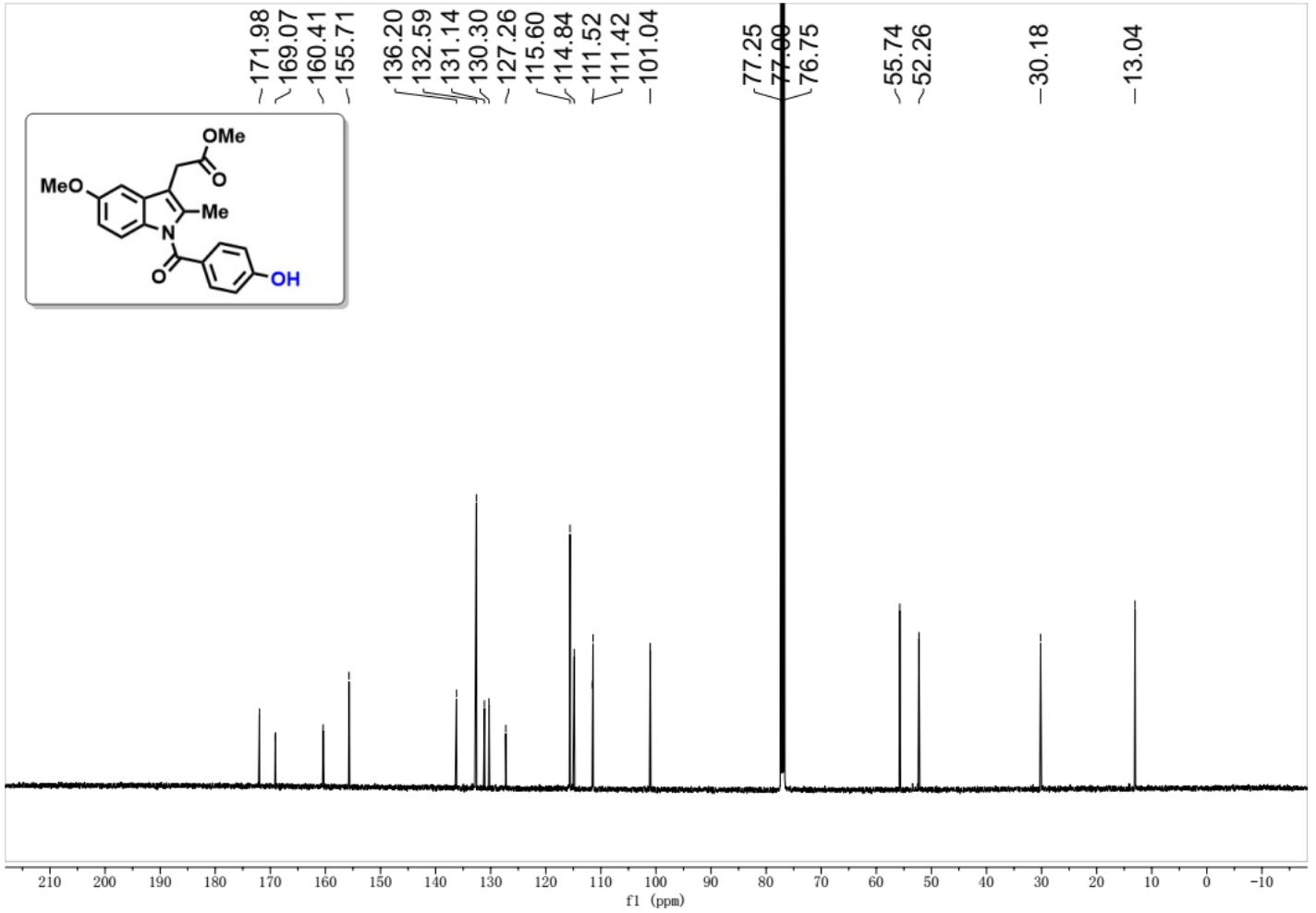
^{13}C NMR of 2au



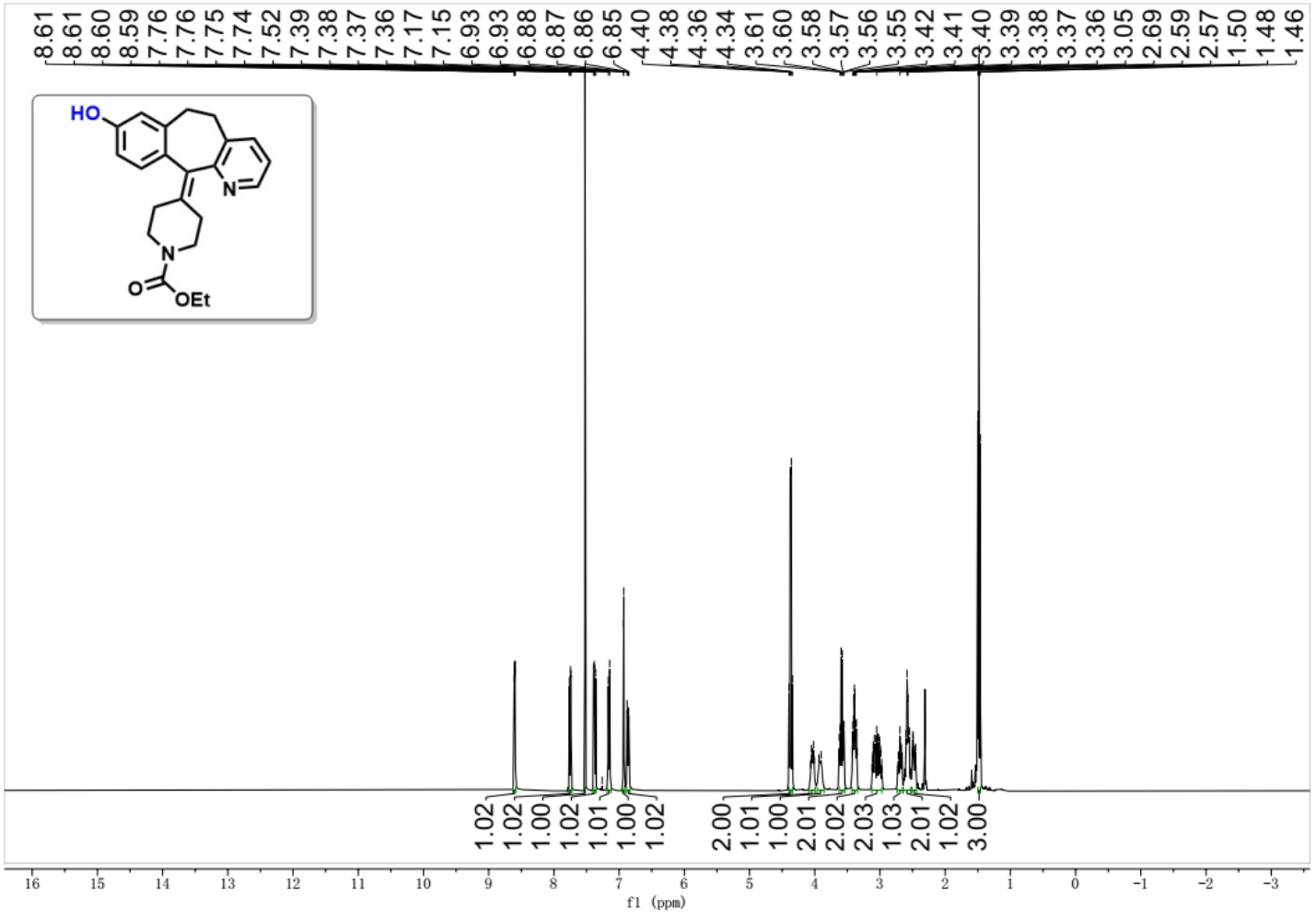
^{1}H NMR of 2av


OMe
MeO
Me
N
O
OH
7.63
7.60
7.26
6.96
6.95
6.90
6.87
6.84
6.67
6.66
6.64
6.63
3.83
3.72
3.69
2.39
2.00
1.01
3.02
1.01
3.00
3.01
2.00
3.00
f1 (ppm)

