

Electroreductive Carboxylation of Benzylphosphine Salts with CO₂ through the Cleavage of the C(sp³)-P Bond

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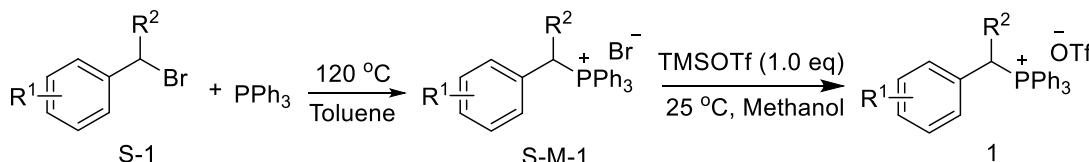
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1. General Information

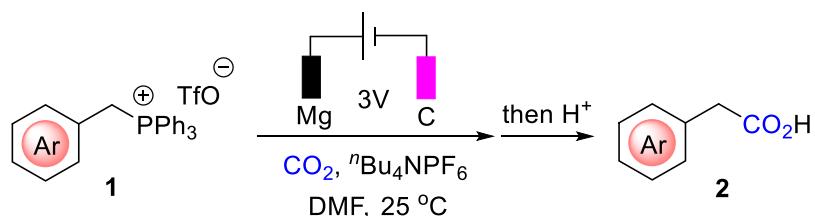
All the electrochemical reactions were performed in an oven-dried undivided electrochemical cell unless otherwise noted. The amount of electricity consumed per mole of reactant is measured by an ampere hour meter. THF was distilled from sodium/benzophenone. Anhydrous DMF, DMA, NMP and DMSO were purchased from Adamas-beta and used without further purification. All commercial reagents were purchased from Acros Organics, Sigma-Aldrich, Alfa Aesar, Adamas-beta, Bidepharm, and Energy Chemical of the highest purity grade. ^1H NMR and ^{13}C NMR spectra were recorded respectively at 400 MHz and 100 MHz on a Bruker AVANCE 400 and chemical shifts are reported in δ (ppm) referenced to residual undeuterated solvent signal for ^1H NMR (7.26 ppm for CDCl_3 or 2.54 ppm for $\text{DMSO}-d_6$), ^{13}C NMR (77.16 ppm for CDCl_3 or 40.45 ppm for $\text{DMSO}-d_6$). HRMS was conducted on a Thermo Scientific LTQ Orbitrap XL apparatus using an electrospray (ESI) or MALDI ionization source. The following abbreviations were used to designate chemical shift multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. All the electrochemical reactions were performed with an HY3005B DC (30 V) power supply purchased from Zhejiang Huayi Electronic Industry Co., Ltd. Carbon rod and graphite felt (GF) was purchased from Beijing Jinglong special carbon technology Co., Ltd. Platinum electrode, Al electrode, and Zn electrode are all purchased from Shanghai yueci Electronic Technology Co., Ltd. Magnesium plate (The purity is 99.95%) was purchased from Xintong Weiye Metal Material Sales Co., Ltd.

2. General procedure for the synthesis of 1.



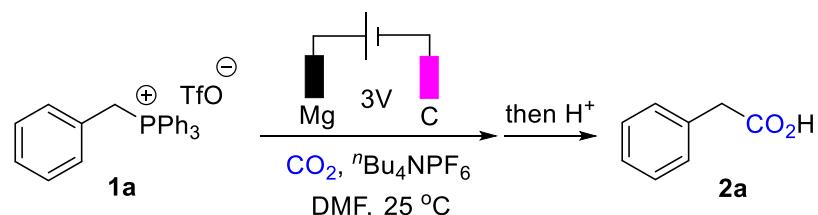
To a solution of substituted benzyl bromide **S-1** (3.4 g, 20.0 mmol) in toluene (20 mL) was added triphenylphosphine (5.2 g, 20.0 mmol). The solution was refluxed for 16 h to form a White suspension. After filtration at room temperature, the White powder was washed with cyclohexane and dried under vaccuo to give the pure phosphonium bromide salt **S-M-1**¹. To a solution of phosphonium bromide salt **S-M-1** (2.2 g, 5.0 mmol) in methanol (10 mL) was added trimethylsilyl trifluoromethanesulfonate (TMSOTf, 0.9 mL, 5.0 mmol). The solution was stirred for 4 hours, and concentrated under reduced pressure. Dissolve the crude product in a small amount of acetone and add ether for recrystallization to give the corresponding phosphonium trifluoromethanesulfonate salt **1**.

3. General procedure for the synthesis of aryl acetic acids 2.



The electrolysis was conducted in an undivided cell equipped with a Mg anode (15 mm x 10 mm x 2 mm) and a C cathode ($\varphi = 6$ mm) with a distance of about 1.0 cm between the two electrodes, and the depth of each electrode immersed in the reaction mixture is about 10 mm. To a 15 mL oven-dried undivided electrochemical cell were added benzyl triphenyl phosphonium salt **1** (0.2 mmol), $^n\text{Bu}_4\text{NPF}_6$ (193.7 mg, 0.5 mmol) and followed by the addition of solvent DMF (5 mL, 0.1 M) in the glove box. Then, seal the tube and remove it from the glove box. After bubbling CO_2 gas into the mixture for 10 min, the electrolysis was carried out with constant voltage electrolysis (cell voltage = 5 V) at 25 °C for 7 h under a CO_2 balloon. The resulting mixture is transferred to a separator funnel for preparing the reaction solution at the beginning. The cell was washed with ethyl acetate (2 mL x 2) twice. The combined organic solution was acidified ($\text{pH} < 2$) by the addition of 1 M HCl and extracted with ethyl acetate (5 x 3 mL). The combined organic layers were washed with water, brine, dried over anhydrous Na_2SO_4 , filtered, and concentrated in vacuo. The residue was purified by flash chromatography (PE/EA) to afford aryl acetic acids **2**.

4. Experiment details for gram-scale synthesis of product



The electrolysis was conducted in an undivided cell equipped with a Mg anode (50 mm x 30 mm x 2 mm) and a C cathode ($\varphi = 6$ mm) with a distance of about 5.0 cm between the two electrodes, and the depth of each electrode immersed in the reaction mixture is about 20 mm. To a 250 mL oven-dried undivided electrochemical cell were added benzyltriphenylphosphonium trifluoromethanesulfonate **1a** (2.1 g, 4.0 mmol), $^n\text{Bu}_4\text{NPF}_6$ (3.9 g, 10.0 mmol) and followed by the addition of solvent DMF (100 mL, 0.1 M) in the glove box. Then, seal the tube and remove it from the glove box. After bubbling CO_2 gas into the mixture for 10 min, the electrolysis was carried out with constant voltage electrolysis (cell voltage = 5 V) at 25 °C for 7 h under a CO_2 balloon. The resulting mixture is transferred to a separator funnel for preparing the reaction solution at the beginning. The cell was washed with ethyl acetate (5 mL x 2) twice. The combined organic solution was acidified ($\text{pH} < 2$) by addition of 1 M HCl and extracted with ethyl acetate (30 x 3 mL). The combined organic layers were washed with water, brine, dried over anhydrous Na_2SO_4 , filtered and concentrated in vacuo. The residue was purified by flash chromatography (PE/EA) to afford aryl acetic acids **2a**.

5. Procedure of controlled experiments.

5.1 Radical trapping experiment with TEMPO.

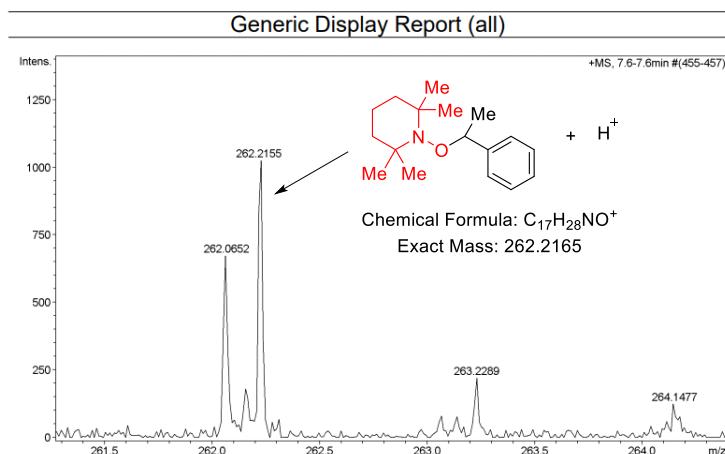
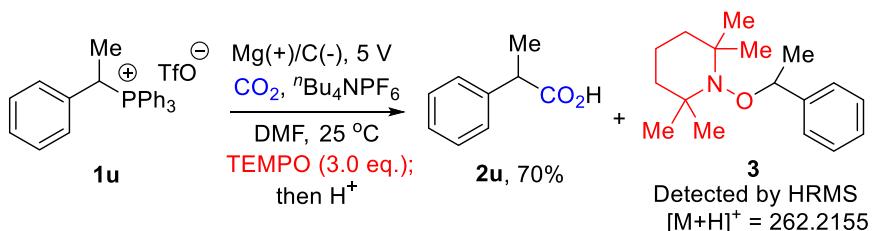
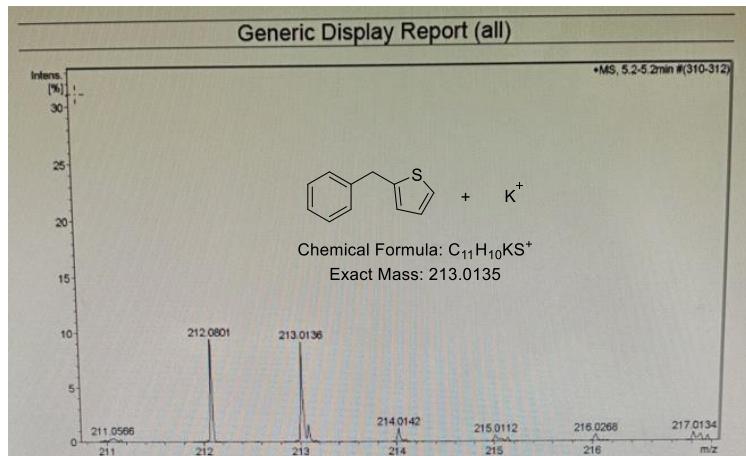
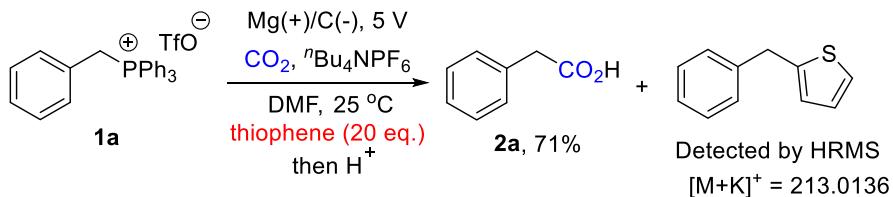


Figure S1 The HRMS spectra of compound 3.

The electrolysis was conducted in an undivided cell equipped with a Mg anode (15 mm x 10 mm x 2 mm) and a C cathode ($\varphi = 6$ mm) with a distance of about 1.0 cm between the two electrodes, and the depth of each electrode immersed in the reaction mixture is about 10 mm. To a 15 mL oven-dried undivided electrochemical cell were added triphenyl(1-phenylethyl)phosphonium trifluoromethanesulfonate **1u** (0.2 mmol), Bu_4NPF_6 (193.7 mg, 0.5 mmol), TEMPO (3.0 equiv.), and followed by the addition of solvent DMF (5 mL, 0.1 M) in the glove box. Then, seal the tube and remove it from the glove box. After bubbling CO_2 gas into the mixture for 10 min, the electrolysis was carried out with constant voltage electrolysis (cell voltage = 5V) at 25 °C for 7 h under a CO_2 balloon. The resulting mixture is transferred to a separator funnel for preparing the reaction solution at the beginning. The cell was washed with ethyl acetate (2 mL x 2) twice. The combined organic solution was acidified ($pH < 2$) by addition of 1 M HCl and extracted with ethyl acetate (5 x 3 mL). The combined organic layers were washed with water, brine, dried over anhydrous Na_2SO_4 , filtered and concentrated in vacuo to achieve the crude residue. The crude yield was 70% which was determined by 1H NMR analysis using 1,3,5-trimethoxybenzene as internal standard. Also, the crude residue was tested with HRMS. The adduct of TEMPO with benzyl radical was detected by HRMS (ESI): HRMS (ESI) m/z : $[M+H]^+$ Calcd for $C_{17}H_{28}NO^+$ 262.2165; Found = 262.2155.

5.2 Radical trapping experiment with thiophene.



The radical trapping experimental procedure with thiophene (4.0 mmol, 20 eq.) as the radical scavenger was the same as **procedure 5.1**. The crude yield was 71%.

5.3 Deuteration experiment.

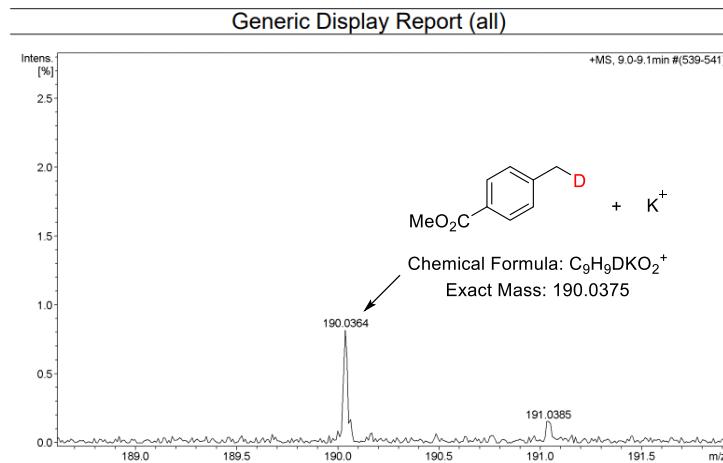


Figure S2 The HRMS spectra of compound 4.

The electrolysis was conducted in an undivided cell equipped with a Mg anode (15 mm x 10 mm x 2 mm) and a C cathode ($\varphi = 6$ mm) with a distance of about 1.0 cm between the two electrodes, and the depth of each electrode immersed in the reaction mixture is about 10 mm. To a 15 mL oven-dried undivided electrochemical cell were added (4-(methoxycarbonyl)benzyl)triphenylphosphonium trifluoromethanesulfonate

1k (0.2 mmol), $^n\text{Bu}_4\text{NPF}_6$ (193.7 mg, 0.5 mmol), D_2O (4.0 mmol, 20.0 eq.), and followed by the addition of solvent DMF (5 mL, 0.1 M) in the glove box. Then, seal the tube and remove it from the glove box. The electrolysis was carried out with constant voltage electrolysis (cell voltage = 5 V) at 25 °C for 7 h under argon atmosphere. Then, the resulting mixture is transferred to a separator funnel for preparing the reaction solution at the beginning. The cell was washed with ethyl acetate (2 mL x 2) twice. The combined organic solution was extracted with ethyl acetate (5 x 3 mL). The combined organic layers were washed with water, brine, dried over anhydrous Na_2SO_4 , filtered and concentrated in vacuo to achieve the crude residue. The crude residue was tested with HRMS. The deuterated product was detected by HRMS (ESI): HRMS (ESI) m/z: $[\text{M}+\text{K}]^+$ Calcd for $\text{C}_9\text{H}_9\text{DKO}_2$ 190.0375; Found 190.0364.

6. Cyclic voltammetry experiments.

Electrochemical studies were carried out with a CHI600D electrochemical workstation. All cyclic voltammograms were measured at 25 °C using an Ag/Ag^+ reference electrode, a platinum (Pt) wire counter electrode and a glassy carbon working electrode (3mm-diameter, disc-electrode). The measurements were carried out at a scan rate of 100 mV s⁻¹ in DMF/ $^n\text{Bu}_4\text{NPF}_6$ (0.1 M). All data are measured in Ar atmosphere.

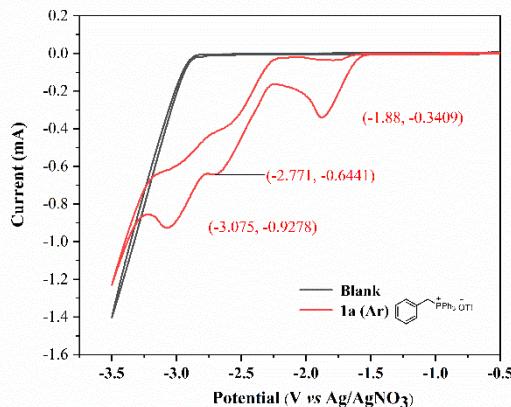


Figure S3. Cyclic voltammetry of Blank: (none); **1a** (40 mM);

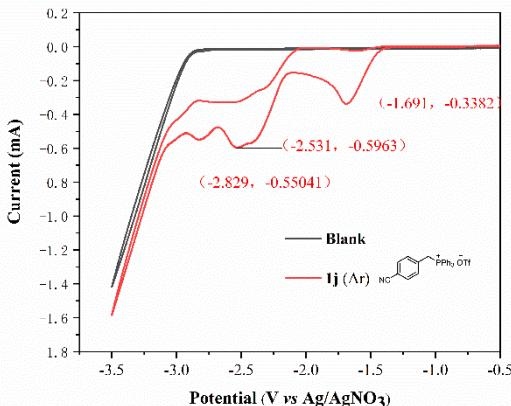


Figure S4. Cyclic voltammetry of Blank: (none); **1j** (40 mM);

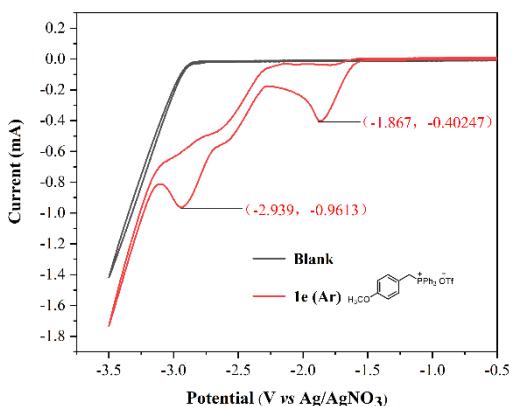


Figure S5. Cyclic voltammetry of Blank: (none); **1e** (40 mM);

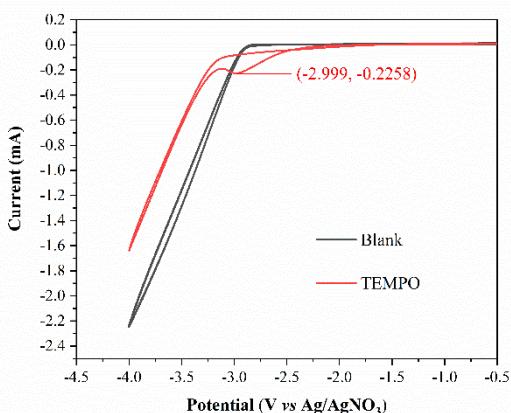
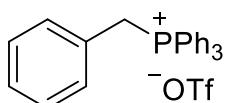


Figure S6. Cyclic voltammetry of Blank: (none); Tempo (20 mM);

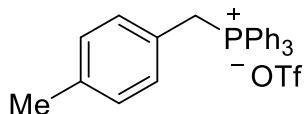
7. Characterization data for compounds **1** and **2**.

benzyltriphenylphosphonium trifluoromethanesulfonate (**1a**).



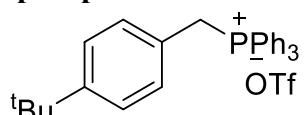
White powder. ¹H NMR (400 MHz, DMSO-*d*₆) δ 7.92 – 7.89 (m, 3H), 7.77 – 7.72 (m, 6H), 7.69 – 7.64 (m, 6H), 7.32 – 7.28 (m, 1H), 7.25 – 7.21 (m, 2H), 6.98 – 6.96 (m, 2H), 5.14 (d, *J* = 15.6 Hz, 2H) ppm. ¹³C NMR (100 MHz, DMSO-*d*₆) δ 135.1 (d, *J* = 2.8 Hz), 134.0 (d, *J* = 9.9 Hz), 130.8 (d, *J* = 5.7 Hz), 130.1 (d, *J* = 12.3 Hz), 128.8 (d, *J* = 2.9 Hz), 128.3 (d, *J* = 3.6 Hz), 127.8 (d, *J* = 8.6 Hz), 120.7 (q, *J* = 320.6 Hz) 117.8 (d, *J* = 85 Hz), 28.2 (d, *J* = 46.6 Hz) ppm. ³¹P NMR (162 MHz, DMSO-*d*₆) δ 23.1 ppm. ¹⁹F NMR (376 MHz, DMSO-*d*₆) δ -77.7 ppm. HRMS (ESI) m/z: [M-OTf]⁺ Calcd for C₂₅H₂₂P 353.1454; Found 353.1450.

methylbenzyl)triphenylphosphonium trifluoromethanesulfonate (**1b**).



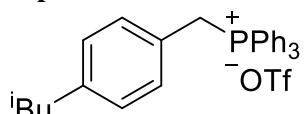
¹H NMR (400 MHz, DMSO-*d*₆) δ 7.92 – 7.89 (m, 3H), 7.77 – 7.72 (m, 6H), 7.69 – 7.63 (m, 6H), 7.04 (d, *J* = 7.6 Hz, 2H), 6.86 – 6.84 (m, 2H), 5.08 (d, *J* = 15.6 Hz, 2H), 2.23 (d, *J* = 2.0 Hz, 3H) ppm. ¹³C NMR (100 MHz, DMSO-*d*₆) δ 137.8 (d, *J* = 4.0 Hz), 135.0 (d, *J* = 2.7 Hz), 134.0 (d, *J* = 9.8 Hz), 130.7 (d, *J* = 4.6 Hz), 130.0 (d, *J* = 12.3 Hz), 129.3 (d, *J* = 4.0 Hz), 124.5 (d, *J* = 8.6 Hz), 120.7 (q, 320.5 Hz), 117.8 (d, *J* = 84.8 Hz), 27.9 (d, *J* = 46.3 Hz) ppm. ³¹P NMR (162 MHz, DMSO-*d*₆) δ 22.6 ppm. ¹⁹F NMR (376 MHz, DMSO-*d*₆) δ -77.7 ppm. HRMS (ESI) m/z: [M-OTf]⁺ Calcd for C₂₆H₂₄P 367.1610; Found 367.1606.

(4-(tert-butyl)benzyl)triphenylphosphonium trifluoromethanesulfonate (1c).



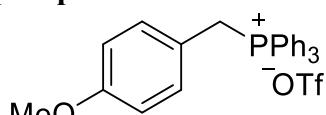
¹H NMR (400 MHz, DMSO-*d*₆) δ 7.93 – 7.89 (m, 3H), 7.76 – 7.72 (m, 6H), 7.67 – 7.62 (m, 6H), 7.25 (d, *J* = 8.4 Hz, 2H), 6.91 – 6.88 (m, 2H), 5.09 (d, *J* = 15.6 Hz, 2H), 1.22 (s, 9H) ppm. ¹³C NMR (100 MHz, DMSO-*d*₆) δ 151.6 (d, *J* = 4.1 Hz), 135.6 (d, *J* = 2.8 Hz), 134.4 (d, *J* = 9.7 Hz), 130.9 (d, *J* = 5.6 Hz), 130.6 (d, *J* = 12.3 Hz), 126.0 (d, *J* = 3.1 Hz), 125.2 (d, *J* = 8.6 Hz), 121.2 (q, *J* = 320.5 Hz), 118.4 (d, *J* = 84.9 Hz), 34.8, 31.4, 28.2 (d, *J* = 46.6 Hz) ppm. ³¹P NMR (162 MHz, DMSO-*d*₆) δ 23.03 ppm. ¹⁹F NMR (376 MHz, DMSO-*d*₆) δ -77.69 ppm. HRMS (ESI) m/z: [M-OTf]⁺ Calcd for C₂₉H₃₀P 409.2080; Found 409.2078.

(4-isobutylbenzyl)triphenylphosphonium trifluoromethanesulfonate (1d).



¹H NMR (400 MHz, DMSO-*d*₆) δ 7.93 – 7.89 (m, 3H), 7.76 – 7.71 (m, 6H), 7.67 – 7.62 (m, 6H), 7.02 (d, *J* = 7.6 Hz, 2H), 6.88 – 6.86 (m, 2H), 5.09 (d, *J* = 15.2 Hz, 2H), 2.39 (d, *J* = 6.8 Hz, 2H), 1.80 – 1.74 (m, 1H), 0.82 (d, *J* = 6.4 Hz, 6H) ppm. ¹³C NMR (100 MHz, DMSO-*d*₆) δ 142.4 (d, *J* = 4.1 Hz), 136.0 (d, *J* = 2.6 Hz), 134.9 (d, *J* = 9.7 Hz), 131.5 (d, *J* = 5.5 Hz), 131.0 (d, *J* = 12.4 Hz), 130.3 (d, *J* = 3.1 Hz), 125.9 (d, *J* = 8.4 Hz), 121.6 (q, *J* = 320.3 Hz), 118.8 (d, *J* = 84.8 Hz), 44.9, 30.4, 28.7 (d, *J* = 46.2 Hz), 22.9 ppm. ³¹P NMR (162 MHz, DMSO-*d*₆) δ 23.0 ppm. ¹⁹F NMR (376 MHz, DMSO-*d*₆) δ -77.7 ppm. HRMS (ESI) m/z: [M-OTf]⁺ Calcd for C₂₉H₃₀P 409.2080; Found 409.2078.

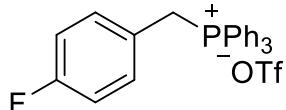
(4-methoxybenzyl)triphenylphosphonium trifluoromethanesulfonate (1e).



¹H NMR (400 MHz, DMSO-*d*₆) δ 7.92 – 7.88 (m, 3H), 7.77 – 7.72 (m, 6H), 7.68 – 7.63

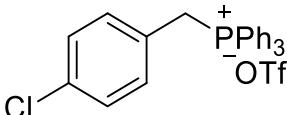
(m, 6H), 6.90 – 6.87 (m, 2H), 6.79 (d, J = 8.8 Hz, 2H), 5.05 (d, J = 14.8 Hz, 2H), 3.69 (s, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 159.3 (d, J = 3.6 Hz), 135.2 (d, J = 2.7 Hz), 134.1 (d, J = 9.8 Hz), 132.1 (d, J = 5.5 Hz), 130.2 (d, J = 12.3 Hz), 120.8 (q, J = 320.5 Hz), 119.1 (d, J = 2.9 Hz), 118.0 (d, J = 84.8 Hz), 114.4 (d, J = 2.9 Hz), 55.2, 27.6 (d, J = 46.4 Hz). ^{31}P NMR (162 MHz, DMSO- d_6) δ 22.3 ppm. ^{19}F NMR (376 MHz, DMSO- d_6) δ -77.7 ppm. HRMS (ESI) m/z: [M-OTf]⁺ Calcd for C₂₆H₂₄OP 383.1559; Found 383.1559.

(4-fluorobenzyl)triphenylphosphonium trifluoromethanesulfonate (1f).



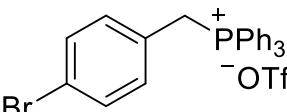
^1H NMR (400 MHz, DMSO- d_6) δ 7.93 – 7.90 (m, 3H), 7.78 – 7.74 (m, 6H), 7.70 – 7.65 (m, 6H), 7.12 – 7.08 (m, 2H), 7.03 – 6.97 (m, 2H), 5.14 (d, J = 15.6 Hz, 2H) ppm. ^{13}C NMR (100 MHz, DMSO- d_6) δ 161.9 (d, J = 248.4 Hz), 135.1 (d, J = 2.8 Hz), 134.0 (d, J = 9.7 Hz), 132.8 (dd, J = 8.3, 5.5 Hz), 130.1 (d, J = 12.3 Hz), 124.0 (dd, J = 8.6, 2.9 Hz), 120.7 (q, 320.2 Hz), 117.6 (d, J = 85.1 Hz), 115.8 (d, J = 21.6, 3.0 Hz), 27.6, 27.1 ppm. ^{31}P NMR (162 MHz, DMSO- d_6) δ 23.1 ppm. ^{19}F NMR (376 MHz, DMSO- d_6) δ -77.7, -113.1 ppm. HRMS (ESI) m/z: [M-OTf]⁺ Calcd for C₂₅H₂₁FP 371.1359; Found 371.1356.

(4-chlorobenzyl)triphenylphosphonium trifluoromethanesulfonate (1g).



^1H NMR (400 MHz, DMSO- d_6) δ 7.93 – 7.90 (m, 3H), 7.77 – 7.68 (m, 12H), 7.32 (d, J = 8.0 Hz, 2H), 7.00 (d, J = 6.8 Hz, 2H), 5.18 (d, J = 16.0 Hz, 2H) ppm. ^{13}C NMR (100 MHz, DMSO- d_6) δ 135.2 (d, J = 2.7 Hz), 134.0 (d, J = 9.8 Hz), 133.3 (d, J = 4.5 Hz), 132.5 (d, J = 5.6 Hz), 130.1 (d, J = 12.4 Hz), 128.8 (d, J = 3.0 Hz), 126.9 (d, J = 8.6 Hz), 120.7 (q, J = 320.4 Hz), 117.5 (d, J = 85.0 Hz), 27.5 (d, J = 47.0 Hz) ppm. ^{31}P NMR (162 MHz, DMSO- d_6) δ 23.0 ppm. ^{19}F NMR (376 MHz, DMSO- d_6) δ -77.7 ppm. HRMS (ESI) m/z: [M-OTf]⁺ Calcd for C₂₅H₂₁ClP 387.1064; Found 387.1062.

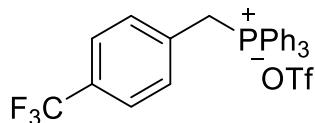
(4-bromobenzyl)triphenylphosphonium trifluoromethanesulfonate (1h).



^1H NMR (400 MHz, DMSO- d_6) δ 7.92 – 7.90 (m, 3H), 7.76 – 7.70 (m, 12H), 7.46 (d, J = 6.4 Hz, 2H), 6.91 (d, J = 6.4 Hz, 2H), 5.17 (d, J = 16.0 Hz, 2H) ppm. ^{13}C NMR (100 MHz, DMSO- d_6) δ 135.2 (d, J = 2.7 Hz), 134.0 (d, J = 9.7 Hz), 132.87 (d, J = 5.4 Hz), 131.7 (d, J = 2.9 Hz), 130.1 (d, J = 12.3 Hz), 127.5 (d, J = 8.6 Hz), 121.8 (d, J = 4.7 Hz), 117.6 (d, J = 85.0 Hz), 27.5 (d, J = 46.6 Hz) ppm. ^{31}P NMR (162 MHz, DMSO- d_6) δ 22.9 ppm. ^{19}F NMR (376 MHz, DMSO- d_6) δ -77.7 ppm. HRMS (ESI) m/z: [M-OTf]⁺ Calcd for C₂₅H₂₁BrP 387.1064; Found 387.1062.

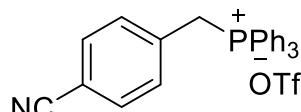
$\text{OTf}]^+$ Calcd for $\text{C}_{25}\text{H}_{21}\text{BrP}$ 431.0559; Found 431.0559.

triphenyl(4-(trifluoromethyl)benzyl)phosphonium trifluoromethanesulfonate (1i).



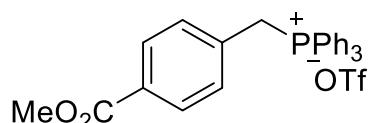
^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 7.92 – 7.91 (m, 3H), 7.76 – 7.63 (m, 14H), 7.20 – 7.18 (m, 2H), 5.29 (d, J = 16.0 Hz, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 135.3 (d, J = 3.0 Hz), 134.1 (d, J = 9.6 Hz), 131.7 (d, J = 5.3 Hz), 131.6 (d, J = 7.9 Hz), 130.7 (d, J = 3.8 Hz), 130.3 (d, J = 12.5 Hz), 125.6 (q, J = 3.6 Hz), 123.7 (q, J = 270.2 Hz), 120.8 (q, J = 318.7 Hz), 117.0 (d, J = 85.8 Hz), 29.6 (d, J = 48.0 Hz) ppm. ^{31}P NMR (162 MHz, $\text{DMSO}-d_6$) δ 23.5 ppm. ^{19}F NMR (376 MHz, $\text{DMSO}-d_6$) δ -61.1, -77.8 ppm. HRMS (ESI) m/z: [M-OTf] $^+$ Calcd for $\text{C}_{26}\text{H}_{21}\text{F}_3\text{P}$ 421.1327; Found 421.1326.

(4-methylbenzyl)triphenylphosphonium trifluoromethanesulfonate (1j).



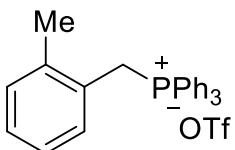
^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 7.94 – 7.90 (m, 3H), 7.79 – 7.66 (m, 14H), 7.16 – 7.14 (m, 2H), 5.28 (d, J = 15.6 Hz, 2H). ^{13}C NMR (100 MHz, $\text{DMSO}-d_6$) δ 135.3 (d, J = 2.9 Hz), 134.0 (d, J = 9.8 Hz), 132.6 (d, J = 4.2 Hz), 131.7 (d, J = 4.5 Hz), 130.2 (d, J = 12.5 Hz), 120.7 (d, J = 320.5 Hz), 118.2 (d, J = 7.8 Hz), 117.3 (d, J = 85.3 Hz), 111.3 (d, J = 4.1 Hz), 28.2 (d, J = 46.8 Hz) ppm. ^{31}P NMR (162 MHz, $\text{DMSO}-d_6$) δ 23.5 ppm. ^{19}F NMR (376 MHz, $\text{DMSO}-d_6$) δ -77.7 ppm. HRMS (ESI) m/z: [M-OTf] $^+$ Calcd for $\text{C}_{26}\text{H}_{21}\text{NP}$ 378.1406; Found 378.1402.

(4-(methoxycarbonyl)benzyl)triphenylphosphonium trifluoromethanesulfonate (1k).



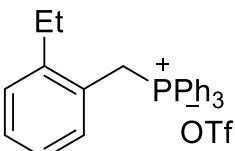
^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 7.95 – 7.91 (m, 3H), 7.83 – 7.67 (m, 14H), 7.13 (d, J = 6.4 Hz, 2H), 5.27 (d, J = 16.0 Hz, 2H), 3.82 (s, 3H) ppm. ^{13}C NMR (100 MHz, $\text{DMSO}-d_6$) δ 176.5 (d, J = 1.0 Hz), 135.2 (d, J = 2.6 Hz), 134.0 (d, J = 9.9 Hz), 133.6 (d, J = 8.5 Hz), 131.2 (d, J = 5.4 Hz), 130.2 (d, J = 12.5 Hz), 129.4 (d, J = 2.5 Hz), 120.8 (q, J = 320.2 Hz), 117.4 (d, J = 85.3 Hz), 52.2, 28.2 (d, J = 46.6 Hz) ppm. ^{31}P NMR (162 MHz, $\text{DMSO}-d_6$) δ 23.3 ppm. ^{19}F NMR (376 MHz, $\text{DMSO}-d_6$) δ -77.7 ppm. HRMS (ESI) m/z: [M-OTf] $^+$ Calcd for $\text{C}_{27}\text{H}_{24}\text{O}_2\text{P}$ 411.1508; Found 411.1509.

(2-methylbenzyl)triphenylphosphonium trifluoromethanesulfonate (1l)



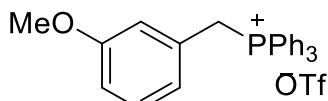
¹H NMR (400 MHz, DMSO-*d*₆) δ 7.94 – 7.90 (m, 3H), 7.76 – 7.71 (m, 6H), 7.64 – 7.59 (m, 6H), 7.25 – 7.21 (m, 1H), 7.11 (d, *J* = 7.6 Hz, 1H), 7.06 – 7.03 (m, 1H), 6.94 (d, *J* = 8.4 Hz, 1H), 5.00 (d, *J* = 14.8 Hz, 2H), 1.64 (s, 3H) ppm. ¹³C NMR (100 MHz, DMSO-*d*₆) δ 138.8 (d, *J* = 5.9 Hz), 135.4 (d, *J* = 2.8 Hz), 134.1 (d, *J* = 9.8 Hz), 131.1 (d, *J* = 3 Hz), 130.9 (d, *J* = 5.0 Hz), 130.3 (d, *J* = 12.3 Hz), 128.8 (d, *J* = 3.7 Hz), 126.4 (d, *J* = 3.1 Hz), 126.1 (d, *J* = 8.5 Hz), 120.8 (q, *J* = 318.6 Hz), 117.6 (d, *J* = 84.6 Hz), 26.1 (d, *J* = 47.0 Hz), 18.9 ppm. ³¹P NMR (162 MHz, DMSO-*d*₆) δ 22.3 ppm. ¹⁹F NMR (376 MHz, DMSO-*d*₆) δ -77.7 ppm. HRMS (ESI) m/z: [M-OTf]⁺ Calcd for C₂₆H₂₄P 367.1610; Found 367.1606.

(2-ethylbenzyl)triphenylphosphonium trifluoromethanesulfonate (1m).



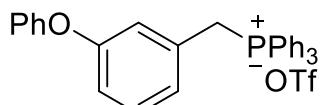
¹H NMR (400 MHz, DMSO-*d*₆) δ 7.94 – 7.90 (m, 3H), 7.76 – 7.71 (m, 6H), 7.63 – 7.58 (m, 6H), 7.31 – 7.28 (m, 1H), 7.18 (d, *J* = 7.6 Hz, 1H), 7.06 – 7.02 (m, 1H), 6.92 (d, *J* = 8.0 Hz, 1H), 4.99 (d, *J* = 15.2 Hz, 2H), 1.95 (q, *J* = 7.6 Hz, 2H), 0.84 (t, *J* = 7.6 Hz, 3H) ppm. ¹³C NMR (100 MHz, DMSO-*d*₆) δ 144.2 (d, *J* = 6.2 Hz), 135.3 (d, *J* = 2.8 Hz), 134.1 (d, *J* = 9.7 Hz), 131.0 (d, *J* = 4.8 Hz), 130.2 (d, *J* = 12.3 Hz), 129.0 (d, *J* = 3.8 Hz), 128.9 (d, *J* = 2.9 Hz), 126.2 (d, *J* = 3.4 Hz), 125.3 (d, *J* = 8.6 Hz), 120.7 (q, *J* = 320.2 Hz), 117.6 (d, *J* = 84.8 Hz), 25.6 (d, *J* = 46.8 Hz), 24.4, 14.3 ppm. ³¹P NMR (162 MHz, DMSO-*d*₆) δ 22.5 ppm. ¹⁹F NMR (376 MHz, DMSO-*d*₆) δ -77.8 ppm. HRMS (ESI) m/z: [M-OTf]⁺ Calcd for C₂₇H₂₆P 381.1767; Found 381.1766.

(3-methoxybenzyl)triphenylphosphonium trifluoromethanesulfonate (1n)



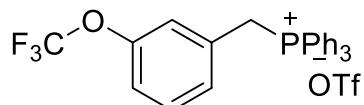
¹H NMR (400 MHz, DMSO-*d*₆) δ 7.93 – 7.90 (m, 3H), 7.78 – 7.73 (m, 6H), 7.71 – 7.66 (m, 6H), 7.19 – 7.15 (m, 1H), 6.87 (d, *J* = 8.0 Hz, 1H), 6.61 (d, *J* = 7.6 Hz, 1H), 6.48 (d, *J* = 1.6 Hz, 1H), 5.10 (d, *J* = 15.6 Hz, 2H), 3.50 (s, 3H) ppm. ¹³C NMR (100 MHz, DMSO-*d*₆) δ 159.3 (d, *J* = 3.4 Hz), 135.2 (d, *J* = 2.7 Hz), 134.1 (d, *J* = 9.7 Hz), 130.2 (d, *J* = 12.3 Hz), 130.0 (d, *J* = 3.0 Hz), 129.3 (d, *J* = 8.5 Hz), 123.1 (d, *J* = 5.7 Hz), 117.9 (d, *J* = 85.2 Hz), 120.8 (q, *J* = 320.3 Hz), 116.3 (d, *J* = 5.4 Hz), 114.4 (d, *J* = 3.6 Hz), 55.0, 28.4 (d, *J* = 46.8 Hz) ppm. ³¹P NMR (162 MHz, DMSO-*d*₆) δ 23.1 ppm. ¹⁹F NMR (376 MHz, DMSO-*d*₆) δ -77.7 ppm. HRMS (ESI) m/z: [M-OTf]⁺ Calcd for C₂₆H₂₄OP 383.1559; Found 383.1559.

(3-phenoxybenzyl)triphenylphosphonium trifluoromethanesulfonate (1o).



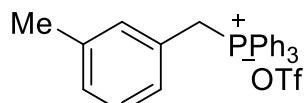
^1H NMR (400 MHz, DMSO- d_6) δ 7.94 – 7.90 (m, 3H), 7.75 – 7.65 (m, 12H), 7.36 – 7.32 (m, 2H), 7.29 – 7.25 (m, 1H), 7.17 – 7.13 (m, 1H), 7.00 (d, $J = 8.0$ Hz, 1H), 6.83 – 6.77 (m, 3H), 6.58 (s, 1H), 5.20 (d, $J = 16.0$ Hz, 2H) ppm. ^{13}C NMR (100 MHz, DMSO- d_6) δ 156.9 (d, $J = 3.3$ Hz), 155.7, 135.1 (d, $J = 2.7$ Hz), 134.0 (d, $J = 9.7$ Hz), 130.4 (d, $J = 3.2$ Hz), 130.11 (d, $J = 12.3$ Hz), 130.08, 129.9 (d, $J = 8.7$ Hz), 125.5 (d, $J = 5.5$ Hz), 123.8, 120.2 (d, $J = 5.6$ Hz), 118.8, 118.6 (d, $J = 3.5$ Hz), 117.6 (d, $J = 85.2$ Hz), 28.0 (d, $J = 46.6$ Hz) ppm. ^{31}P NMR (162 MHz, DMSO- d_6) δ 23.3 ppm. ^{19}F NMR (376 MHz, DMSO- d_6) δ -77.7 ppm. HRMS (ESI) m/z: [M-OTf] $^+$ Calcd for $\text{C}_{31}\text{H}_{26}\text{OP}$ 445.1716 Found 445.1717.

triphenyl(3-(trifluoromethoxy)benzyl)phosphonium trifluoromethanesulfonate (1p).



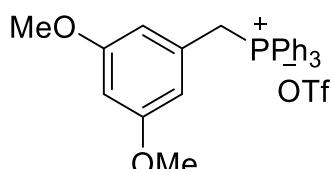
^1H NMR (400 MHz, DMSO- d_6) δ 7.94 – 7.90 (m, 3H), 7.78 – 7.66 (m, 12H), 7.42 – 7.38 (m, 1H), 7.31 (d, $J = 8.4$ Hz, 1H), 7.06 (d, $J = 7.6$ Hz, 1H), 6.94 (s, 1H), 5.28 (d, $J = 15.6$ Hz, 2H) ppm. ^{13}C NMR (100 MHz, DMSO- d_6) δ 149.1, 136.1 (d, $J = 2.8$ Hz), 134.9 (d, $J = 10.0$ Hz), 131.7 (q, $J = 3.2$ Hz), 131.6, 131.0 (d, $J = 12.4$ Hz), 130.7 (d, $J = 5.3$ Hz), 124.1 (d, $J = 5.4$ Hz), 121.9 (d, $J = 3.2$ Hz), 120.7 (q, $J = 255.4$ Hz), 118.3 (d, $J = 85.3$ Hz), 28.7 (d, $J = 47.1$ Hz) ppm. ^{31}P NMR (162 MHz, DMSO- d_6) δ 23.5 ppm. ^{19}F NMR (376 MHz, DMSO- d_6) δ -56.8, -77.8 ppm. HRMS (ESI) m/z: [M-OTf] $^+$ Calcd for $\text{C}_{26}\text{H}_{21}\text{F}_3\text{OP}$ 437.1277; Found 437.1277.

methylbenzyltriphenylphosphonium trifluoromethanesulfonate (1q).



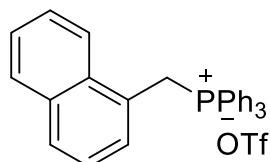
^1H NMR (400 MHz, DMSO- d_6) δ 7.93 – 7.90 (m, 3H), 7.78 – 7.73 (m, 6H), 7.69 – 7.64 (m, 6H), 7.13 (d, $J = 6.0$ Hz, 2H), 6.81 (d, $J = 6.4$ Hz, 1H), 6.65 (s, 1H), 5.08 (d, $J = 15.6$ Hz, 2H), 2.09 (s, 3H) ppm. ^{13}C NMR (100 MHz, DMSO- d_6) δ 138.8 (d, $J = 3.2$ Hz), 136.0 (d, $J = 2.7$ Hz), 134.9 (d, $J = 9.9$ Hz), 132.5 (d, $J = 5.5$ Hz), 131.0 (d, $J = 12.4$ Hz), 129.8 (d, $J = 3.8$ Hz), 129.6 (d, $J = 3.0$ Hz), 128.8 (d, $J = 5.6$ Hz), 128.5 (d, $J = 8.5$ Hz), 121.6 (d, $J = 320.5$ Hz), 118.7 (d, $J = 8.5$ Hz), 28.1 (d, $J = 46.4$ Hz), 20.7 ppm. ^{31}P NMR (162 MHz, DMSO- d_6) δ 23.0 ppm. ^{19}F NMR (376 MHz, DMSO- d_6) δ -77.7 ppm. HRMS (ESI) m/z: [M-OTf] $^+$ Calcd for $\text{C}_{26}\text{H}_{24}\text{P}$ 367.1610; Found 367.1606.

(3,5-dimethoxybenzyl)triphenylphosphonium trifluoromethanesulfonate (1r).



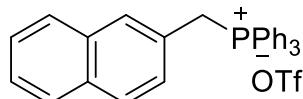
¹H NMR (400 MHz, DMSO-*d*₆) δ 7.93 – 7.89 (m, 3H), 7.78 – 7.73 (m, 6H), 7.69 – 7.65 (m, 6H), 6.42 (s, 1H), 6.12 (s, 2H), 5.02 (d, *J* = 15.6 Hz, 2H), 3.50 (s, 9H) ppm. ¹³C NMR (100 MHz, DMSO-*d*₆) δ 160.5 (d, *J* = 3.1 Hz), 135.2 (d, *J* = 2.7 Hz), 134.1 (d, *J* = 9.7 Hz), 130.2 (d, *J* = 12.3 Hz), 129.9 (d, *J* = 8.4 Hz), 120.8 (q, *J* = 320.4 Hz), 117.8 (d, *J* = 85.2 Hz), 109.0 (d, *J* = 5.4 Hz), 100.3 (d, *J* = 3.3 Hz), 55.2, 28.6 (d, *J* = 46.1 Hz) ppm. ³¹P NMR (162 MHz, DMSO-*d*₆) δ 23.0 ppm. ¹⁹F NMR (376 MHz, DMSO-*d*₆) δ -77.8 ppm. HRMS (ESI) m/z: [M-OTf]⁺ Calcd for C₂₇H₂₆O₂P 413.1665 Found 413.1664.

(naphthalen-1-ylmethyl)triphenylphosphonium trifluoromethanesulfonate (1s).



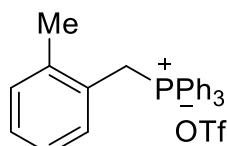
¹H NMR (400 MHz, DMSO-*d*₆) δ 7.91 – 7.81 (m, 5H), 7.69 – 7.60 (m, 13H), 7.41 – 7.34 (m, 2H), 7.31 – 7.29 (m, 1H), 7.13 – 7.09 (m, 1H), 5.58 (d, *J* = 15.6 Hz, 2H) ppm. ¹³C NMR (100 MHz, DMSO-*d*₆) δ 135.0 (d, *J* = 2.8 Hz), 134.0 (d, *J* = 9.9 Hz), 133.2 (d, *J* = 2.7 Hz), 132.0 (d, *J* = 4.6 Hz), 129.9 (d, *J* = 12.4 Hz), 129.7 (d, *J* = 6.8 Hz), 129.2 (d, *J* = 4.2 Hz), 128.4, 125.9, 125.1 (d, *J* = 3.9 Hz), 123.9 (d, *J* = 9.0 Hz), 123.7, 120.6 (d, *J* = 320.4 Hz), 117.6 (d, *J* = 84.6 Hz), 25.2 (d, *J* = 47.3 Hz) ppm. ³¹P NMR (162 MHz, DMSO-*d*₆) δ 22.8 ppm. ¹⁹F NMR (376 MHz, DMSO-*d*₆) δ -77.7 ppm. HRMS (ESI) m/z: [M-OTf]⁺ Calcd for C₂₉H₂₄P 403.1610 Found 403.1610.

(naphthalen-2-ylmethyl)triphenylphosphonium trifluoromethanesulfonate (1t).



¹H NMR (400 MHz, DMSO-*d*₆) δ 7.93 – 7.86 (m, 4H), 7.79 – 7.68 (m, 13H), 7.62 – 7.59 (m, 1H), 7.53 – 7.47 (m, 3H), 7.06 (d, *J* = 8.4 Hz, 1H), 5.32 (d, *J* = 15.6 Hz, 2H) ppm. ¹³C NMR (100 MHz, DMSO-*d*₆) δ 135.1 (d, *J* = 2.8 Hz), 134.0 (d, *J* = 9.9 Hz), 132.5 (d, *J* = 3.1 Hz), 132.2 (d, *J* = 2.6 Hz), 130.3 (d, *J* = 7.3 Hz), 130.1 (d, *J* = 12.3 Hz), 128.3 (d, *J* = 2.3 Hz), 128.0 (d, *J* = 4.2 Hz), 127.6, 127.4, 126.8 (d, *J* = 8.9 Hz), 125.4 (d, *J* = 9.0 Hz), 120.8 (q, *J* = 320.3 Hz), 117.8 (d, *J* = 85.1 Hz), 28.4 (d, *J* = 46.4 Hz) ppm. ³¹P NMR (162 MHz, DMSO-*d*₆) δ 22.9 ppm. ¹⁹F NMR (376 MHz, DMSO-*d*₆) δ -77.7 ppm. HRMS (ESI) m/z: [M-OTf]⁺ Calcd for C₂₉H₂₄P 403.1610 Found 403.1610.

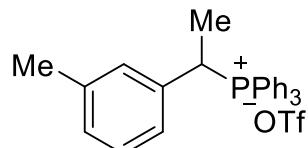
triphenyl(1-phenylethyl)phosphonium trifluoromethanesulfonate (1u).



¹H NMR (400 MHz, DMSO-*d*₆) δ 7.93 – 7.89 (m, 3H), 7.70 – 7.72 (m, 6H), 7.71 – 7.64

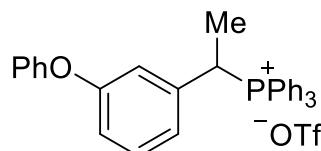
(m, 6H), 7.35 – 7.34 (m, 1H), 7.28 (t, J = 7.6 Hz, 2H), 6.96 – 6.94 (m, 2H), 5.65 – 5.56 (m, 1H), 1.74 (dd, J = 18.8, 7.2 Hz, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 135.2 (d, J = 2.7 Hz), 134.3 (d, J = 9.1 Hz), 133.7 (d, J = 5.2 Hz), 130.3 (d, J = 12.0 Hz), 129.7 (d, J = 5.8 Hz), 129.05, 128.98 (d, J = 2.1 Hz), 120.6 (q, J = 320.6 Hz), 117.2 (d, J = 82.1 Hz), 34.2 (d, J = 43.6 Hz), 16.8 ppm. ^{31}P NMR (162 MHz, DMSO- d_6) δ 26.3 ppm. ^{19}F NMR (376 MHz, DMSO- d_6) δ -77.7 ppm. [M-OTf] $^+$ Calcd for $\text{C}_{26}\text{H}_{24}\text{P}$ 367.1610; Found 367.1606.

triphenyl(1-(m-tolyl)ethyl)phosphonium trifluoromethanesulfonate (1v).



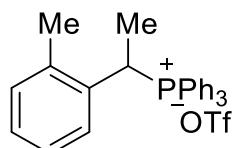
^1H NMR (400 MHz, DMSO- d_6) δ 7.95 – 7.91 (m, 3H), 7.79 – 7.68 (m, 12H), 7.22 – 7.18 (m, 2H), 6.87 (d, J = 6.8 Hz, 1H), 6.58 (s, 1H), 5.61 – 5.52 (m, 1H), 2.12 (s, 3H), 1.73 (dd, J = 18.8, 7.2 Hz, 3H) ppm. ^{13}C NMR (100 MHz, DMSO- d_6) δ 138.1 (d, J = 2.4 Hz), 135.0 (d, J = 2.8 Hz), 134.3 (d, J = 9.1 Hz), 134.0 (d, J = 9.8 Hz), 133.3 (d, J = 5.2 Hz), 130.3 (d, J = 5.6 Hz), 130.1 (d, J = 12 Hz), 129.4 (d, J = 3.1 Hz), 128.7 (d, J = 2.1 Hz), 126.8 (d, J = 5.9 Hz), 120.8 (d, J = 320.5 Hz), 117.1 (d, 82.0 Hz), 34.1 (d, J = 43.4 Hz), 20.8, 16.6 ppm. ^{31}P NMR (162 MHz, DMSO- d_6) δ 26.3 ppm. ^{19}F NMR (376 MHz, DMSO- d_6) δ -77.7 ppm. [M-OTf] $^+$ Calcd for $\text{C}_{27}\text{H}_{26}\text{P}$ 381.1767; Found 381.1765.

(1-(3-phenoxyphenyl)ethyl)triphenylphosphonium trifluoromethanesulfonate (1w).



^1H NMR (400 MHz, DMSO- d_6) δ 7.94 – 7.90 (m, 3H), 7.77 – 7.72 (m, 6H), 7.70 – 7.65 (m, 6H), 7.41 – 7.30 (m, 3H), 7.20 – 7.16 (m, 1H), 7.05 (d, J = 8.0 Hz, 1H), 6.89 (d, J = 7.6 Hz, 2H), 6.70 (d, J = 7.6 Hz, 1H), 6.58 (d, J = 1.6 Hz, 1H), 5.62 – 5.52 (m, 1H), 1.71 (dd, J = 18.8, 7.2 Hz, 3H) ppm. ^{13}C NMR (100 MHz, DMSO- d_6) δ 157.1 (d, J = 2.4 Hz), 155.7, 135.7 (d, J = 5.1 Hz), 135.1 (d, J = 2.7 Hz), 134.2 (d, J = 9.2 Hz), 130.7 (d, J = 2.0 Hz), 130.3, 130.2 (d, J = 1.0 Hz), 124.2 (d, J = 5.6 Hz), 124.0, 119.25 (d, J = 5.9 Hz), 119.15 (d, J = 2.9 Hz), 119.0, 116.9 (d, J = 82.3 Hz), 34.20 (d, J = 44.3 Hz), 16.8 ppm. ^{31}P NMR (162 MHz, DMSO- d_6) δ 26.2 ppm. ^{19}F NMR (376 MHz, DMSO- d_6) δ -77.7 ppm. [M-OTf] $^+$ Calcd for $\text{C}_{32}\text{H}_{28}\text{OP}$ 459.1872; Found 459.1872.

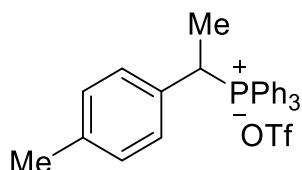
triphenyl(1-(o-tolyl)ethyl)phosphonium trifluoromethanesulfonate (1x)



^1H NMR (400 MHz, DMSO- d_6) δ 7.97 – 7.94 (m, 3H), 7.77 – 7.72 (m, 6H), 7.52 – 7.47

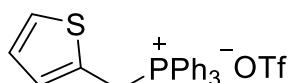
(m, 6H), 7.33 – 7.30 (m, 1H), 7.20 (d, J = 7.6 Hz, 1H), 7.13–7.09 (m, 1H), 6.65 (d, J = 8.0 Hz, 1H), 5.16 – 5.07 (m, 1H), 1.89 – 1.81 (m, 6H) ppm. ^{13}C NMR (100 MHz, DMSO- d_6) δ 138.4 (d, J = 7.3 Hz), 135.5 (d, J = 2.7 Hz), 134.5 (d, J = 9.1 Hz), 131.9 (d, J = 5.2 Hz), 131.3, 130.4 (d, J = 11.9 Hz), 129.2 (d, J = 2.9 Hz), 128.2 (d, J = 4.5 Hz), 126.8 (d, J = 2.7 Hz), 120.9 (q, J = 320.3 Hz), 116.7 (d, J = 81.9 Hz), 32.6 (d, J = 44.6 Hz), 19.1, 17.4 ppm. ^{31}P NMR (162 MHz, DMSO- d_6) δ 24.9 ppm. ^{19}F NMR (376 MHz, DMSO- d_6) δ -77.7 ppm. [M-OTf] $^+$ Calcd for C₂₇H₂₆P 381.1767; Found 381.1765.

triphenyl(1-(p-tolyl)ethyl)phosphonium trifluoromethanesulfonate (1y).



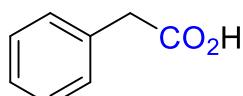
^1H NMR (400 MHz, DMSO- d_6) δ 7.92 – 7.89 (m, 3H), 7.78 – 7.66 (m, 12H), 7.08 (d, J = 8.0 Hz, 2H), 6.84 – 6.82 (m, 2H), 5.56 – 5.52 (m, 1H), 2.26 (d, J = 1.6 Hz, 3H), 1.70 (dd, J = 18.8, 7.2, Hz, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 139.0 (d, J = 3.3 Hz), 135.5 (d, J = 2.7 Hz), 134.7 (d, J = 9.2 Hz), 130.9 (d, J = 5.3 Hz), 130.7 (d, J = 12.0 Hz), 130.1 (d, J = 5.7 Hz), 129.9 (d, J = 2.3 Hz), 121.2 (q, J = 320.2 Hz) 117.7 (d, J = 82.1 Hz), 34.3 (d, J = 43.4 Hz), 21.1, 17.3 ppm. ^{31}P NMR (162 MHz, DMSO- d_6) δ 26.0 ppm. ^{19}F NMR (376 MHz, DMSO- d_6) δ -77.7 ppm. [M-OTf] $^+$ Calcd for C₂₇H₂₆P 381.1767; Found 381.1765.

triphenyl(thiophen-2-ylmethyl)phosphonium trifluoromethanesulfonate (1a')



^1H NMR (400 MHz, DMSO- d_6) δ 7.87 – 7.84 (m, 3H), 7.72 – 7.68 (m, 6H), 7.65 – 7.60 (m, 7.6 Hz, 6H), 7.46 – 7.44 (m, 1H), 7.11 (s, 1H), 6.52 (d, J = 5.2, Hz, 1H), 5.20 (d, J = 15.2 Hz, 2H) ppm. ^{13}C NMR (100 MHz, DMSO- d_6) δ 135.0 (d, J = 2.8 Hz), 133.8 (d, J = 9.7 Hz), 130.1 (d, J = 12.3 Hz), 128.7 (d, J = 3.3 Hz), 127.4 (d, J = 1.2 Hz), 126.9, 126.8, 118.0 (d, J = 85.0 Hz), 23.5 (d, J = 48.3 Hz) ppm. ^{19}F NMR (377 MHz, DMSO- d_6) δ -77.72 (s). ^{31}P NMR (162 MHz, DMSO) δ 21.79 ppm. ^{19}F NMR (376 MHz, DMSO- d_6) δ -77.72 ppm.

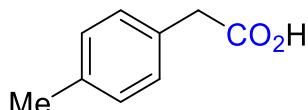
2-phenylacetic acid (2a)².



White solid (22.3 mg, 82% yield). Conformed the documental reports. ^1H NMR (400 MHz, CDCl₃) δ 7.38 – 7.29 (m, 5H), 3.66 (s, 2H) ppm; ^{13}C NMR (100 MHz, CDCl₃) δ

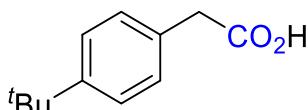
178.1, 133.4, 129.5, 128.8, 127.5, 41.2 ppm.

2-(*p*-tolyl)acetic acid (2b)³.



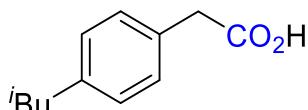
White solid (20.3 mg, 68% yield). Conformed the documental reports. ¹H NMR (400 MHz, CDCl₃) δ 7.18 (d, *J* = 7.6 Hz, 2H), 7.16 (d, *J* = 8.4 Hz, 2H), 3.62 (s, 2H), 2.34 (s, 3H), 0.09 (s, 3H) ppm; ¹³C NMR (100 MHz, CDCl₃) δ 178.4, 137.2, 130.3, 129.5, 129.37, 40.8, 21.2, 1.2 ppm.

2-(4-(*tert*-butyl)phenyl)acetic acid (2c)².



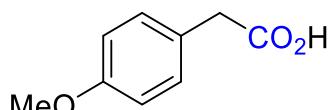
Colorless oil (19.0 mg, 50% yield). Conformed the documental reports. ¹H NMR (400 MHz, CDCl₃) δ 7.37 (d, *J* = 7.6 Hz, 2H), 7.23 (d, *J* = 8.0 Hz, 2H), 3.63 (s, 2H), 1.32 (s, 9H) ppm; ¹³C NMR (100 MHz, CDCl₃) δ 178.1, 150.4, 130.4, 129.2, 125.8, 40.7, 34.6, 31.5 ppm.

2-(4-isobutylphenyl)acetic acid (2d)⁸.



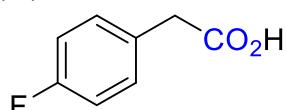
White solid (30.9 mg, 75% yield). Conformed the documental reports. ¹H NMR (400 MHz, CDCl₃) δ 7.20 (d, *J* = 8.0 Hz, 2H), 7.12 (d, *J* = 8.0 Hz, 2H), 3.62 (s, 2H), 2.46 (d, *J* = 7.2 Hz, 2H), 1.93 – 1.81 (m, 1H), 0.91 (d, *J* = 6.8 Hz, 6H) ppm; ¹³C NMR (100 MHz, CDCl₃) δ 178.2, 141.0, 130.6, 129.5, 129.2, 45.2, 40.8, 30.3, 22.5 ppm.

2-(4-methoxyphenyl)acetic acid (2e)².



White solid (24.6 mg, 74% yield). Conformed the documental reports. ¹H NMR (400 MHz, CDCl₃) δ 7.21 (d, *J* = 8.8 Hz, 2H), 6.88 (d, *J* = 8.4 Hz, 2H), 3.80 (s, 3H), 3.59 (s, 2H) ppm; ¹³C NMR (100 MHz, CDCl₃) δ 178.4, 159.0, 130.5, 125.5, 114.2, 55.4, 40.3 ppm.

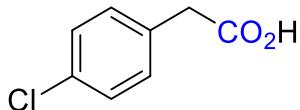
2-(4-fluorophenyl)acetic acid (2f)².



White solid (21.4 mg, 69% yield). Conformed the documental reports. ¹H NMR (400 MHz, CDCl₃) δ 7.25 (dd, *J* = 7.6, 4.6 Hz, 2H), 7.02 (t, *J* = 8.6 Hz, 2H), 3.63 (s, 2H)

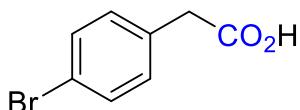
ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 177.4, 162.3 (d, $J = 244.5\text{Hz}$), 131.1 (d, $J = 8\text{ Hz}$), 129.1 (d, $J = 3.4\text{ Hz}$), 115.7 (d, $J = 21.3\text{ Hz}$), 40.2 ppm. ^{19}F NMR (376 MHz, CDCl_3) δ -120.0 ppm.

2-(4-chlorophenyl)acetic acid (2g)⁴.



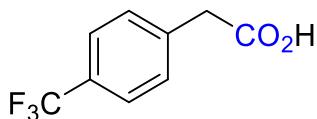
White solid (15.0 mg, 44% yield). Conformed the documental reports. ^1H NMR (400 MHz, CDCl_3) δ 7.31 (d, $J = 8.4\text{ Hz}$, 2H), 7.21 (d, $J = 8.4\text{ Hz}$, 2H), 3.62 (s, 2H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 177.6, 133.6, 131.8, 130.9, 129.0, 40.4 ppm.

2-(4-bromophenyl)acetic acid (2h)².



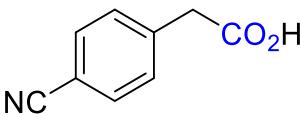
White solid (13.6 mg, 51% yield). Conformed the documental reports. ^1H NMR (400 MHz, CDCl_3) δ 7.46 (d, $J = 8.4\text{ Hz}$, 2H), 7.16 (d, $J = 8.4\text{ Hz}$, 2H), 3.60(s, 2H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 177.3, 132.3, 131.9, 131.2, 121.6, 40.5 ppm.

2-(4-(trifluoromethyl)phenyl)acetic acid (2i)³.



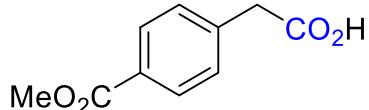
White solid (28.7 mg, 70% yield). Conformed the documental reports. ^1H NMR (400 MHz, CDCl_3) δ 7.60 (d, $J = 7.2\text{ Hz}$, 2H), 7.41 (d, $J = 7.2\text{ Hz}$, 2H), 3.72 (s, 2H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 177.82, 137.65, 130.3 (q, $J = 32.4\text{ Hz}$), 126.1 (q, $J = 3.7\text{ Hz}$), 124.6 (q, $J = 270.4\text{ Hz}$), 41.33 ppm.

2-(4-cyanophenyl)acetic acid (2j)³.



White solid (22.7 mg, 71% yield). Conformed the documental reports. ^1H NMR (400 MHz, CDCl_3) δ 7.64 (d, $J = 8.4\text{ Hz}$, 2H), 7.41 (d, $J = 8.0\text{ Hz}$, 2H), 3.72 (s, 2H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 174.9, 138.6, 132.6, 130.4, 118.7, 111.7, 40.9 ppm.

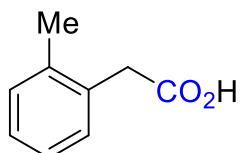
2-(4-(methoxycarbonyl)phenyl)acetic acid (2k)⁵.



White solid (25.3 mg, 65% yield). Conformed the documental reports. ^1H NMR (400 MHz, CDCl_3) δ 8.00 (d, $J = 8.4\text{ Hz}$, 2H), 7.36 (d, $J = 8.0\text{ Hz}$, 2H), 3.91 (s, 3H), 3.71

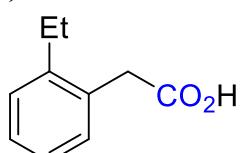
(s, 2H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 176.5, 167.0, 138.5, 130.1, 129.6, 129.5, 52.3, 41.0 ppm.

