Supporting Information

Uranyl-Photocatalyzed Hydrolysis of Diaryl Ethers at Ambient Environment for Directional Degradation of 4-O-5 Lignin

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I. General Information

NMR Spectrum:

^{1}H and ^{13}C spectra were collected 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, DMSO = δ 2.50). 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, DMSO = δ 39.52). Dates are represented as follows: chemical shift, multiplicity (brs = broad single , s = singlet, d = double, t = triplet, q = quartet, m = multiplet), coupling constants in Hertz (Hz), integration.

Mass Spectroscopy:

Mass spectra were in general recorded on a Shimadzu GCMS-QP2010 Ultra and a HP 5989A mass selective detector.

Chromatography:

Column chromatography was performed with silica gel (300 – 400 mesh ASTM).

IR:

SHIMADZU IR Tracer-100 Spectrometers.

Solvent:

Acetonitrile (CH_3CN) was bought and used without further purification, and acetone was freshly distilled with $CaSO_4$ at 80^{o}C.

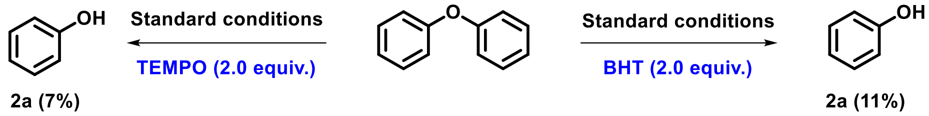
Starting materials:

Except for those commercially available, other diphenyl ether derivatives were prepared adopting reported procedures.[1]

II. Mechanistic Study

1) Radical Quenching Experience

All reactions were operated under standard conditions with extra 2,2,6,6-tetramethyl-1-piperinedinyloxy (TEMPO) or butylated hydroxytoluene (BHT). The yields were determined by NMR yields with CH_2Br_2 as the internal standard.



Radical quenching experiments revealed that TEMPO or BHT could efficiently quench the process of the cleavage of C-O bond, suggested the radical property of this system.

2) Ultraviolet-Visible 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 4 x 10^{-3} M.

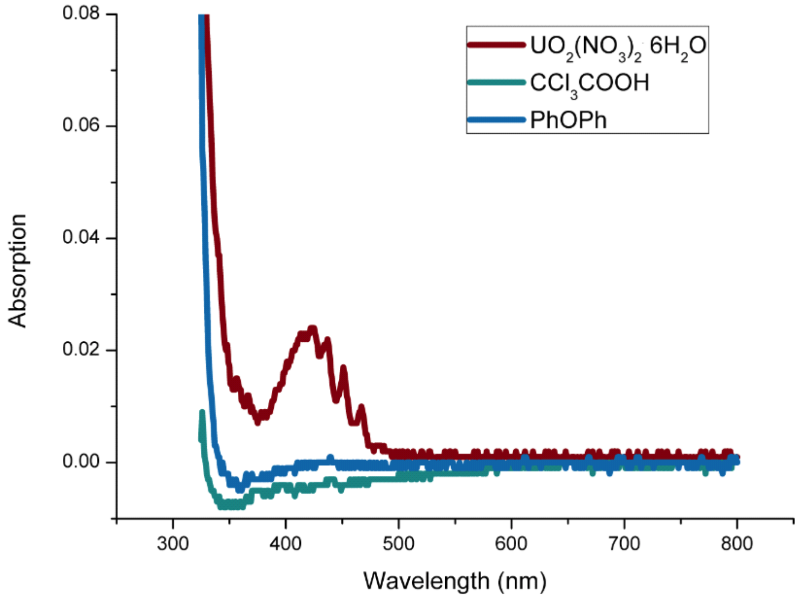


Figure S1. UV-Vis experiments of $UO_2(NO_3)_2 \cdot 6H_2O$, CCl_3COOH and PhOPh.

Uranyl cation was approved to serve as photosensor at 424 nm.

3) Stern–Volmer Fluorescence Quenching Experiments with $UO_2(NO_3)_2·6H_2O$

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 $UO_2(NO_3)_2·6H_2O$, the photoredox catalyst concentration was 4 x 10^{-3} M, the solution was irradiated at 424 nm.

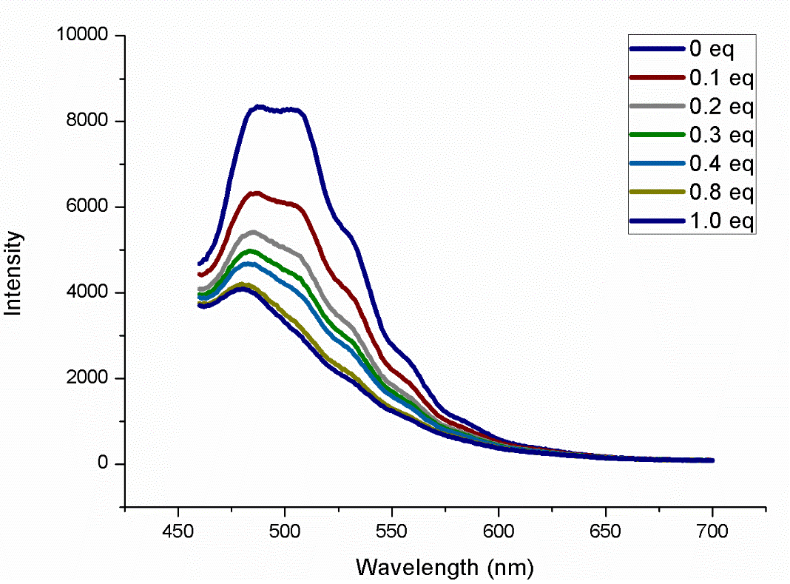


Figure S2. Quenching experiments of $UO_2(NO_3)_2·6H_2O$ with PhOPh.

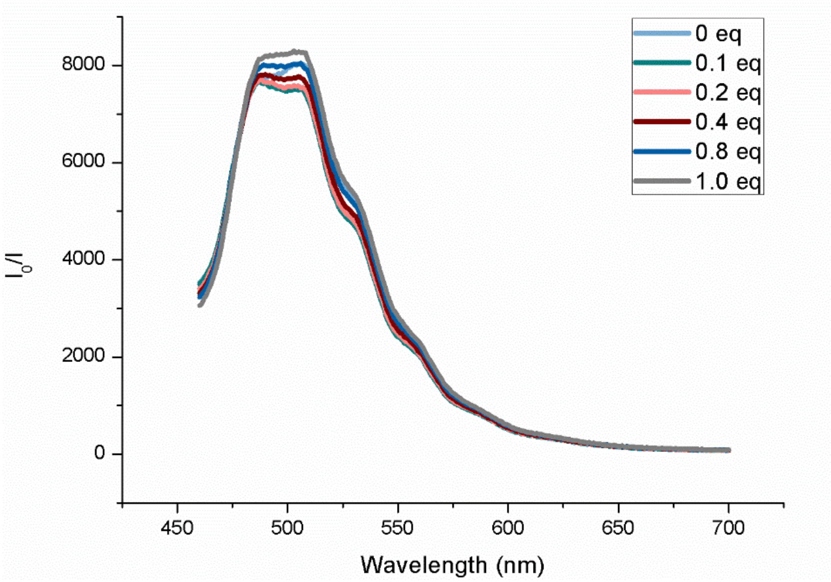


Figure S3. Quenching experiments of $UO_2(NO_3)_2·6H_2O$ with CCl_3COOH.

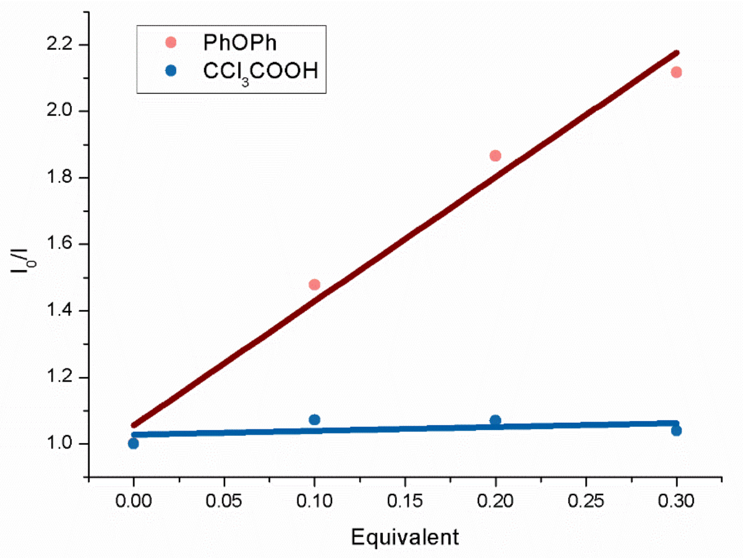


Figure S4. Stern-Volmer plot of Fluorescence Quenching Experiments

The Stern-Volmer analysis revealed that the excited state of $UO_2(NO_3)_2{\cdot}6H_2O$ *photoredox catalysis is efficiently quenched by PhOPh in acetone at room temperature.*

4) Oxygen Labelling Experiment

To a 25 mL Schlenk tube, diaryl ether (0.2 mmol), $UO_2(NO_3)_2{\cdot}6H_2O$ (4 mol%/0.008 mmol, 4 mg), CCl_3COOH (0.4 mmol, 65.2 mg) and $H_2{}^{18}O$ (4 mmol) were stirred in CH_3CN (2 mL) at room temperature for 48 h under blue light (460 nm) in a paralleled reactor in N_2. The yields of each phenol were determined were by ^1H NMR with CH_2Br_2 as the internal standard and GC-MS.

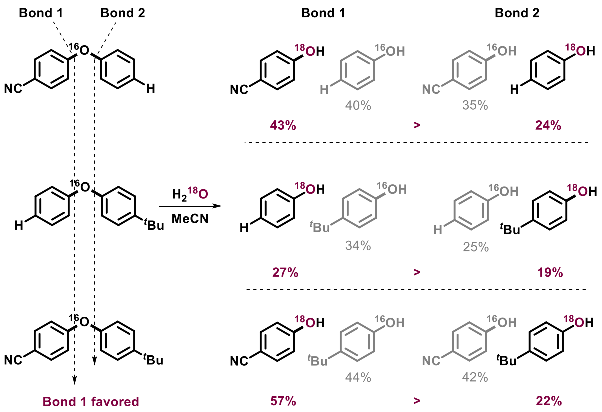


Figure S5. Investigation of Cut-off Bond.

Investigation of Cut-off Bond via ^{18}O*-labeling experiment depicted the cleavage trend on the bias of low electron cloud density side.*

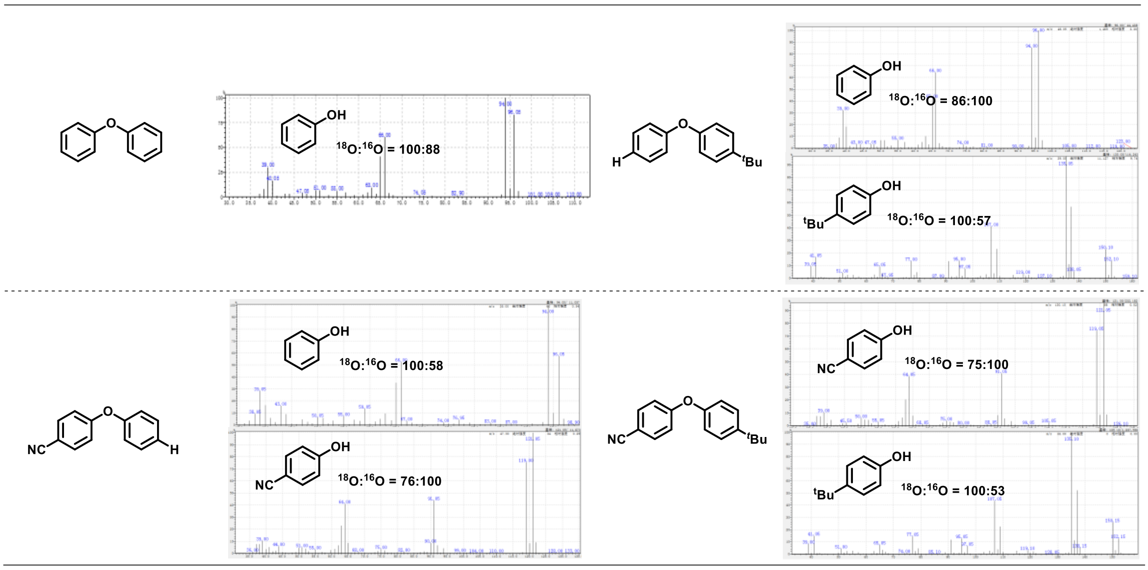
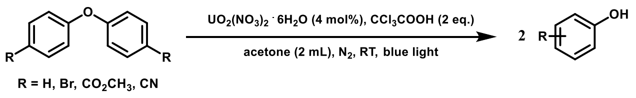
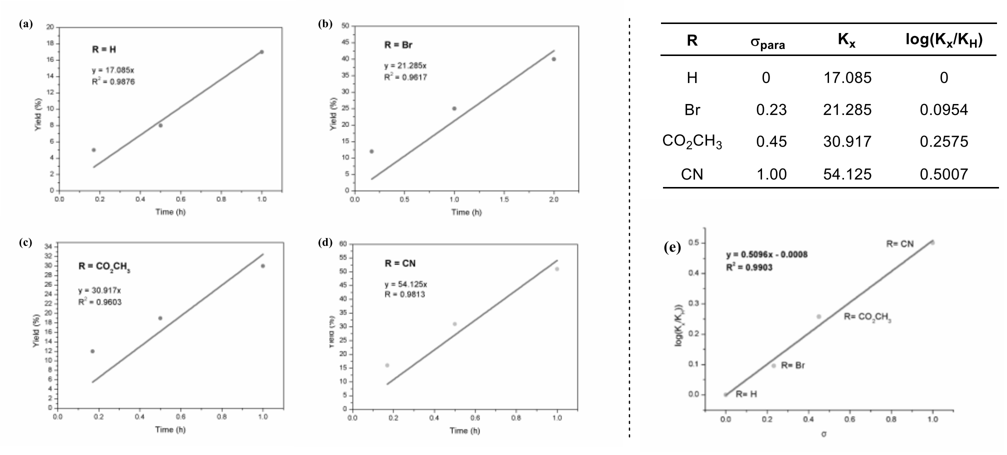


Figure S6. Ratio of phenol products with ^{18}O and ^{16}O.

5) Hammett Plot

To a 25 mL Schlenk tube, diaryl ether (0.1 mmol), $UO_2(NO_3)_2 \cdot 6H_2O$ (4 mol%/0.004 mmol, 2.0 mg) and CCl_3COOH (0.2 mmol, 32.6 mg) were stirred in freshly-distilled acetone (2 mL) at room temperature for 48 h under blue light (460 nm, 12W) in a paralleled reactor in N_2. The yields were determined by ^1H NMR with CH_2Br_2 as the internal standard.



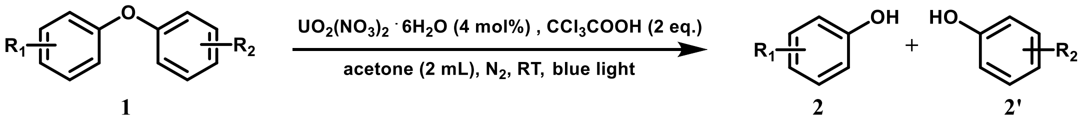


R	σ_{para}	K_x	$\log(K_x/K_H)$
H	0	17.085	0
Br	0.23	21.285	0.0954
CO_2CH_3	0.45	30.917	0.2575
CN	1.00	54.125	0.5007

Figure S7. Hammett plot. a) Rate for diphenyl ether. b) Rate for diaryl ether with -Br. c) Rate for diaryl ether with -CO_2CH_3. d) Rate for diaryl ether with -CN. e) Hammett plot for cleavage of C-O bond. Equation: y =0.5096x – 0.0008; R^2 = 0.9903.

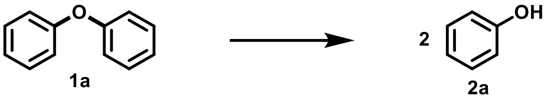
ρ value of +0.5096 for reactions showed that electron-withdrawing groups promoted the transformation and the decisive step was the process of negative charge accumulation.

III. General Procedures



To a 25 mL Schlenk tube, diphenyl ether (0.4 mmol), $UO_2(NO_3)_2 \cdot 6H_2O$ (4 mol%/0.008 mmol, 4 mg), CCl_3COOH (0.8 mmol, 130.7 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 48 h under blue LEDs (460 nm, 9 W) in a paralleled reactor. After evaporation of solvent, the residue was purified by column chromatography on silica gel using PE/EA to afford the corresponding phenols. For detailed modification, please see the corresponding procedure.

IV. Procedures and Data



To a 25 mL Schlenk tube, diphenyl ether (0.4 mmol, 68.0 mg), $UO_2(NO_3)_2{\cdot}6H_2O$ (4 mol%/0.016 mmol, 8 mg), CCl_3COOH (0.8 mmol, 130.7 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78°C then stirred for 48 h under blue LEDs (3x3W, 460 nm) in a paralleled reactor. **2a**[2] (60.2 mg, 80%) was obtained through column chromatography (V_{PE}/V_{EA} = 10/1) as a brown liquid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.28 - 7.24 (m, 2H), 6.95 (t, J = 8.0 Hz, 1H), 6.85 (d, J = 8.0 Hz, 2H), 5.02 (brs, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 155.3, 129.7, 120.8, 115.3. **IR** (neat) 3237, 1594, 1498, 1472, 1221, 1071, 810 cm^{-1}. **MS** (EI) m/z 94.

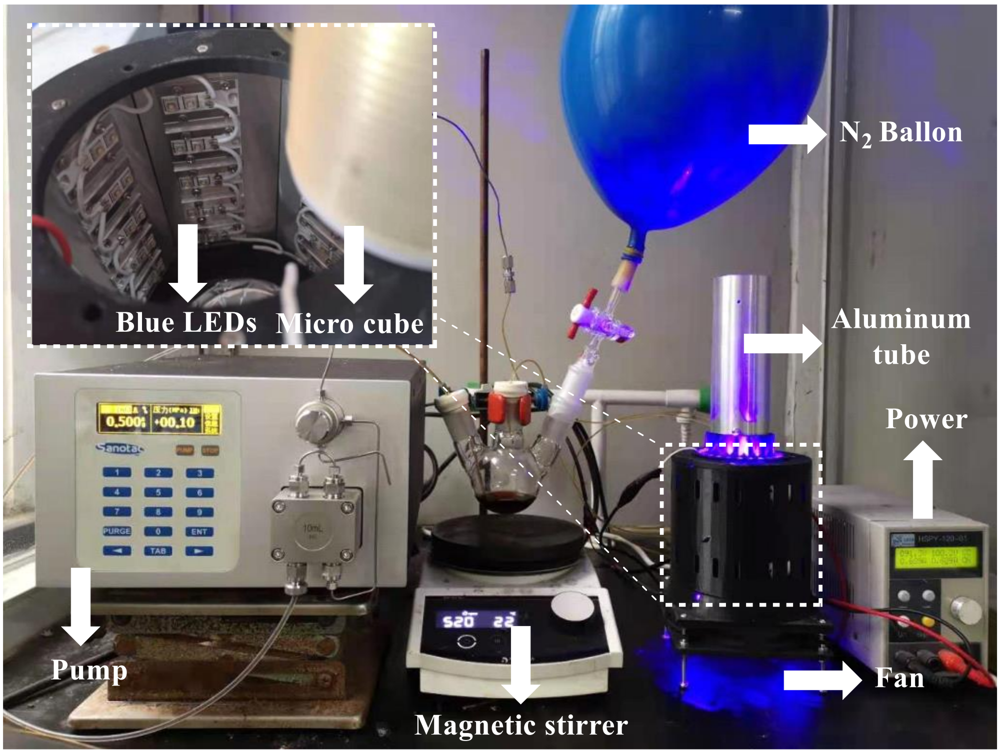
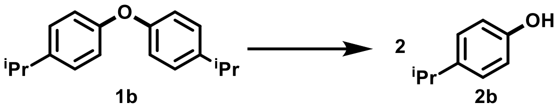
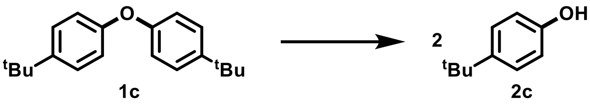


Figure S8. Flow Reaction Photoreactor.

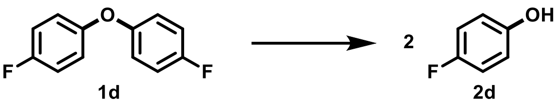
Flow reaction: To a 250 mL three-necked flask, a solution of diphenyl ether (10.0 mmol, 1.70g), $UO_2(NO_3)_2{\cdot}6H_2O$ (4 mol%/0.4 mmol, 201 mg) and CCl_3COOH (20.0 mmol, 3.26 g) in freshly-distilled acetone (50 mL) was added. The flask was equipped with rubber plugs, with inlet and outlet of micro tube, which was made of PTFE tubing (O.D. = 2 mm, I.D. = 1 mm, length = 5.68 m, volume = 4.45 mL). Then the flask along with the micro cube were evacuated and filled with N_2 three times at -78 °C. The solution was pumped by a pump (0.5 mL/min) into the micro tube, then returned to flask. This circulatory system was irradiated by blue LEDs (430 nm, 54 W totally) for 72 h (the temperature was below 30 °C). After the reaction, acetone (10 mL) was pumped into the tube to flush out residual fluid. The solvent was removed in vacuo and the crude residue was purified through column chromatography on silica gel using PE/EA (10/1) to afford **2a** (1.16 g, 62%) as a brown liquid.



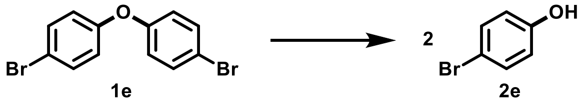
To a 25 mL Schlenk tube, 4,4'-oxybis(isopropylbenzene) (0.2 mmol, 50.8 mg), $UO_2(NO_3)_2{\cdot}6H_2O$ (4 mol%/0.008 mmol, 4 mg), CCl_3COOH (0.4 mmol, 65.4 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 48 h under blue light (460nm, 9W) in a paralleled reactor. **2b**[3] (34.8 mg, 64%) was obtained through column chromatography (V_{PE}/V_{EA} = 10/1) as a brown solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ7.11 (d, *J* = 8.0 Hz, 2H), 6.78 (d, *J* = 8.0 Hz, 2H), 4.43 (brs, 1H), 2.89 - 2.83 (m, 1H), 1.23 (d, *J* = 8.0 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 153.3, 127.4, 115.1, 33.3, 24.2. **IR** (neat) 3317, 2959, 1612, 1512, 1225, 1174, 827 cm^{-1}. **MS** (EI) m/z 136.



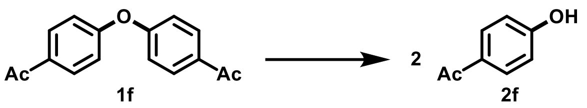
To a 25 mL Schlenk tube, 4,4'-oxybis(tert-butylbenzene) (0.2 mmol, 56.4 mg), $UO_2(NO_3)_2{\cdot}6H_2O$ (4 mol%/0.008 mmol, 4 mg), CCl_3COOH (0.4 mmol, 65.4 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 48 h under blue LEDs (460 nm, 9W) in a paralleled reactor. **2c**[4] (40.8 mg, 68 %) was obtained through column chromatography (V_{PE}/V_{EA} =) as a white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.32 - 7.28 (m, 2H), 6.84 - 6.80 (m, 2H), 4.95 (brs, 1H), 1.34 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ153.1, 143.6, 126.4, 114.7, 34.0, 31.5. **IR** (neat) 3235, 2961, 1703, 1515, 1361, 1232, 1181 cm^{-1}. **MS** (EI) m/z 150.



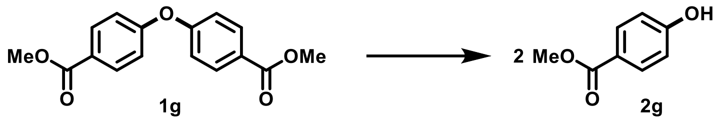
To a 25 mL Schlenk tube, 4,4'-oxybis(fluorobenzene) (0.2 mmol, 41.2 mg), $UO_2(NO_3)_2 \cdot 6H_2O$ (4 mol%/0.008 mmol, 4 mg), CCl_3COOH (0.4 mmol, 65.4 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 48 h under blue light (460nm, 9W) in a paralleled reactor. **2d**[4] (21.9 mg, 49%) was obtained through column chromatography (V_{PE}/V_{EA} = 10/1) as a brown liquid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.95 - 6.91 (m, 2H), 6.79 - 6.75 (m, 2H), 4.71 (brs, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 157.3 (d, *J* = 237 Hz), 151.4 (d, *J* = 2.0 Hz), 116.2 (d, *J* = 8.0 Hz), 116.0 (d, *J* = 23.0 Hz). **^{19}F NMR** (376 MHz, $CDCl_3$) δ -124.16. **IR** (neat) 3346, 1507, 1223, 1195, 1091, 829, 746 cm^{-1}. **MS** (EI) m/z 112.



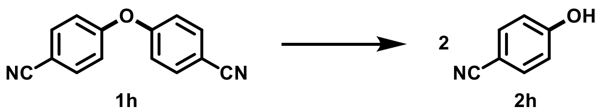
To a 25 mL Schlenk tube, 4,4'-oxybis(bromobenzene) (0.2 mmol, 65.2 mg), $UO_2(NO_3)_2 \cdot 6H_2O$ (4 mol%/0.008 mmol, 4 mg), CCl_3COOH (0.4 mmol, 65.4 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 72 h under blue light (460nm, 9W) in a paralleled reactor. **2e**[5] (49.5 mg, 72%) was obtained through column chromatography (V_{PE}/V_{EA} = 10/1) as a brown liquid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.35 - 7.32 (m, 2H), 6.74 - 6.70 (m, 2H), 4.96 (brs, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 154.5, 132.5, 117.2, 112.9. **IR** (neat) 3322, 1559, 1491, 1432, 1165, 1091, 1009 cm^{-1}. **MS** (EI) m/z 172.



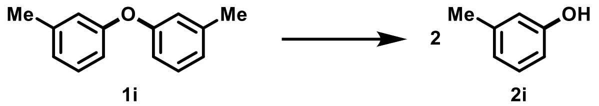
To a 25 mL Schlenk tube, 1,1'-(oxybis(4,1-phenylene))bis(ethan-1-one) (0.2 mmol, 50.8 mg), $UO_2(NO_3)_2{\cdot}6H_2O$ (4 mol%/0.008 mmol, 4 mg), CCl_3COOH (0.4 mmol, 65.4 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 48 h under blue light (460nm, 9W) in a paralleled reactor. **2f**[5] (39.2 mg, 72%) was obtained through column chromatography (V_{PE}/V_{EA} = 5/1) as a brown solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.0 Hz, 2H), 6.90 (d, *J* = 8.0 Hz, 2H), 6.24 (brs, 1H), 2.56 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 198.0, 160.9, 131.1, 129.8, 115.5, 26.3. **IR** (neat) 3384, 1587, 1525, 1488, 1350, 1069, 1006 cm^{-1}. **MS** (EI) m/z 136.



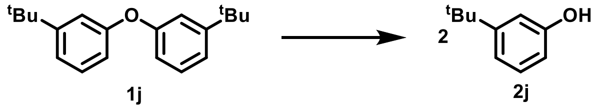
To a 25 mL Schlenk tube, dimethyl 4,4'-oxydibenzoate (0.2 mmol, 57.2 mg), $UO_2(NO_3)_2{\cdot}6H_2O$ (4 mol%/0.008 mmol, 4 mg), CCl_3COOH (0.4 mmol, 65.4 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 48 h under blue light (460nm, 9W) in a paralleled reactor. **2g**[5] (53.5 mg, 88%) was obtained through column chromatography (V_{PE}/V_{EA} = 5/1) as a white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.95 (d, *J* = 8.0 Hz, 2H), 6.88 (d, *J* = 8.0 Hz, 2H), 3.90 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.4, 160.1, 131.9, 122.3, 115.2, 52.0. **IR** (neat) 3276, 1688, 1438, 1235, 1172, 859, 770 cm^{-1}. **MS** (EI) m/z 152.



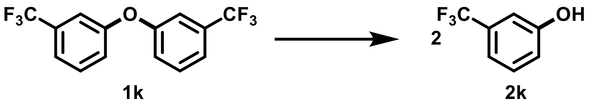
To a 25 mL Schlenk tube, 4,4'-oxydibenzonitrile (0.2 mmol, 44.0 mg), $UO_2(NO_3)_2{\cdot}6H_2O$ (4 mol%/0.008 mmol, 4 mg), CCl_3COOH (0.4 mmol, 65.4 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 48 h under blue light (460nm, 9W) in a paralleled reactor. **2h**[5] (46.2 mg, 97%) was obtained through column chromatography (V_{PE}/V_{EA} = 5/1) as a white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.55 (d, *J* = 8.7 Hz, 2H), 6.93 (d, *J* = 8.7 Hz, 2H), 6.49 (brs, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 160.0, 134.3, 119.2, 116.4, 103.2. **IR** (neat) 3293, 2235, 1610, 1587, 1510, 1286, 1167 cm^{-1}. **MS** (EI) m/z 119.



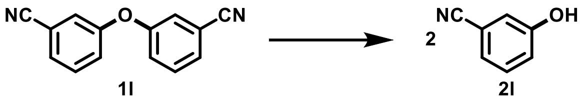
To a 25 mL Schlenk tube, 3,3'-oxybis(methylbenzene) (0.2 mmol, 39.6 mg), $UO_2(NO_3)_2{\cdot}6H_2O$ (4 mol%/0.008 mmol, 4 mg), CCl_3COOH (0.4 mmol, 65.4 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 48 h under blue light (460nm, 9W) in a paralleled reactor. **2i**[5] (35.8 mg, 83%) was obtained through column chromatography (V_{PE}/V_{EA} = 10/1) as a pink liquid .**^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.13 (t, *J* = 8.0 Hz, 1H), 6.75 (d, *J* = 8.0 Hz, 1H), 6.65 (d, *J* = 12.0 Hz, 2H), 4.87 (brs, 1H), 2.31 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 155.3, 139.8, 129.4, 121.6, 116.0, 112.2, 21.3. **IR** (neat) 3314, 1589, 1491, 1278, 1154, 926, 773 cm^{-1}. **MS** (EI) m/z 108.



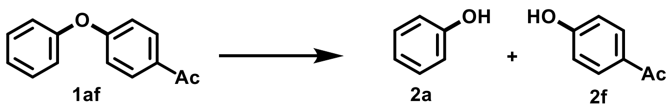
To a 25 mL Schlenk tube, 3,3'-oxybis(tert-butylbenzene) (0.4 mmol, 113.0 mg), $UO_2(NO_3)_2{\cdot}6H_2O$ (4 mol%/0.016 mmol, 8 mg), CCl_3COOH (0.8 mmol, 130.7 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 72 h under blue light (460nm, 9W) in a paralleled reactor. **2j**[6] (84.1 mg, 74%) was obtained through column chromatography (V_{PE}/V_{EA} = 10/1) as a white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.18 (t, *J* = 8.0 Hz, 1H), 6.99 - 6.97 (m, 1H), 6.88 - 6.87 (m, 1H), 6.67 - 6.64 (m, 1H), 4.81 (brs, 1H), 1.31 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 155.2, 153.3, 129.1, 117.8, 112.5, 112.2, 34.6, 31.2. **IR** (neat) 3307, 2963, 1451, 1280, 914, 780, 699 cm^{-1}. **MS** (EI) m/z 150.



To a 25 mL Schlenk tube, 3,3'-oxybis((trifluoromethyl)benzene) (0.4 mmol, 122.4 mg), $UO_2(NO_3)_2\cdot 6H_2O$ (4 mol%/0.016 mmol, 8 mg), CCl_3COOH (0.8 mmol, 130.7 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 72 h under blue light (460nm, 9W) in a paralleled reactor. **2k**[7] (106.3 mg, 82%) was obtained through column chromatography (V_{PE}/V_{EA} = 10/1) as a brown liquid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ7.36 (t, J = 8.0 Hz, 1H), 7.22 (d, J = 8.0 Hz, 1H), 7.10 (s, 1H), 7.02 (dd, J = 8.0, 4.0 Hz, 1H), 5.59 (brs, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) 155.3, 132.1 (q, J = 32.4 Hz), 130.3, 123.7 (q, J = 272.3 Hz), 118.8 (d, J = 1.0 Hz), 117.8 (q, J = 3.9 Hz), 112.3 (q, J = 3.8 Hz). **^{19}F NMR** (376 MHz, $CDCl_3$) δ -62.80. **IR** (neat) 3356, 1460, 1332, 1128, 1064, 894, 791 cm^{-1}. **MS** (EI) m/z 162.

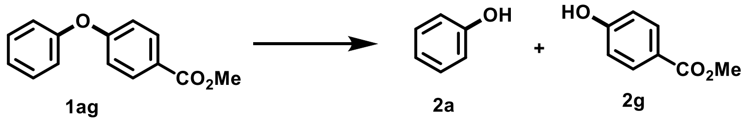


To a 25 mL Schlenk tube, 3,3'-oxydibenzonitrile (0.4 mmol, 88.0 mg), $UO_2(NO_3)_2\cdot 6H_2O$ (4 mol%/0.016 mmol, 8 mg), CCl_3COOH (0.8 mmol, 130.7 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 72 h under blue light (430 nm, 12W) in a paralleled reactor. **2l**[5] (78.0 mg, 82%) was obtained through column chromatography (V_{PE}/V_{EA} = 5/1) as a brown solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.34 (t, J = 8.0 Hz, 1H), 7.26 - 7.22 (m, 1H), 7.14 (m, 1H), 7.11 - 7.09 (m, 1H), 5.90 (brs, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 156.1, 130.6, 124.5, 120.7, 118.7, 118.5, 112.8. **IR** (neat) 3410, 2924, 2242, 1599, 1584, 1318, 1287 cm^{-1}. **MS** (EI) m/z 119.

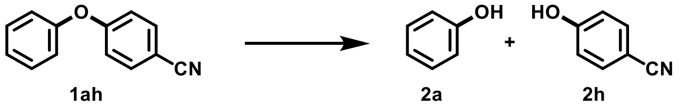


To a 25 mL Schlenk tube, 1-(4-phenoxyphenyl)ethan-1-one (0.2 mmol, 42.4 mg),

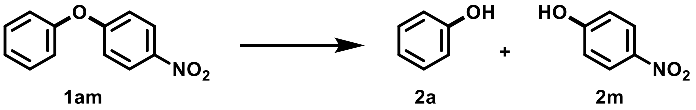
$UO_2(NO_3)_2{\cdot}6H_2O$ (4 mol%/0.008 mmol, 4 mg), CCl_3COOH (0.4 mmol, 65.4 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 72 h under blue light (460nm, 9W) in a paralleled reactor. **2f** was obtained through column chromatography (V_{PE}/V_{EA} = 5/1) as a brown solid (21.5 mg, 79%). **2a** was obtained through column chromatography (V_{PE}/V_{EA} = 10/1) as a brown liquid (7.5 mg, 40%).



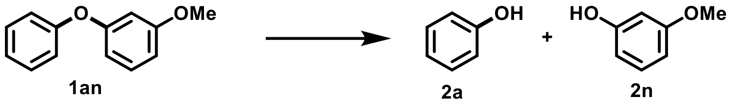
To a 25 mL Schlenk tube, methyl 4-phenoxybenzoate (0.2 mmol, 45.6 mg), $UO_2(NO_3)_2{\cdot}6H_2O$ (4 mol%/0.008 mmol, 4 mg), CCl_3COOH (0.4 mmol, 65.4 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 72 h under blue light (460nm, 9W) in a paralleled reactor. **2g** was obtained through column chromatography (V_{PE}/V_{EA} = 5/1) as a white solid (21.3 mg, 70%). **2a** was obtained through column chromatography (V_{PE}/V_{EA} = 10/1) as a brown liquid (11.7 mg, 62%).



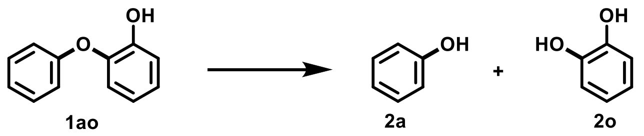
To a 25 mL Schlenk tube, 4-phenoxybenzonitrile (0.2 mmol, 39.0 mg), $UO_2(NO_3)_2{\cdot}6H_2O$ (4 mol%/0.008 mmol, 4 mg), CCl_3COOH (0.4 mmol, 65.4 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 72 h under blue light (460nm, 9W) in a paralleled reactor. **2h** was obtained through column chromatography (V_{PE}/V_{EA} = 5/1) as a white solid (23.4 mg, 99%). **2a** was obtained through column chromatography (V_{PE}/V_{EA} = 10/1) as a brown liquid (13.2 mg, 70%).