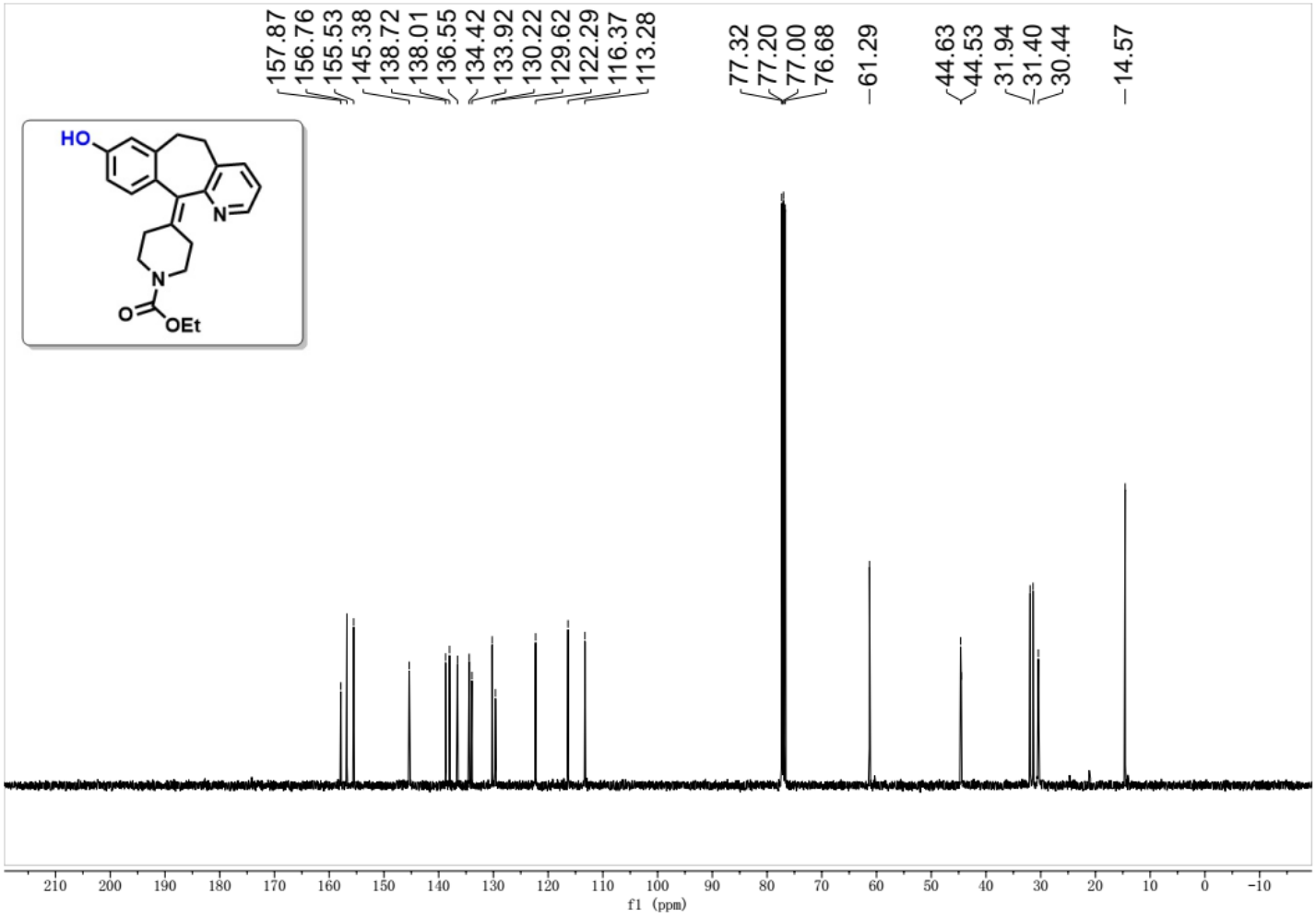
^{13}C NMR of 2av



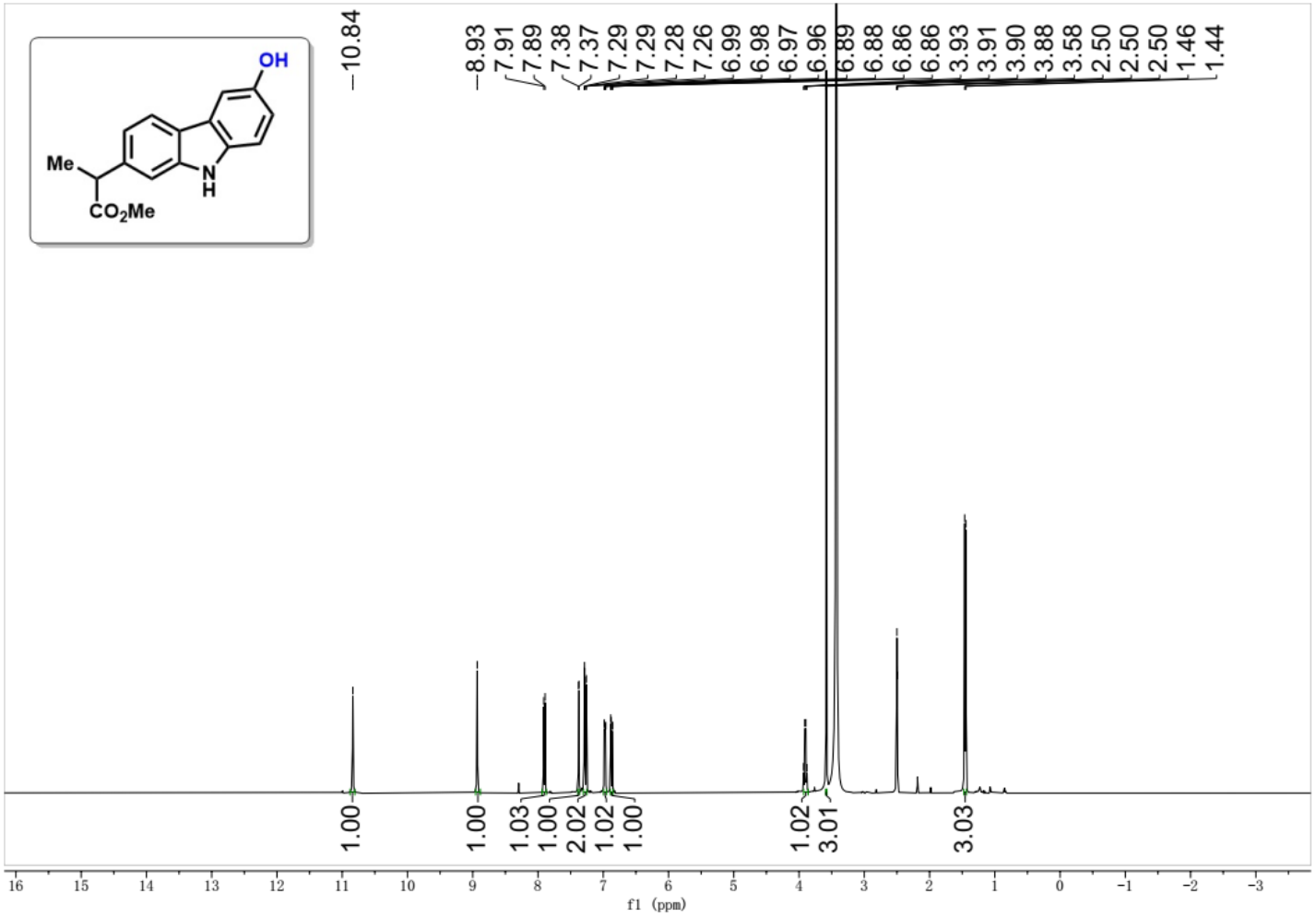
^{1}H NMR of 2aw


8.61
8.61
8.60
8.59
7.76
7.76
7.75
7.74
7.52
7.39
7.38
7.37
7.36
7.17
7.15
6.93
6.93
6.88
6.87
6.86
6.85
4.40
4.38
4.36
4.34
3.61
3.60
3.58
3.57
3.56
3.55
3.42
3.41
3.40
3.39
3.38
3.37
3.36
3.05
2.69
2.59
2.57
1.50
1.48
1.46
HO
N
N
O
OEt
1.02
1.02
1.00
1.02
1.01
1.00
1.02
2.00
1.01
1.00
2.01
2.02
2.03
1.03
2.01
1.02
3.00
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
−1
−2
−3
f1 (ppm)