2-(*o*-tolyl)acetic acid (2l**)².**



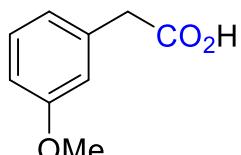
White solid (18.2 mg, 61% yield). Conformed the documental reports. ^1H NMR (400 MHz, CDCl_3) δ 7.24 – 7.16 (m, 4H), 3.68 (s, 2H), 2.34 (s, 3H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 178.0, 137.1, 132.1, 130.6, 130.4, 127.8, 126.3, 39.0, 19.7 ppm.

2-(2-ethylphenyl)acetic acid (2m**)⁷.**



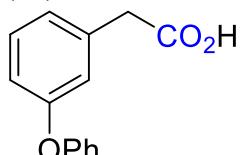
White solid (16.7 mg, 51% yield). Conformed the documental reports. ^1H NMR (400 MHz, CDCl_3) δ 7.26 – 7.14 (m, 4H), 3.68 (s, 2H), 2.66 (q, $J = 7.6$ Hz, 2H), 1.21 (t, $J = 7.6$ Hz, 3H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 178.0, 142.8, 131.4, 130.7, 128.8, 128.0, 126.2, 38.37, 25.9, 14.9 ppm.

2-(3-methoxyphenyl)acetic acid (2n**)³.**



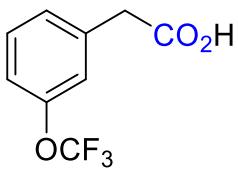
White solid (17.6 mg, 53% yield). Conformed the documental reports. ^1H NMR (400 MHz, CDCl_3) δ 7.30 – 7.28 (m, 1H), 6.91 – 6.85 (m, 3H), 3.83 (s, 3H), 3.66 (s, 2H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 177.4, 159.9, 134.8, 129.8, 121.9, 115.2, 113.0, 55.4, 41.2 ppm.

2-(3-phenoxyphenyl)acetic acid (2o**)³.**



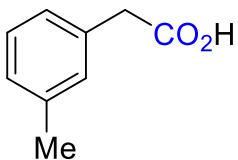
White solid (42.6 mg, 88% yield). Conformed the documental reports. ^1H NMR (400 MHz, CDCl_3) δ 7.37 – 7.28 (m, 3H), 7.12 (t, $J = 7.4$ Hz, 1H), 7.03 (d, $J = 8.0$ Hz, 3H), 6.97 – 6.92 (m, 2H), 3.64 (s, 2H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 177.7, 157.6, 157.0, 135.2, 130.0, 129.9, 124.3, 123.6, 119.9, 119.2, 117.7, 41.0 ppm.

2-(3-(trifluoromethoxy)phenyl)acetic acid (2p**).**



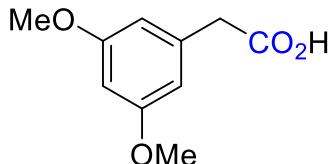
White solid (35.7 mg, 81% yield). Conformed the documental reports. ^1H NMR (400 MHz, CDCl_3) δ 7.46 (t, $J = 7.6$ Hz, 1H), 7.36 – 7.31 (m, 1H), 7.26 – 7.24 (m, 2H), 3.77 (s, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 177.40, 149.5, 135.4, 130.1, 128.0, 122.2, 120.6 (q, $J = 255.6$ Hz), 120.0, 40.74 ppm. HRMS (ESI) m/z: [M-H] $^-$ Calcd for $\text{C}_9\text{H}_6\text{F}_3\text{O}_3^-$ 219.0275; Found 219.0270.

2-(m-tolyl)acetic acid (2q)².



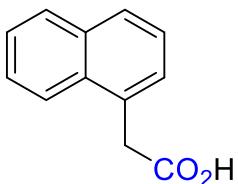
White solid (25.2 mg, 84% yield). Conformed the documental reports. ^1H NMR (400 MHz, CDCl_3) δ 7.24 – 7.21 (m, 1H), 7.10 – 7.08 (m, 3H), 3.61 (s, 2H), 2.34 (s, 3H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 177.4, 138.5, 133.3, 130.3, 128.7, 128.3, 126.5, 41.1, 21.5 ppm.

2-(3, 5-dimethoxyphenyl)acetic acid (2r)⁴.



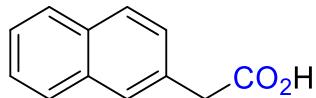
White solid (25.9 mg, 66% yield). Conformed the documental reports. ^1H NMR (400 MHz, CDCl_3) δ 6.44 (d, $J = 2.4$ Hz, 2H), 6.39 (t, $J = 2.2$ Hz, 1H), 3.78 (s, 6H), 3.58 (s, 2H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 177.3, 161.1, 135.4, 107.6, 99.6, 55.5, 41.4 ppm.

2-(naphthalen-1-yl)acetic acid (2s).⁹



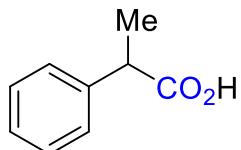
White solid (23.5 mg, 63% yield). Conformed the documental reports. ^1H NMR (400 MHz, CDCl_3) δ 7.96 (d, $J = 8.0$ Hz, 1H), 7.87 (d, $J = 7.6$ Hz, 1H), 7.82 (d, $J = 8.0$ Hz, 1H), 7.56 – 7.48 (m, 2H), 7.46 – 7.40 (m, 2H), 4.09 (s, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 177.8, 133.9, 132.1, 129.9, 128.9, 128.5, 128.4, 126.7, 126.0, 125.6, 123.8, 38.9 ppm.

2-(naphthalen-2-yl)acetic acid (2t)².



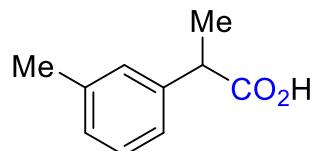
White solid (19.7 mg, 53% yield). Conformed the documental reports. ^1H NMR (400 MHz, CDCl_3) δ 7.81 (t, $J = 8.0$ Hz, 3H), 7.74 (s, 1H), 7.49 – 7.44 (m, 2H), 7.41 (dd, $J = 8.4, 1.6$ Hz, 1H), 3.82 (s, 2H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 177.3, 133.6, 132.7, 130.9, 128.5, 128.3, 127.8, 127.8, 127.4, 126.4, 126.1, 41.3 ppm.

2-phenylpropanoic acid (2u)².



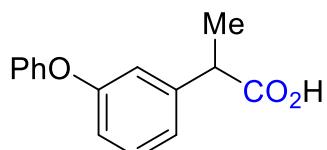
Colorless oil (24.5 mg, 82% yield). Conformed the documental reports. ^1H NMR (400 MHz, CDCl_3) δ 7.34 (d, $J = 4.0$ Hz, 4H), 7.30 – 7.27 (m, 1H), 3.76 (q, $J = 7.2$ Hz, 1H), 1.53 (d, $J = 7.2$ Hz, 3H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 180.9, 139.9, 128.8, 127.8, 127.5, 45.5, 18.2 ppm.

2-(*m*-tolyl)propanoic acid (2v)⁵.



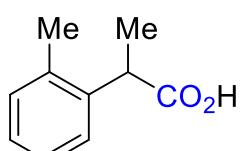
Colorless oil (15.7 mg, 48% yield). Conformed the documental reports. ^1H NMR (400 MHz, CDCl_3) δ 7.23 (t, $J = 7.6$ Hz, 1H), 7.14 – 7.08 (m, 3H), 3.71 (q, $J = 7.2$ Hz, 1H), 2.36 (s, 3H), 1.51 (d, $J = 7.2$ Hz, 3H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 180.8, 139.9, 138.5, 128.7, 128.5, 128.3, 124.8, 45.4, 21.6, 18.2 ppm.

2-(3-phenoxyphenyl)propanoic acid (2w)³.



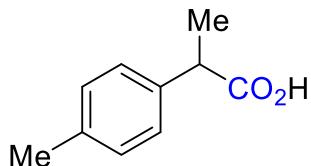
White solid (40.6 mg, 83% yield). Conformed the documental reports. ^1H NMR (400 MHz, CDCl_3) δ 7.31 – 7.27 (m, 2H), 7.24 – 7.21 (m, 1H), 7.08 – 7.01 (m, 2H), 6.99 – 6.96 (m, 3H), 6.86 – 6.84 (m, 1H), 3.68 (q, $J = 7.2$ Hz, 1H), 1.46 (d, $J = 7.2$ Hz, 3H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 180.3, 157.6, 157.1, 141.8, 130.0, 129.9, 123.5, 122.5, 119.1, 118.4, 117.6, 45.3, 18.2 ppm.

2-(*o*-tolyl)propanoic acid (2x)⁵.



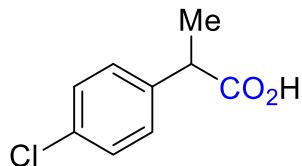
White solid (24.3 mg, 74% yield). Conformed the documental reports. ^1H NMR (400 MHz, CDCl_3) δ 7.30 (d, $J = 6.8$ Hz, 1H), 7.23 – 7.17 (m, 3H), 3.99 (q, $J = 7.2$ Hz, 1H), 2.39 (s, 3H), 1.50 (d, $J = 6.8$ Hz, 3H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 180.9, 138.5, 136.1, 130.7, 127.3, 126.7, 126.6, 41.2, 19.8, 17.7 ppm.

2-(*p*-tolyl)propanoic acid (2y)².



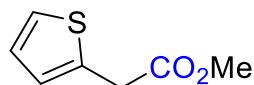
Colorless oil (17.3 mg, 53% yield). Conformed the documental reports. ^1H NMR (400 MHz, CDCl_3) δ 7.22 (d, $J = 8.0$ Hz, 2H), 7.15 (d, $J = 8.0$ Hz, 2H), 3.71 (q, $J = 7.2$ Hz, 1H), 2.33 (s, 3H), 1.50 (d, $J = 7.2$ Hz, 3H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 181.0, 137.2, 136.9, 129.5, 127.6, 45.0, 21.2, 18.2 ppm.

2-(4-chlorophenyl)propanoic acid (2z)⁶.



White solid (22.9 mg, 62% yield). Conformed the documental reports. ^1H NMR (400 MHz, CDCl_3) δ 7.31 (d, $J = 8.8$ Hz, 2H), 7.26 (d, $J = 8.8$ Hz, 2H), 3.72 (q, $J = 7.2$ Hz, 1H), 1.50 (d, $J = 7.2$ Hz, 3H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 180.3, 138.3, 133.5, 129.2, 129.0, 44.9, 18.2 ppm.

methyl 2-(thiophen-2-yl)acetate. (2a')¹⁰



Brown oily liquid (11.0 mg, 35% yield). ^1H NMR (400 MHz, CDCl_3) δ 7.29 (dd, $J = 4.8, 2.8$ Hz, 1H), 7.15 – 7.14 (d, $J = 1.8$ Hz, 1H), 7.04 (d, $J = 4.8$ Hz, 1H), 3.71 (s, 3H), 3.67 (s, 2H) ppm. ^{13}C NMR (100 MHz, CDCl_3) δ 171.7, 133.7, 128.6, 125.9, 123.0, 52.2, 35.8 ppm.

8. References.

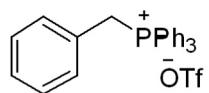
- [1] M. S. M. Pearson, D. R. Carbery, *J. Org. Chem.* **2009**, *74*, 5320-5325.
- [2] D.-T. Yang, M. Zhu, Z. J. Schiffer, K. Williams, X. Song, X. Liu, K. Manthiram, *ACS Catal.* **2019**, *9*, 4699-4705.
- [3] C.-K. Ran, Y.-N. Niu, L. Song, M.-K. Wei, Y.-F. Cao, S.-P. Luo, Y.-M. Yu, L.-L. Liao, D.-G. Yu, *ACS Catal.* **2022**, *12*, 18-24.
- [4] J.-S. Zhong, Z.-X. Yang, C.-L. Ding, Y.-F. Huang, Y. Zhao, H. Yan, K.-Y. Ye, *J.*

- Org. Chem.* **2021**, *86*, 16162-16170.
- [5] Z. Huang, Y. Cheng, X. Chen, H.-F. Wang, C.-X. Du, Y. Li, *Chem. Commun.* **2018**, *54*, 3967-3970.
- [6] J. Li, J. Shen, C. Xia, Y. Wang, D. Liu, W. Zhang, *Org. Lett.* **2016**, *18*, 2122-2125.
- [7] J. M. Burns, E. H. Krenske, R. P. McGeary, *Eur. J. Org. Chem.* **2017**, *2017*, 252-256.
- [8] L. Tao, W. Yang, W. Zhao, *ChemistrySelect* **2021**, *6*, 8532-8536.
- [9] C. Li, P. Zhao, R. Li, B. Zhang, W. Zhao, *Angew. Chem. Int. Ed.* **2020**, *59*, 10913-10917.
- [10] Y. Li, Z. Wang, X.-F. Wu, *ACS Catal.* **2018**, *8*, 738-741.

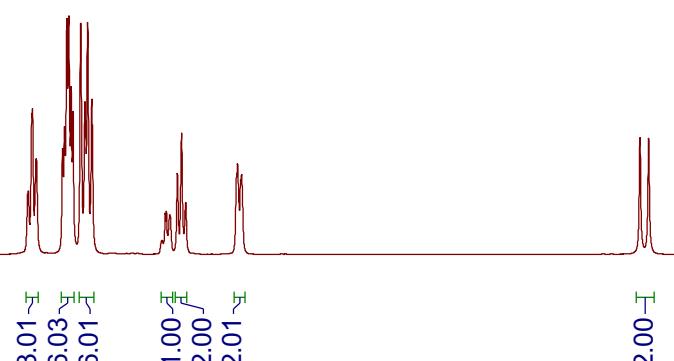
9. NMR Spectra of Coupled Products

7.909
7.906
7.761
7.750
7.742
7.731
7.722
7.688
7.669
7.656
7.638
7.306
7.300
7.285
7.251
7.232
7.213
6.982
6.979
6.965
6.960

5.158
5.119



(**1a**, ^1H NMR, 400 MHz, $\text{DMSO}-d_6$)

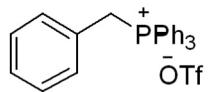


9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5

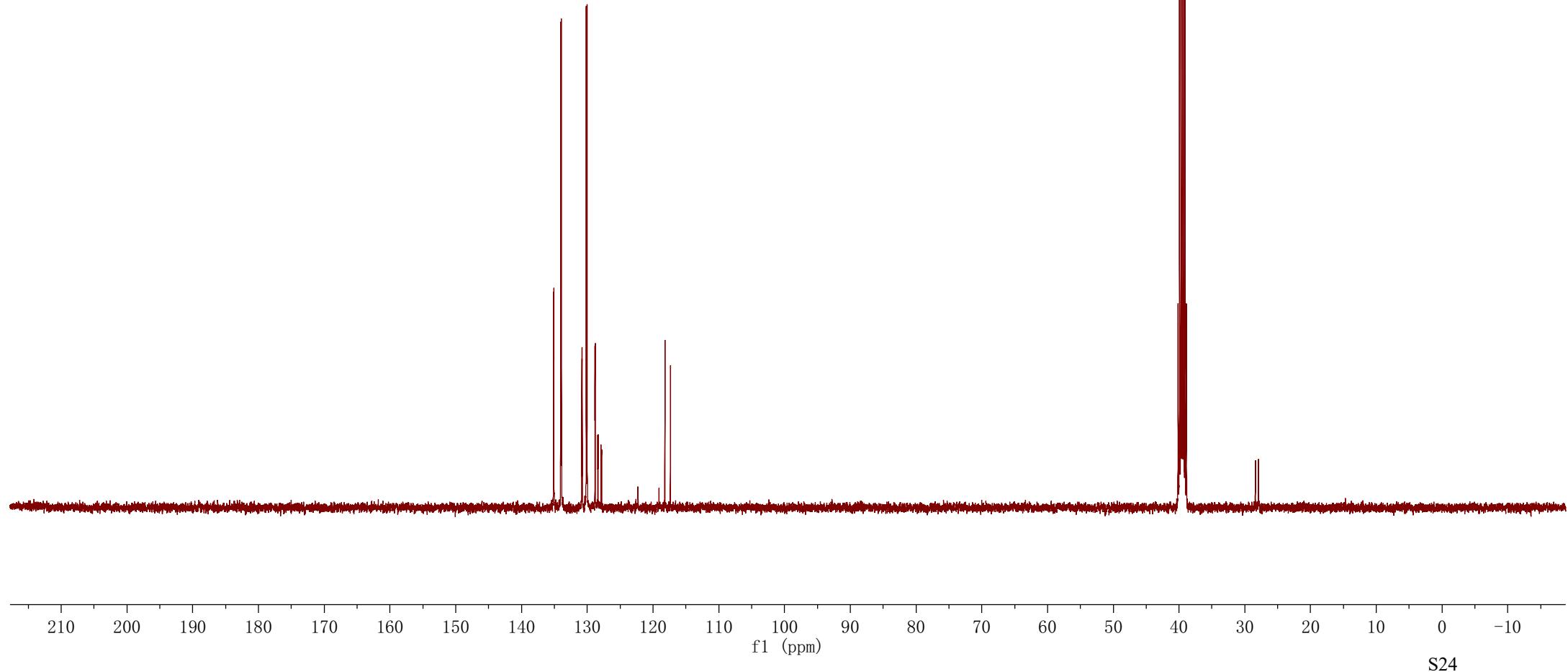
f1 (ppm)

135.112
135.084
134.028
133.929
130.821
130.764
130.151
130.028
128.823
128.794
128.389
128.353
127.899
127.813
122.285
119.079
118.199
117.349

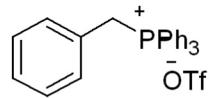
28.385
27.919



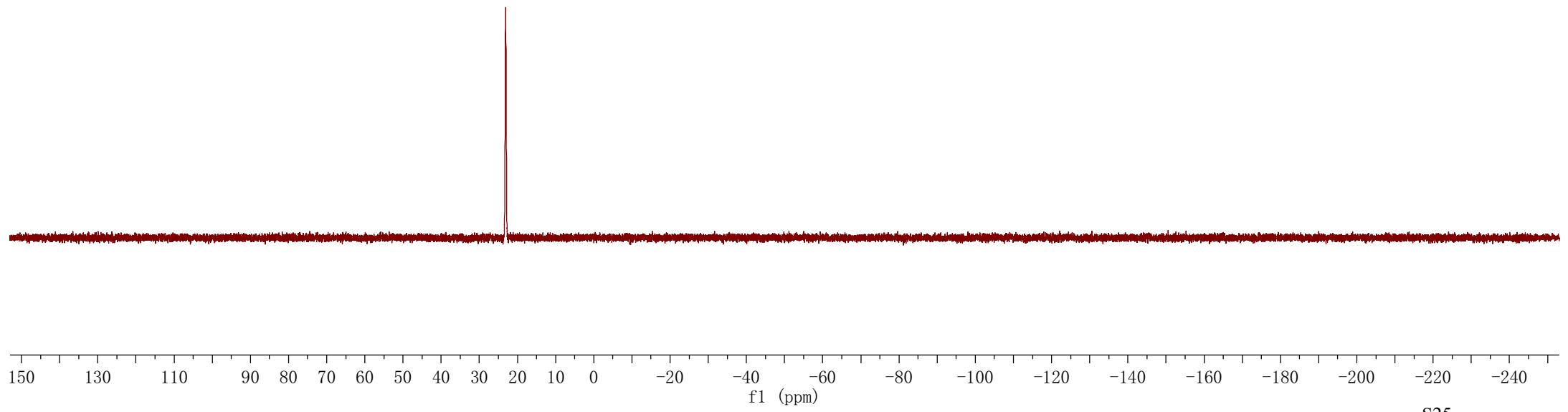
(1a, ¹³C NMR, 100 MHz, DMSO-*d*₆)



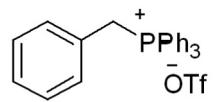
-23.087



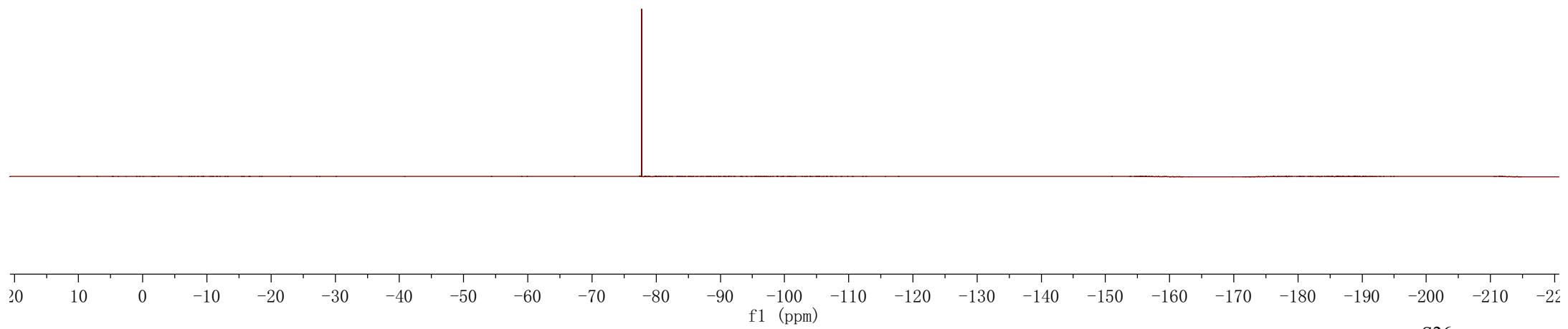
(**1a**, ^{31}P NMR, 162 MHz, $\text{DMSO}-d_6$)



-77.729



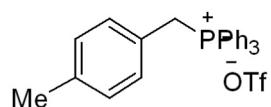
(1a, ¹⁹F NMR, 376 MHz, DMSO-d₆)



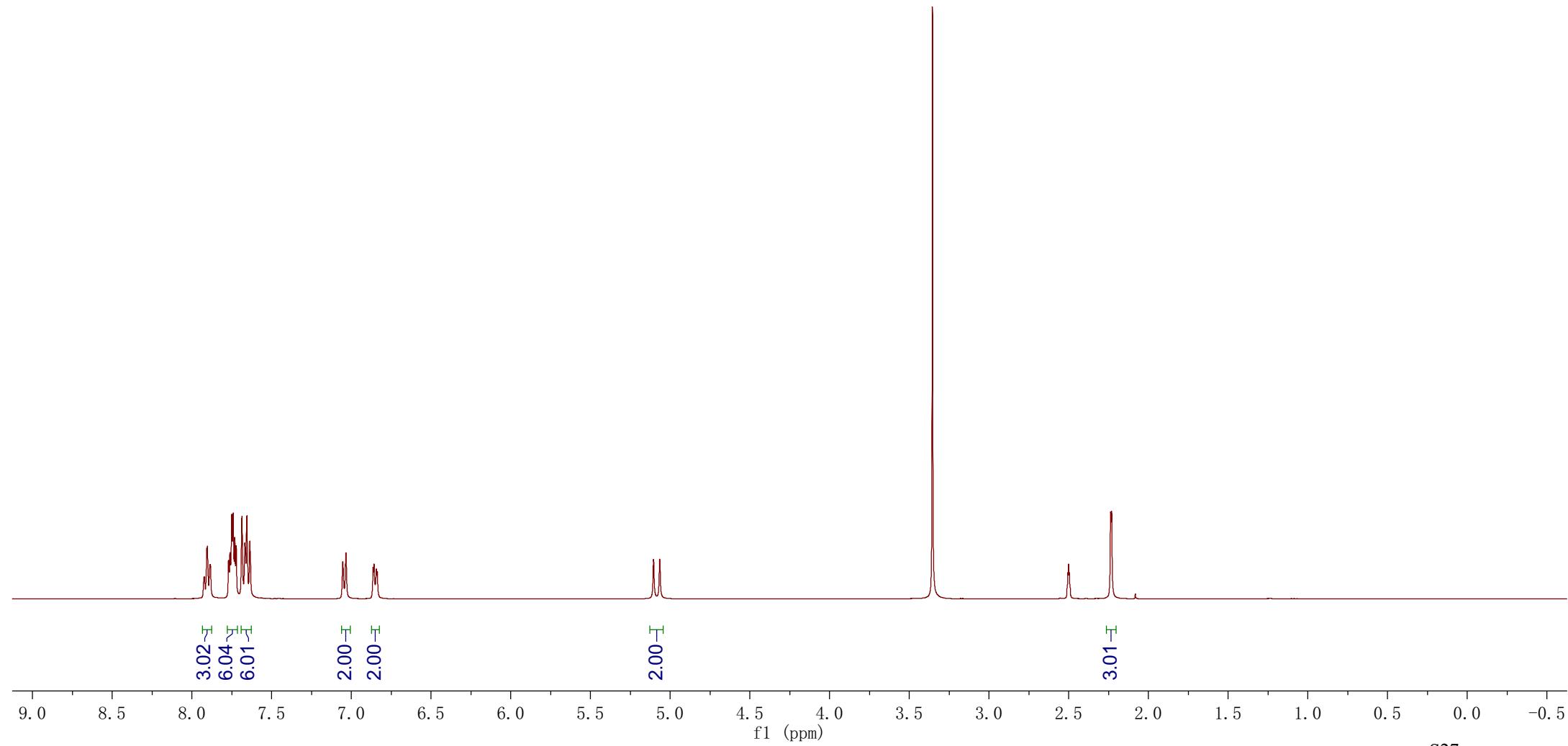
7.907
7.902
7.750
7.741
7.731
7.722
7.686
7.667
7.664
7.655
7.636
7.634
7.633
7.034
6.863
6.857
6.843
6.837

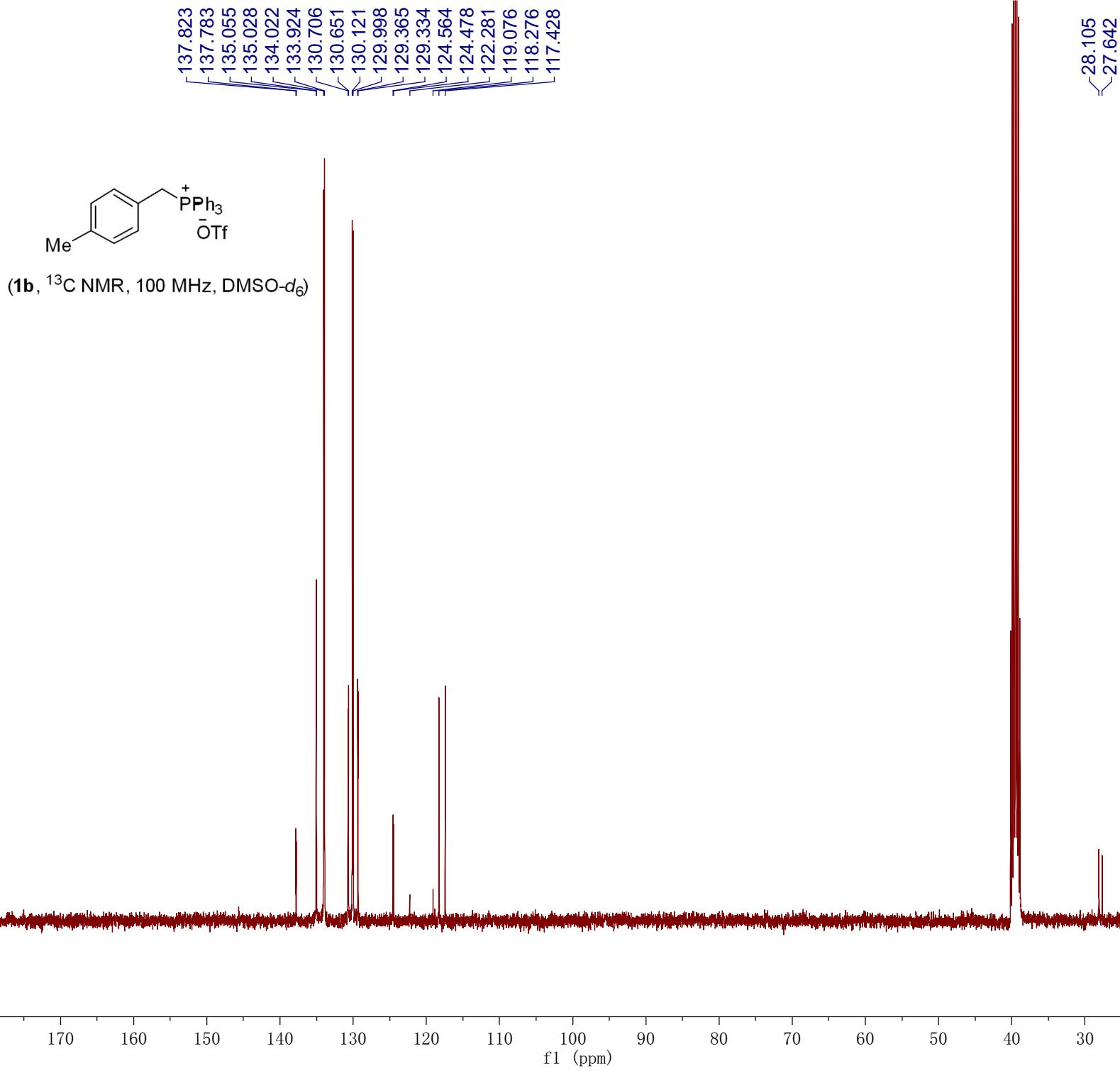
5.104
5.065

2.236
2.231

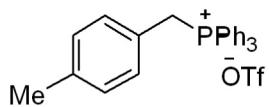


(**1b**, ^1H NMR, 400 MHz, DMSO- d_6)

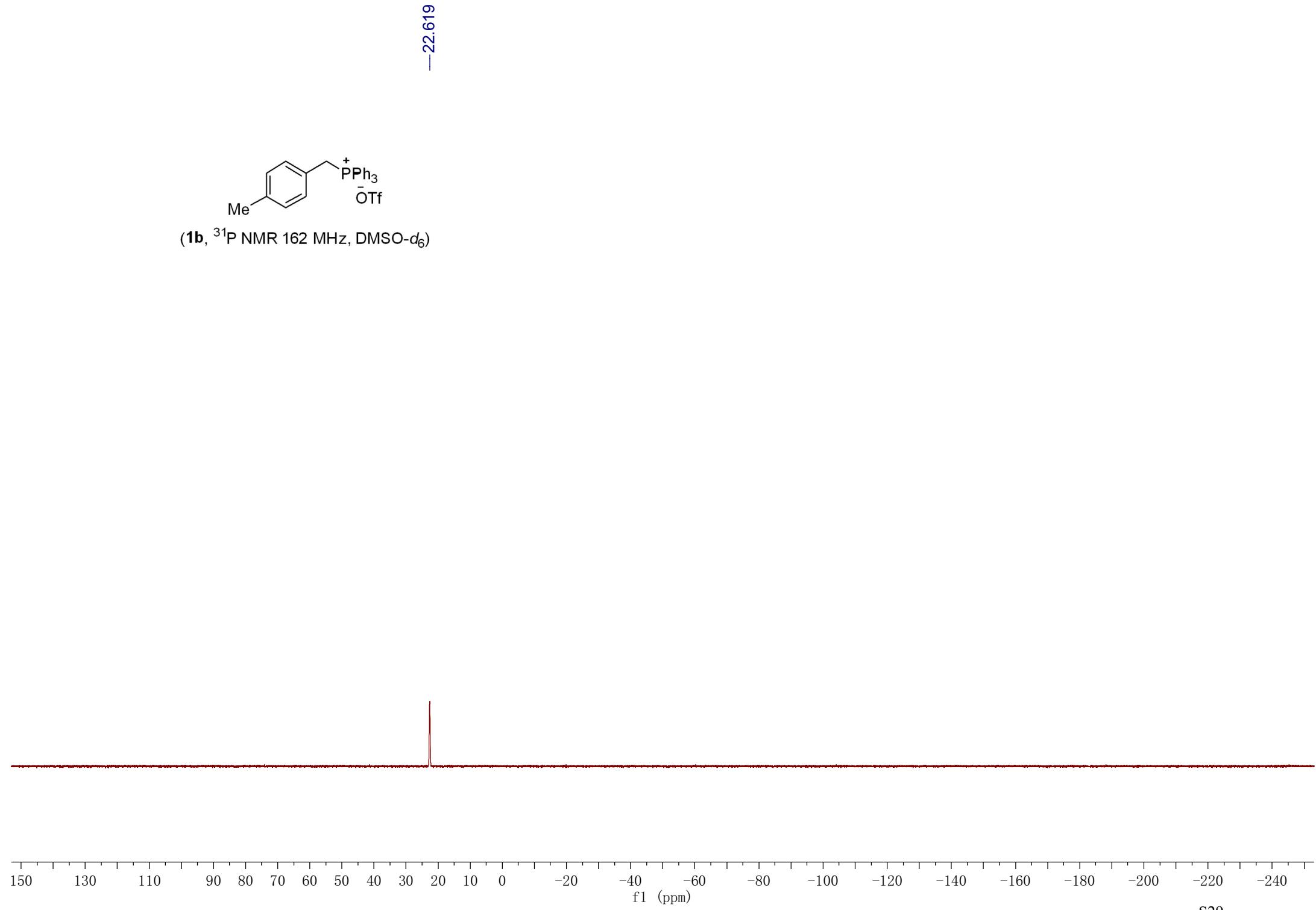




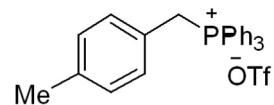
-22.619



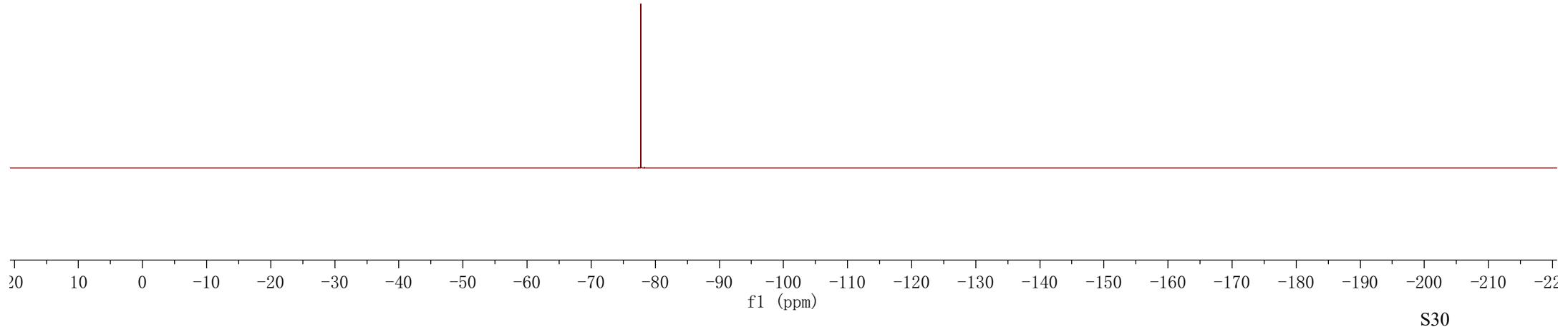
(**1b**, ^{31}P NMR 162 MHz, $\text{DMSO}-d_6$)



-77.722



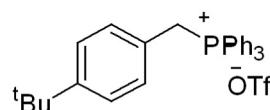
(**1b**, ^{19}F NMR, 376 MHz, DMSO- d_6)



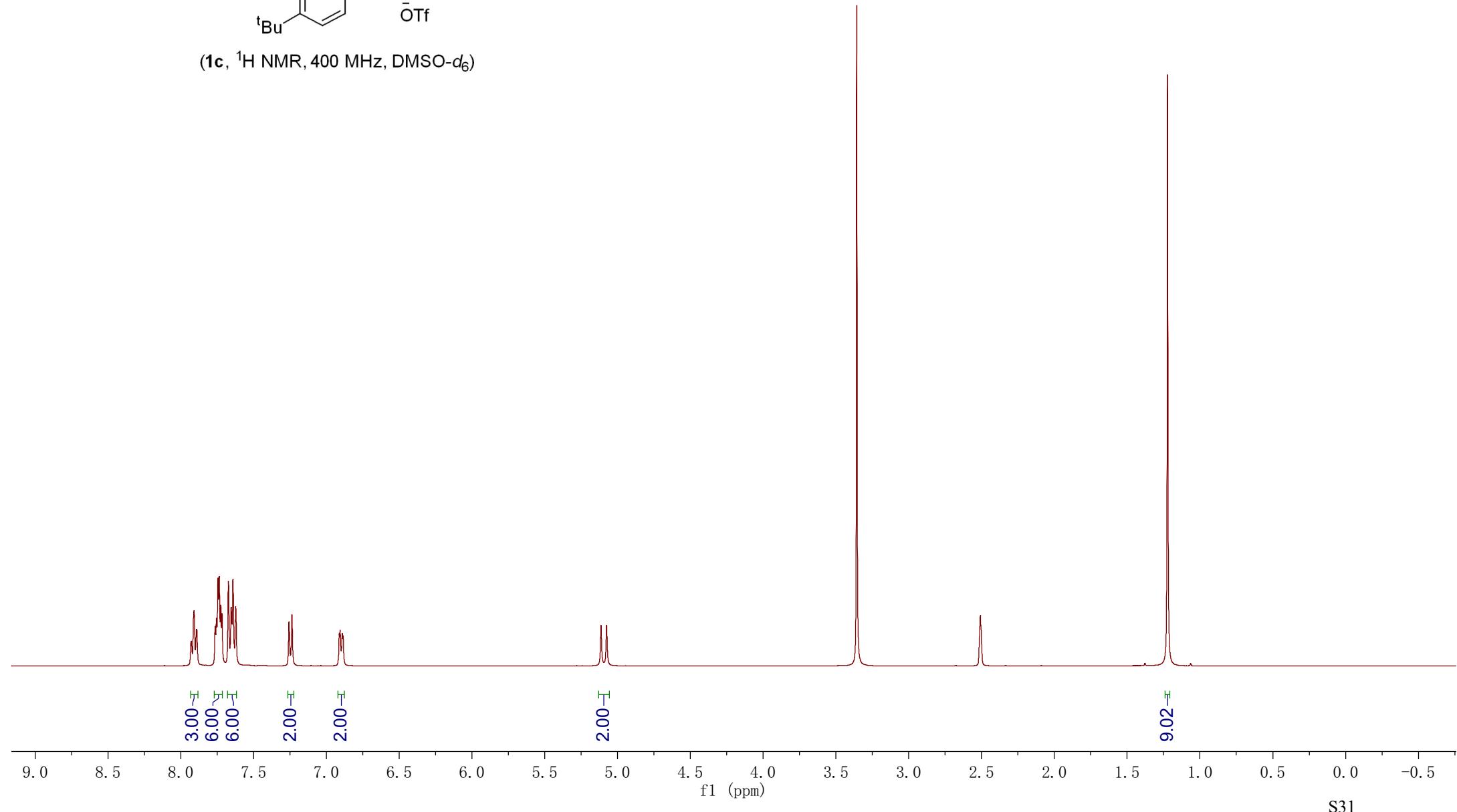
7.911
7.908
7.764
7.755
7.744
7.736
7.725
7.717
7.714
7.673
7.654
7.642
7.623
7.236
6.912
6.906
6.891
6.885

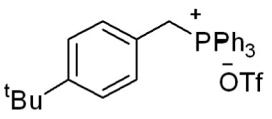
5.114
5.075

-1.223

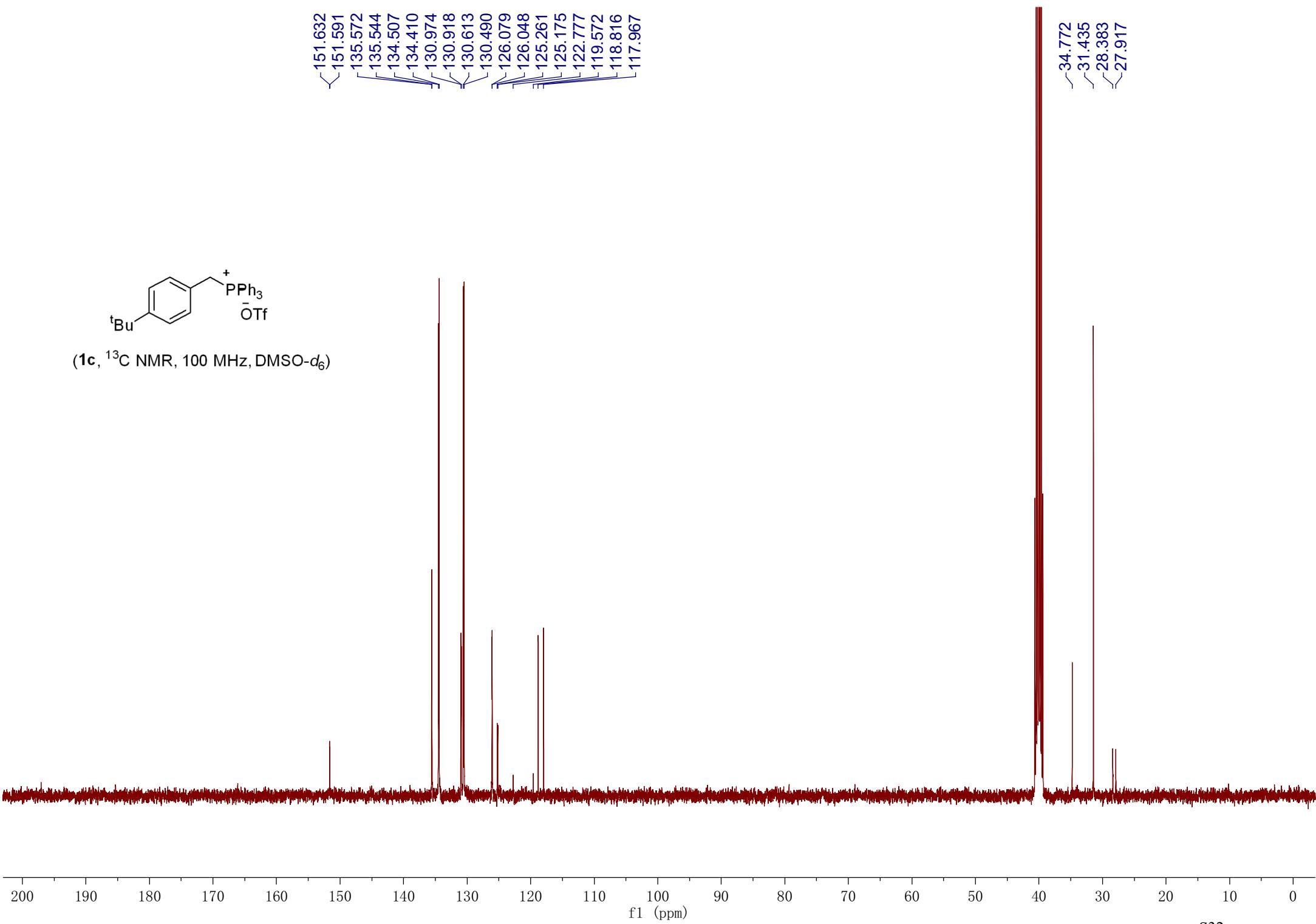


(**1c**, ^1H NMR, 400 MHz, DMSO- d_6)

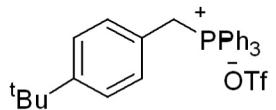




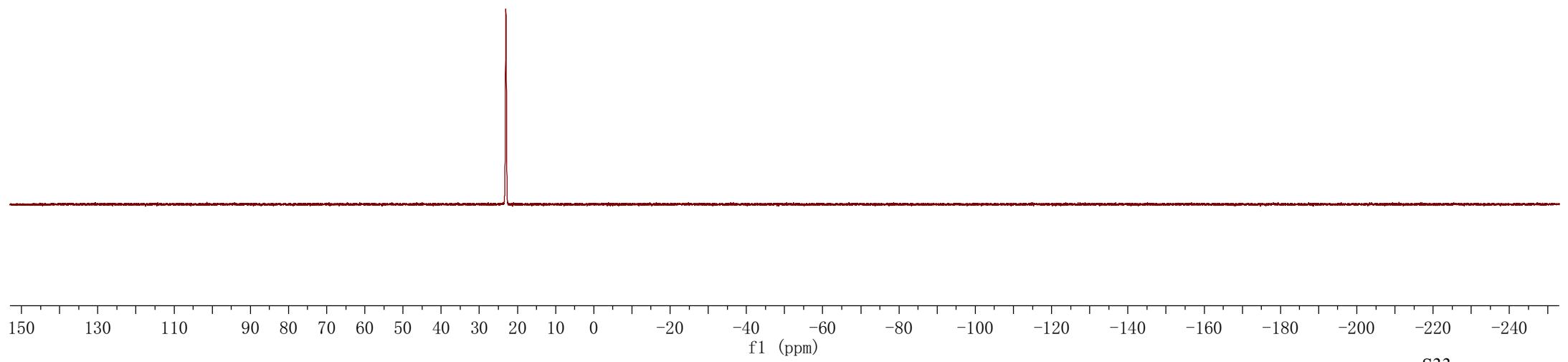
(**1c**, ^{13}C NMR, 100 MHz, $\text{DMSO}-d_6$)



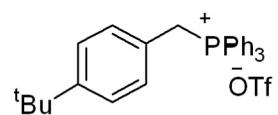
-23.030



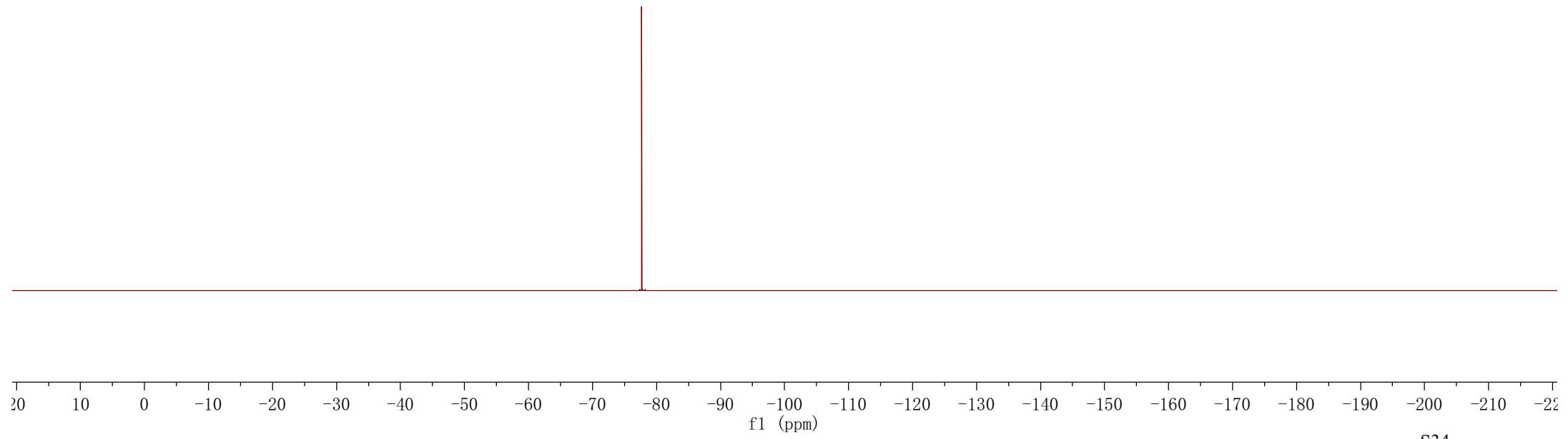
(**1c**, ^{31}P NMR, 162 MHz, $\text{DMSO}-d_6$)



-77.689



(**1c**, ^{19}F NMR, 376 MHz, DMSO- d_6)

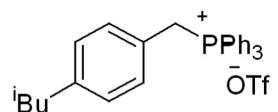


7.913
7.910
7.761
7.752
7.741
7.732
7.722
7.713
7.670
7.650
7.639
7.618
7.619
7.010
6.885
6.880
6.865
6.860

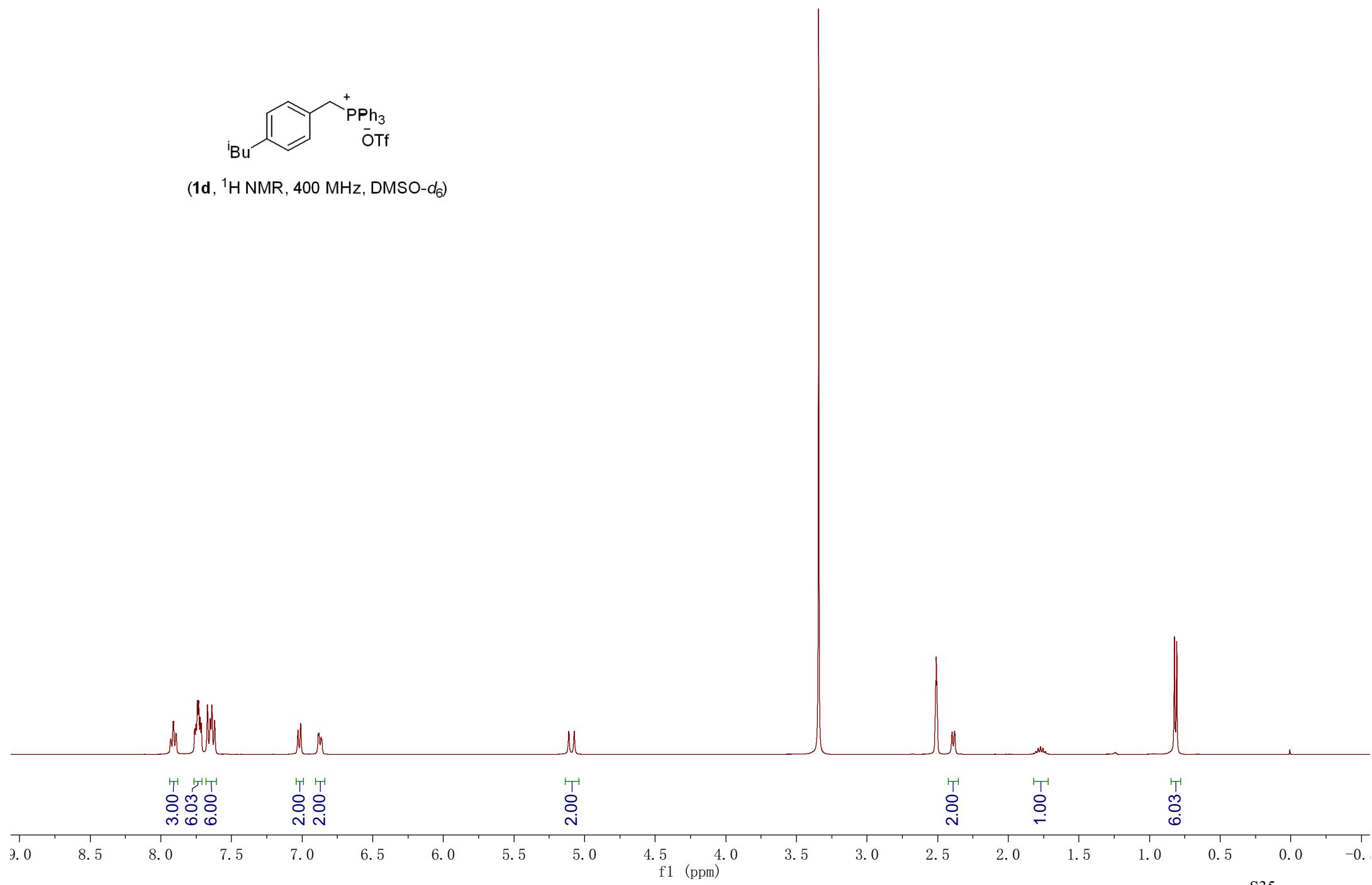
5.111
5.073

2.398
2.381
1.805
1.788
1.771
1.755
1.738

0.824
0.808



(**1d**, ^1H NMR, 400 MHz, $\text{DMSO}-d_6$)

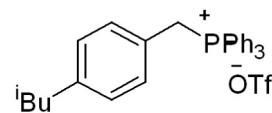


HF-Y-1d-C
HF-Y-1d-C

142.420
142.379
136.016
135.990
134.964
134.867
131.493
131.438
131.042
130.918
130.319
130.288
125.969
125.885
123.226
120.023
119.216
118.368

-44.907

30.364
28.979
28.517
22.881



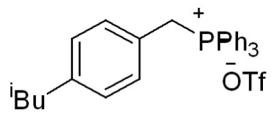
(**1d**, ^{13}C NMR, 100 MHz, $\text{DMSO}-d_6$)

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

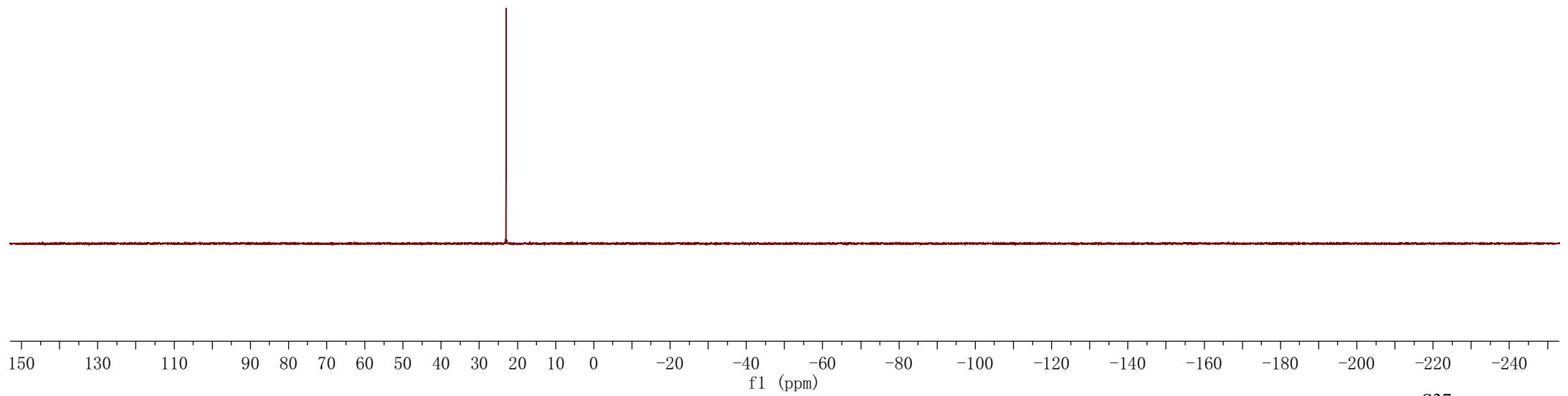
f1 (ppm)

S36

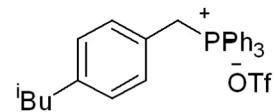
-22.994



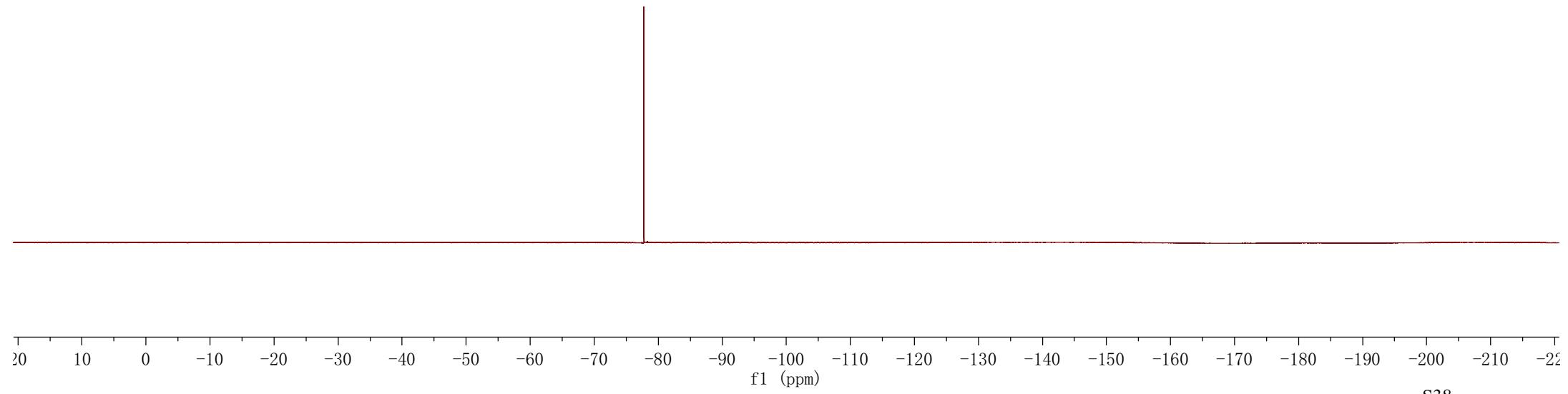
(**1d**, ^{31}P NMR, 162 MHz, DMSO- d_6)



—77.740



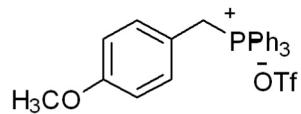
(**1d**, ^{19}F NMR, 376 MHz, DMSO- d_6)



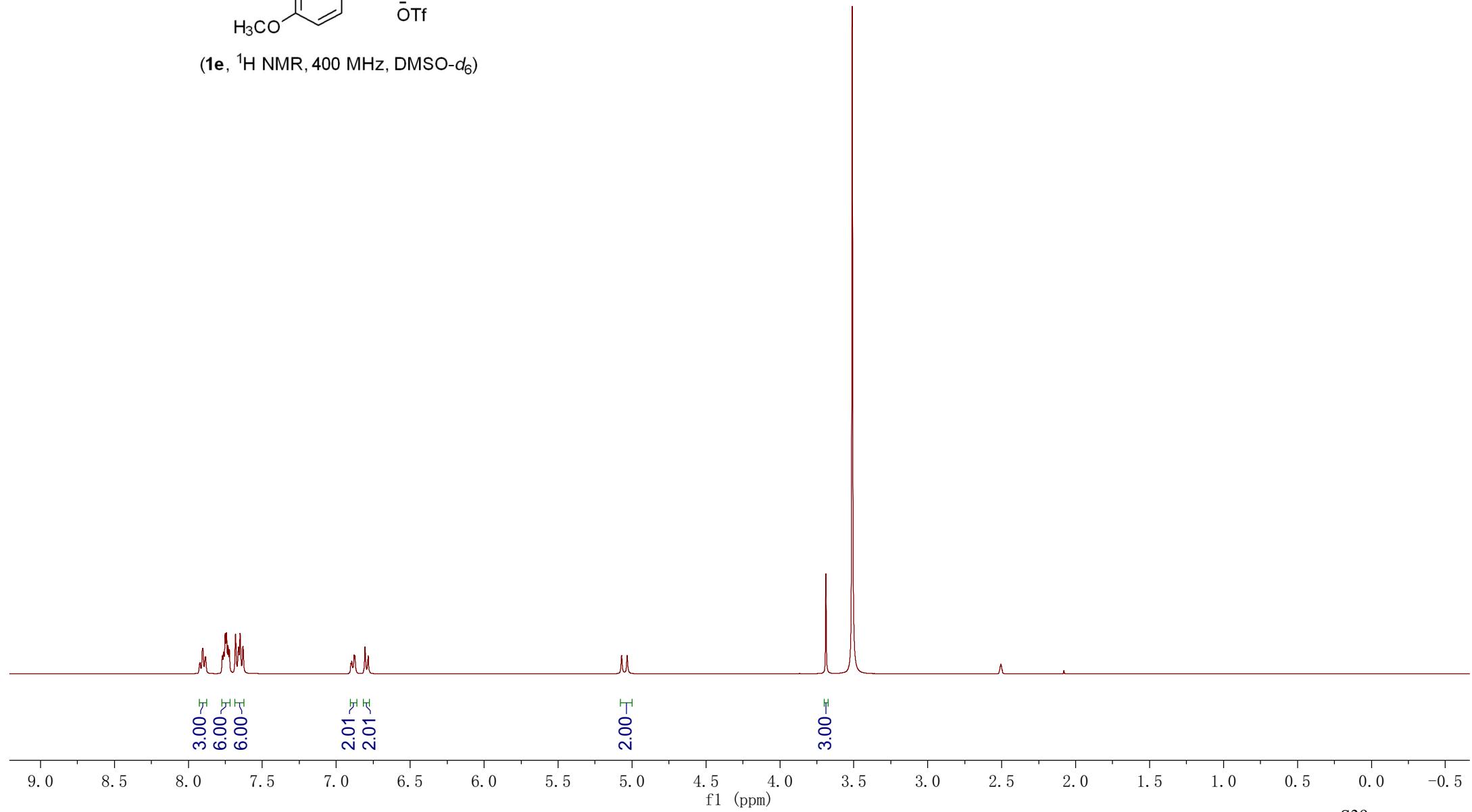
7.921
7.902
7.885
7.771
7.762
7.751
7.742
7.732
7.723
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7.662
7.650
7.631
6.901
6.895
6.879
6.873
6.806
6.784

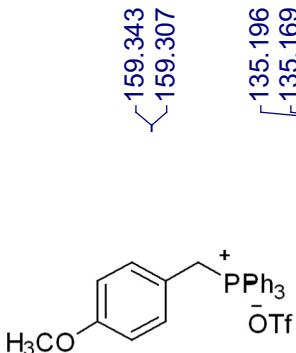
5.070
5.033

—3.689

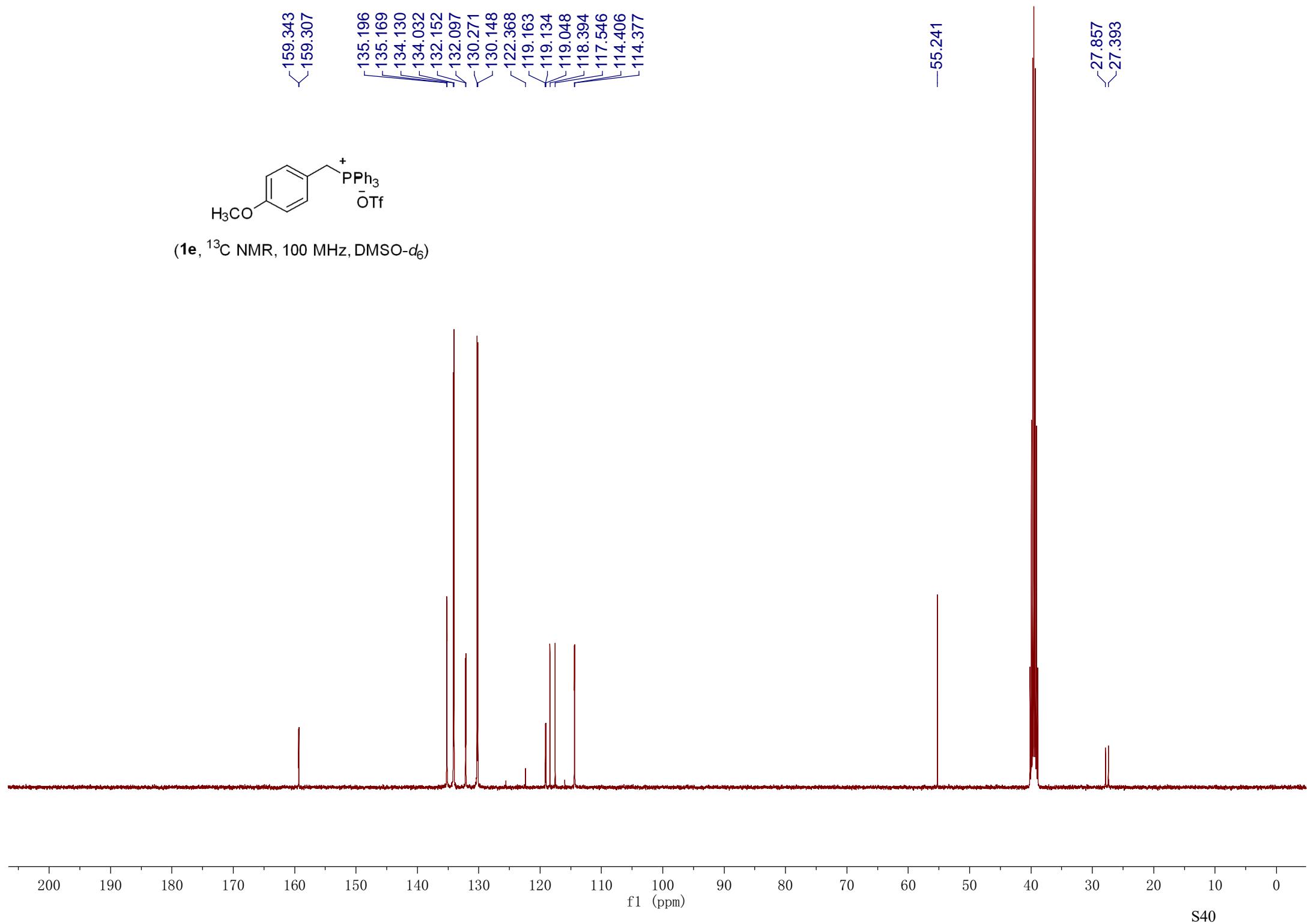


(**1e**, ^1H NMR, 400 MHz, DMSO- d_6)

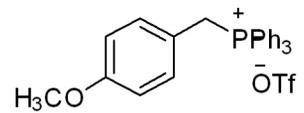




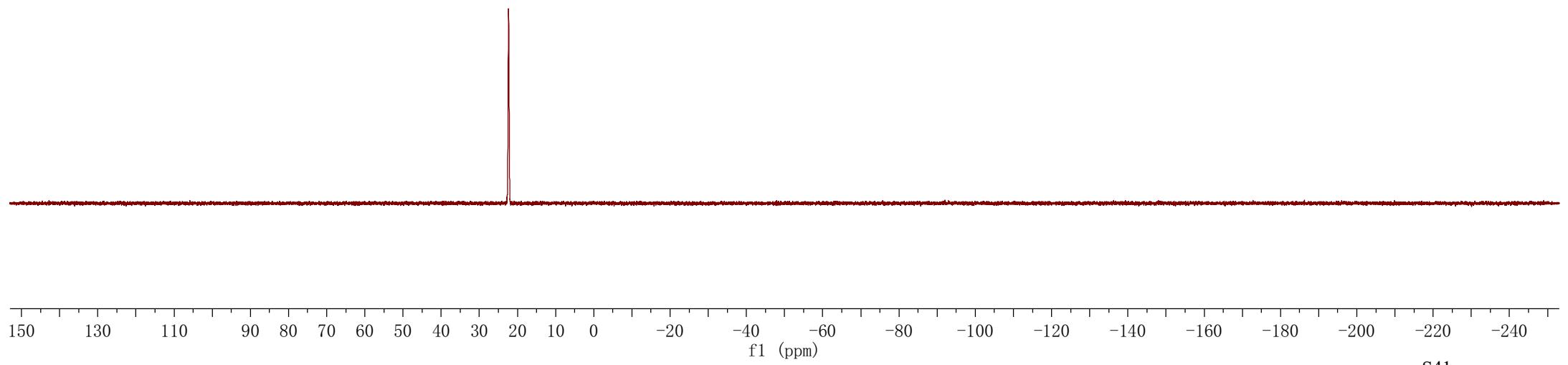
(**1e**, ^{13}C NMR, 100 MHz, $\text{DMSO}-d_6$)