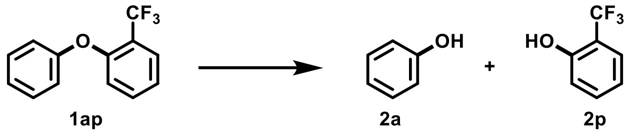
To a 25 mL Schlenk tube, 1-nitro-4-phenoxybenzene (0.2 mmol, 43.0 mg), $UO_2(NO_3)_2\cdot 6H_2O$ (4 mol%/0.008 mmol, 4 mg), CCl_3COOH (0.4 mmol, 65.4 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 72 h under blue light (460nm, 9W) in a paralleled reactor. **2a** was obtained through column chromatography (V_{PE}/V_{EA} = 10/1) as a brown liquid (13.7 mg, 73%). **2m**[8] was obtained through column chromatography (V_{PE}/V_{EA} = 5/1) as a yellow solid (19.2 mg, 69%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.19 - 8.15 (m, 2H), 6.94 - 6.90 (m, 2H), 6.20 (brs, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 161.5, 141.5, 126.2, 115.7. **IR** (neat) 3366, 1613, 1595, 1335, 1297, 1114, 844 cm^{-1}. **MS** (EI) m/z 139.



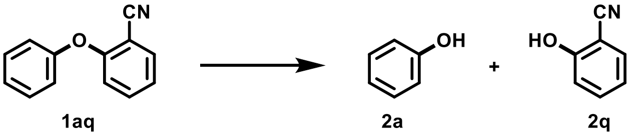
To a 25 mL Schlenk tube, 1-methoxy-3-phenoxybenzene (0.4 mmol, 80.0 mg), $UO_2(NO_3)_2\cdot 6H_2O$ (4 mol%/0.016 mmol, 8 mg), CCl_3COOH (0.8 mmol, 130.7 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 72 h under blue light (430 nm, 12W) in a paralleled reactor. **2a** was obtained through column chromatography (V_{PE}/V_{EA} = 10/1) as a brown liquid (32.3 mg, 56%). **2n**[5] was obtained through column chromatography (V_{PE}/V_{EA} = 10/1) as a brown liquid (mg, 59%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.14 (t, J = 8.1 Hz, 1H), 6.53 - 6.47 (m, 1H), 6.45 - 6.40 (m, 2H), 4.96 (brs, 1H), 3.79 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 160.8, 156.6, 130.1, 107.7, 106.4, 101.4, 55.2. **IR** (neat) 1584, 1479, 1451, 1292, 1250, 1057, 747 cm^{-1}. **MS** (EI) m/z 124.



To a 25 mL Schlenk tube, 2-phenoxyphenol (0.2 mmol, 37.2 mg), $UO_2(NO_3)_2\cdot 6H_2O$ (4 mol%/0.008 mmol, 4 mg), CCl_3COOH (0.4 mmol, 65.4 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 72 h under blue light (460nm, 9W) in a paralleled reactor. **2a** was obtained through column chromatography ($V_{PE}/V_{EA} = 10/1$) as a brown liquid (10.9 mg, 58%). **2o**[11] was obtained through column chromatography ($V_{PE}/V_{EA} = 5/1$) as a white solid (13.4 mg, 61 %). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.88 - 6.86 (m, 2H), 6.84 - 6.81 (m, 2H), 5.23 (brs, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 143.4, 121.2, 115.4. **IR** (neat) 3450, 3327, 1513, 1470, 1364, 1282, 1256 cm^{-1}. **MS** (EI) m/z 110.

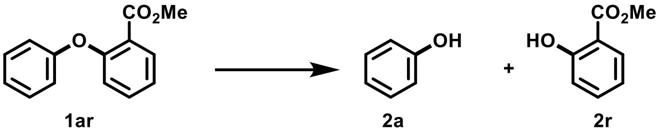


To a 25 mL Schlenk tube, 1-phenoxy-2-(trifluoromethyl)benzene (0.4 mmol, 68.0 mg), $UO_2(NO_3)_2 \cdot 6H_2O$ (4 mol%/0.016 mmol, 8 mg), CCl_3COOH (0.8 mmol, 130.7 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 72 h under blue light (460nm, 9W) in a paralleled reactor. **2a** was obtained through column chromatography ($V_{PE}/V_{EA} = 10/1$) as a brown liquid (22.6 mg, 60%). **2p**[9] was obtained through column chromatography ($V_{PE}/V_{EA} = 10/1$) as a white solid (42.1 mg, 65%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.52 (d, *J* = 8.0 Hz, 1H), 7.43 (t, *J* = 7.6 Hz, 1H), 7.01 (t, *J* = 8.0 Hz, 1H), 6.96 (d, *J* = 8.0 Hz, 1H), 5.52 (brs, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 153.4 (d, *J* = 2.0 Hz), 133.5, 126.8 (q, *J* = 4.8 Hz), 124.1 (q, *J* = 271.0 Hz), 119.1 (d, *J* = 298.7 Hz), 116.4 (q, *J* = 30.3 Hz). **^{19}F NMR** (376 MHz, $CDCl_3$) δ -60.85. **IR** (neat) 3459, 1616, 1464, 1322, 1263, 1163, 1106 cm^{-1}. **MS** (EI) m/z 162.



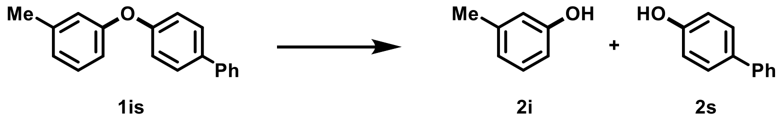
To a 25 mL Schlenk tube, 2-phenoxybenzonitrile (0.2 mmol, 39.0 mg), $UO_2(NO_3)_2 \cdot 6H_2O$ (4 mol%/0.008 mmol, 4 mg), CCl_3COOH (0.4 mmol, 65.4 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 72 h under blue light (460nm, 9W) in a paralleled reactor. **2a** was obtained through column chromatography ($V_{PE}/V_{EA} = 10/1$) as a brown liquid (15.0 mg, 80%). **2q**[5] was obtained through column chromatography ($V_{PE}/V_{EA} =$

5/1) as a brown solid (19.2 mg, 81%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.52 - 7.45 (m, 2H), 7.02 - 6.97 (m, 2H), 6.67 (brs, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.7, 134.7, 132.9, 120.8, 116.6, 116.3, 99.3. **IR** (neat) 3262, 2229, 1603, 1503, 1234, 1160, 1100 cm^{-1}. **MS** (EI) m/z 119.

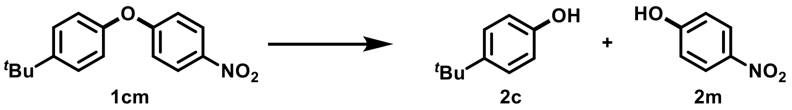


To a 25 mL Schlenk tube, methyl 2-phenoxybenzoate (0.4 mmol, 91.2mg), $UO_2(NO_3)_2 \cdot 6H_2O$ (4 mol%/0.016 mmol, 8 mg), CCl_3COOH (0.8 mmol, 130.7 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 72 h under blue light (430 nm, 12 W) in a paralleled reactor. **2a** was obtained through column chromatography (V_{PE}/V_{EA} = 10/1) as a brown liquid (30.5 mg, 81%). **2r**[10] was obtained through column chromatography (V_{PE}/V_{EA} = 5/1) as a yellow liquid (55.9 mg, 92%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 10.77 (brs, 1H), 7.84 (dd, J = 8.0, 1.7 Hz, 1H), 7.48 - 7.44 (m, 1H), 6.98 (d, J = 8.4 Hz, 1H), 6.90 - 6.86 (m, 1H), 3.95 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.5, 161.5, 135.6, 129.8, 119.1, 117.5, 112.3, 52.2. **IR** (neat) 1674, 1614, 1485, 1438, 1301, 1089, 848 cm^{-1}. **MS** (EI) m/z 152.

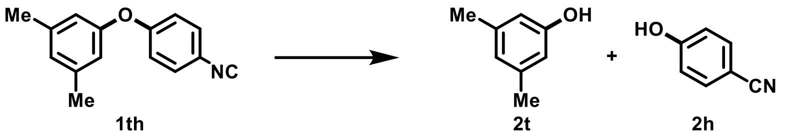
Flow reaction: To a 250 mL three-necked flask, a solution of methyl 2-phenoxybenzoate (10.0 mmol, 2.28 g), $UO_2(NO_3)_2 \cdot 6H_2O$ (4 mol%/0.4 mmol, 201 mg) and CCl_3COOH (20.0 mmol, 3.26 g) in freshly-distilled acetone (50 mL) was added. The flask was equipped with rubber plugs, with inlet and outlet of micro tube, which was made of PTFE tubing (O.D. = 2 mm, I.D. = 1 mm, length = 5.68 m, volume = 4.45 mL). Then the flask along with the micro cube were evacuated and filled with N_2 three times at -78 °C. The solution was pumped by a pump (0.5 mL/min) into the micro tube, then returned to flask. This circulatory system was irradiated by blue light (430 nm, 54 W totally) for 48 h (the temperature was below 30 °C). After the reaction, acetone (10 mL) was pumped into the tube to flush out residual fluid. The solvent was removed in vacuo. **2a** was obtained through column chromatography ($V_{PE}/V_{EA} = 10/1$) as a brown liquid (0.67 g, 71%). **2r** was obtained through column chromatography ($V_{PE}/V_{EA} = 5/1$) as a yellow liquid (1.09 g, 72%).



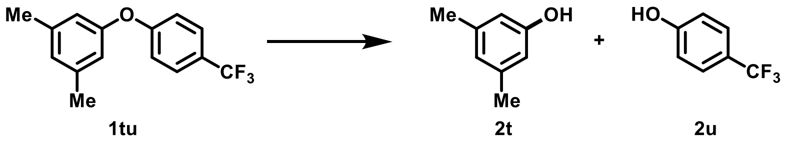
To a 25 mL Schlenk tube, 4-(m-tolyloxy)-1,1'-biphenyl (0.2 mmol, 52.0 mg), $UO_2(NO_3)_2{\cdot}6H_2O$ (4 mol%/0.008 mmol, 4 mg), CCl_3COOH (0.4 mmol, 65.3 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 48 h under blue light (460 nm, 9 W) in a paralleled reactor. **2i** was obtained through column chromatography ($V_{PE}/V_{EA} = 10/1$) as a pink liquid (16.4 mg, 76%). **2s**^{2} was obtained through column chromatography ($V_{PE}/V_{EA} = 5/1$) as a yellow solid (27.8 mg, 82%). ^{1}**H NMR** (400 MHz, $CDCl_3$) δ7.58 – 7.52 (m, 2H), 7.49 (d, J = 8.7 Hz, 2H), 7.42 (t, J = 7.6 Hz, 2H), 7.31 (t, J = 7.3 Hz, 1H), 6.91 (d, J = 8.7 Hz, 2H), 4.83 (s, 1H). ^{13}**C NMR** (100 MHz, $CDCl_3$) δ 155.0, 140.7, 134.0, 128.7, 128.3, 126.7, 115.6. **IR** (neat) 1739, 1711, 1597, 1488, 1373, 1239, 832 cm^{-1}. **MS** (EI) m/z 170.



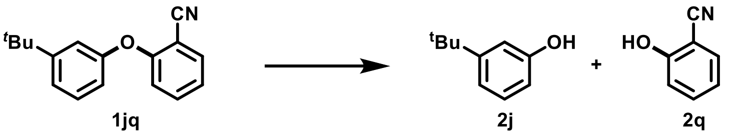
To a 25 mL Schlenk tube, 1-(tert-butyl)-4-(4-nitrophenoxy)benzene (0.4 mmol, 108.4 mg), $UO_2(NO_3)_2{\cdot}6H_2O$ (4 mol%/0.016 mmol, 8 mg), CCl_3COOH (0.8 mmol, 130.7 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 72 h under blue light (430 nm, 12 W) in a paralleled reactor. **2c** was obtained through column chromatography ($V_{PE}/V_{EA} = 10/1$) as a white solid (39.0 mg, 65%). **2m** was obtained through column chromatography ($V_{PE}/V_{EA} = 5/1$) as a yellow solid (28.3 mg, 51%).



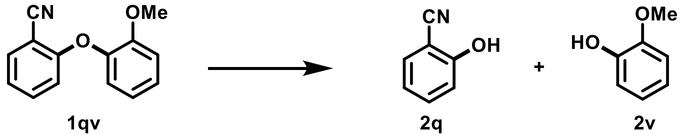
To a 25 mL Schlenk tube, 4-(3,5-dimethylphenoxy)benzonitrile (0.4 mmol, 89.2 mg), $UO_2(NO_3)_2{\cdot}6H_2O$ (4 mol%/0.016 mmol, 8 mg), CCl_3COOH (0.8 mmol, 130.7 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 72 h under blue light (430 nm, 12 W) in a paralleled reactor. **2h** was obtained through column chromatography ($V_{PE}/V_{EA} = 5/1$) as a white solid (43.3 mg, 91%). **2t**[5] was obtained through column chromatography ($V_{PE}/V_{EA} = 10/1$) as a brown solid (23.9 mg, 49%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.58 (s, 1H), 6.46 (s, 2H), 4.63 (brs, 1H), 2.27 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 155.3, 139.5, 122.5, 112.9, 21.2. **IR** (neat) 3269, 2920, 1632, 1598, 1312, 1158, 835, cm^{-1}. **MS** (EI) m/z 122.



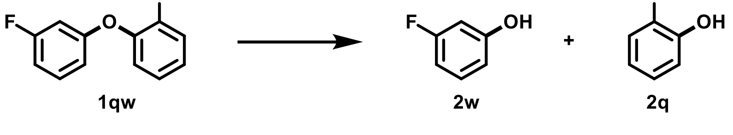
To a 25 mL Schlenk tube, 1,3-dimethyl-5-(4-(trifluoromethyl)phenoxy)benzene (0.4 mmol, 106.0 mg), $UO_2(NO_3)_2{\cdot}6H_2O$ (4 mol%/0.016 mmol, 8 mg), CCl_3COOH (0.8 mmol, 130.7 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 72 h under blue light (430 nm, 12 W) in a paralleled reactor. **2t** was obtained through column chromatography ($V_{PE}/V_{EA} = 20/1$) as a brown solid (19.0 mg, 39%). **2u**[13] was obtained through column chromatography ($V_{PE}/V_{EA} = 10/1$) as a brown solid (46.7 mg, 72%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.51 (d, *J* = 12.0 Hz, 2H), 6.91 (d, *J* = 8.0 Hz, 2H), 5.66 (brs, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.0, 127.2 (q, *J* = 3.7 Hz), 124.3(q, *J* = 269.0 Hz), 123.3 (q, *J* = 32.8 Hz), 115.4. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -61.54. **IR** (neat) 3357, 1617, 1604, 1522, 1319, 1247, 1158 cm^{-1}. **MS** (EI) m/z 162.



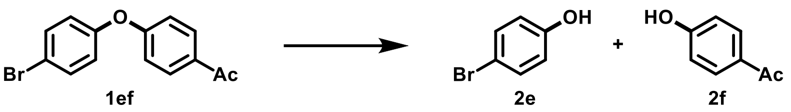
To a 25 mL Schlenk tube, 2-(3-(tert-butyl)phenoxy)benzonitrile (0.4 mmol, 100.4 mg), $UO_2(NO_3)_2{\cdot}6H_2O$ (4 mol%/0.016 mmol, 8 mg), CCl_3COOH (0.8 mmol, 130.7 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 72 h under blue light (460nm, 9W) in a paralleled reactor. **2j** was obtained through column chromatography ($V_{PE}/V_{EA} = 10/1$) as a white solid (44.4 mg, 74%). **2q** was obtained through column chromatography ($V_{PE}/V_{EA} = 5/1$) as a brown solid (43.4 mg, 92%).



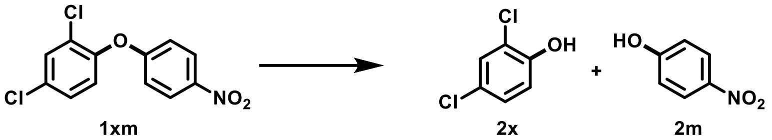
To a 25 mL Schlenk tube, 2-(2-methoxyphenoxy)benzonitrile (0.4 mmol, 90.0 mg), $UO_2(NO_3)_2{\cdot}6H_2O$ (4 mol%/0.016 mmol, 8 mg), CCl_3COOH (0.8 mmol, 130.7 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 72 h under blue light (460nm, 9W) in a paralleled reactor. **2q** was obtained through column chromatography ($V_{PE}/V_{EA} = 10/1$) as a brown solid (30.2 mg, 64%). **2v**[8] was obtained through column chromatography ($V_{PE}/V_{EA} = 50/1$) as a brown liquid (19.2 mg, 39%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.95 - 6.91 (m, 1H), 6.90 – 6.83 (m, 3H), 5.65 (brs, 1H), 3.89 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 146.5, 145.5, 121.3, 120.0, 114.4, 110.6, 55.7. **IR** (neat) 3510, 3449, 2932, 2842, 1596, 1499, 1443 cm^{-1}. **MS** (EI) m/z 124.



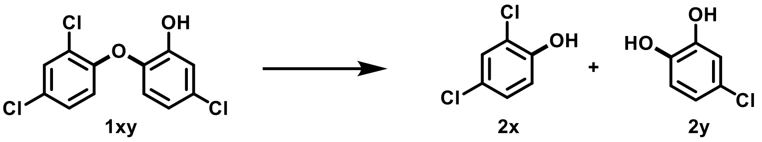
To a 25 mL Schlenk tube, 2-(3-fluorophenoxy)benzonitrile (0.2 mmol, 42.6 mg), $UO_2(NO_3)_2{\cdot}6H_2O$ (4 mol%/0.008 mmol, 4 mg), CCl_3COOH (0.4 mmol, 65.4 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 72 h under blue light (460nm, 9W) in a paralleled reactor. **2q** was obtained through column chromatography (V_{PE}/V_{EA} = 10/1) as a brown solid (27.4 mg, 88%). **2w**[8] was obtained through column chromatography (V_{PE}/V_{EA} = 40/1) as a brown liquid (15.0 mg, 67 %).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.22 - 7.16 (m, 1H), 6.65 - 6.59 (m, 3H), 5.95 (brs, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 164.7, 162.3, 156.5 (d, J = 11.3 Hz), 130.5 (d, J = 10.1 Hz), 111.1 (d, J = 2.9 Hz), 107.8 (d, J = 21.3 Hz), 103.2 (d, J = 24.6 Hz). **^{19}F NMR** (376 MHz, $CDCl_3$) δ -111.57. **IR** (neat) 3374, 1609, 1490, 1460, 1282, 1160, 1129 cm^{-1}. **MS** (EI) m/z 112.



To a 25 mL Schlenk tube, 1-(4-(4-bromophenoxy)phenyl)ethan-1-one (0.4 mmol, 116.0 mg), $UO_2(NO_3)_2 \cdot 6H_2O$ (4 mol%/0.016 mmol, 8 mg), CCl_3COOH (0.8 mmol, 130.7 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 72 h under blue light (430 nm, 12W) in a paralleled reactor. **2e** was obtained through column chromatography (V_{PE}/V_{EA} = 10/1) as a brown liquid(49.2 mg, 72%). **2f** was obtained through column chromatography (V_{PE}/V_{EA} = 5/1) as a brown solid (44.6 mg, 82%).

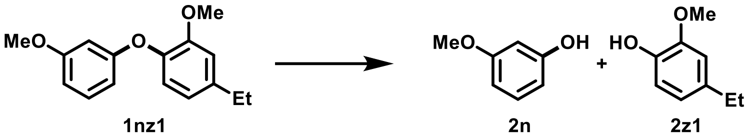


To a 25 mL Schlenk tube, nitrofen (0.4 mmol, 113.6 mg), $UO_2(NO_3)_2 \cdot 6H_2O$ (4 mol%/0.016 mmol, 8 mg), CCl_3COOH (0.8 mmol, 130.7 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 72 h under blue light (430 nm, 12W) in a paralleled reactor. **2m** was obtained through column chromatography (V_{PE}/V_{EA} = 10/1) as a yellow solid (35.0 mg, 54%). **2x**[14] was obtained through column chromatography (V_{PE}/V_{EA} = 30/1) as a white solid (28.4 mg, 51%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.33 (d, *J* = 4.0 Hz, 1H), 7.17 - 7.14 (m, 1H), 6.95 (d, *J* = 8.0 Hz, 1H), 5.53 (brs, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 150.1, 128.5, 128.5, 125.5, 120.3, 117.1. **IR** (neat) 3426, 1477, 1406, 1328, 1276, 1184, 1094 cm^{-1}. **MS** (EI) m/z 162.



To a 25 mL Schlenk tube, triclosan (0.4 mmol, 115.8 mg), $UO_2(NO_3)_2{\cdot}6H_2O$ (4 mol%/0.016 mmol, 8 mg), CCl_3COOH (0.8 mmol, 130.7 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 72 h under blue light (430 nm, 12W) in a paralleled reactor. **2x** was obtained through column chromatography (V_{PE}/V_{EA} = 20/1) as a white solid (38.2 mg, 59%). **2y**[15] was obtained through column chromatography (V_{PE}/V_{EA} = 5/1) as a brown solid (32.3 mg, 56%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.89 - 6.84 (m, 2H), 6.78 (t, *J* = 8.0 Hz, 1H), 5.56 (brs, 1H), 5.54 (brs, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 144.7, 139.2, 121.2, 120.4, 119.7, 114.2. **IR** (neat) 3409, 2925, 1596, 1490, 1464, 1326, 1158 cm^{-1}. **MS** (EI) m/z 144.

Flow reaction: To a 250 mL three-necked flask, a solution of triclosan (10.0 mmol, 2.88 g), $UO_2(NO_3)_2{\cdot}6H_2O$ (4 mol%/0.4 mmol, 201 mg) and CCl_3COOH (20.0 mmol, 3.26 g) in freshly-distilled acetone (50 mL) was added. The flask was equipped with rubber plugs, with inlet and outlet of micro tube, which was made of PTFE tubing (O.D. = 2 mm, I.D. = 1 mm, length = 5.68 m, volume = 4.45 mL). Then the flask along with the micro cube were evacuated and filled with N_2 three times at -78 °C. The solution was pumped by a pump (0.5 mL/min) into the micro tube, then returned to flask. This circulatory system was irradiated by blue light (430 nm, 54 W) for 48 h (the temperature was below 30 °C). After the reaction, acetone (10 mL) was pumped into the tube to flush out residual fluid. The solvent was removed in vacuo. **2x** was obtained through column chromatography (V_{PE}/V_{EA} = 20/1) as a white solid (0.91g, 56%). **2y** was obtained through column chromatography (V_{PE}/V_{EA} = 5/1) as a brown solid (0.69 mg, 48%).

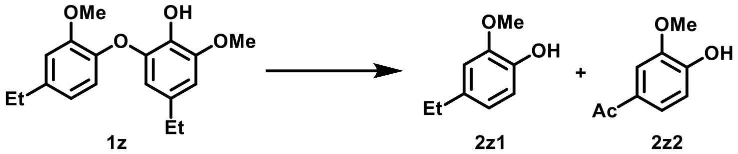


1nz1 **2n** **2z1**

To a 25 mL Schlenk tube, **1nz1** (0.4 mmol, 103.2 mg), $UO_2(NO_3)_2 \cdot 6H_2O$ (4 mol%/0.016 mmol, 8 mg), CCl_3COOH (0.8 mmol, 130.7 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 72 h under blue light (430 nm, 12 W) in a paralleled reactor. **2n** was obtained through column chromatography (V_{PE}/V_{EA} = 20/1) (23.8 mg, 48%) as a brown liquid. **2z1**[16] was obtained through column chromatography (V_{PE}/V_{EA} = 5/1) (32.8 mg, 54%) as a brown liquid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.85 (d, *J* = 8.4 Hz, 1H), 6.70 (d, *J* = 7.2 Hz, 2H), 5.48 (brs, 1H), 3.89 (s, 3H), 2.59 (q, *J* = 7.6 Hz, 2H), 1.23 (t, *J* = 7.6 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 146.2, 143.4, 136.2, 120.2, 114.1, 110.4, 55.8, 28.5, 15.9. **IR** (neat) 1598, 1287, 1198, 1149, 1041, 945, 686 cm^{-1}. **MS** (EI) m/z 152.

Flow reaction: To a 250 mL three-necked flask, a solution of **1nz1** (10.0 mmol, 2.58 g), $UO_2(NO_3)_2 \cdot 6H_2O$ (4 mol%/0.4 mmol, 201 mg) and CCl_3COOH (20.0 mmol, 3.26 g) in freshly-distilled acetone (50 mL) was added. The flask was equipped with rubber plugs, with inlet and outlet of micro tube, which was made of PTFE tubing (O.D. = 2 mm, I.D. = 1 mm, length = 5.68 m, volume = 4.45 mL). Then the flask along with the micro cube were evacuated and filled with N_2 three times at -78 °C. The solution was pumped by a pump (0.5 mL/min) into the micro tube, then returned to flask. This circulatory system was irradiated by blue LEDs (430 nm, 54 W totally) for 48 h (the temperature was below 30 °C). After the reaction, acetone (10 mL) was pumped into the tube to flush out residual fluid. The solvent was removed in vacuo. **2n** was obtained through column chromatography (V_{PE}/V_{EA} = 20/1) as a brown liquid (0.47 g, 65%). **2z1** was obtained through column chromatography (V_{PE}/V_{EA} = 5/1) as a brown liquid (0.71

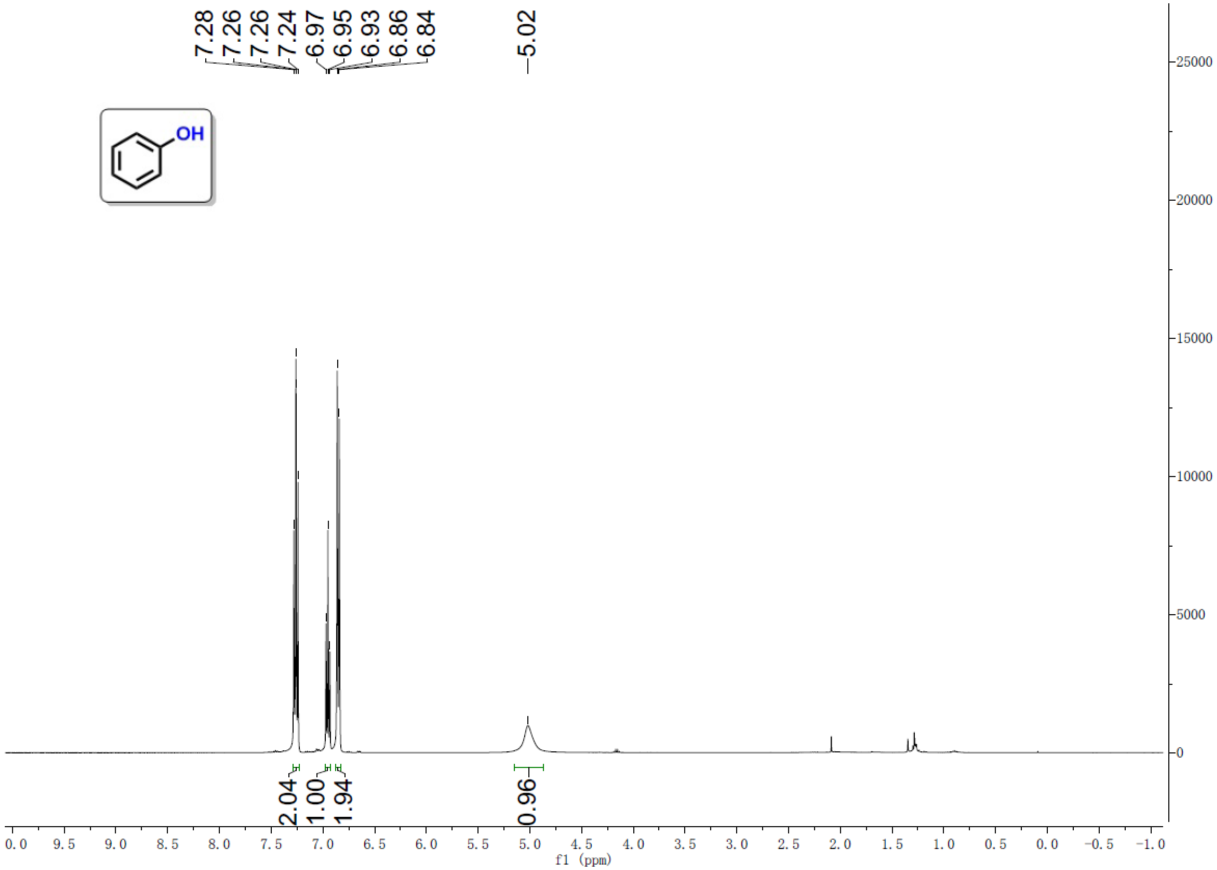
g, 81%) based on starting material recovery.



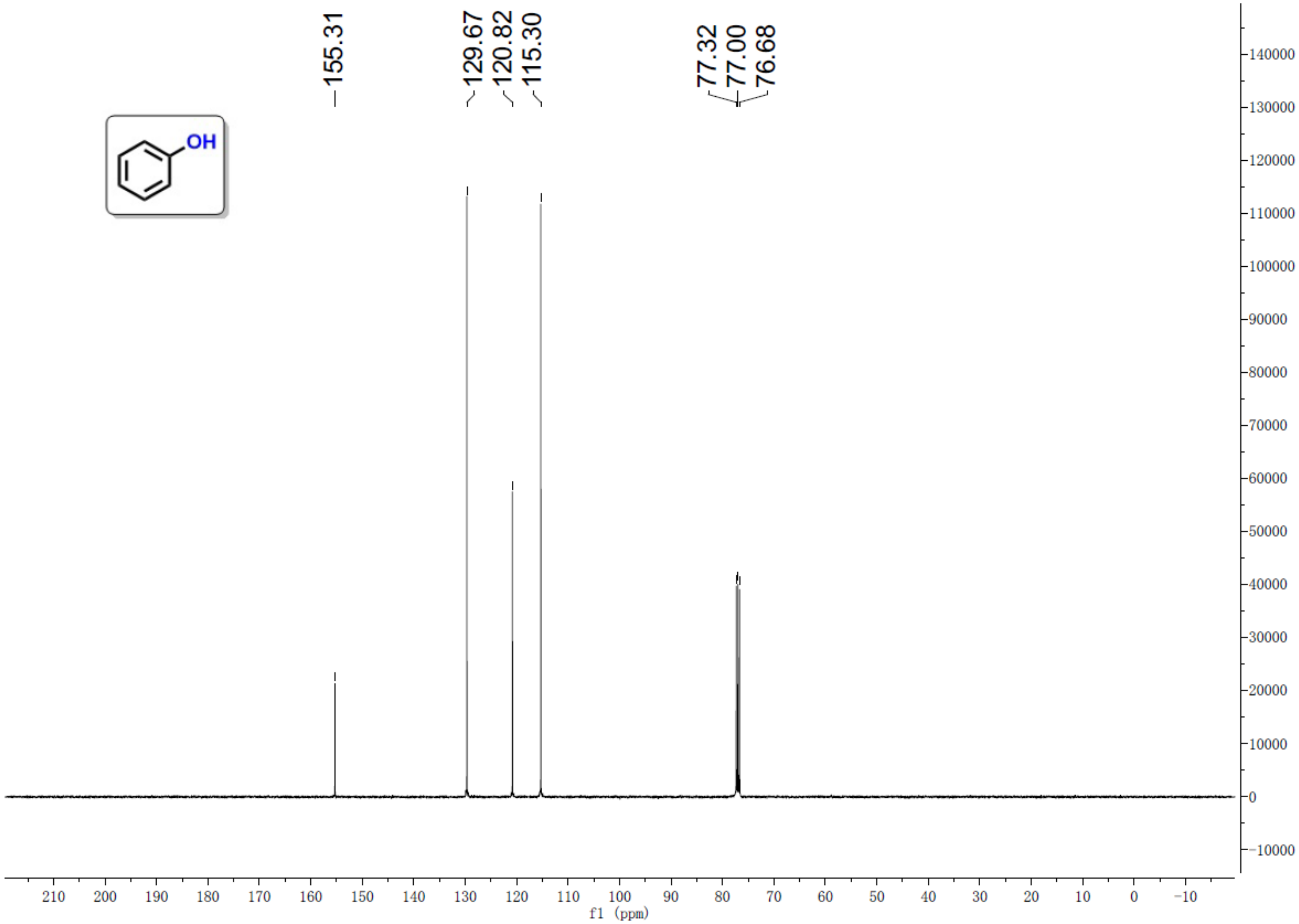
To a 25 mL Schlenk tube, **1z** (0.4 mmol, 120.8 mg), $UO_2(NO_3)_2 \cdot 6H_2O$ (4 mol%/0.016 mmol, 8 mg), CCl_3COOH (0.8 mmol, 130.7 mg) and freshly distilled acetone (2 mL) were added. The tube was evacuated and filled with N_2 three times at -78 °C then stirred for 72 h under blue light (430 nm, 12 W) in a paralleled reactor. **2z1** was obtained through column chromatography ($V_{PE}/V_{EA} = 5/1$) as a brown liquid (8.5 mg, 14%). **2z2**[17] was obtained through column chromatography ($V_{PE}/V_{EA} = 2/1$) as a brown liquid (14.6 mg, 22%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.55-7.52 (m, 2H), 6.95 (d, J= 8.0 Hz, 1H), 6.13 (brs, 1H), 3.95 (s, 3H), 2.55 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 196.9, 150.4, 146.6, 130.0, 123.9, 113.8, 109.7, 55.9, 26.0. **IR** (neat) 3324, 1659, 1577, 1518, 1292, 1222, 851 cm^{-1}. **MS** (EI) m/z 166.