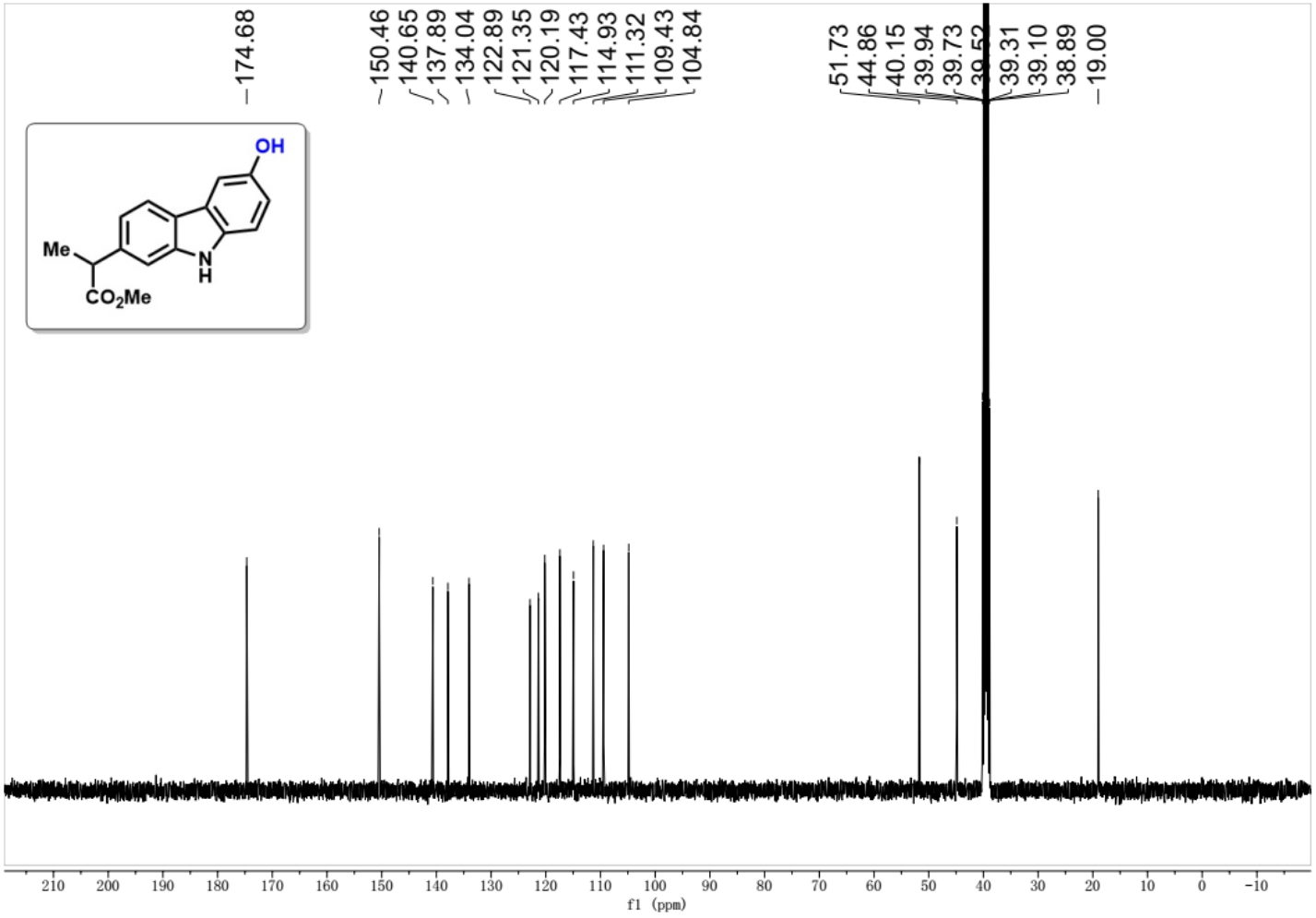
^{13}C NMR of 2aw



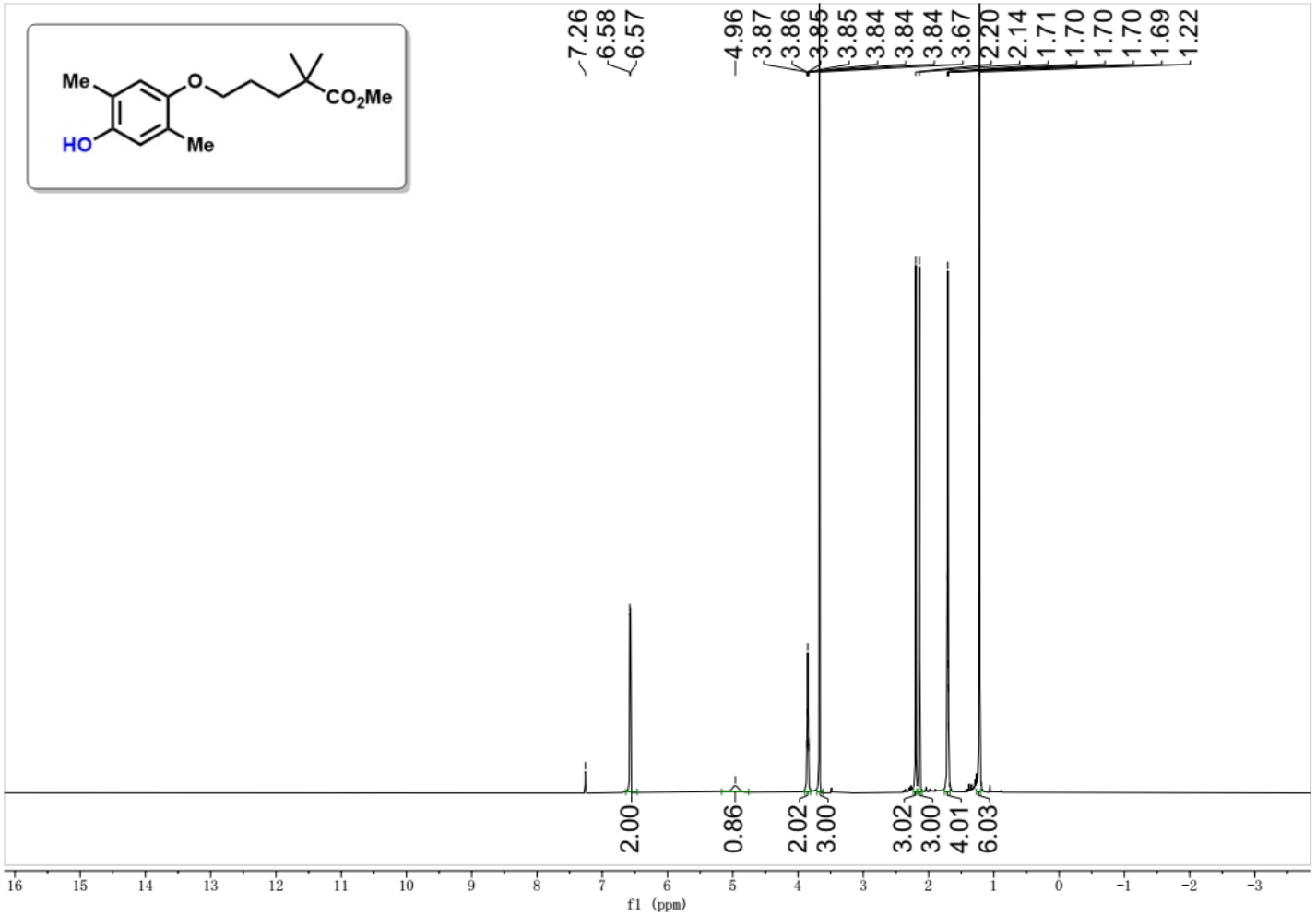
^{1}H NMR of 2ax


OH
Me
N
H
CO2Me
10.84
8.93
7.91
7.89
7.38
7.37
7.29
7.29
7.28
7.26
6.99
6.98
6.97
6.96
6.89
6.88
6.86
6.86
3.93
3.91
3.90
3.88
3.58
2.50
2.50
2.50
1.46
1.44
1.00
1.00
1.03
1.00
2.02
1.02
1.00
1.02
3.01
3.03
f1 (ppm)