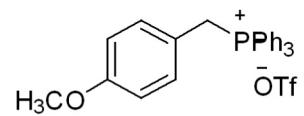
-22.320



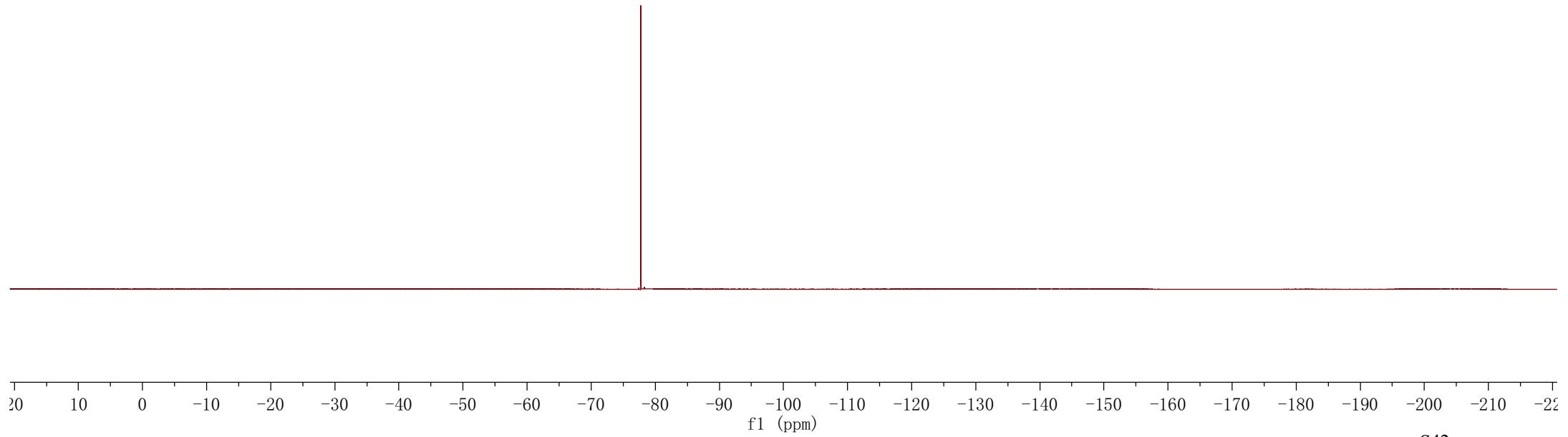
(**1e**, ^{31}P NMR, 162 MHz, $\text{DMSO}-d_6$)



—77.741

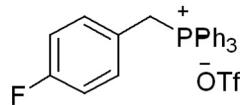


(**1e**, ^{19}F NMR, 376 MHz, $\text{DMSO}-d_6$)

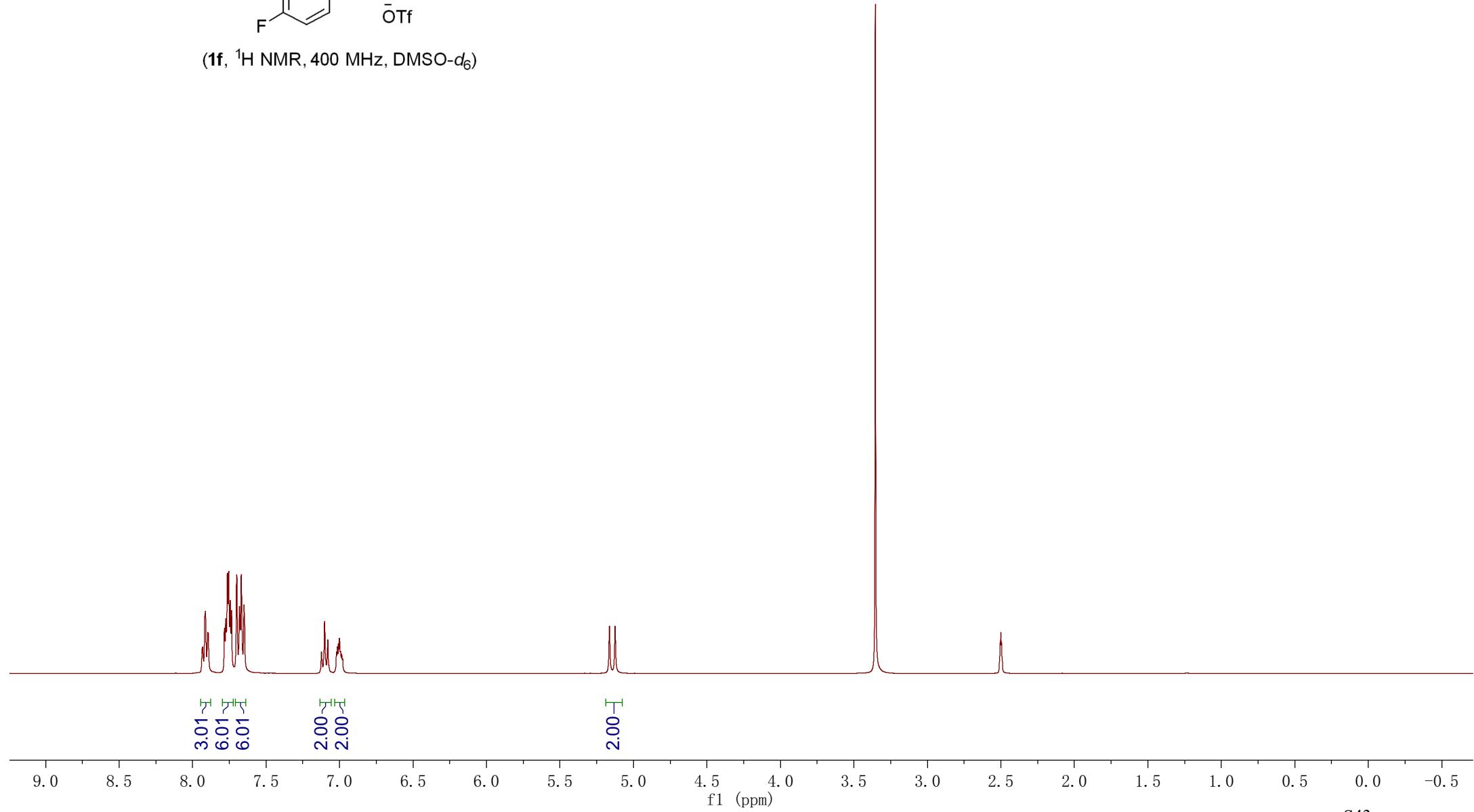


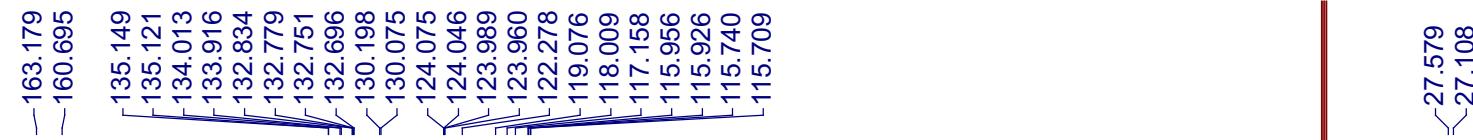
7.916
7.912
7.783
7.774
7.763
7.754
7.744
7.735
7.699
7.681
7.678
7.668
7.659
7.623
7.102
7.080
7.021
7.015
7.008
7.001
6.993
6.986
6.980

5.164
5.125

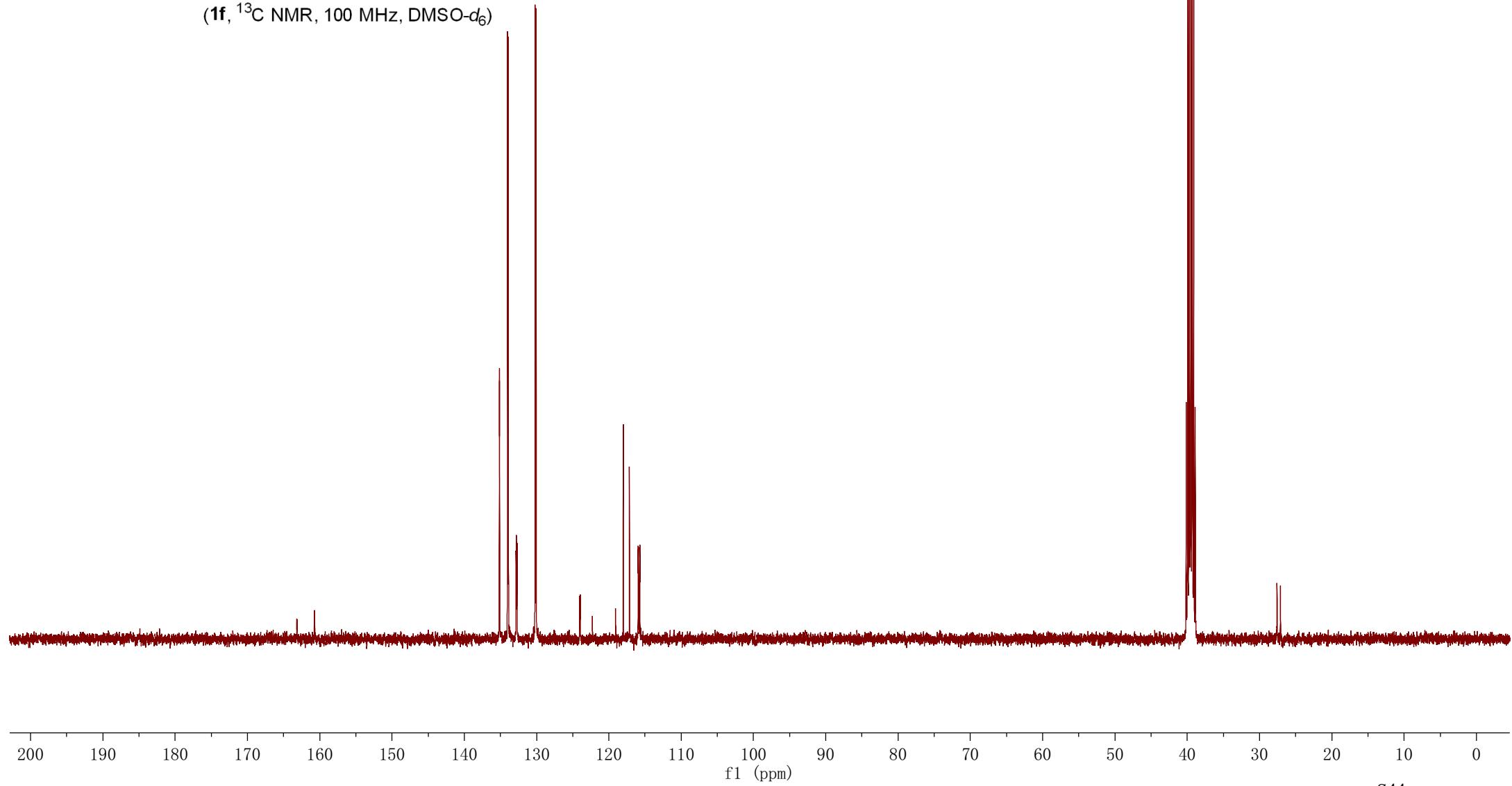
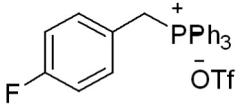


(**1f**, ^1H NMR, 400 MHz, DMSO- d_6)

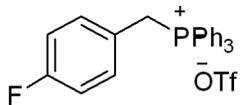




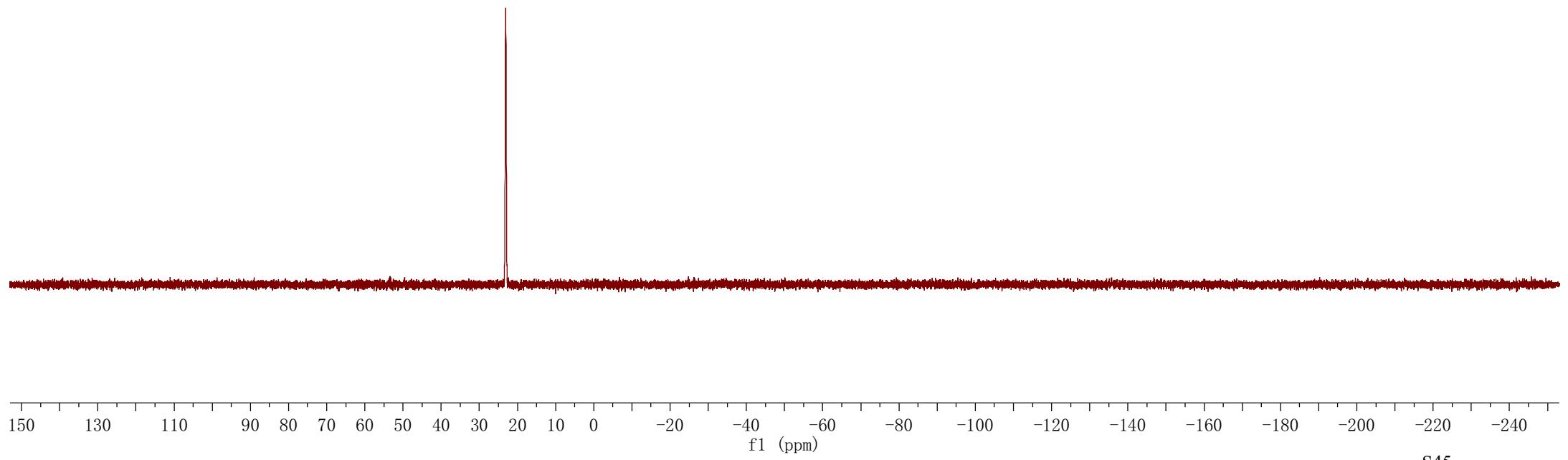
(**1f**, ¹³C NMR, 100 MHz, DMSO-*d*₆)

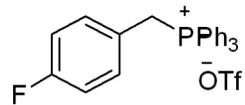


-23.077



(**1f**, ^{31}P NMR, 162 MHz, DMSO- d_6)

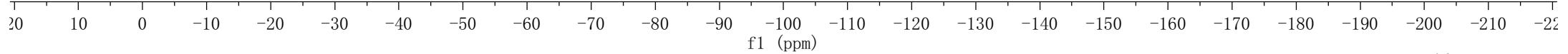




(**1f**, ^{19}F NMR, 376 MHz, DMSO- d_6)

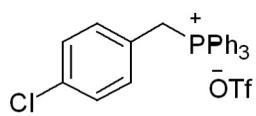
—77.725

—113.137

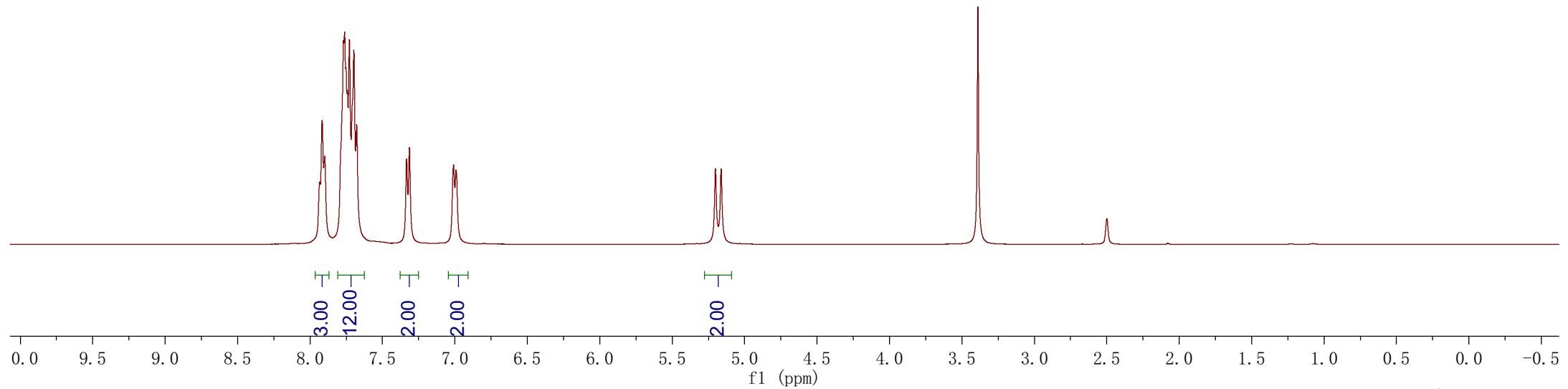


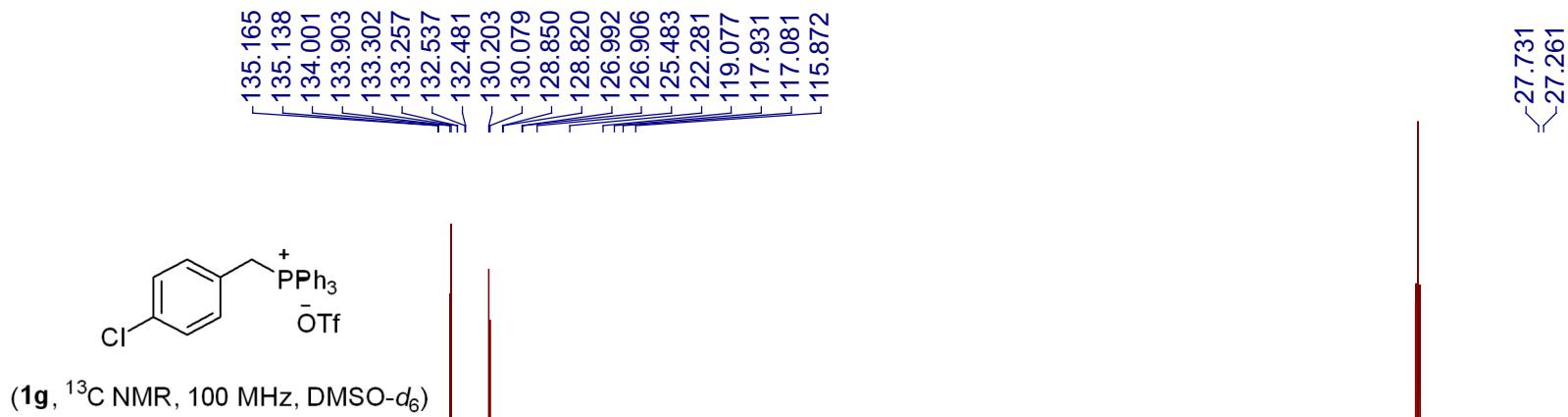
7.933
7.916
7.899
7.769
7.761
7.752
7.742
7.728
7.698
7.678
7.334
7.314
7.009
6.992

5.202
5.162

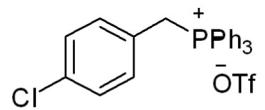


(**1g**, ^1H NMR, 400 MHz, DMSO- d_6)

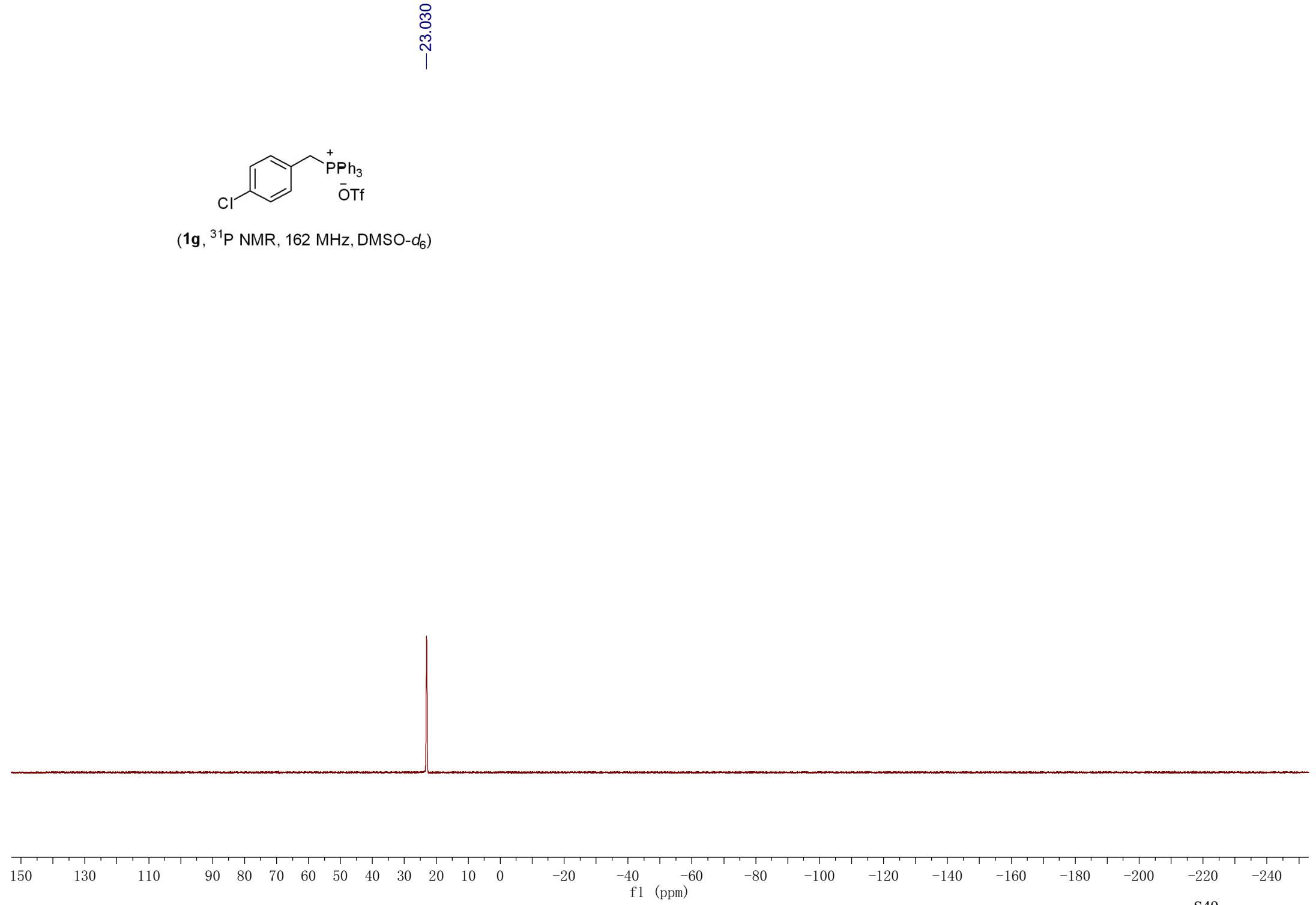




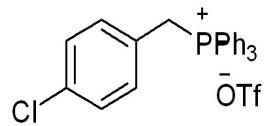
-23.030



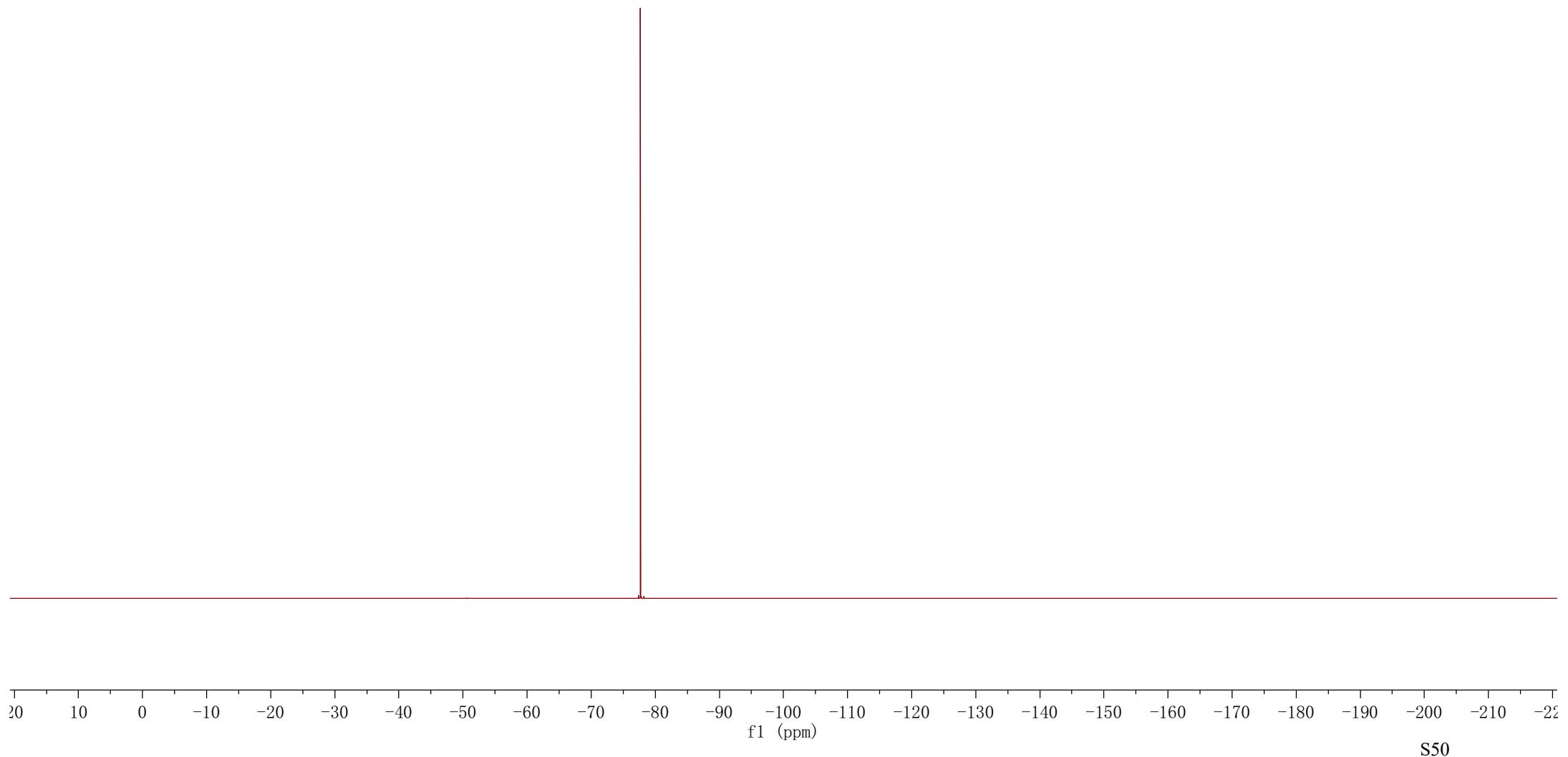
(**1g**, ^{31}P NMR, 162 MHz, DMSO- d_6)



-77.689

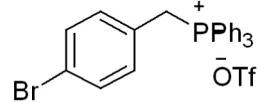


(**1g**, ¹⁹F NMR, 376 MHz, DMSO-d₆)

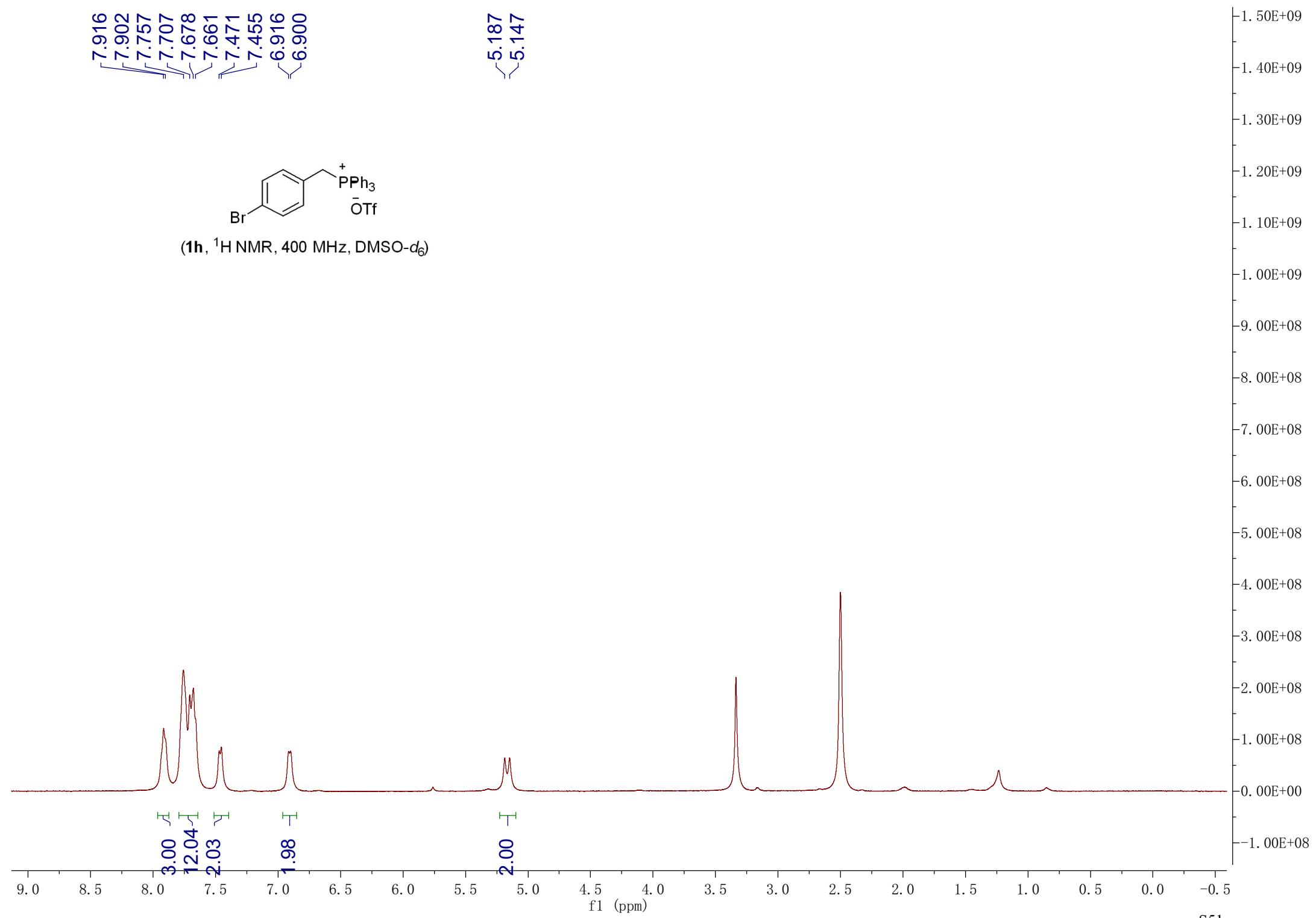


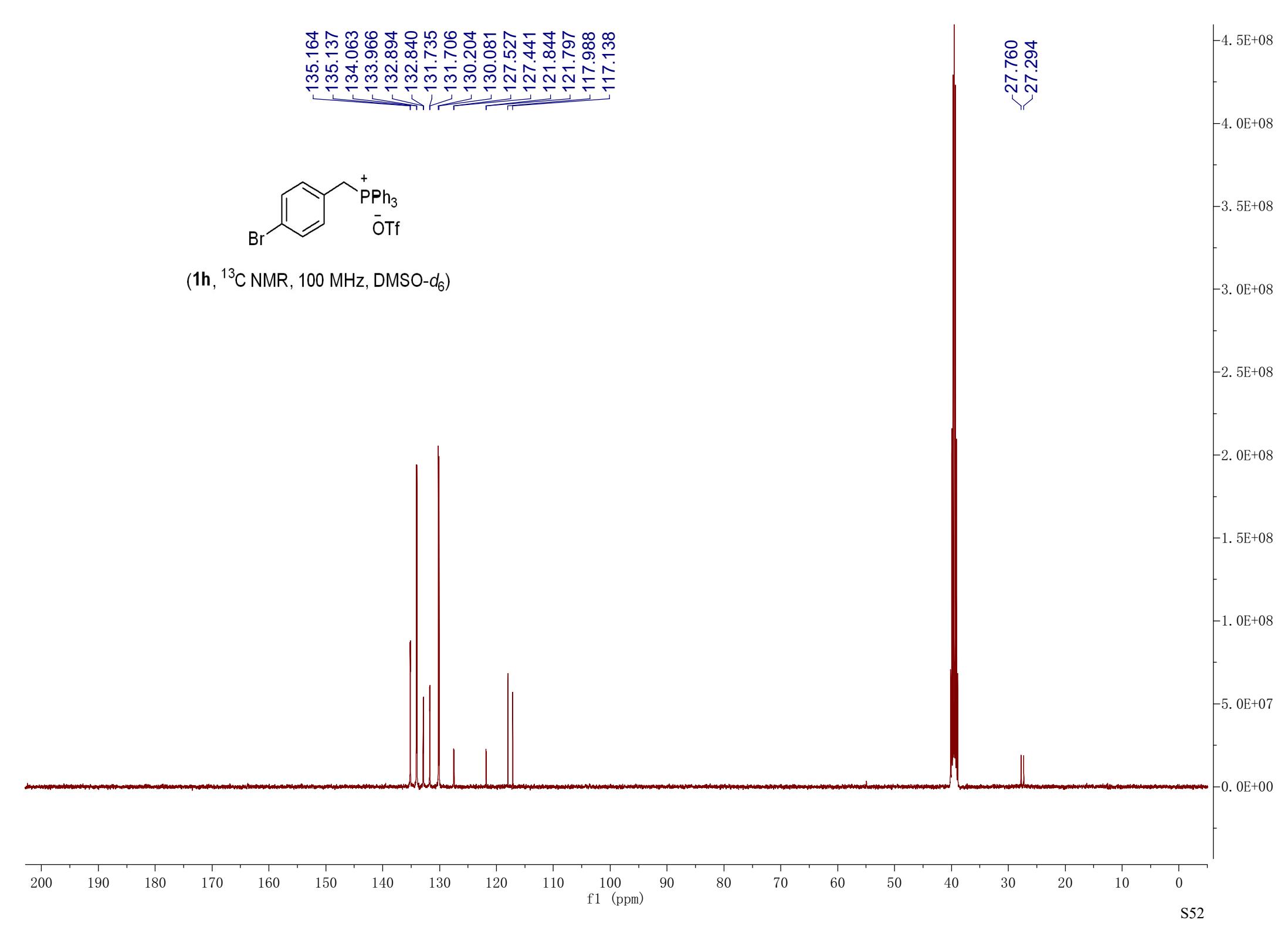
7.916
7.902
7.757
7.707
7.678
7.661
7.471
7.455
6.916
6.900

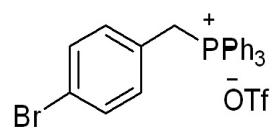
5.187
5.147



(**1h**, ^1H NMR, 400 MHz, DMSO- d_6)

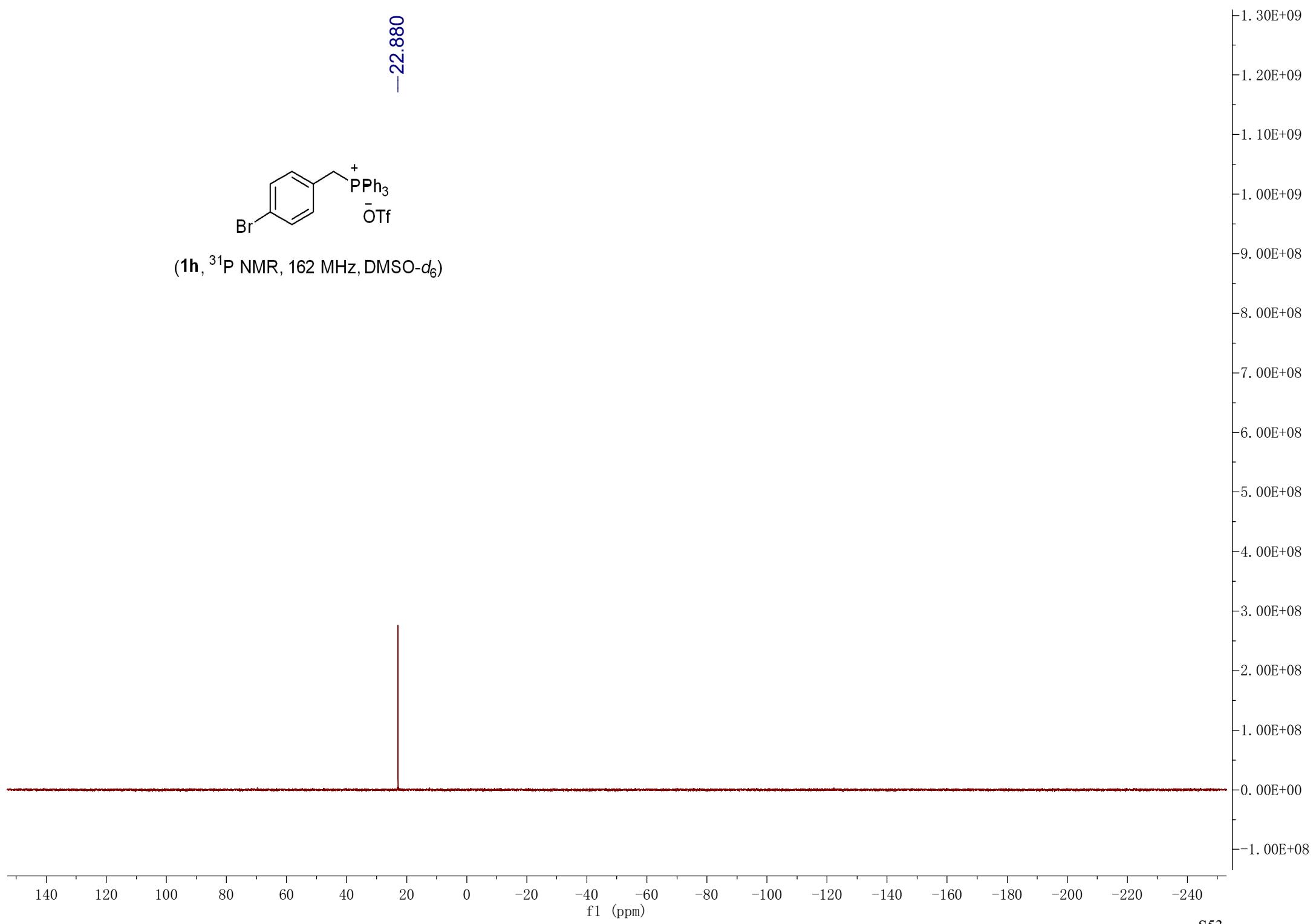






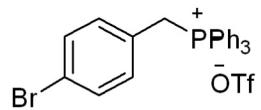
(**1h**, ^{31}P NMR, 162 MHz, DMSO- d_6)

-22.880

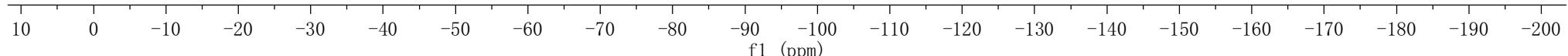


-2.8E+09
-2.6E+09
-2.4E+09
-2.2E+09
-2.0E+09
-1.8E+09
-1.6E+09
-1.4E+09
-1.2E+09
-1.0E+09
-8.0E+08
-6.0E+08
-4.0E+08
-2.0E+08
0.0E+00
-2.0E+08

-77.720

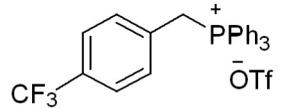


(**1h**, ^{19}F NMR, 376 MHz, DMSO- d_6)

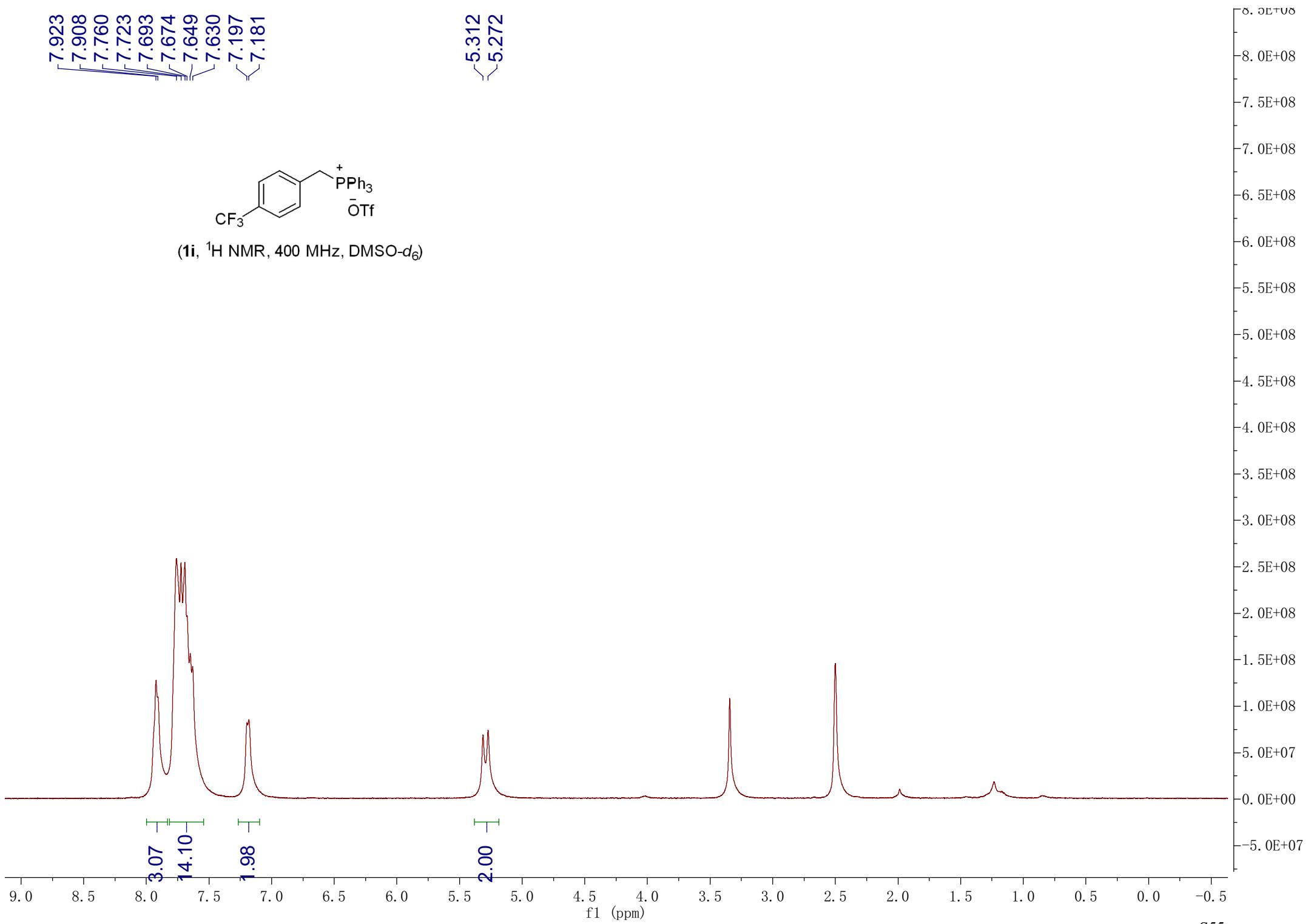


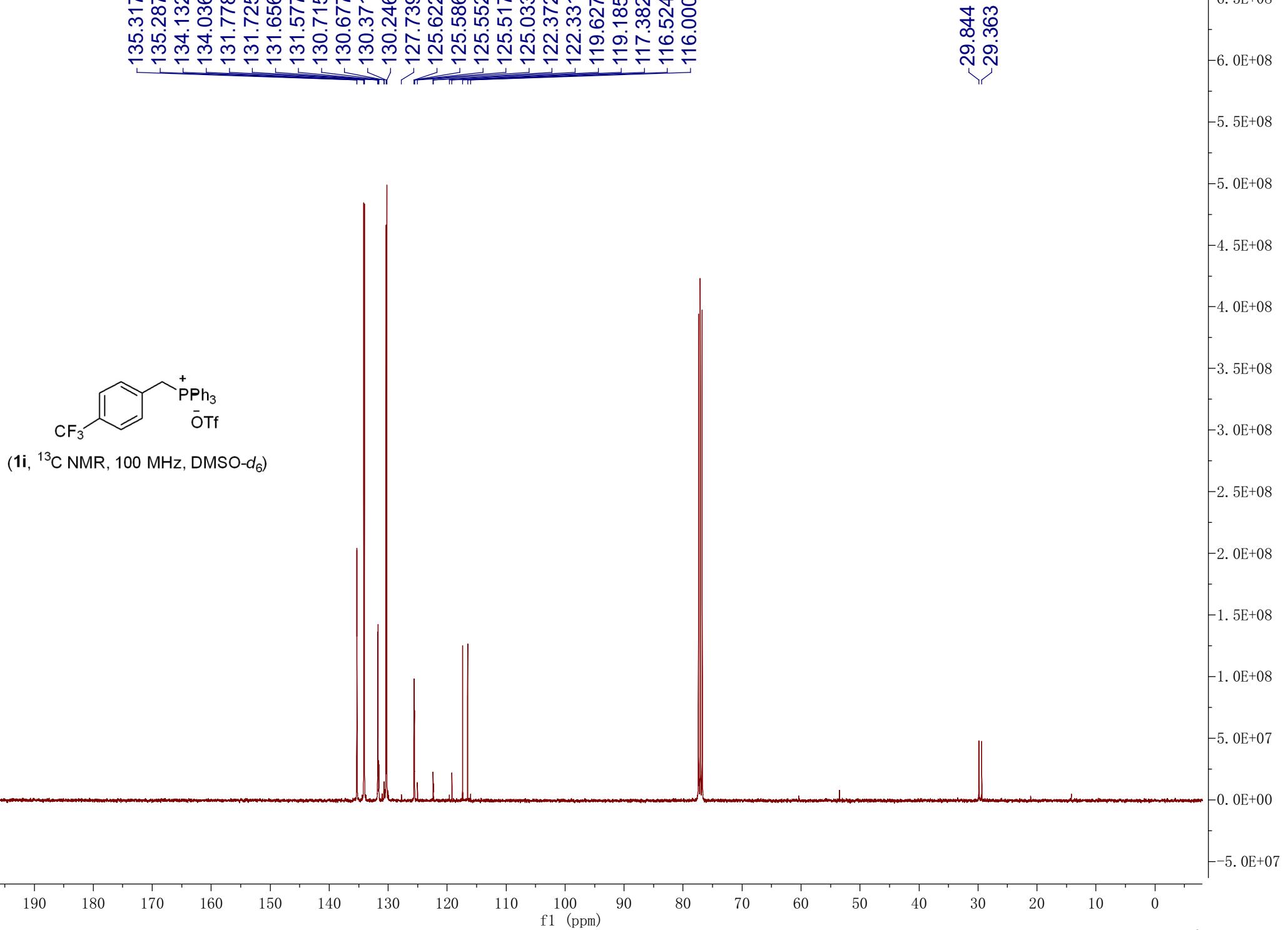
7.923
7.908
7.760
7.723
7.693
7.674
7.649
7.630
7.197
7.181

5.312
5.272



(**1i**, ^1H NMR, 400 MHz, $\text{DMSO}-d_6$)





1.10E+09

1.00E+09

9.00E+08

8.00E+08

7.00E+08

6.00E+08

5.00E+08

4.00E+08

3.00E+08

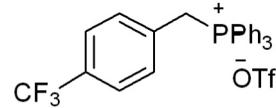
2.00E+08

1.00E+08

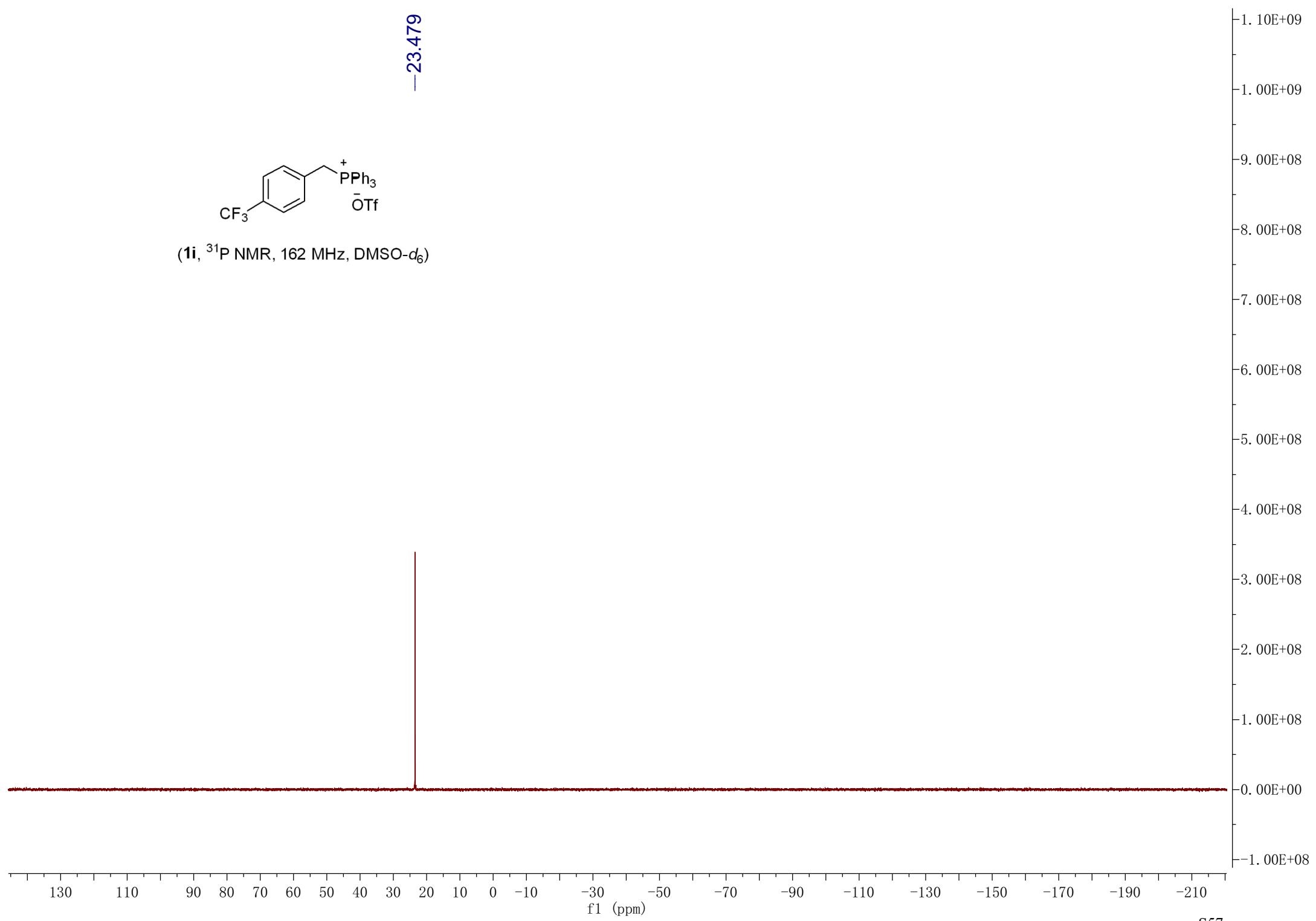
0.00E+00

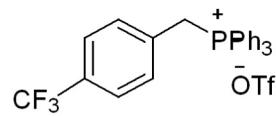
-1.00E+08

-23.479



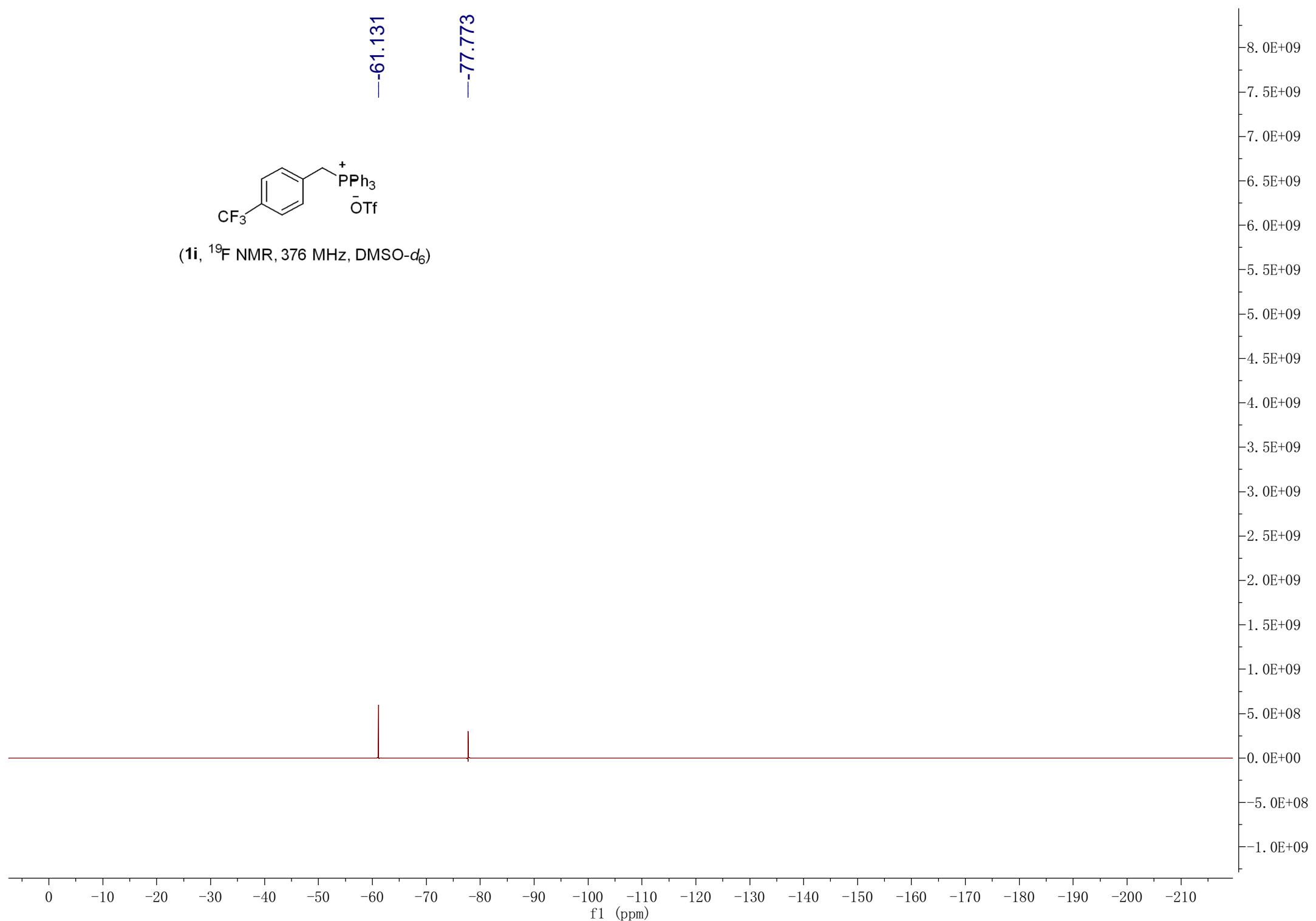
(**1i**, ^{31}P NMR, 162 MHz, DMSO- d_6)





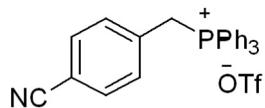
(**1i**, ^{19}F NMR, 376 MHz, DMSO- d_6)

—61.131
—77.773

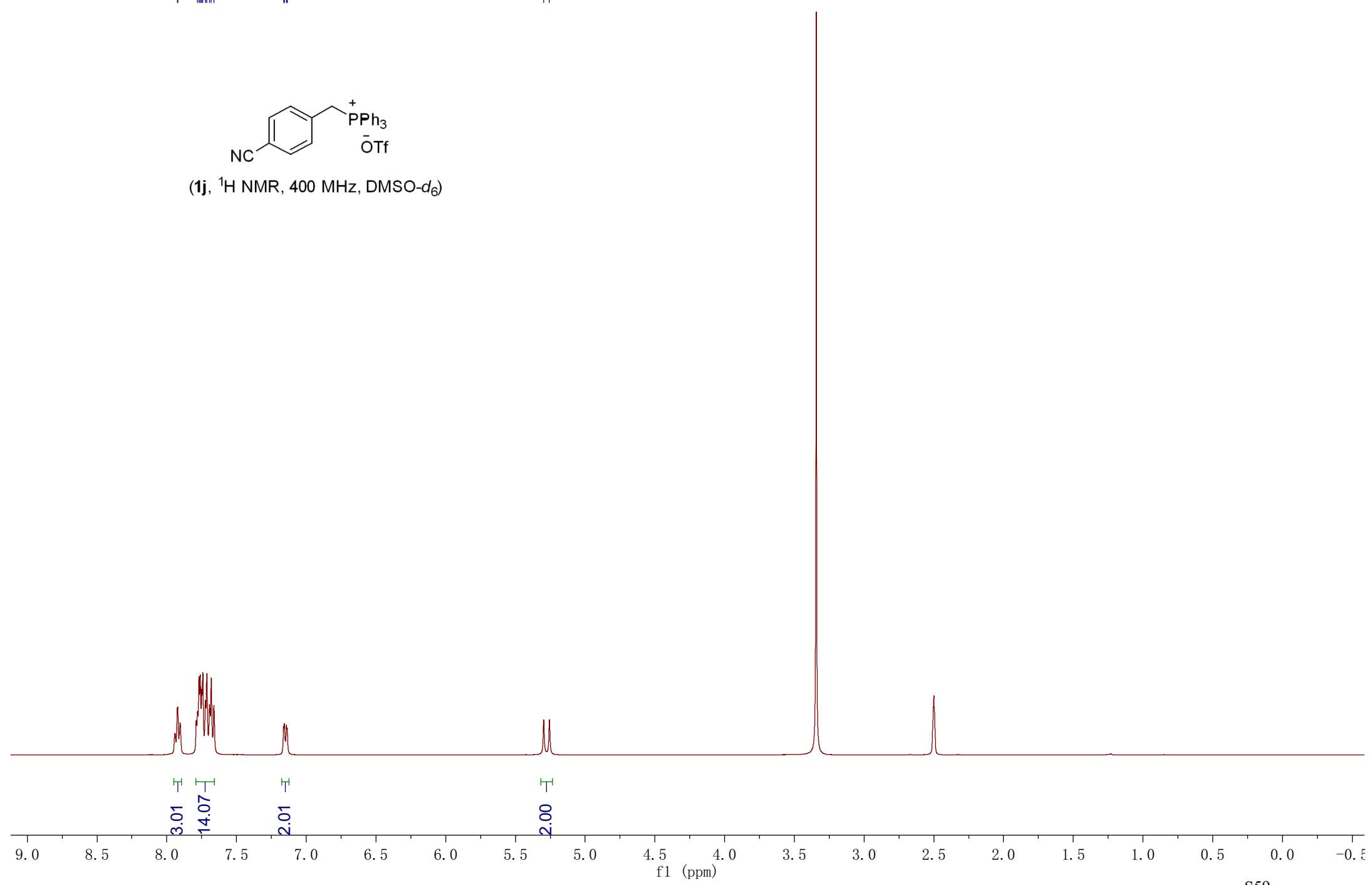


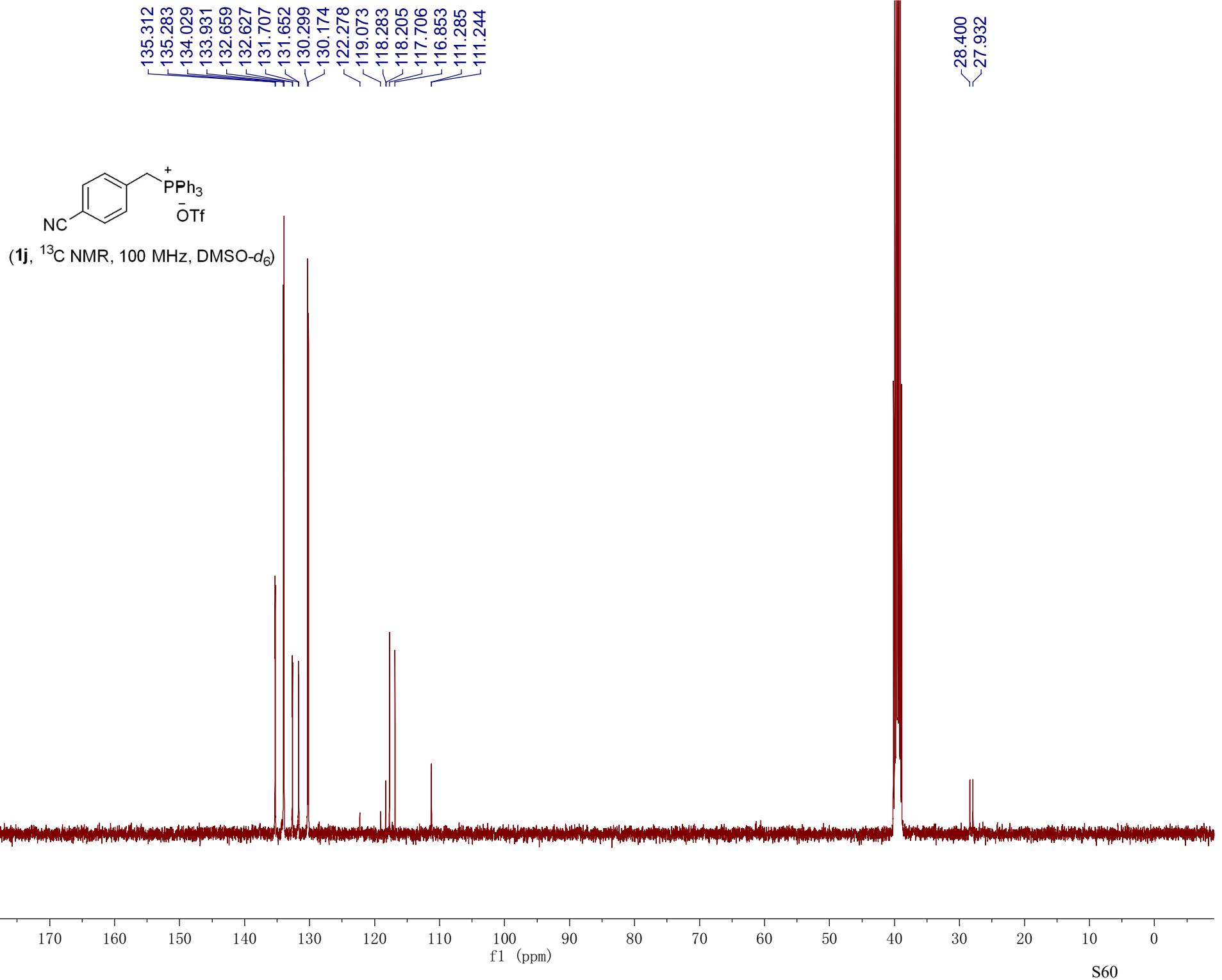
7.924
7.921
7.780
7.769
7.760
7.750
7.742
7.722
7.713
7.694
7.681
7.662
7.157
7.142
7.136

5.297
5.256

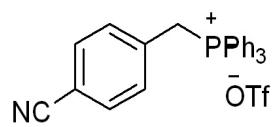


(**1j**, ^1H NMR, 400 MHz, DMSO- d_6)

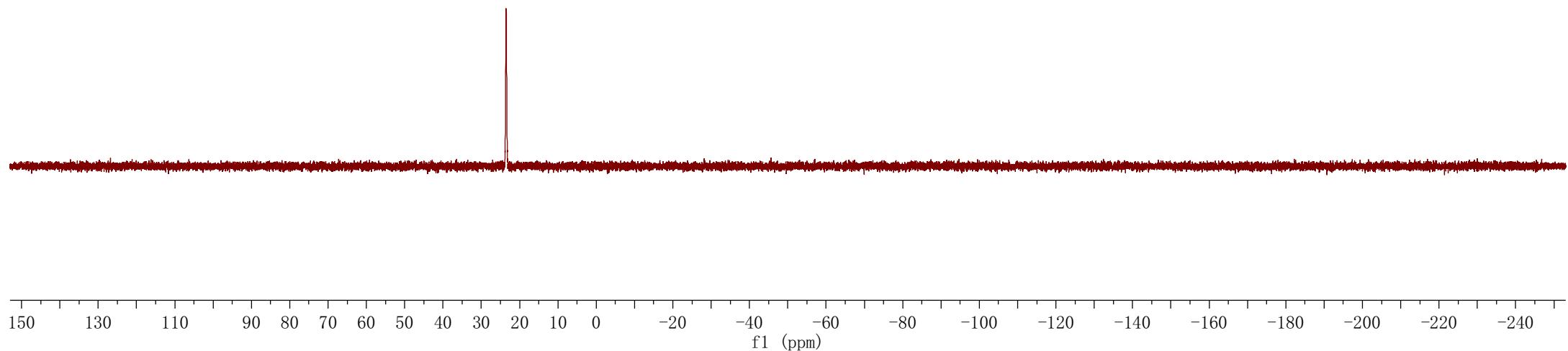




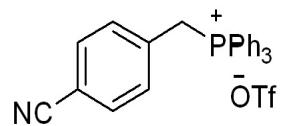
-23.500



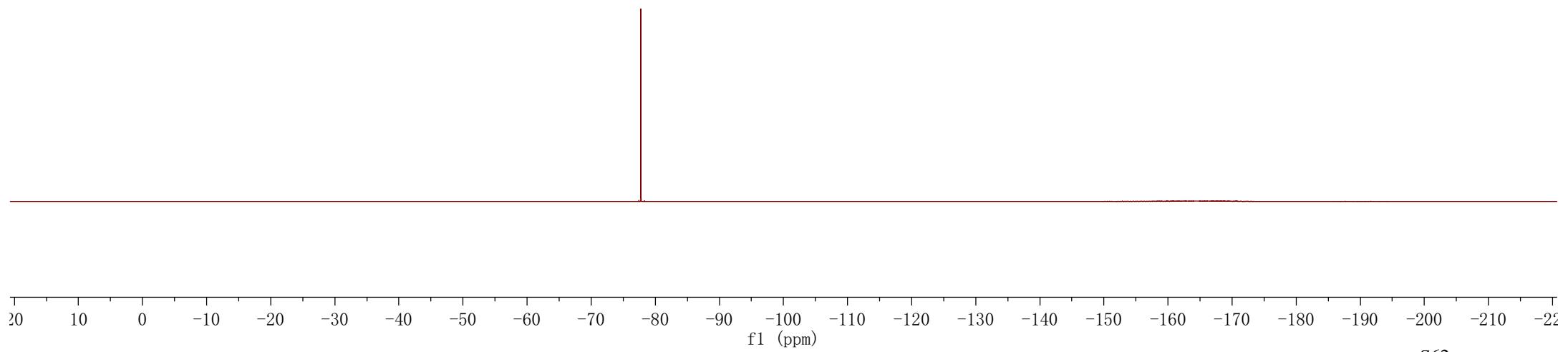
(**1j**, ^{31}P NMR, 162 MHz, DMSO- d_6)