V. NMR Spectra

^{1}H NMR of 2a



^{13}C NMR of 2a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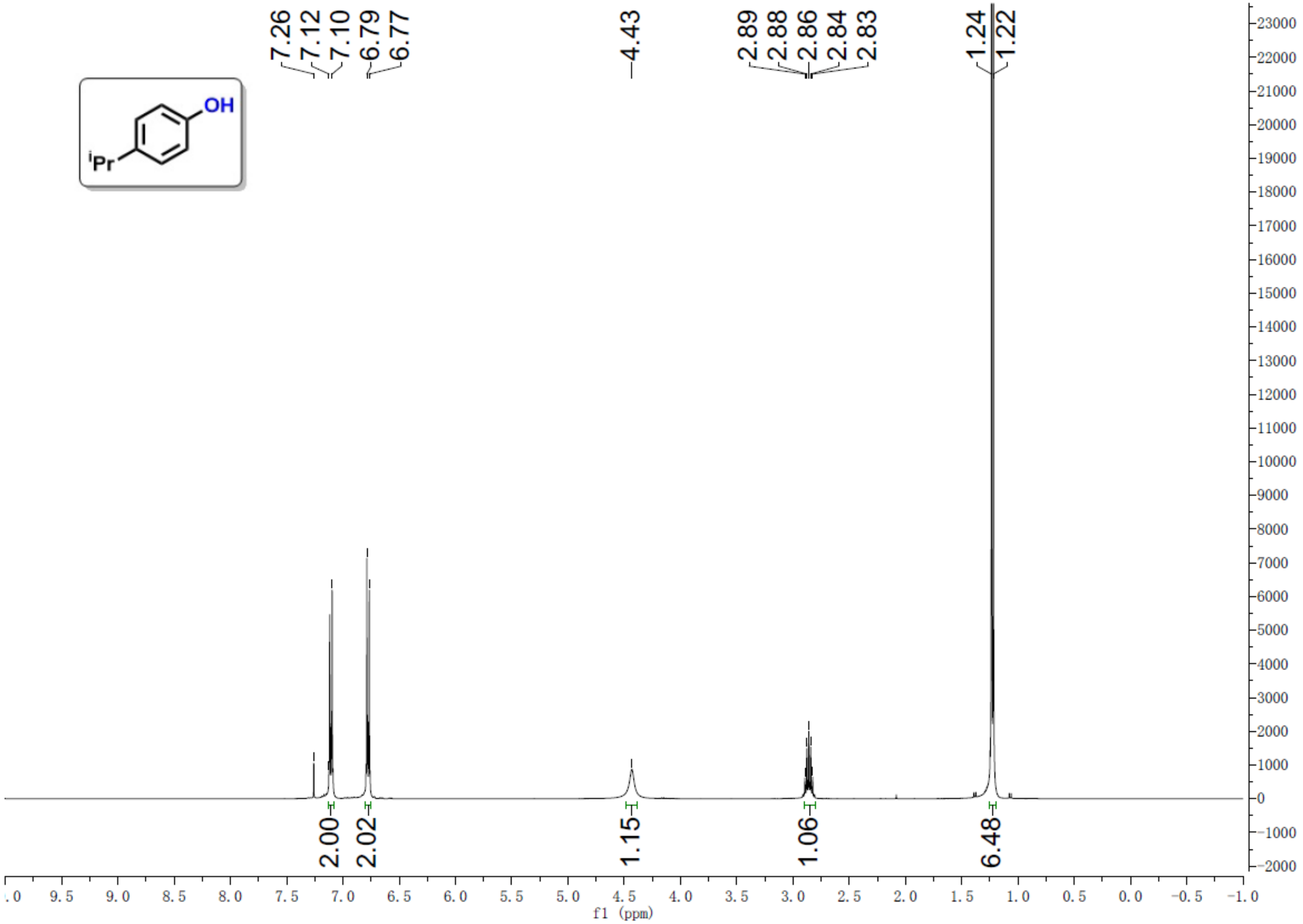
115.30
77.32
77.00
76.68
OH
140000
130000
120000
110000
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

^{1}H NMR of 2b



OH
^{i}Pr
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7.12
7.10
6.79
6.77
4.43
2.89
2.88
2.86
2.84
2.83
1.24
1.22
2.00
2.02
1.15
1.06
6.48
.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
−1.0
f1 (ppm)
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22000
21000
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
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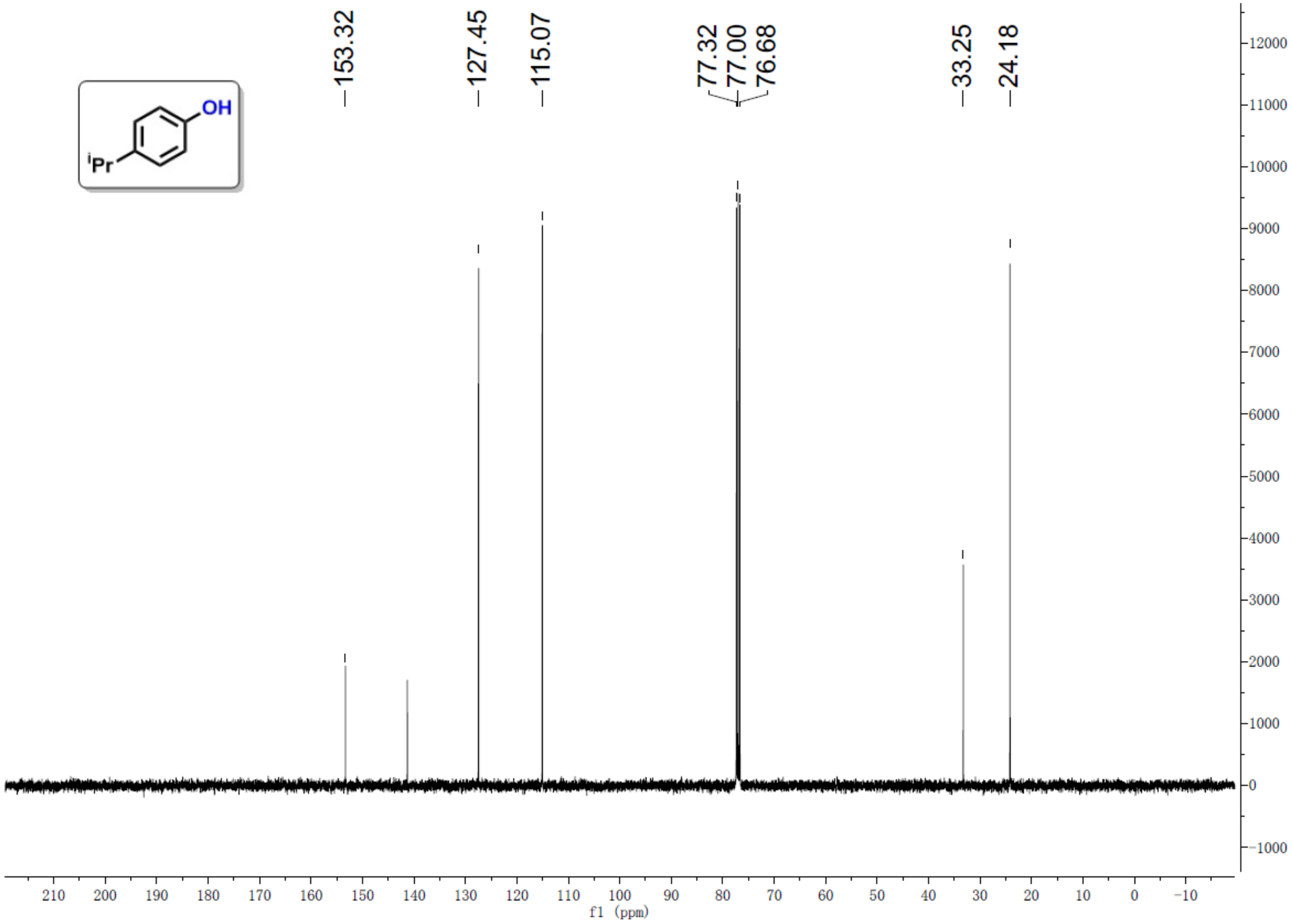
8000
7000
6000
5000
4000
3000
2000
1000
0
−1000
−2000

^{13}C NMR of 2b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115.07
77.32
77.00
76.68
33.25
24.18
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11000
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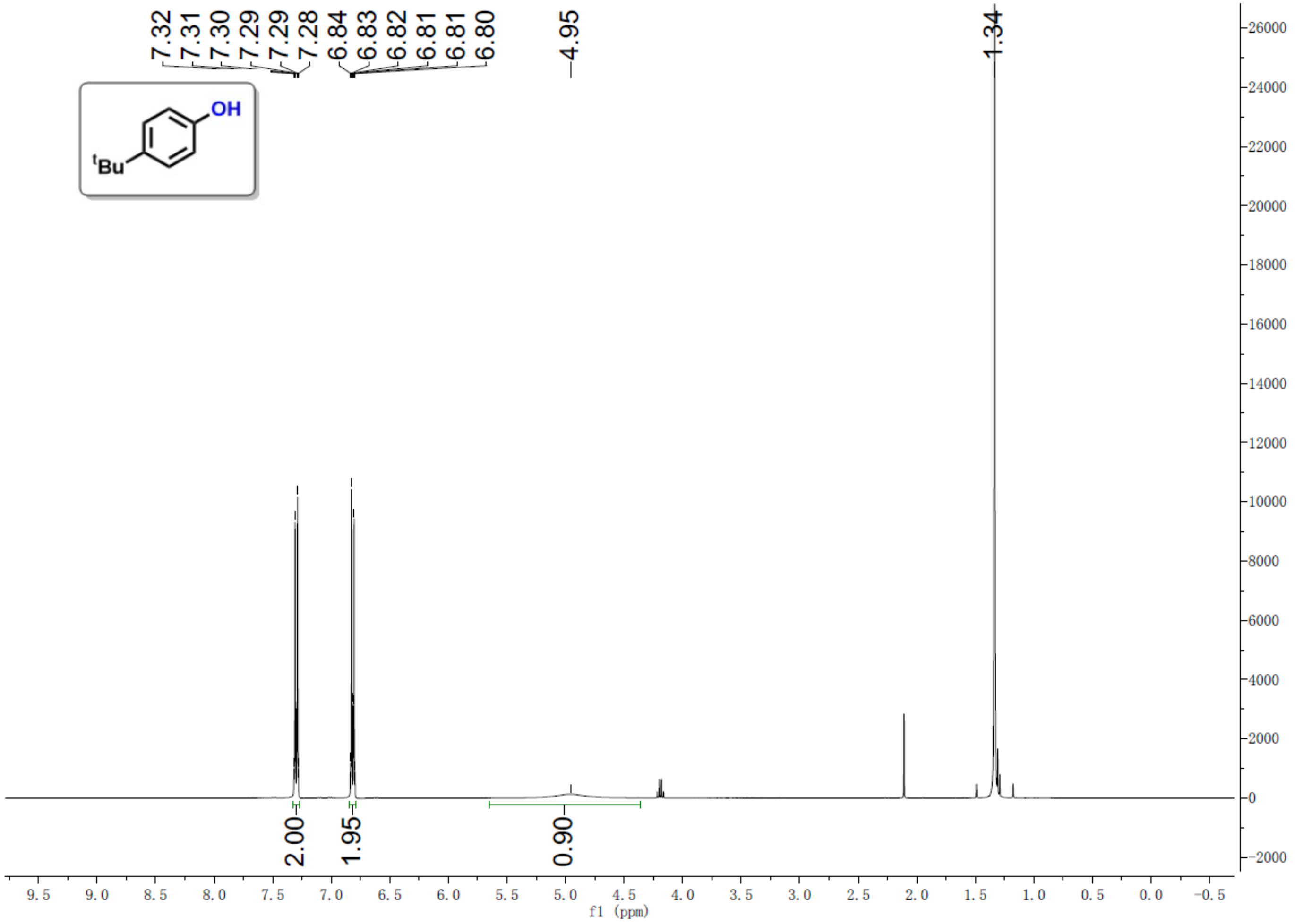
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

^{1}H NMR of 2c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7.28
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6.82
6.81
6.81
6.80
4.95
1.34
OH
tBu
2.00
1.95
0.90
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)
26000
24000
22000
20000
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12000
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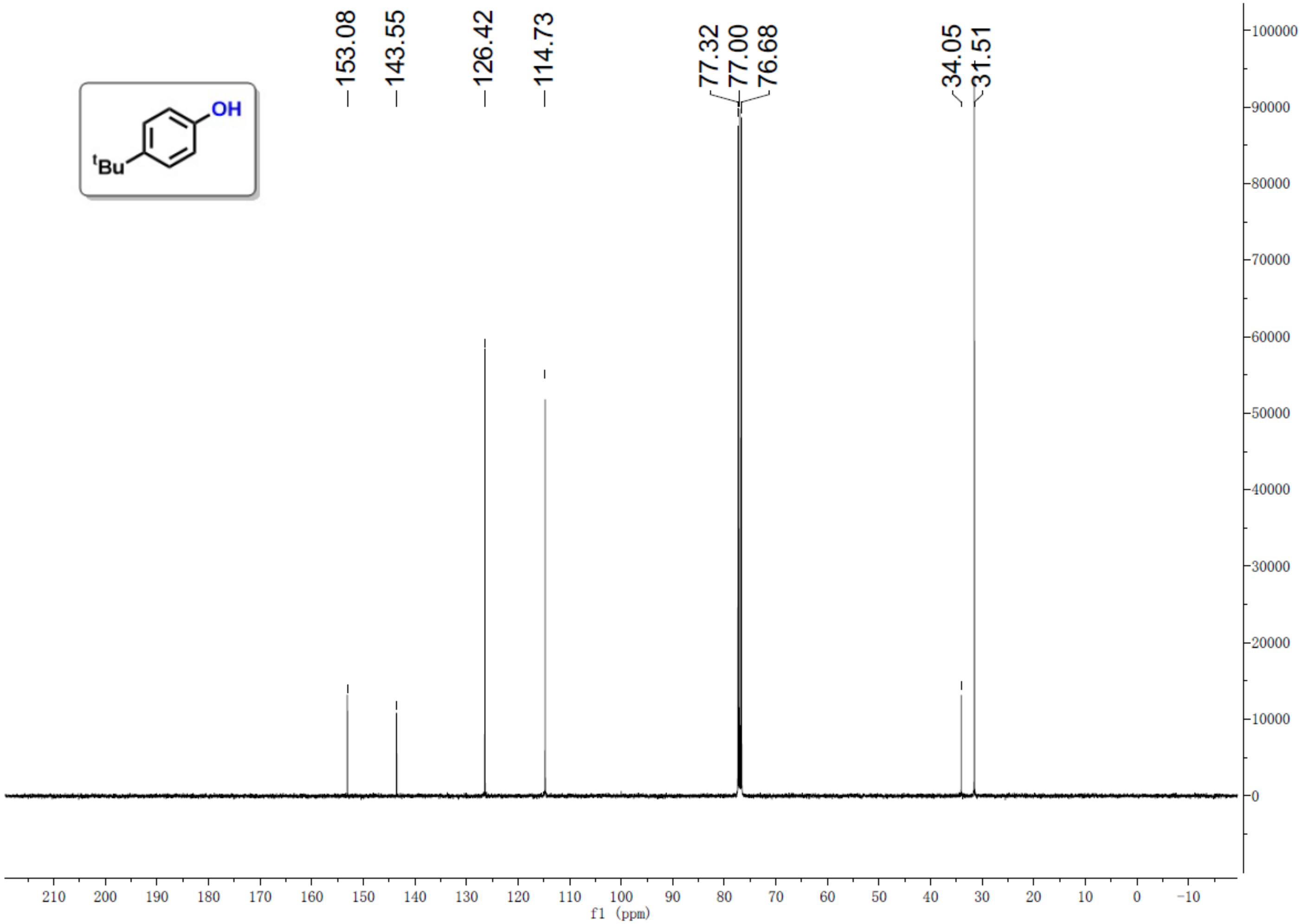
8000
6000
4000
2000
0
-2000

^{13}C NMR of 2c



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31.51
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70000
60000
50000
40000
30000
20000
10000
0
210
200
190
180
170
160
150
140
130
120
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80
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50
40
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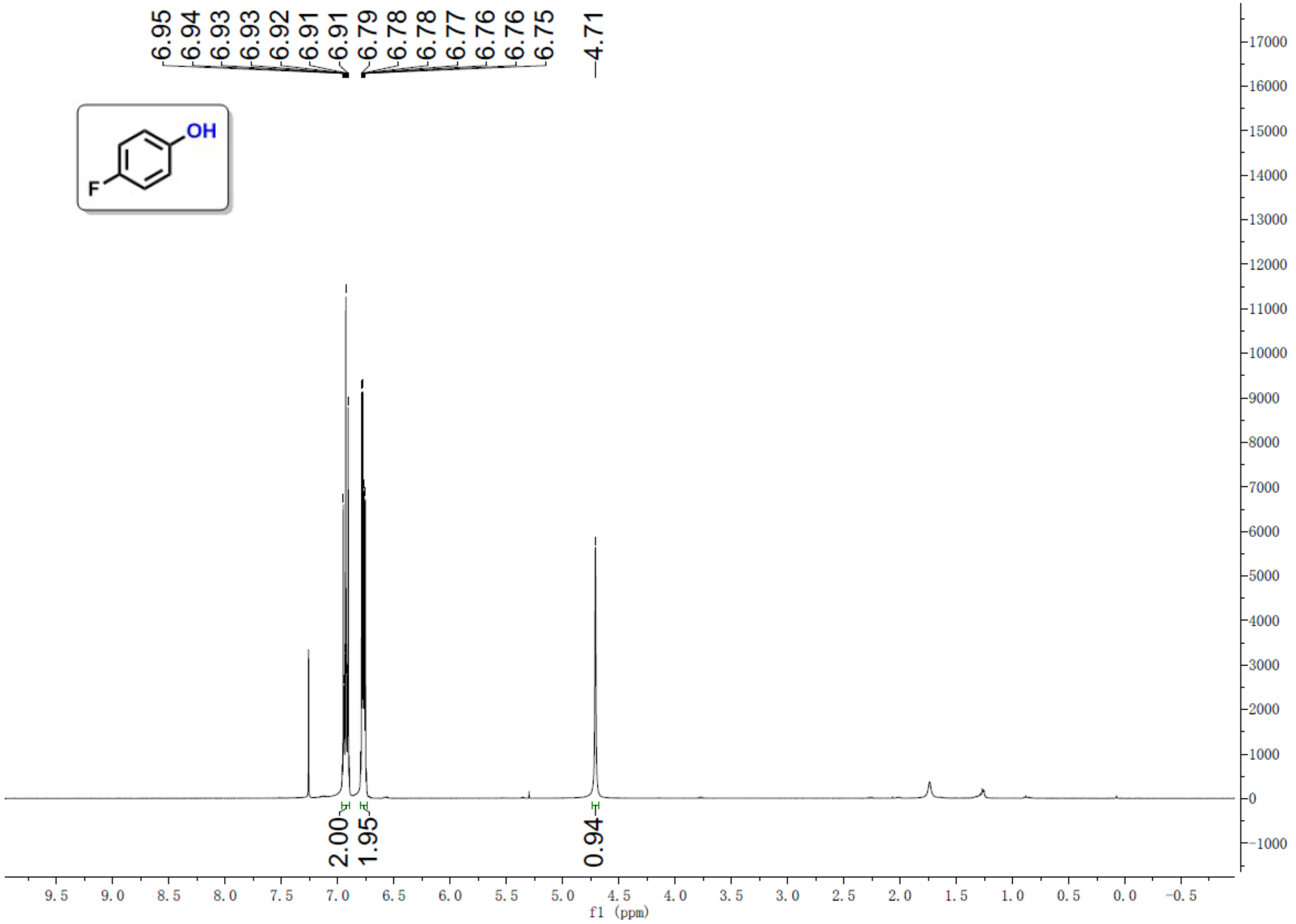
f1 (ppm)

^{1}H NMR of 2d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6.76
6.75
4.71
OH
F
2.00
1.95
0.94
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)
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16000
15000
14000
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12000
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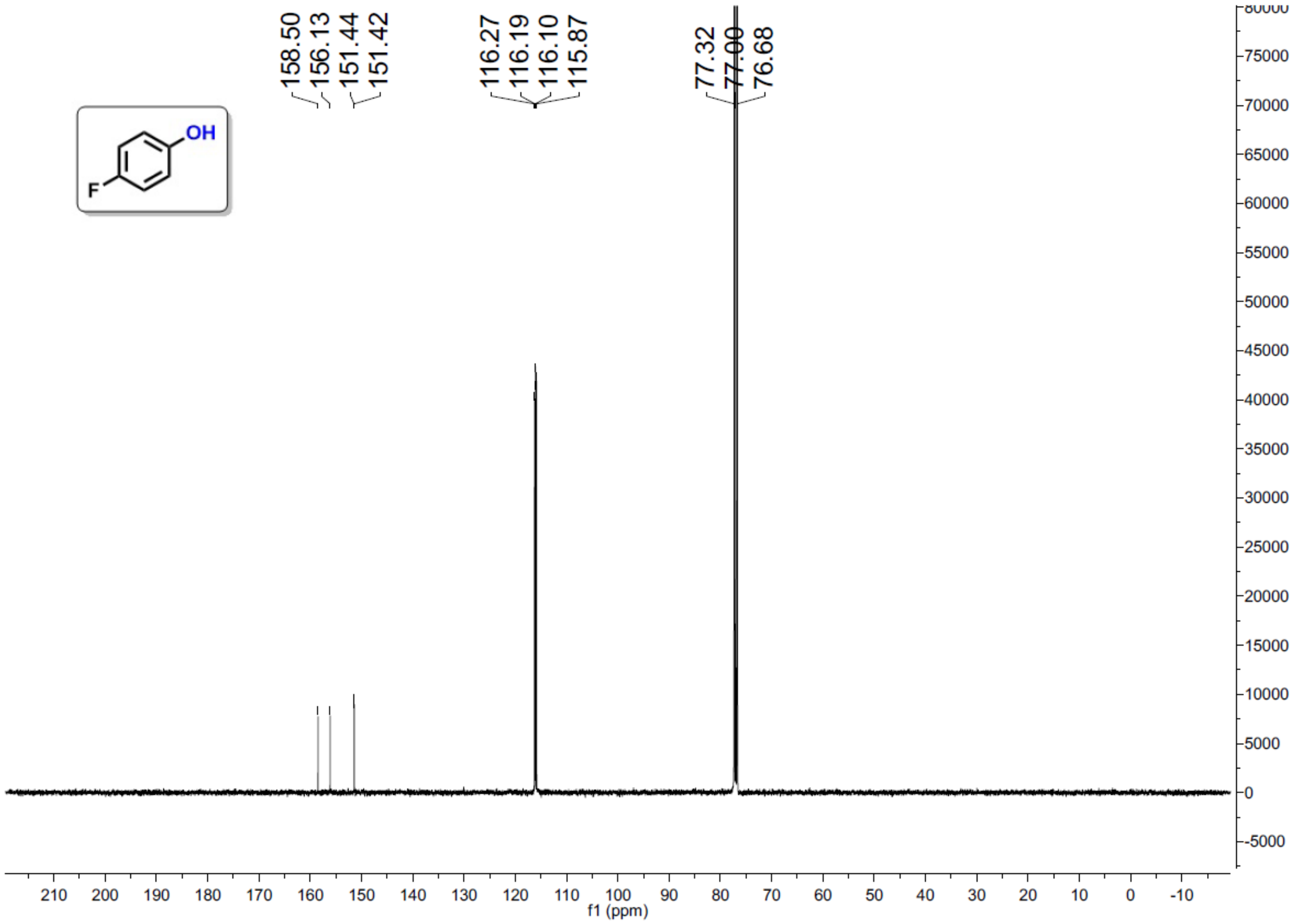
7000
6000
5000
4000
3000
2000
1000
0
-1000

^{13}C NMR of 2d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75000
70000
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
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0
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210
200
190
180
170
160
150
140
130
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80
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40
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20
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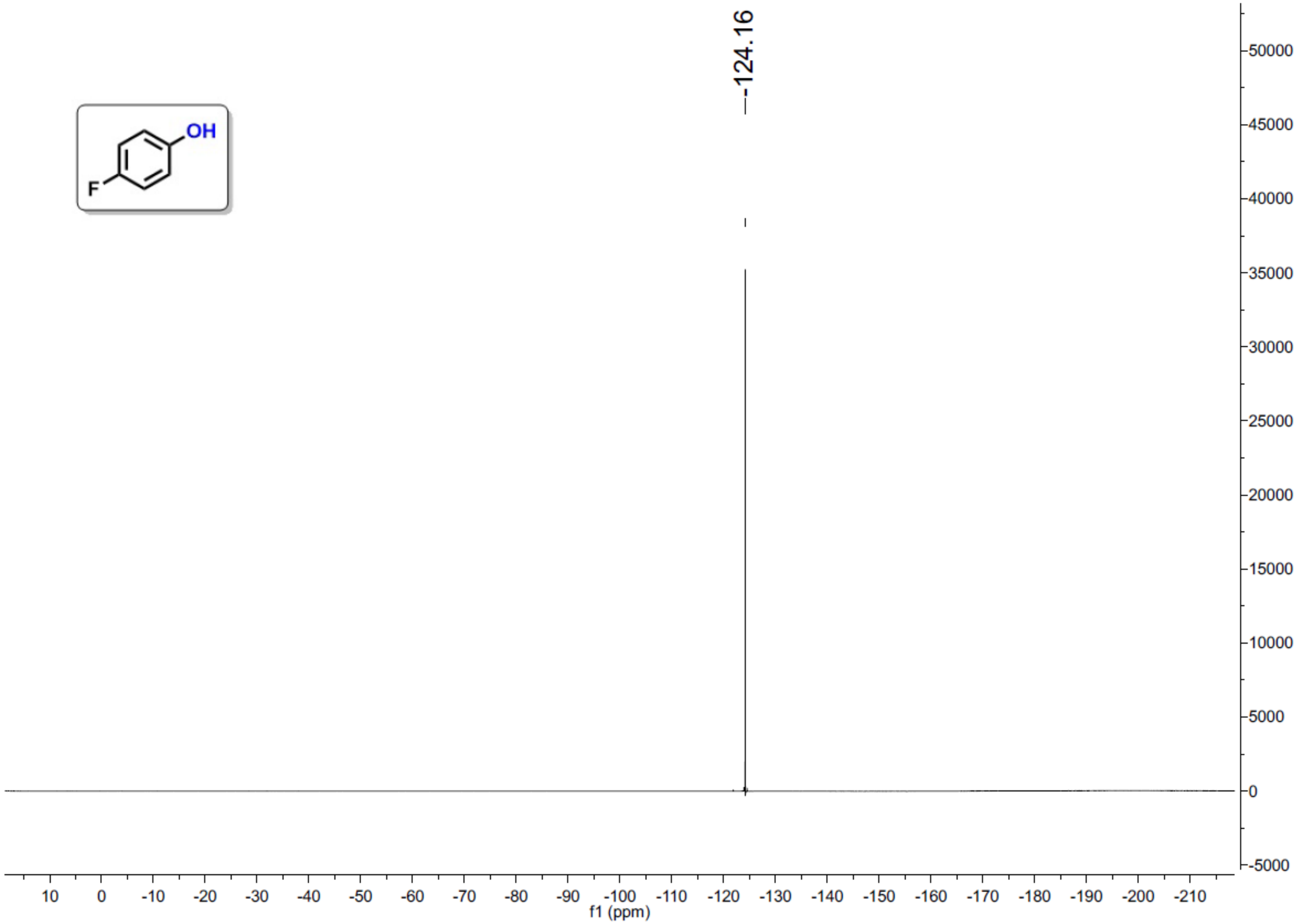
f1 (ppm)

^{19}F NMR of 2d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-100
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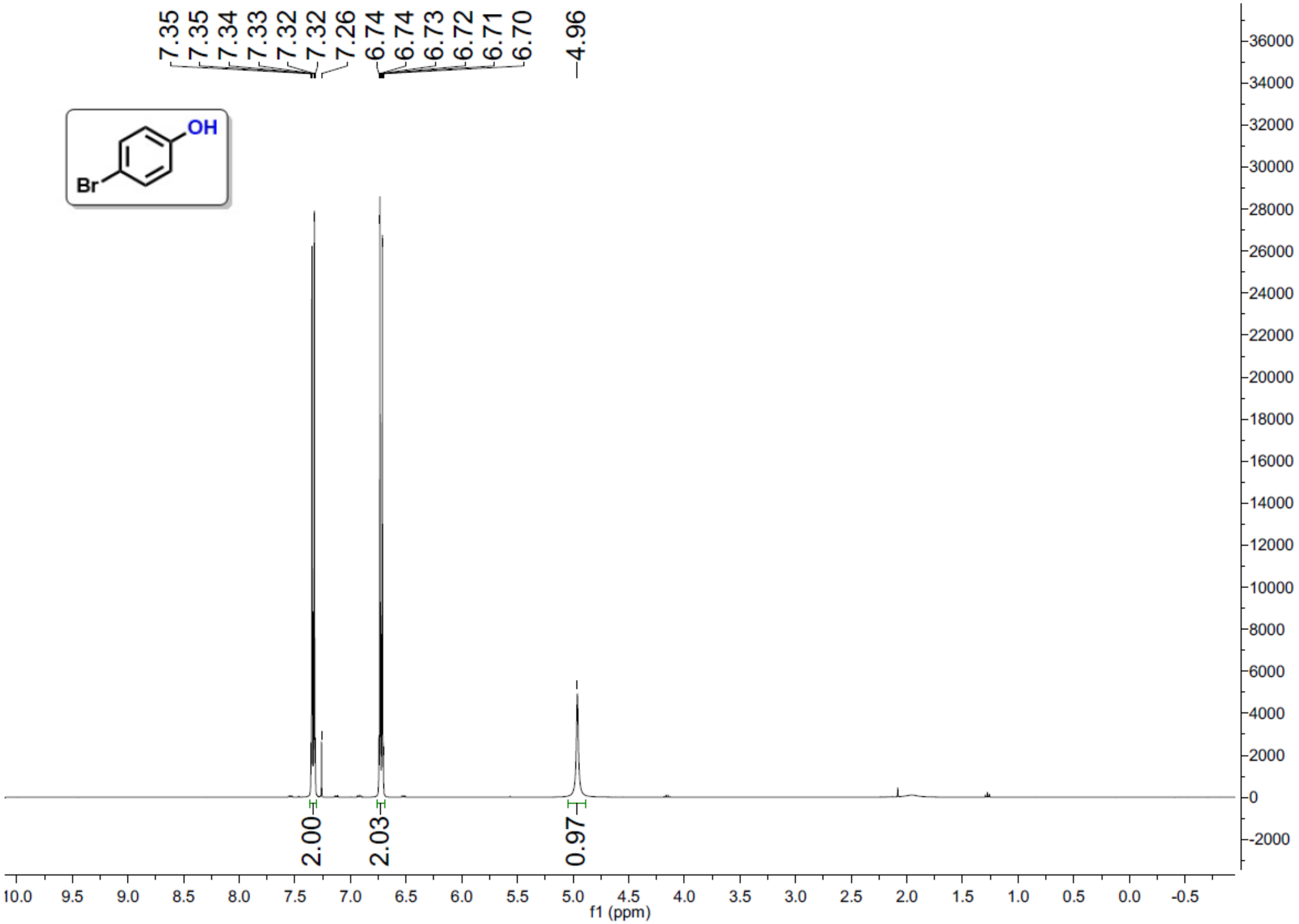
f1 (ppm)

^{1}H NMR of 2e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7.33
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7.32
7.26
6.74
6.74
6.73
6.72
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6.70
4.96
OH
Br
2.00
2.03
0.97
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34000
32000
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
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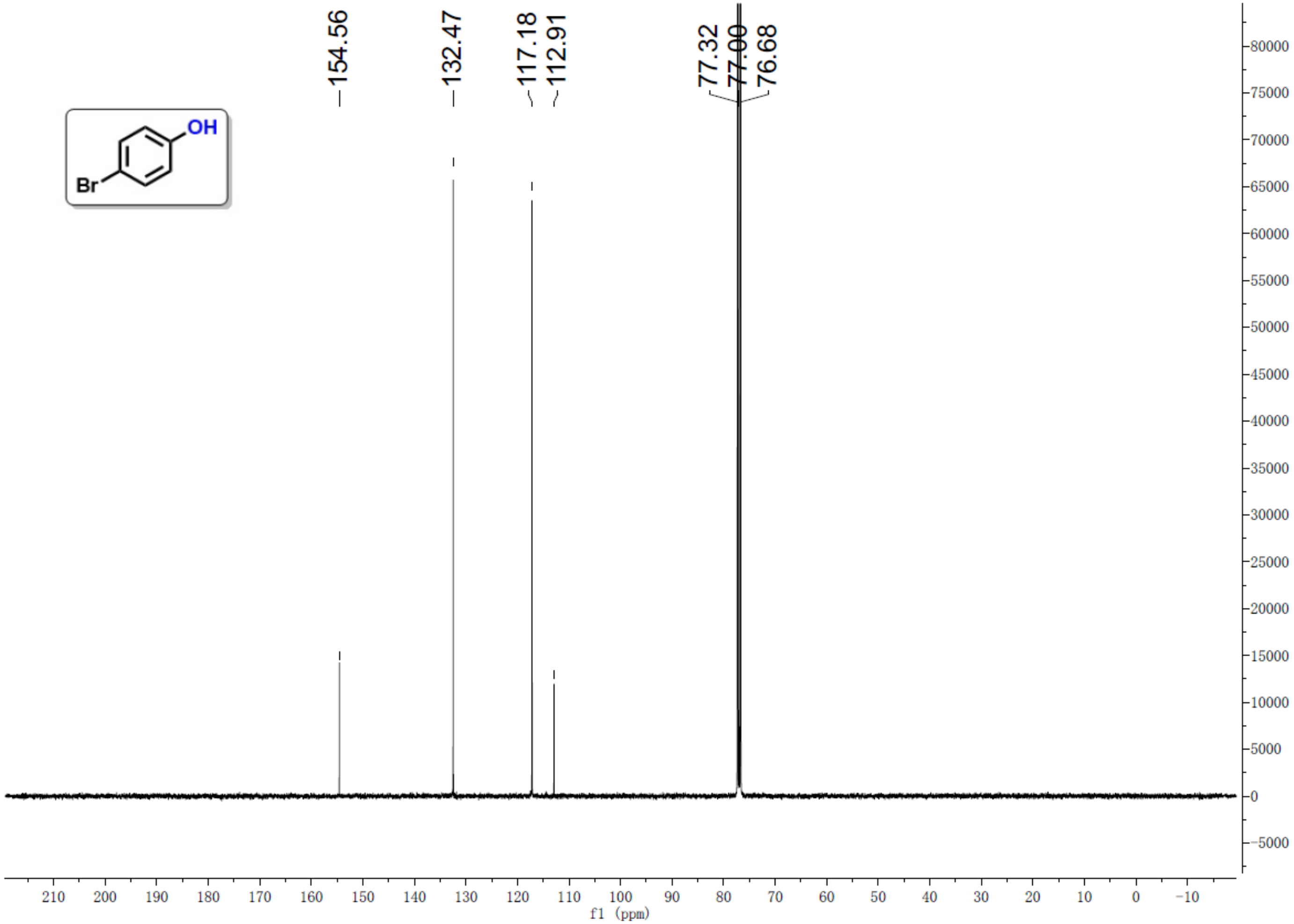
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

^{13}C NMR of 2e



154.56
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76.68
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Br
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70000
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
−5000
210
200
190
180
170
160
150
140
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80
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50
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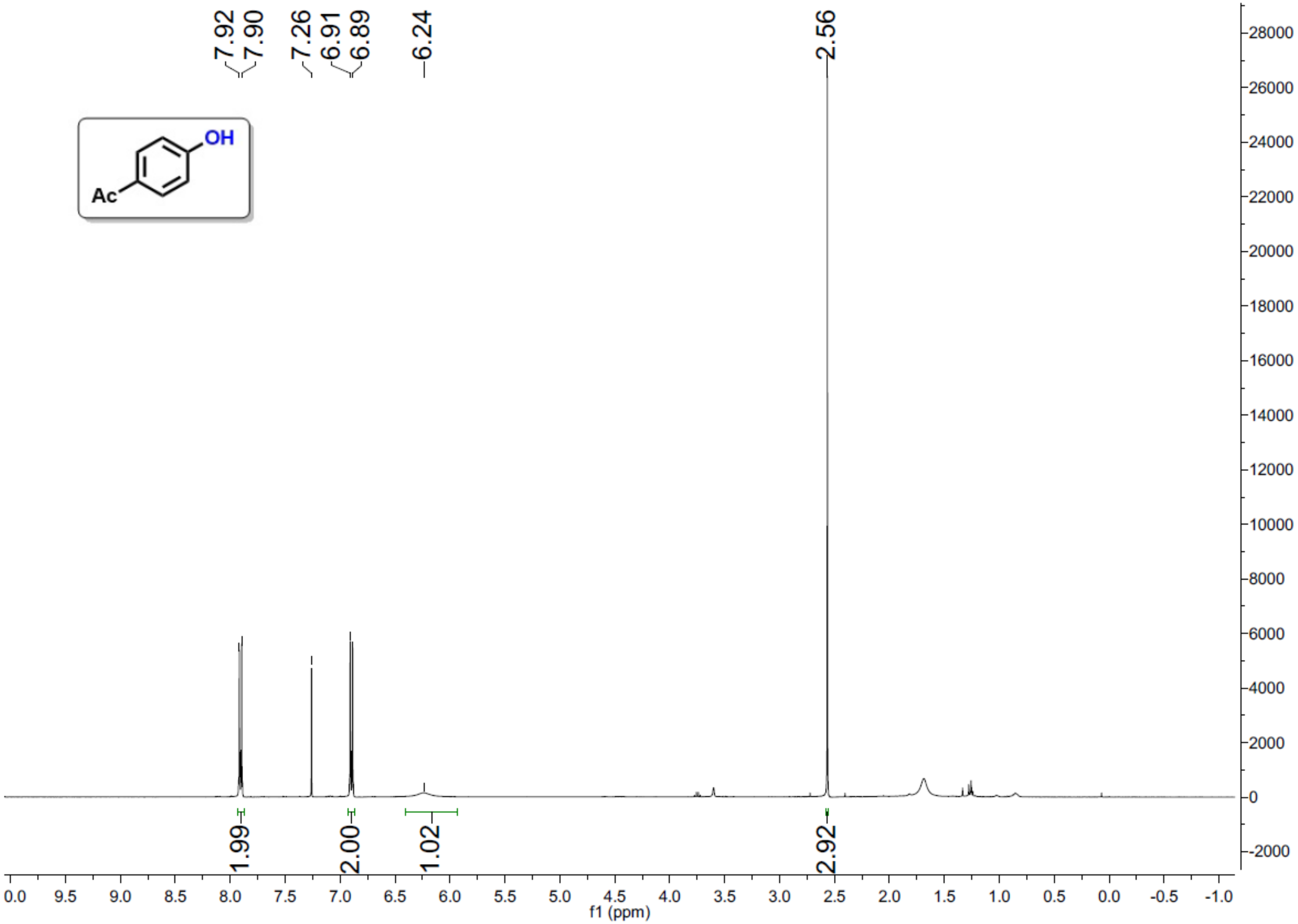
20
10
0
−10
f1 (ppm)