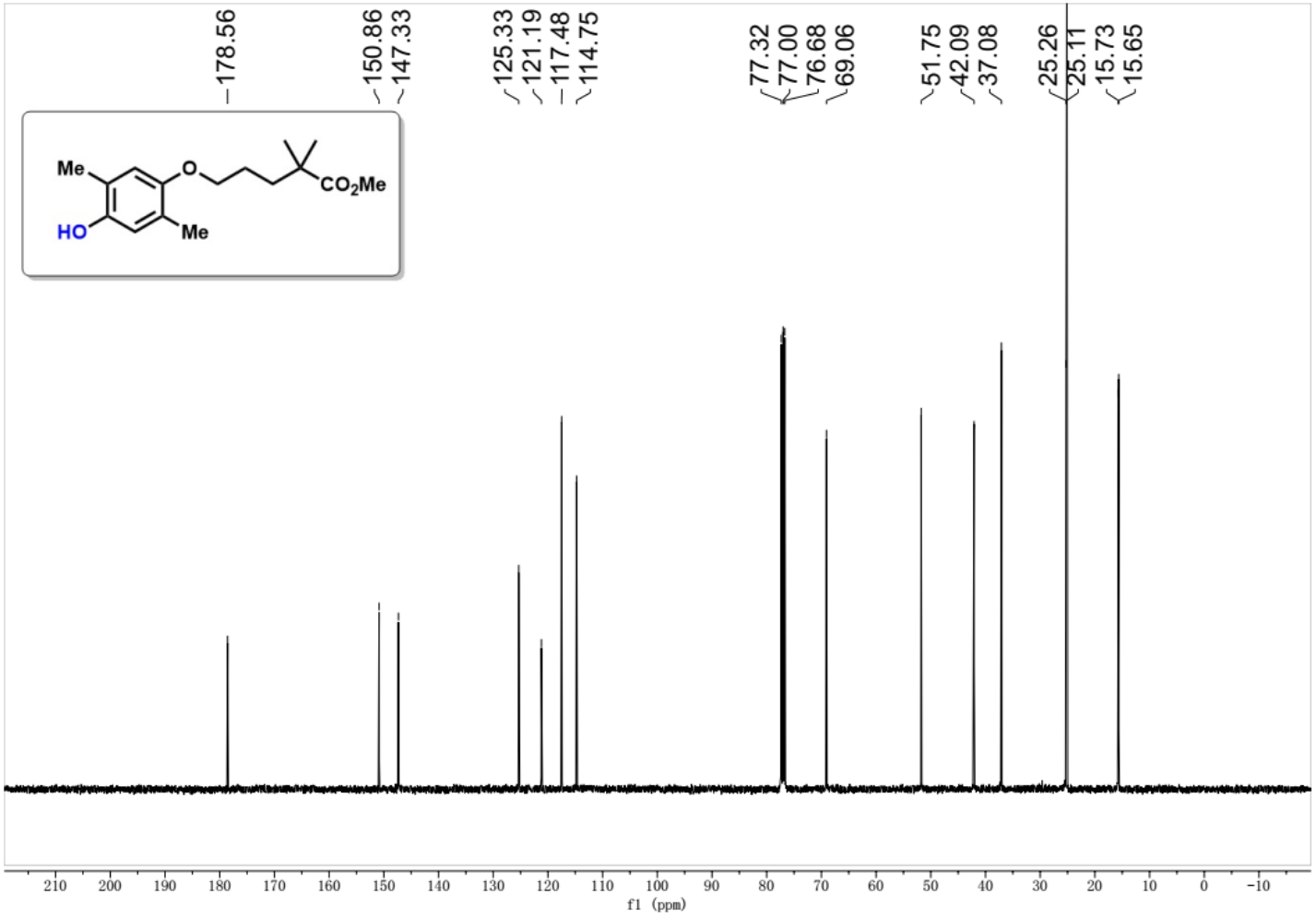
^{13}C NMR of 2ax



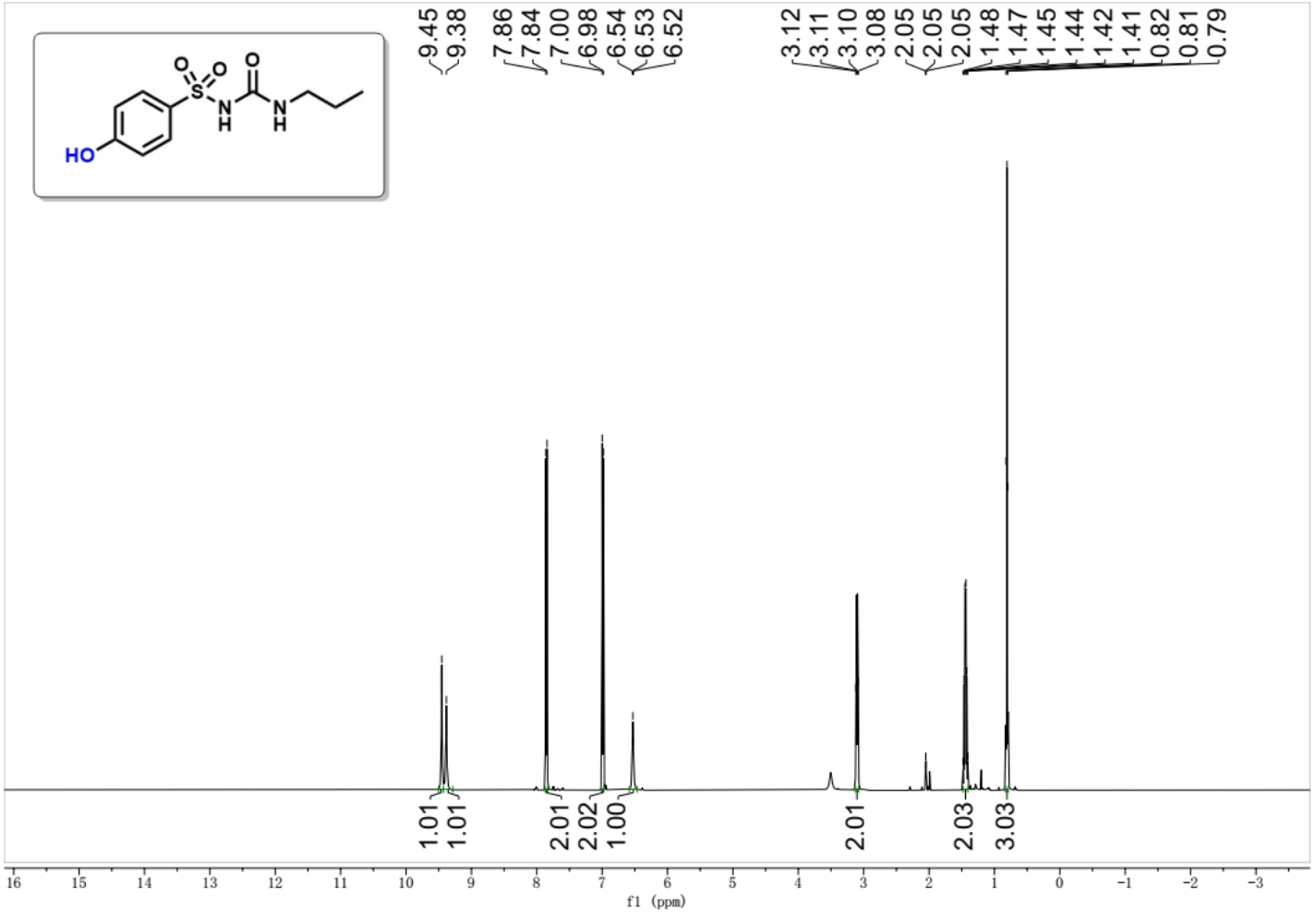
^{1}H NMR of 2ay


Me
O
CO2Me
HO
Me
7.26
6.58
6.57
4.96
3.87
3.86
3.85
3.85
3.84
3.84
3.84
3.67
2.20
2.14
1.71
1.70
1.70
1.70
1.69
1.22
2.00
0.86
2.02
3.00
3.02
3.00
4.01
6.03
f1 (ppm)