—77.736



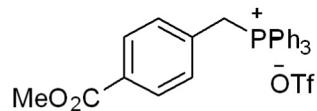
(**1j**, ^{19}F NMR, 376 MHz, DMSO- d_6)



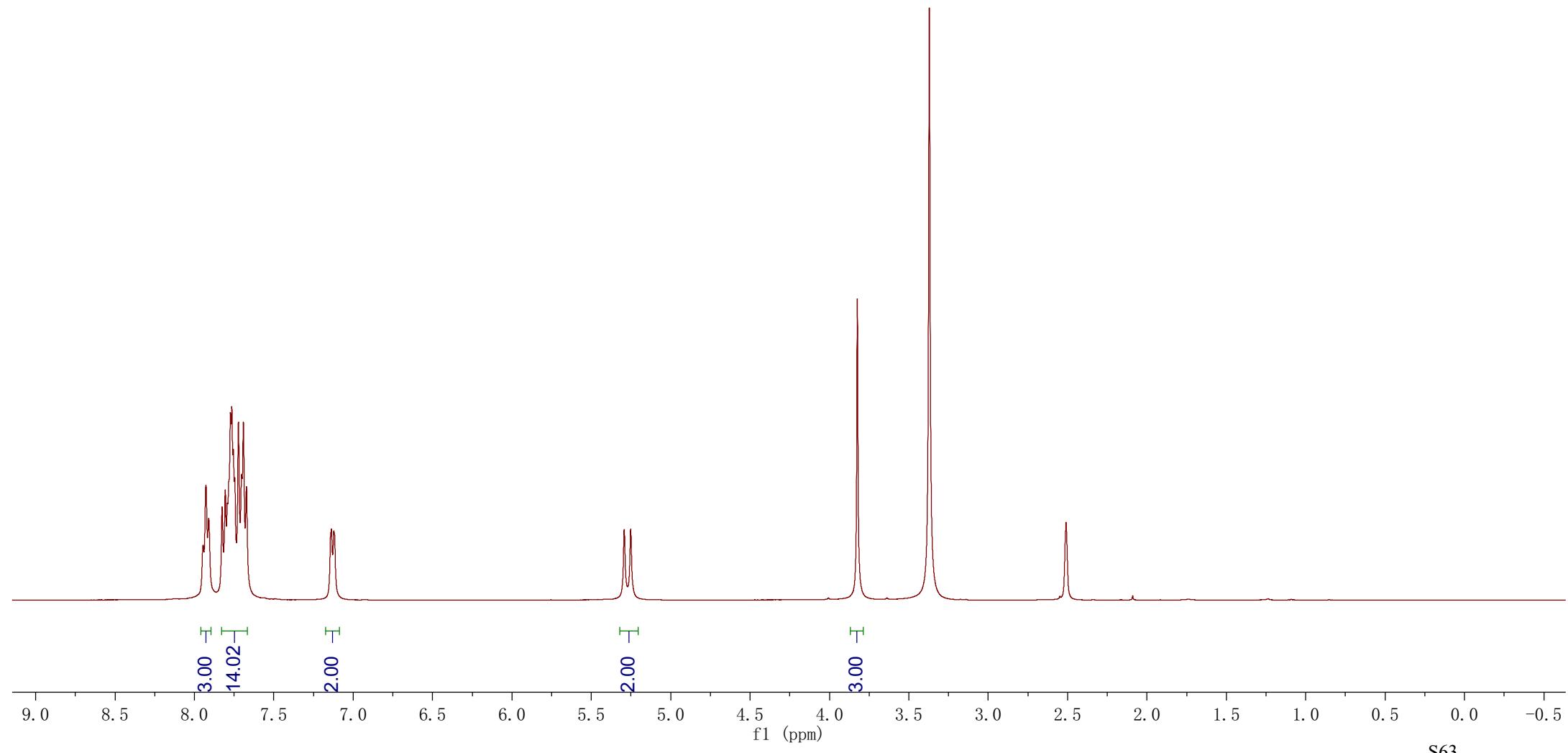
7.927
7.910
7.826
7.806
7.792
7.773
7.765
7.755
7.746
7.723
7.702
7.692
7.672
7.658
7.638
7.122

5.292
5.252

-3.824



(**1k**, ^1H NMR, 400 MHz, DMSO- d_6)

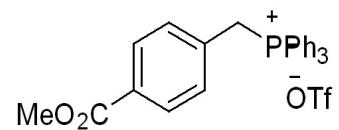


165.646
165.636

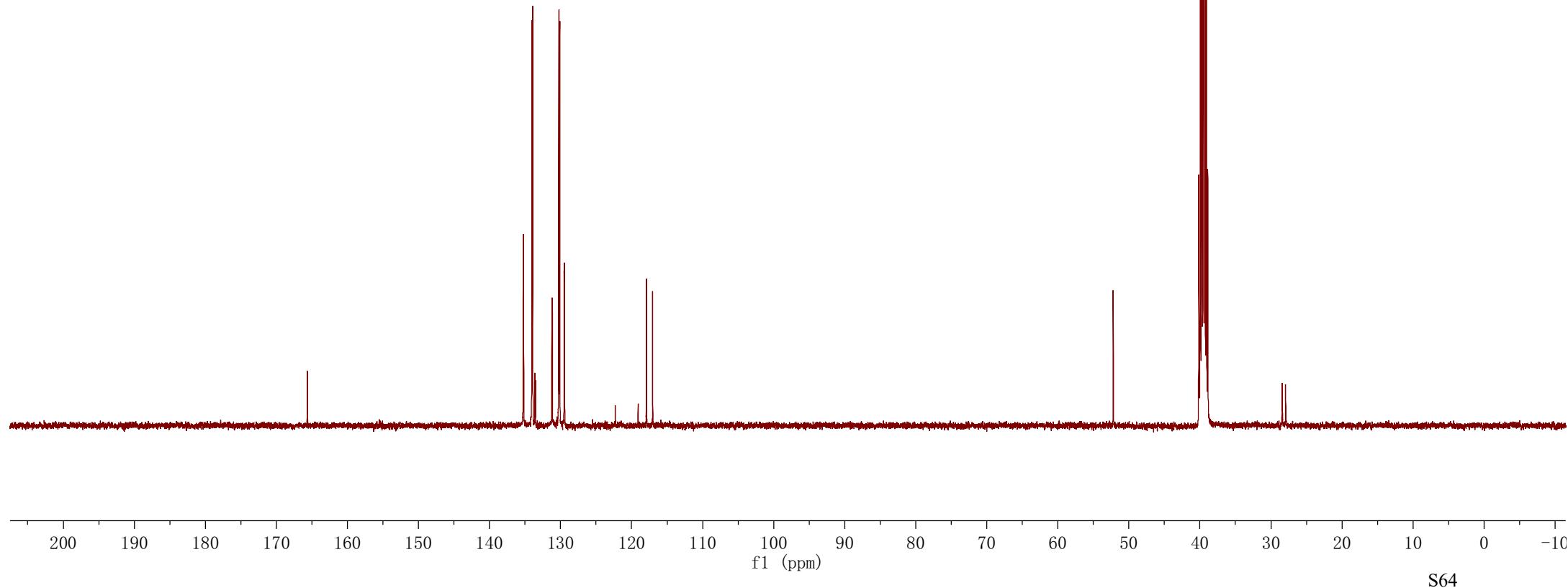
135.233
135.207
134.028
133.929
133.622
131.210
131.156
130.246
130.121
129.479
122.454
119.083
117.893
117.040

—52.201

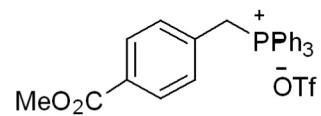
28.411
27.945



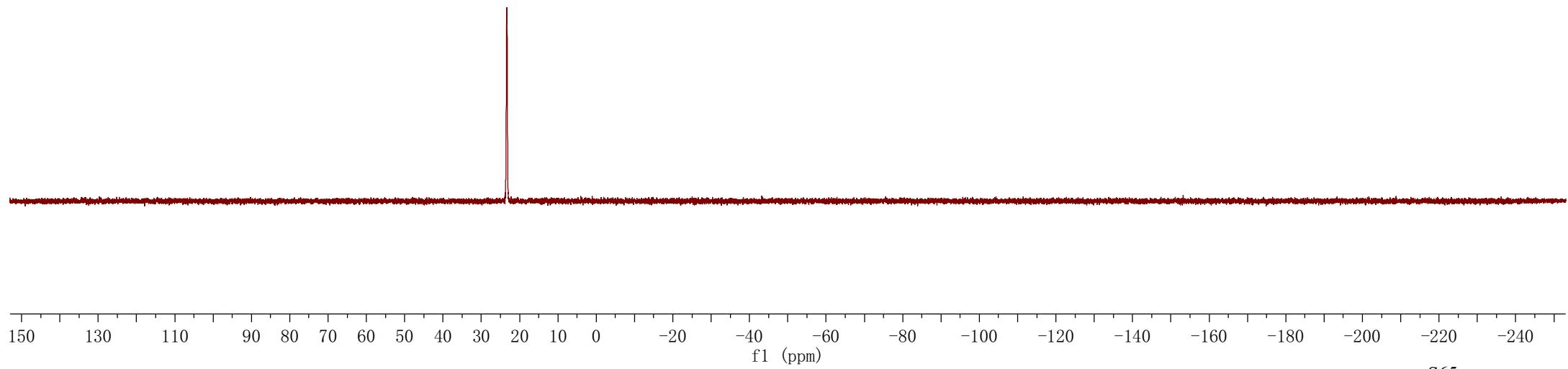
(**1k**, ^{13}C NMR, 100 MHz, DMSO- d_6)



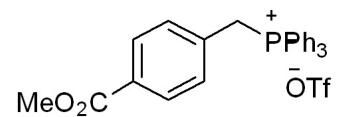
-23.335



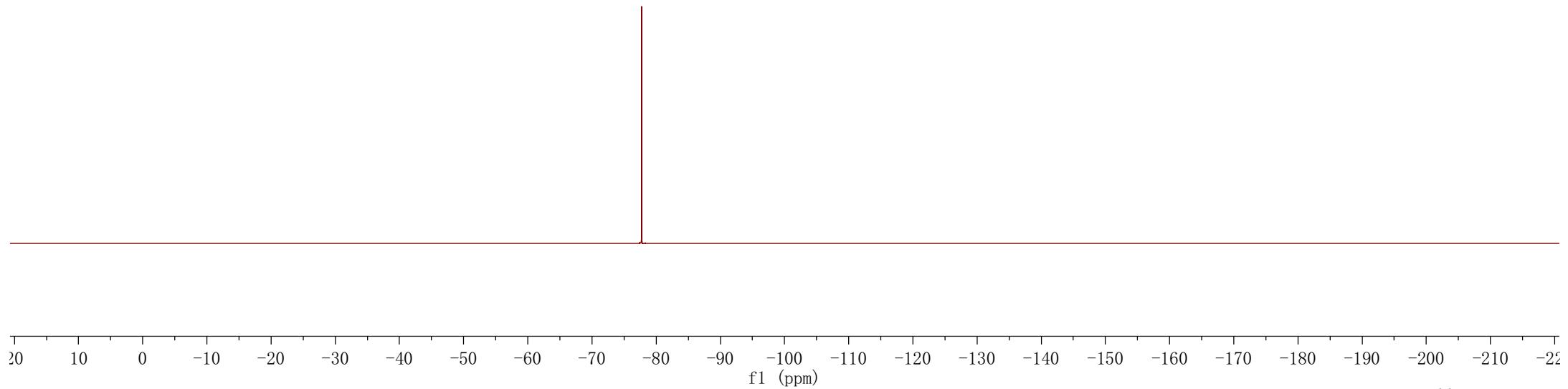
(**1k**, ^{31}P NMR, 162 MHz, $\text{DMSO}-d_6$)



-77.729



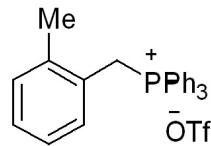
(**1k**, ^{19}F NMR, 376 MHz, $\text{DMSO}-d_6$)



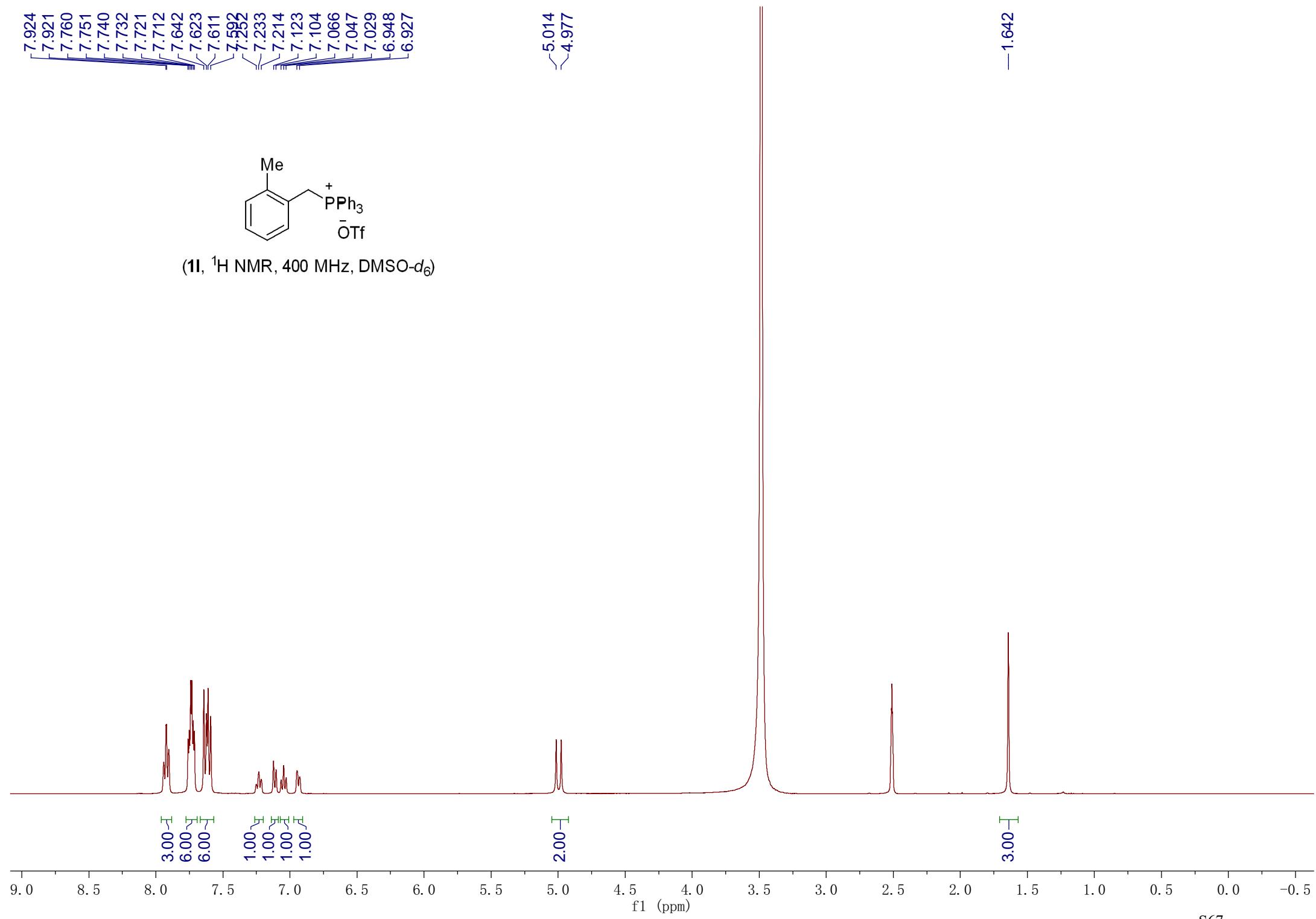
7.924
7.921
7.760
7.751
7.740
7.732
7.721
7.712
7.642
7.623
7.611
7.592
7.233
7.214
7.123
7.104
7.066
7.047
7.029
6.948
6.927

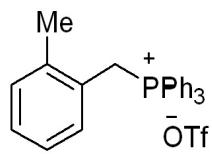
5.014
4.977

—1.642

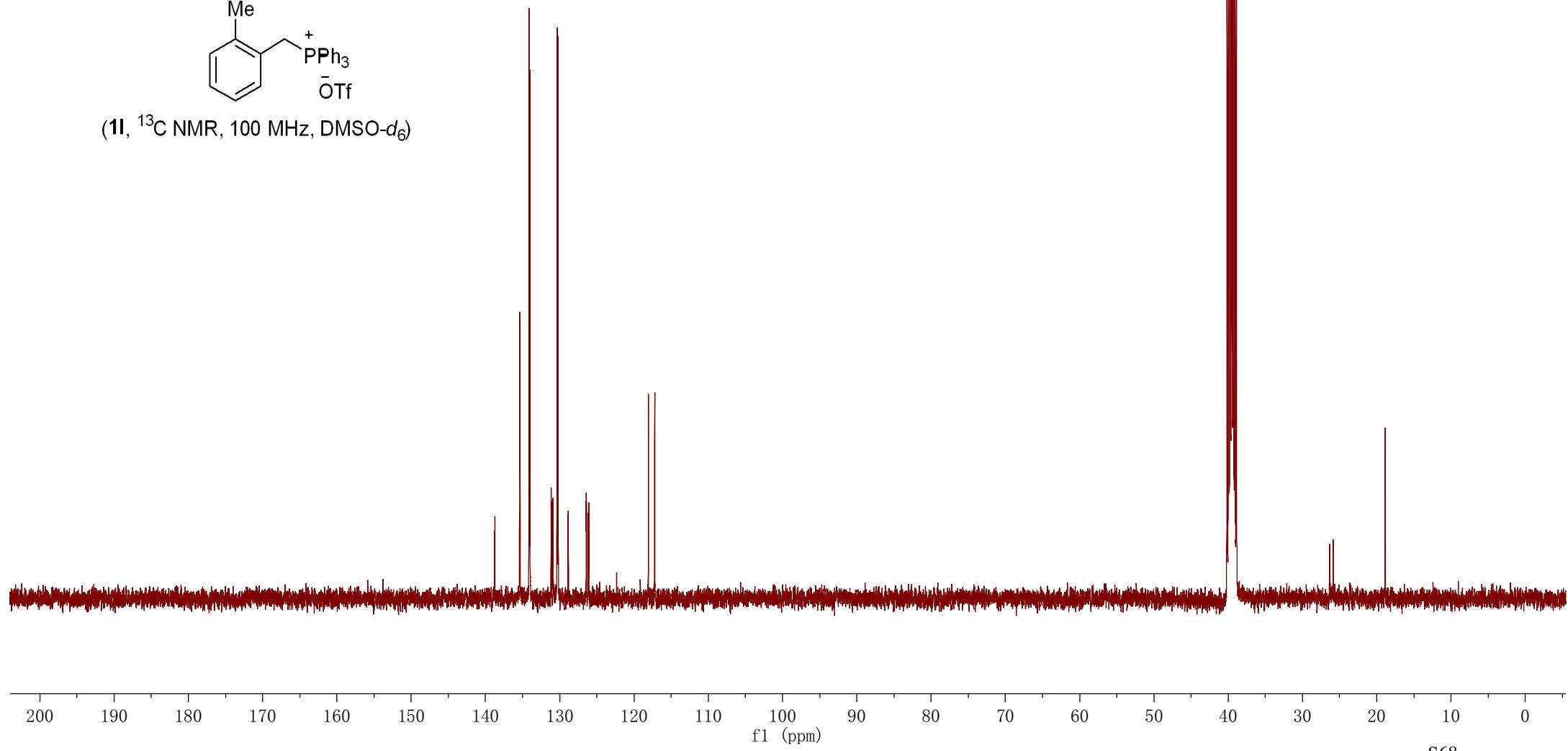


(1I, ^1H NMR, 400 MHz, DMSO- d_6)

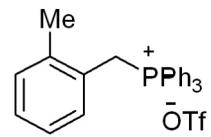




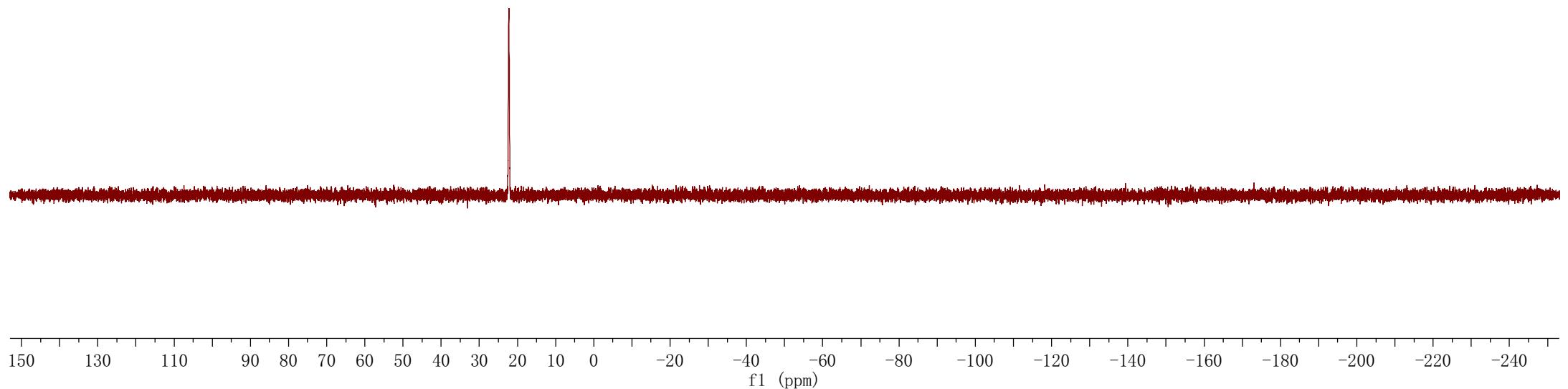
(**1I**, ^{13}C NMR, 100 MHz, $\text{DMSO}-d_6$)



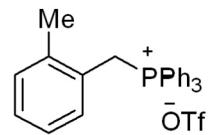
-22.260



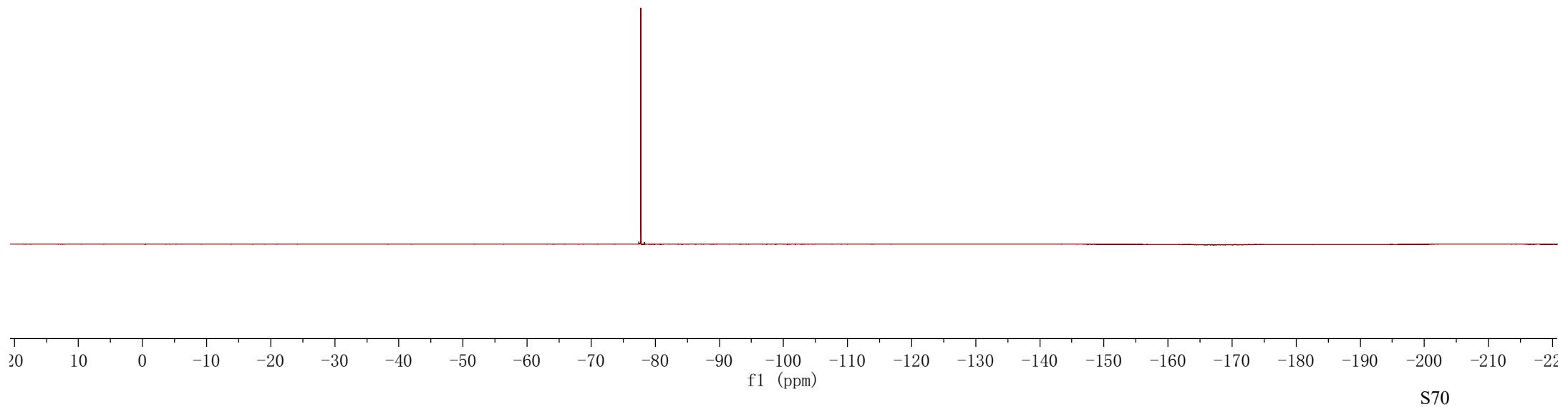
(**1l**, ^{31}P NMR, 162 MHz, $\text{DMSO}-d_6$)



—77.754



(**1I**, ^{19}F NMR, 376 MHz, $\text{DMSO}-d_6$)

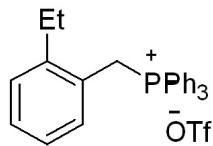


7.935
7.916
7.898
7.758
7.749
7.738
7.730
7.719
7.710
7.635
7.615
7.604
7.584
7.313
7.295
7.276
7.188
7.169
7.055
7.036
7.018
6.931
6.911

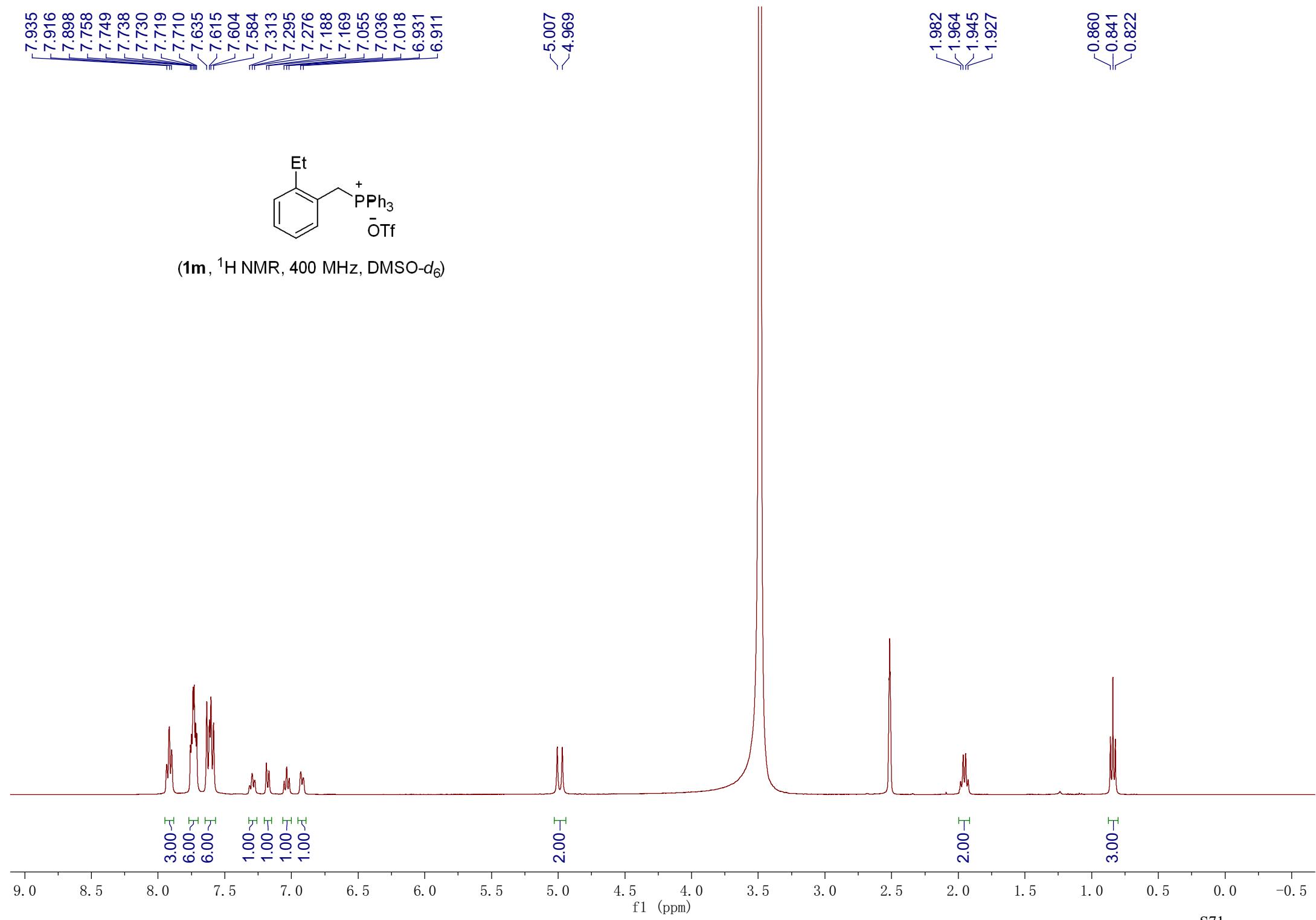
5.007
4.969

1.982
1.964
1.945
1.927

0.860
0.841
0.822



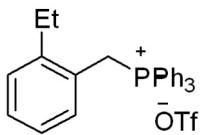
(**1m**, ^1H NMR, 400 MHz, DMSO- d_6)



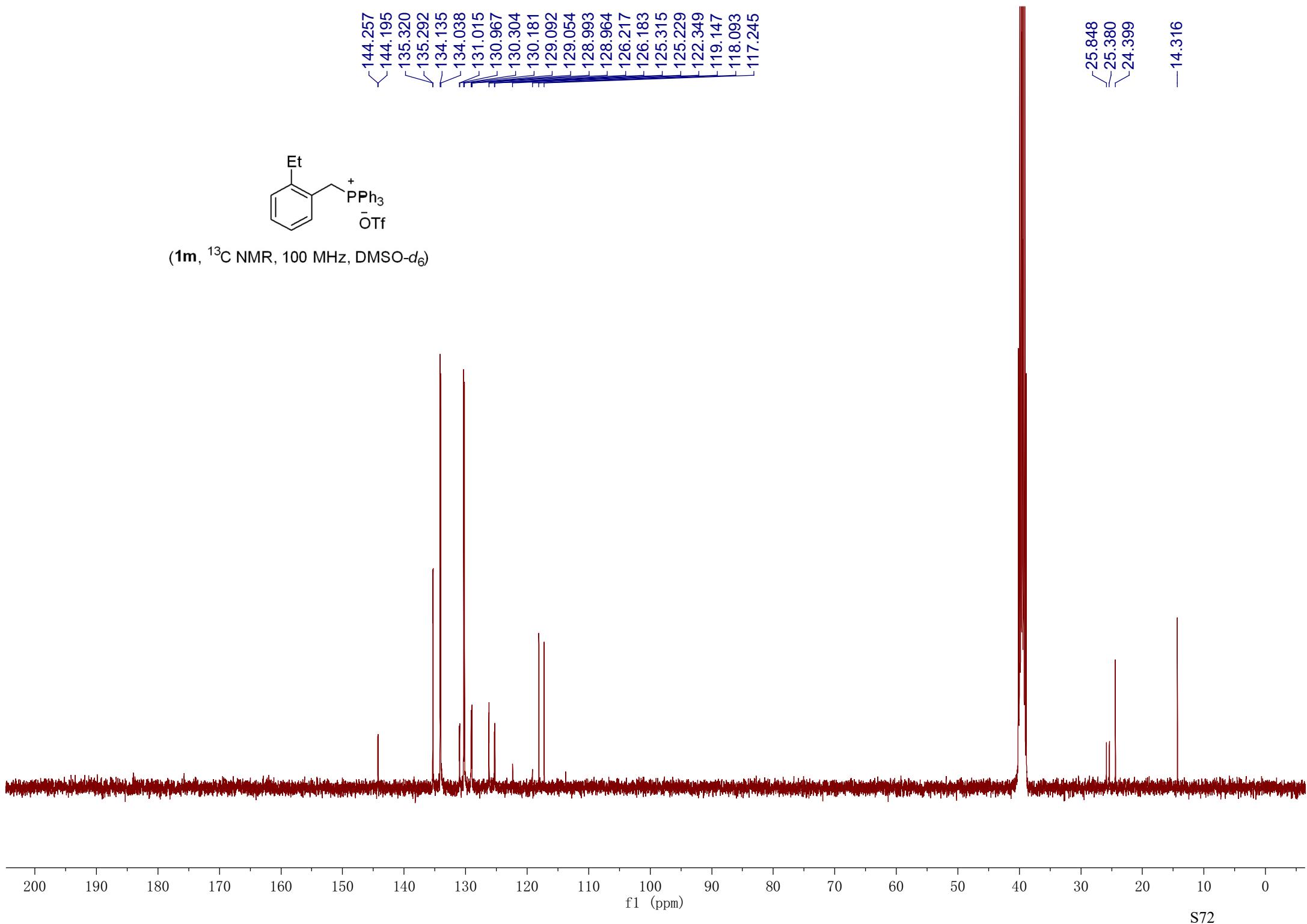
—14.316

25.848
25.380
24.399

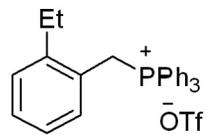
144.257
144.195
135.320
135.292
134.135
134.038
131.015
130.967
130.304
130.181
129.092
129.054
128.993
128.964
126.217
126.183
125.315
125.229
122.349
119.147
118.093
117.245



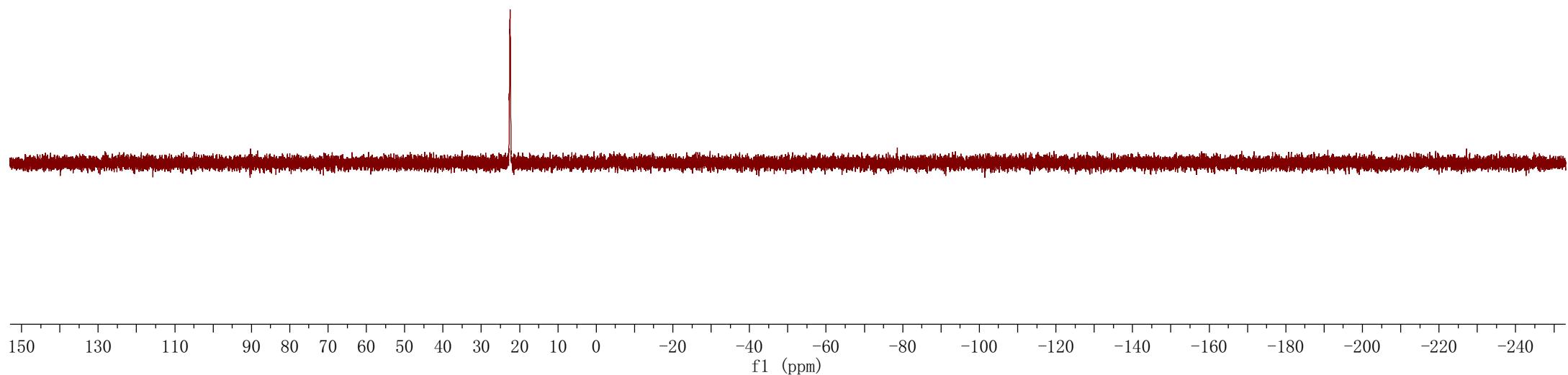
(**1m**, ^{13}C NMR, 100 MHz, $\text{DMSO}-d_6$)



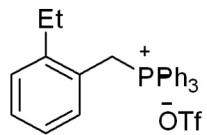
-22.503



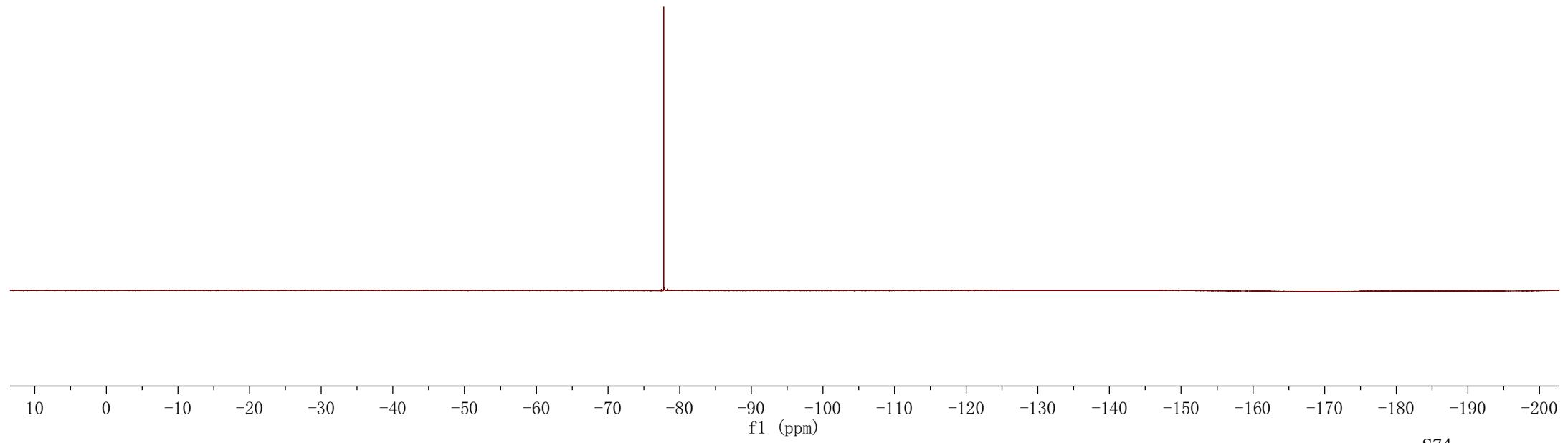
(**1m**, ^{31}P NMR, 162 MHz, DMSO- d_6)



—77.755



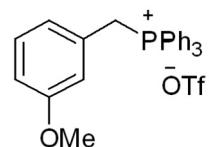
(**1m**, ^{19}F NMR, 376 MHz, $\text{DMSO}-d_6$)



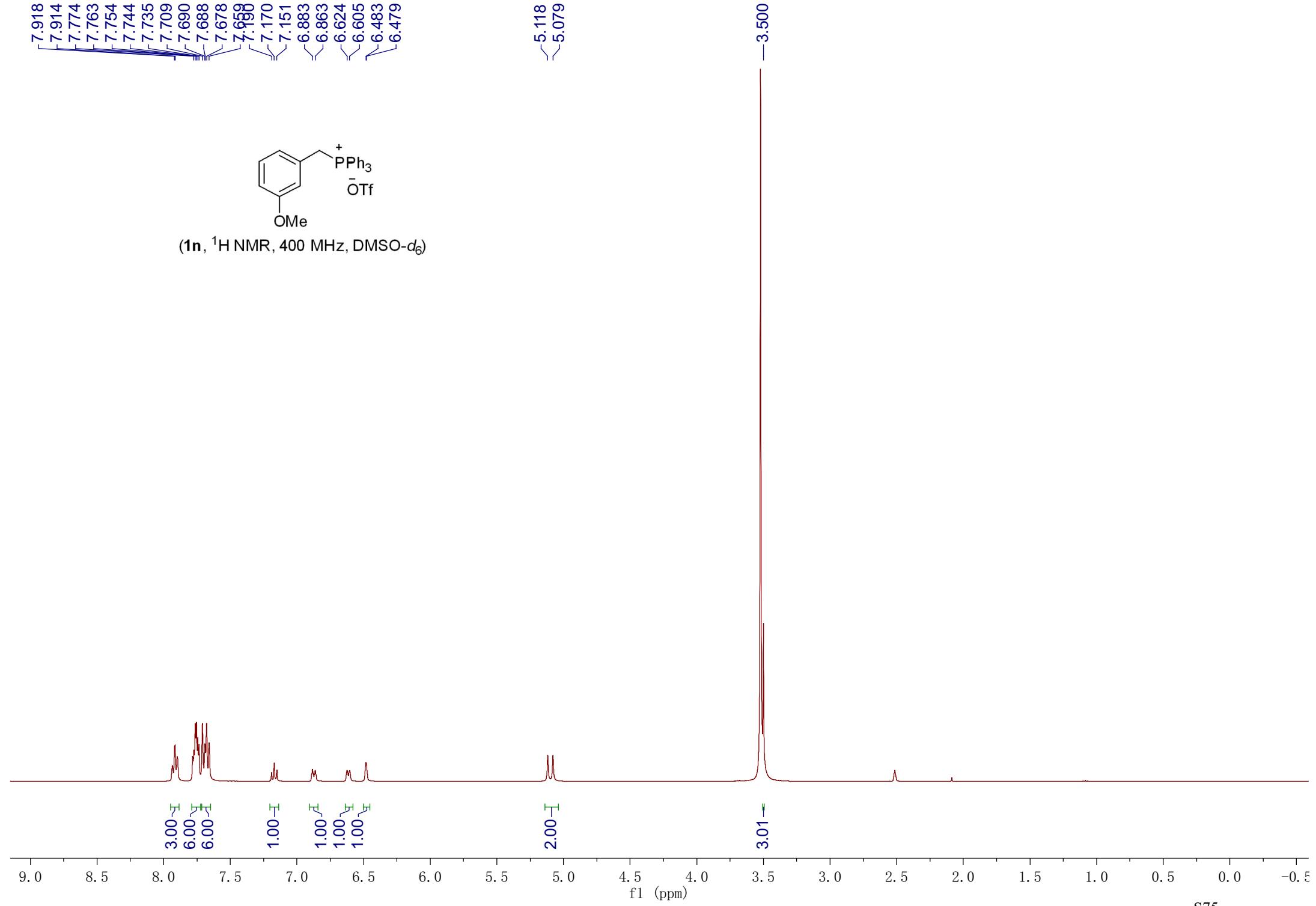
7.918
7.914
7.774
7.763
7.754
7.744
7.735
7.709
7.690
7.688
7.678
7.659
7.643

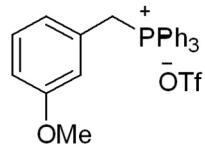
5.118
5.079

3.500

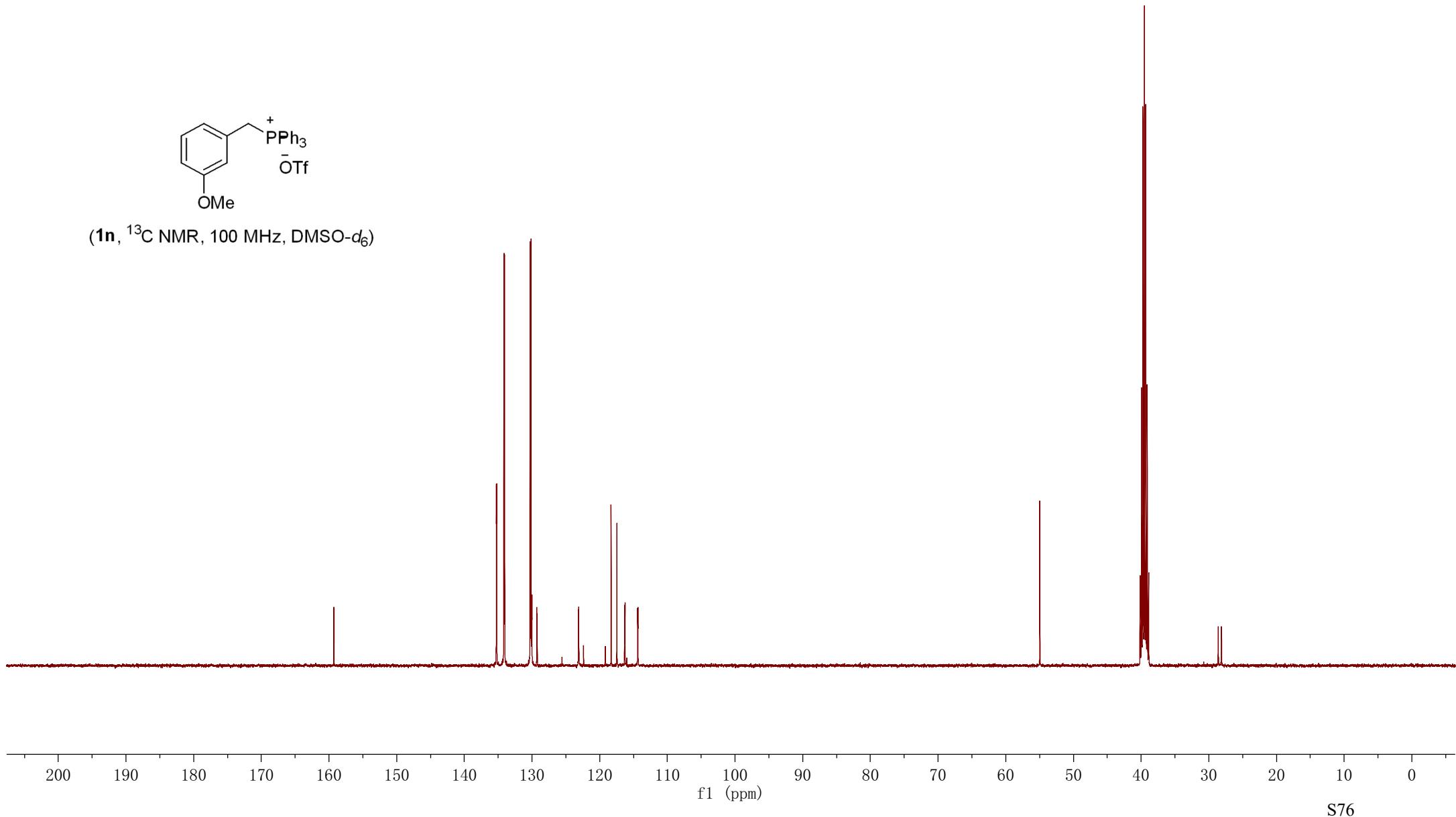
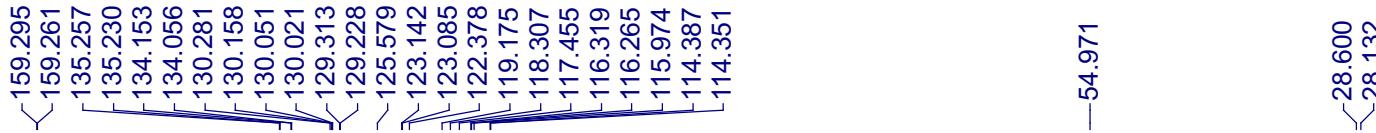


(**1n**, ^1H NMR, 400 MHz, $\text{DMSO}-d_6$)

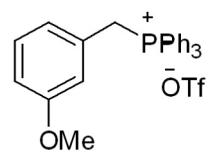




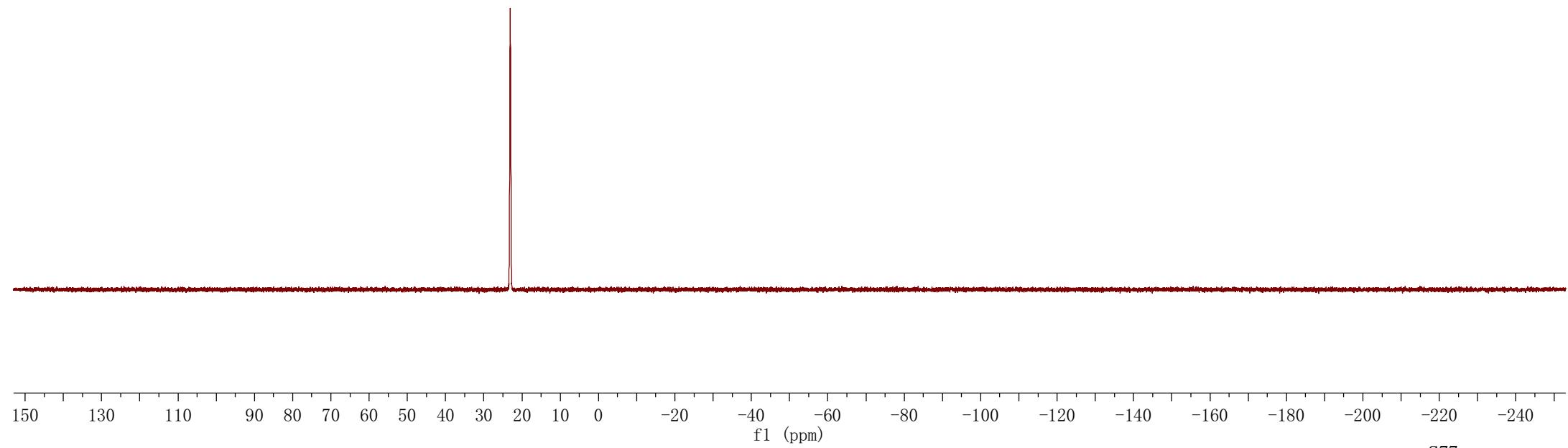
(**1n**, ^{13}C NMR, 100 MHz, DMSO- d_6)



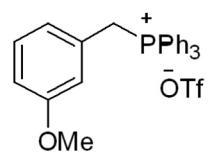
-23.069



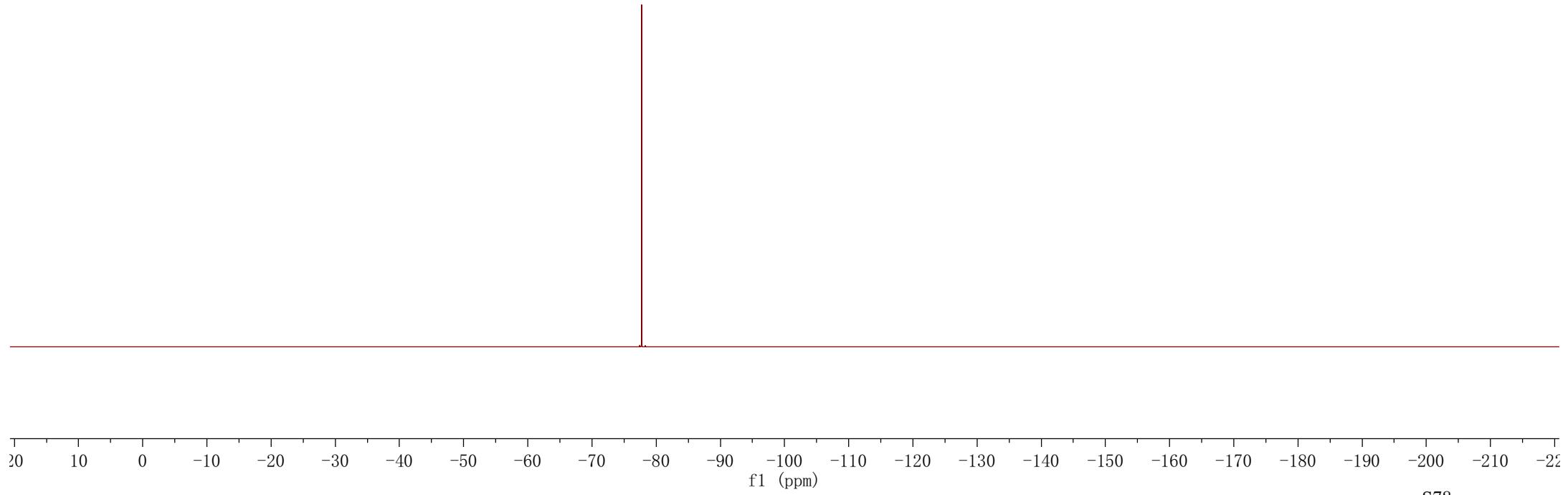
(1n, ^{31}P NMR, 162 MHz, DMSO- d_6)



-77.729

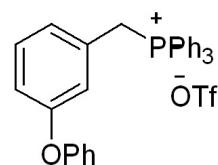


(**1n**, ¹⁹F NMR, 376 MHz, DMSO-*d*₆)

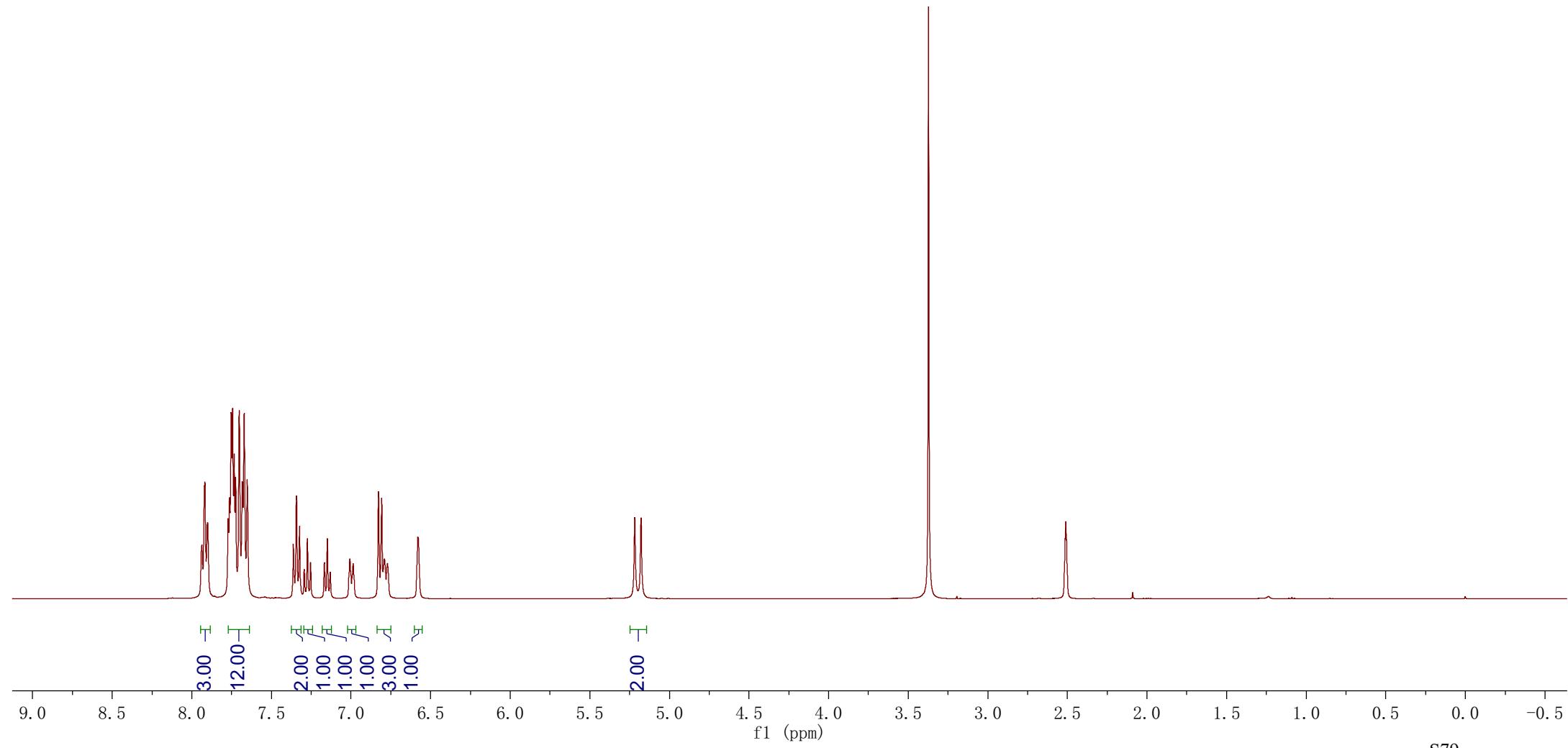


7.921
7.918
7.763
7.752
7.744
7.734
7.724
7.702
7.683
7.671
7.652

7.362
7.343
7.323
7.294
7.274
7.254
7.167
7.149
7.007
6.987
6.827
6.807
6.790
6.771
6.580
5.178

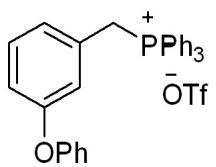


(**1o**, ^1H NMR, 400 MHz, DMSO- d_6)

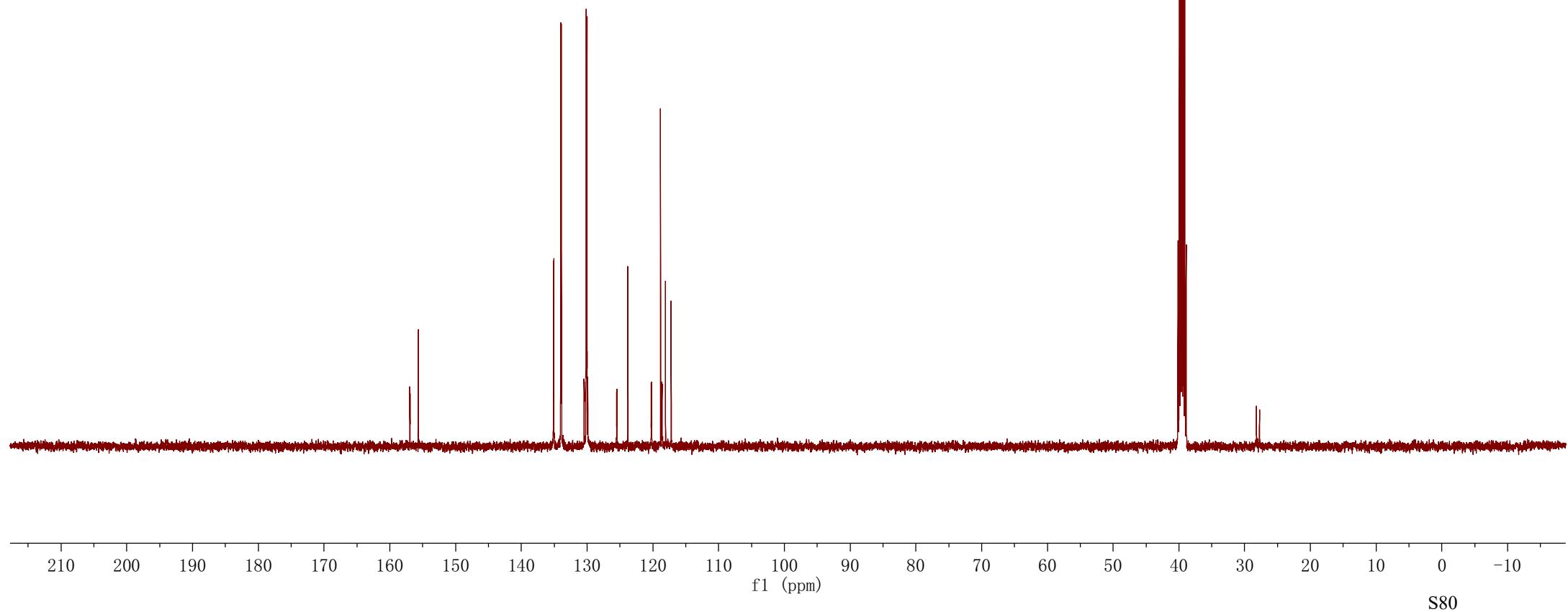


²⁸206
²⁷740

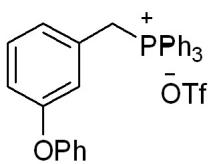
156.962
156.929
155.668
135.097
135.070
133.999
133.902
130.459
130.427
130.147
130.078
130.024
129.984
129.897
125.508
125.453
123.802
120.254
120.198
118.823
118.631
118.596
118.067
117.215



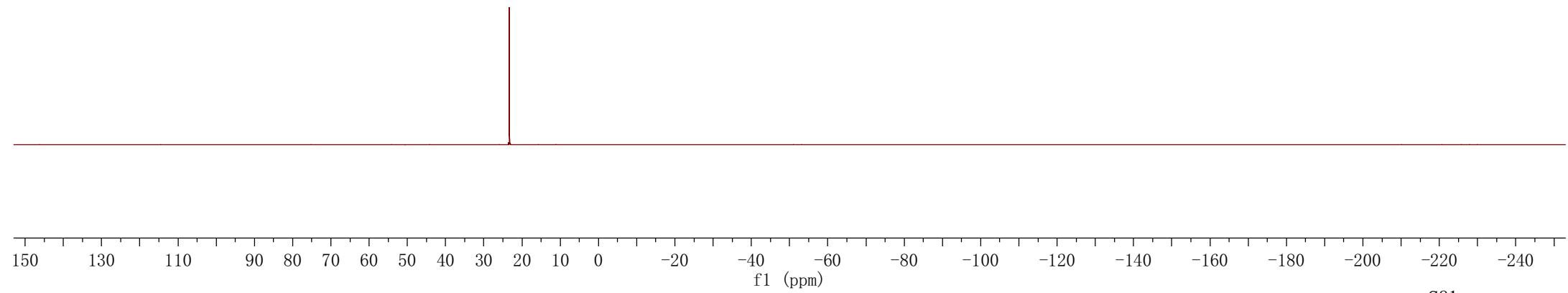
(**1o**, ^{13}C NMR, 100 MHz, $\text{DMSO}-d_6$)



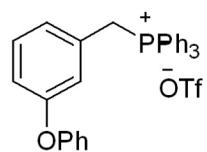
-23.302



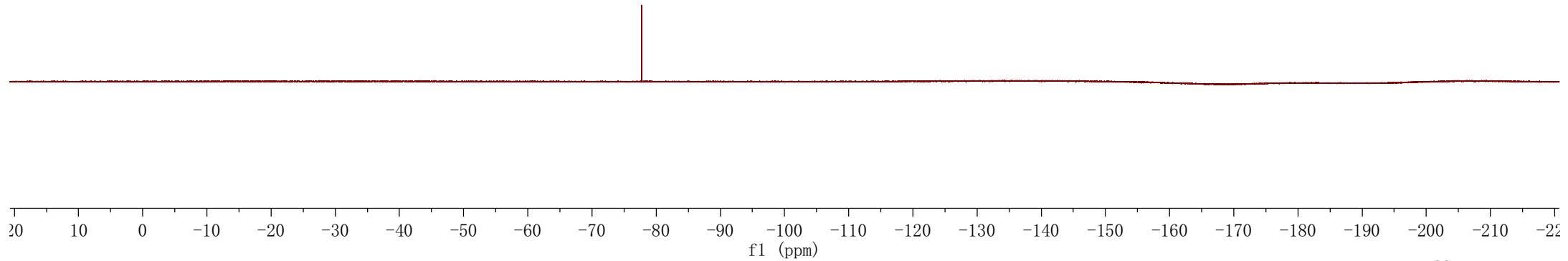
(**1o**, ^{31}P NMR, 162 MHz, DMSO- d_6)



-77.717

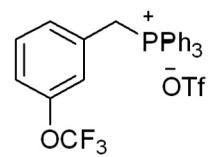


(**1o**, ^{19}F NMR, 376 MHz, DMSO- d_6)

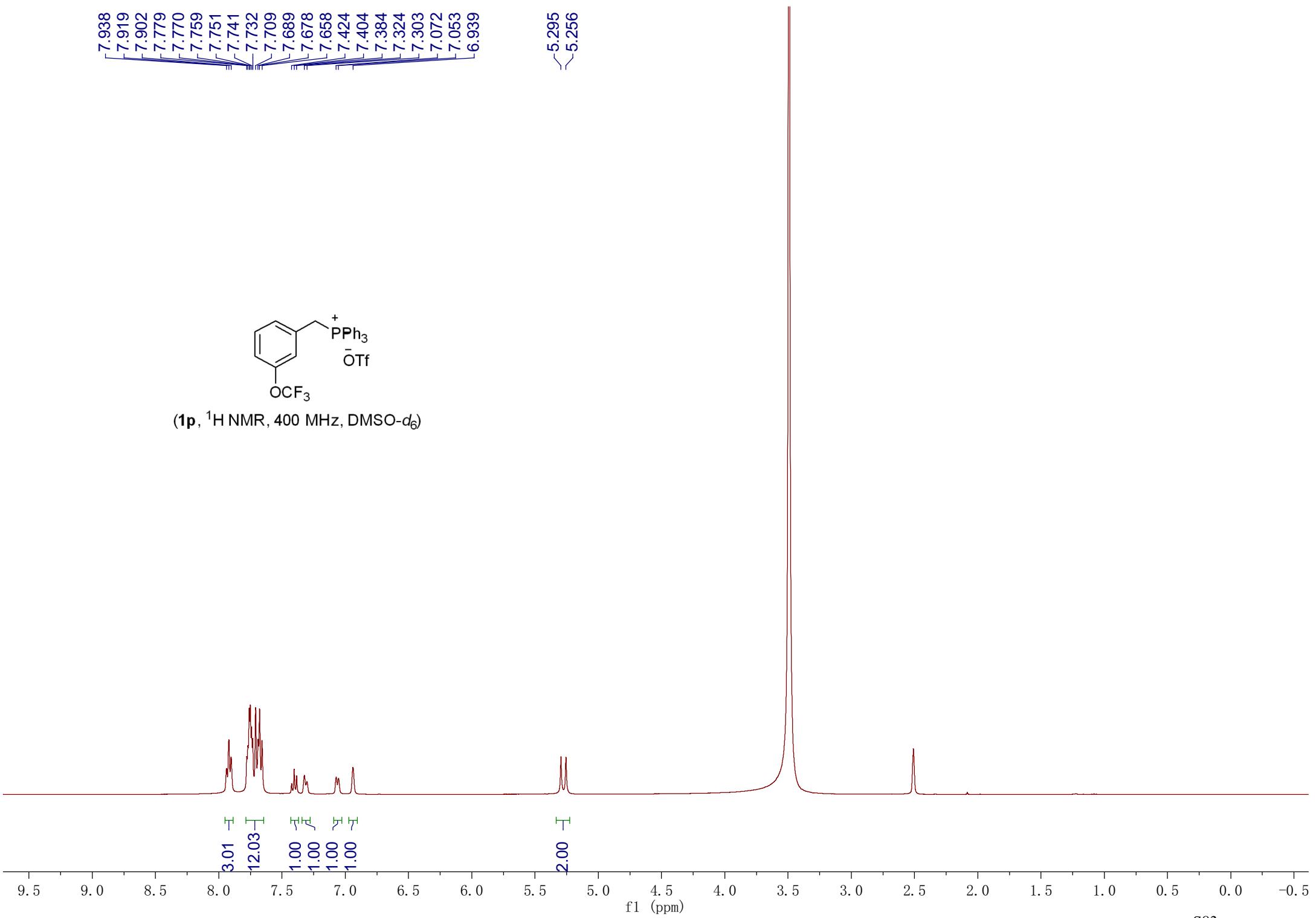


7.938
7.919
7.902
7.779
7.770
7.759
7.751
7.741
7.732
7.709
7.689
7.678
7.658
7.424
7.404
7.384
7.324
7.303
7.072
7.053
6.939

5.295
5.256

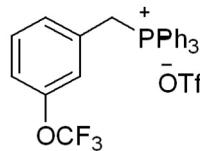


(**1p**, ^1H NMR, 400 MHz, $\text{DMSO}-d_6$)



-149.110
 -136.117
 -136.089
 -134.969
 -134.869
 -131.769
 -131.737
 -131.708
 -131.623
 -131.116
 -130.992
 -130.702
 -130.649
 -124.100
 -124.046
 -121.963
 -121.912
 -121.880
 -119.409
 -118.752
 -117.899