^{1}H NMR of 2f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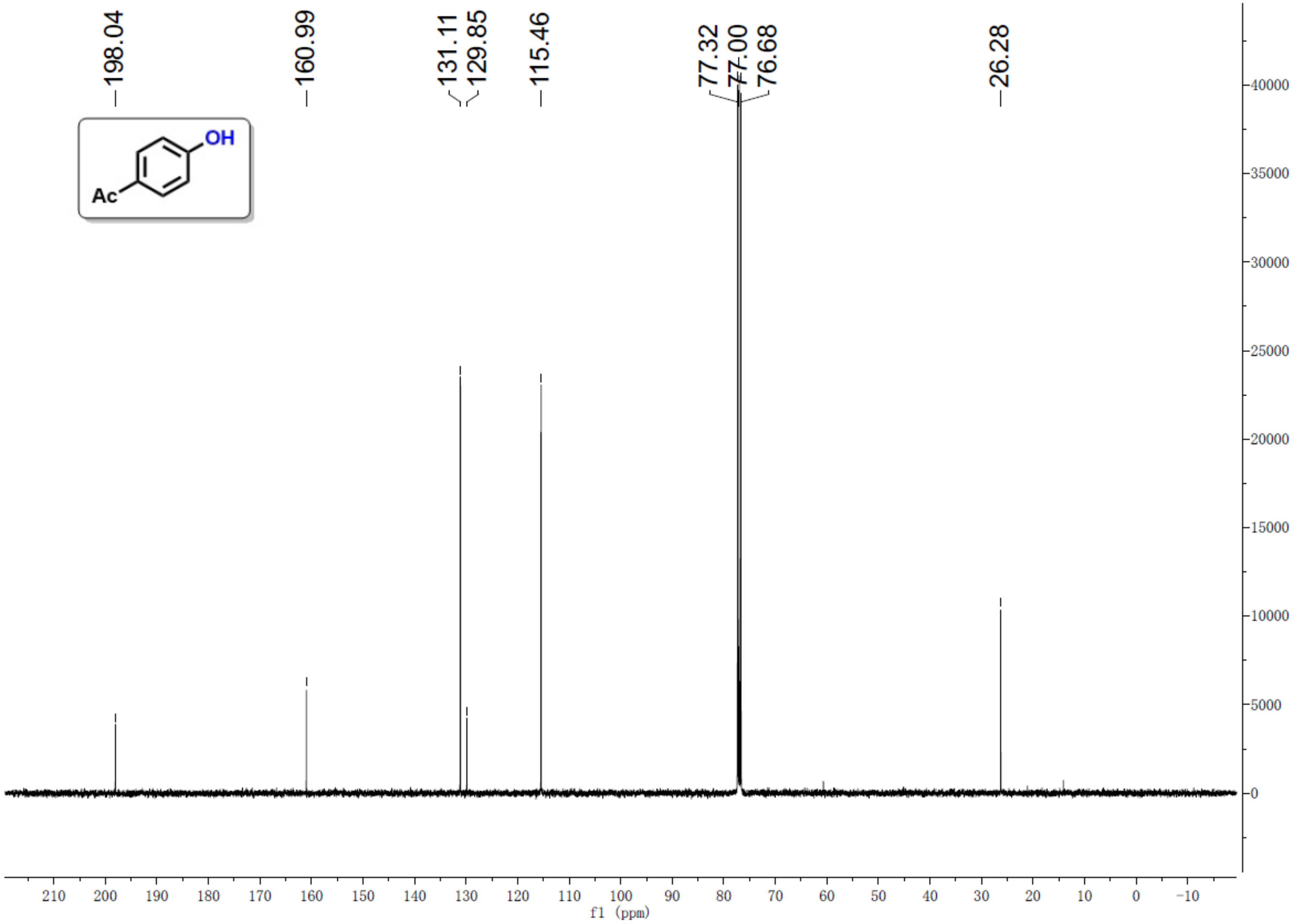
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
0.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

^{13}C NMR of 2f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20000
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10000
5000
0
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200
190
180
170
160
150
140
130
120
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100
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80
70
60
50
40
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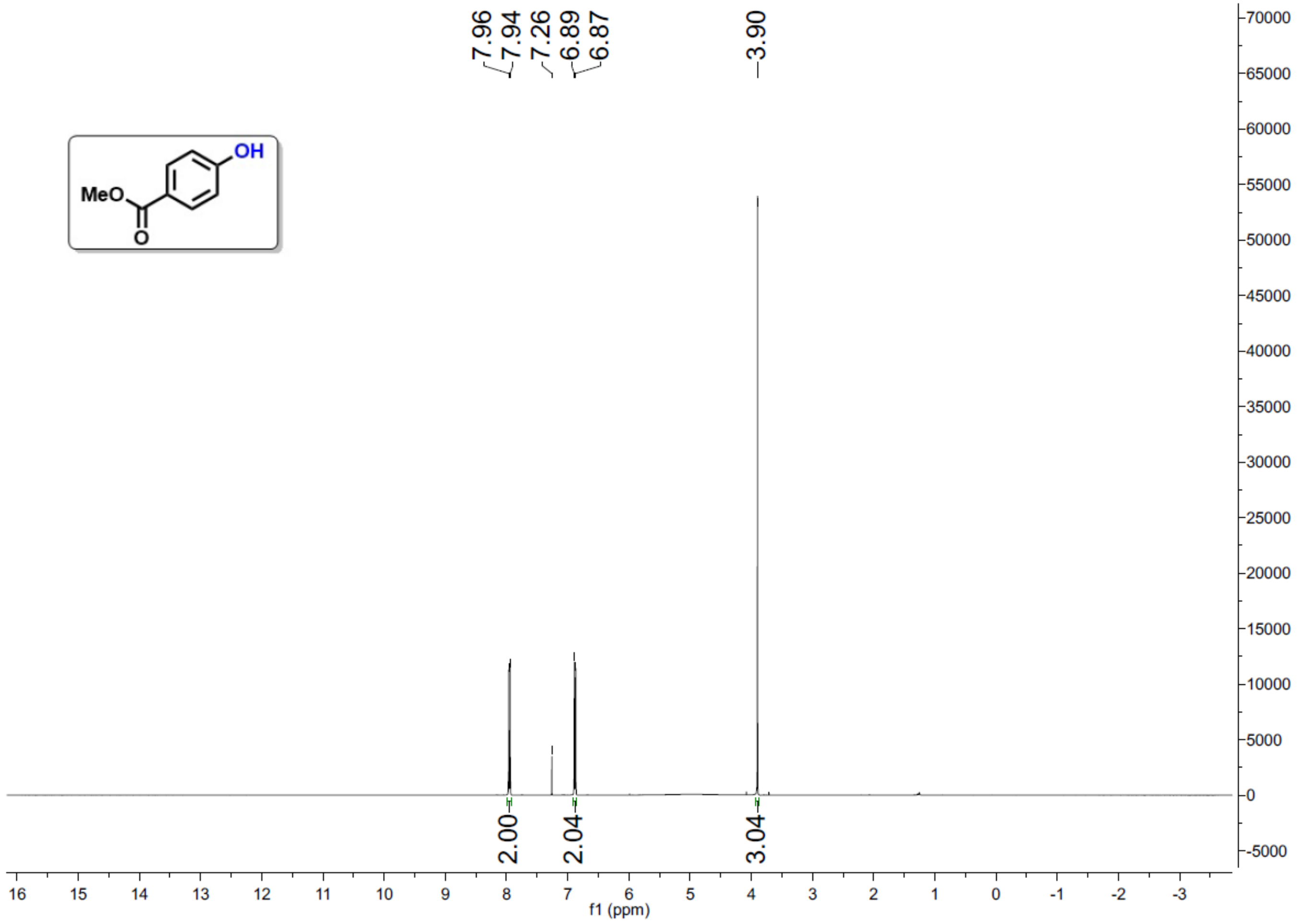
20
10
0
-10
f1 (ppm)

^{1}H NMR of 2g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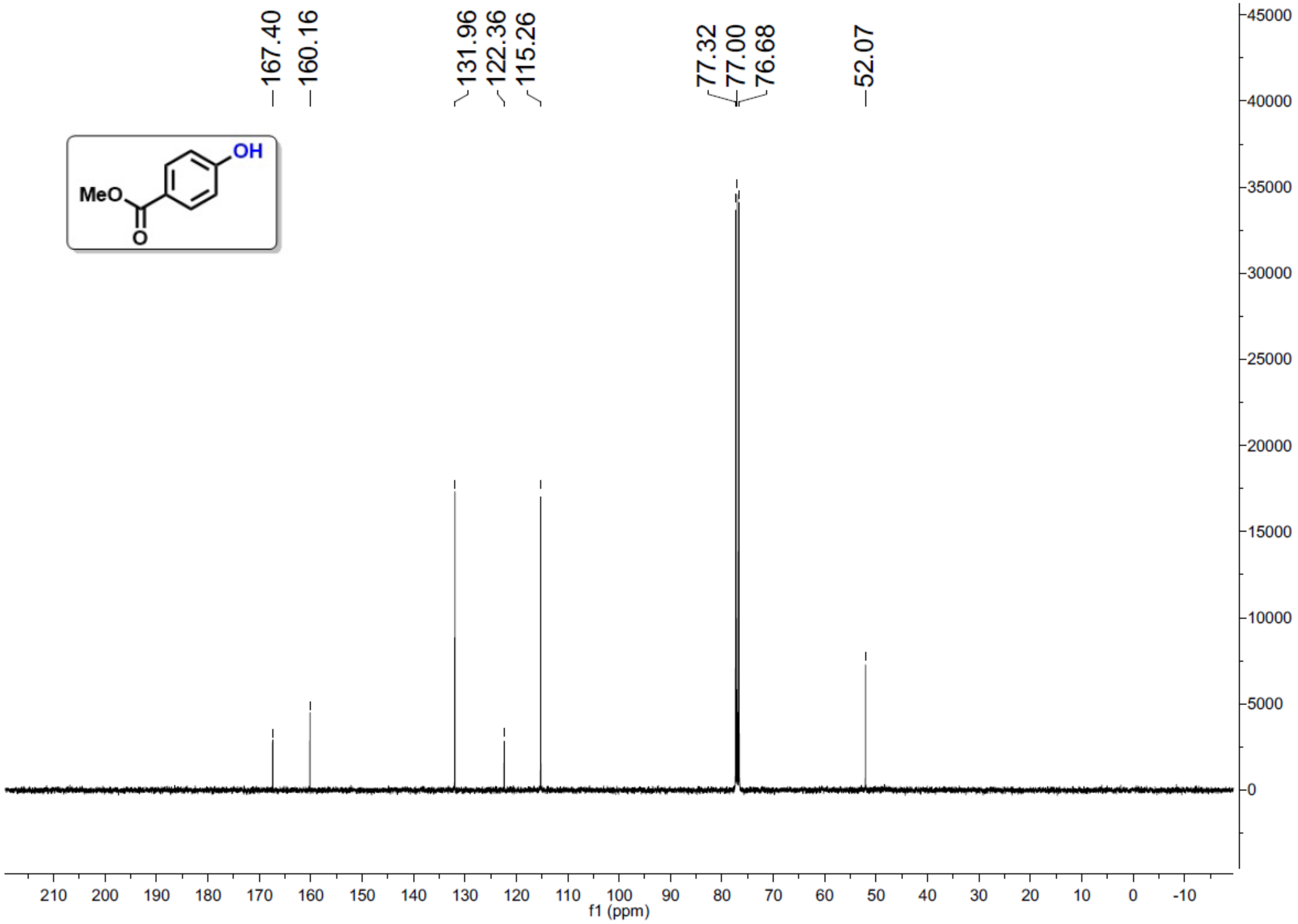
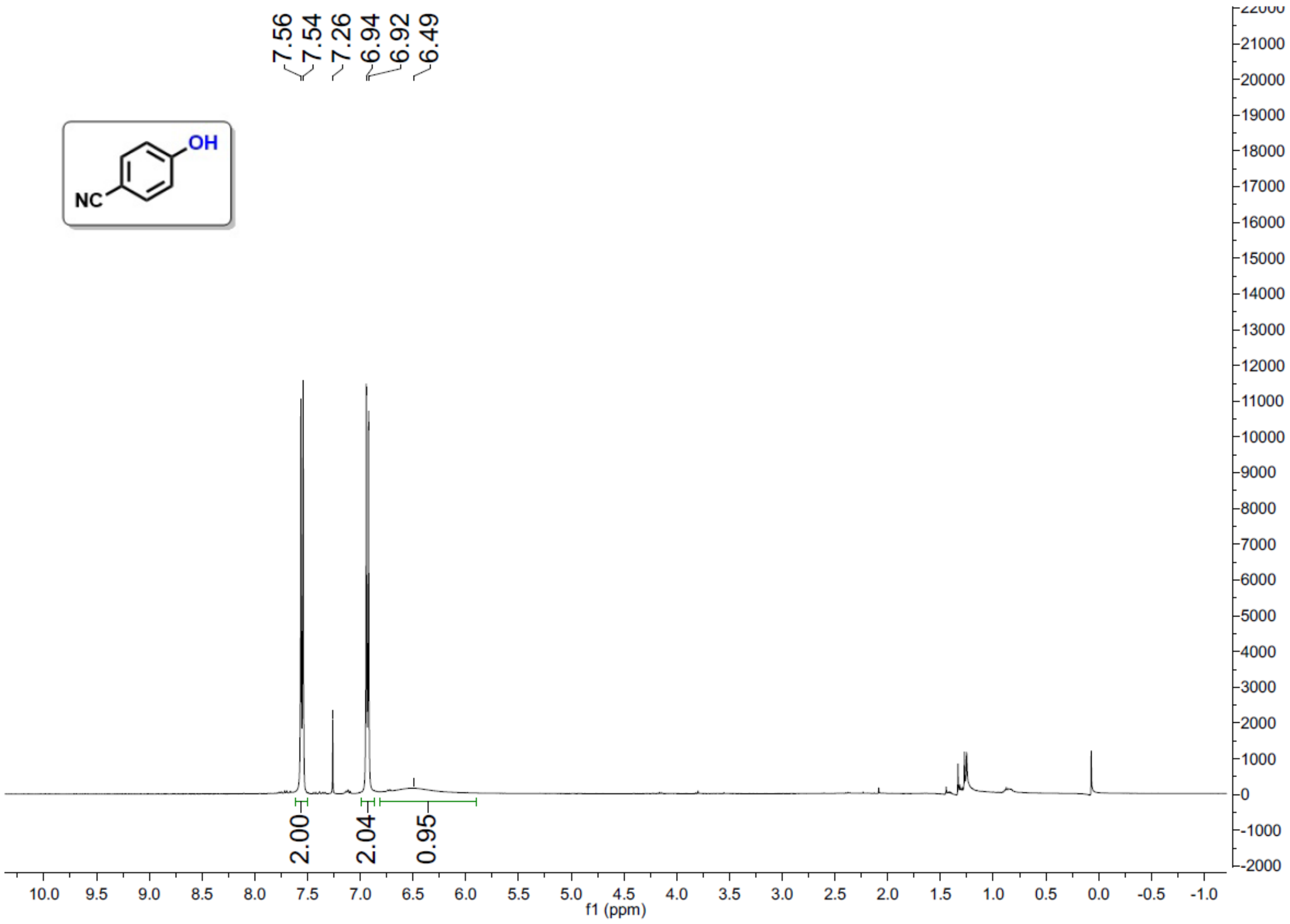
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)

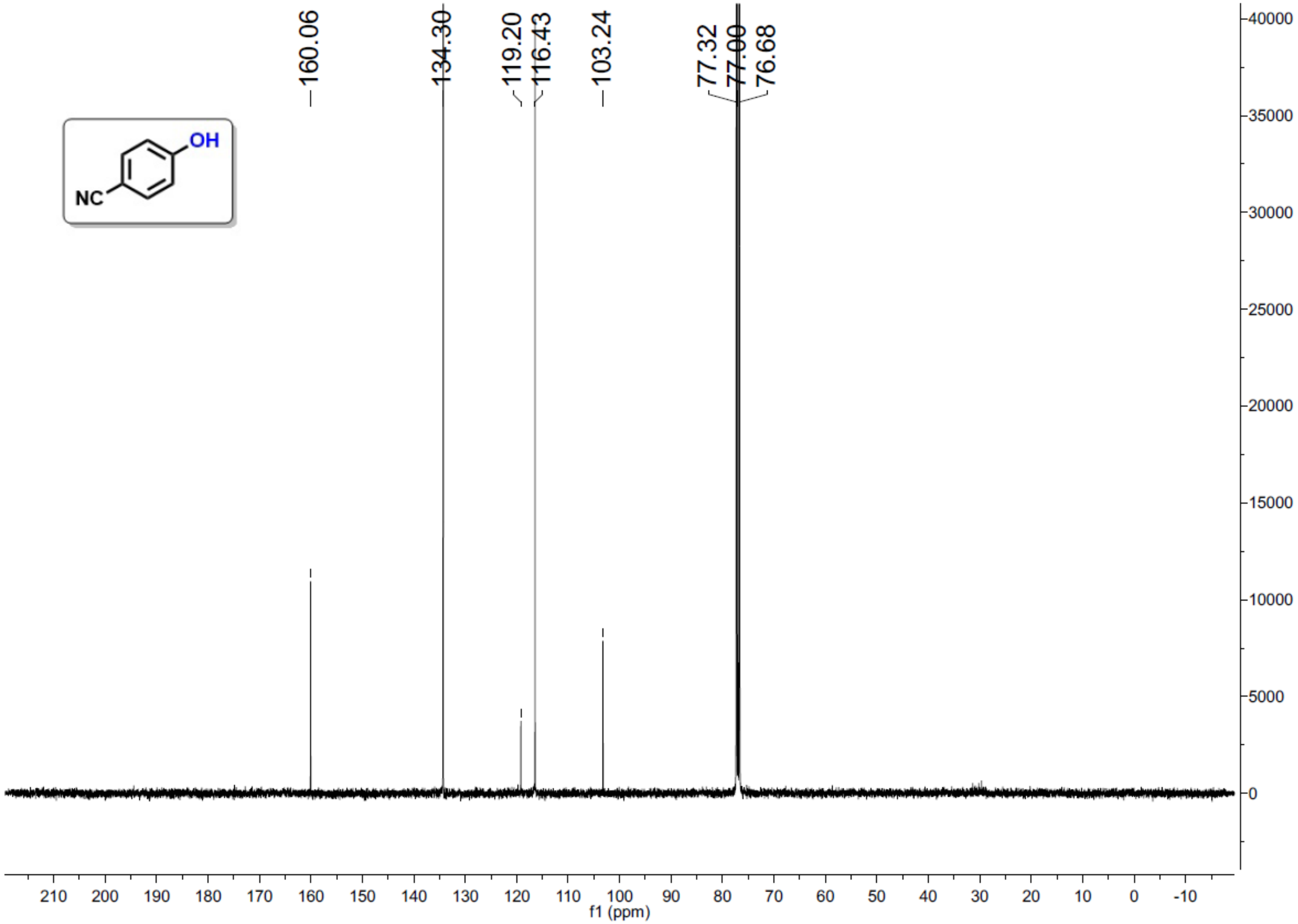
^{13}C NMR of 2g


167.40
160.16
131.96
122.36
115.26
77.32
77.00
76.68
52.07
OH
MeO
O
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
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)

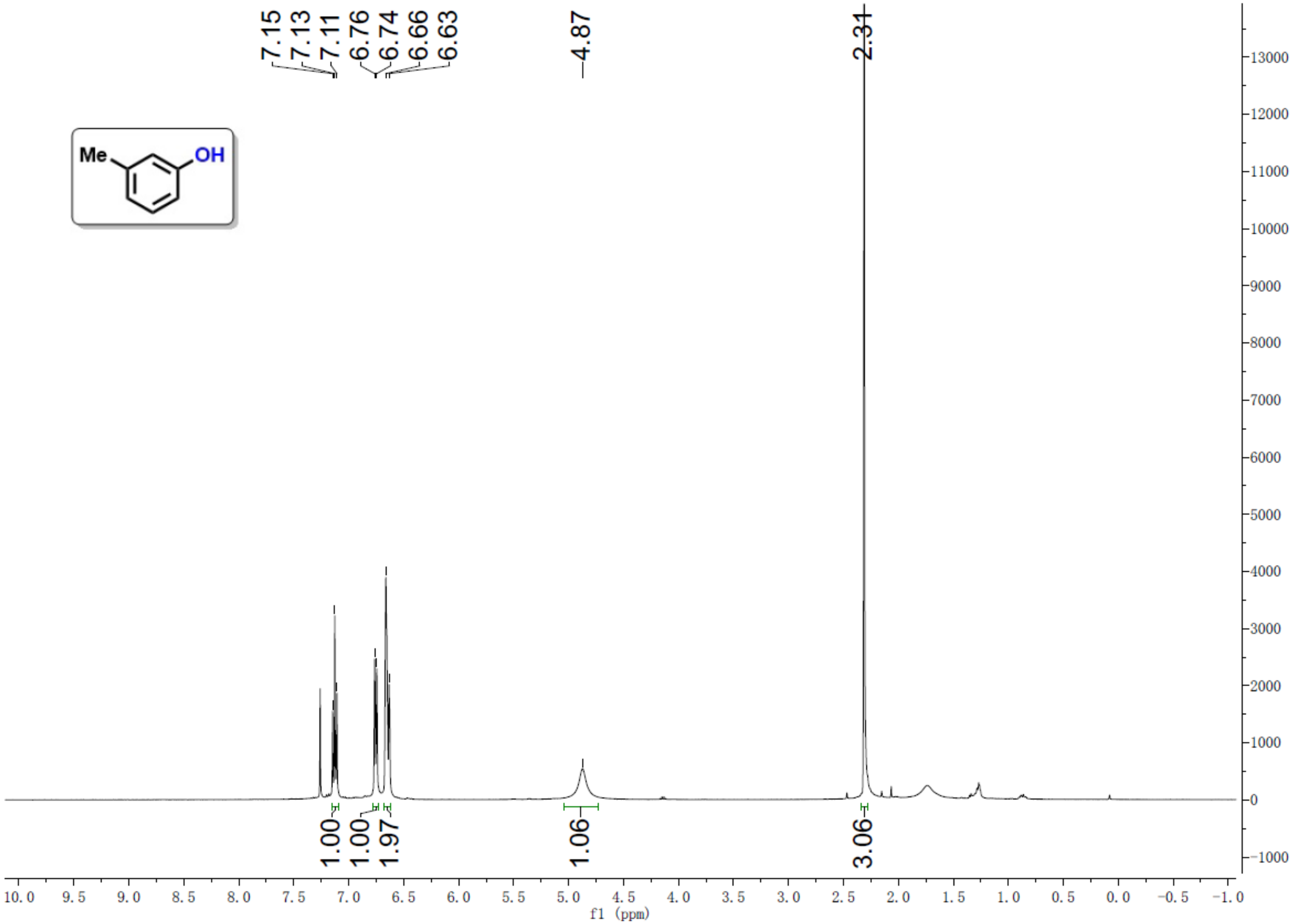
^{1}H NMR of 2h


7.56
7.54
7.26
6.94
6.92
6.49
OH
NC
2.00
2.04
0.95
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)
22000 21000 20000 19000 18000 17000 16000 15000 14000 13000 12000 11000 10000 9000 8000 7000 6000 5000 4000 3000 2000 1000 0 -1000 -2000

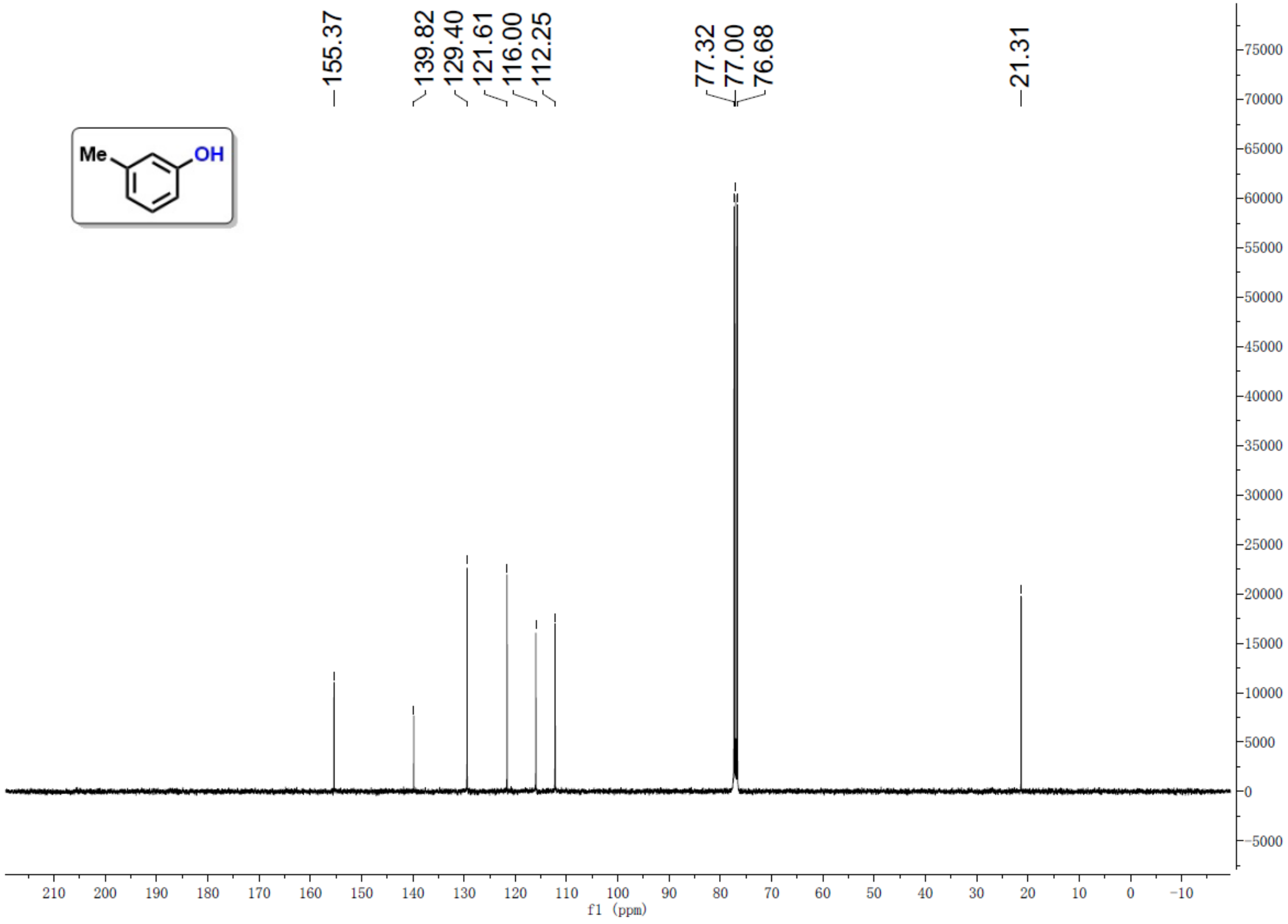
^{13}C NMR of 2h


160.06
134.30
119.20
116.43
103.24
77.32
77.00
76.68
OH
NC
40000
35000
30000
25000
20000
15000
10000
5000
0
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)

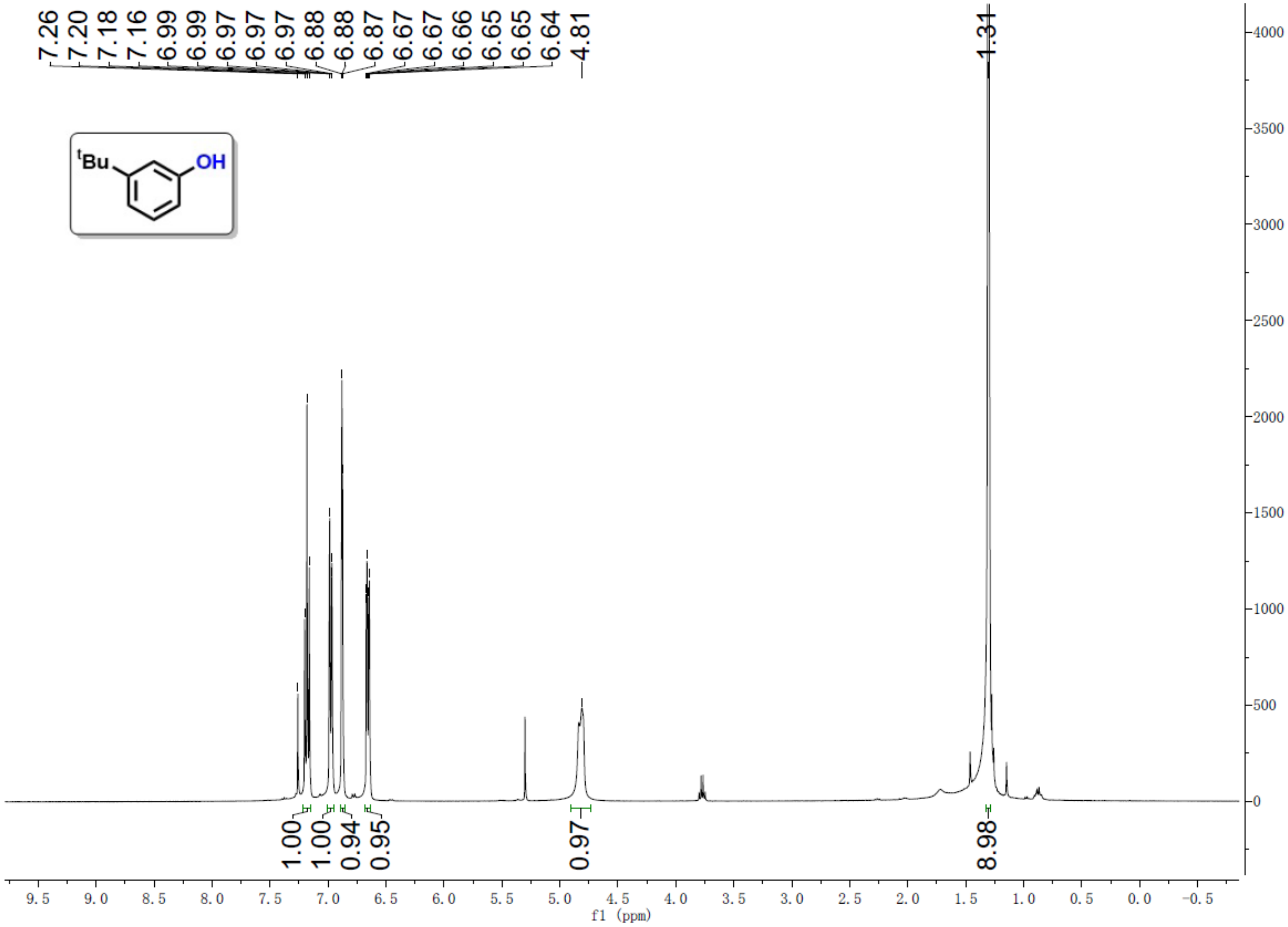
^{1}H NMR of 2i


7.15
7.13
7.11
6.76
6.74
6.66
6.63
4.87
2.31
Me
OH
1.00
1.00
1.97
1.06
3.06
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

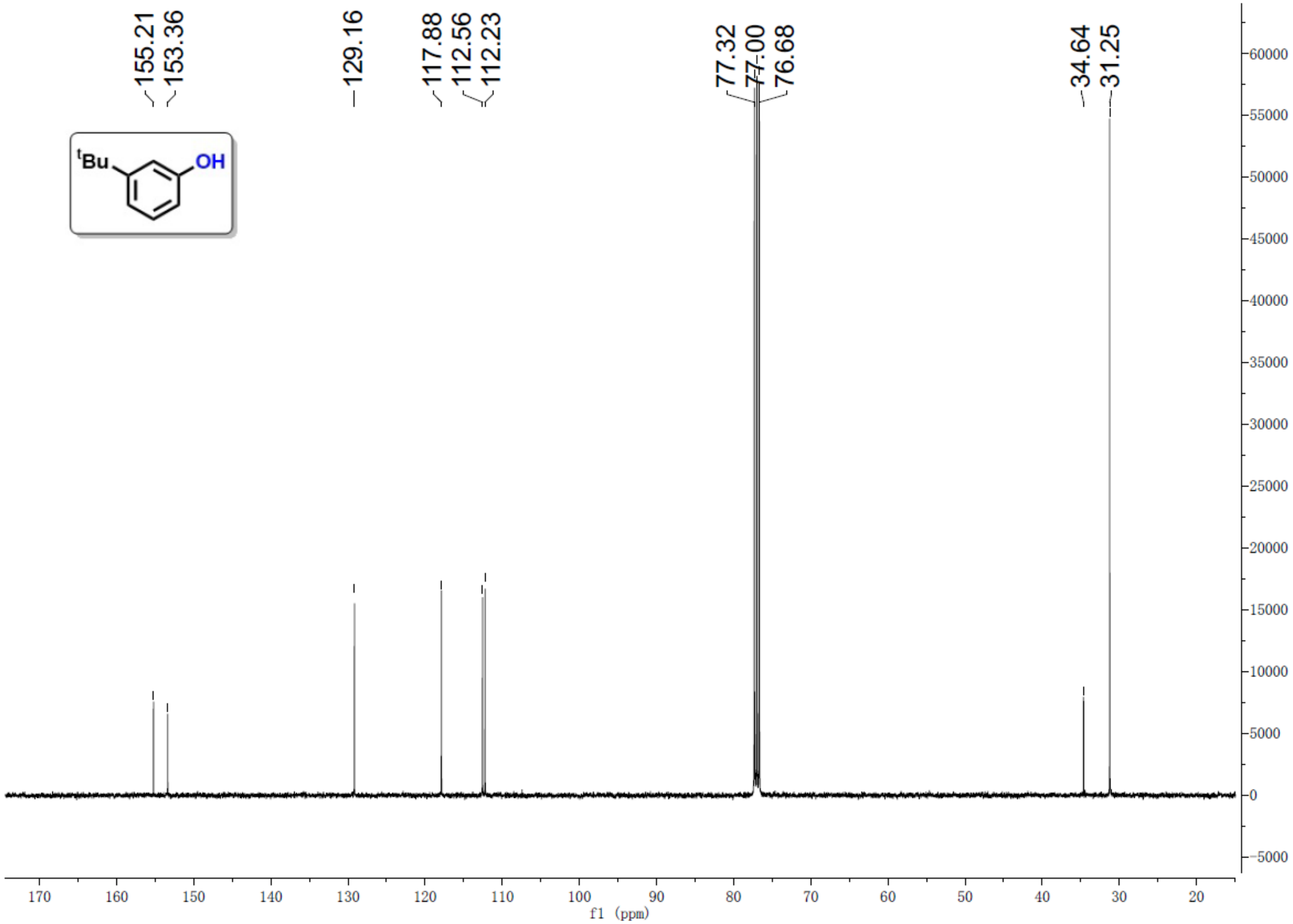
^{13}C NMR of 2i


155.37
139.82
129.40
121.61
116.00
112.25
77.32
77.00
76.68
21.31
Me
OH
75000
70000
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
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)

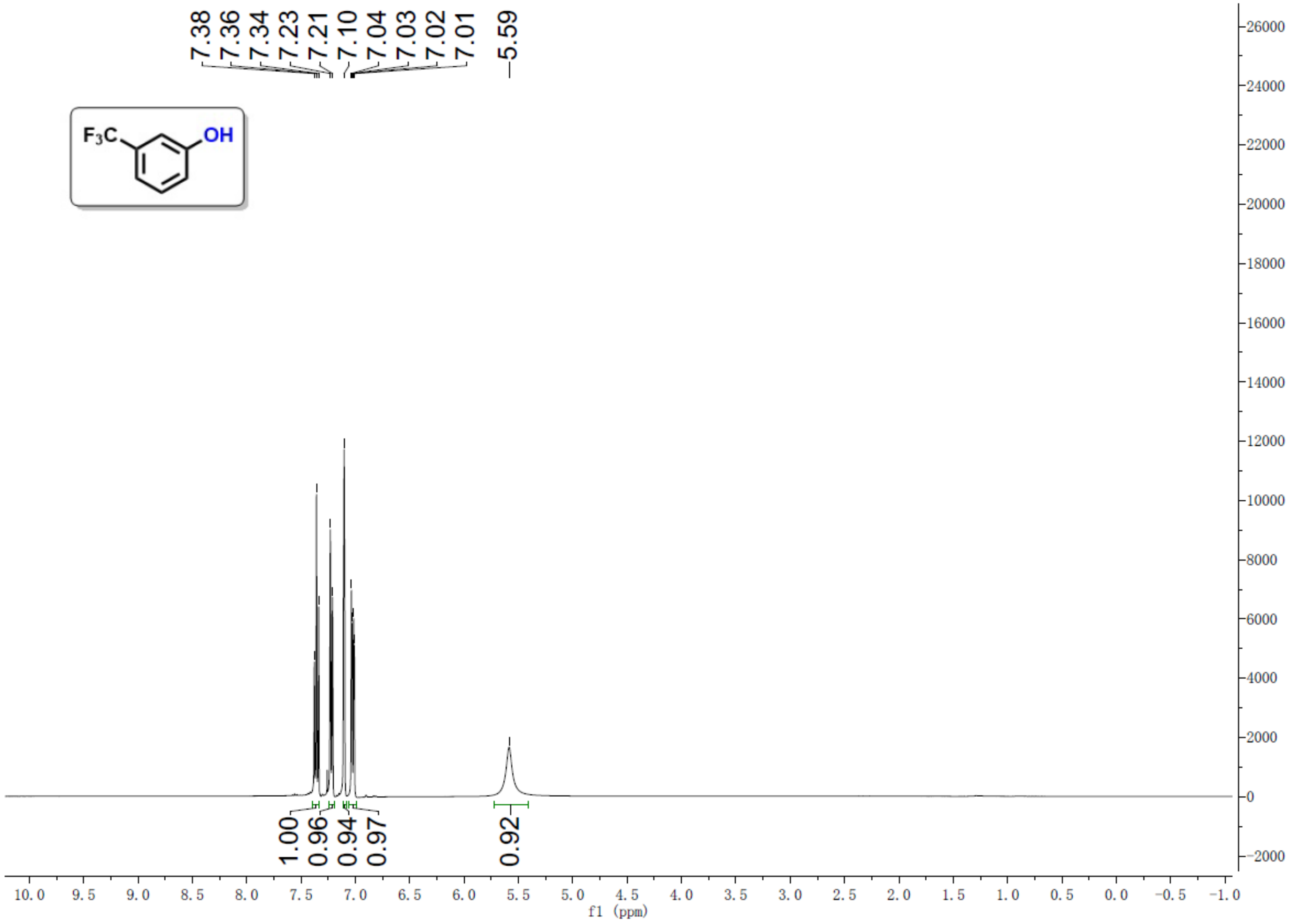
^{1}H NMR of 2j


7.26
7.20
7.18
7.16
6.99
6.99
6.97
6.97
6.97
6.88
6.88
6.87
6.67
6.67
6.66
6.65
6.65
6.64
4.81
1.31
ᵗBu
OH
1.00
1.00
0.94
0.95
0.97
8.98
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)
4000
3500
3000
2500
2000
1500
1000
500
0