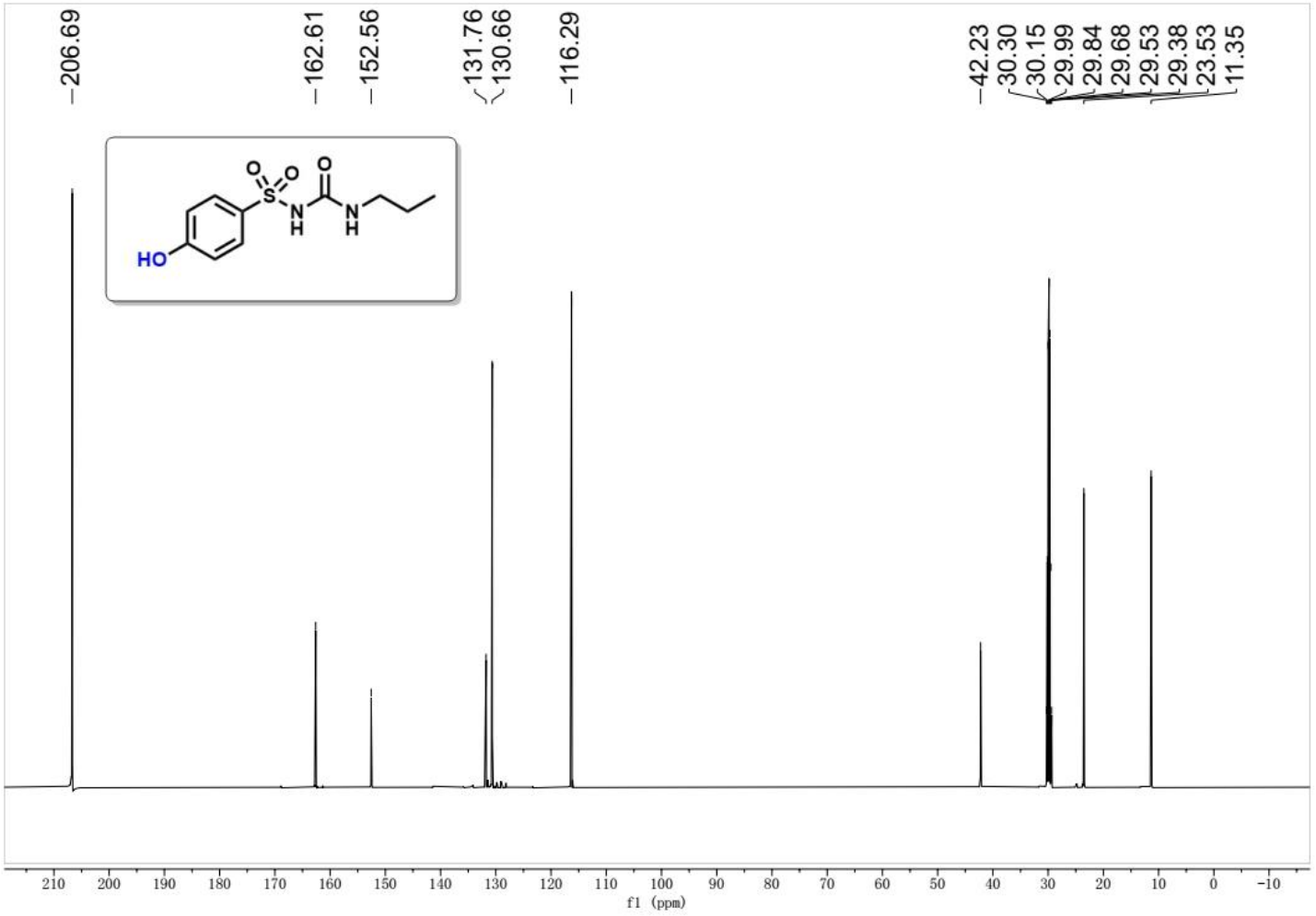
^{13}C NMR of 2ay



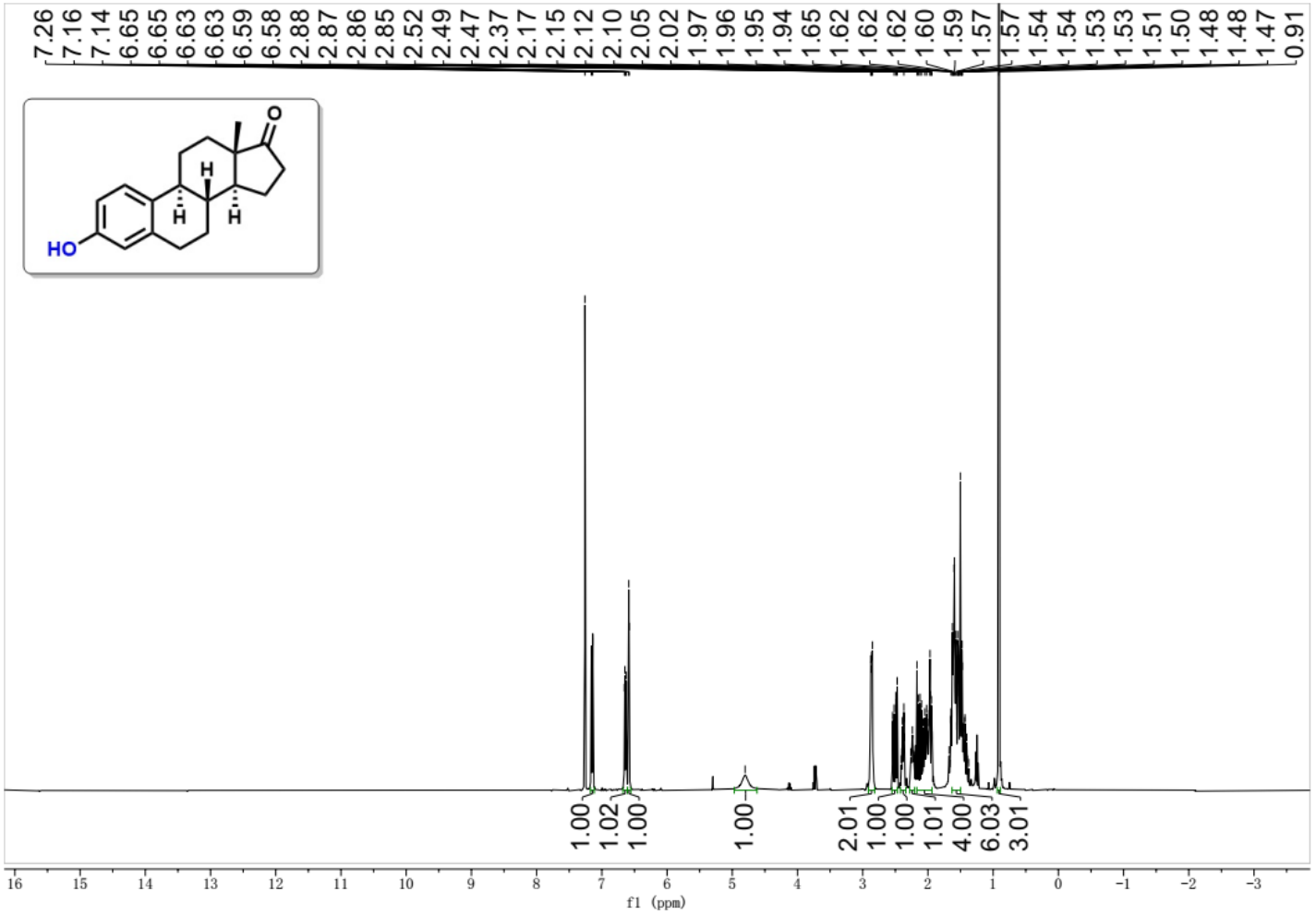
^{1}H NMR of 2az


HO
9.45
9.38
7.86
7.84
7.00
6.98
6.54
6.53
6.52
3.12
3.11
3.10
3.08
2.05
2.05
2.05
1.48
1.47
1.45
1.44
1.42
1.41
0.82
0.81
0.79
1.01
1.01
2.01
2.02
1.00
2.01
2.03
3.03
f1 (ppm)