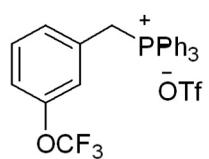
<28.963
 <28.492



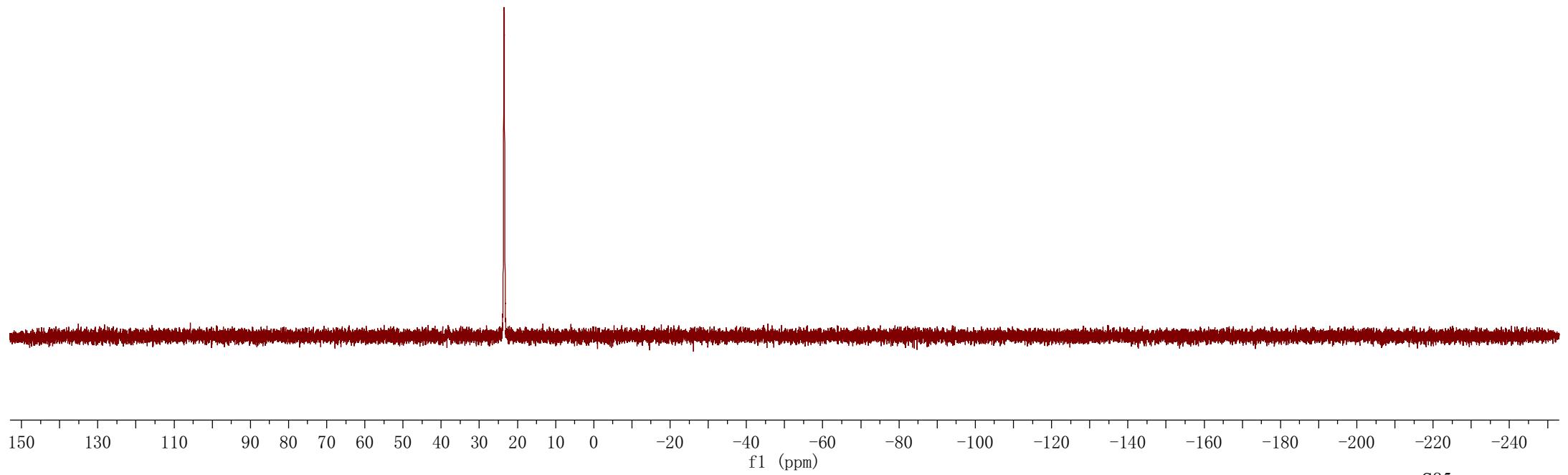
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

f1 (ppm)

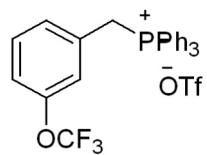
-23.500



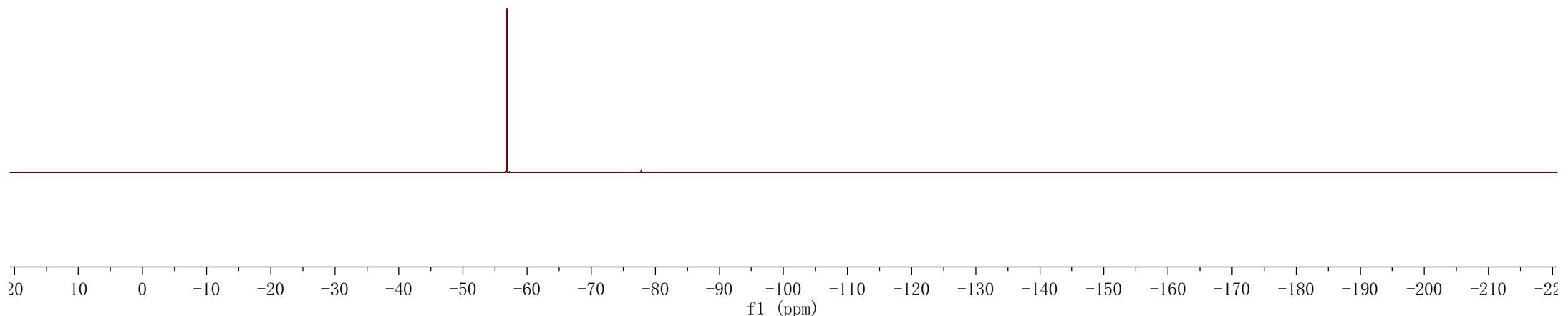
(**1p**, ^{31}P NMR, 162 MHz, DMSO- d_6)



—56.847



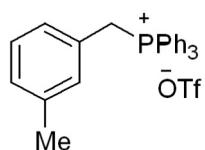
(**1p**, ^{19}F NMR, 376 MHz, $\text{DMSO}-d_6$)



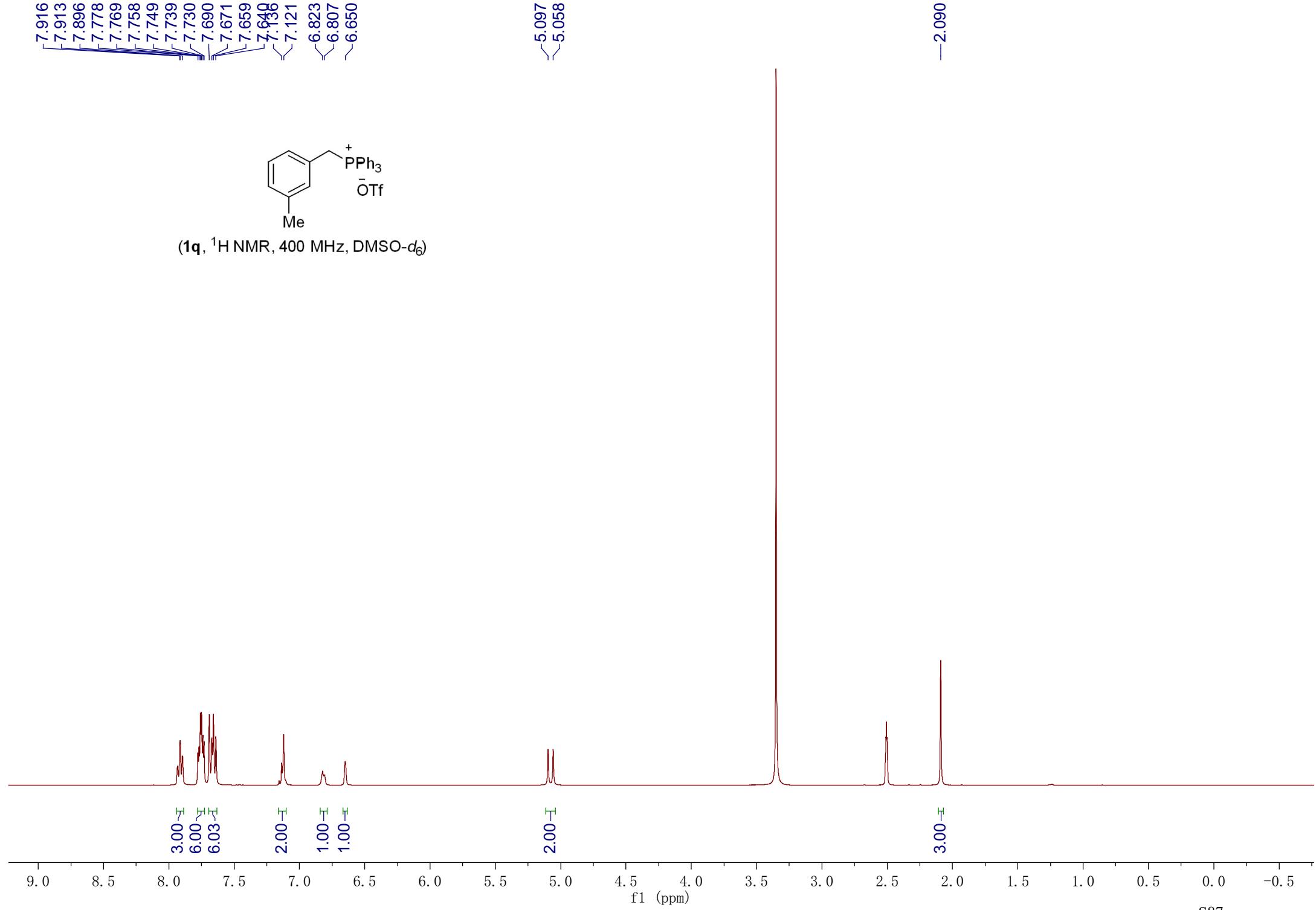
7.916
7.896
7.778
7.769
7.758
7.749
7.739
7.730
7.690
7.671
7.659
7.648
7.36
7.121
6.823
6.807
6.650

5.097
5.058

-2.090

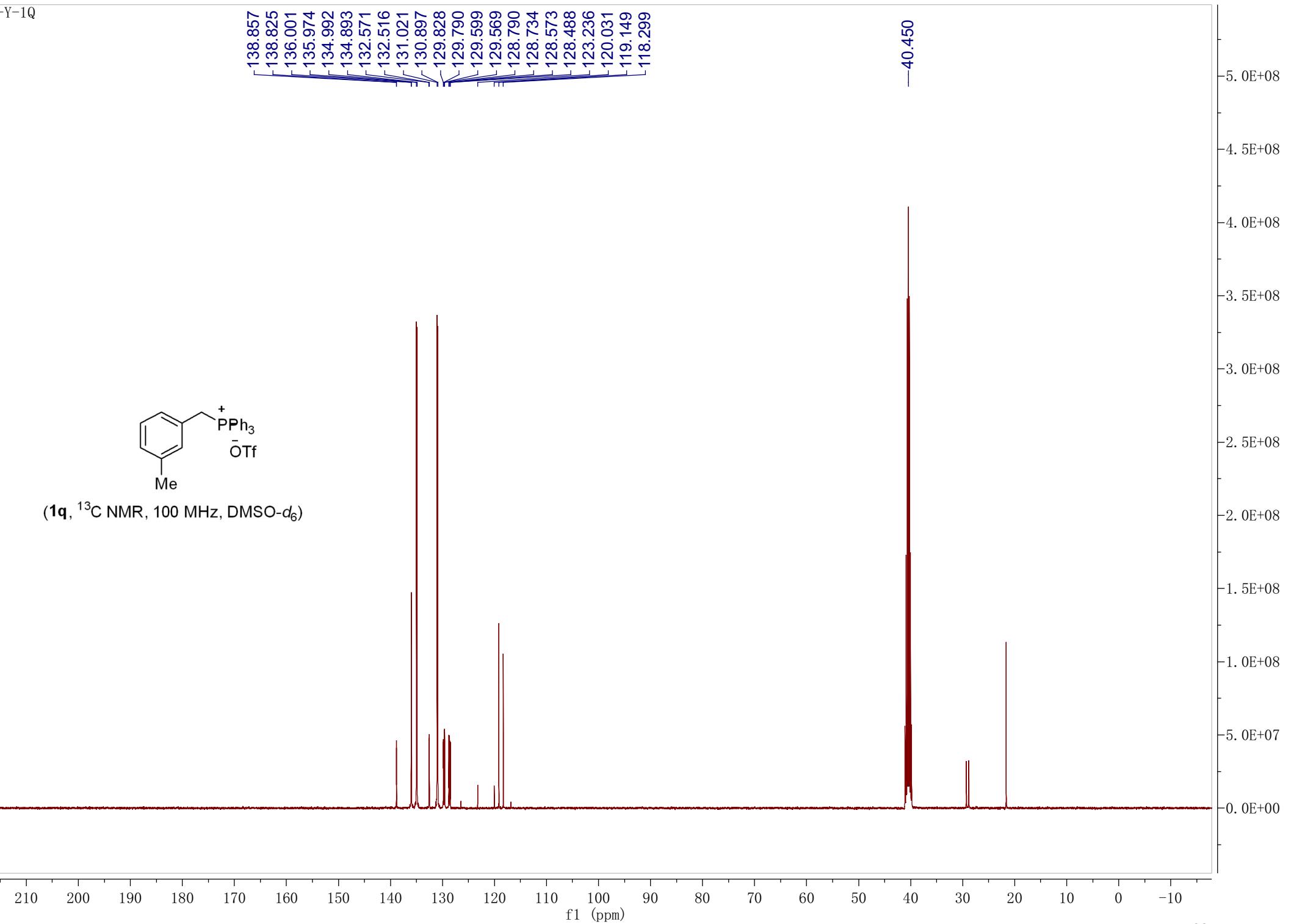
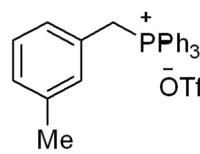


(**1q**, ^1H NMR, 400 MHz, DMSO- d_6)

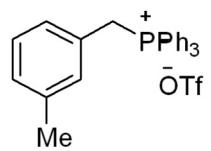


138.857
138.825
136.001
135.974
134.992
134.893
132.571
132.516
131.021
130.897
129.828
129.790
129.599
129.569
128.790
128.734
128.573
128.488
123.236
120.031
119.149
118.299

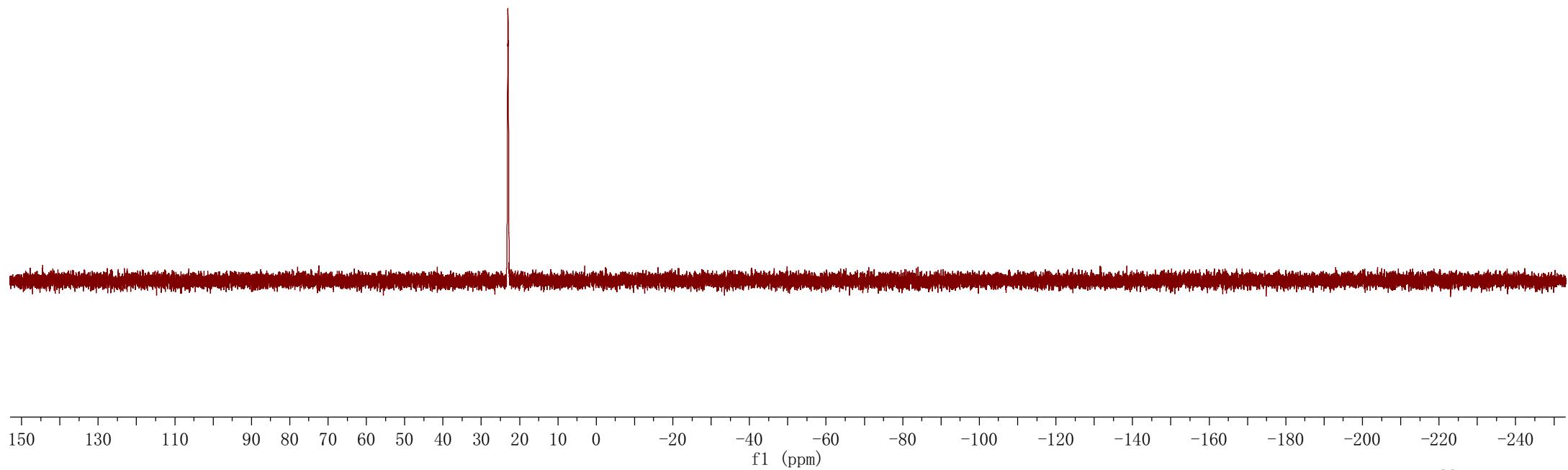
-40.450



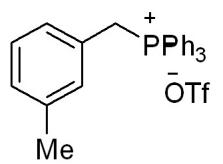
-23.027



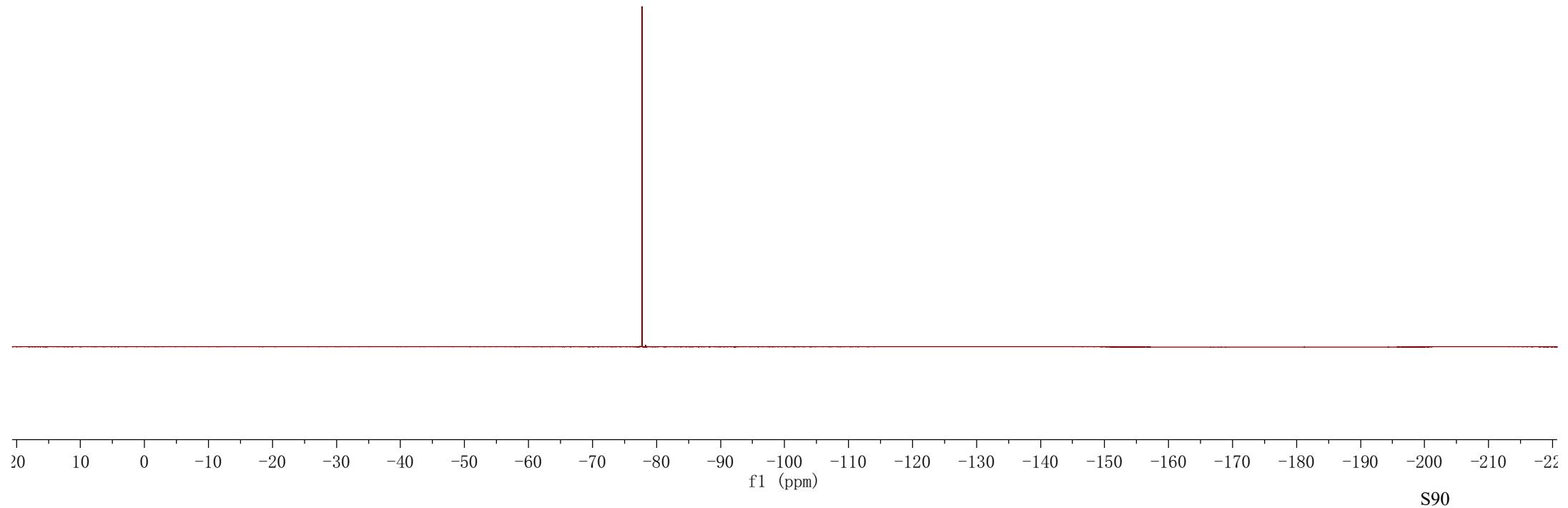
(**1q**, ^{31}P NMR, 162 MHz, DMSO- d_6)



—77.736



(**1q**, ^{19}F NMR, 376 MHz, DMSO- d_6)

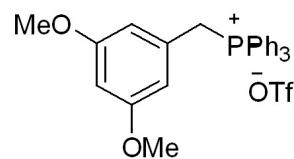


7.931
7.915
7.912
7.895
7.781
7.772
7.761
7.752
7.742
7.733
7.699
7.680
7.668
7.649

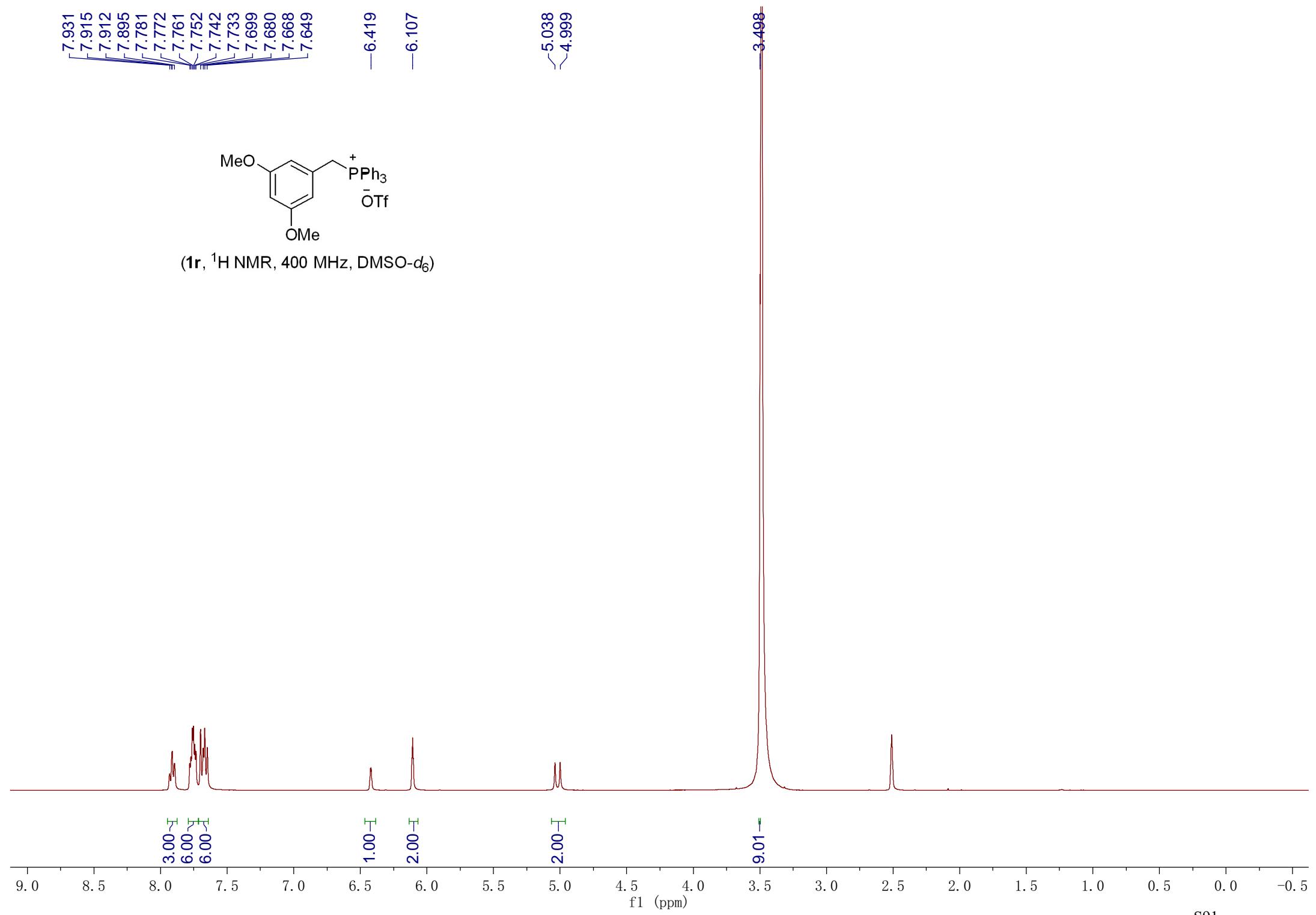
—6.419
—6.107

5.038
4.999

3.498



(**1r**, ^1H NMR, 400 MHz, DMSO- d_6)



28.817

28.348

—55.170

100.342

100.375

108.995

109.049

117.463

118.315

119.153

120.842

129.926

130.151

130.274

134.070

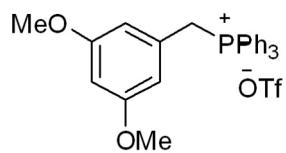
134.167

135.237

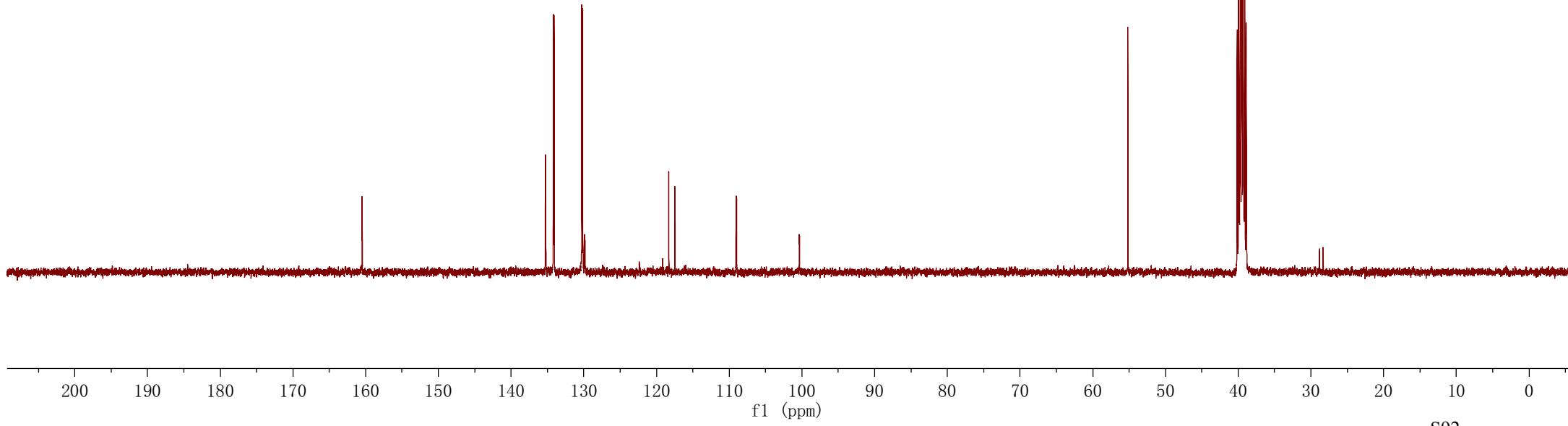
135.264

160.478

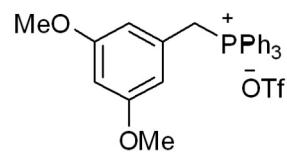
160.509



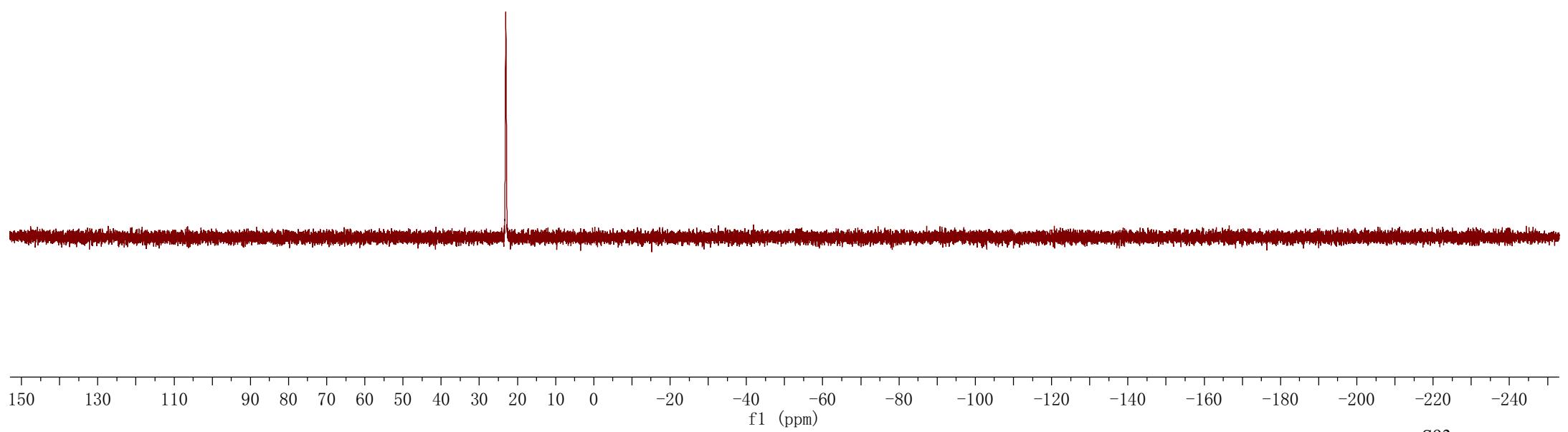
(1r, ¹³C NMR, 100 MHz, DMSO-d₆)



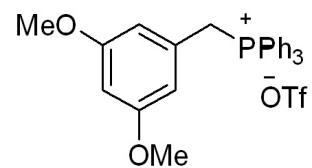
-23.043



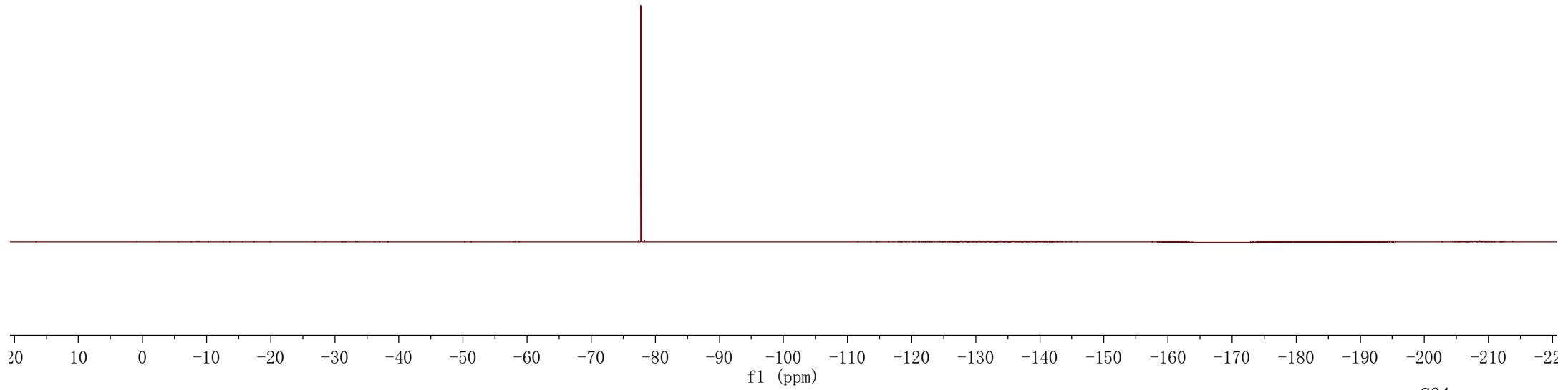
(**1r**, ^{31}P NMR, 162 MHz, $\text{DMSO}-d_6$)



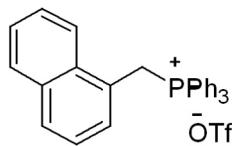
—77.758



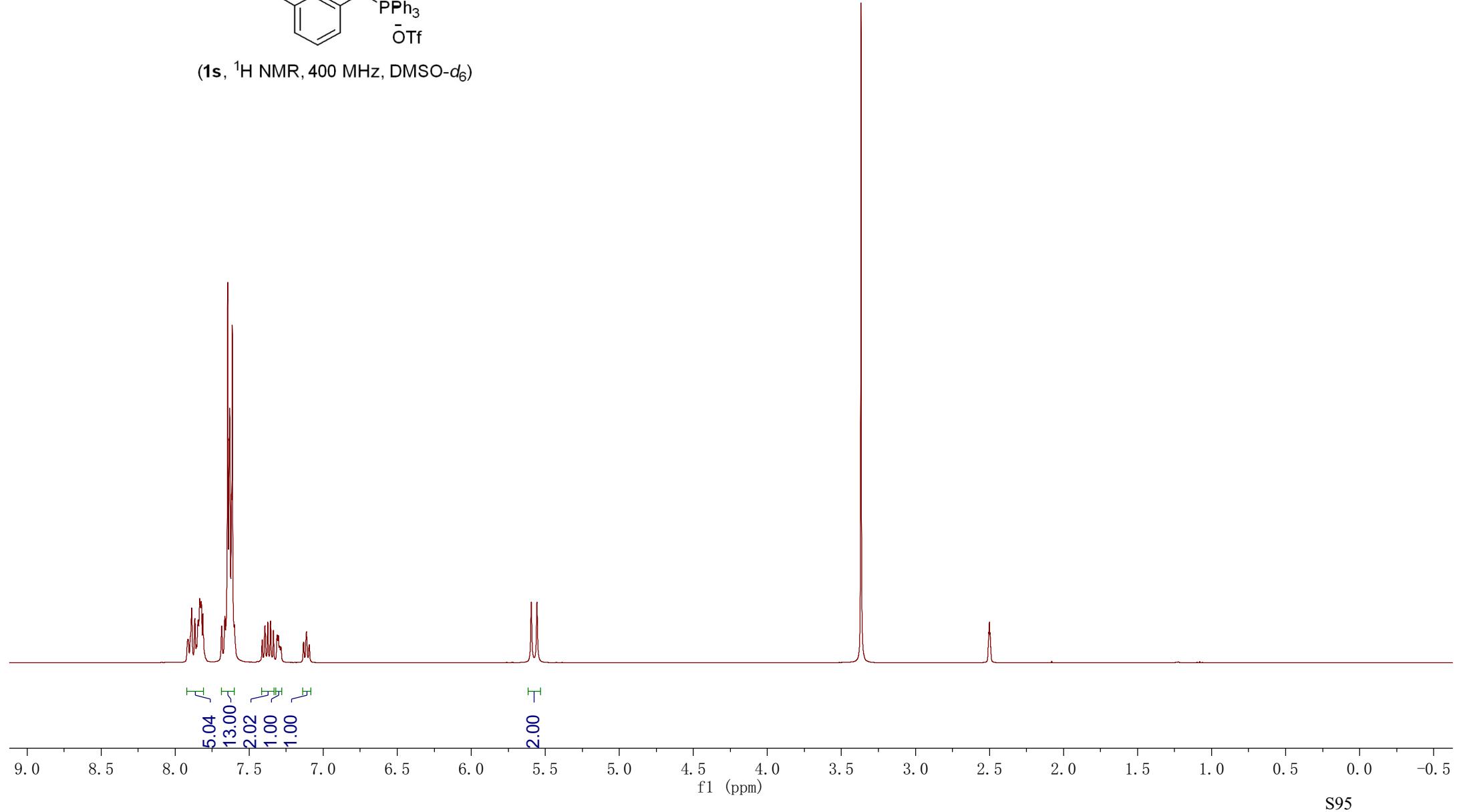
(1r, ^{19}F NMR, 376 MHz, DMSO- d_6)

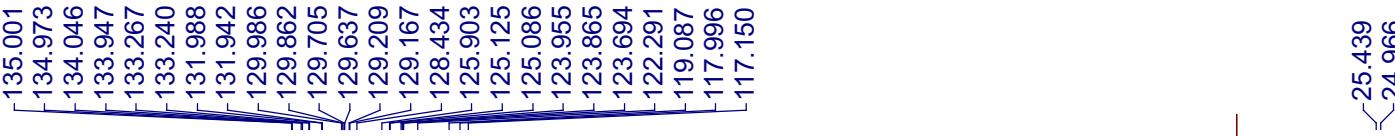


7.914
7.888
7.867
7.844
7.834
7.829
7.823
7.813
7.808
7.686
7.664
7.645
7.635
7.632
7.619
7.614
7.599
7.394
7.374
7.355
7.336
7.310
7.303
7.113
5.556

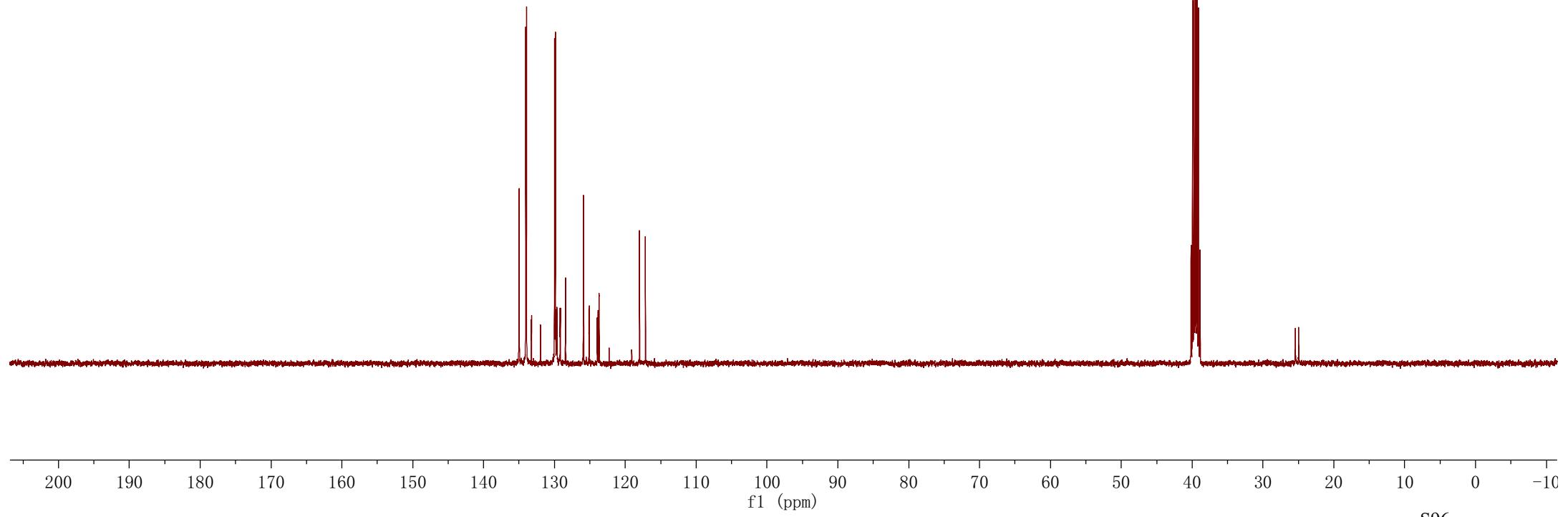
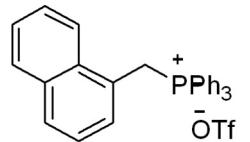


(**1s**, ^1H NMR, 400 MHz, DMSO- d_6)

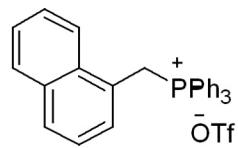




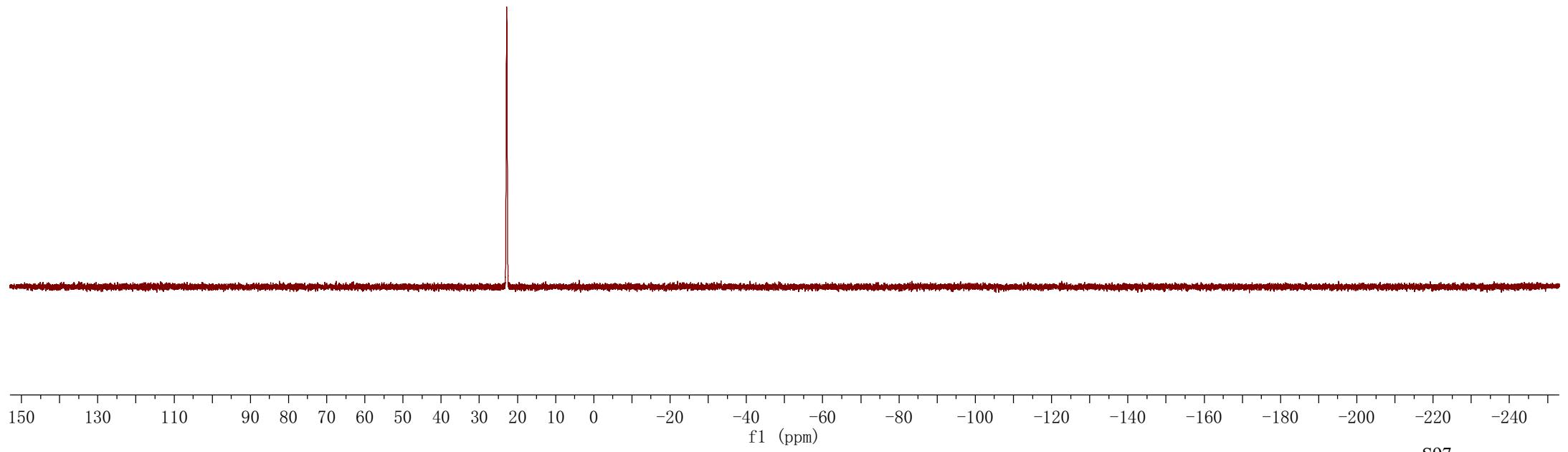
(**1s**, ^{13}C NMR, 100 MHz, DMSO-d_6)



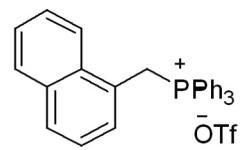
-22.817



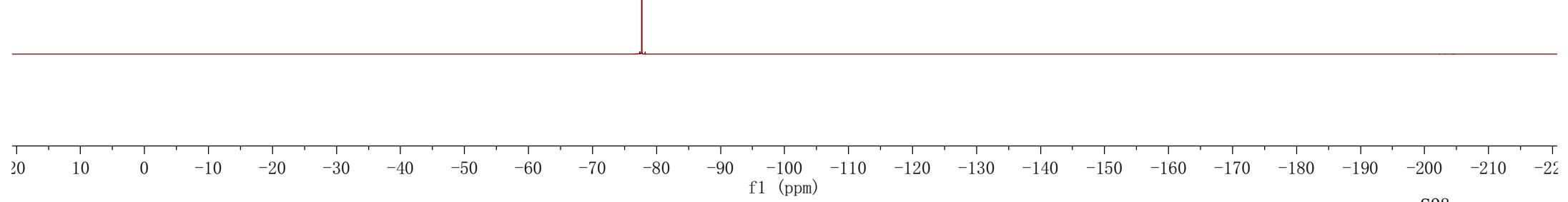
(1s, ^{31}P NMR, 162 MHz, DMSO- d_6)



-77.697

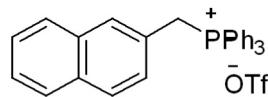


(**1s**, ^{19}F NMR, 376 MHz, $\text{DMSO}-d_6$)

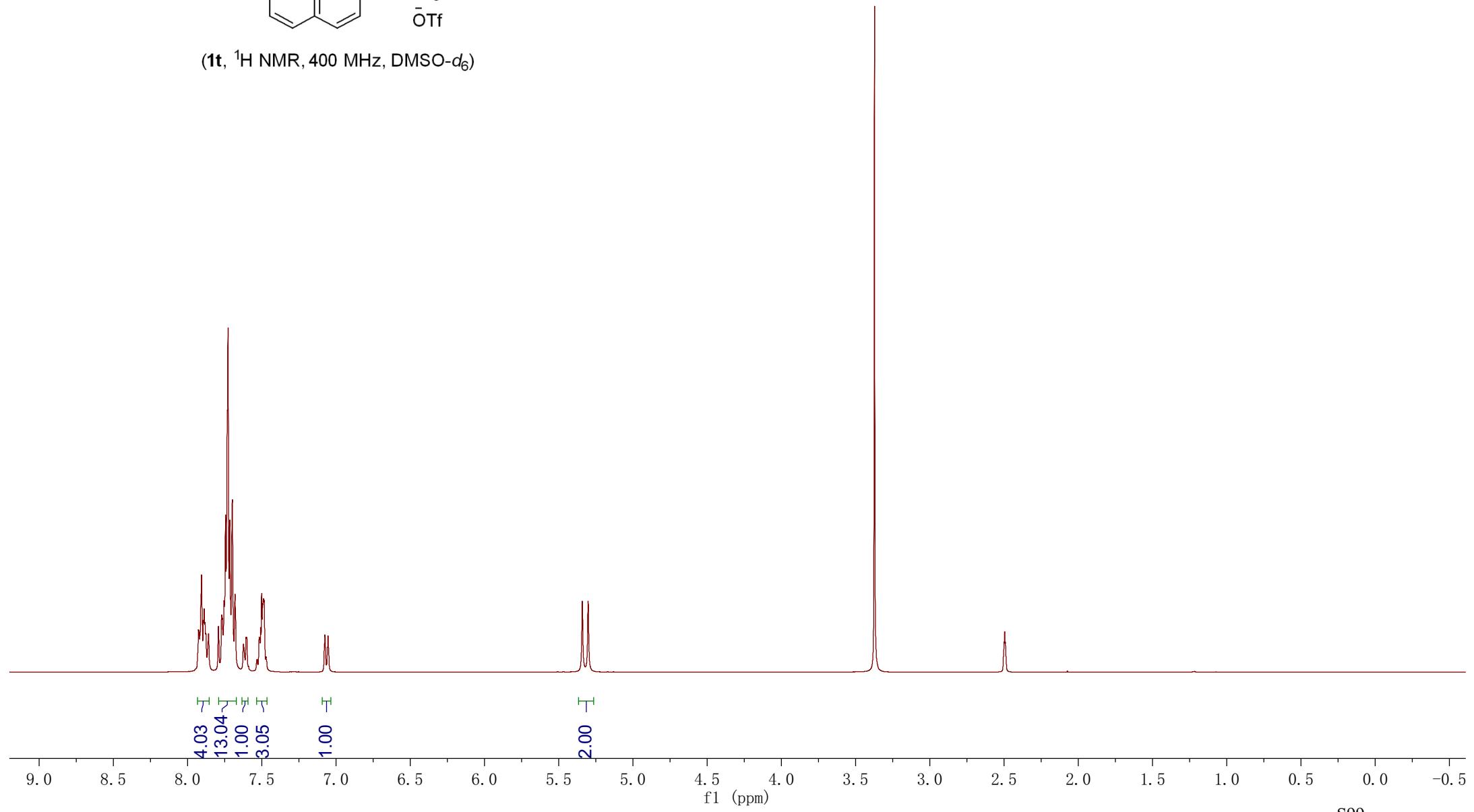


7.906
7.888
7.770
7.755
7.744
7.731
7.727
7.716
7.698
7.680
7.501
7.494
7.488
7.075
7.054

5.341
5.302

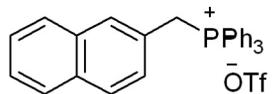


(**1t**, ^1H NMR, 400 MHz, DMSO- d_6)

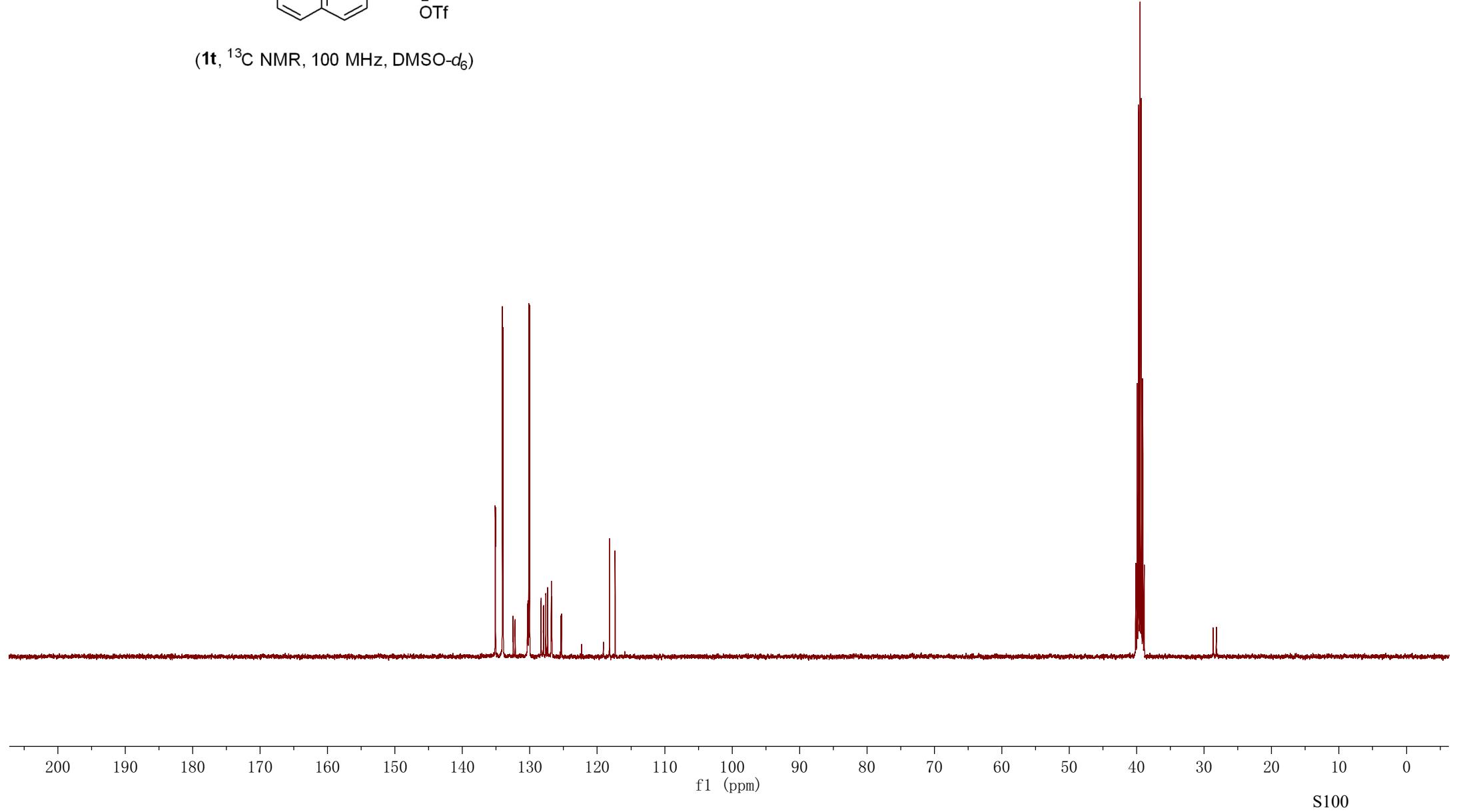


²⁸661
²⁸197

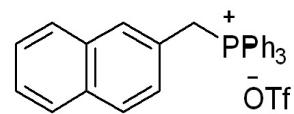
117.348
118.199
119.100
120.303
125.398
127.360
127.635
127.956
127.998
128.323
128.346
130.036
130.159
132.189
132.462
132.493
133.959
134.058
135.091
135.119



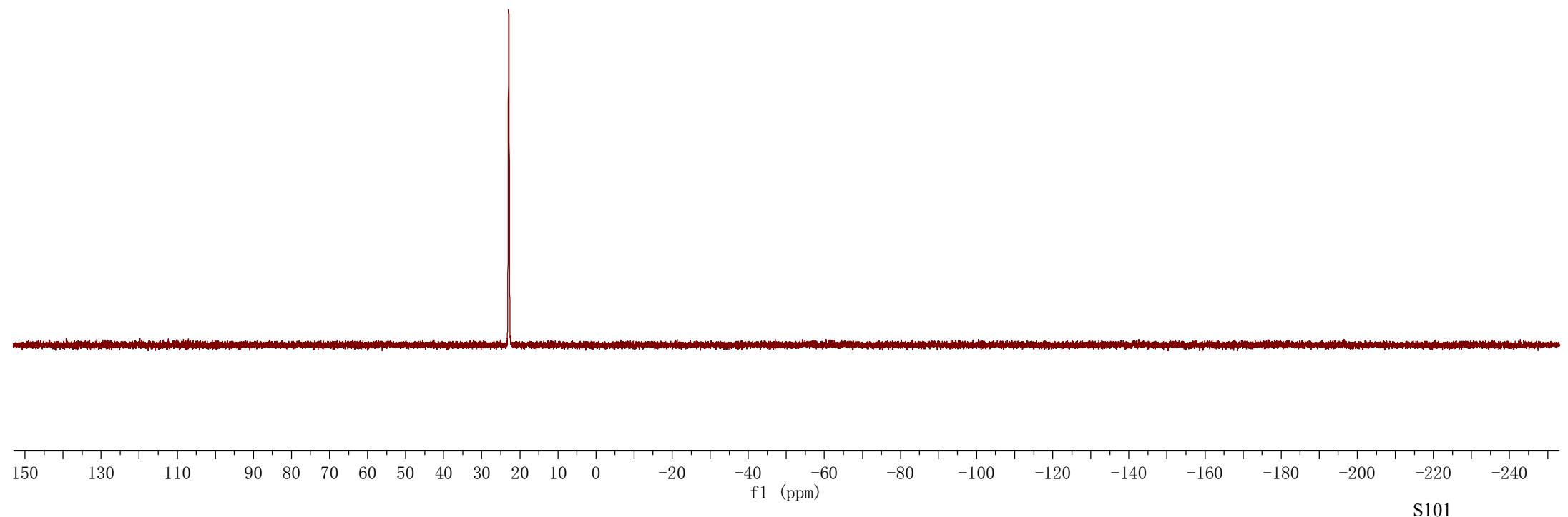
(**1t**, ¹³C NMR, 100 MHz, DMSO-*d*₆)



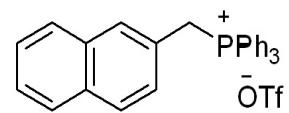
-22.915



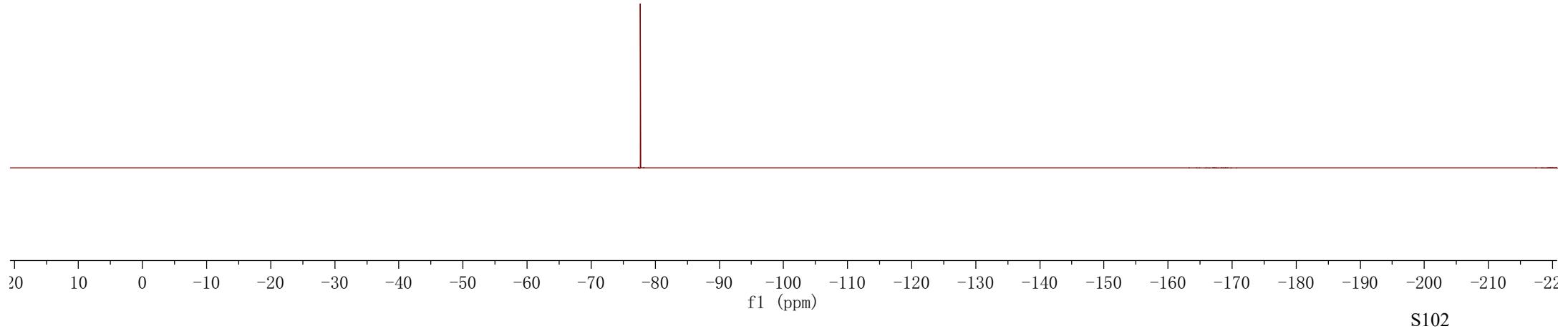
(**1t**, ^{31}P NMR, 162 MHz, $\text{DMSO}-d_6$)

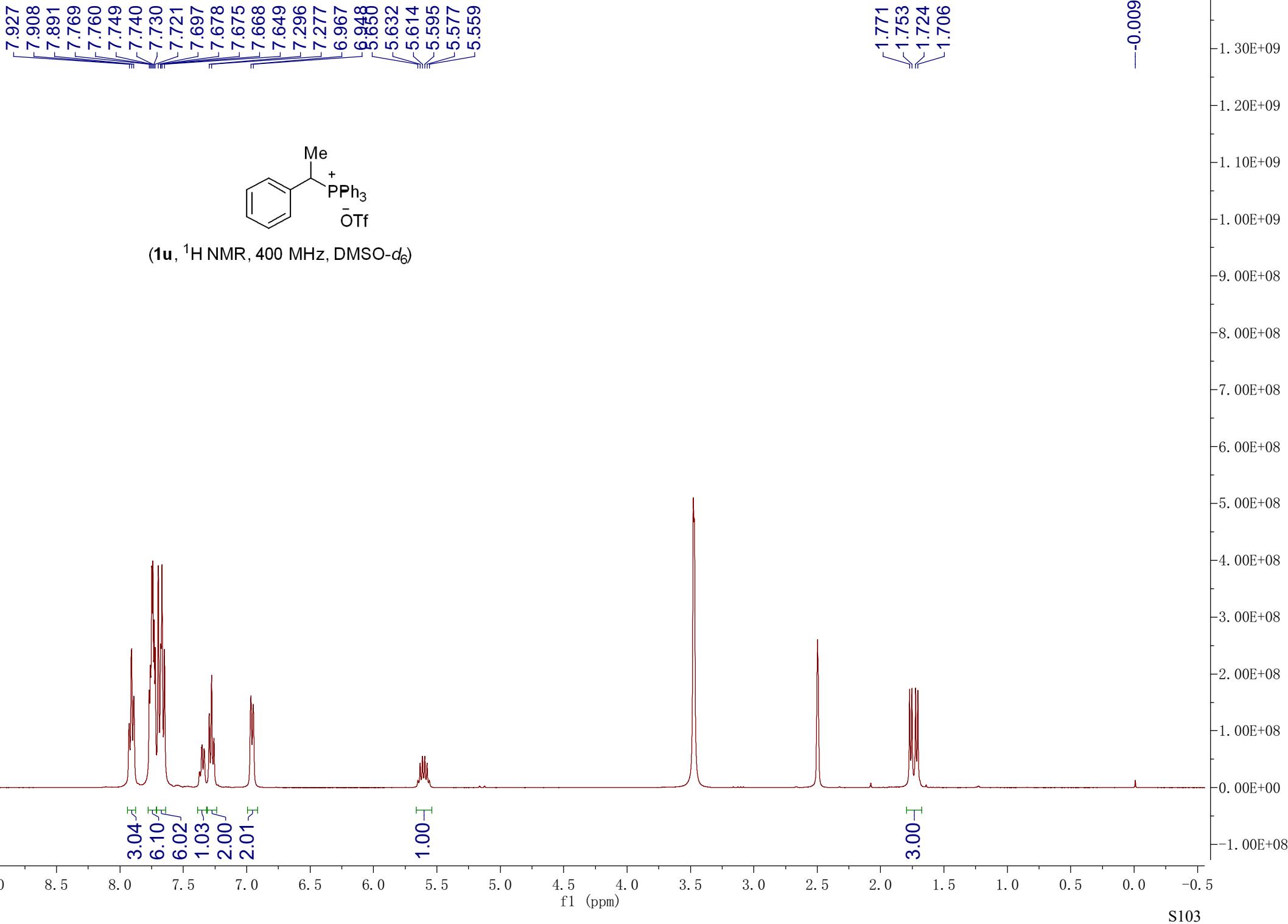


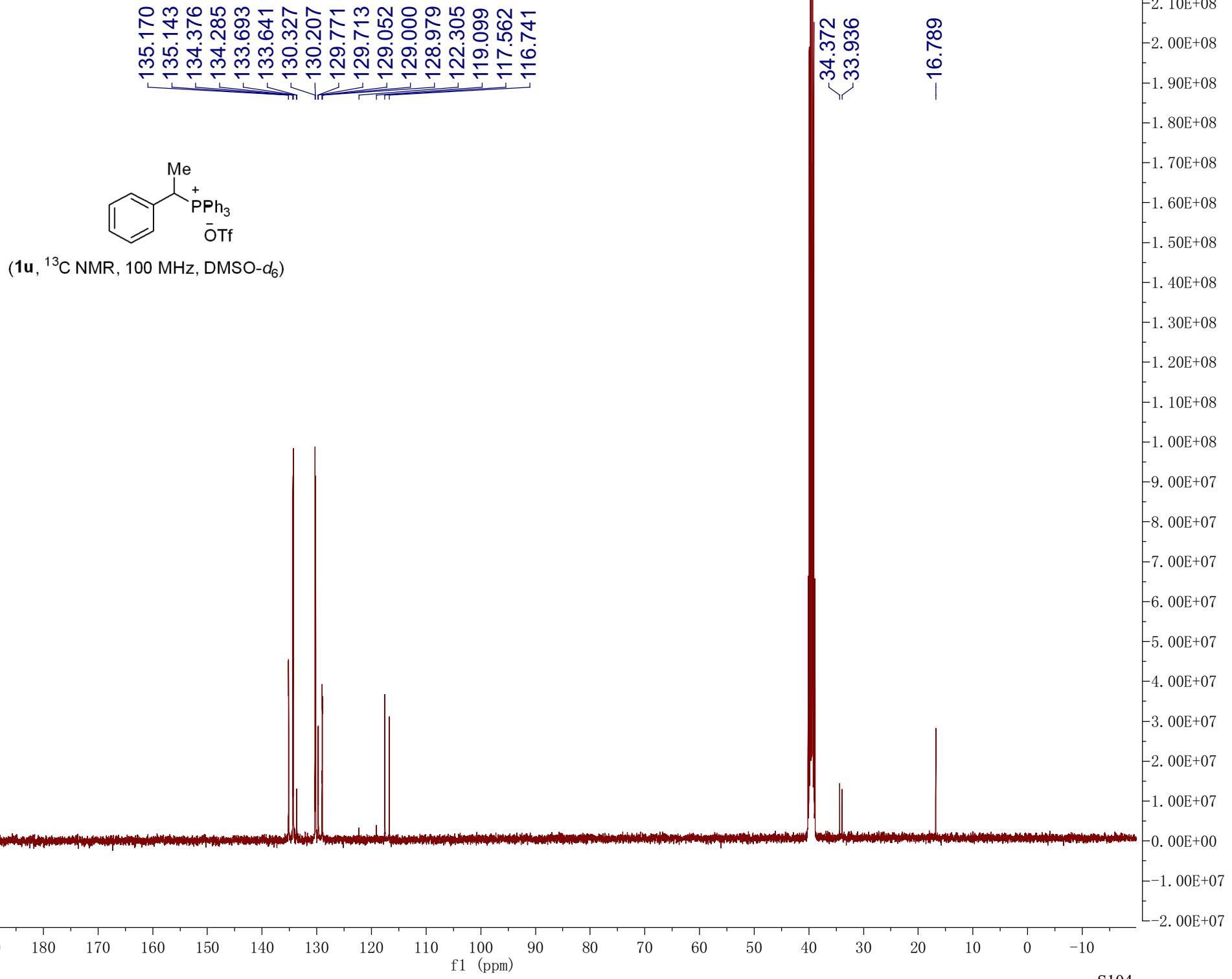
-77.688

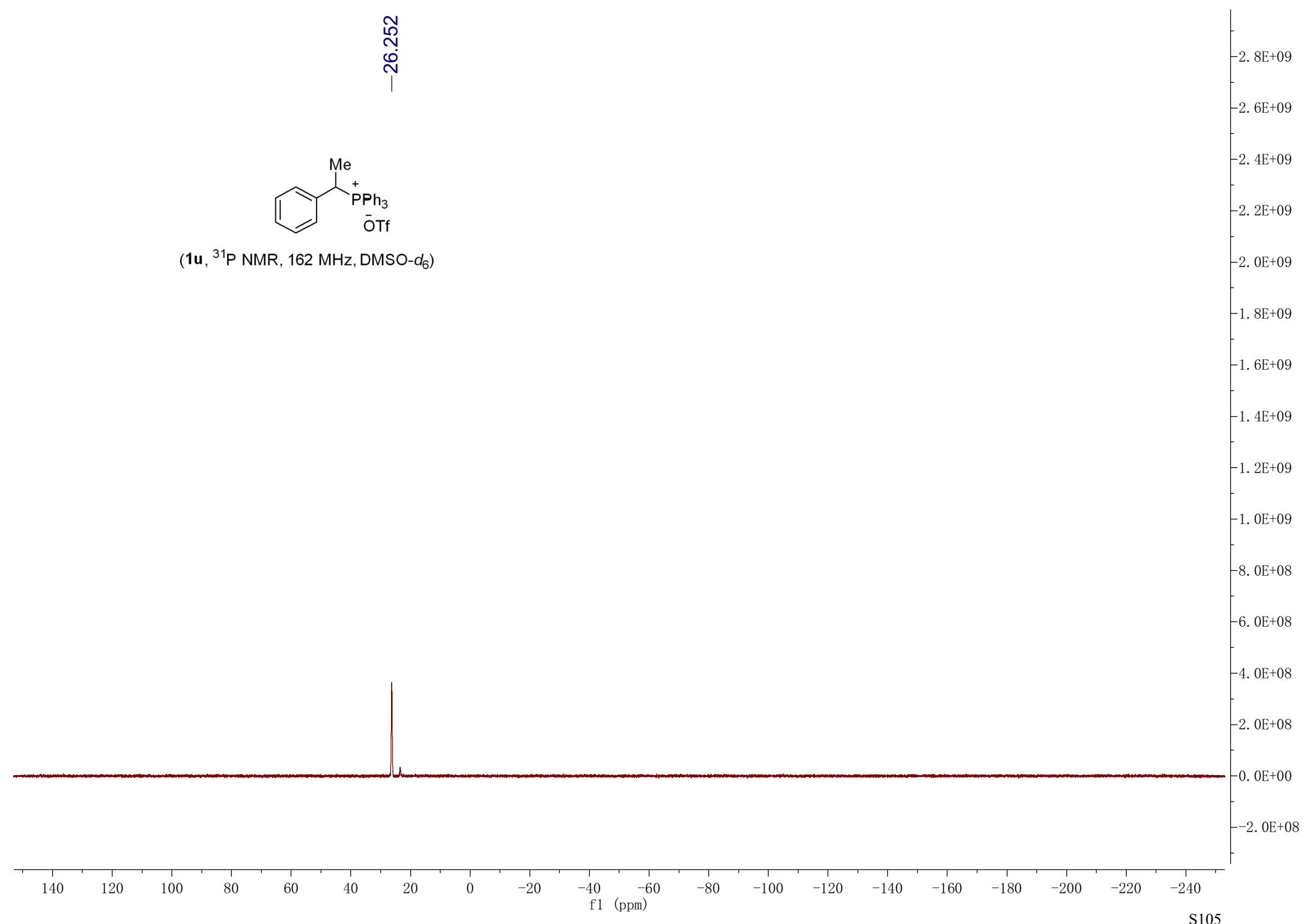


(**1t**, ^{19}F NMR, 376 MHz, $\text{DMSO}-d_6$)



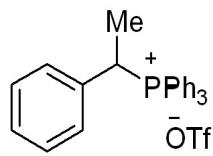






-2.8E+09
-2.6E+09
-2.4E+09
-2.2E+09
-2.0E+09
-1.8E+09
-1.6E+09
-1.4E+09
-1.2E+09
-1.0E+09
-8.0E+08
-6.0E+08
-4.0E+08
-2.0E+08
0.0E+00
-2.0E+08

—77.736



(**1u**, ^{19}F NMR, 376 MHz, $\text{DMSO}-d_6$)

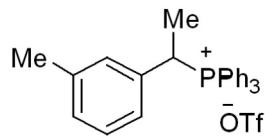
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220

f1 (ppm)

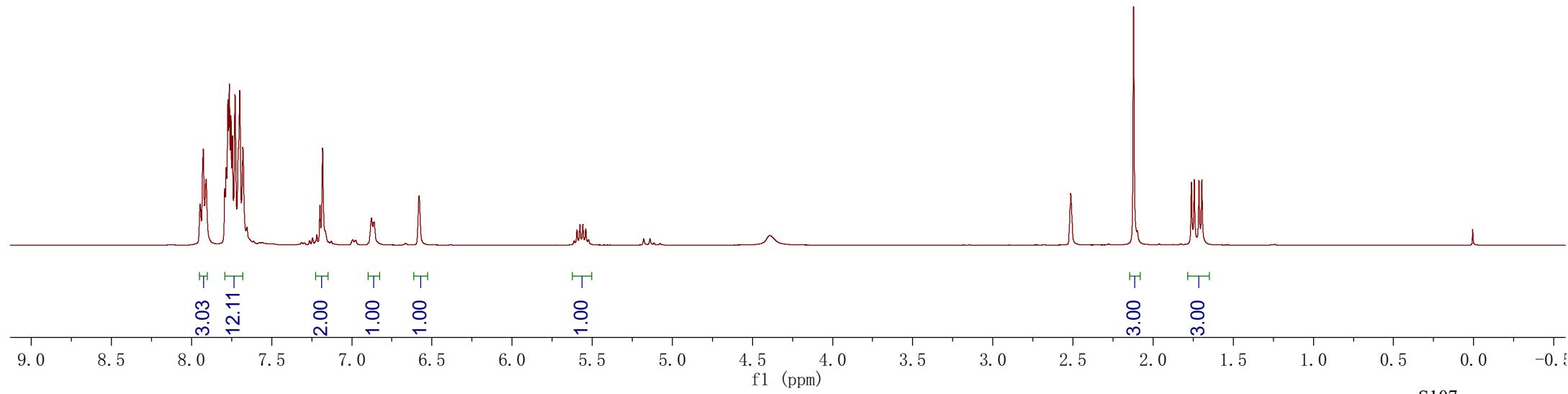
7.946
7.927
7.909
7.793
7.784
7.773
7.764
7.755
7.745
7.728
7.699
7.680
7.218
7.199
7.182
6.877
6.860
-6.580