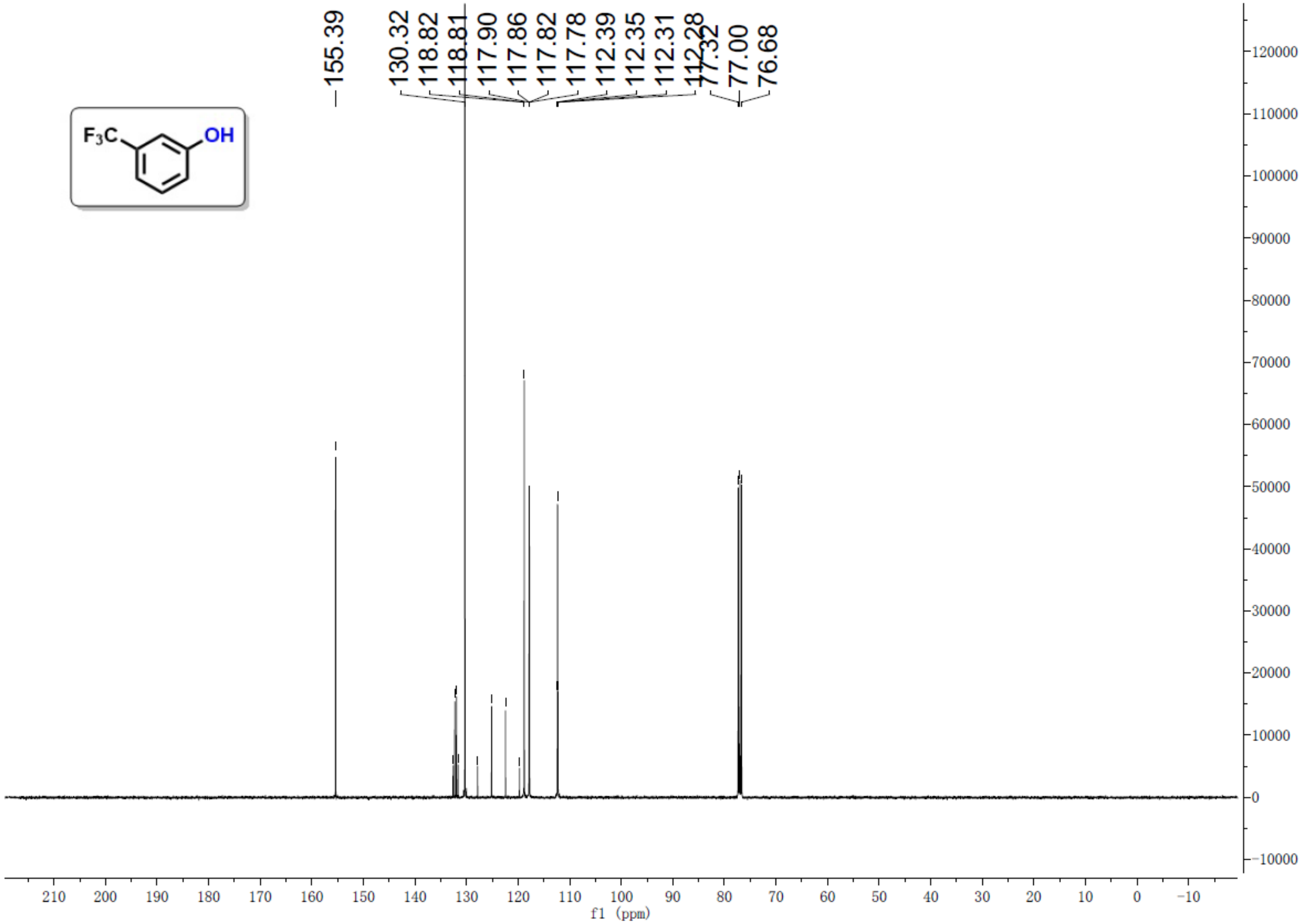
^{13}C NMR of 2j


155.21
153.36
129.16
117.88
112.56
112.23
77.32
77.00
76.68
34.64
31.25
tBu
OH
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)

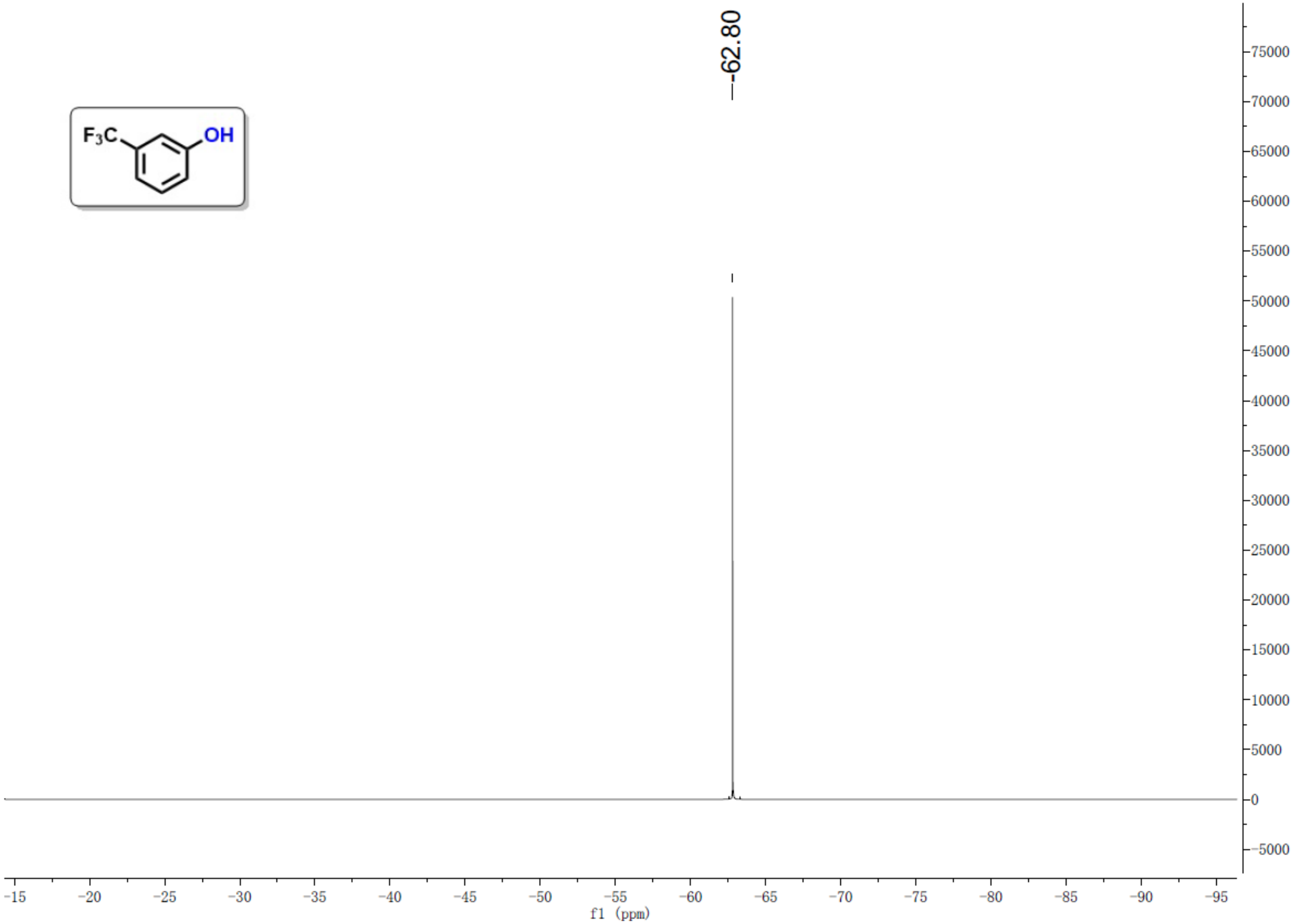
^{1}H NMR of 2k


7.38
7.36
7.34
7.23
7.21
7.10
7.04
7.03
7.02
7.01
5.59
F3C
OH
1.00
0.96
0.94
0.97
0.92
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

^{13}C NMR of 2k

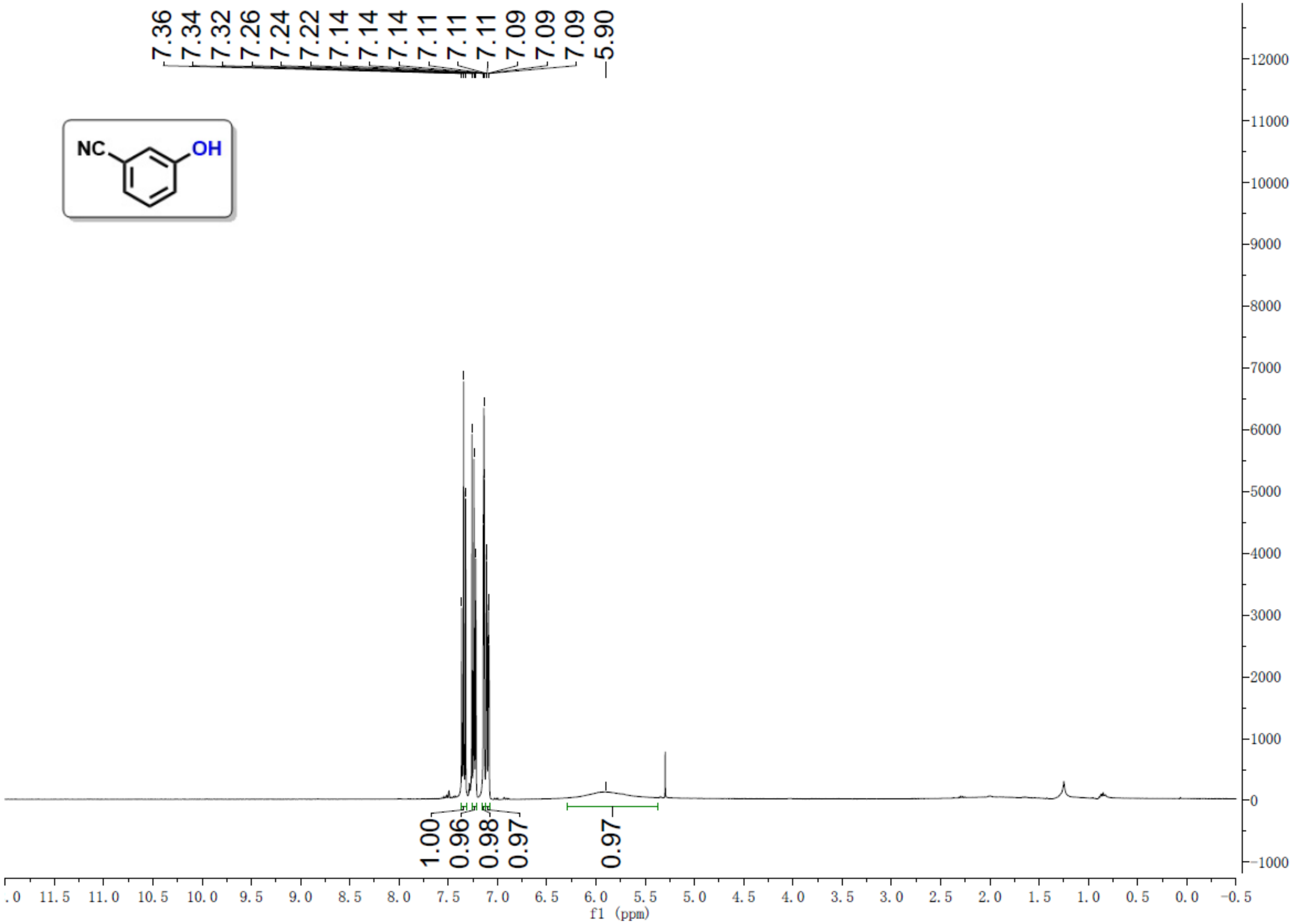

155.39
130.32
118.82
118.81
117.90
117.86
117.82
117.78
112.39
112.35
112.31
112.28
77.32
77.00
76.68
F3C
OH
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)
120000 110000 100000 90000 80000 70000 60000 50000 40000 30000 20000 10000 0 -10000

^{19}F NMR of 2k

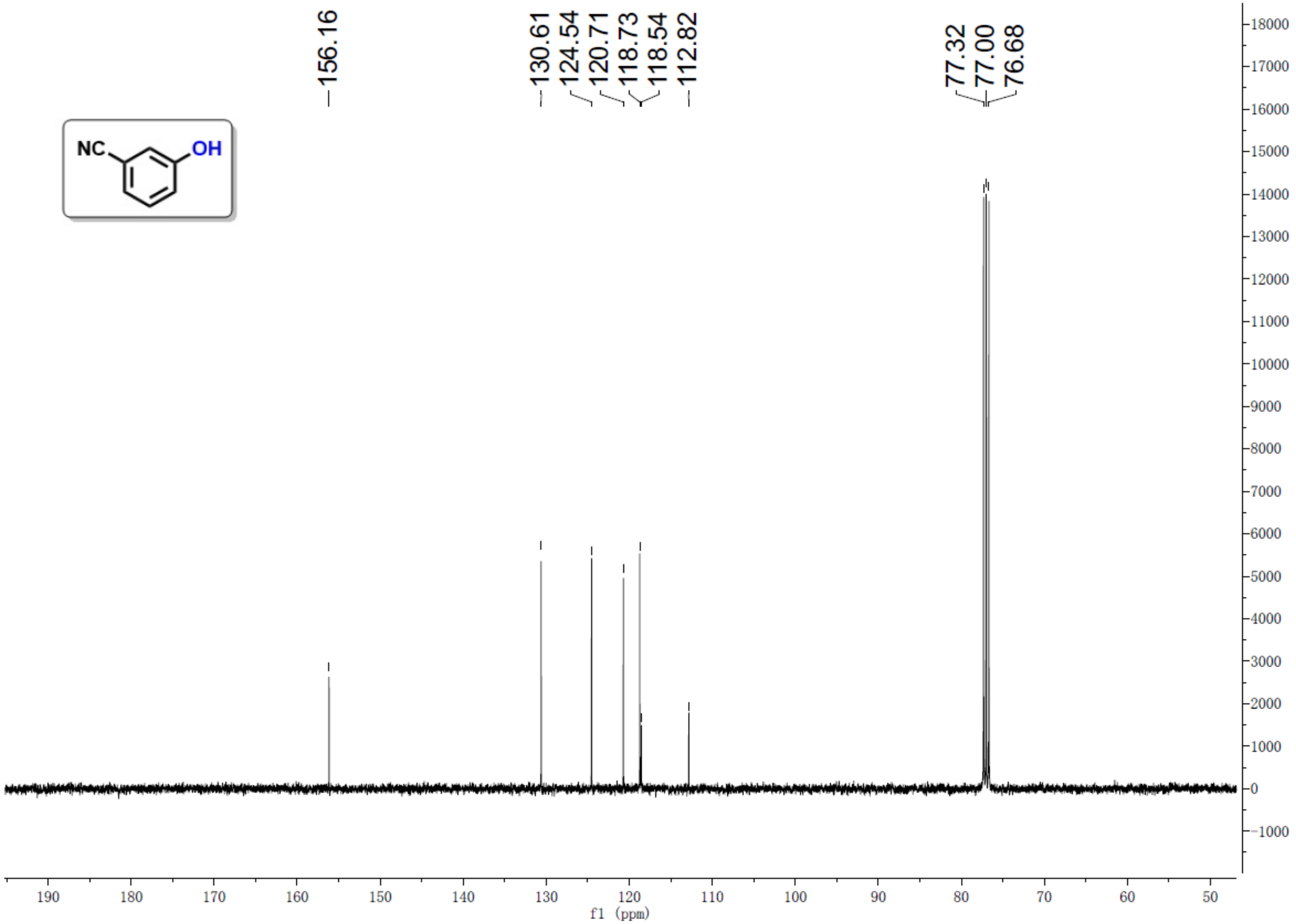


-62.80
F3C
OH
75000
70000
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
-15
-20
-25
-30
-35
-40
-45
-50
-55
-60
-65
-70
-75
-80
-85
-90
-95
f1 (ppm)

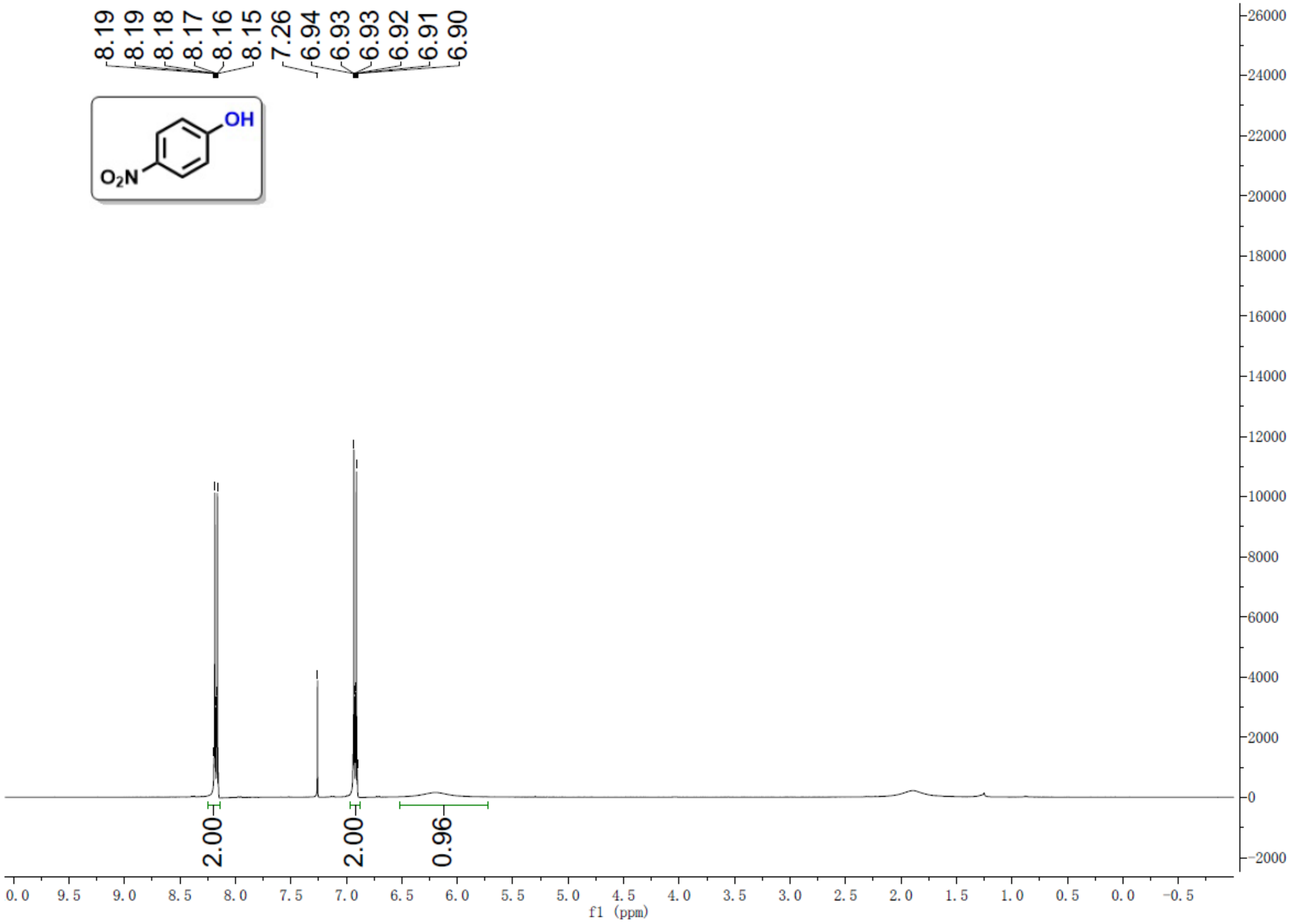
^{1}H NMR of 2l


7.36
7.34
7.32
7.26
7.24
7.22
7.14
7.14
7.14
7.11
7.11
7.11
7.09
7.09
7.09
5.90
NC
OH
1.00
0.96
0.98
0.97
0.97
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
−1000
.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
f1 (ppm)

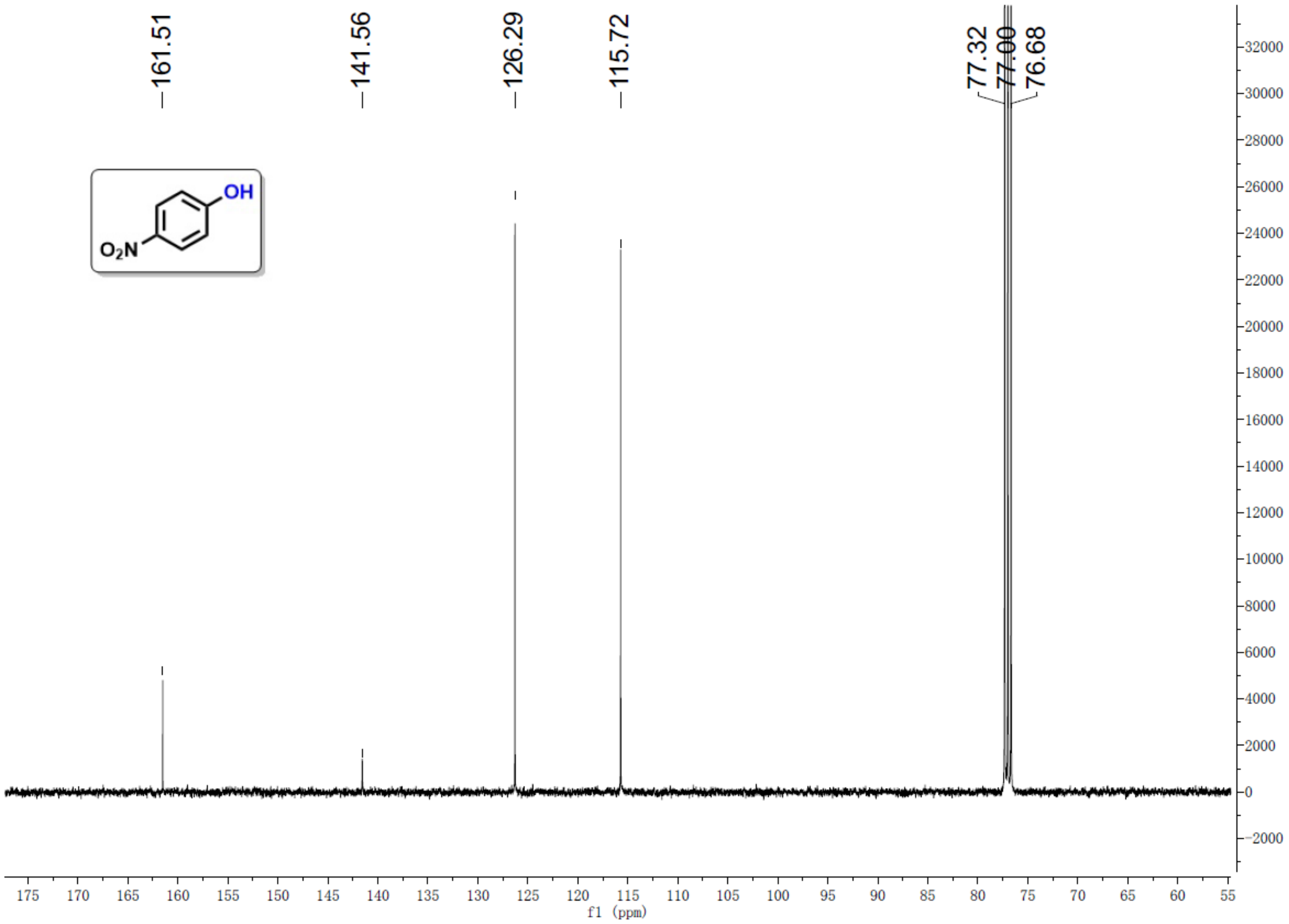
^{13}C NMR of 2l


NC
OH
156.16
130.61
124.54
120.71
118.73
118.54
112.82
77.32
77.00
76.68
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
−1000
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
f1 (ppm)

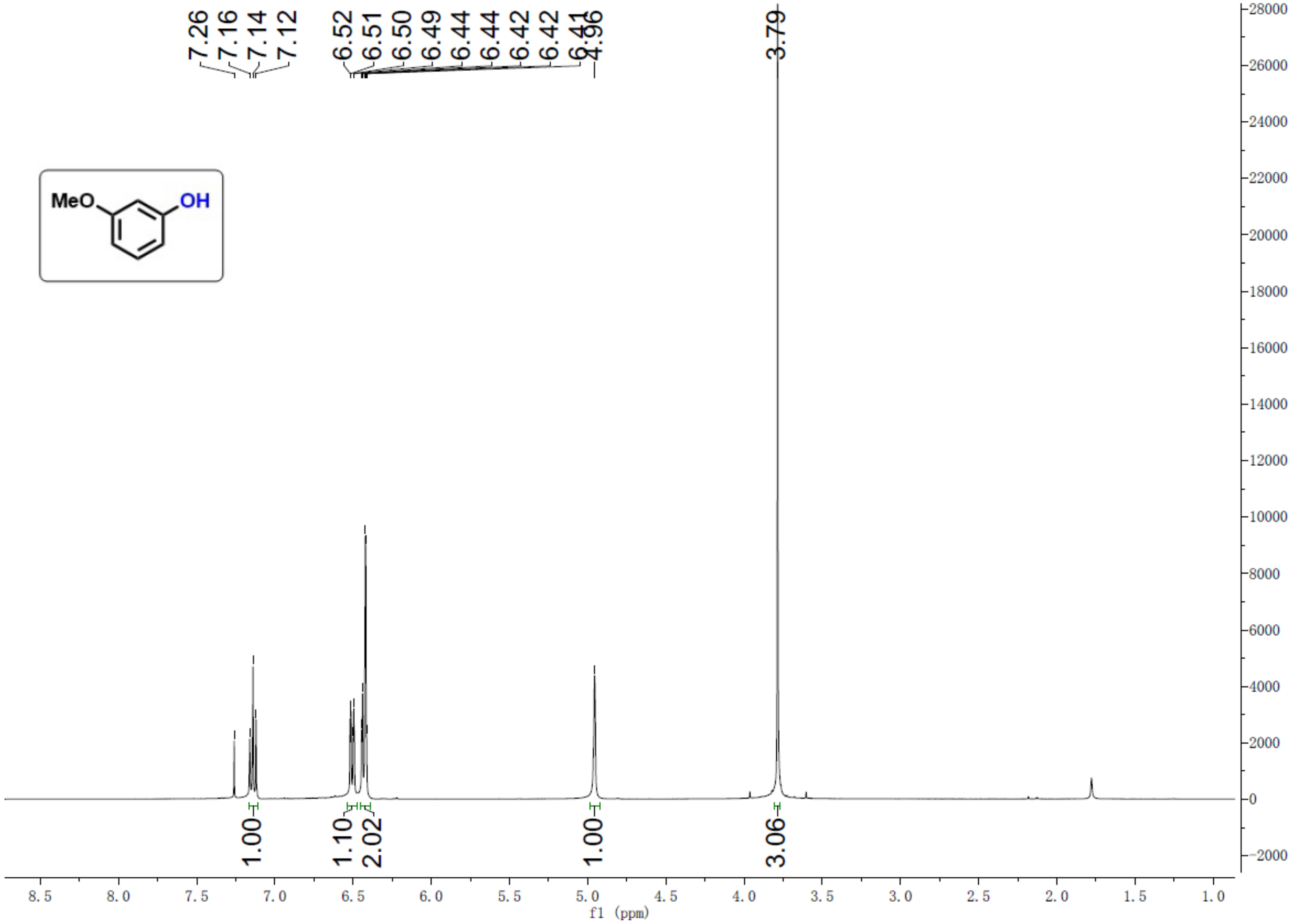
^{1}H NMR of 2m


8.19
8.19
8.18
8.17
8.16
8.15
7.26
6.94
6.93
6.93
6.92
6.91
6.90
OH
O_2N
2.00
2.00
0.96
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
0.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

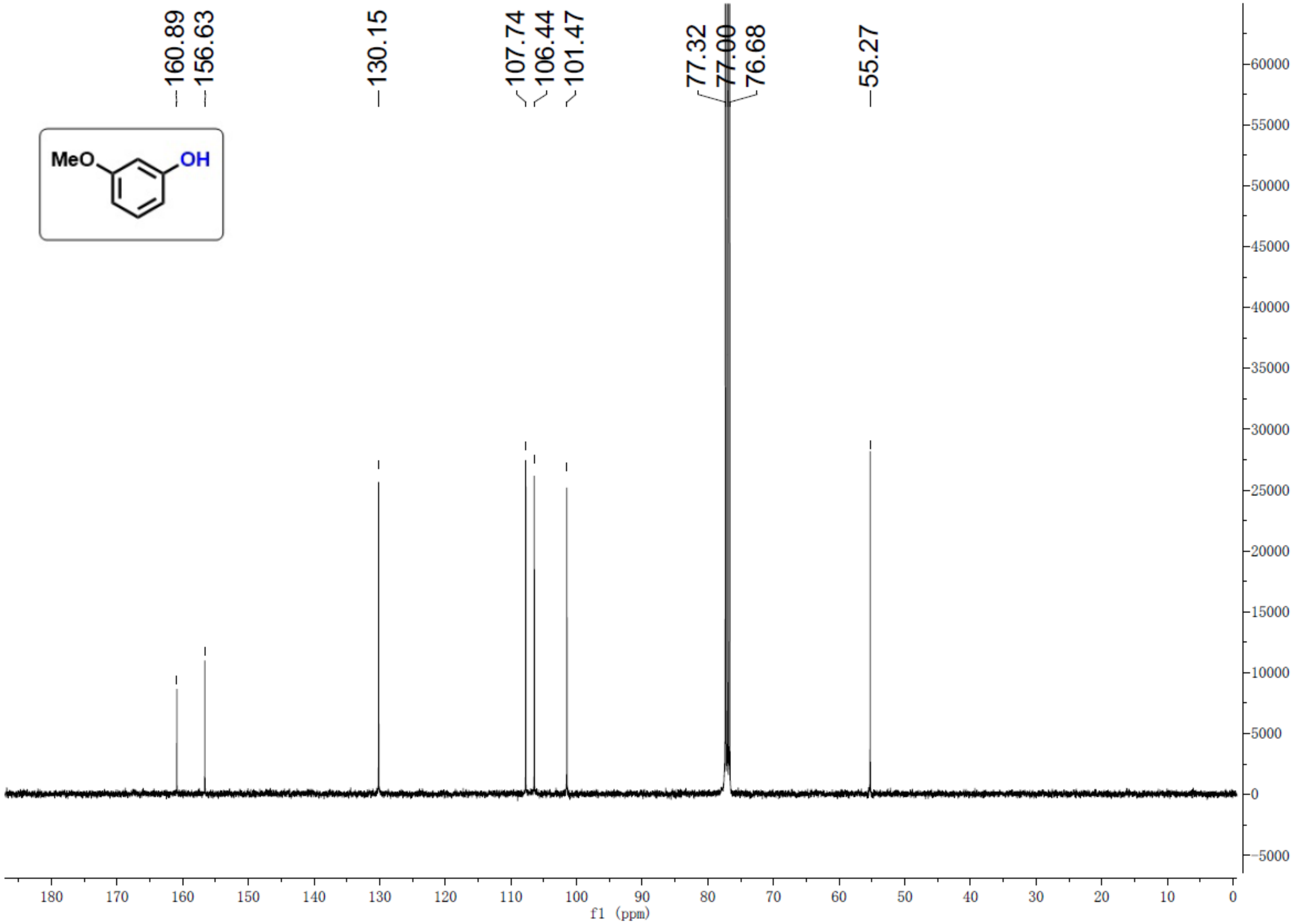
^{13}C NMR of 2m


161.51
141.56
126.29
115.72
77.32
77.00
76.68
OH
O_2N
32000
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
−2000
175
170
165
160
155
150
145
140
135
130
125
120
115
110
105
100
95
90
85
80
75
70
65
60
55
f1 (ppm)

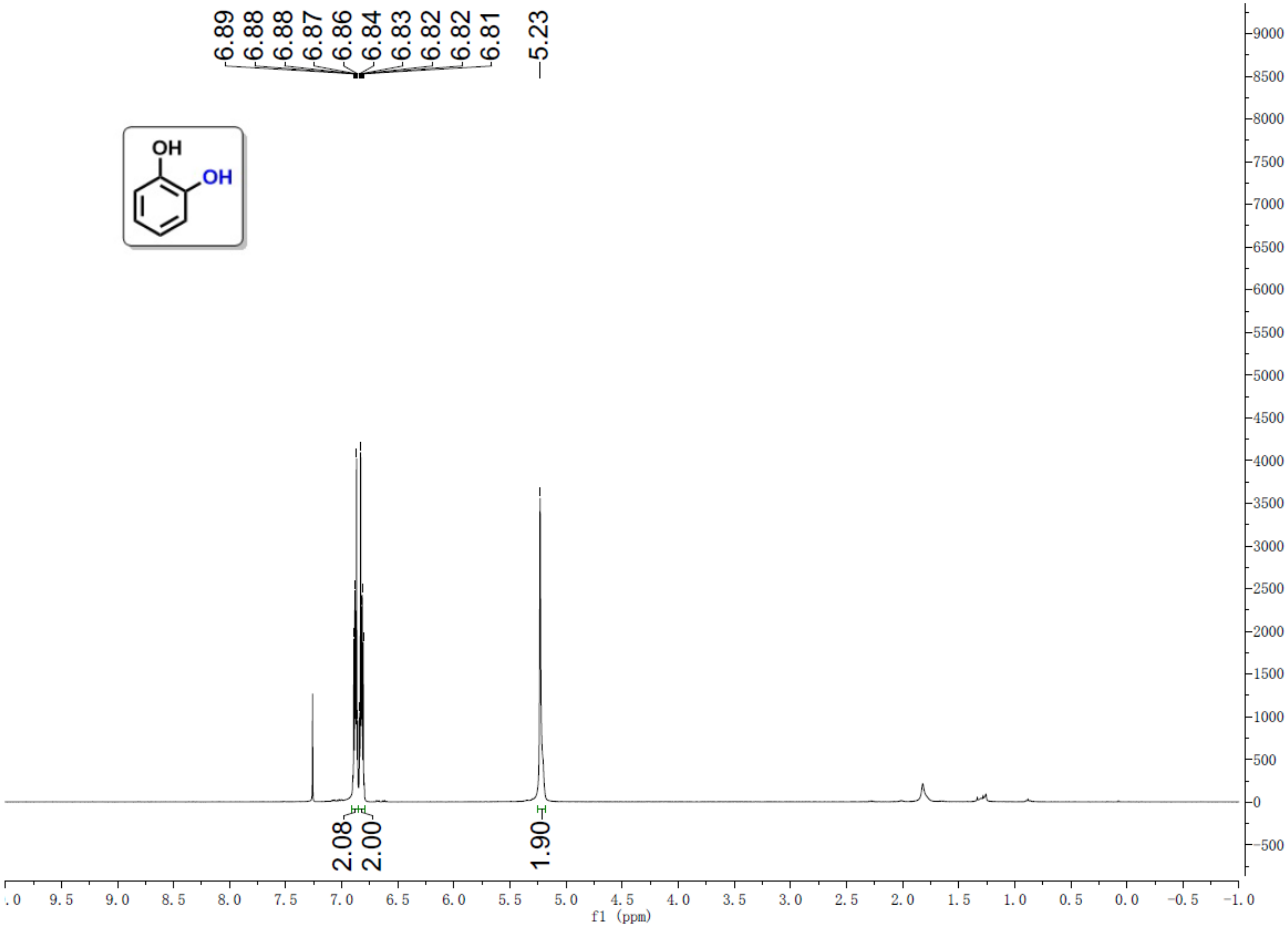
^{1}H NMR of 2n


7.26
7.16
7.14
7.12
6.52
6.51
6.50
6.49
6.44
6.44
6.42
6.42
6.41
4.96
3.79
MeO
OH
1.00
1.10
2.02
1.00
3.06
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
f1 (ppm)
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000