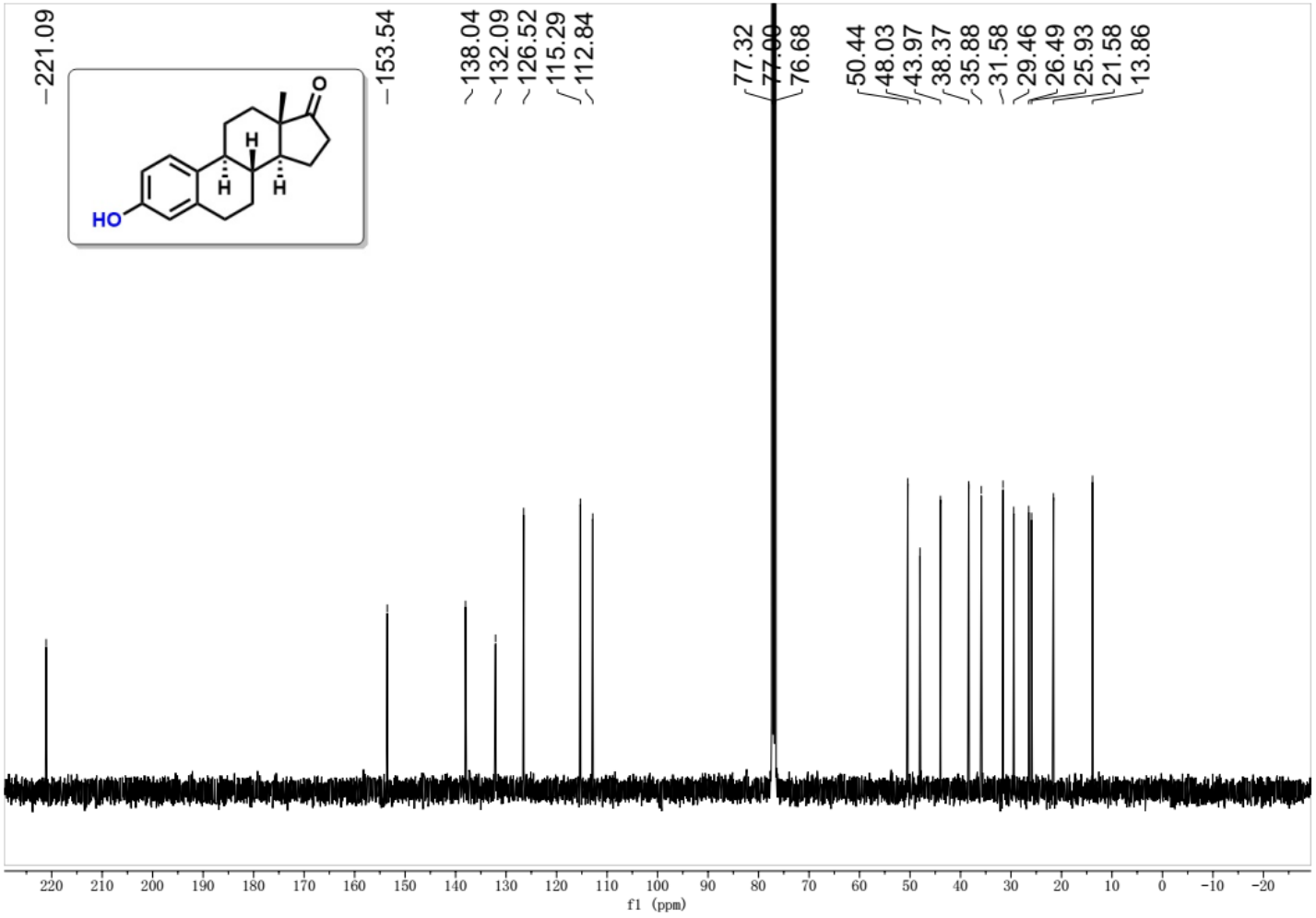
^{13}C NMR of 2az



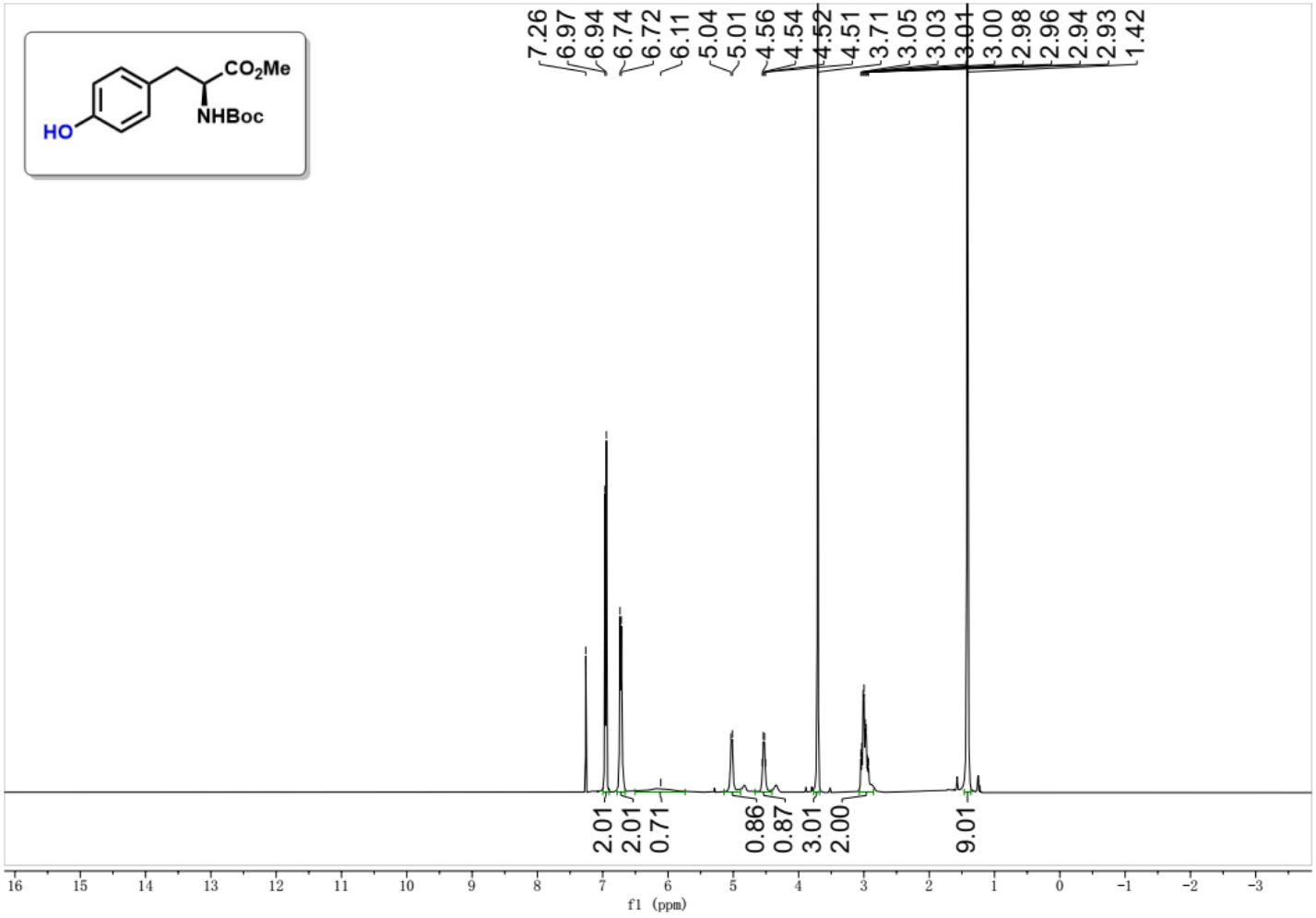
^{1}H NMR of 2ba


7.26
7.16
7.14
6.65
6.65
6.63
6.63
6.59
6.58
2.88
2.87
2.86
2.85
2.52
2.49
2.47
2.37
2.17
2.15
2.12
2.10
2.05
2.02
1.97
1.96
1.95
1.94
1.65
1.62
1.62
1.62
1.60
1.59
1.57
1.57
1.54
1.54
1.53
1.53
1.51
1.50
1.48
1.48
1.47
0.91
HO
O
H
H
H
1.00
1.02
1.00
1.00