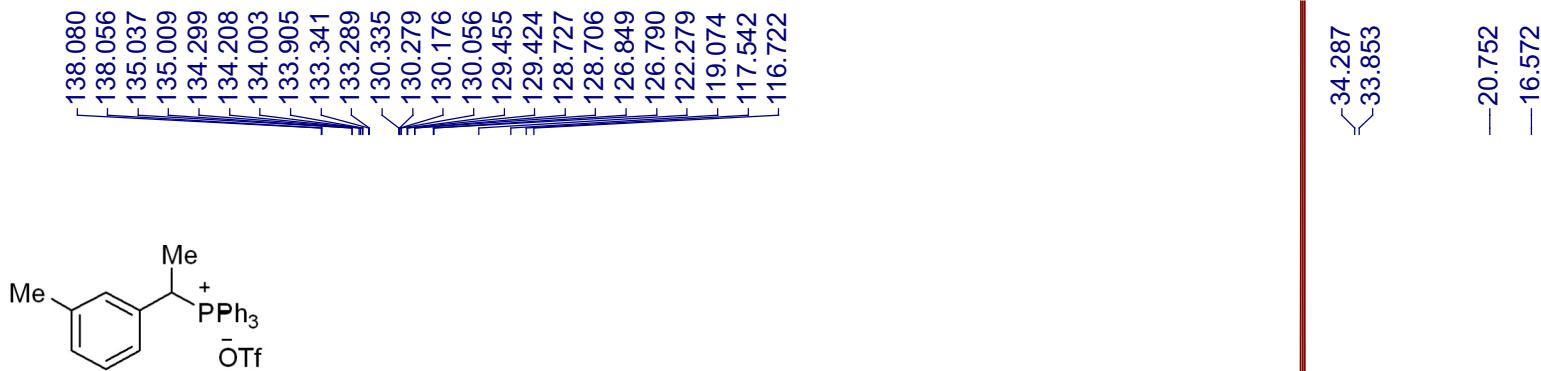
5.613
5.595
5.577
5.559
5.540
5.523

-2.122
-1.761
-1.743
-1.714
-1.696

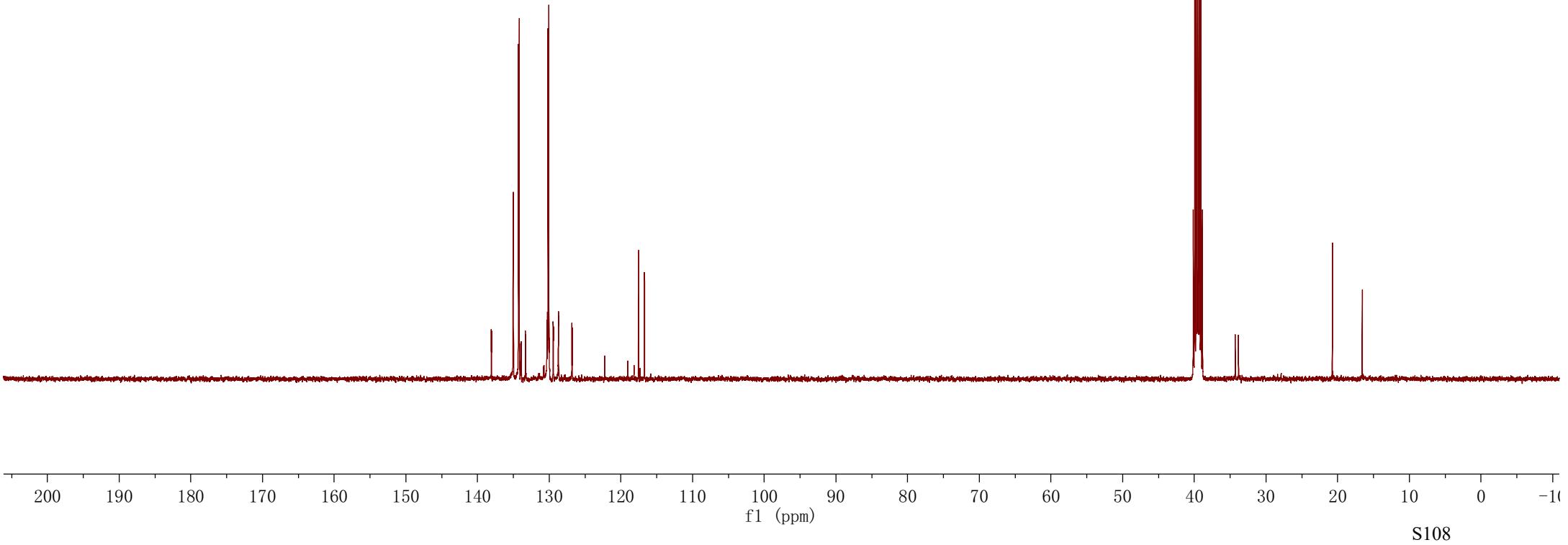
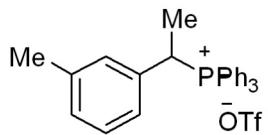


(**1v**, ^1H NMR, 400 MHz, DMSO- d_6)

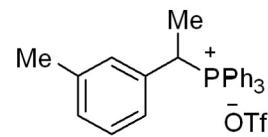




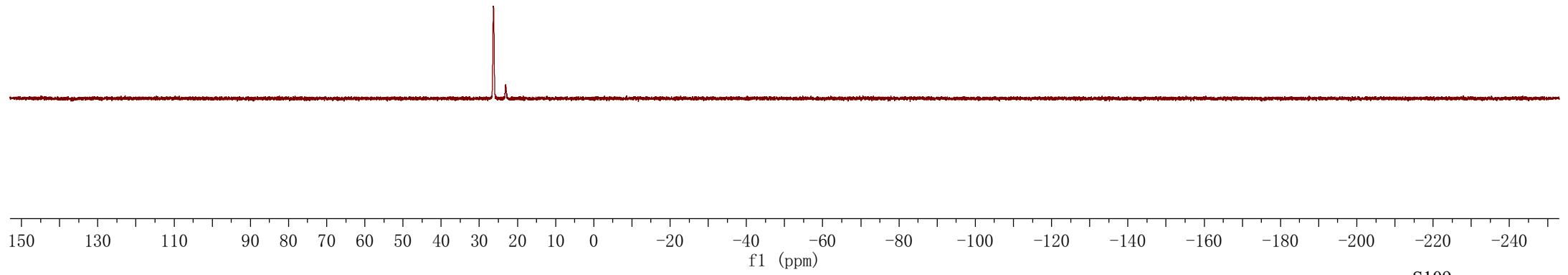
(**1v**, ^{13}C NMR, 100 MHz, DMSO- d_6)



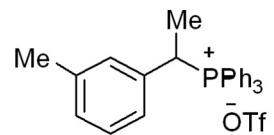
-26.280



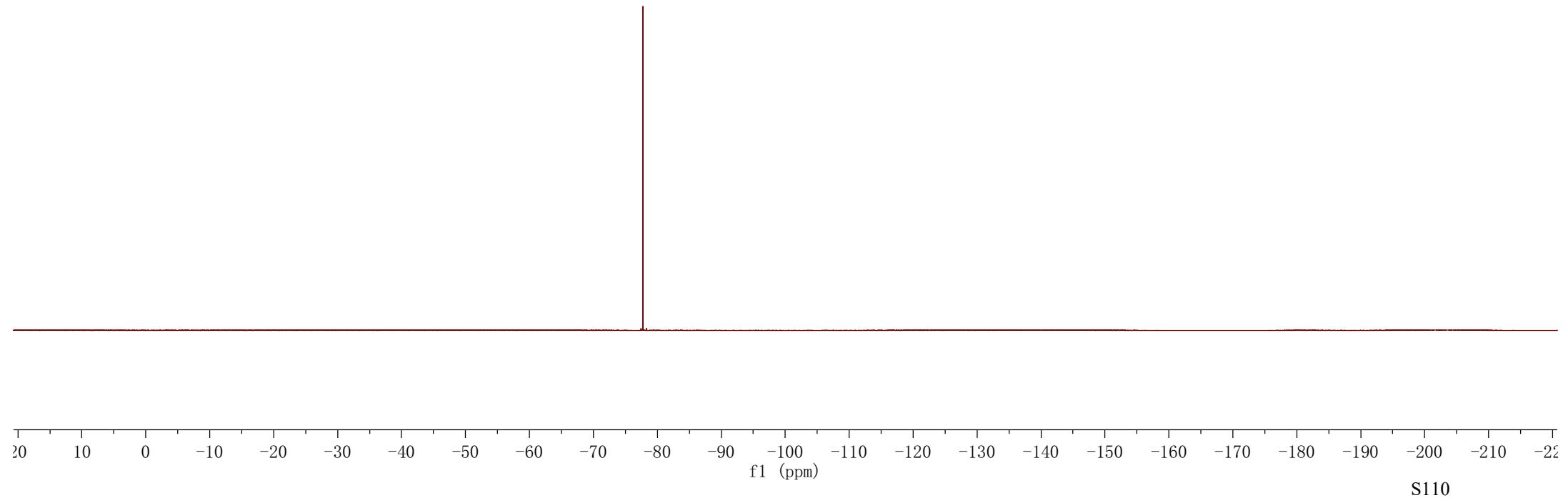
(**1v**, ^{31}P NMR, 162 MHz, $\text{DMSO}-d_6$)



-77.720

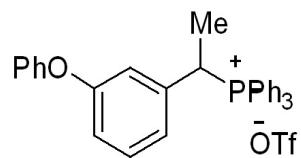


(**1v**, ^{19}F NMR, 376 MHz, $\text{DMSO}-d_6$)

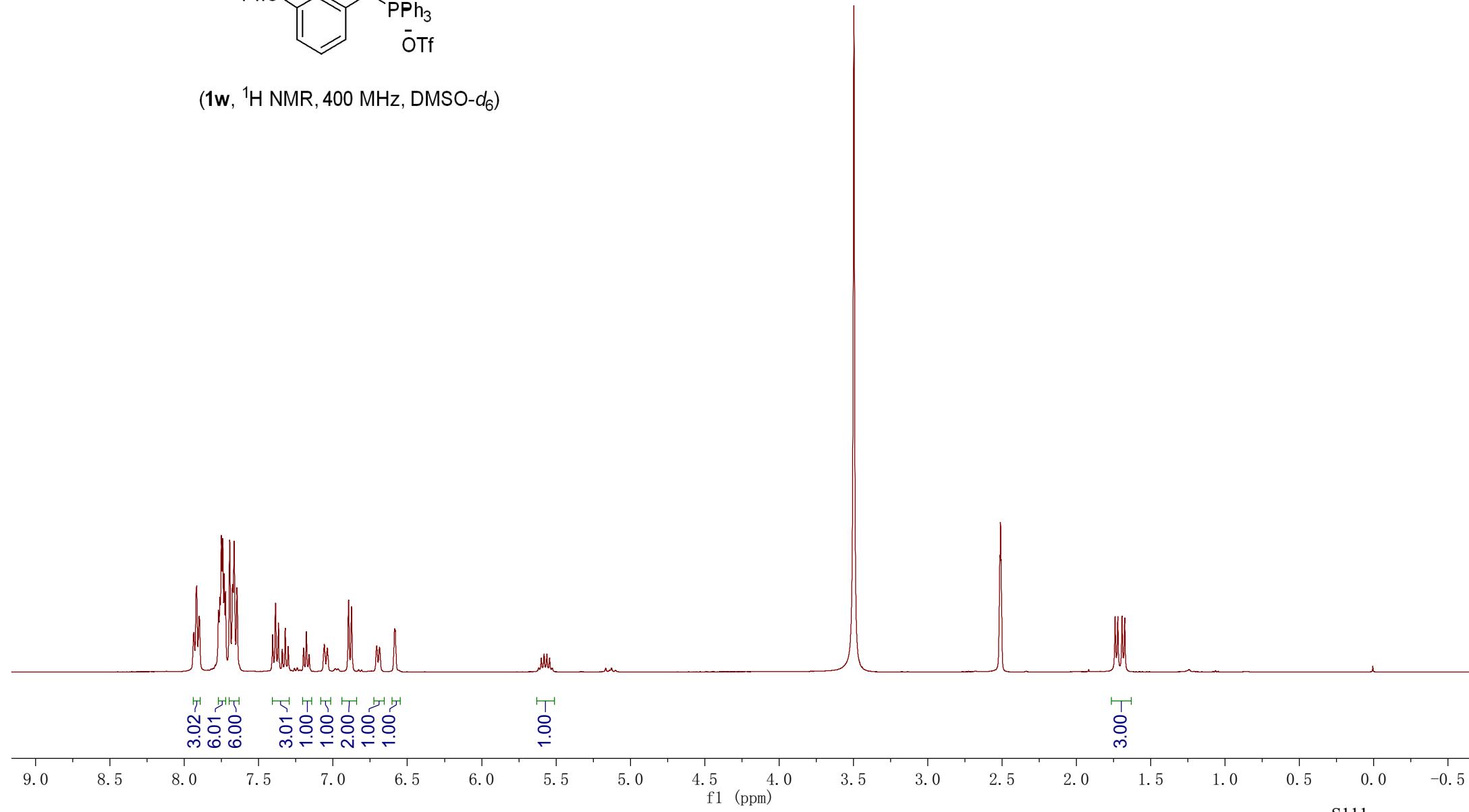


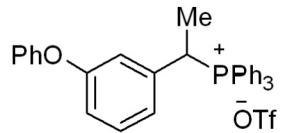
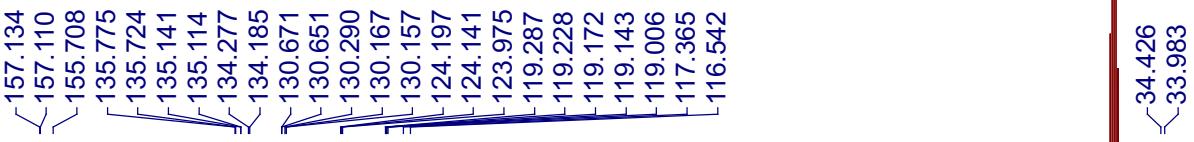
7.936
7.917
7.900
7.771
7.762
7.751
7.742
7.732
7.723
7.695
7.665
7.647
7.676
7.673
7.388
7.367
7.322
7.179
6.895
6.876
6.586
6.582
6.596
5.599
5.580
5.562
5.543
5.525

1.739
1.721
1.692
1.674

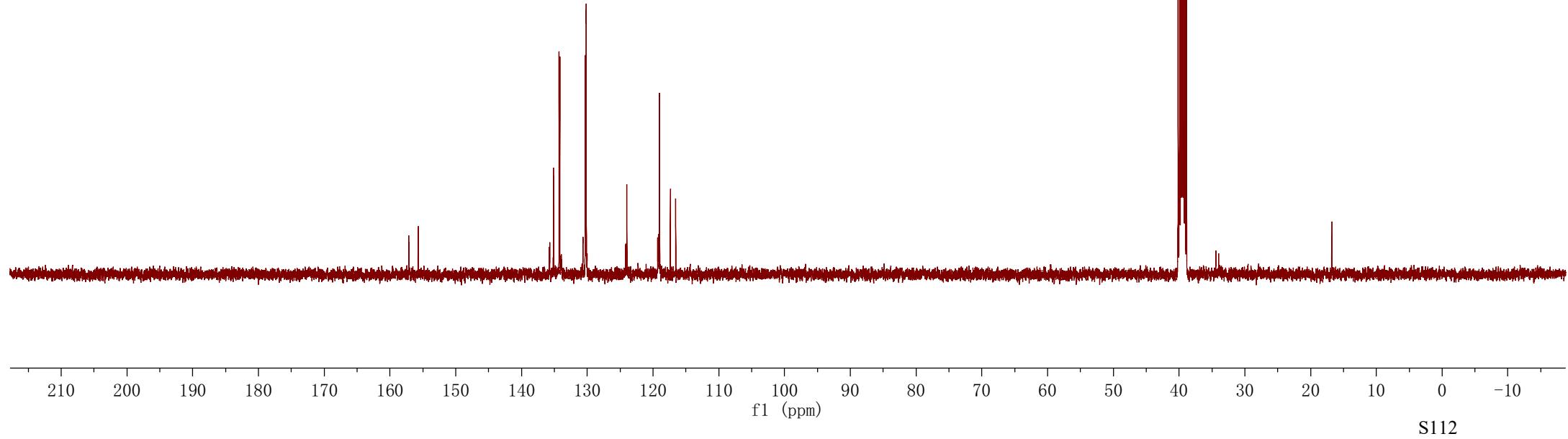


(**1w**, ^1H NMR, 400 MHz, DMSO- d_6)

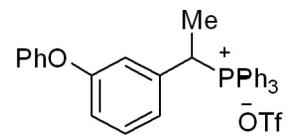




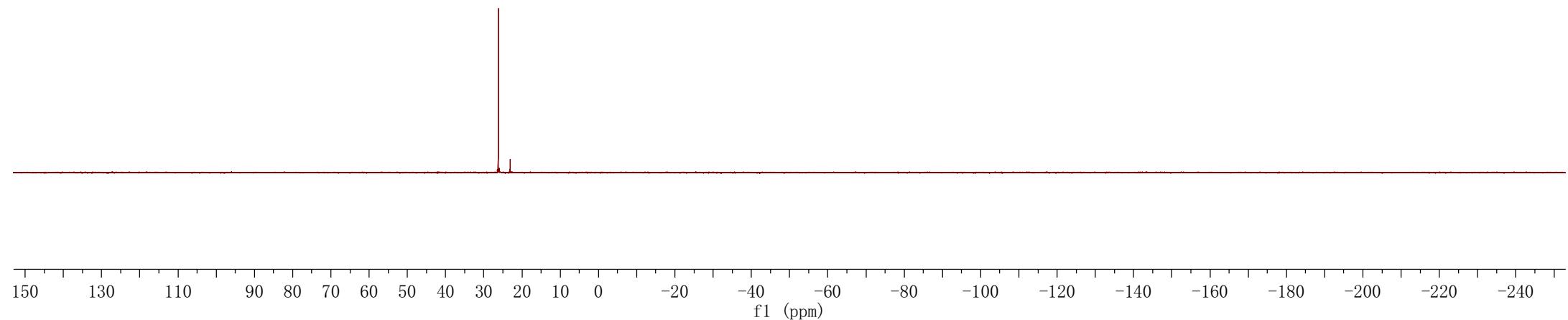
(**1w**, ^{13}C NMR, 100 MHz, $\text{DMSO}-d_6$)



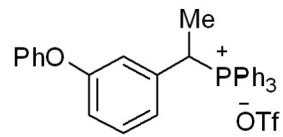
-26.216



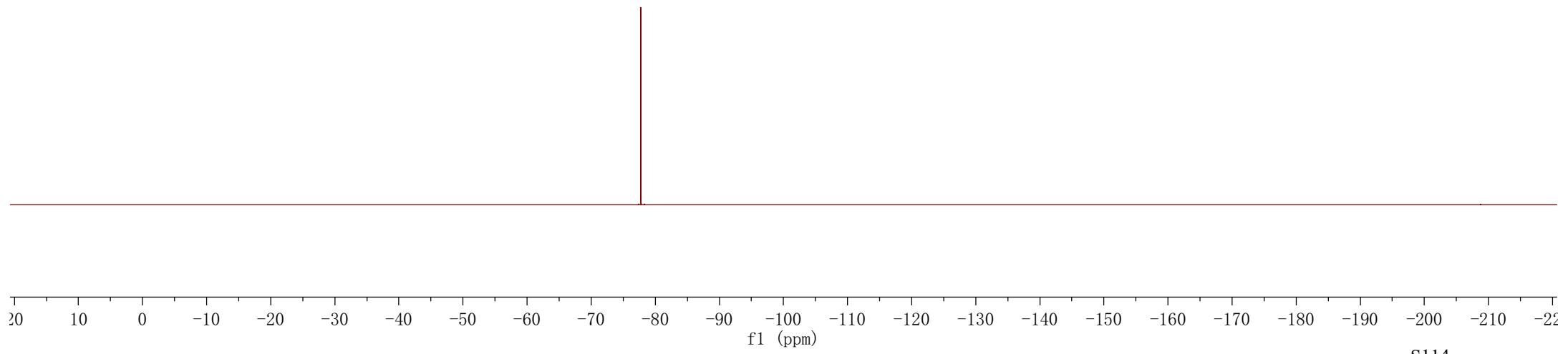
(**1w**, ^{31}P NMR, 162 MHz, DMSO- d_6)



—77.733



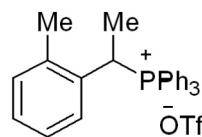
(**1w**, ^{19}F NMR, 376 MHz, $\text{DMSO}-d_6$)



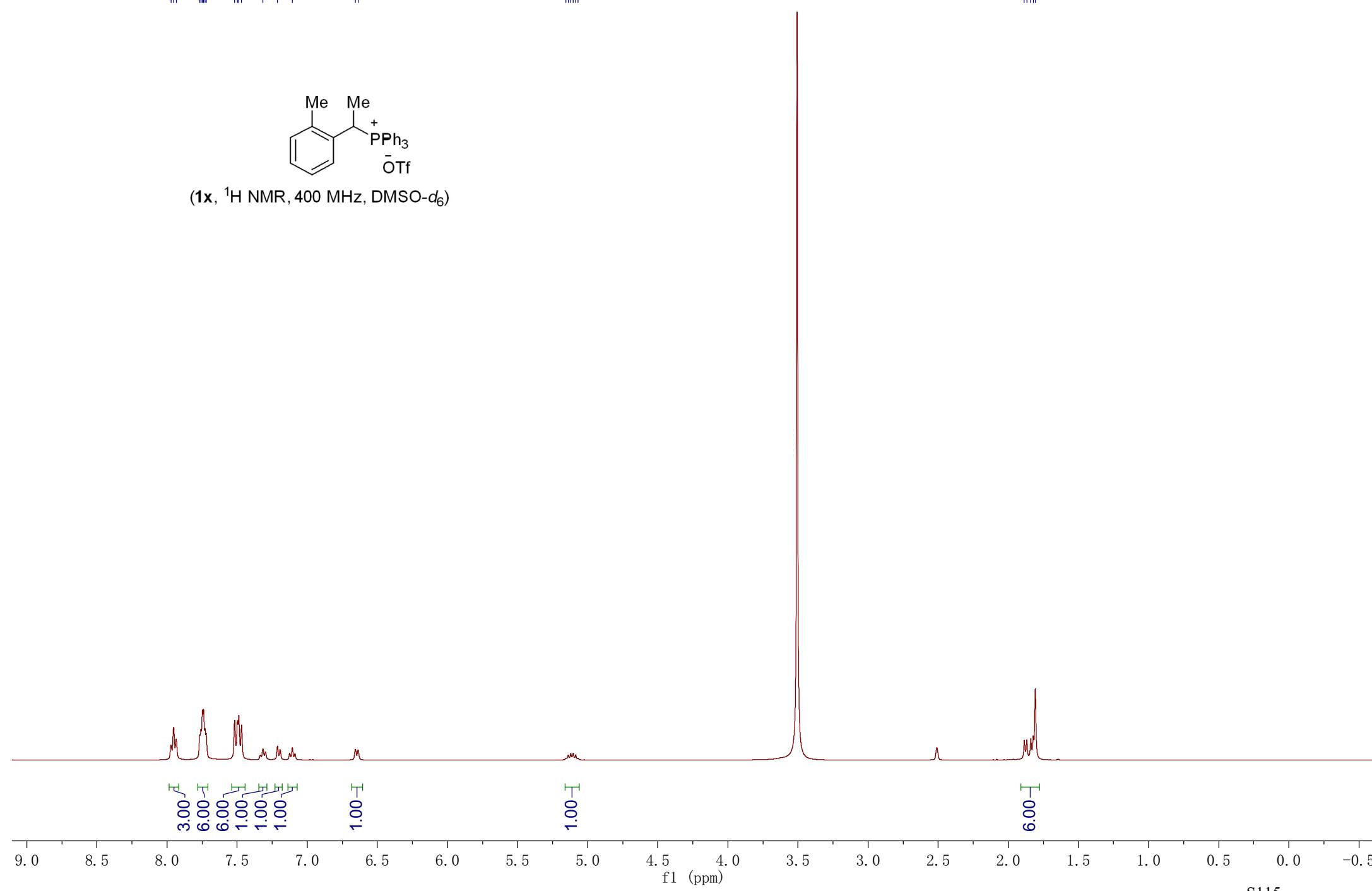
7.971
7.953
7.935
7.767
7.759
7.747
7.740
7.729
7.720
7.518
7.498
7.488
7.468
7.316
7.212
6.699
6.637

5.156
5.139
5.121
5.103
5.085
5.068

1.886
1.869
1.840
1.822
1.808

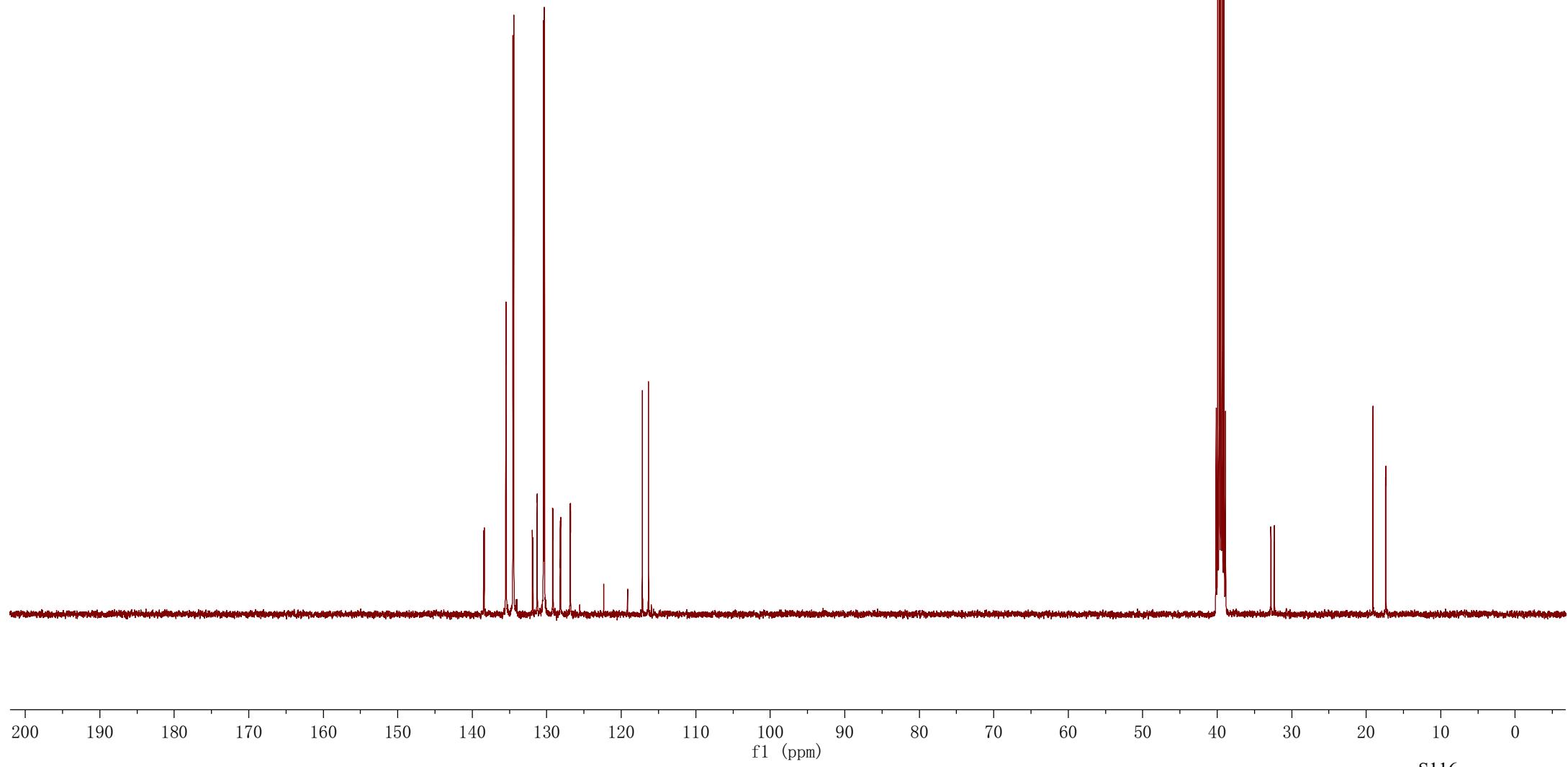
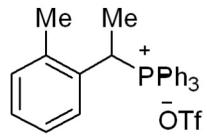


(**1x**, ^1H NMR, 400 MHz, $\text{DMSO}-d_6$)

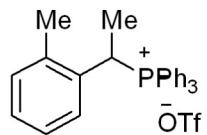




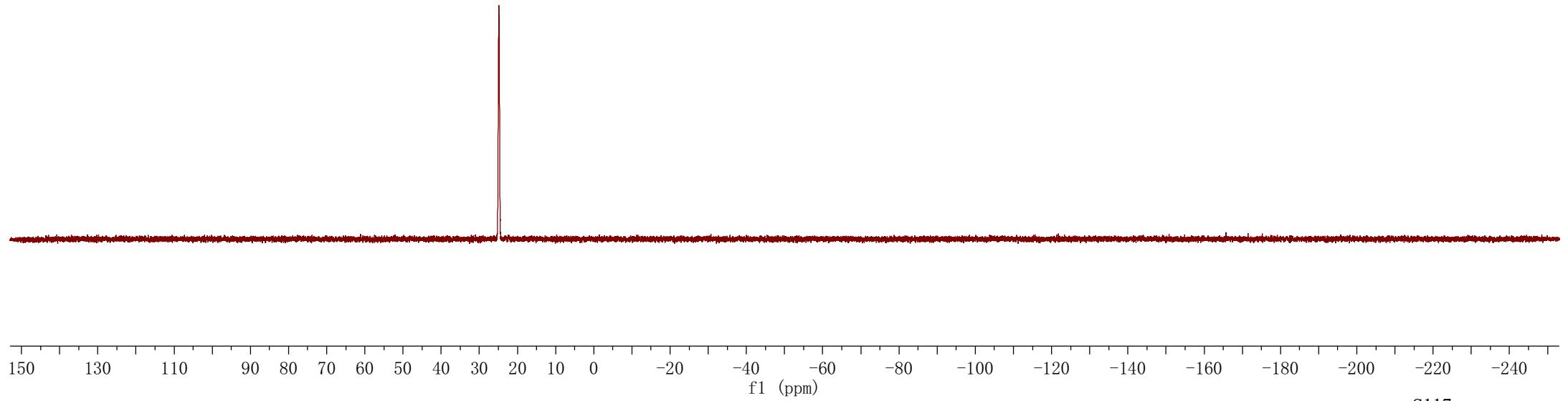
(**1x**, ^{13}C NMR, 100 MHz, $\text{DMSO}-d_6$)



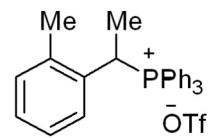
-24.871



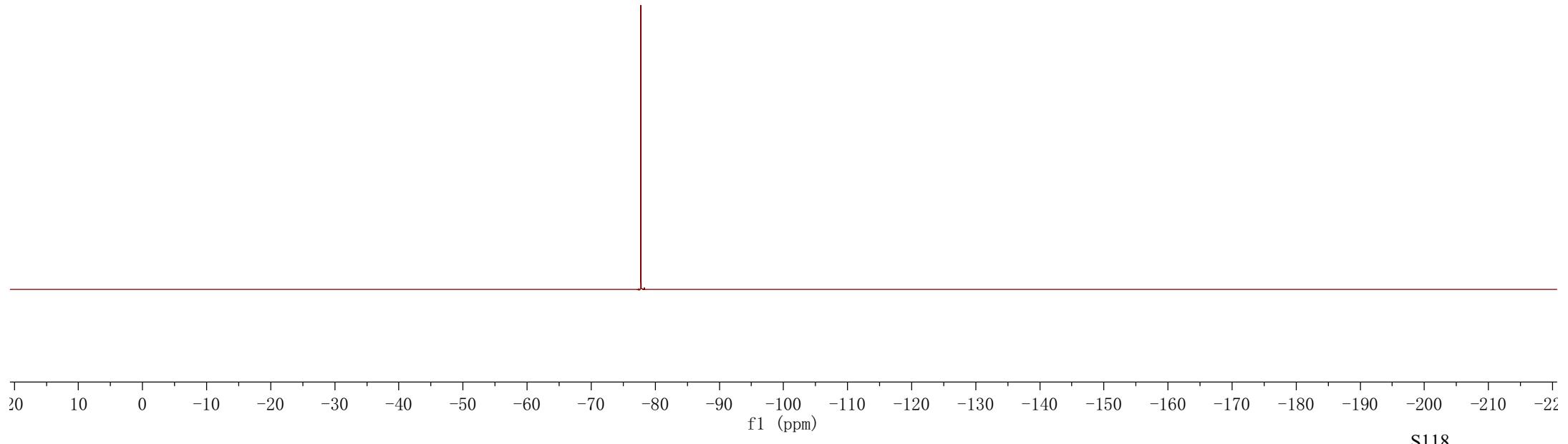
(**1x**, ^{31}P NMR, 162 MHz, DMSO- d_6)

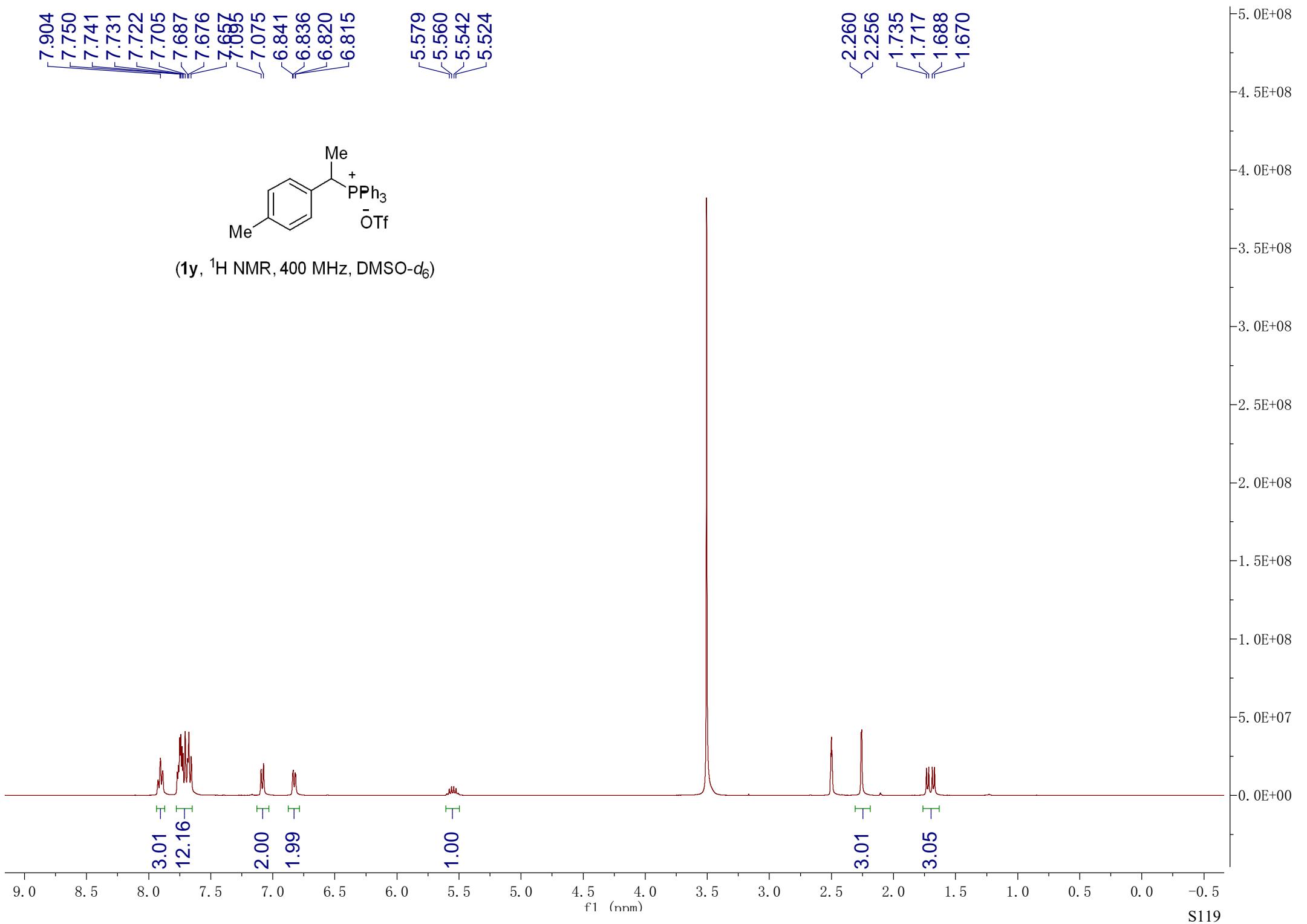


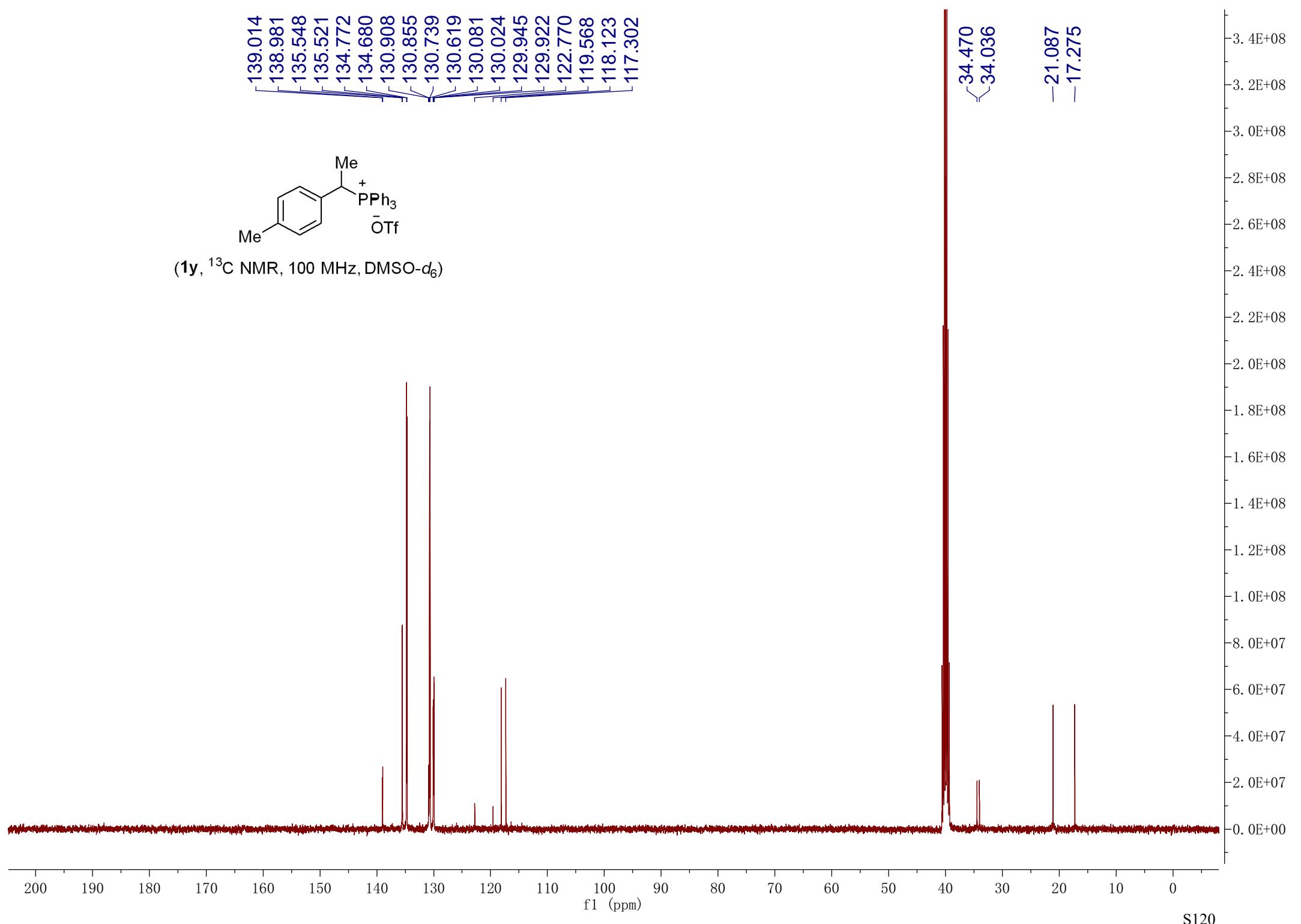
—77.740

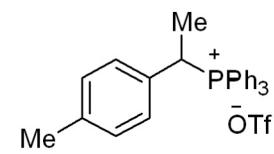


(**1x**, ^{19}F NMR, 376 MHz, DMSO- d_6)



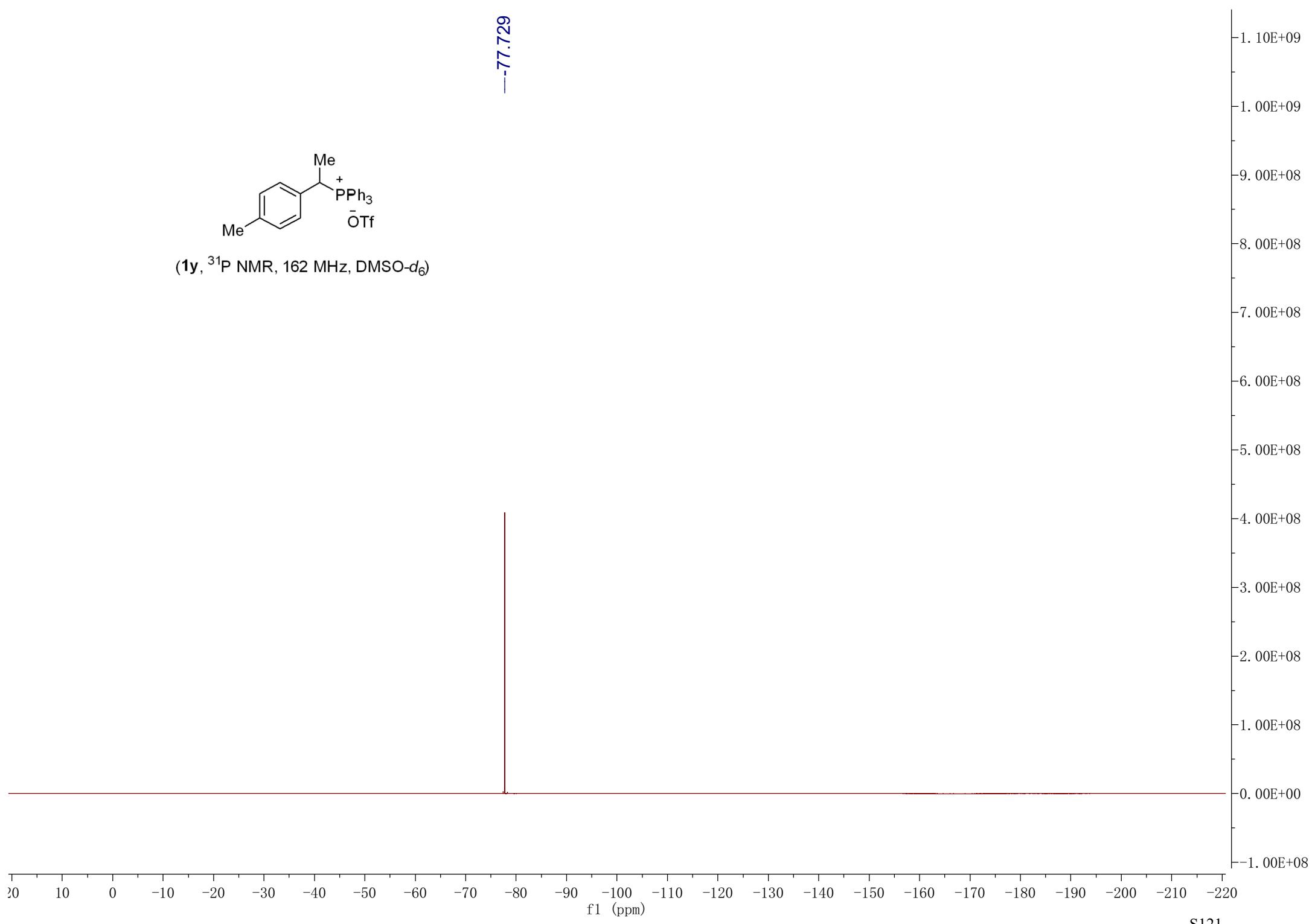




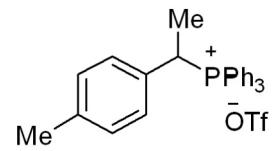


(**1y**, ^{31}P NMR, 162 MHz, $\text{DMSO}-d_6$)

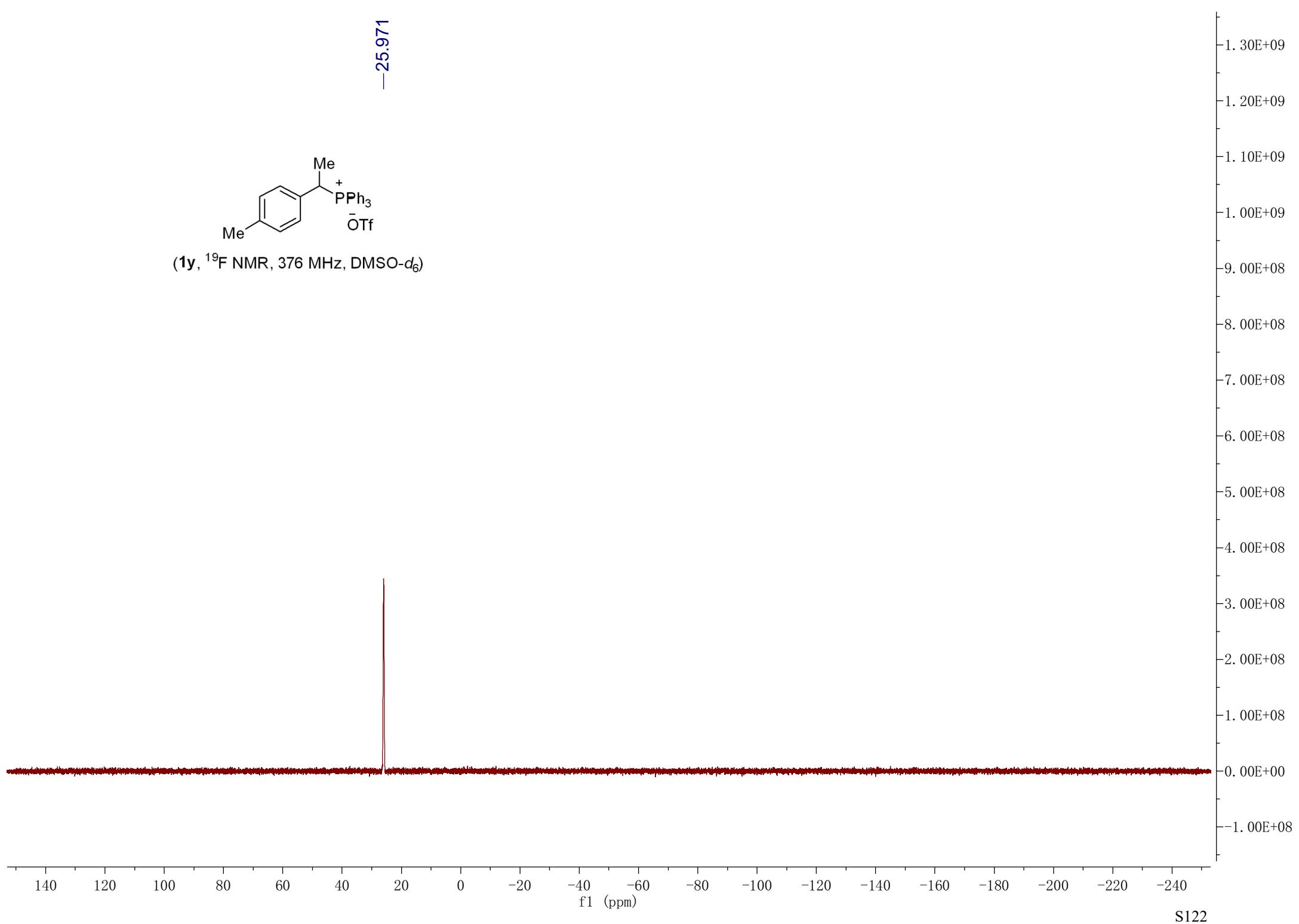
-77.729



-25.971



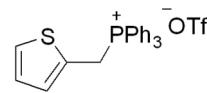
(**1y**, ¹⁹F NMR, 376 MHz, DMSO-*d*₆)



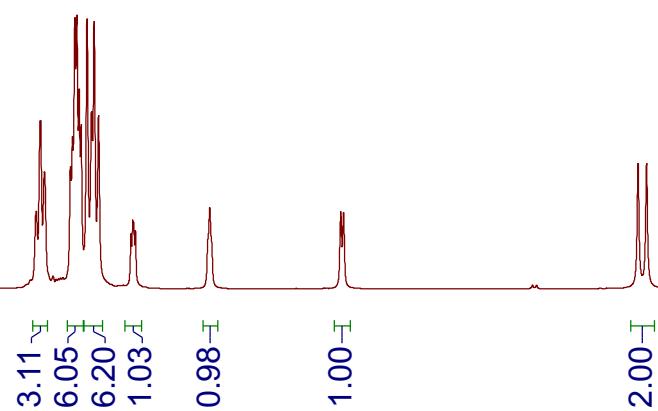
7.857
7.855
7.704
7.695
7.685
7.676
7.650
7.631
7.619
7.608

6.531
6.518

5.219
5.181



(**1a'**, ^1H NMR, 400 MHz, DMSO-d_6)



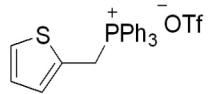
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5

f1 (ppm)

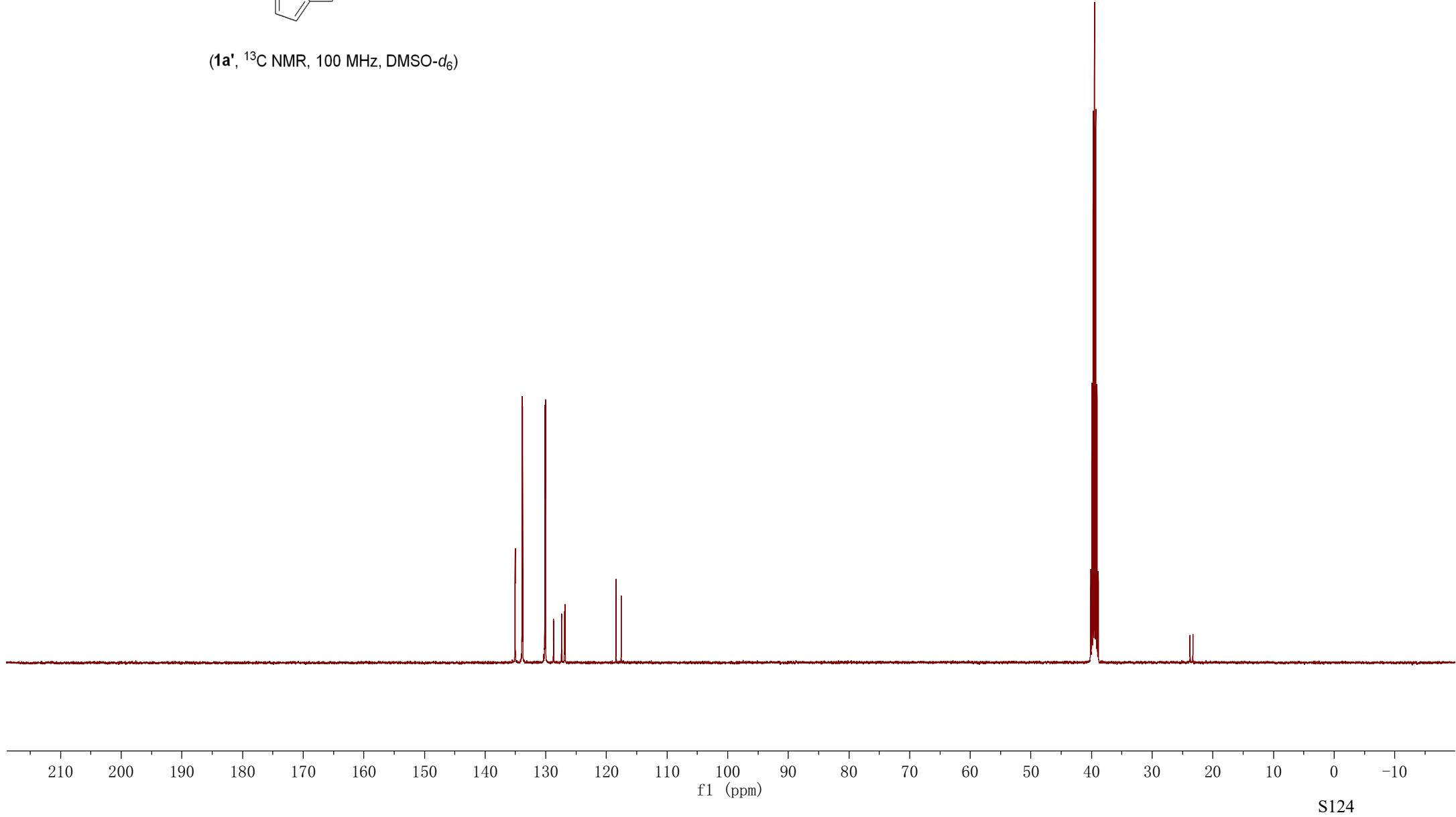
S123

23.772
23.289

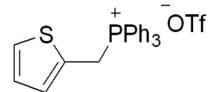
135.065
135.037
133.901
133.804
130.159
130.036
128.740
128.707
127.395
127.383
126.906
126.819
118.408
117.558



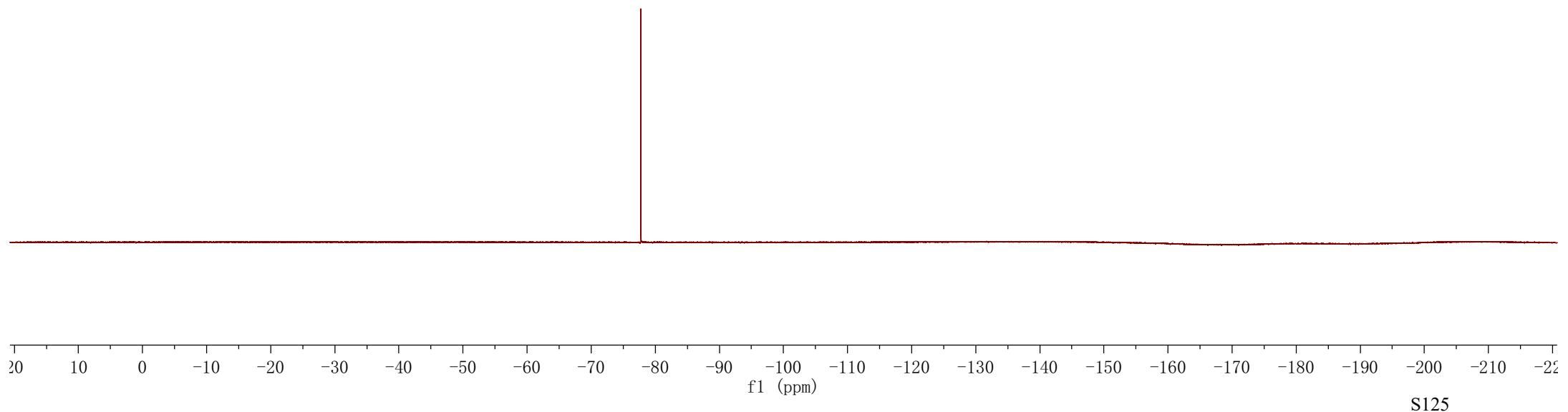
(**1a'**, ^{13}C NMR, 100 MHz, DMSO- d_6)



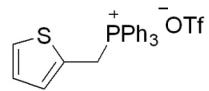
-77.721



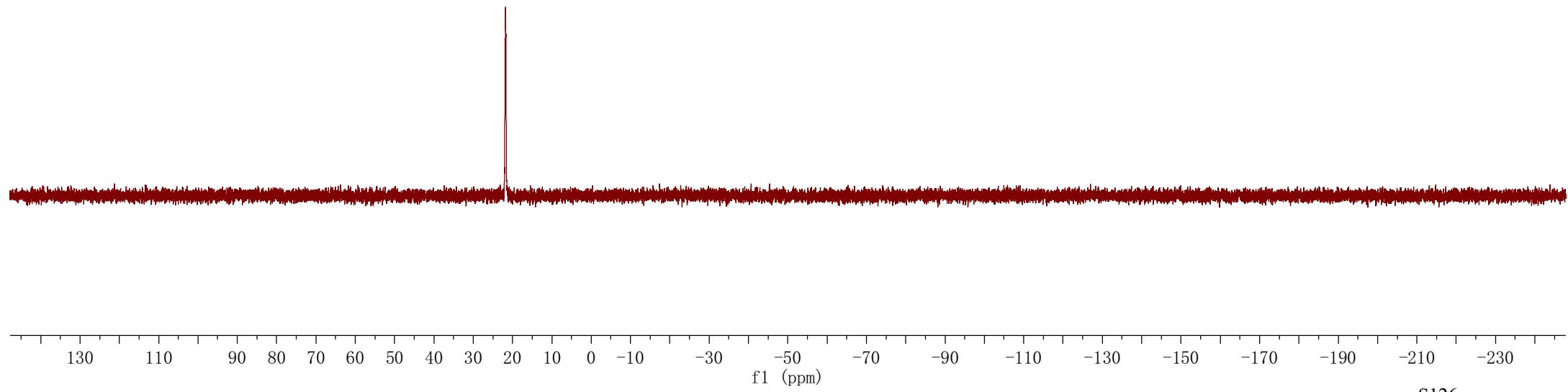
(1a', ¹⁹F NMR, 376 MHz, DMSO-d₆)



-21.792

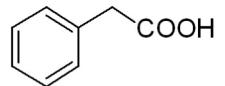


(**1a'**, ^{31}P NMR, 162 MHz, $\text{DMSO}-d_6$)

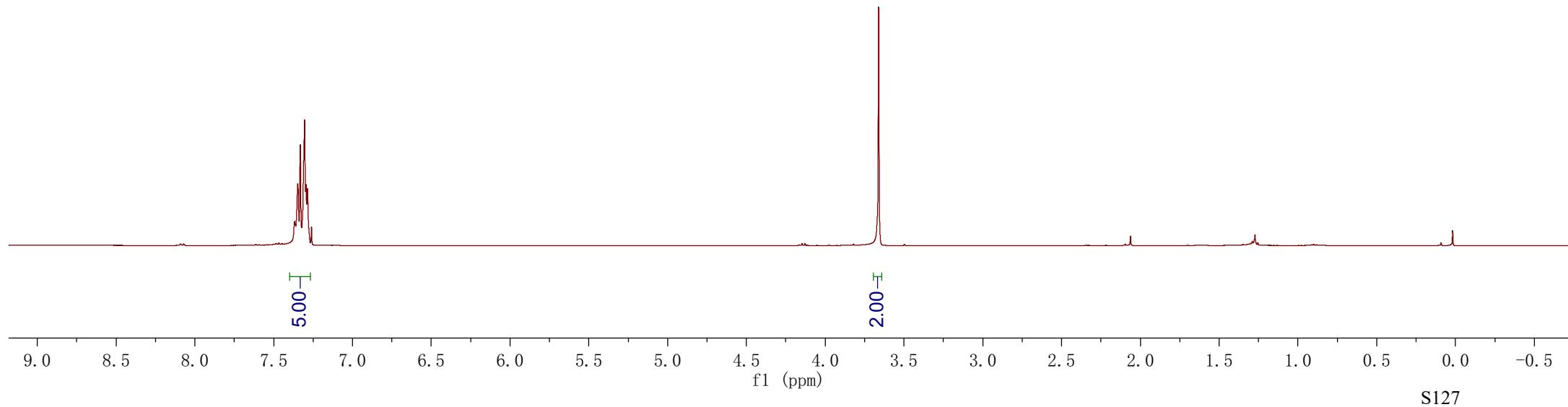


7.367
7.363
7.348
7.331
7.304
7.295
7.286

—3.661



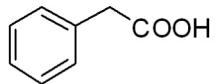
(**2a**, ^1H NMR, 400 MHz, CDCl_3)



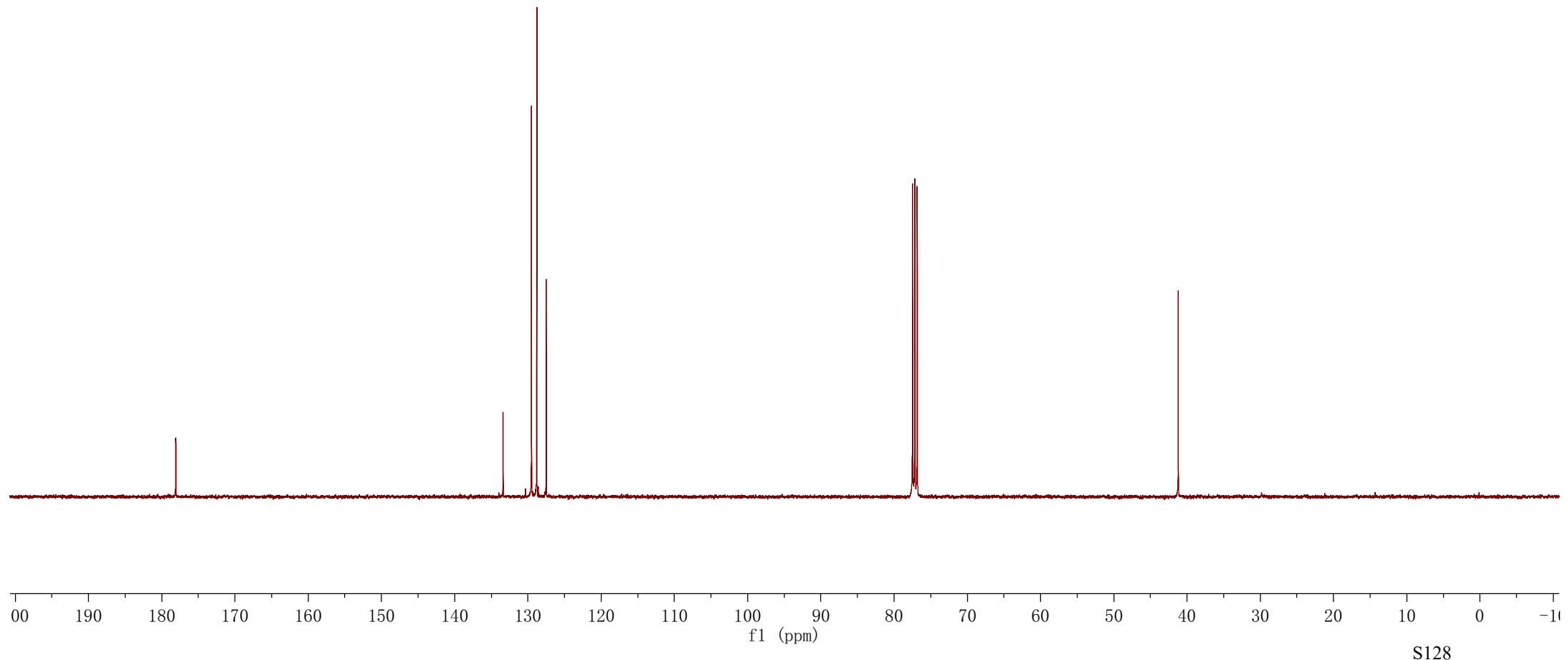
—178.071

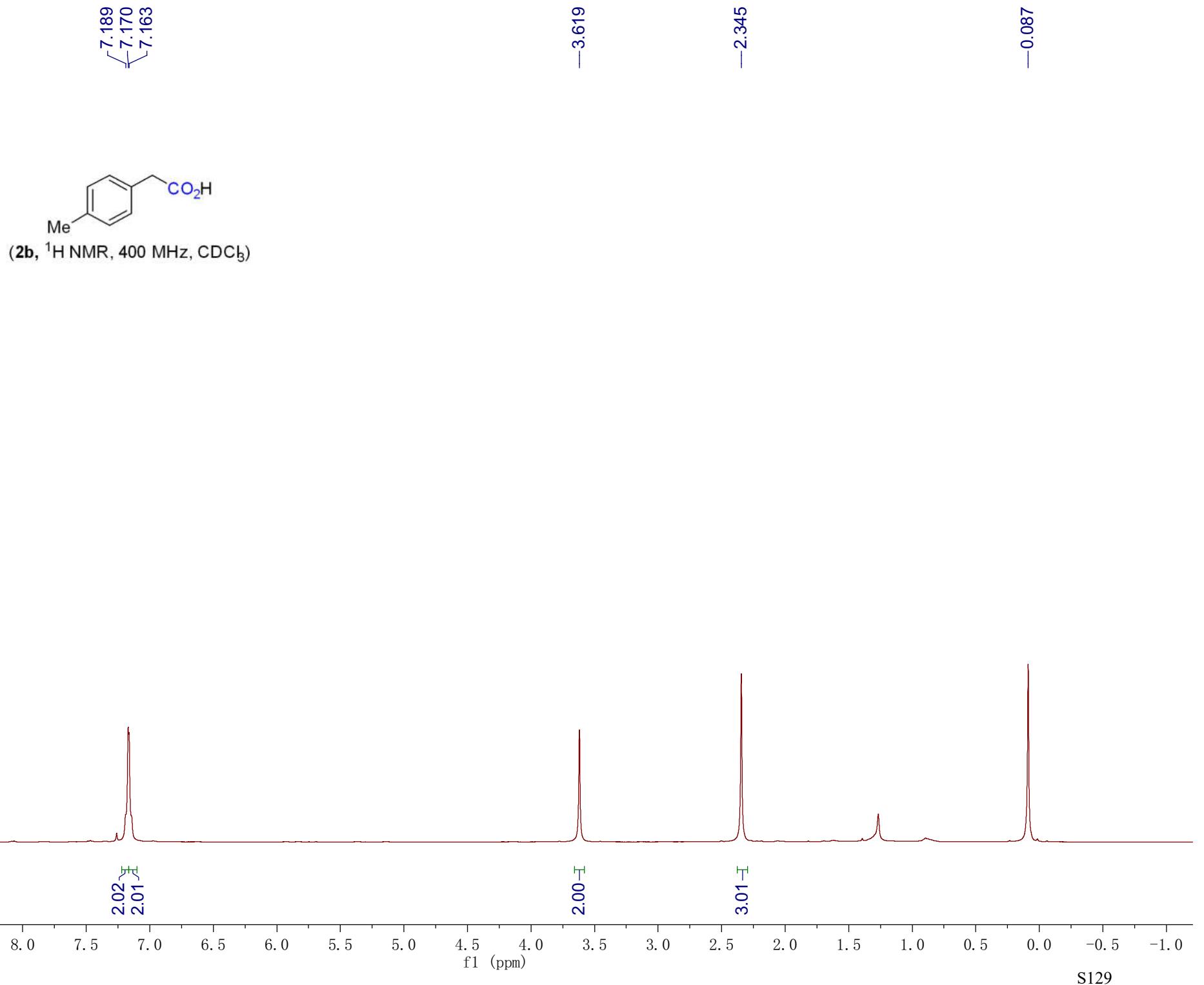
✓133.390
✓129.511
✓128.786
✓127.491

—41.198



(**2a**, ^{13}C NMR, 100 MHz, CDCl_3)