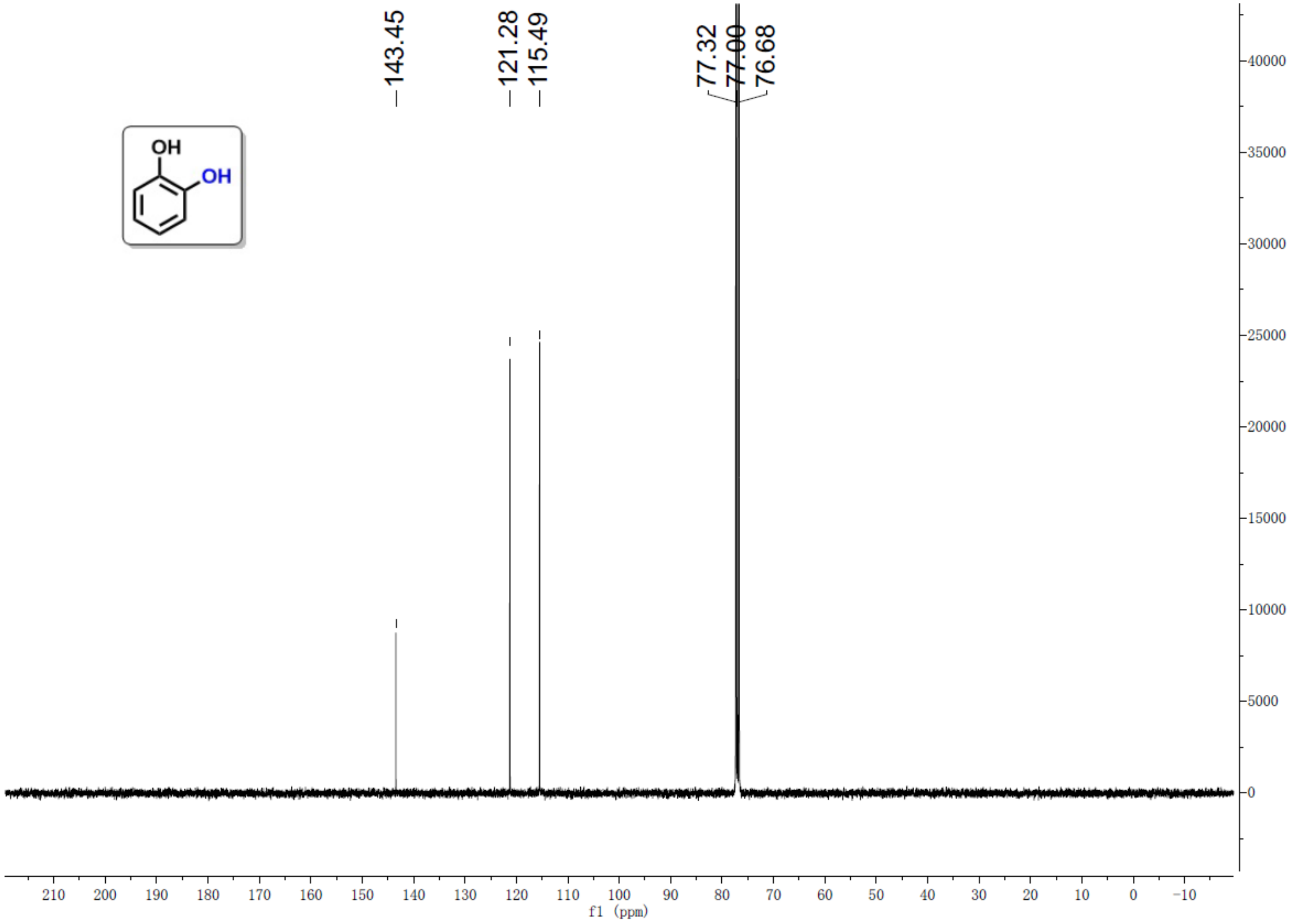
^{13}C NMR of 2n


160.89
156.63
130.15
107.74
106.44
101.47
77.32
77.00
76.68
55.27
MeO
OH
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

^{1}H NMR of 2o

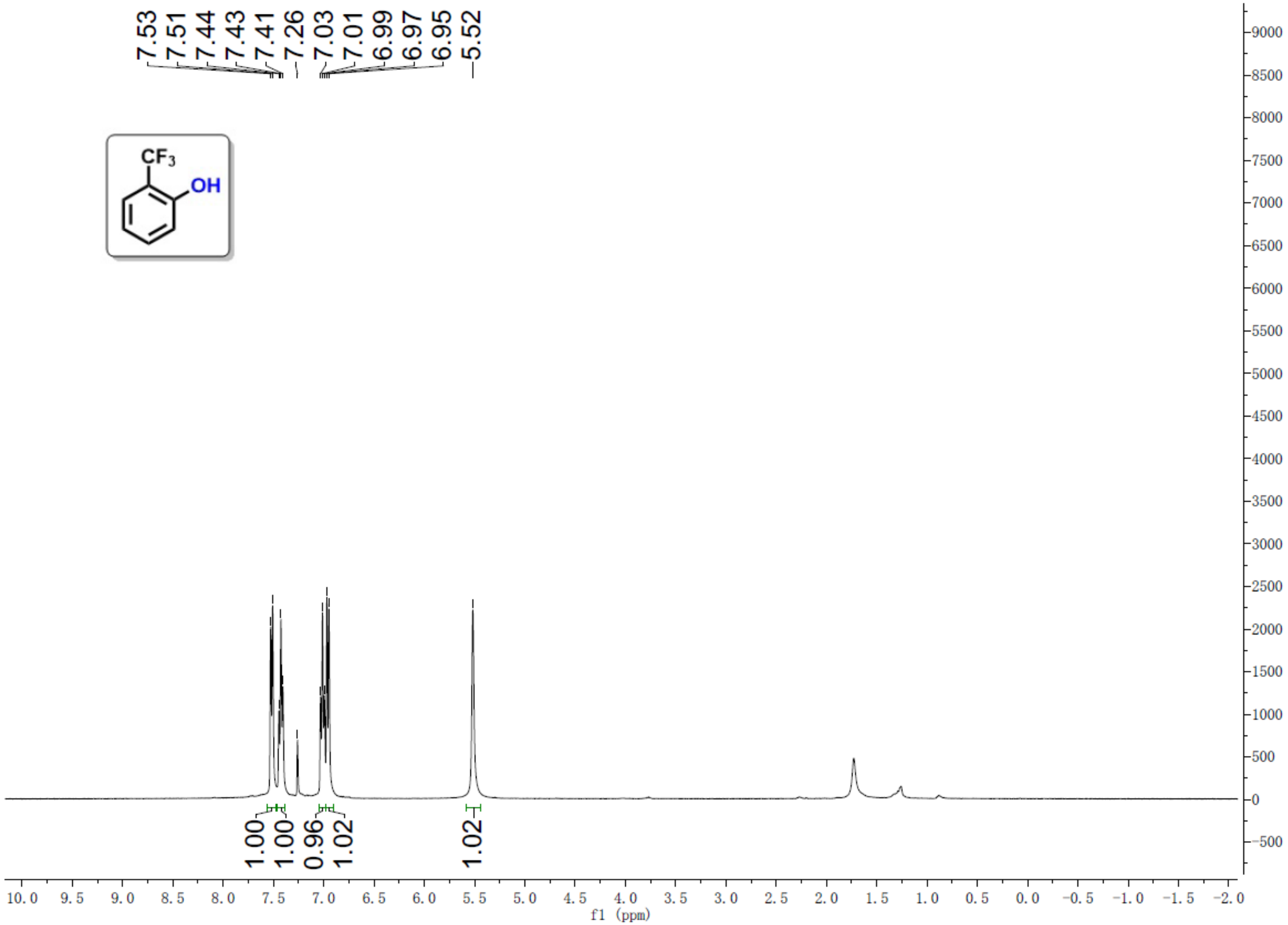

6.89
6.88
6.88
6.87
6.86
6.84
6.83
6.82
6.82
6.81
5.23
OH
OH
2.08
2.00
1.90
f1 (ppm)
.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
9000
8500
8000
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500

^{13}C NMR of 2o

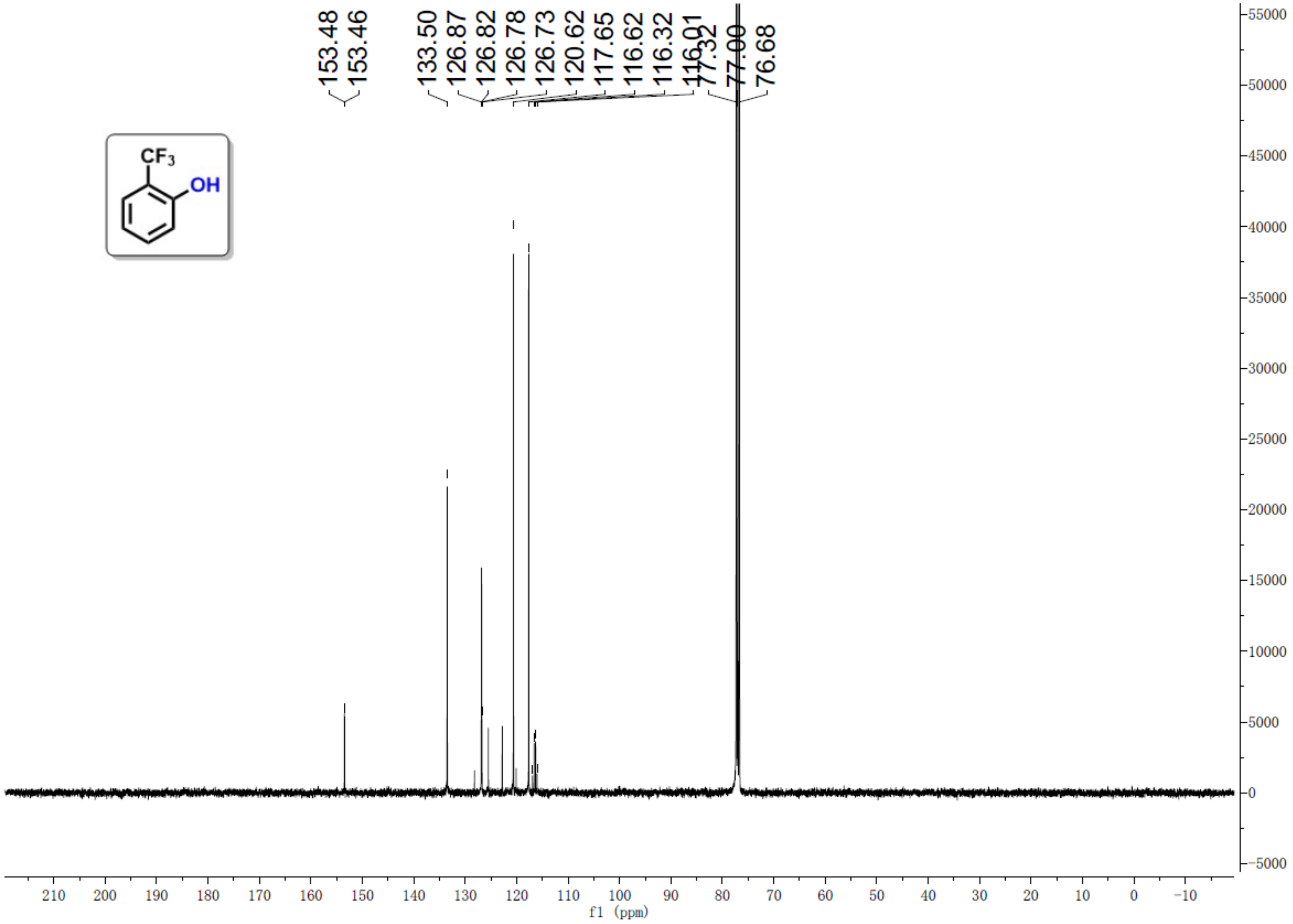


143.45
121.28
115.49
77.32
77.00
76.68
OH
OH
40000
35000
30000
25000
20000
15000
10000
5000
0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

^{1}H NMR of 2p

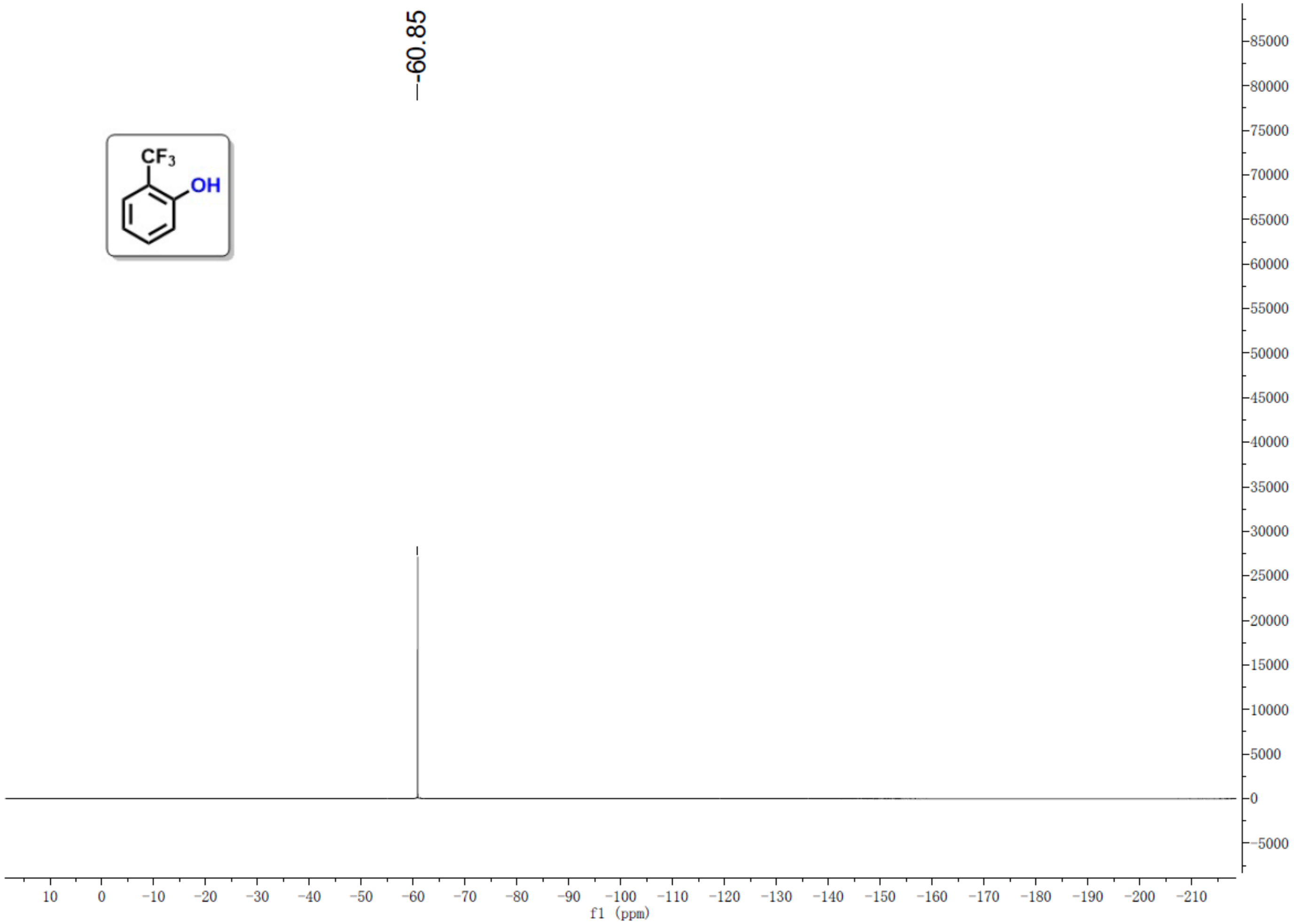

7.53
7.51
7.44
7.43
7.41
7.26
7.03
7.01
6.99
6.97
6.95
5.52
CF3
OH
1.00
1.00
0.96
1.02
1.02
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2.0
f1 (ppm)
9000 8500 8000 7500 7000 6500 6000 5500 5000 4500 4000 3500 3000 2500 2000 1500 1000 500 0 -500

^{13}C NMR of 2p



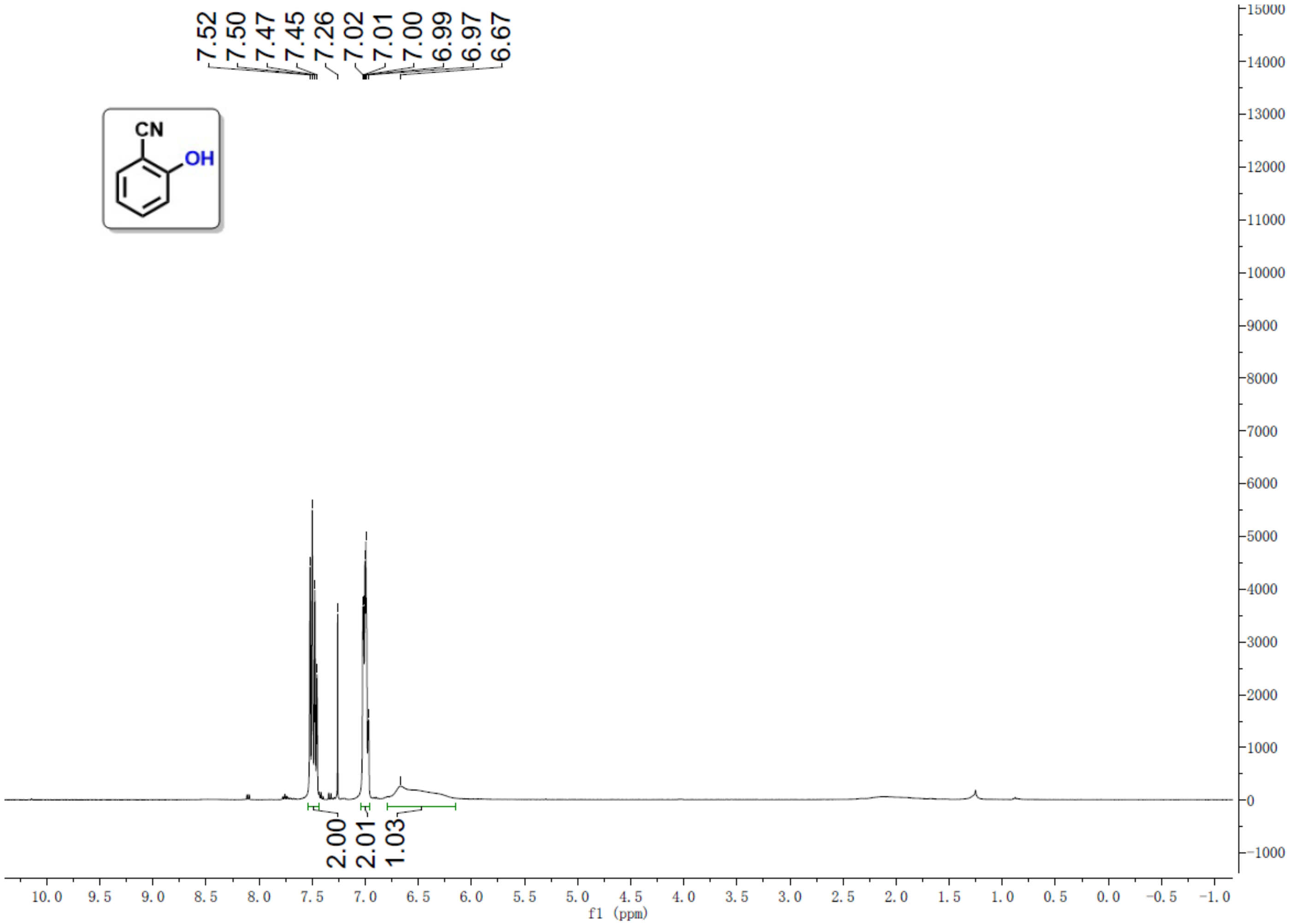
153.48
153.46
133.50
126.87
126.82
126.78
126.73
120.62
117.65
116.62
116.32
116.01
77.32
77.00
76.68
CF3
OH
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

^{19}F NMR of 2p

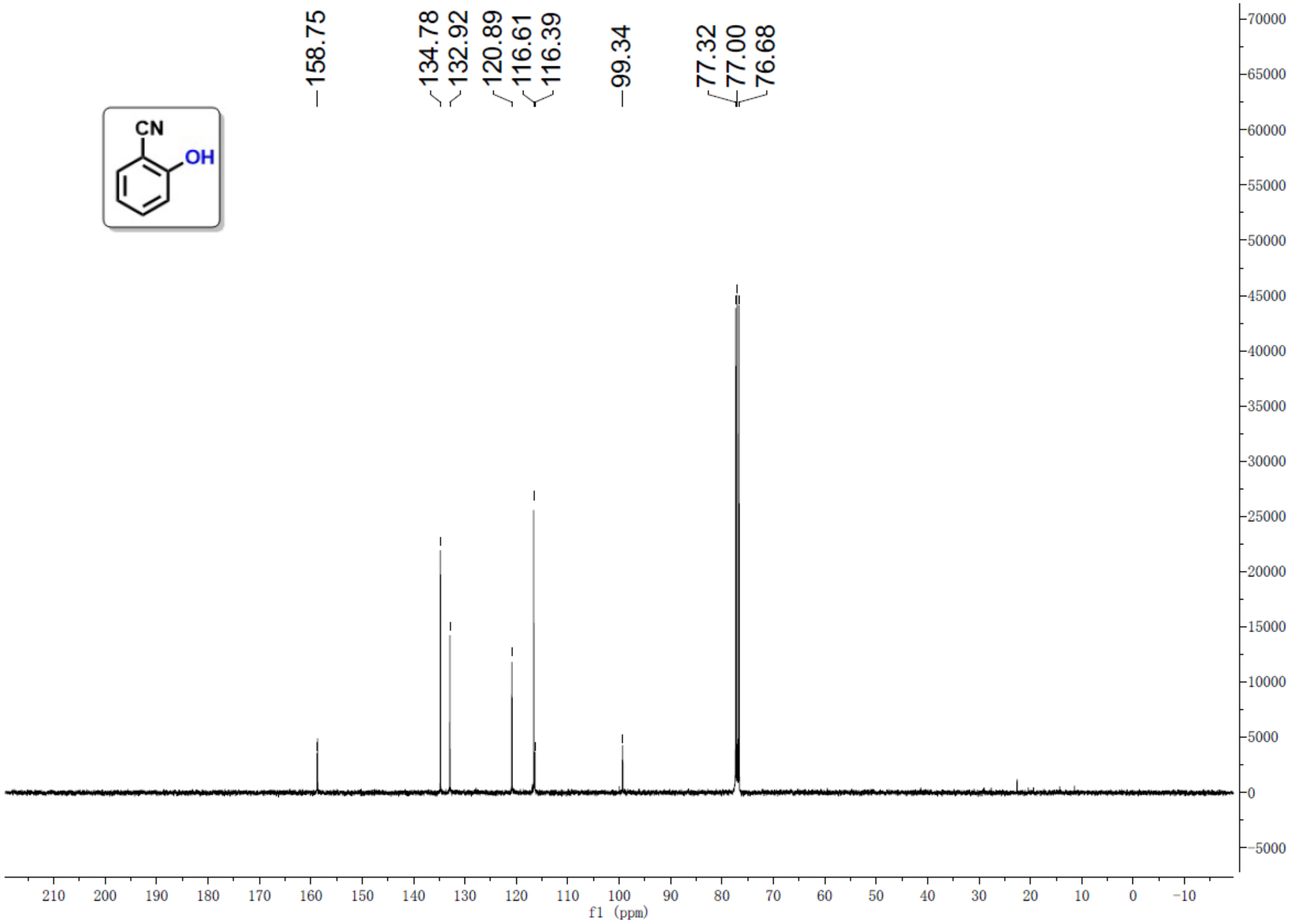


-60.85
CF3
OH
85000
80000
75000
70000
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

^{1}H NMR of 2q

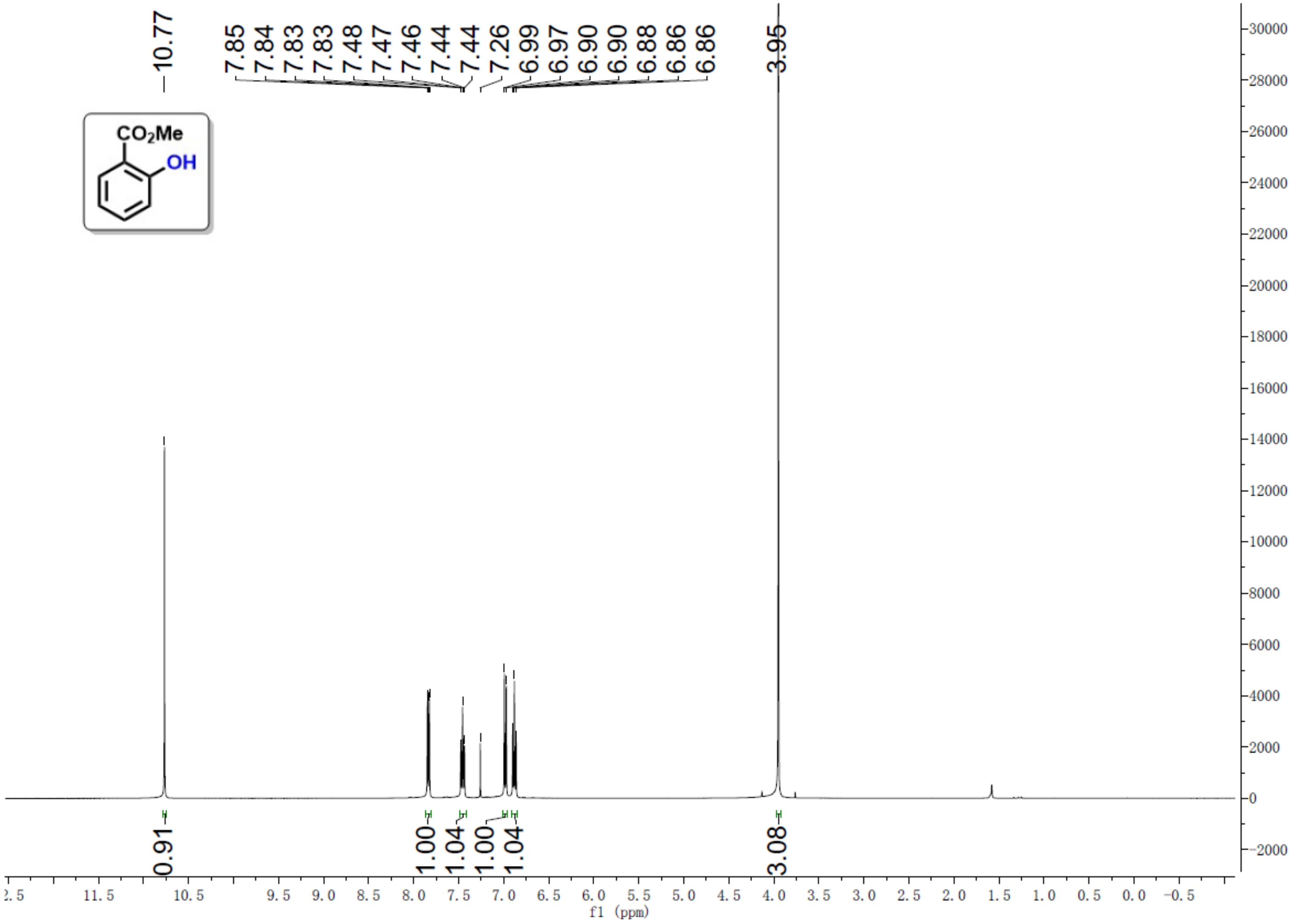

7.52
7.50
7.47
7.45
7.26
7.02
7.01
7.00
6.99
6.97
6.67
CN
OH
2.00
2.01
1.03
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

^{13}C NMR of 2q

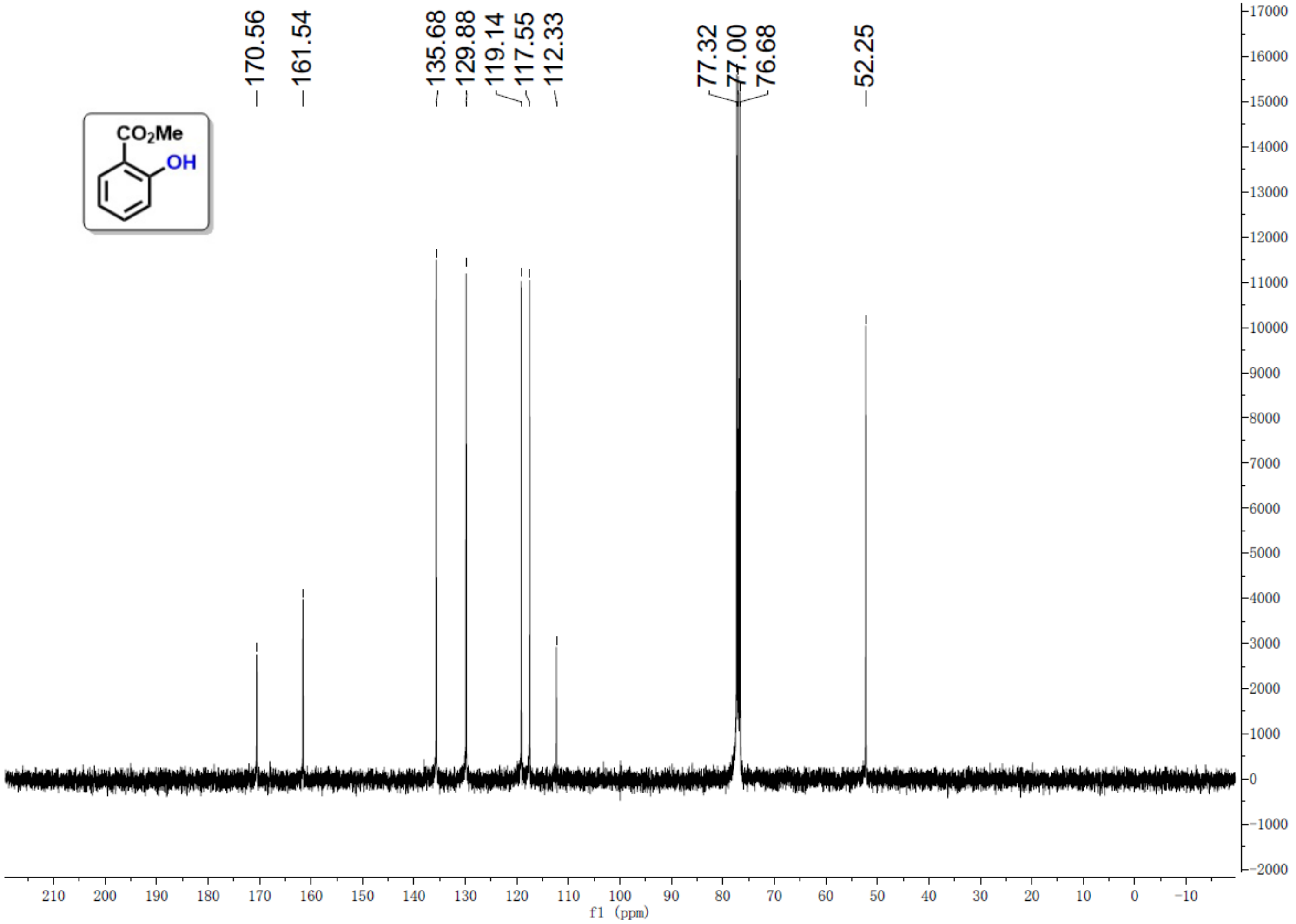


158.75
134.78
132.92
120.89
116.61
116.39
99.34
77.32
77.00
76.68
CN
OH
70000
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

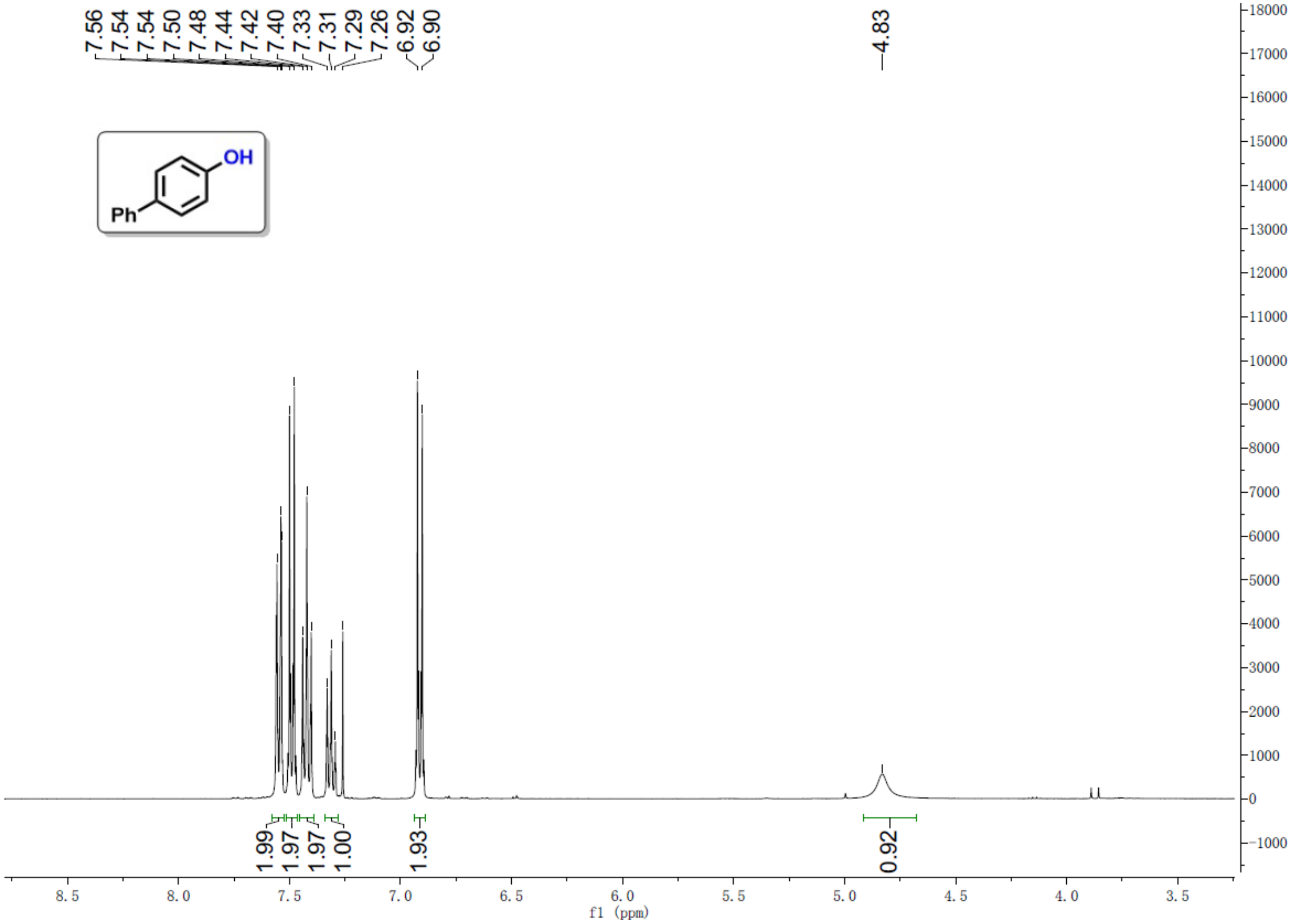
^{1}H NMR of 2r


10.77
7.85
7.84
7.83
7.83
7.48
7.47
7.46
7.44
7.44
7.26
6.99
6.97
6.90
6.90
6.88
6.86
6.86
3.95
CO2Me
OH
0.91
1.00
1.04
1.00
1.04
3.08
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000