2.01
1.00
1.00
1.01
4.00
6.03
3.01
f1 (ppm)

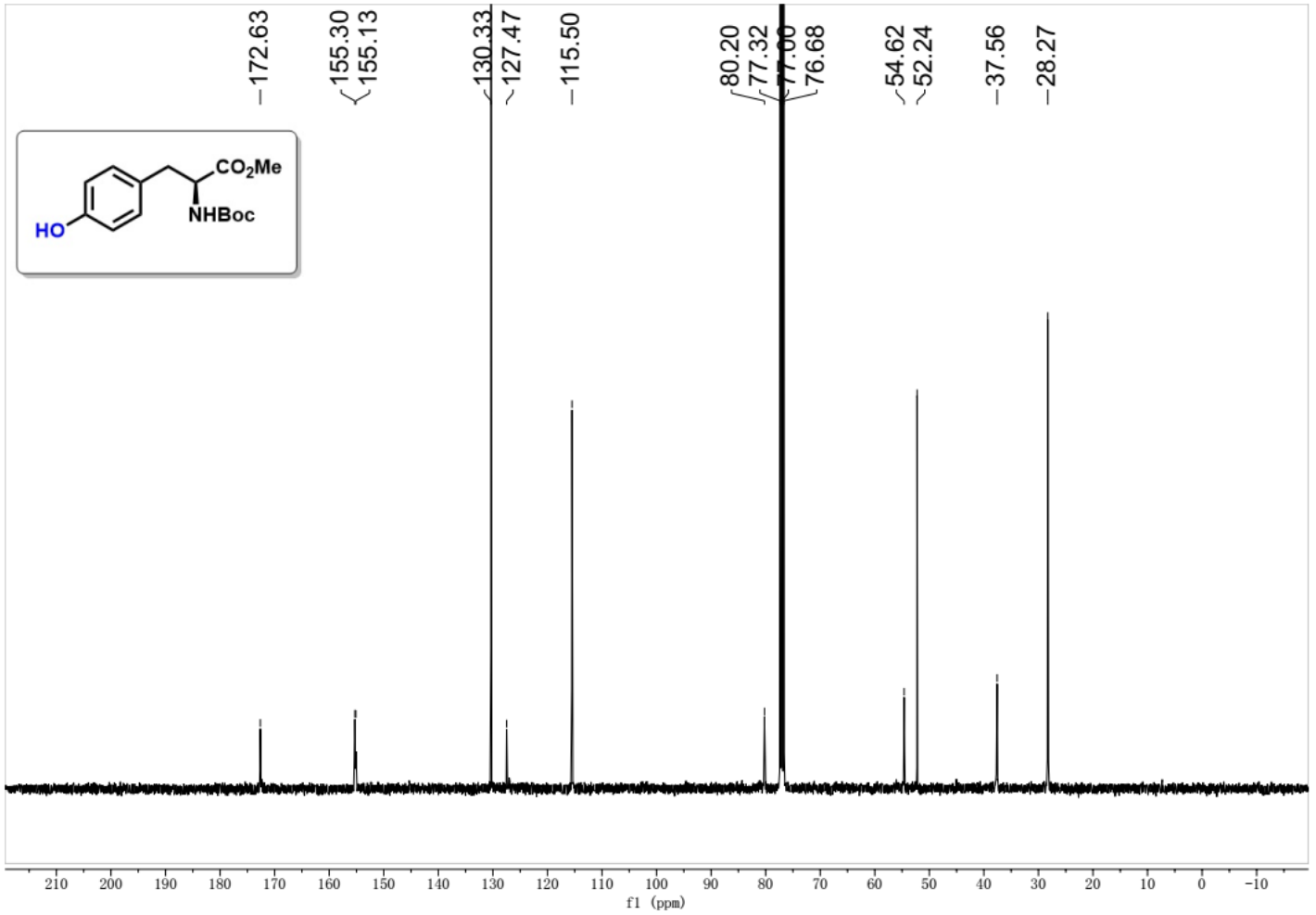
^{13}C NMR of 2ba



^{1}H NMR of 2bb


HO
CO2Me
NHBoc
7.26
6.97
6.94
6.74
6.72
6.11
5.04
5.01
4.56
4.54
4.52
4.51
3.71
3.05
3.03
3.01
3.00
2.98
2.96
2.94
2.93
1.42
2.01
2.01
0.71
0.86
0.87
3.01
2.00
9.01
f1 (ppm)

^{13}C NMR of 2bb



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