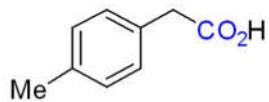


—178.388

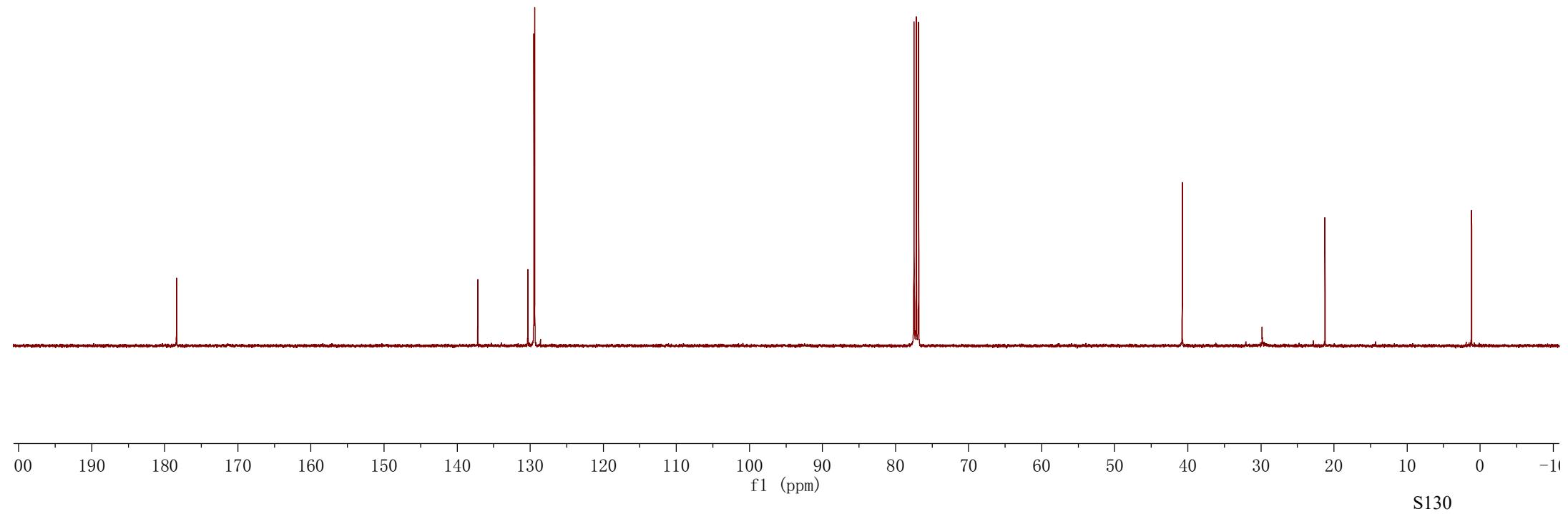
—137.166
—130.316
—129.485
—129.373

—40.759

—21.231



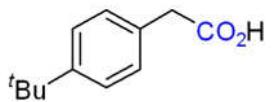
(**2b**, ^{13}C NMR, 100 MHz, CDCl_3)



7.376
7.357
7.239
7.219

—3.627

—1.320



(**2c**, ^1H NMR, 400 MHz, CDCl_3)

2.00 —H
1.97 —H

2.00 —H

9.00 —H

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)

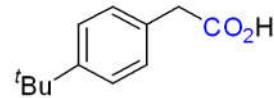
S131

—178.113

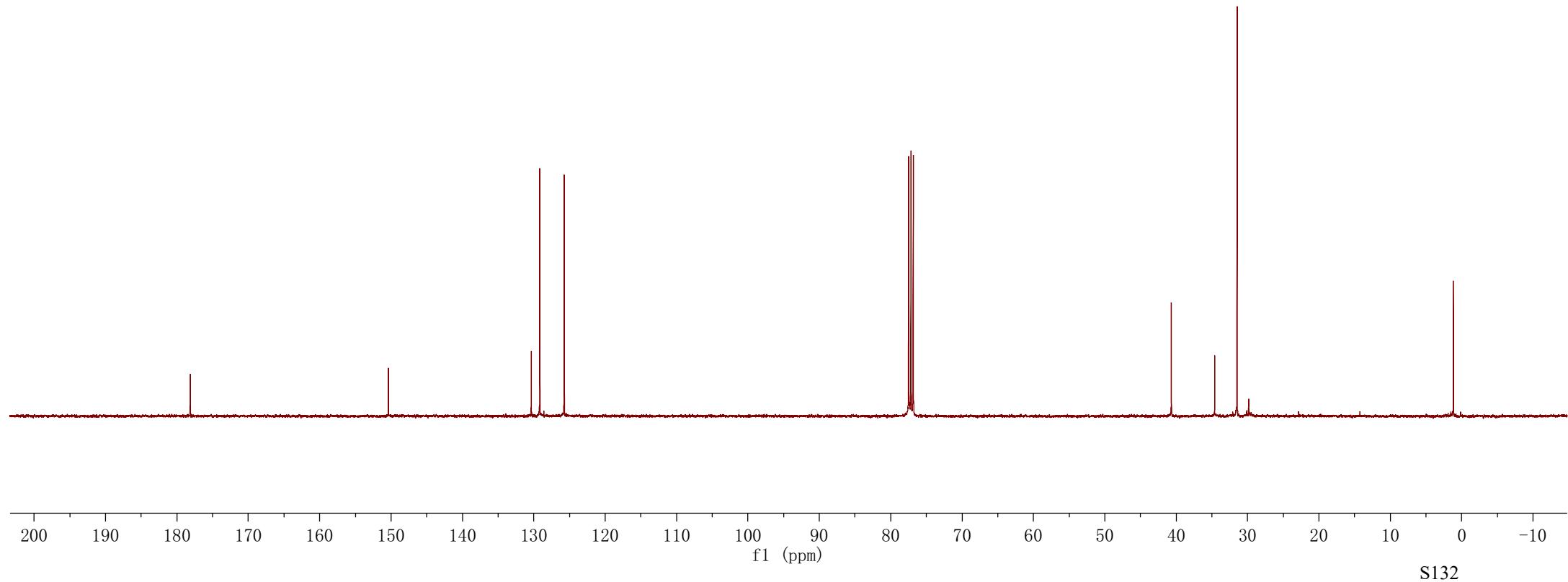
—150.373

~130.353
~129.163
~125.745

~40.680
~34.619
~31.463



(**2c**, ^{13}C NMR, 100 MHz, CDCl_3)

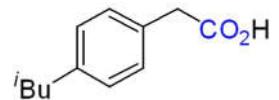


7.208
7.188
7.127
7.107

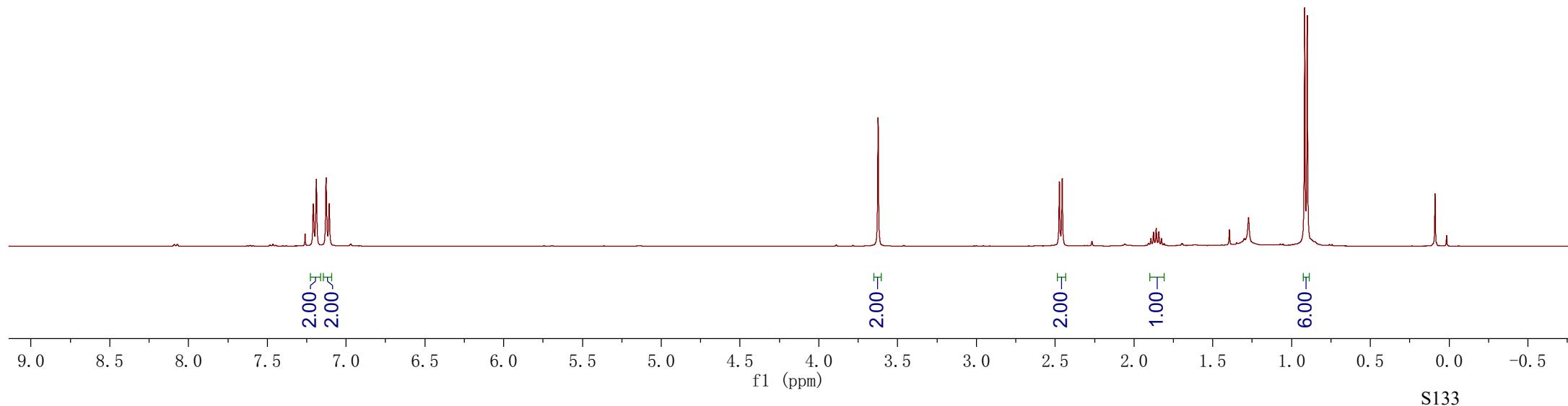
-3.624

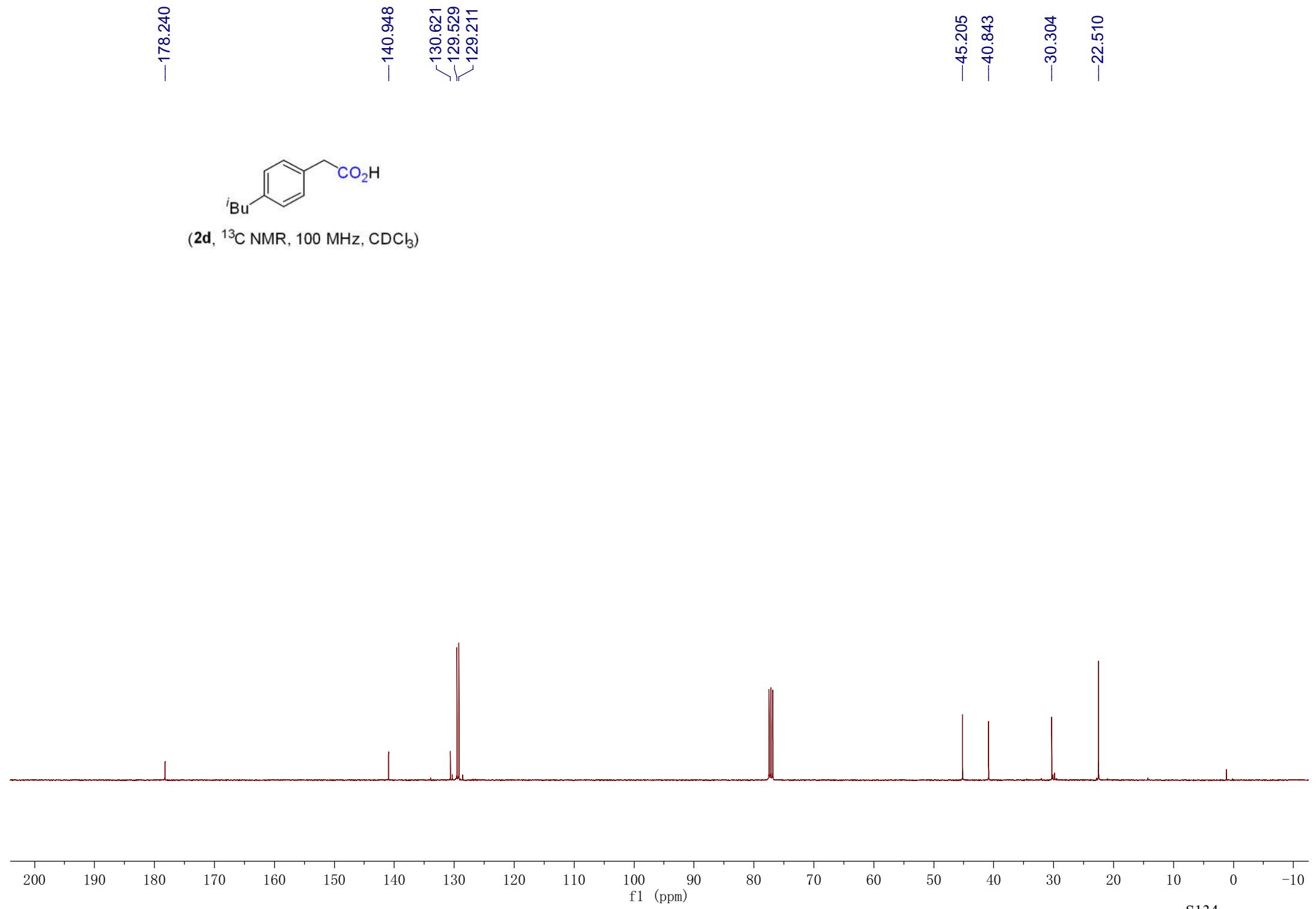
2.473
2.455
1.926
1.909
1.893
1.876
1.859
1.842
1.825
1.808

0.917
0.900



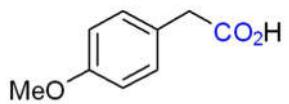
(2d, ^1H NMR, 400 MHz, CDCl_3)



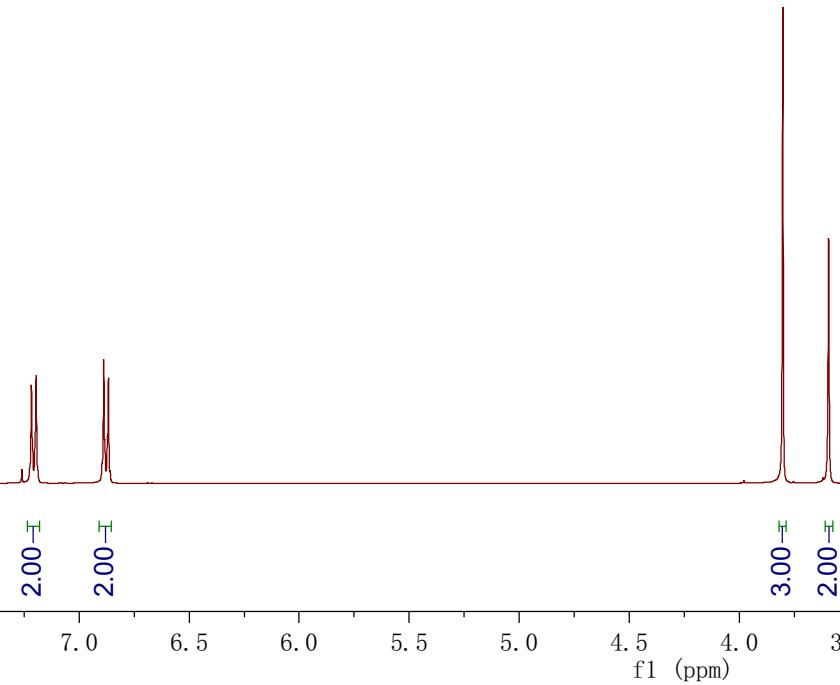


7.217
7.195
6.888
6.867

—3.802
—3.593



(**2e**, ^1H NMR, 400 MHz, CDCl_3)



—178.378

—159.004

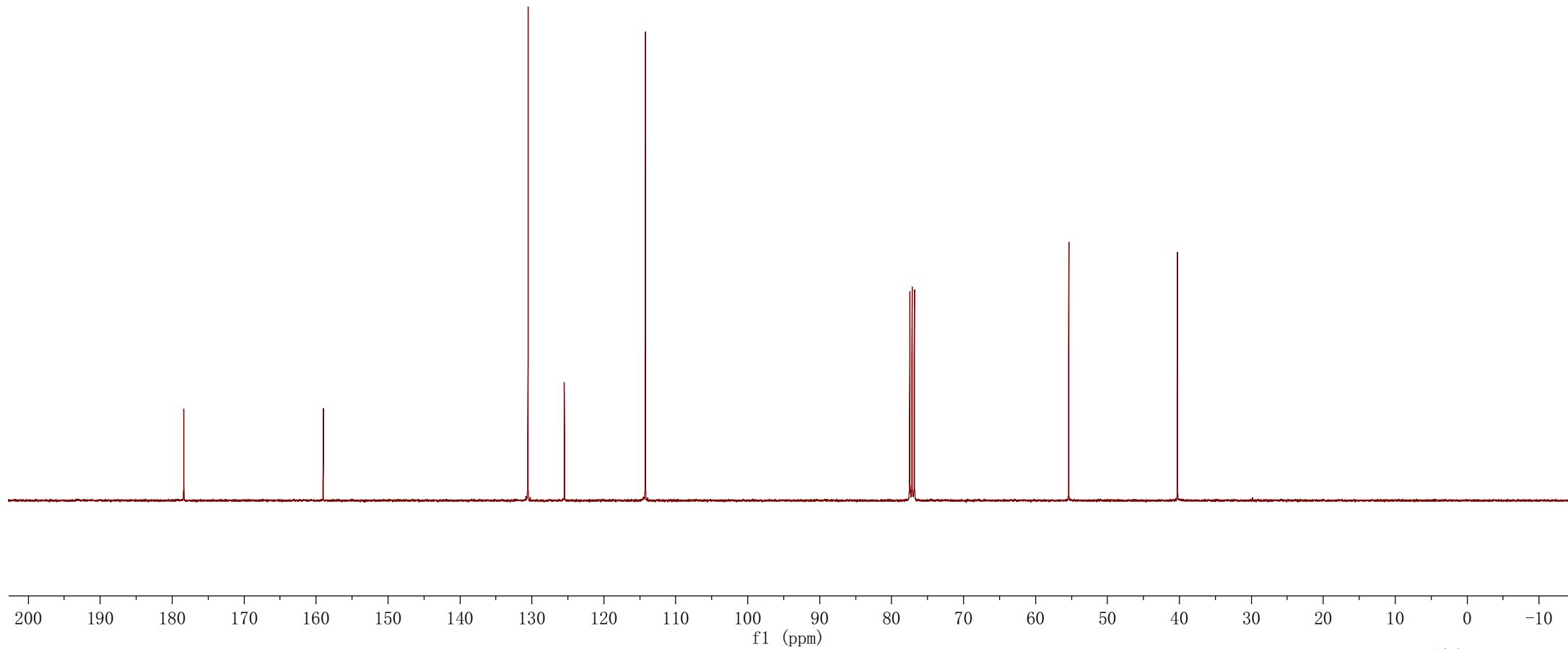
—130.538

—125.482

—114.225

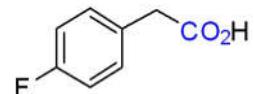
—55.384

—40.282

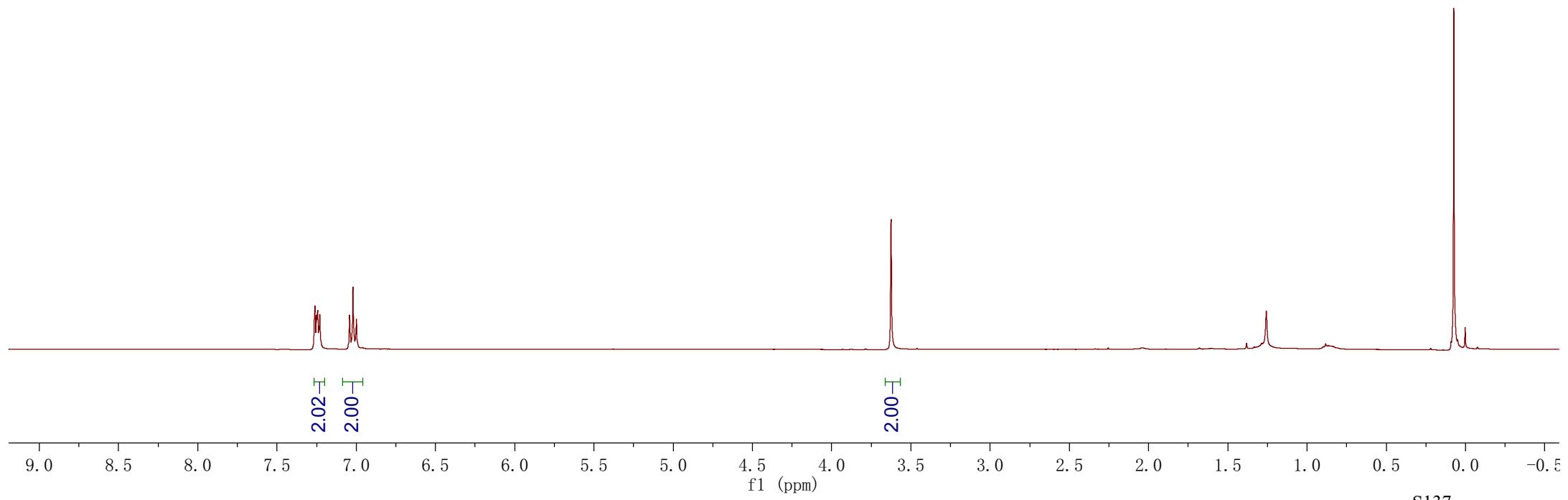


7.260
7.251
7.243
7.230
7.042
7.021
6.999

—3.625



(2f, ^1H NMR, 400 MHz, CDCl_3)



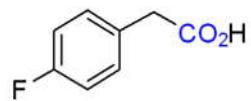
—177.419

—163.537
—161.092

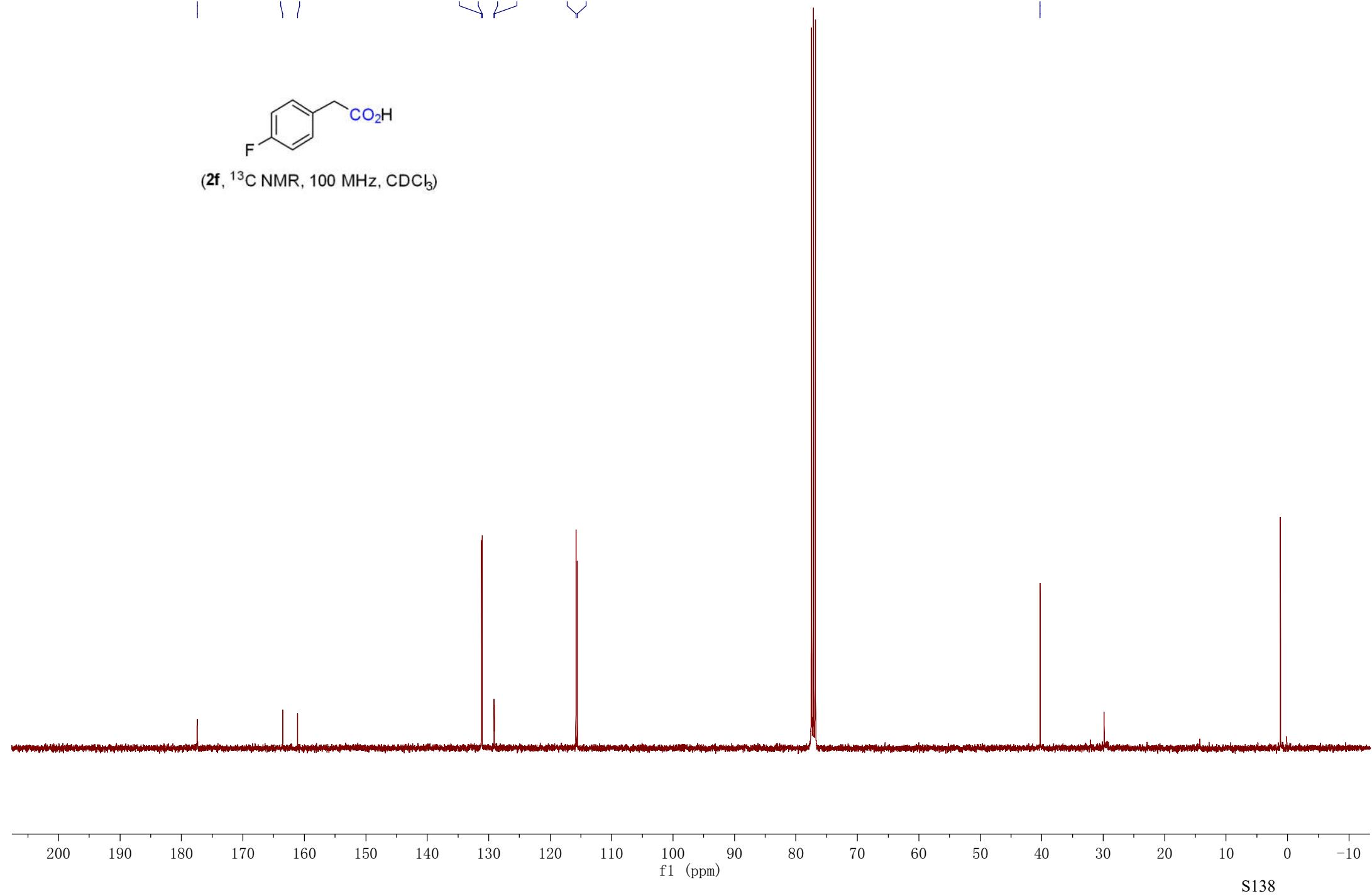
131.144
131.064
129.113
129.079

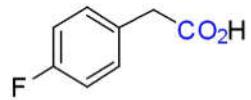
115.790
115.577

—40.253



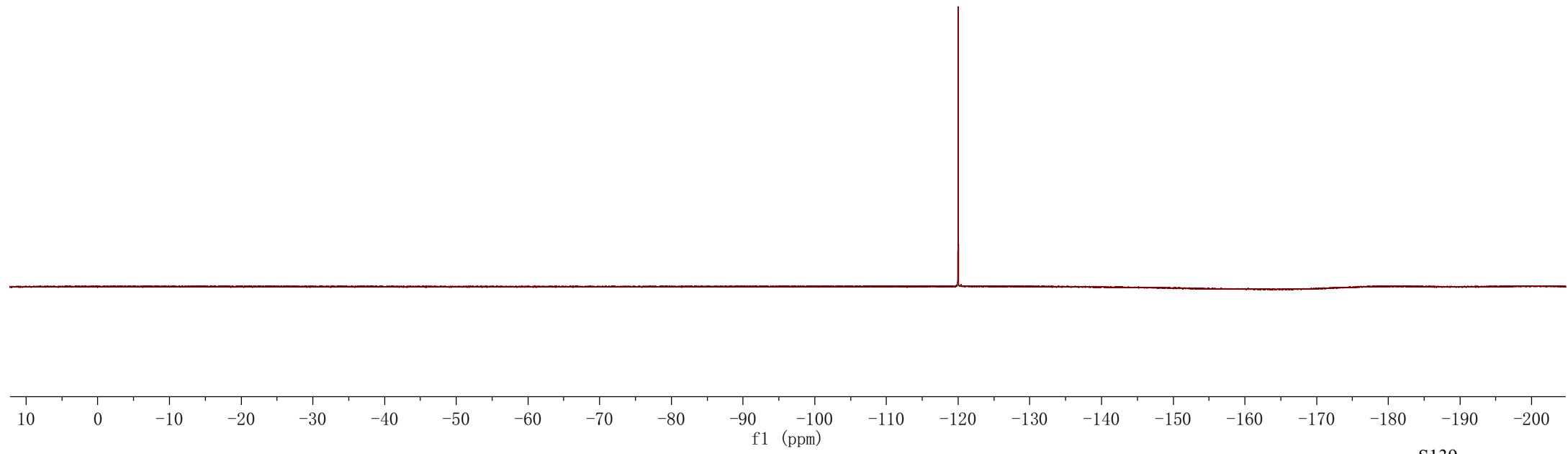
(2f, ¹³C NMR, 100 MHz, CDCl₃)





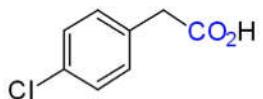
(2f, ¹⁹F NMR, 376 MHz, CDCl₃)

—120.005

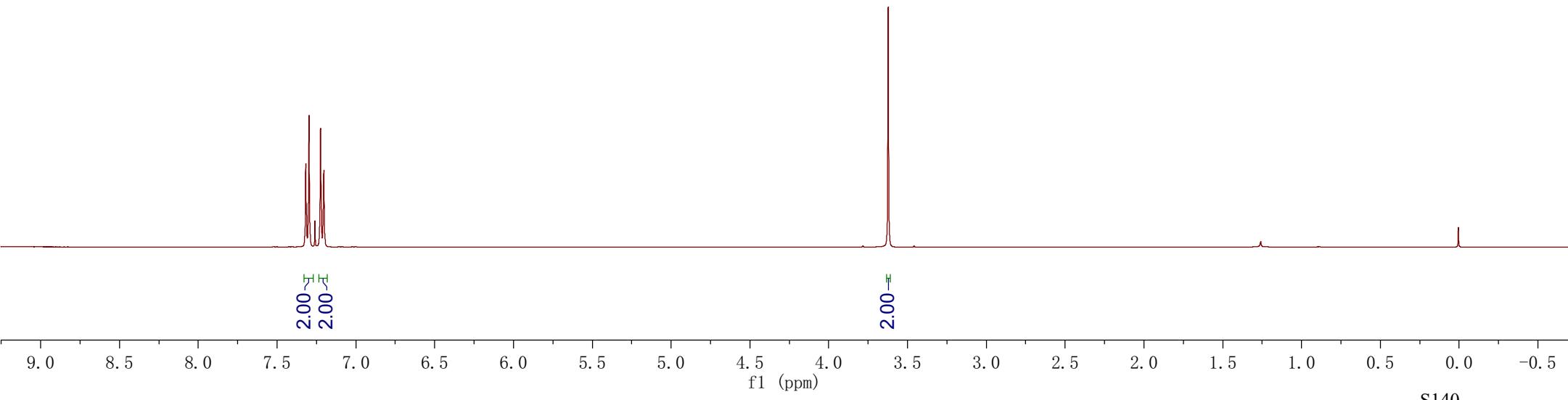


7.318
7.297
7.224
7.203

— 3.622



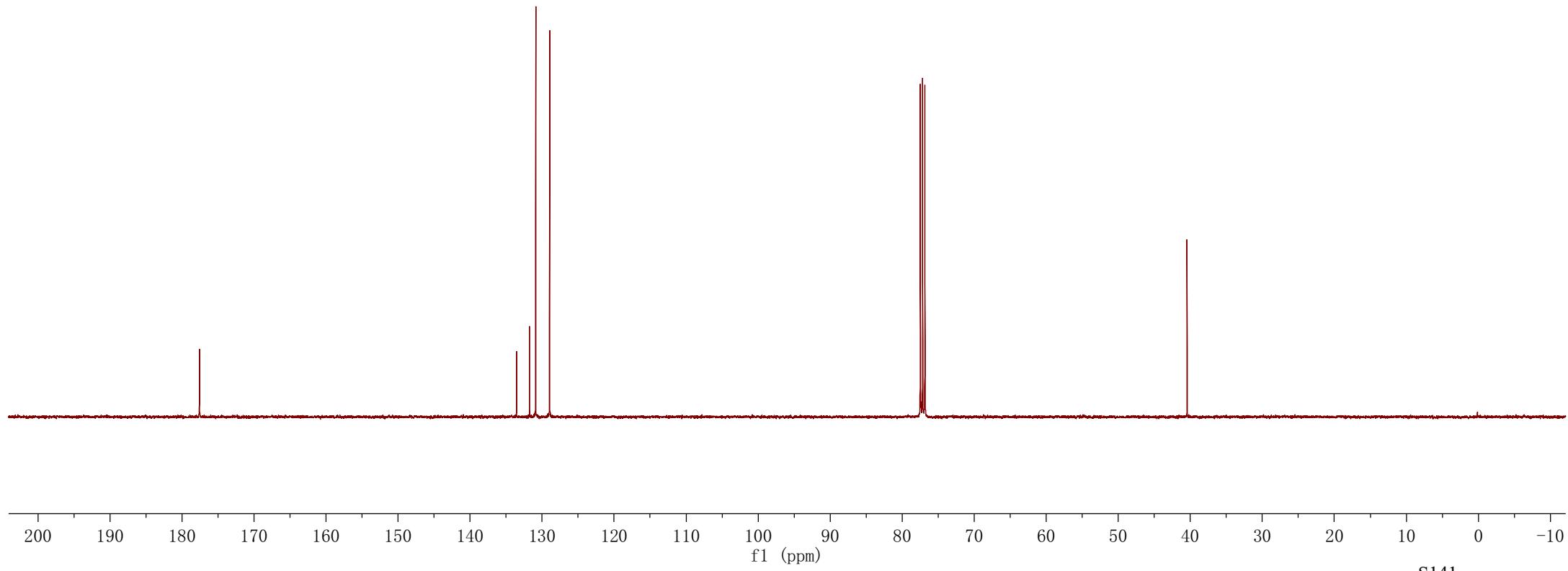
(**2g**, ^1H NMR, 400 MHz, CDCl_3)



—177.558

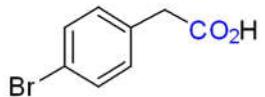
133.551
131.745
130.887
128.952

—40.442

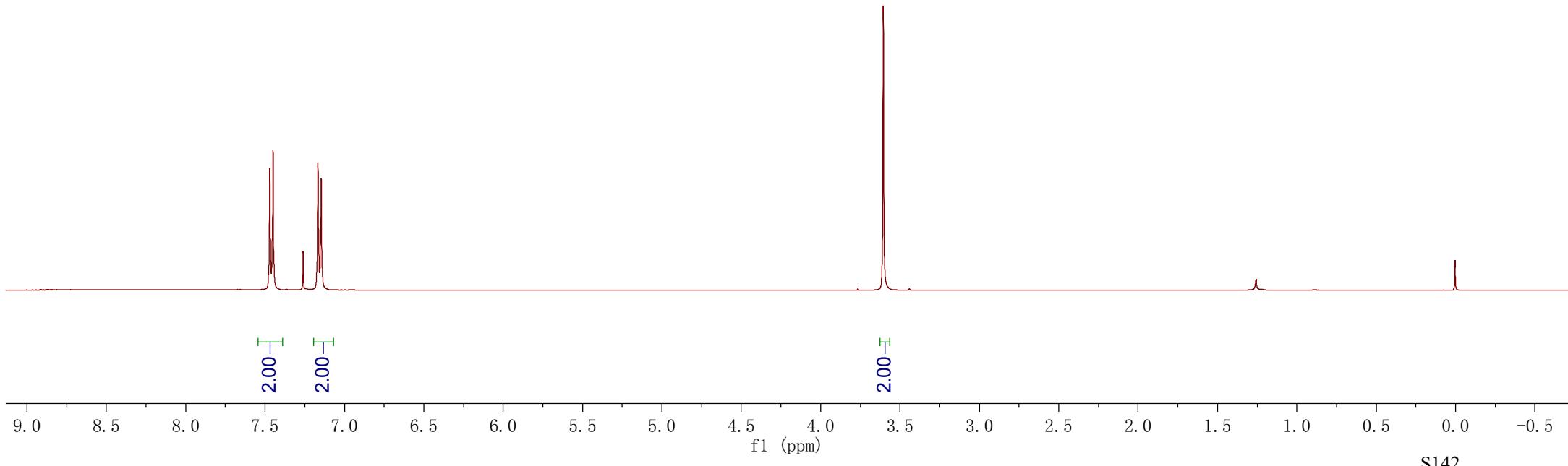


7.471
7.450
7.167
7.146

-3.605



(**2h**, ^1H NMR, 400 MHz, CDCl_3)

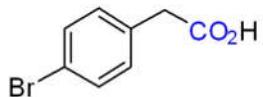


—177.307

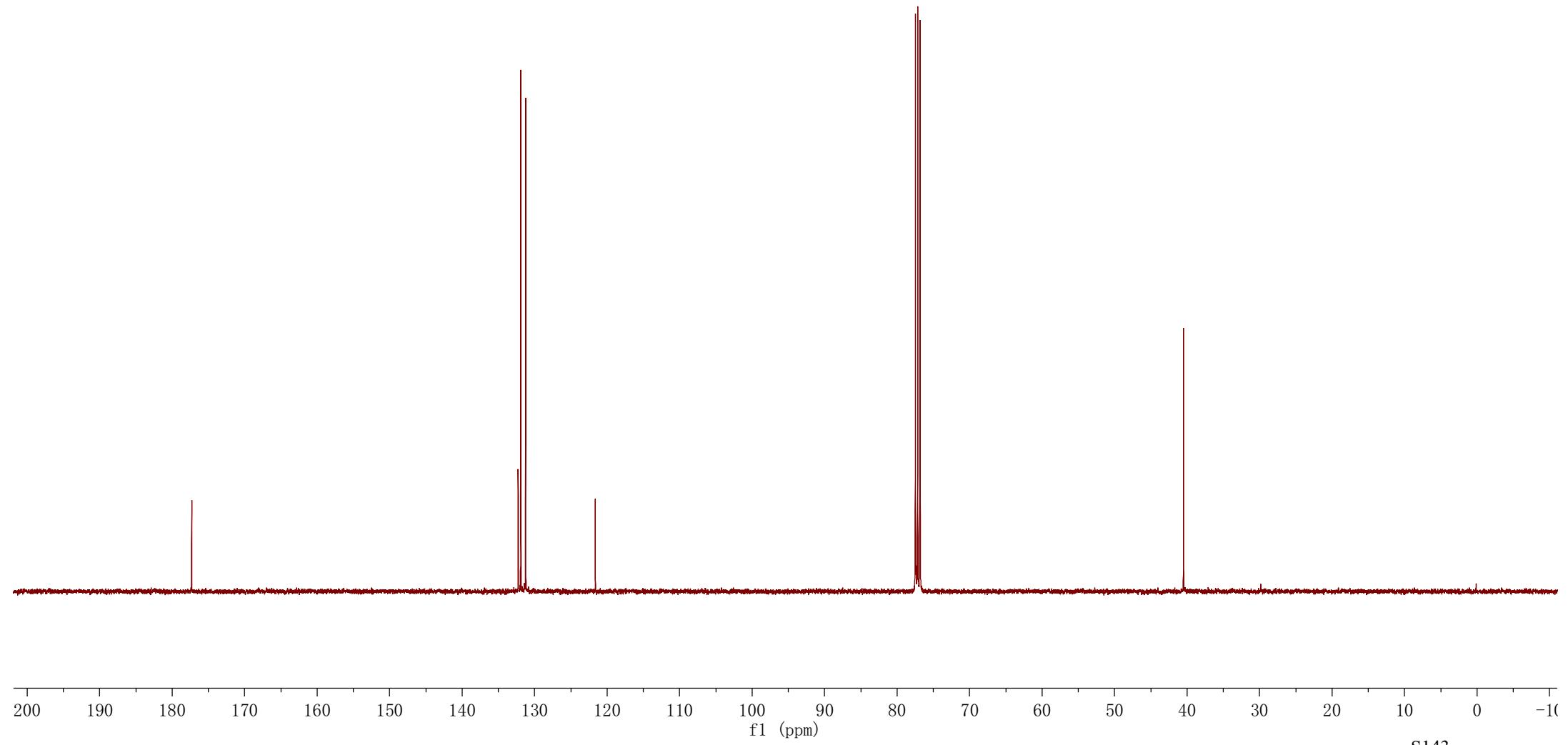
132.270
131.913
131.244

—121.630

—40.501

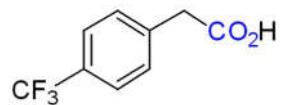


(**2h**, ^{13}C NMR, 100 MHz, CDCl_3)

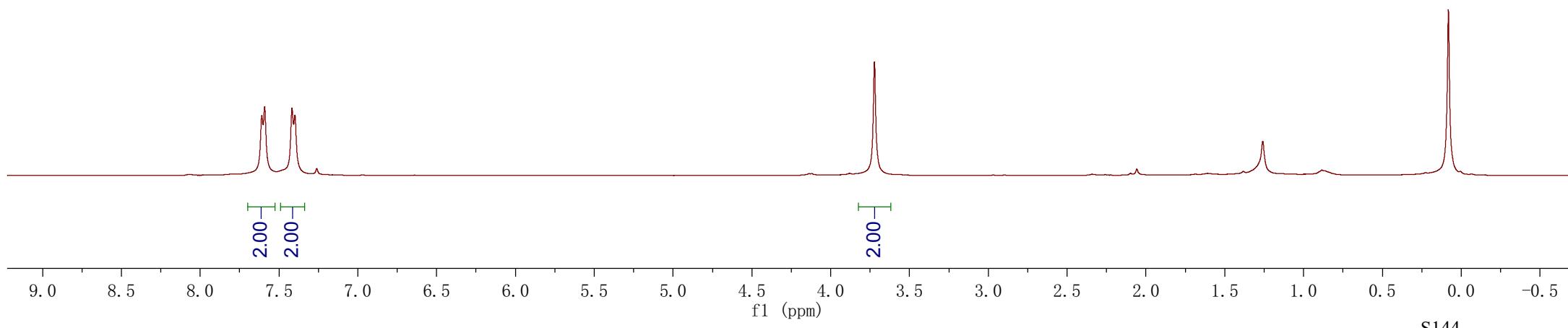


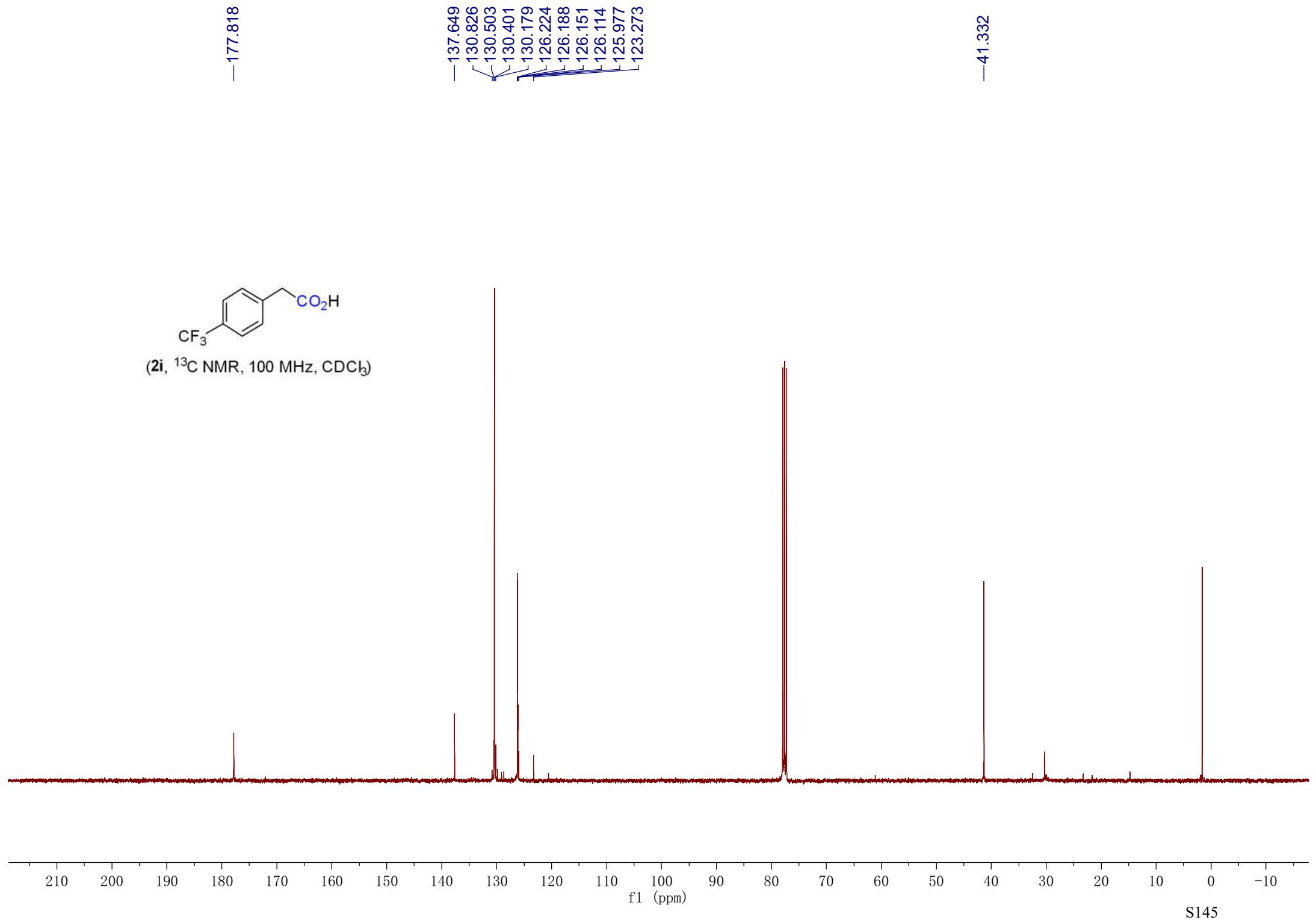
7.609
7.591
7.417
7.399

-3.722



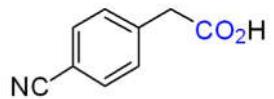
(**2i**, ^1H NMR, 400 MHz, CDCl_3)



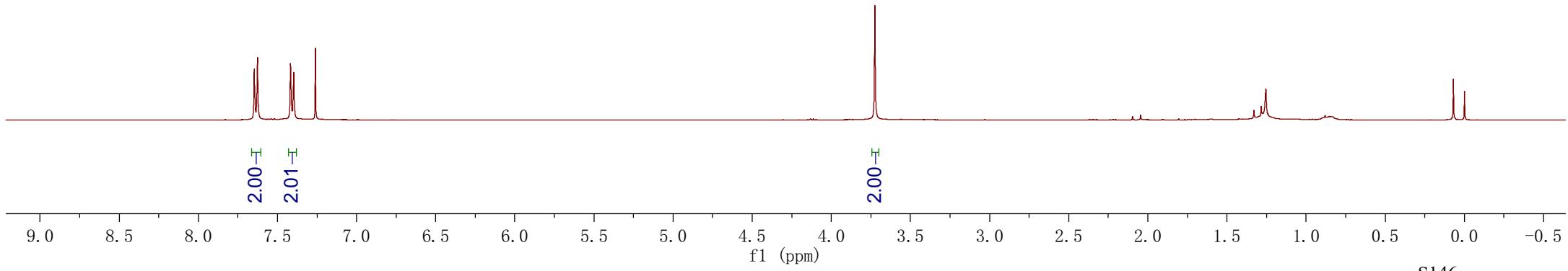


7.647
7.626
7.417
7.397

-3.725



(2j, ¹H NMR, 400 MHz, CDCl₃)



—174.973

—138.643

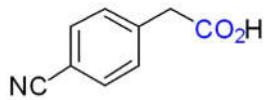
—132.561

~130.417

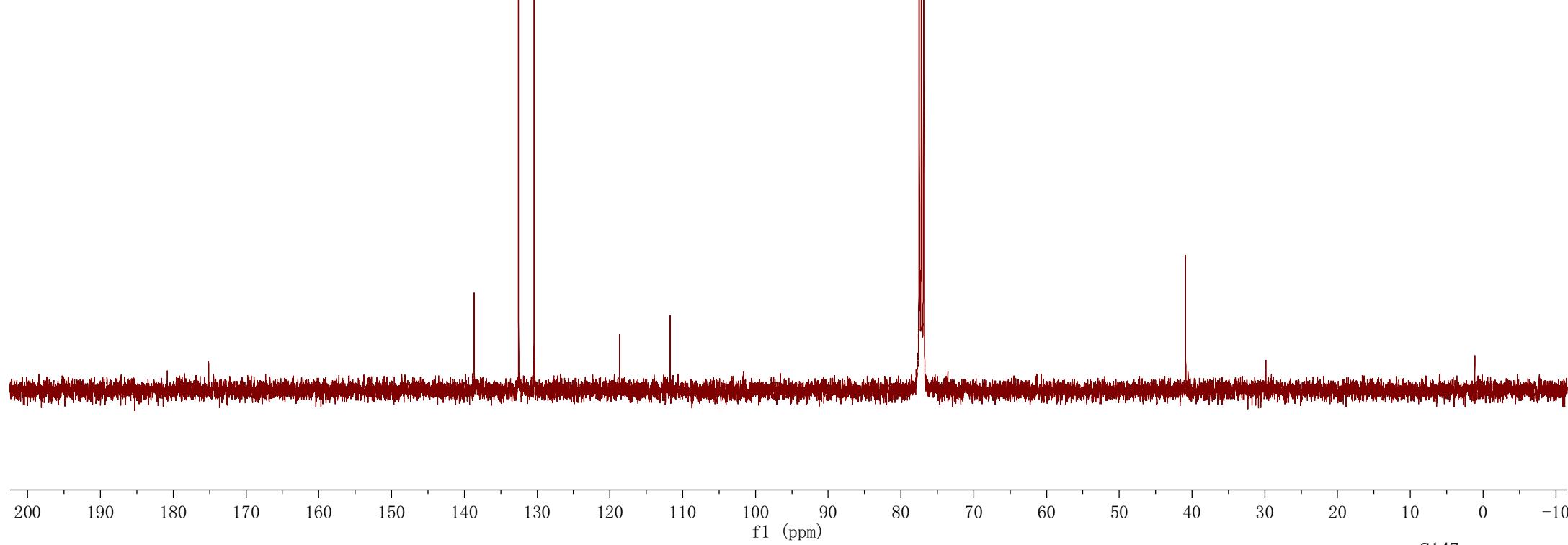
—118.670

—111.700

—40.885



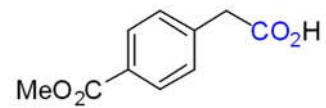
(**2j**, ^{13}C NMR, 100 MHz, CDCl_3)



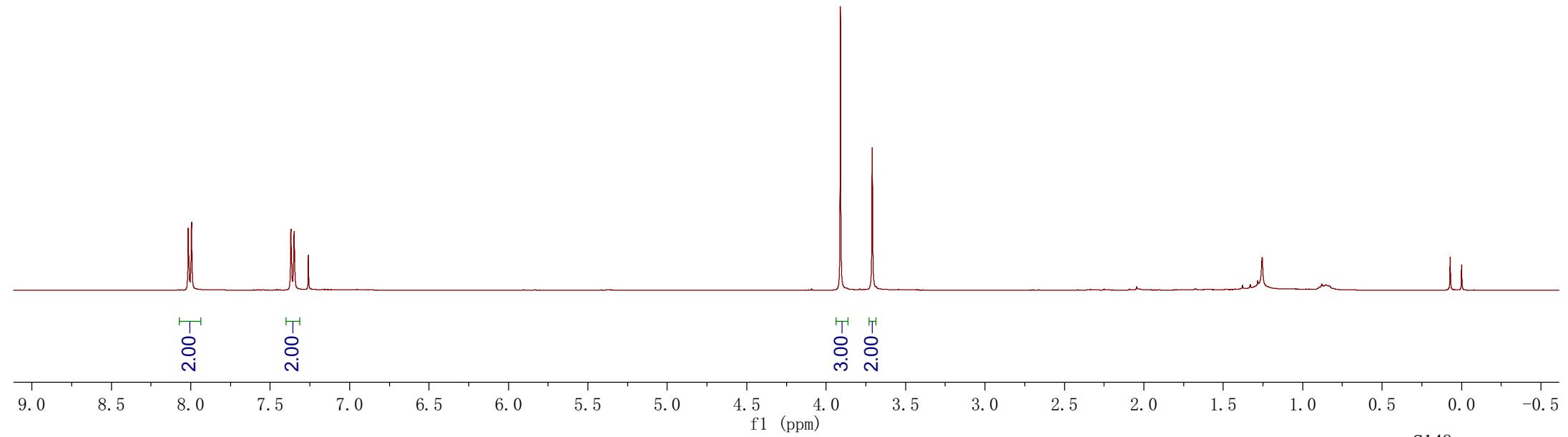
<8.016
<7.995

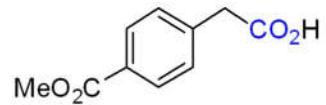
<7.369
<7.349

-3.910
-3.710



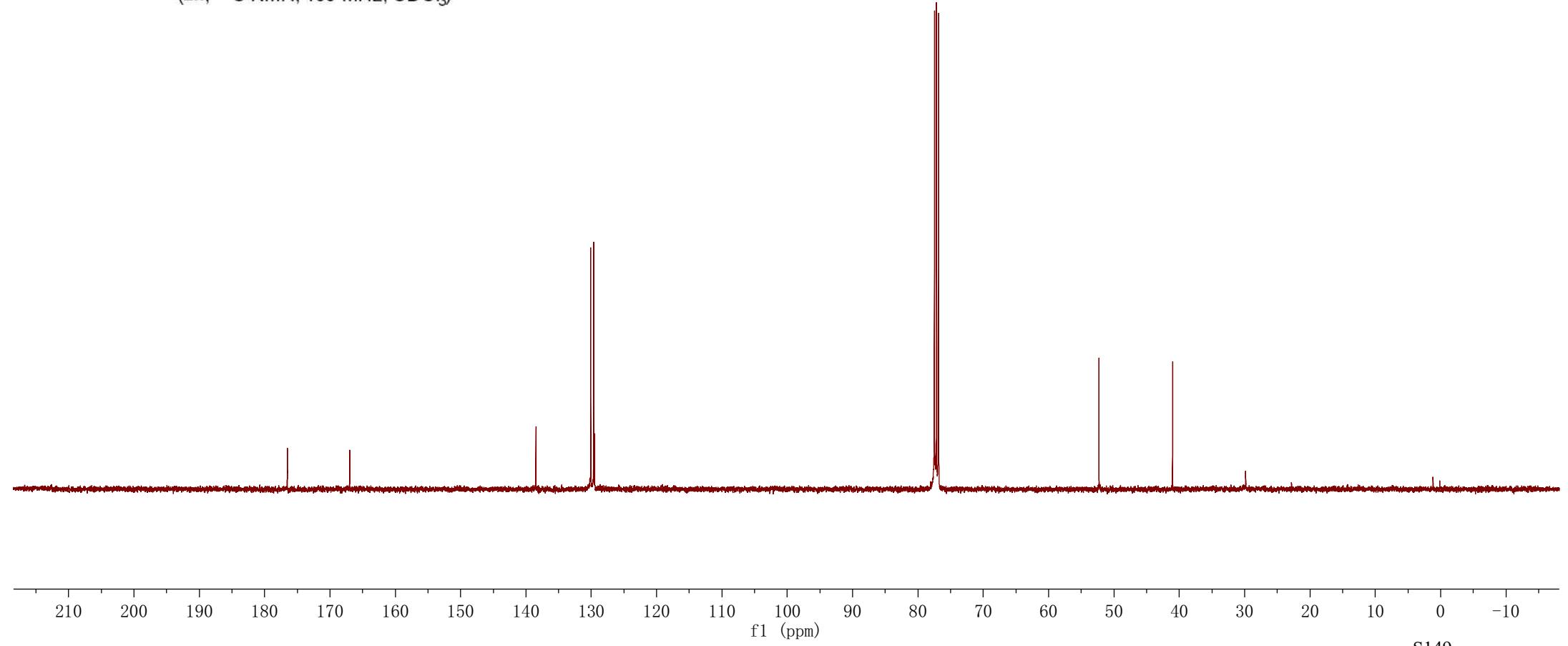
(**2k**, ^1H NMR, 400 MHz, CDCl_3)



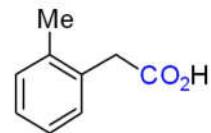


(**2k**, ^{13}C NMR, 100 MHz, CDCl_3)

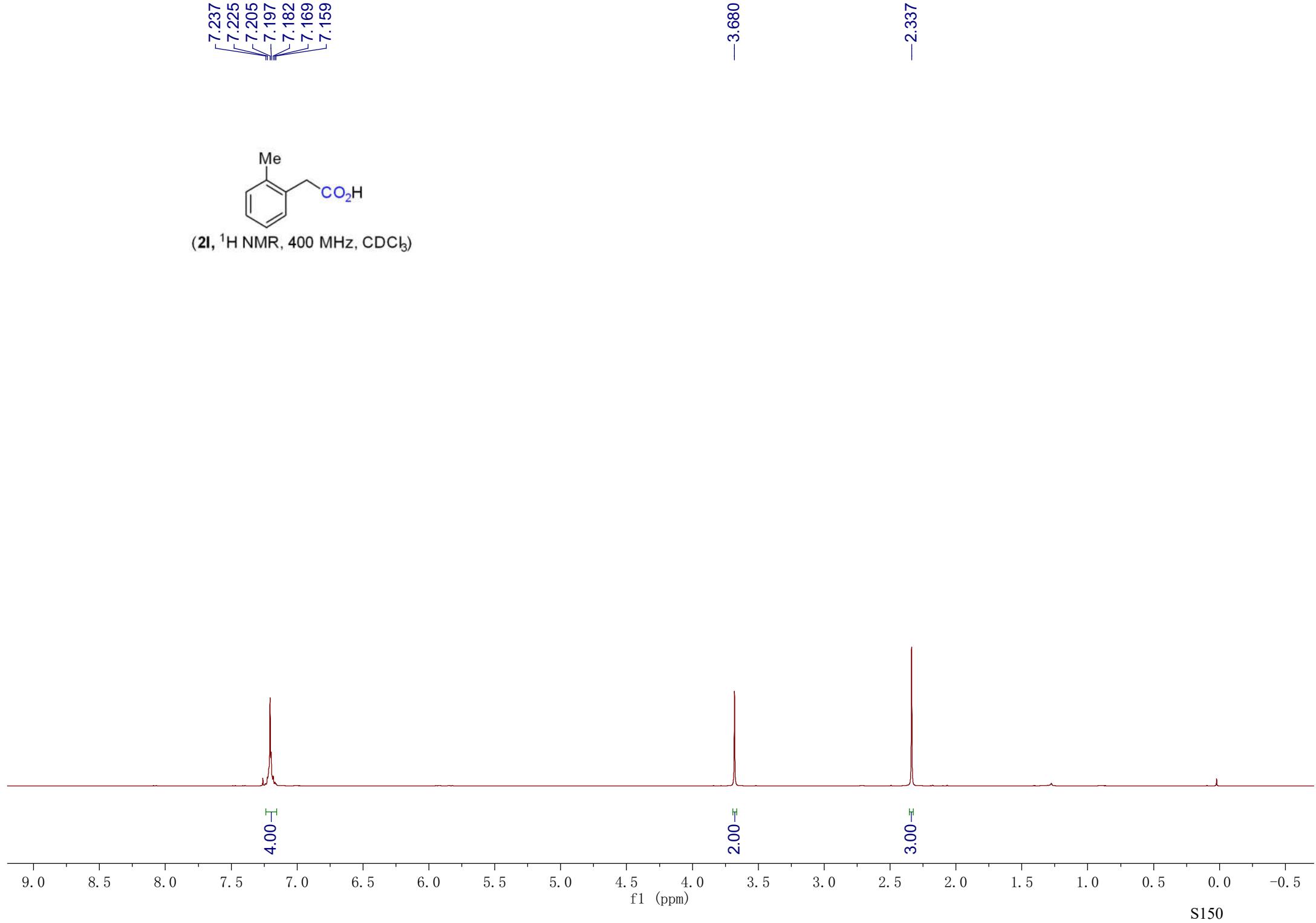
—176.502
—166.973
—138.477
—130.083
—129.613
—129.473
—52.283
—41.024



7.237
7.225
7.205
7.197
7.182
7.169
7.159



(**2l**, ^1H NMR, 400 MHz, CDCl_3)

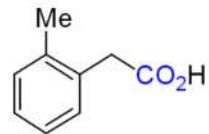


—178.046

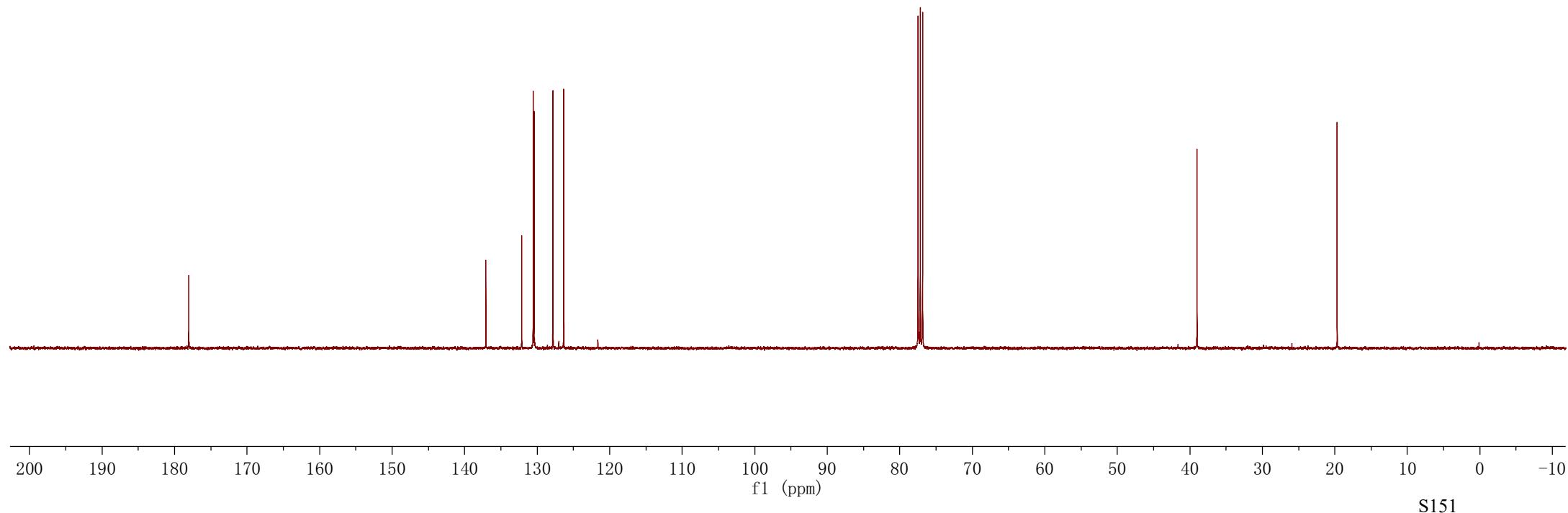
✓137.077
✓132.139
✓130.558
✓130.429
~127.828
~126.341

—39.010

—19.686



(**2l**, ^{13}C NMR, 100 MHz, CDCl_3)

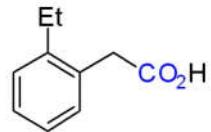


7.264
7.260
7.245
7.241
7.225
7.220
7.213
7.206
7.198
7.181
7.176
7.164
7.161
7.146
7.141

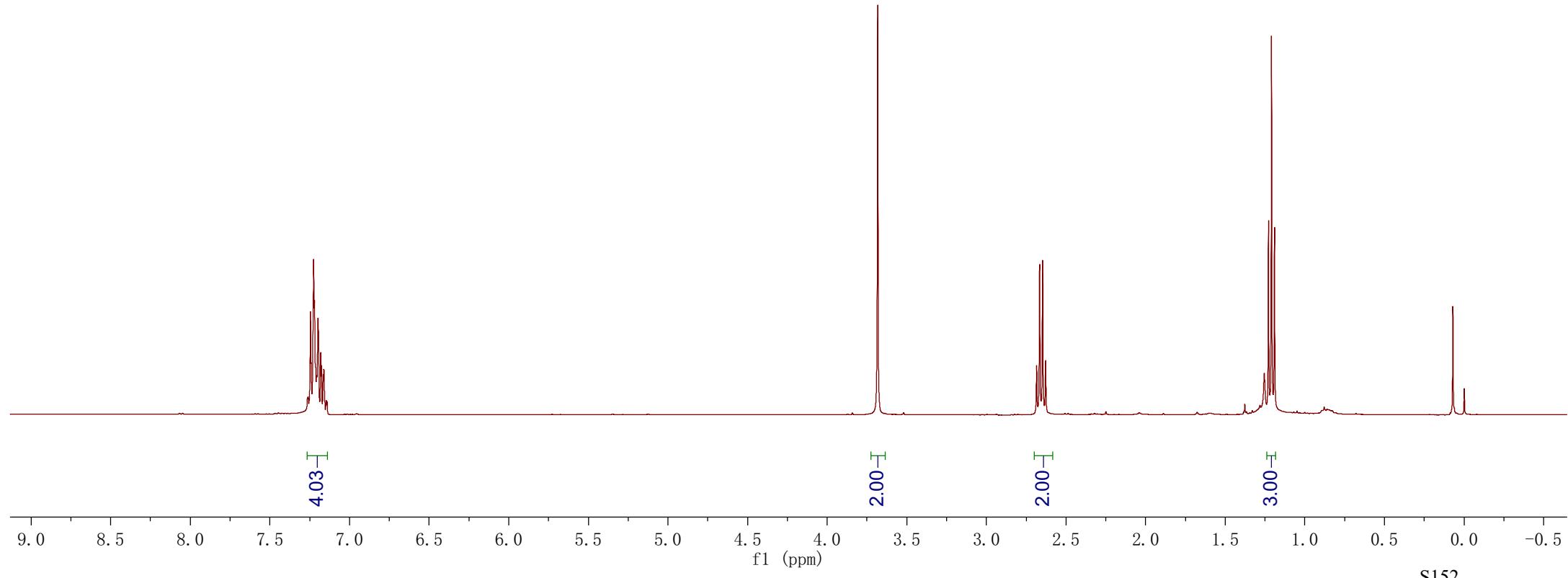
-3.683

2.685
2.666
2.647
2.628

1.228
1.209
1.190



(**2m**, ^1H NMR, 400 MHz, CDCl_3)