^{13}C NMR of 2r


170.56
161.54
135.68
129.88
119.14
117.55
112.33
77.32
77.00
76.68
52.25
CO2Me
OH
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
−1000
−2000
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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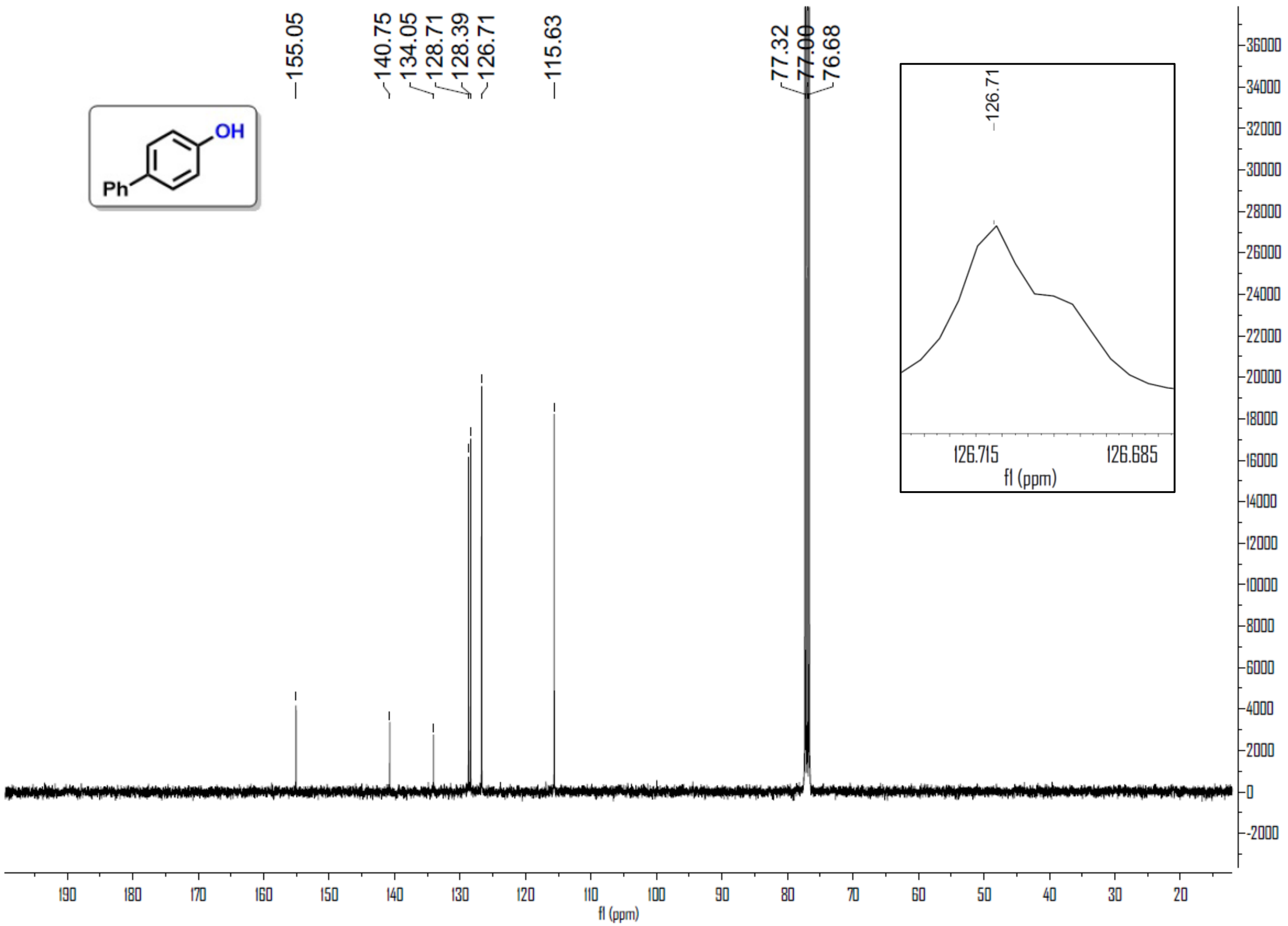
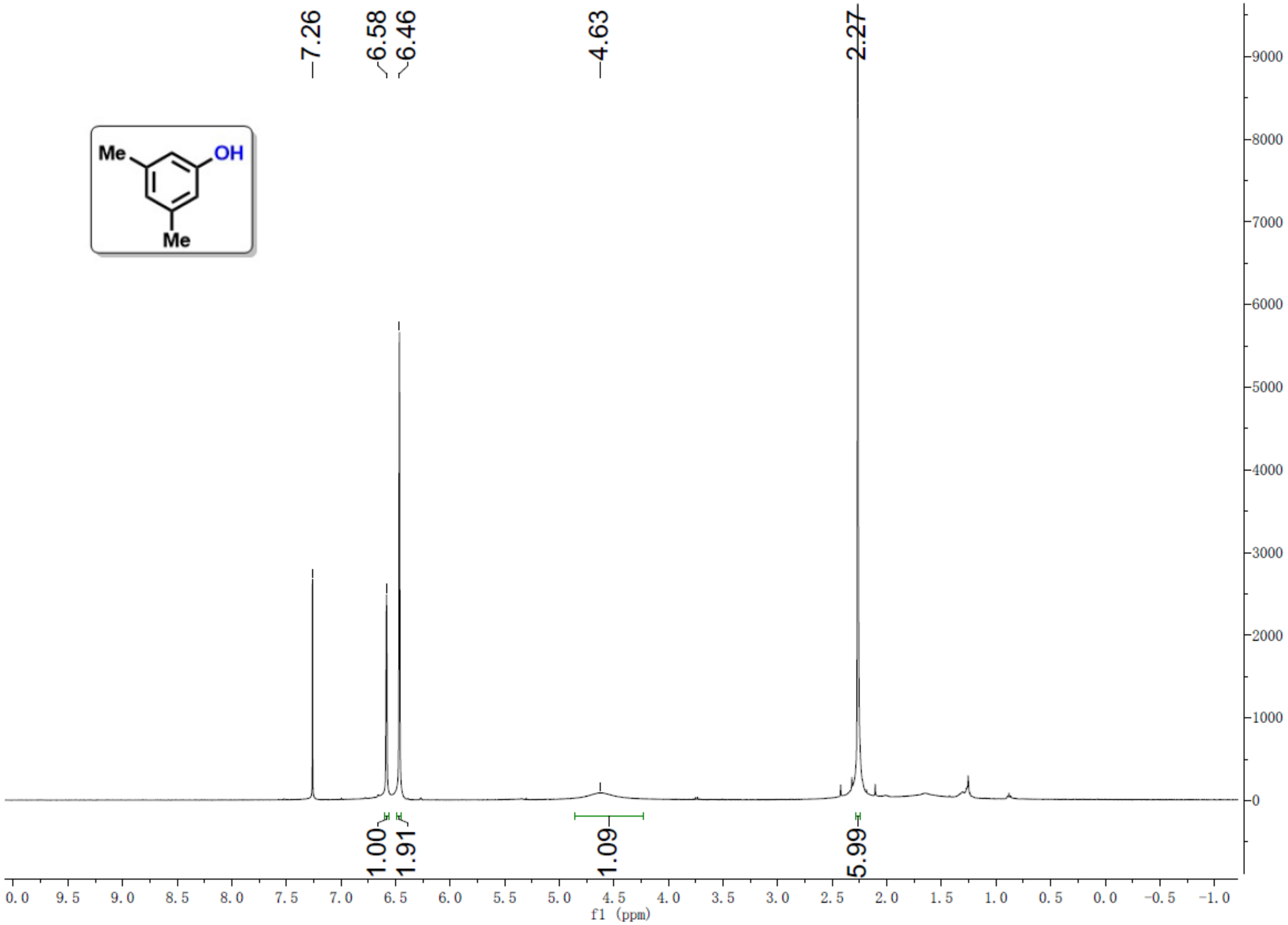
7.54
7.54
7.50
7.48
7.44
7.42
7.40
7.33
7.31
7.29
7.26
6.92
6.90
4.83
OH
Ph
1.99
1.97
1.97
1.00
1.93
0.92
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
f1 (ppm)

^{13}C NMR of 2s

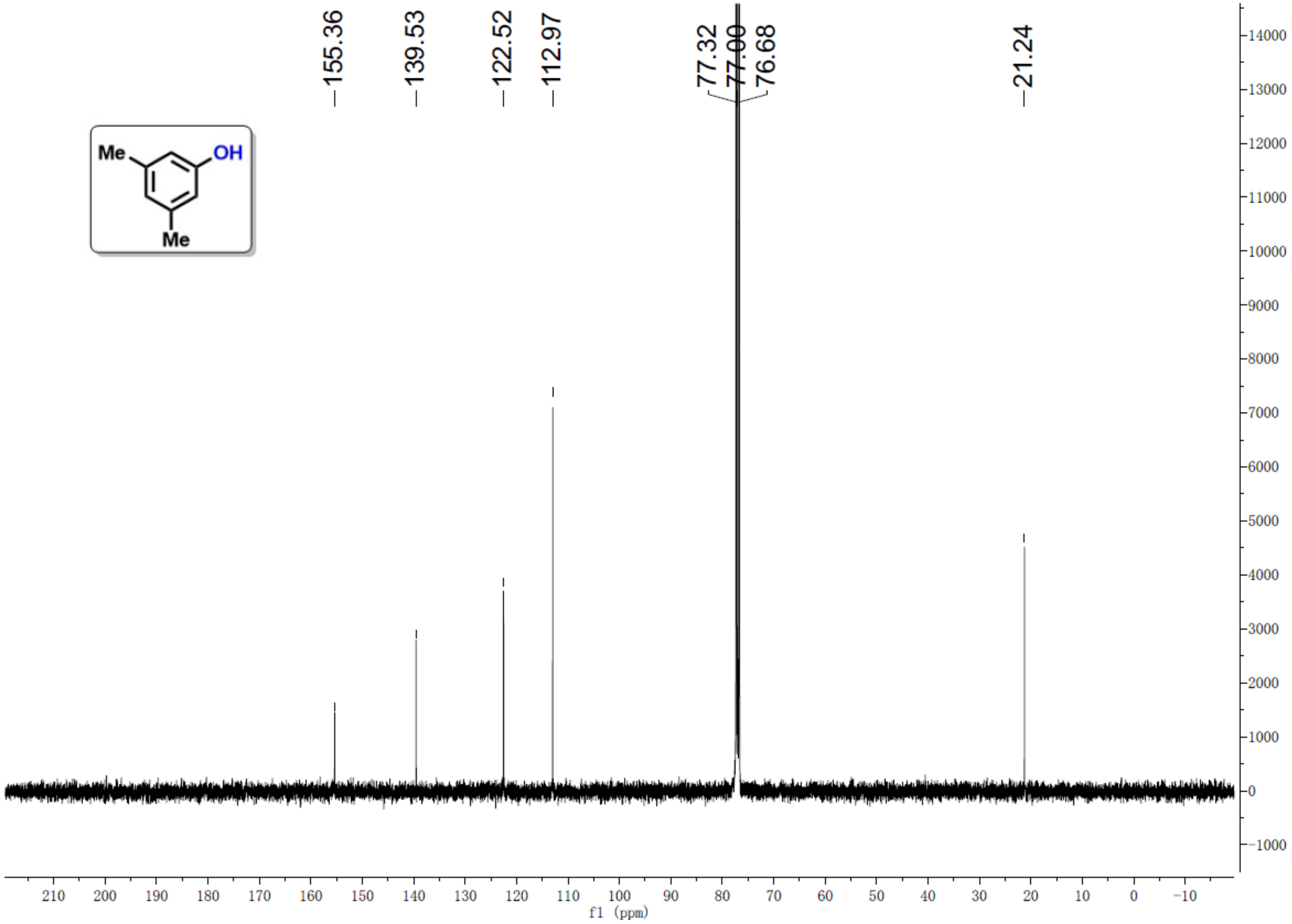

155.05
140.75
134.05
128.71
128.39
126.71
115.63
77.32
77.00
76.68
OH
Ph
126.71
126.715
126.685
fl (ppm)
36000
34000
32000
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
fl (ppm)

^{1}H NMR of 2t



7.26
6.58
6.46
4.63
2.27
Me
OH
Me
1.00
1.91
1.09
5.99
0.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)
9000
8000
7000
6000
5000
4000
3000
2000
1000
0

^{13}C NMR of 2t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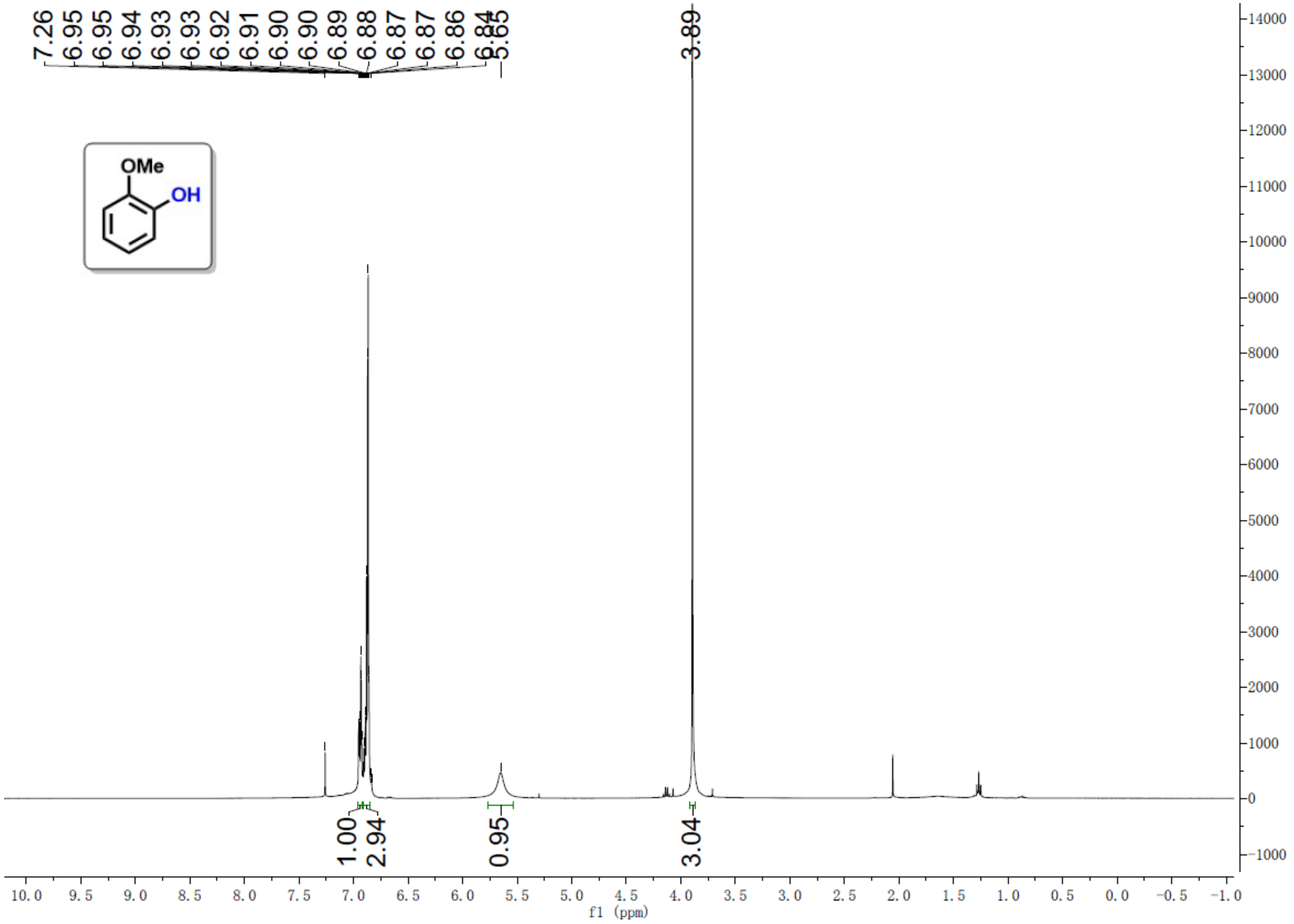
139.53
122.52
112.97
77.32
77.00
76.68
21.24
Me
OH
Me
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

^{1}H NMR of 2u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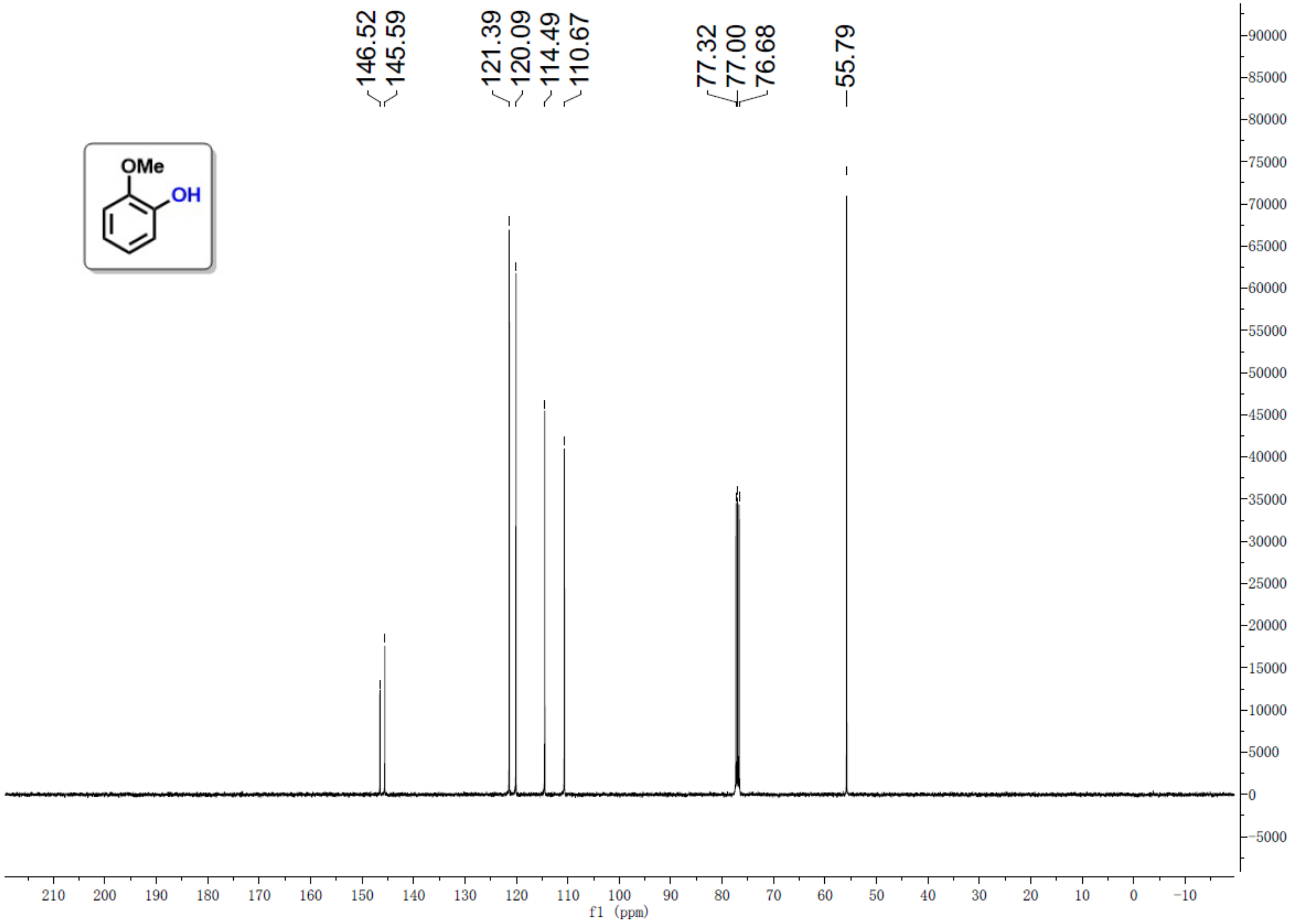
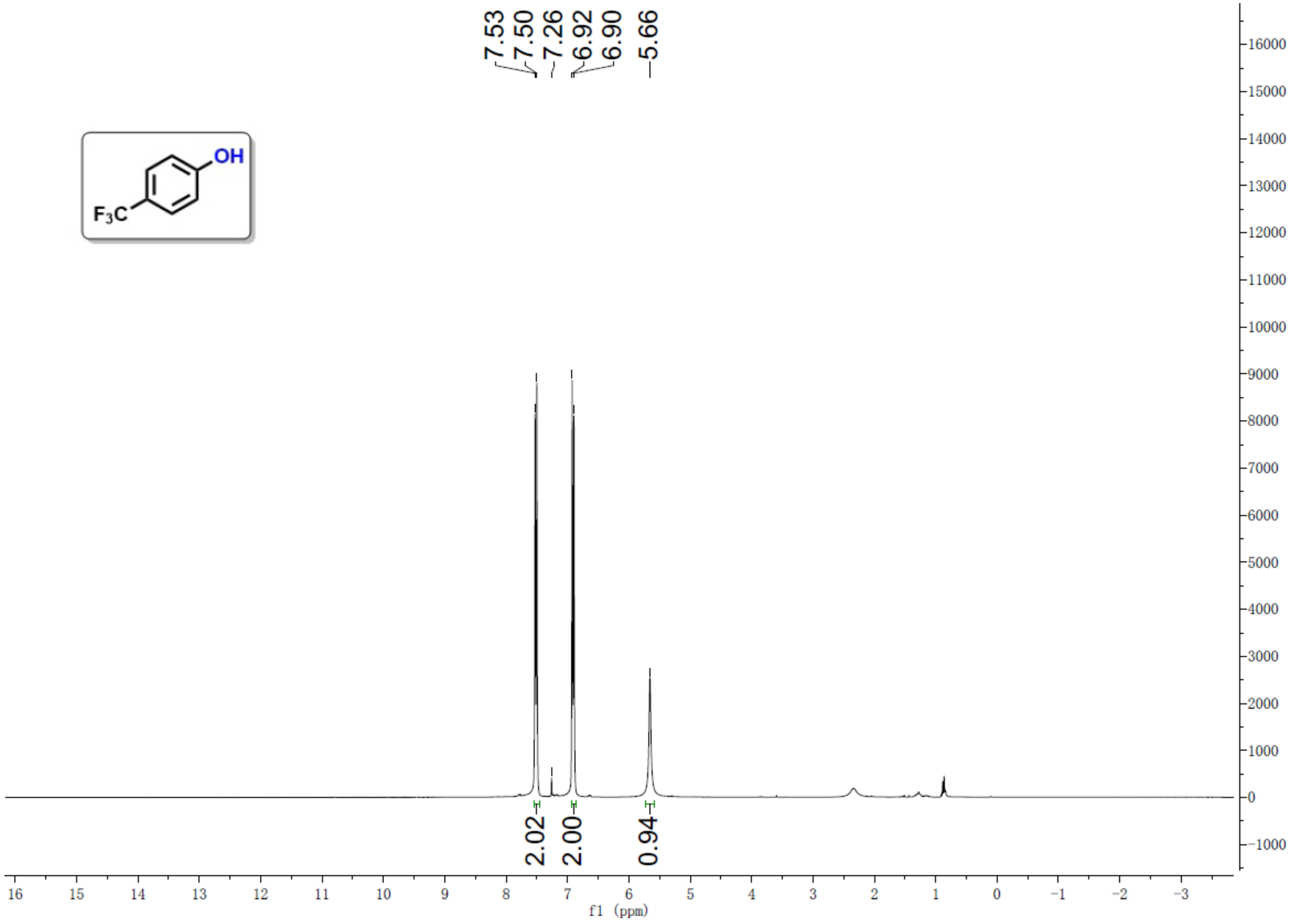
6.95
6.95
6.94
6.93
6.93
6.92
6.91
6.90
6.90
6.89
6.88
6.87
6.87
6.86
6.84
5.65
3.89
OMe
OH
1.00
2.94
0.95
3.04
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000

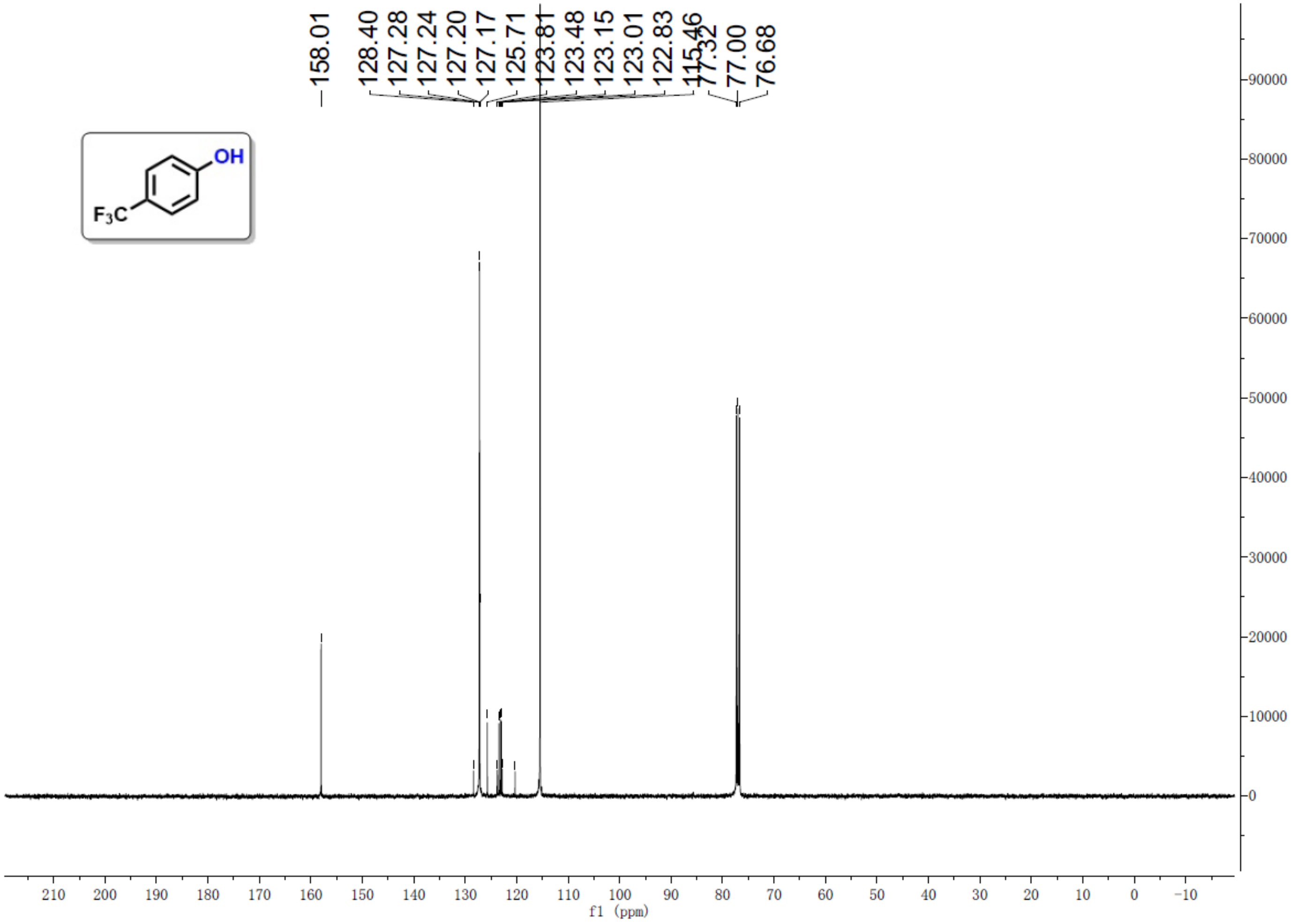
^{13}C NMR of 2u


146.52
145.59
121.39
120.09
114.49
110.67
77.32
77.00
76.68
55.79
OMe
OH
90000
85000
80000
75000
70000
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
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

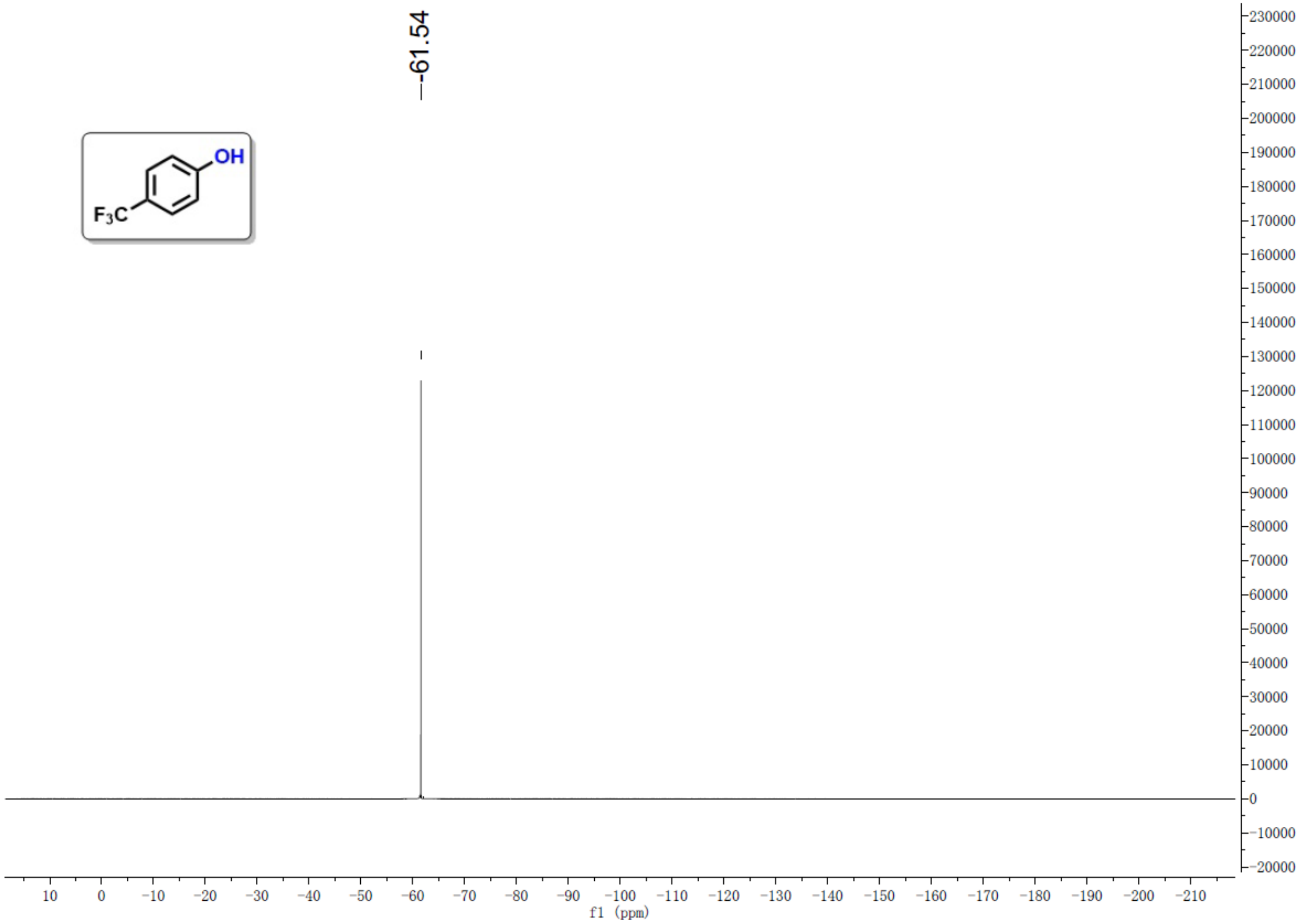

7.53
7.50
7.26
6.92
6.90
5.66
OH
F_3C
2.02
2.00
0.94
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)

^{13}C NMR of 2v



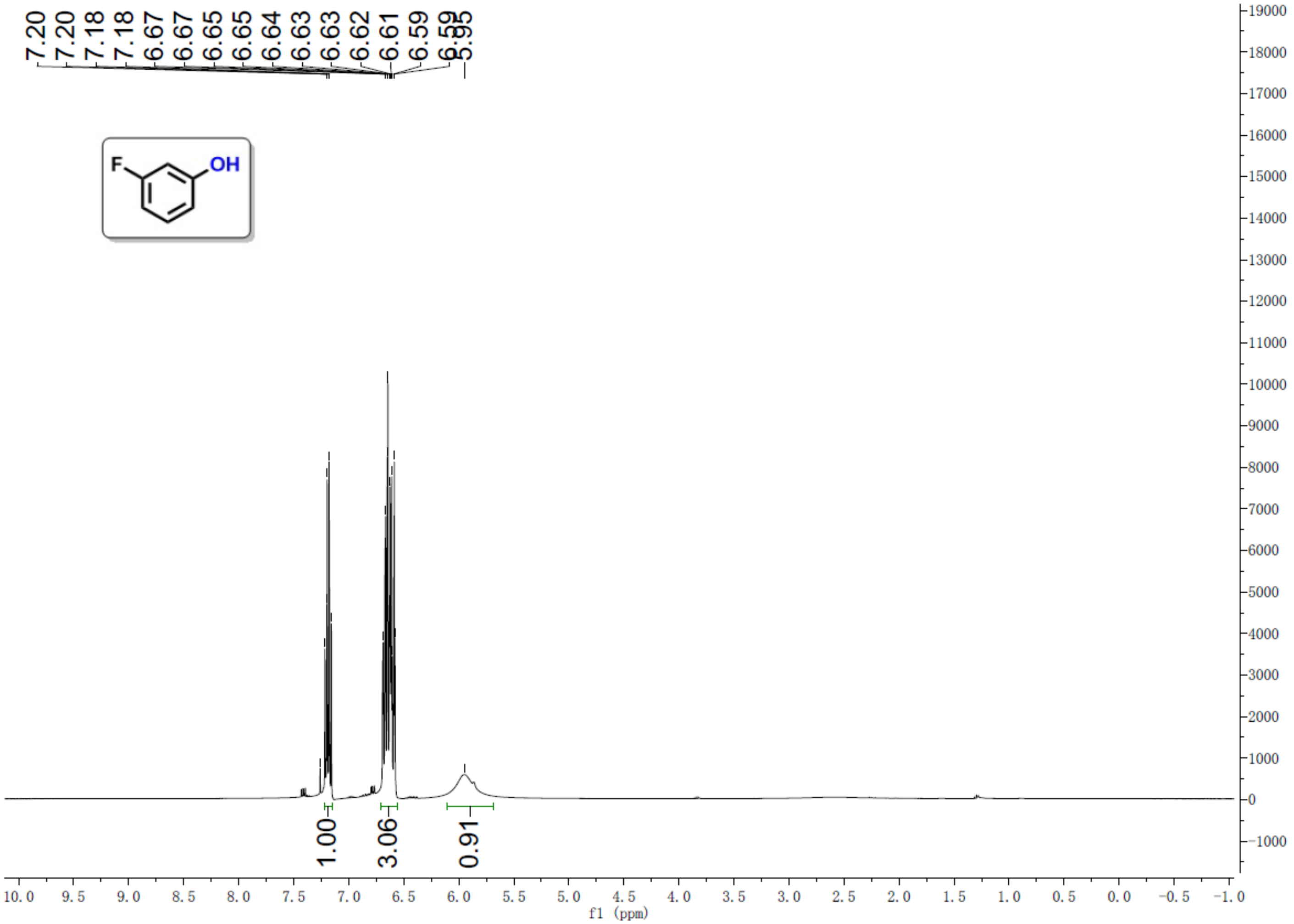
158.01
128.40
127.28
127.24
127.20
127.17
125.71
123.81
123.48
123.15
123.01
122.83
115.46
77.32
77.00
76.68
OH
F3C
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

^{19}F NMR of 2v

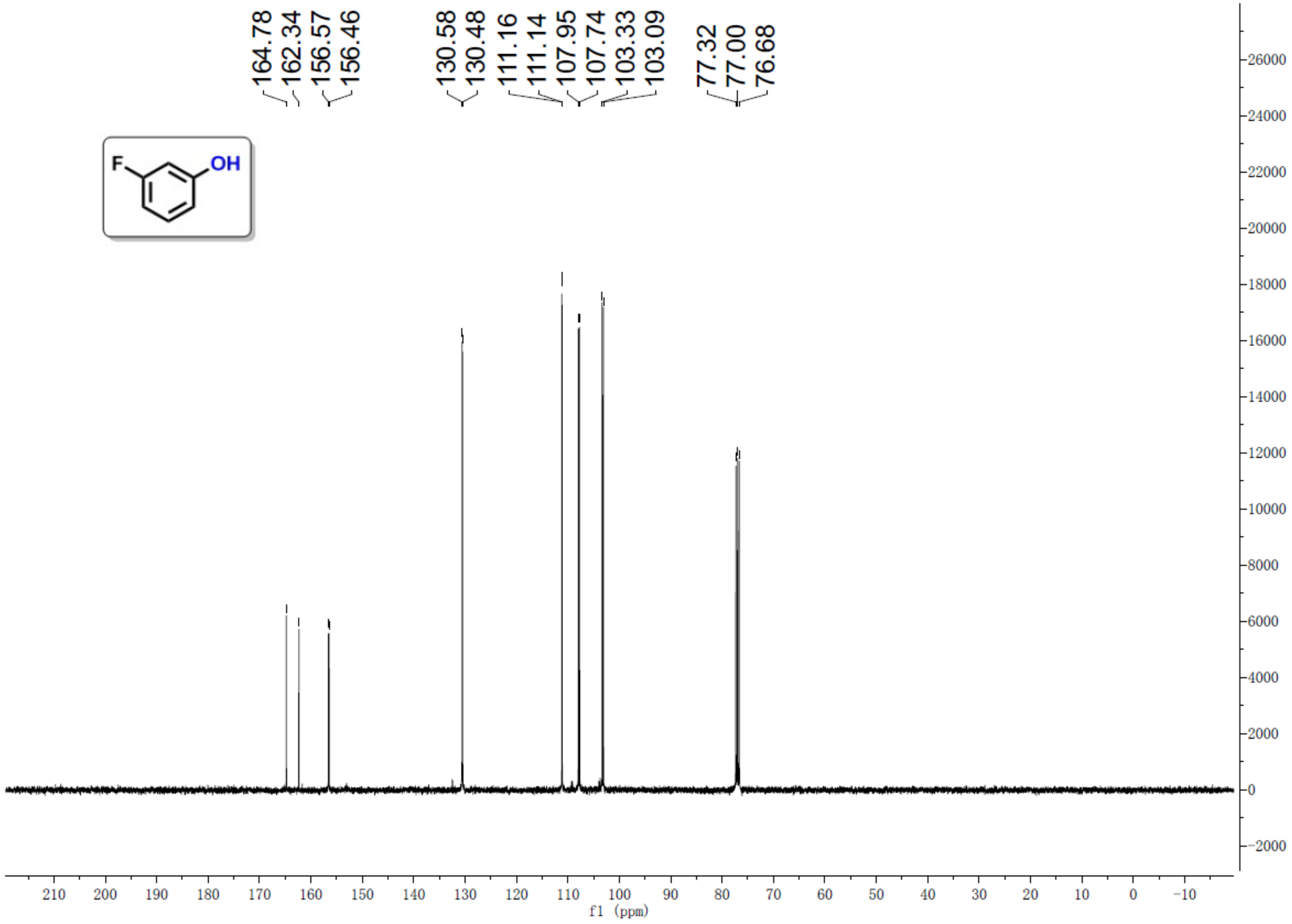


-61.54
OH
F3C
230000
220000
210000
200000
190000
180000
170000
160000
150000
140000
130000
120000
110000
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
-20000
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

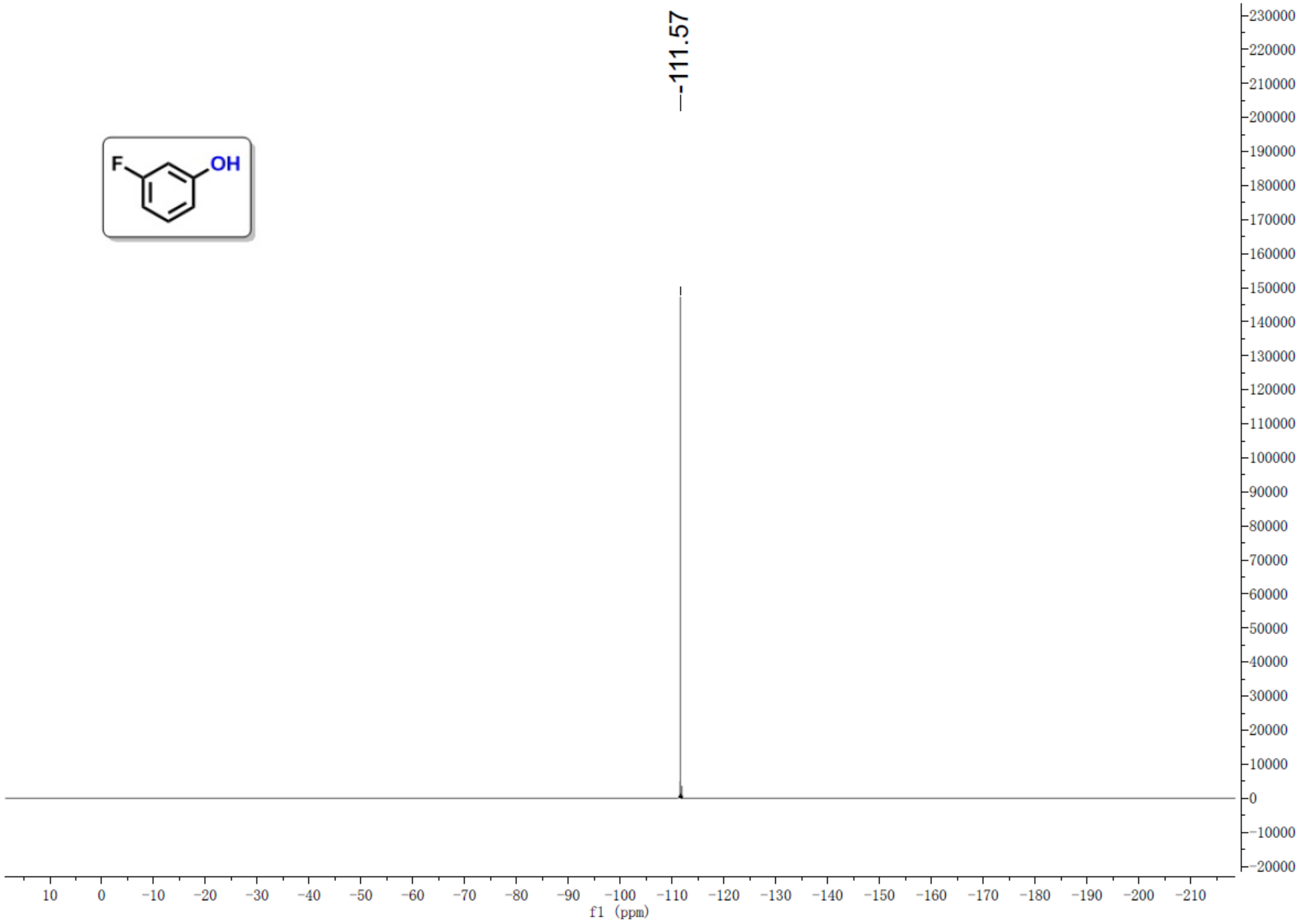
^{1}H NMR of 2w


7.20
7.20
7.18
7.18
6.67
6.67
6.65
6.65
6.64
6.63
6.63
6.62
6.61
6.59
6.59
5.95
F
OH
1.00
3.06
0.91
f1 (ppm)

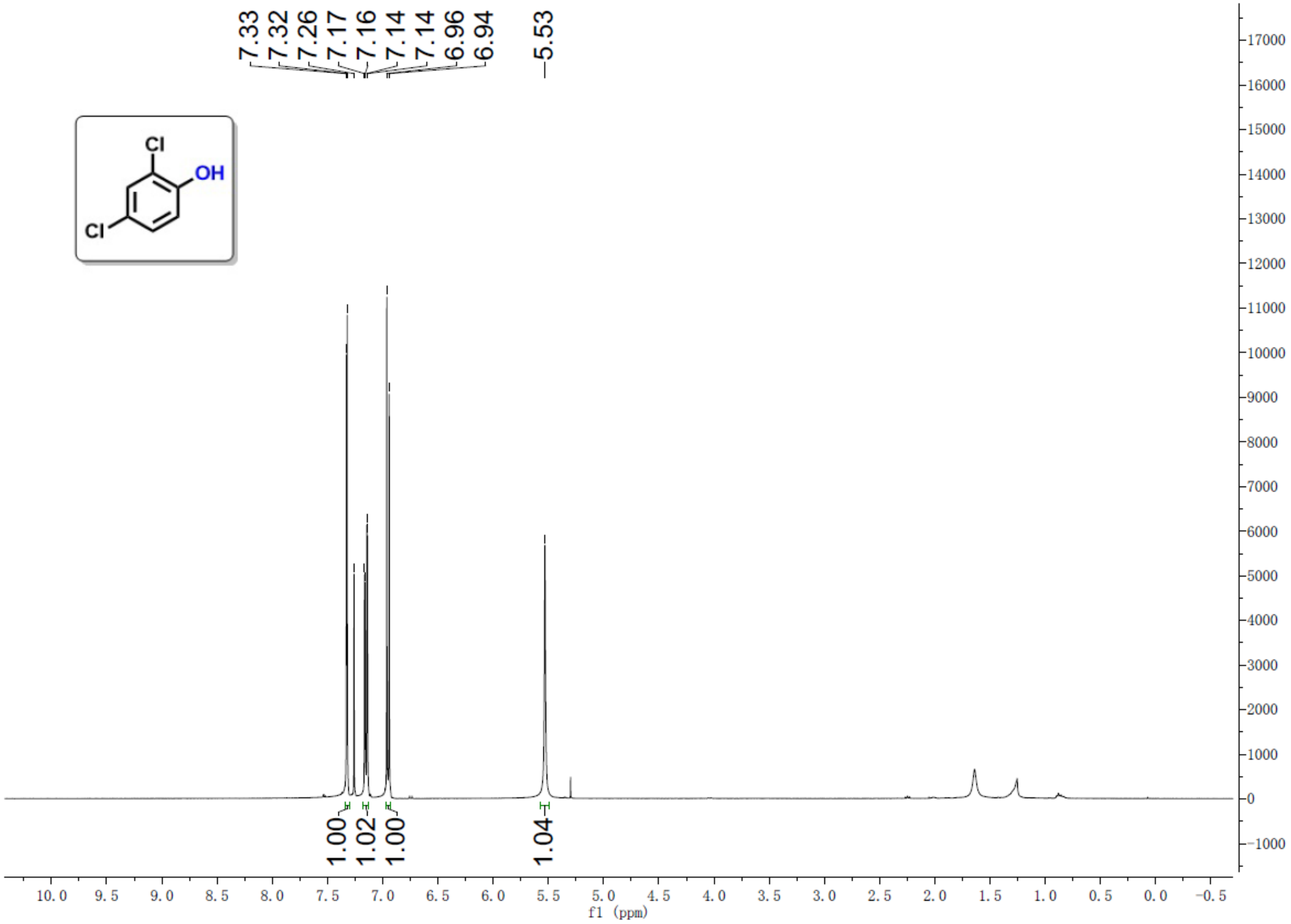
^{13}C NMR of 2w


164.78
162.34
156.57
156.46
130.58
130.48
111.16
111.14
107.95
107.74
103.33
103.09
77.32
77.00
76.68
F
OH
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
−2000
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)

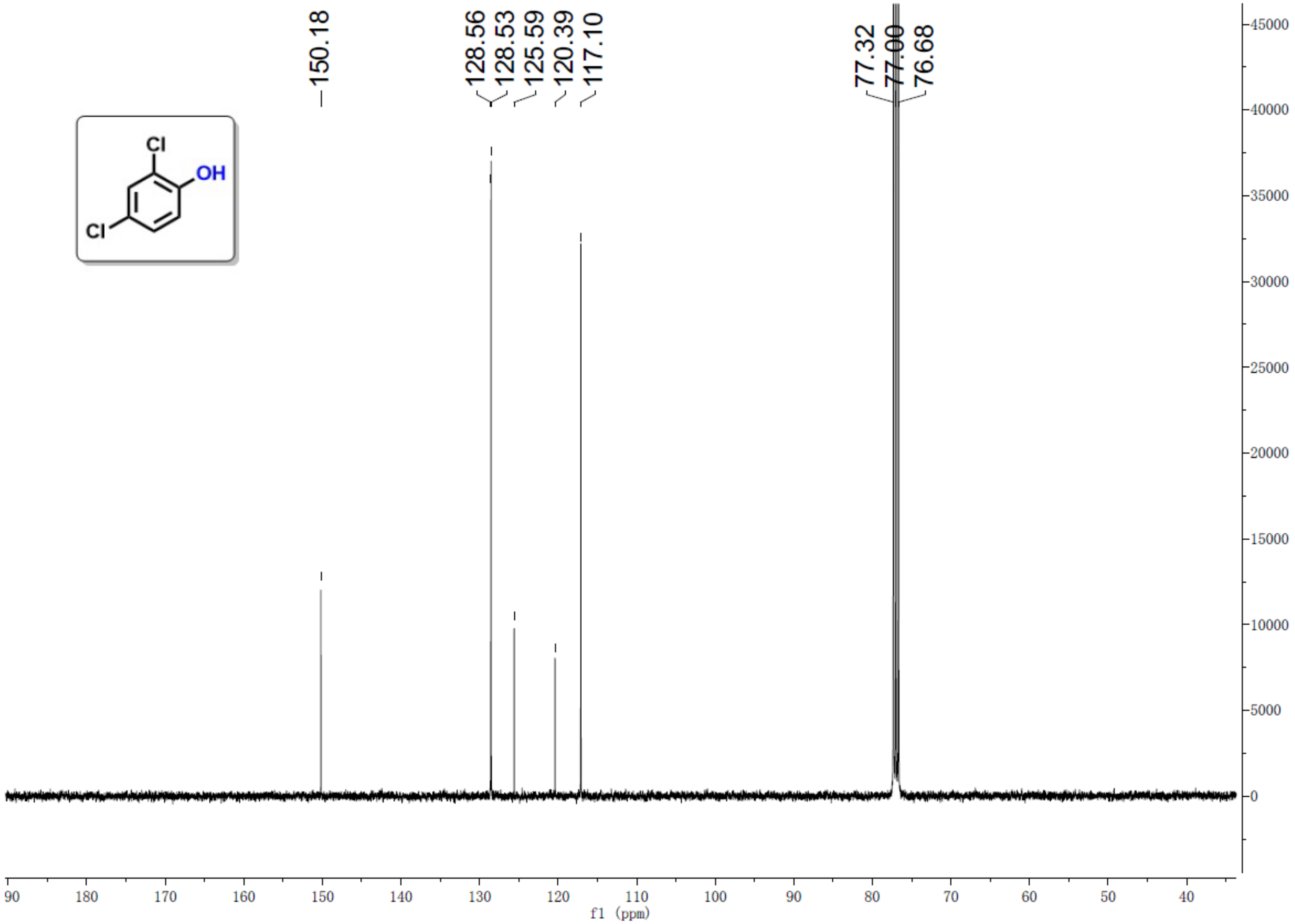
^{19}F NMR of 2w


-111.57
F
OH
f1 (ppm)
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
230000 220000 210000 200000 190000 180000 170000 160000 150000 140000 130000 120000 110000 100000 90000 80000 70000 60000 50000 40000 30000 20000 10000 0 -10000 -20000

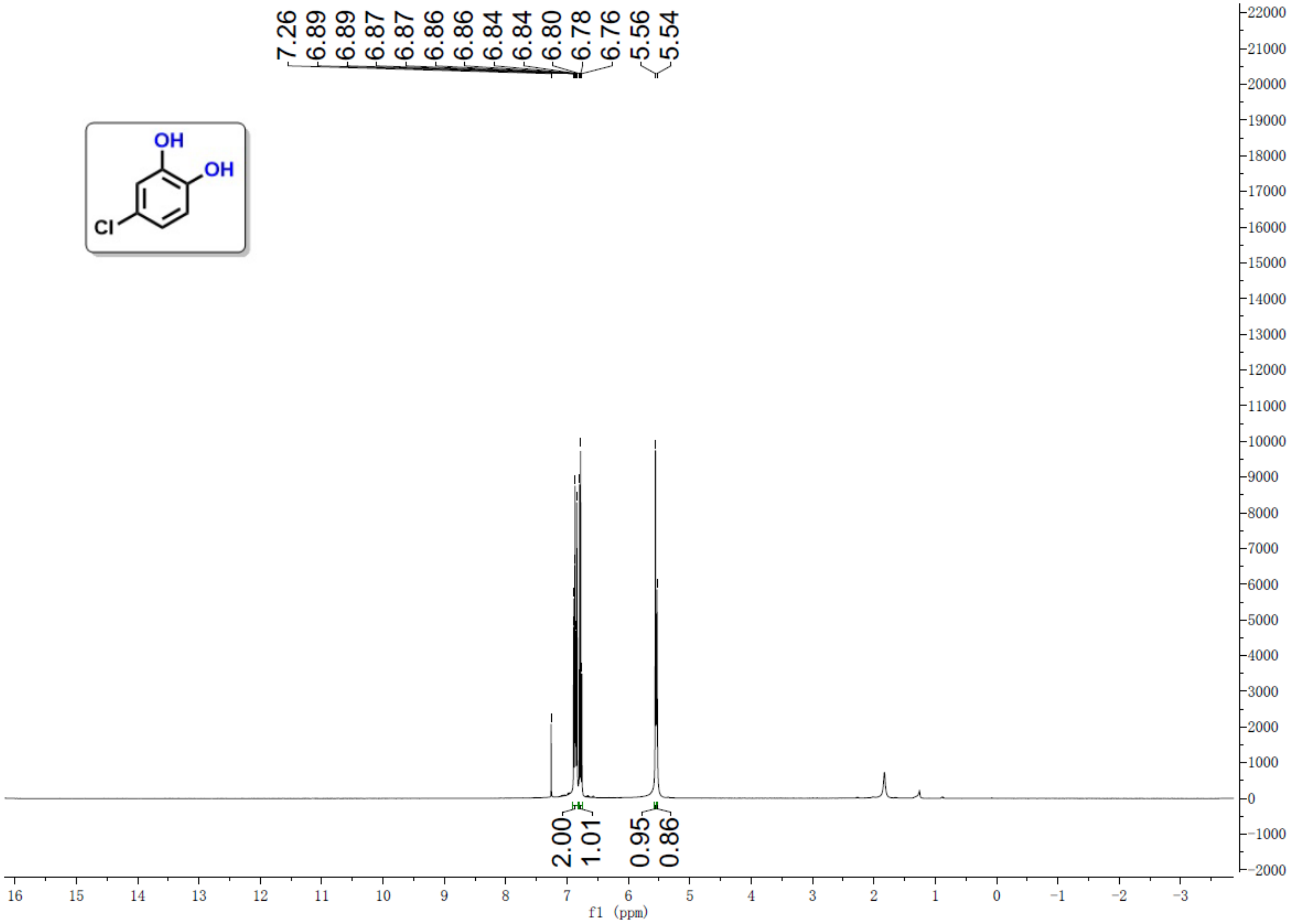
^{1}H NMR of 2x


7.33
7.32
7.26
7.17
7.16
7.14
7.14
6.96
6.94
5.53
Cl
OH
Cl
1.00
1.02
1.00
1.04
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000

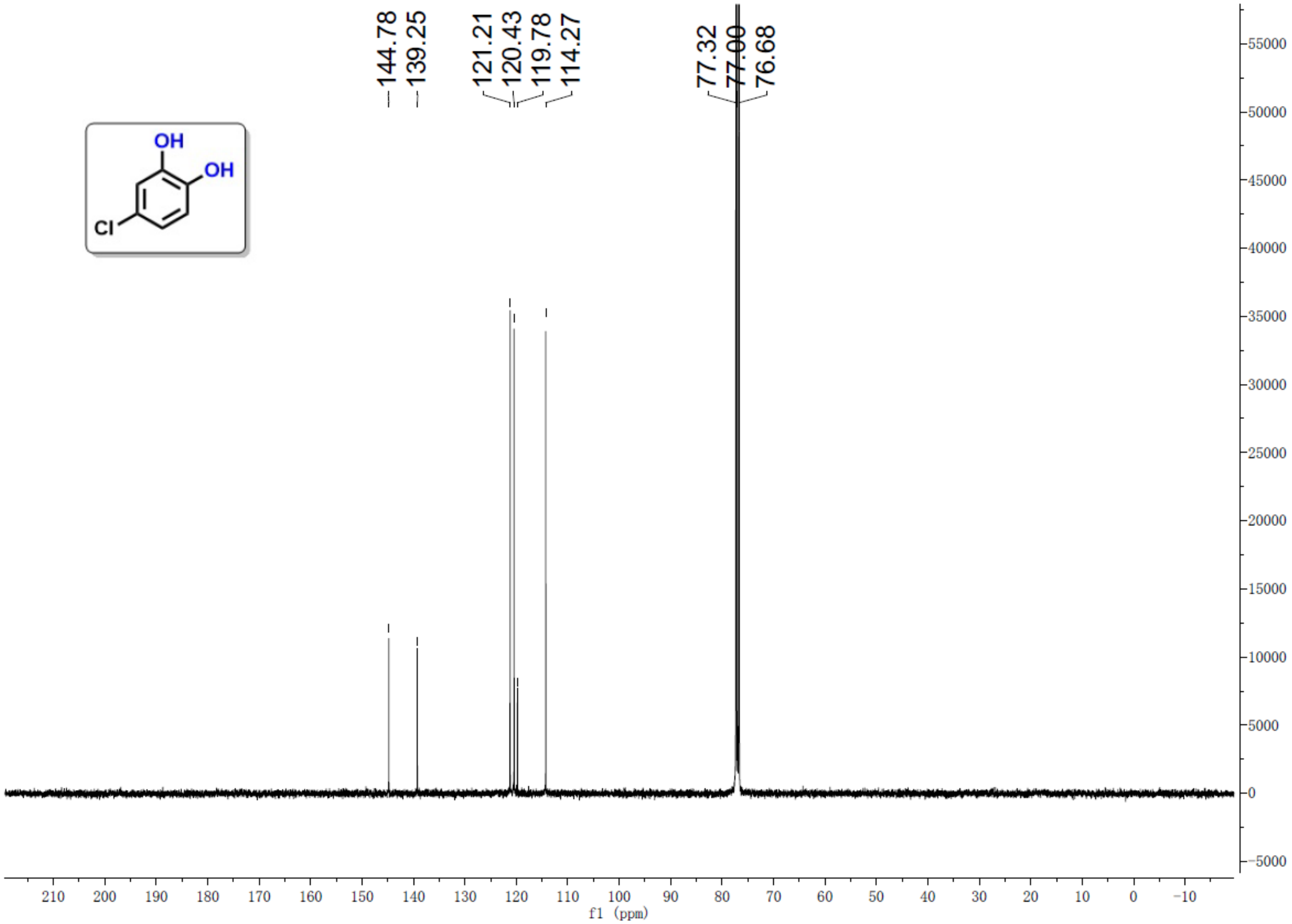
^{13}C NMR of 2x


150.18
128.56
128.53
125.59
120.39
117.10
77.32
77.00
76.68
Cl
OH
Cl
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
90
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
f1 (ppm)

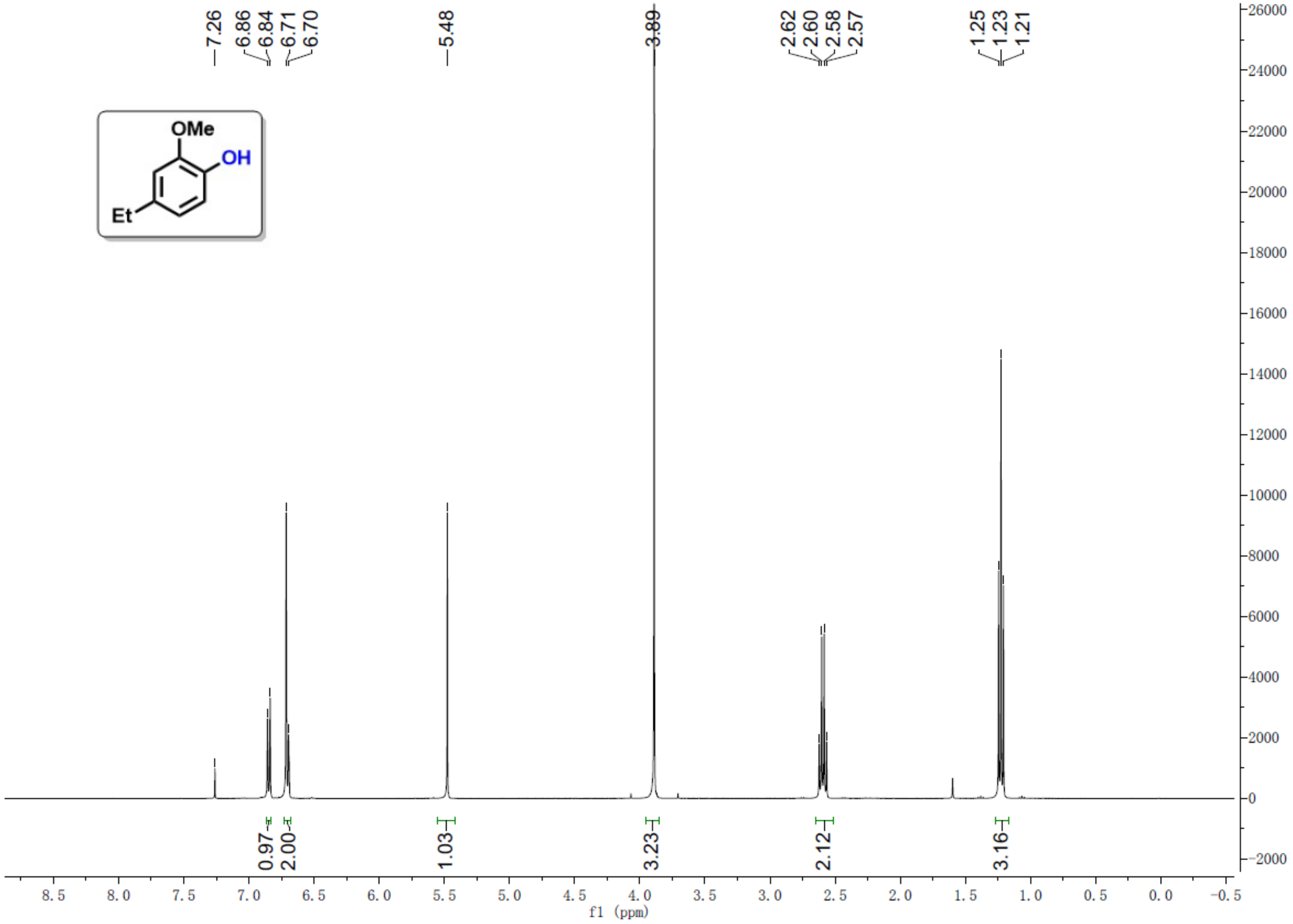
^{1}H NMR of 2y


7.26
6.89
6.89
6.87
6.87
6.86
6.86
6.84
6.84
6.80
6.78
6.76
5.56
5.54
OH
OH
Cl
2.00
1.01
0.95
0.86
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
−1
−2
−3
f1 (ppm)
22000
21000
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
−1000
−2000

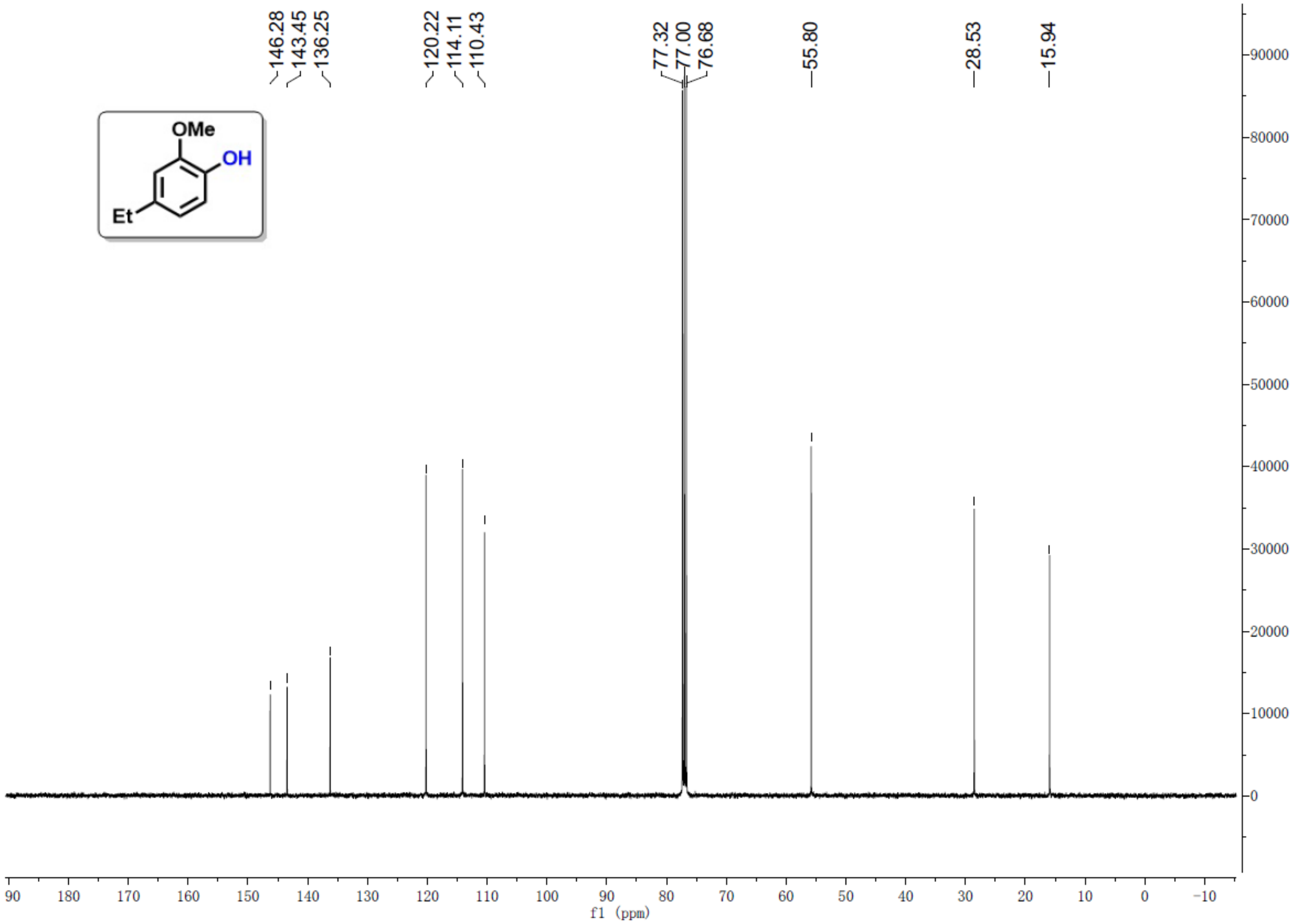
^{13}C NMR of 2y


144.78
139.25
121.21
120.43
119.78
114.27
77.32
77.00
76.68
OH
OH
Cl
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
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)

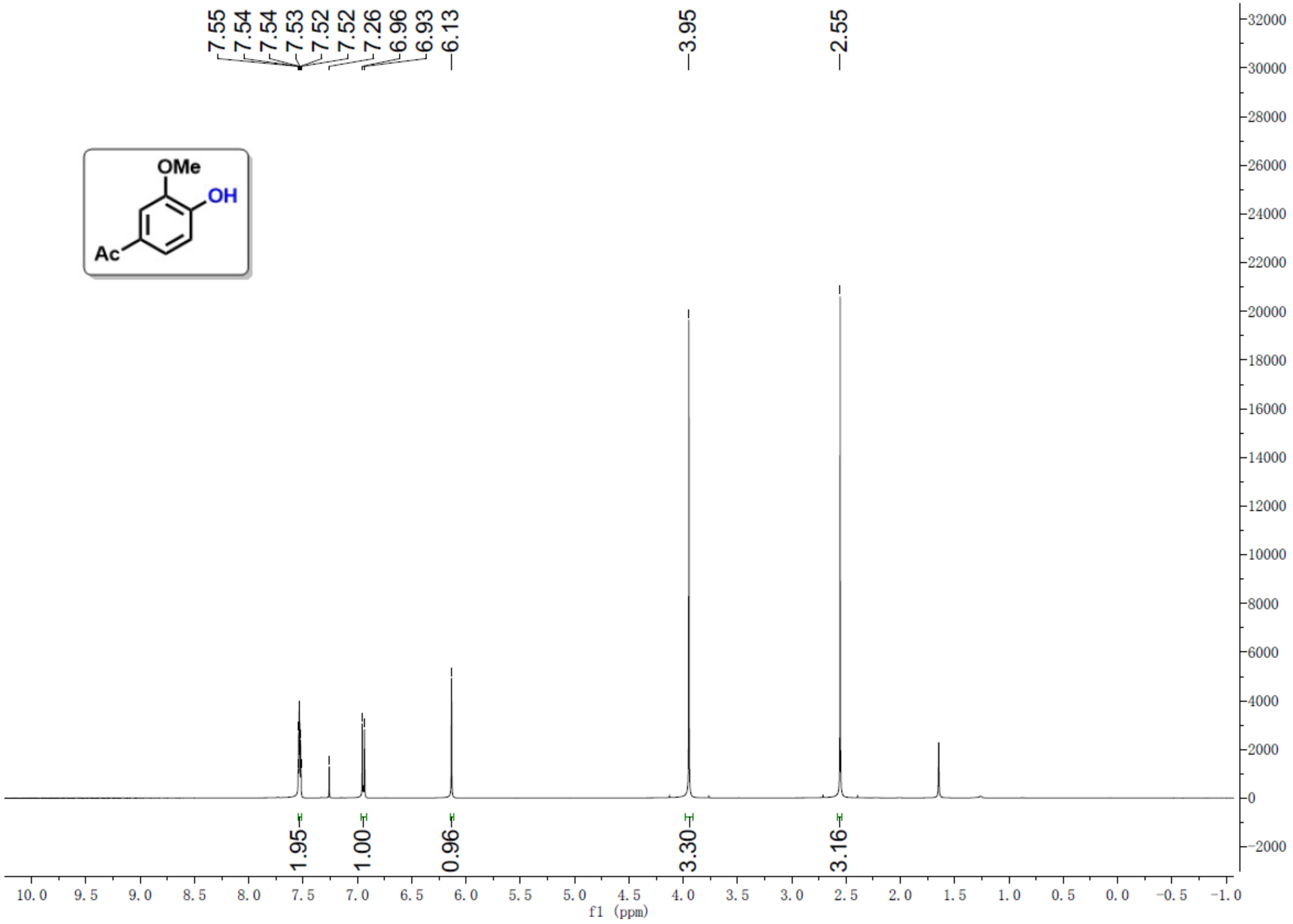
^{1}H NMR of 2z1


7.26
6.86
6.84
6.71
6.70
5.48
3.89
2.62
2.60
2.58
2.57
1.25
1.23
1.21
OMe
OH
Et
0.97
2.00
1.03
3.23
2.12
3.16
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

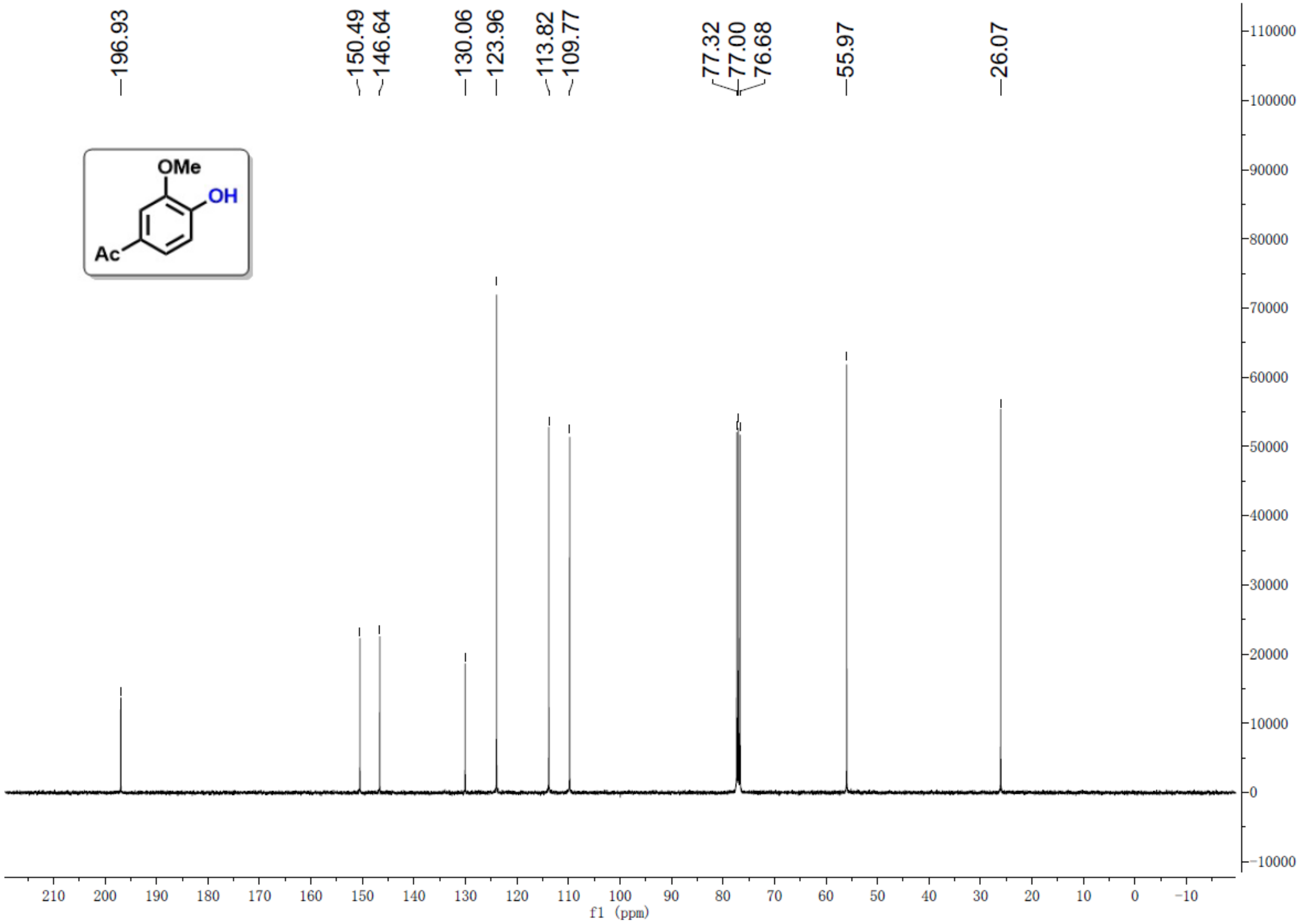
^{13}C NMR of 2z1



^{1}H NMR of 2z2


7.55
7.54
7.54
7.53
7.52
7.52
7.26
6.96
6.93
6.13
3.95
2.55
OMe
OH
Ac
1.95
1.00
0.96
3.30
3.16
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)
32000
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000

^{13}C NMR of 2z2



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