—177.995

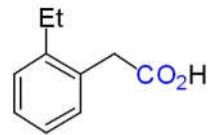
—142.807

131.427
130.711
128.753
128.006
126.250

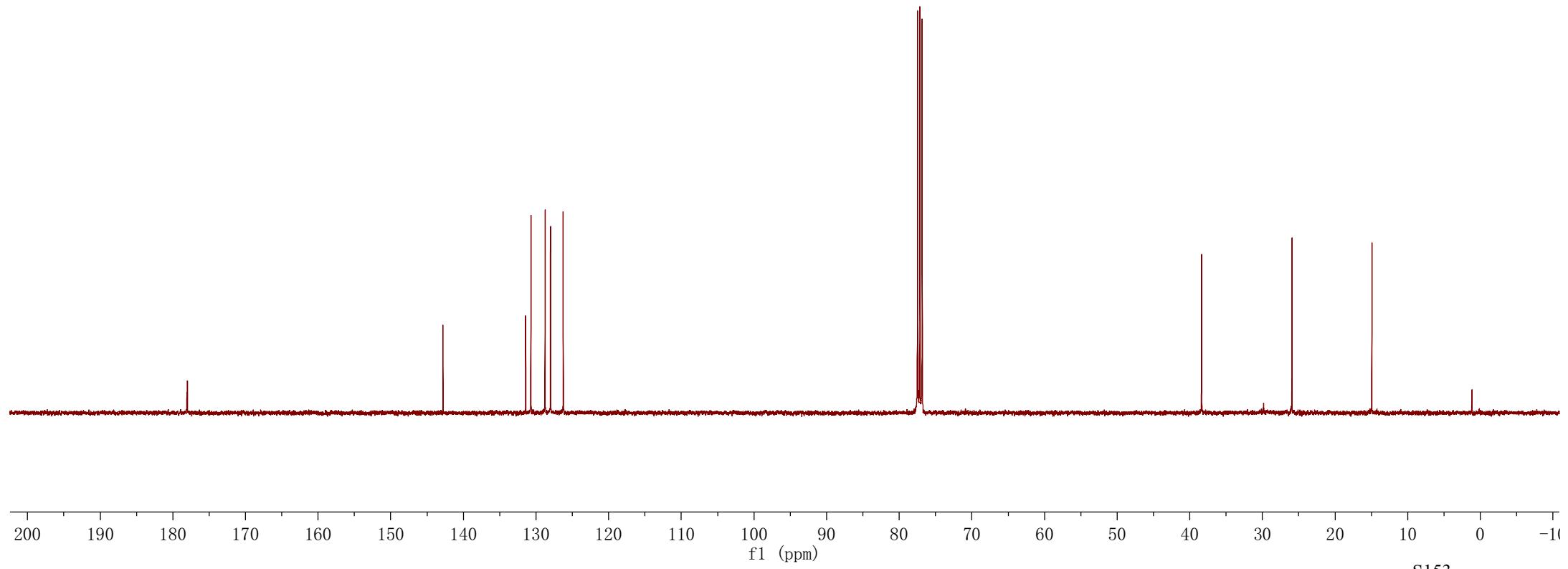
—38.366

—25.926

—14.933

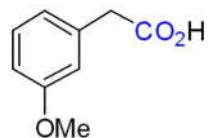


(**2m**, ^{13}C NMR, 100 MHz, CDCl_3)

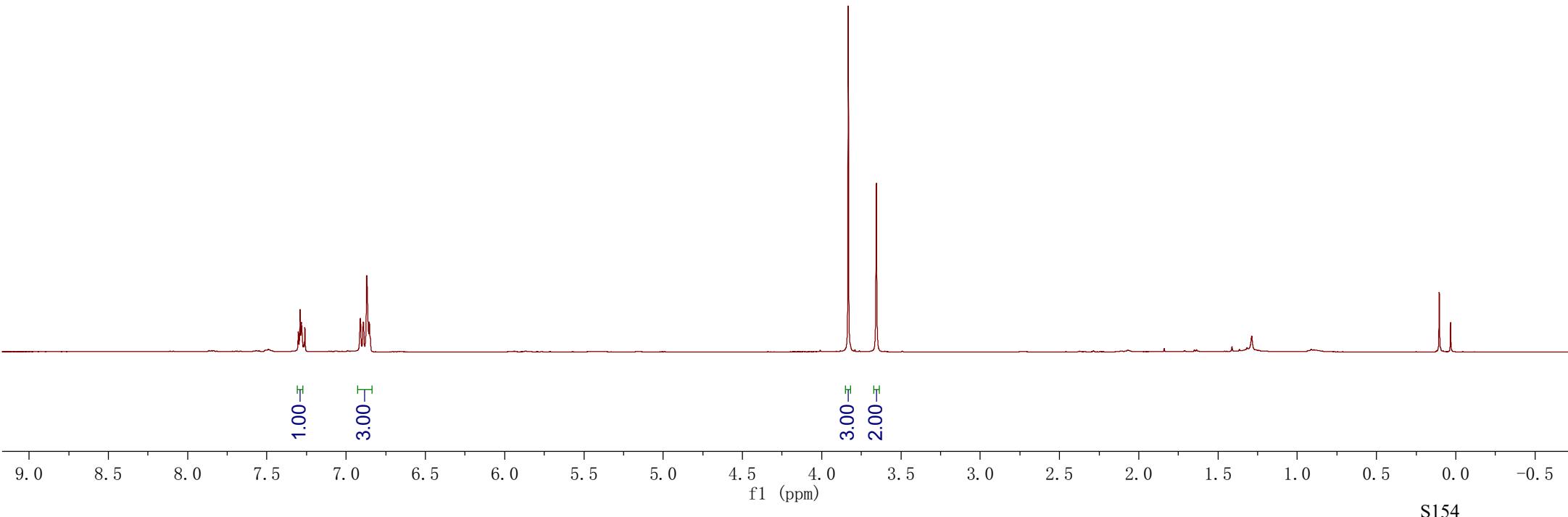


7.302
7.290
7.283
7.279
6.911
6.891
6.870
6.854

— 3.832
— 3.655



(**2n**, ^1H NMR, 400 MHz, CDCl_3)



—177.392

—159.887

—134.820

—129.782

—121.855

—115.195

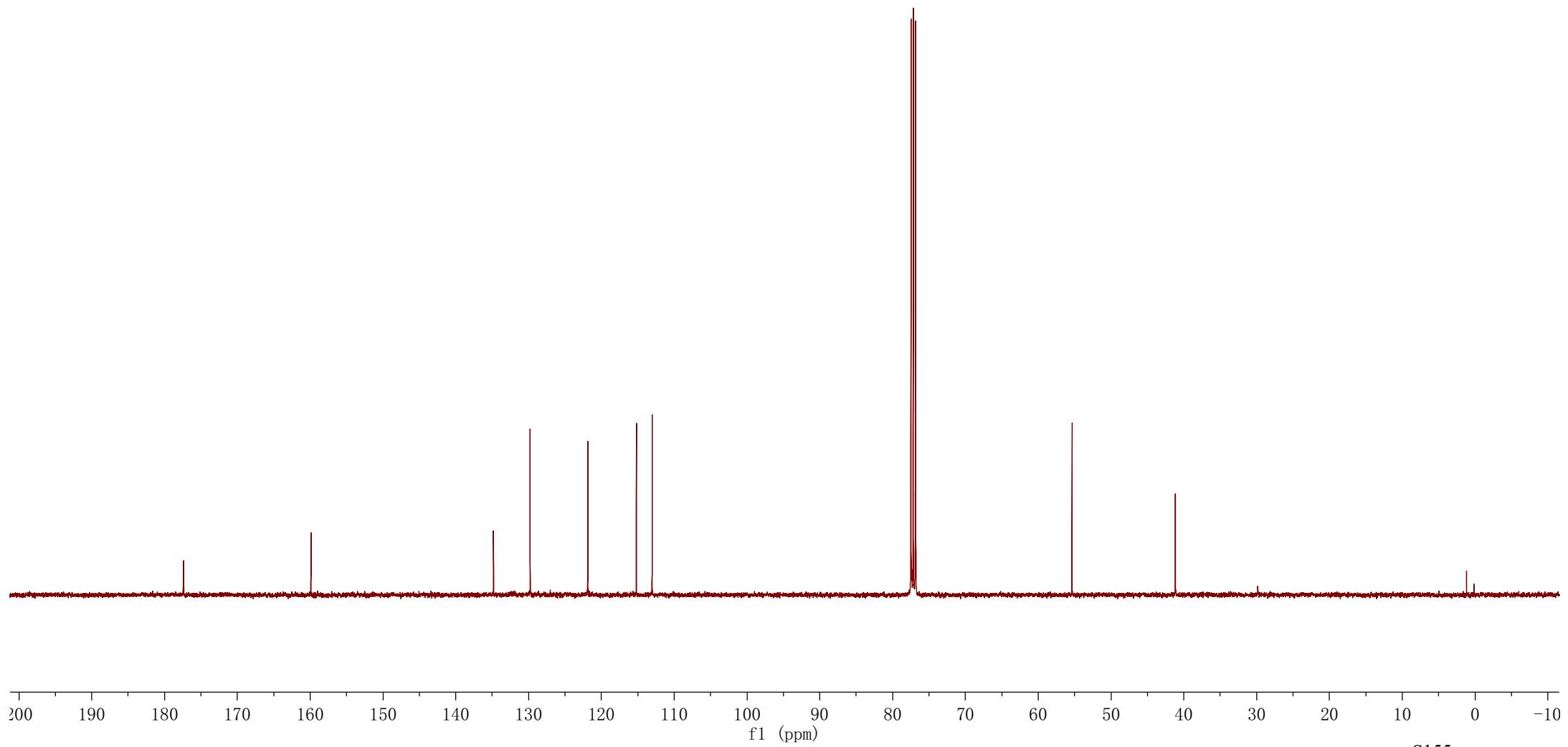
—113.028

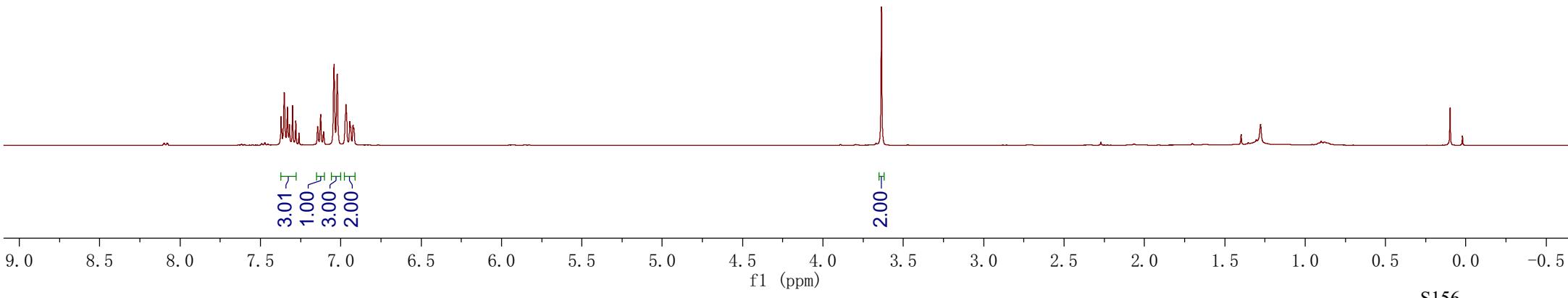
—55.357

—41.178



(**2n**, ¹³C NMR, 100 MHz, CDCl₃)



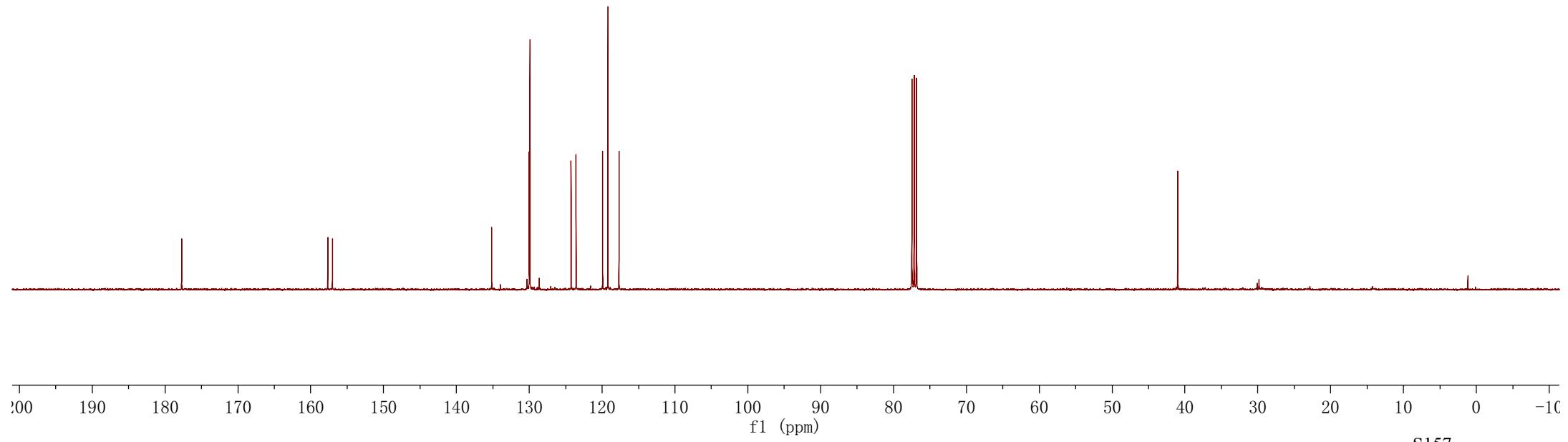
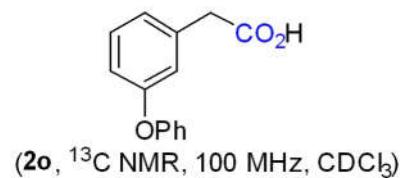


—177.712

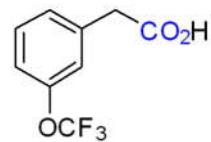
157.655
157.023

135.168
130.007
129.915
124.257
123.572
119.940
119.218
117.694

—40.987



7.385
7.366
7.344
7.260
7.233
7.214
7.165
7.148



(2p, ^1H NMR, 400 MHz, CDCl_3)

-3.679

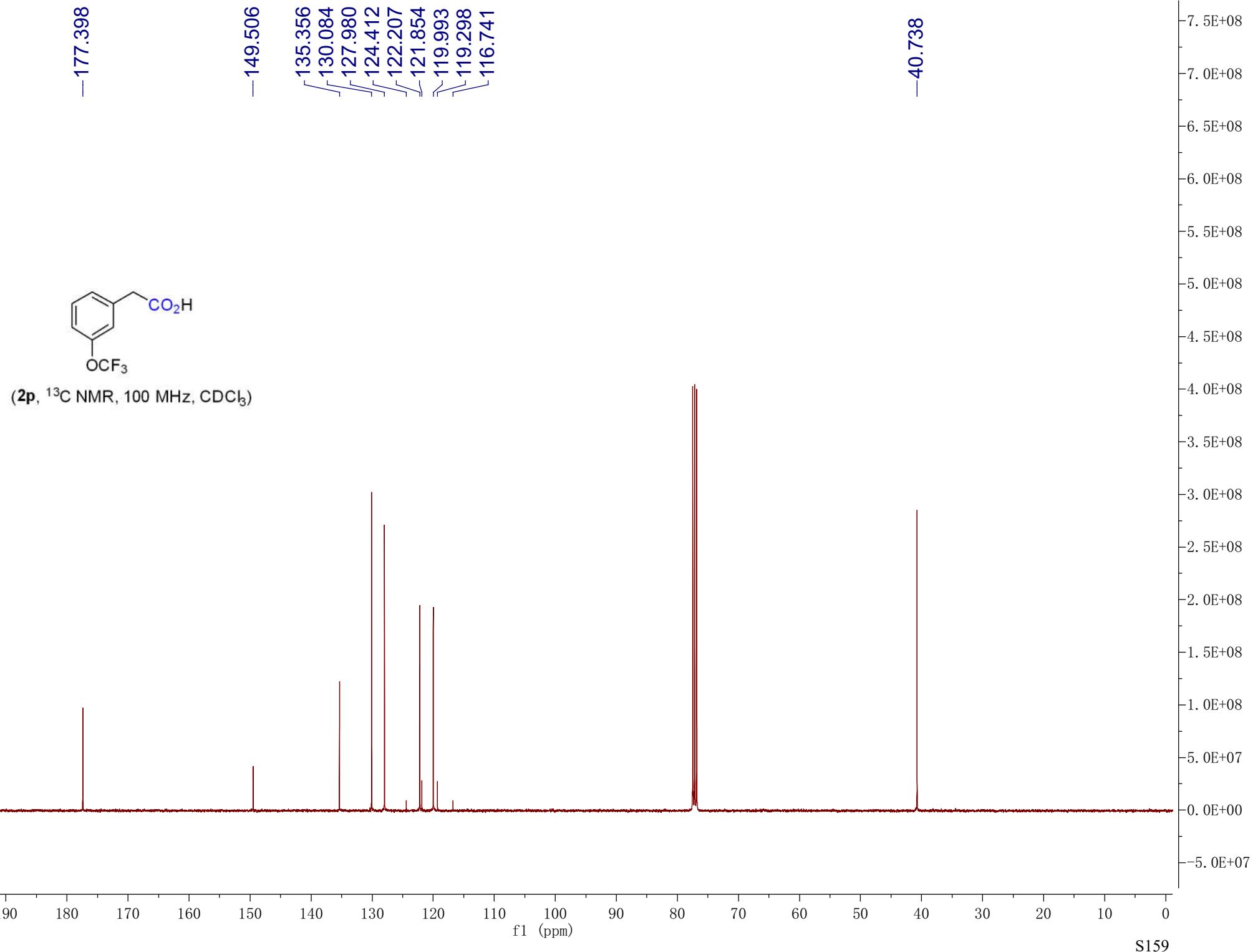
1.00
1.09
2.00

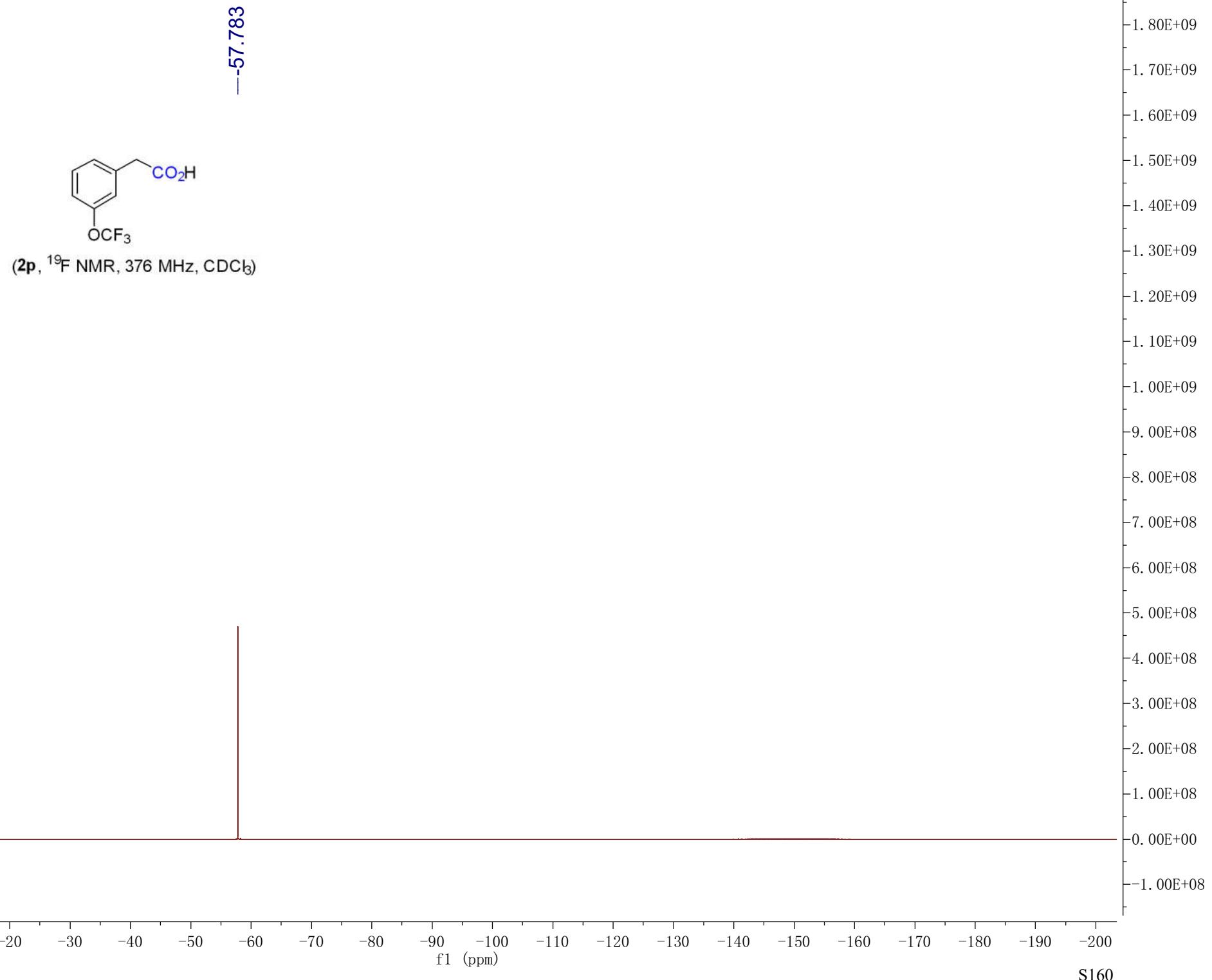
2.00

9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5

f1 (ppm)

S158

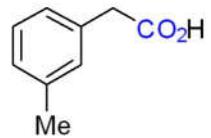




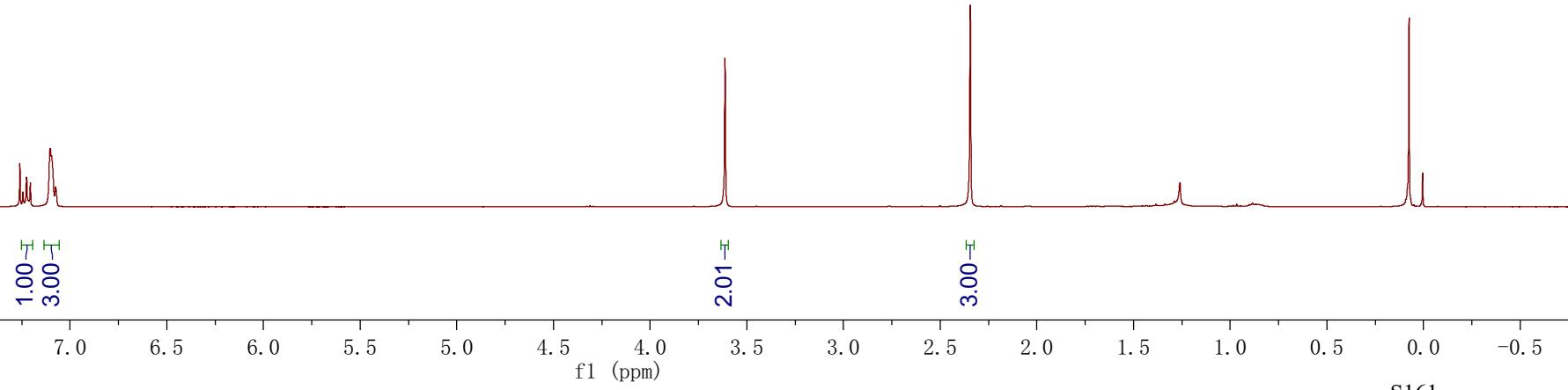
7.244
7.238
7.225
7.218
7.214
7.206
7.103
7.096
7.075

-3.613

-2.344



(2q, ^1H NMR, 400 MHz, CDCl_3)

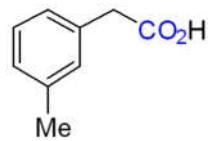


—177.416

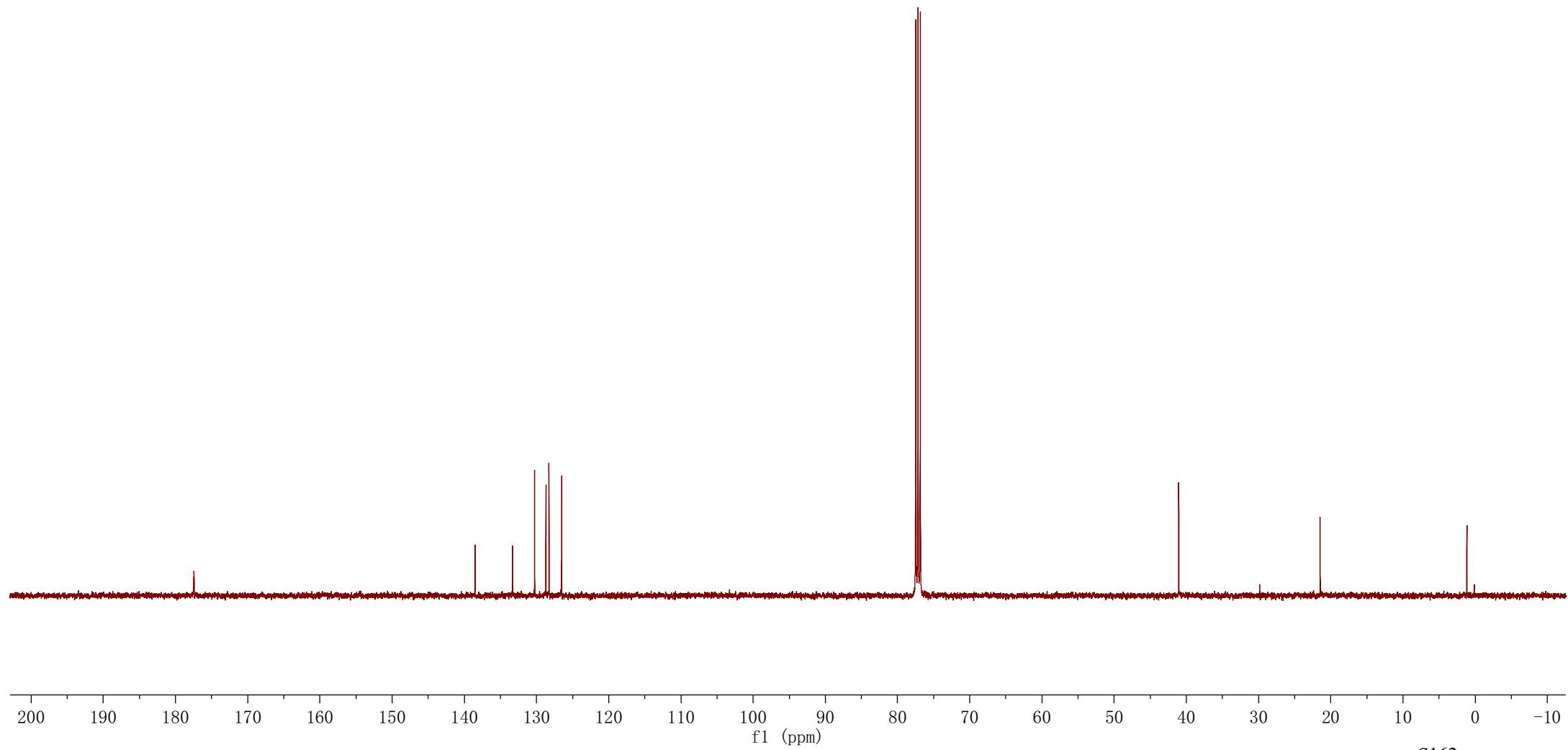
✓138.496
✓133.316
✓130.255
✓128.700
✓128.262
✓126.516

—41.062

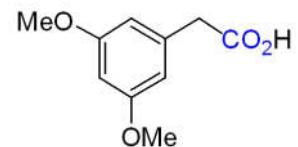
—21.477



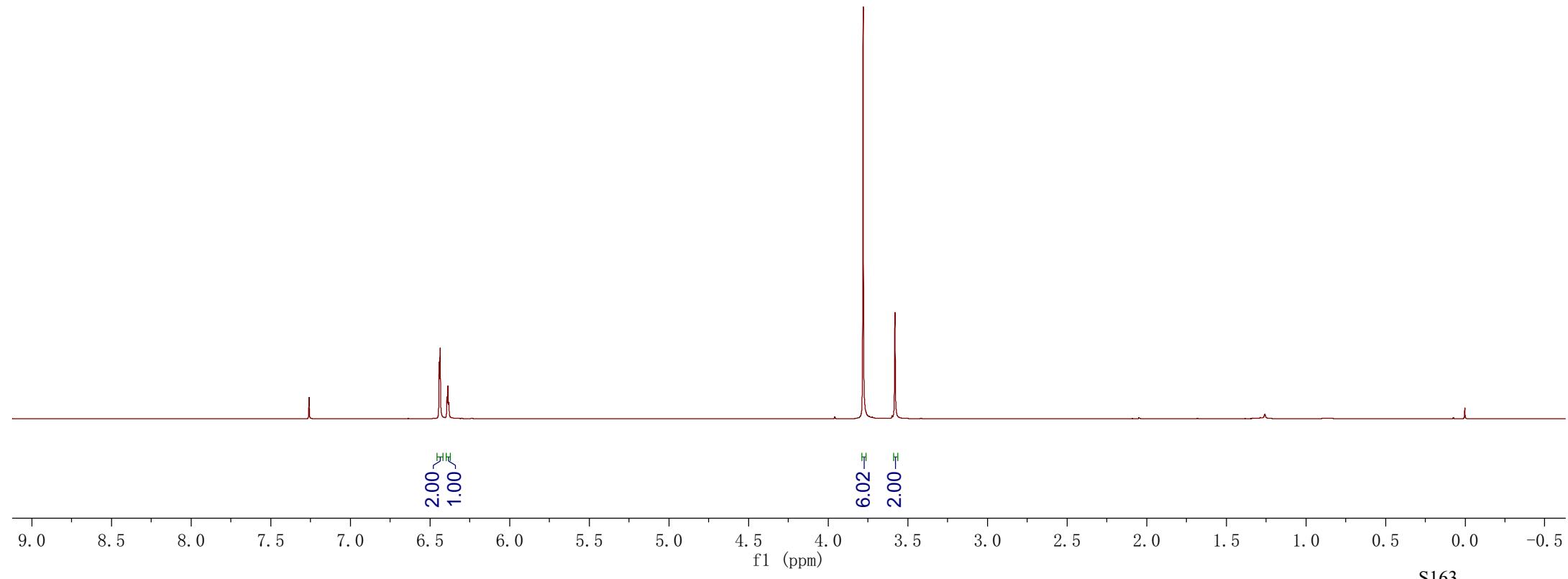
(**2q**, ^{13}C NMR, 100 MHz, CDCl_3)



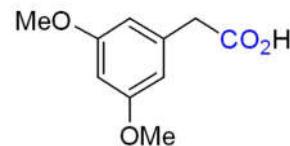
6.442
6.436
6.393
6.388
6.382



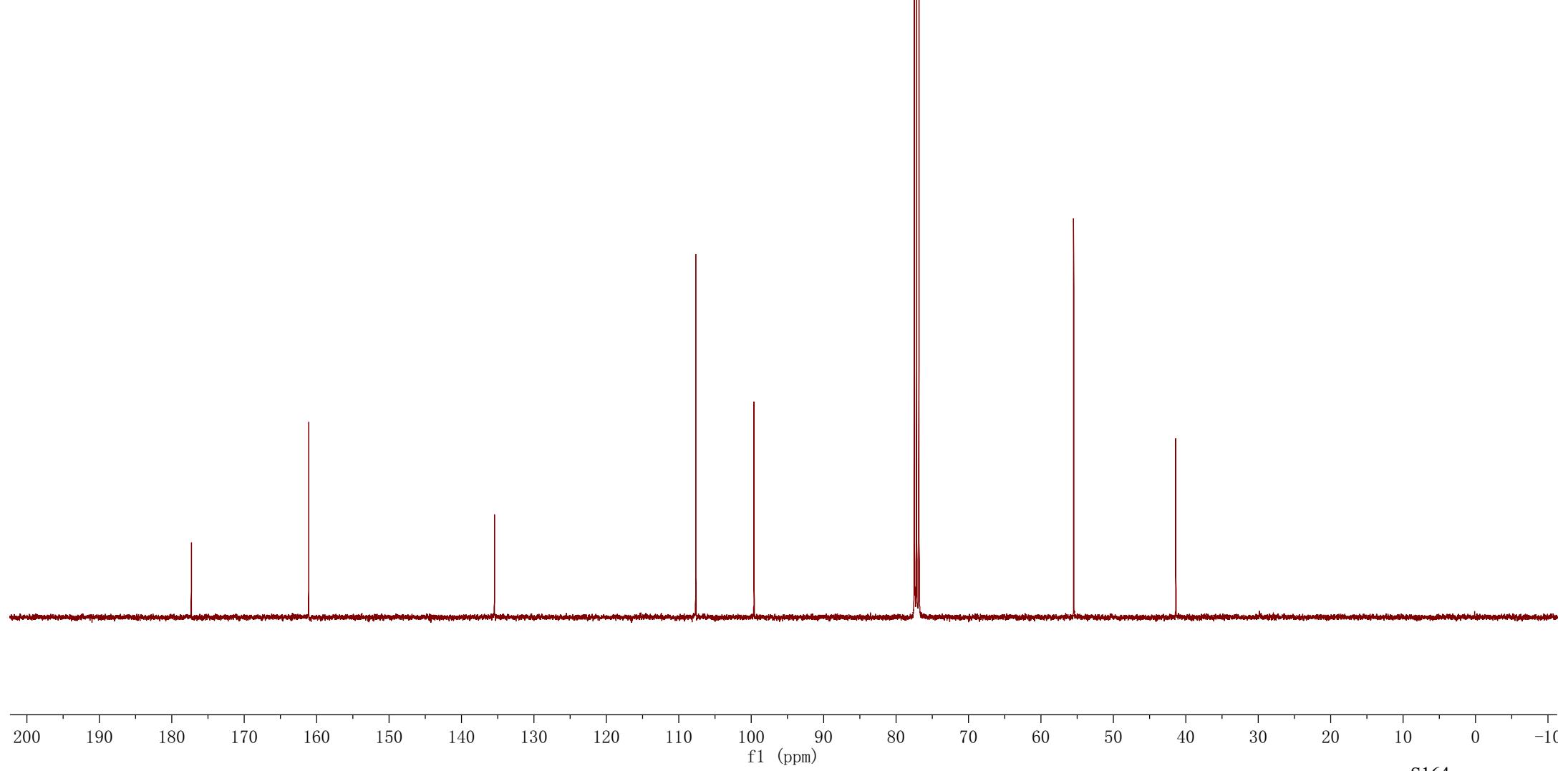
(**2r**, ^1H NMR, 400 MHz, CDCl_3)



—177.262
—161.064
—135.435
—107.642
—99.607
—55.475
—41.382

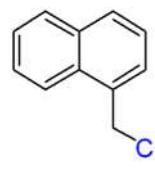


(2r, ¹³C NMR, 100 MHz, CDCl₃)



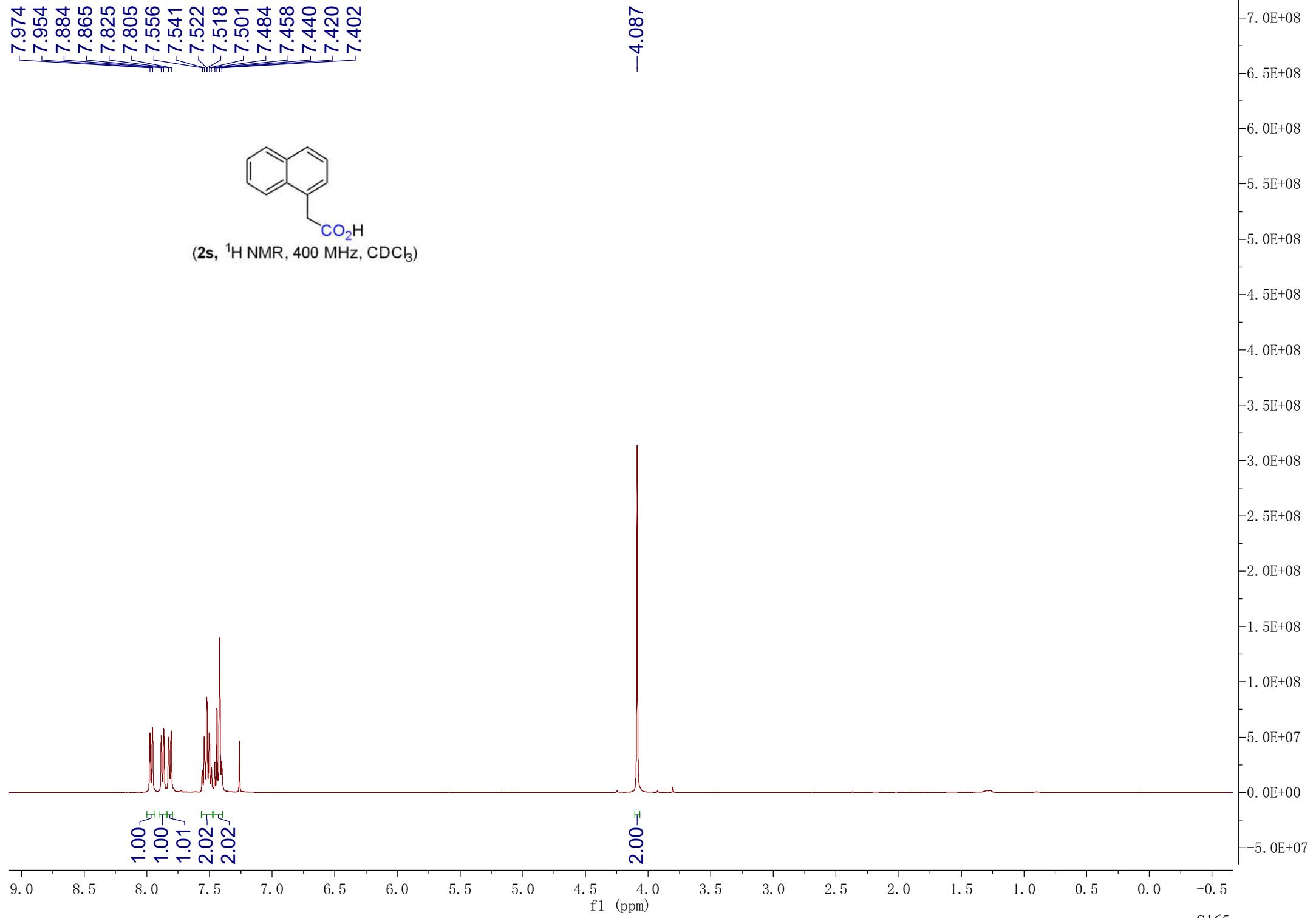
7.974
7.954
7.884
7.865
7.825
7.556
7.522
7.541
7.518
7.805
7.501
7.484
7.458
7.440
7.420
7.402

-4.087



(2s, ^1H NMR, 400 MHz, CDCl_3)

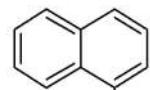
1.00
1.00
1.01
2.02
2.02



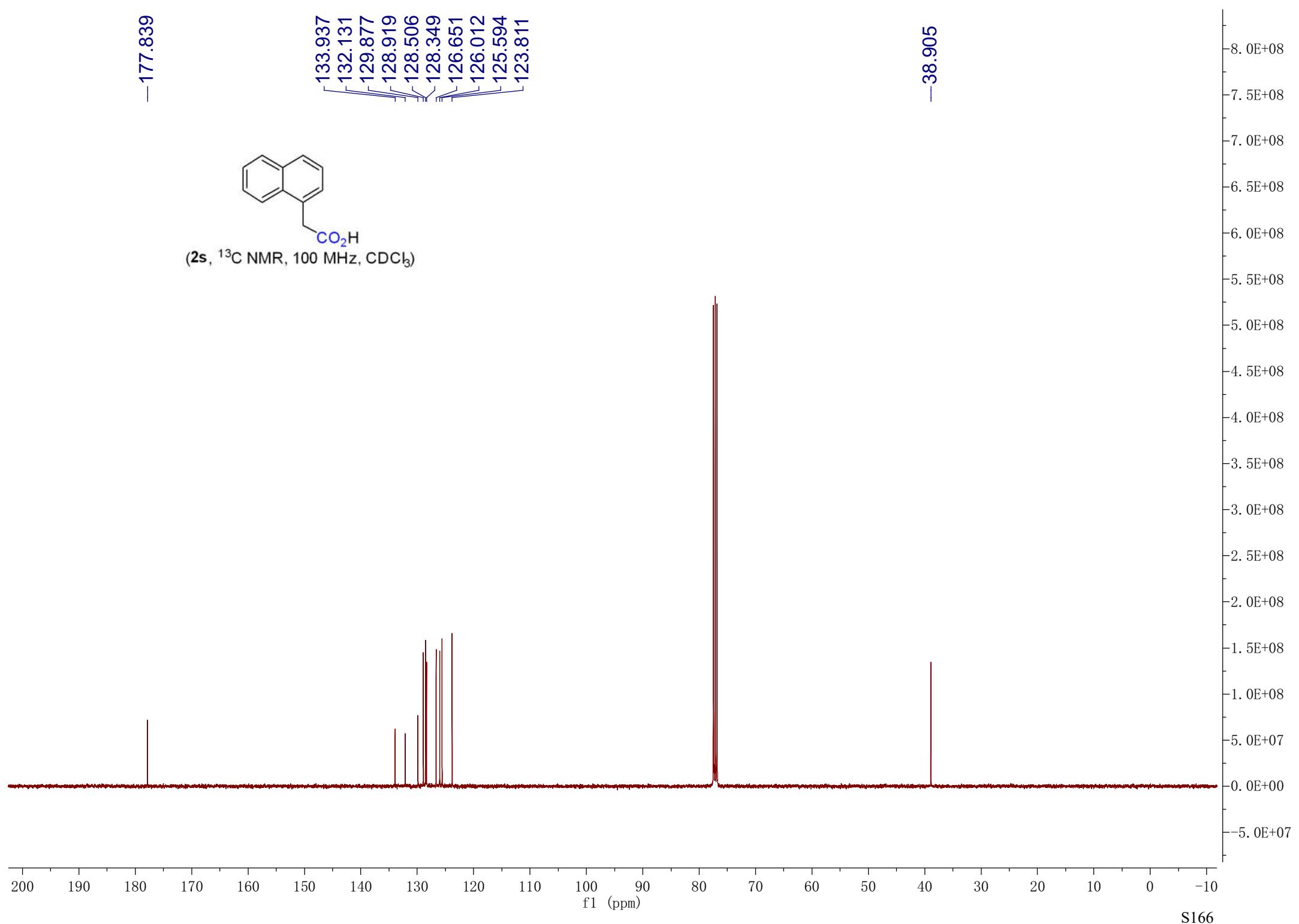
-177.839

133.937
132.131
129.877
128.919
128.506
128.349
126.651
126.012
125.594
123.811

-38.905

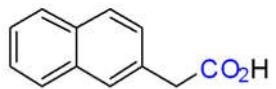


(**2s**, ^{13}C NMR, 100 MHz, CDCl_3)

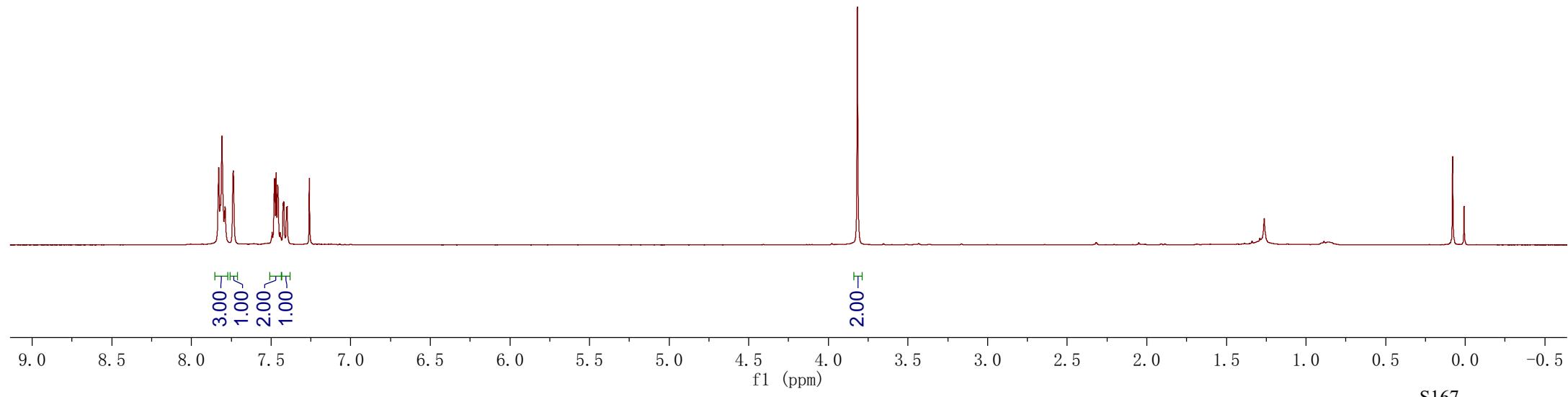


7.829
7.808
7.789
7.738
7.493
7.478
7.468
7.458
7.443
7.424
7.420
7.403
7.399

—3.818



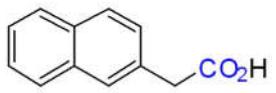
(**2t**, ^1H NMR, 400 MHz, CDCl_3)



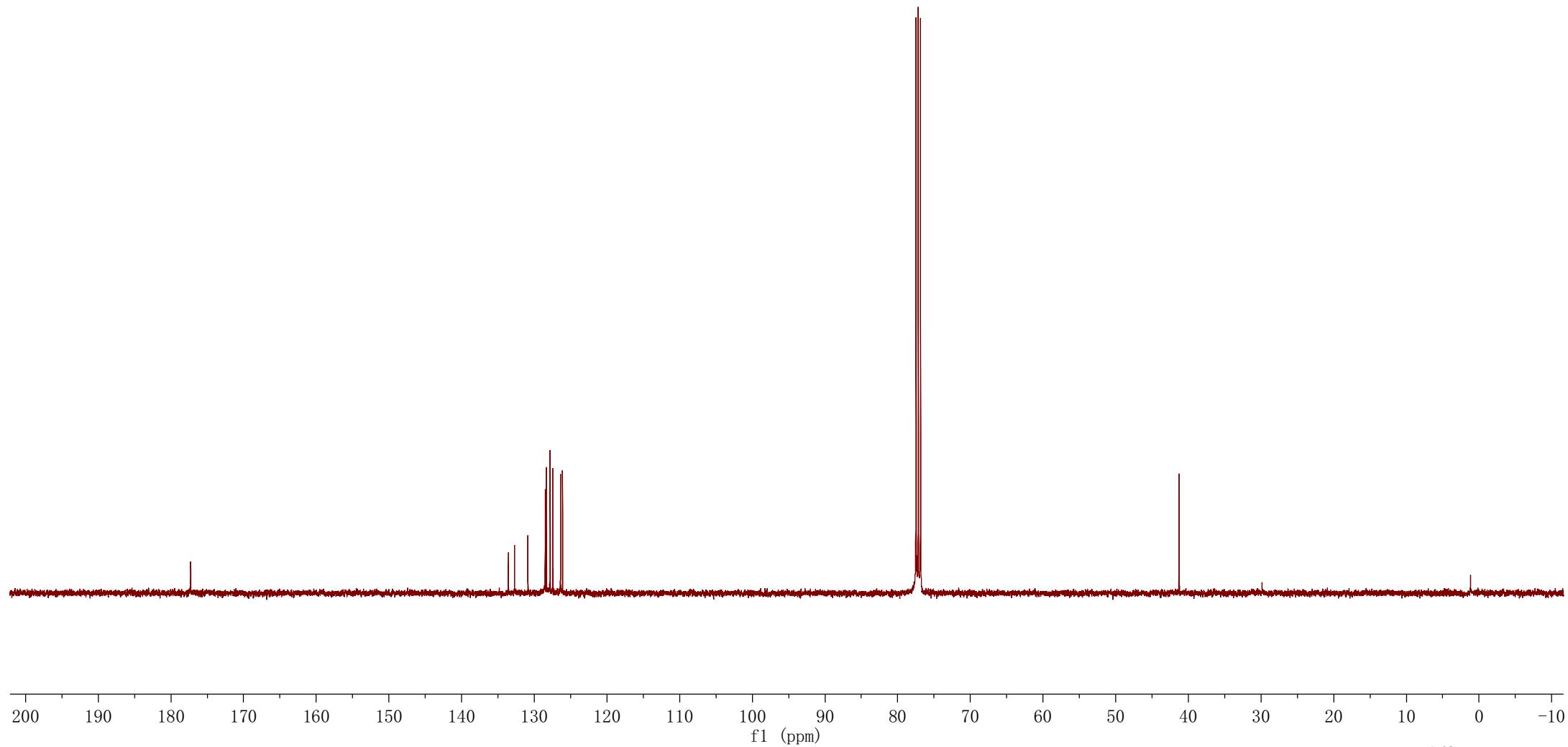
—177.288

133.572
132.723
130.873
128.494
128.331
127.834
127.821
127.450
126.395
126.107

—41.269



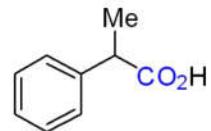
(**2t**, ^{13}C NMR, 100 MHz, CDCl_3)



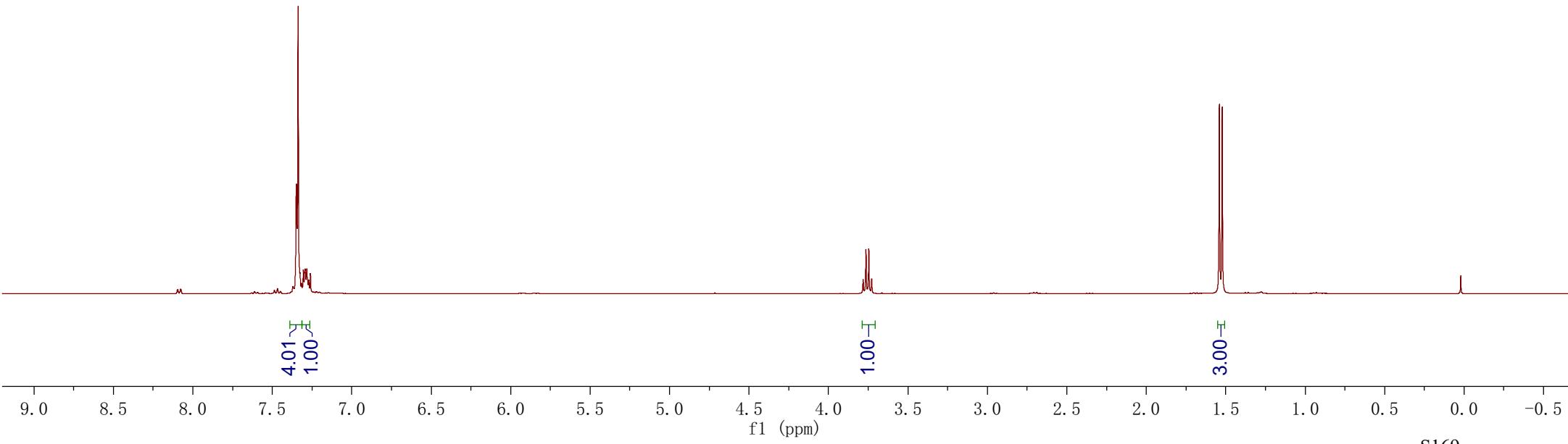
7.347
7.337
7.304
7.297
7.293
7.283
7.270

3.782
3.764
3.746
3.728

1.540
1.522



(**2u**, ¹H NMR, 400 MHz, CDCl₃)



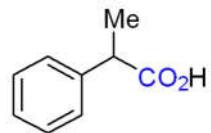
—180.922

—139.910

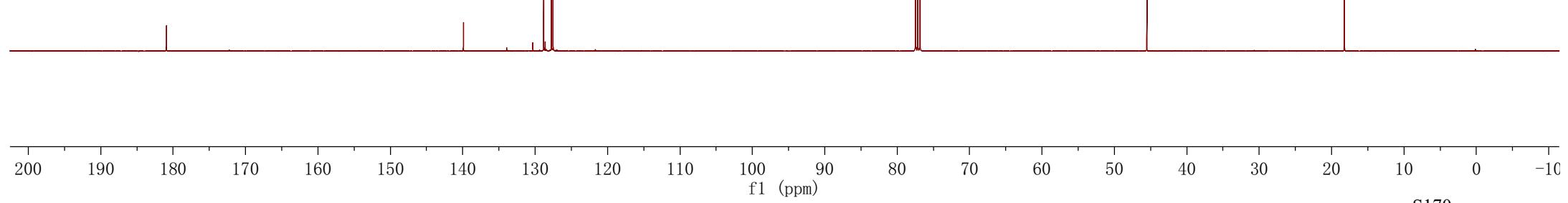
128.821
127.746
127.529

—45.511

—18.232



(**2u**, ^{13}C NMR, 100 MHz, CDCl_3)

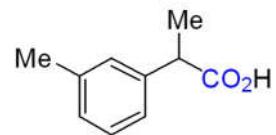


7.250
7.231
7.212
7.139
7.120
7.103
7.085

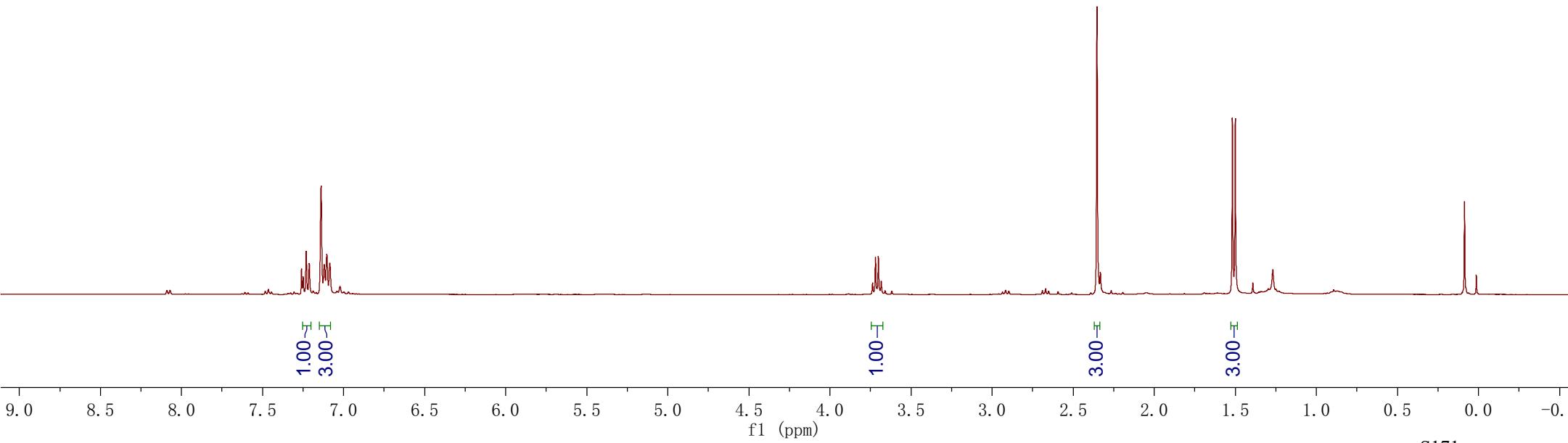
3.737
3.719
3.701
3.684

-2.353

1.518
1.500



(2v, ^1H NMR, 400 MHz, CDCl_3)

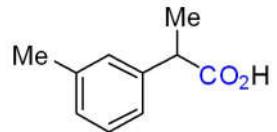


—180.807

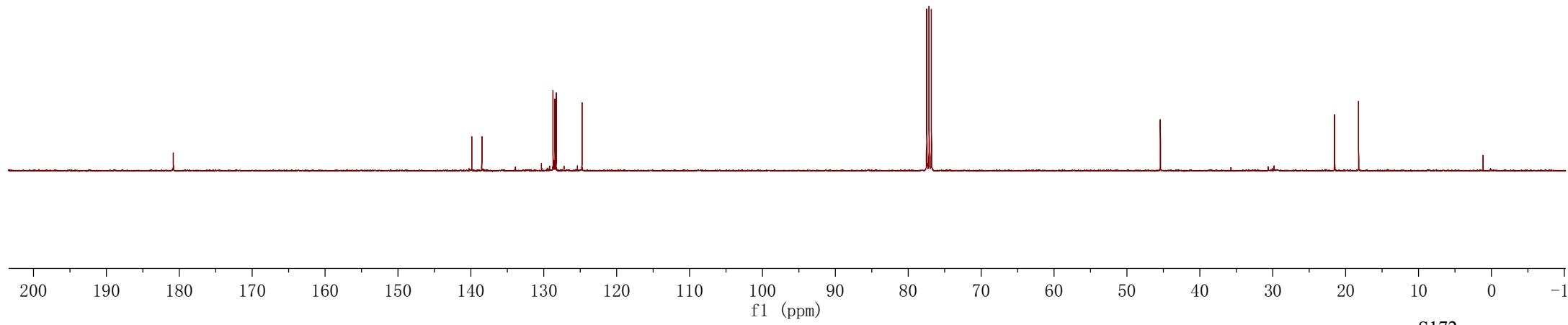
—139.875
—138.491
—128.719
—128.461
—128.299
—124.752

—45.436

—21.546
—18.237



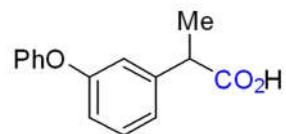
(**2v**, ^{13}C NMR, 100 MHz, CDCl_3)



7.314
7.309
7.295
7.293
7.279
7.274
7.240
7.220
7.214
7.085
7.067
7.048
7.031
7.012
6.986
6.983
6.974
6.969
6.964
6.860
6.856
6.841
6.840
6.836

3.703
3.685
3.667
3.649

1.473
1.455



(2w, ^1H NMR, 400 MHz, CDCl_3)

2.00
1.00
2.00
3.00
1.00

1.00

3.00

9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5

f1 (ppm)

S173

—180.270

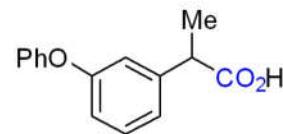
—157.651
—157.098

—141.846

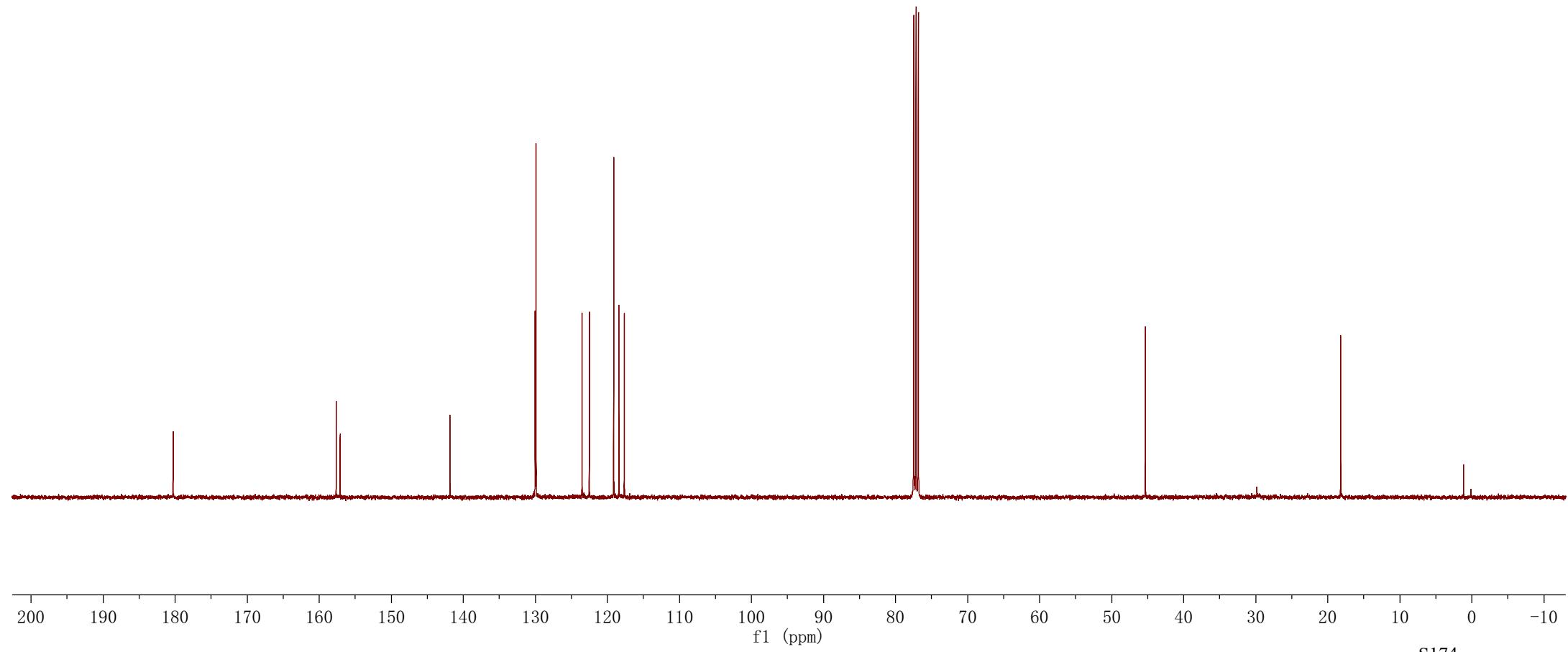
—130.023
—129.911
—123.521
—122.510
—119.127
—118.389
—117.650

—45.327

—18.206



(**2w**, ^{13}C NMR, 100 MHz, CDCl_3)

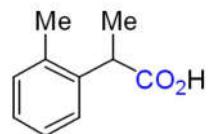


7.312
7.295
7.228
7.216
7.205
7.197
7.183
7.181
7.172

4.020
4.002
3.984
3.966

-2.390

1.509
1.492



(**2x**, ¹H NMR, 400 MHz, CDCl₃)

1.00
3.01

1.02

3.00

3.04

9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5

f1 (ppm)

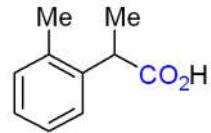
S175

—180.890

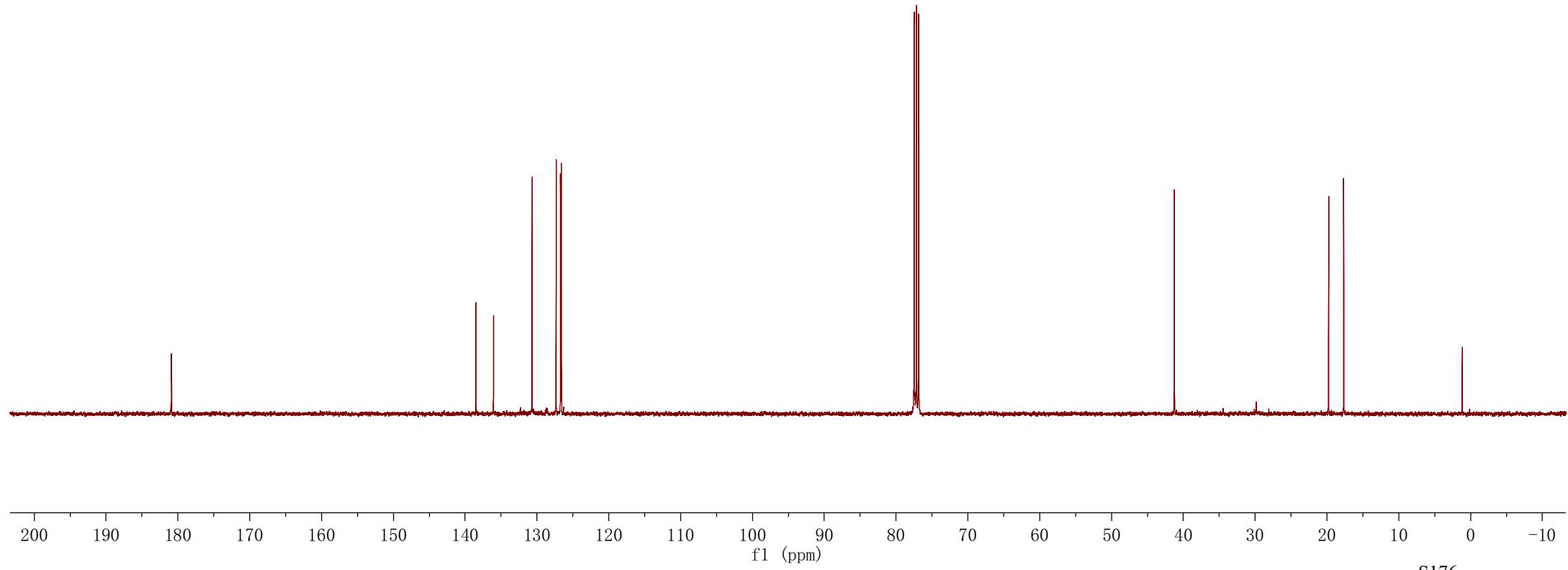
✓138.496
✓136.057
✓130.688
✓127.339
✓126.710
✓126.602

—41.247

✓19.769
✓17.667



(**2x**, ^{13}C NMR, 100 MHz, CDCl_3)

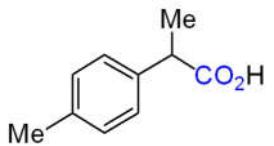


7.227
7.207
7.158
7.138

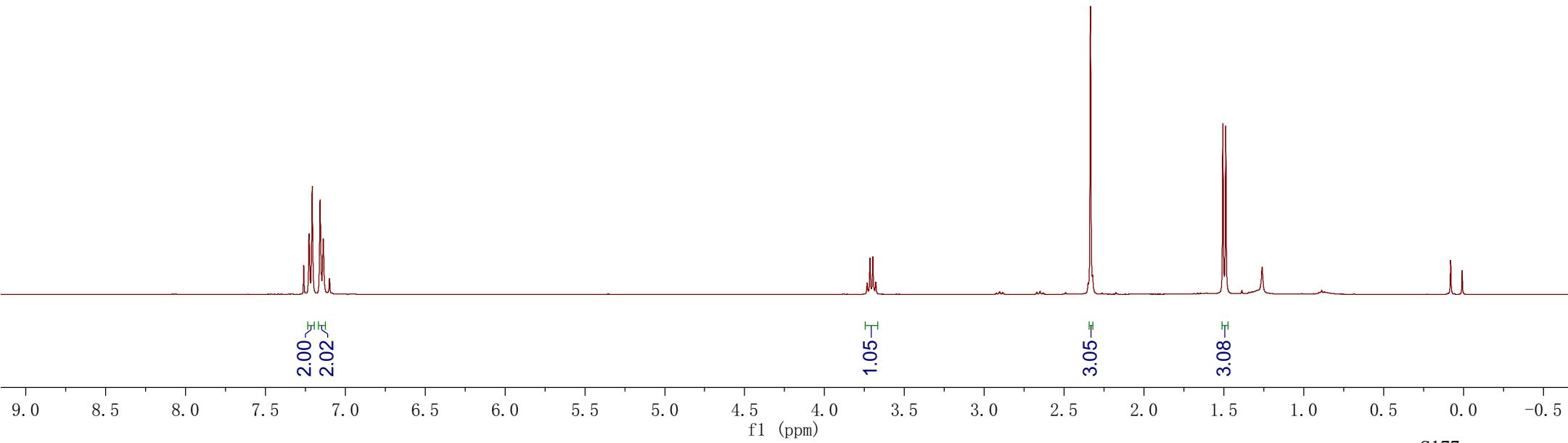
3.733
3.715
3.697
3.679

-2.335

1.506
1.488



(2y, ¹H NMR, 400 MHz, CDCl₃)

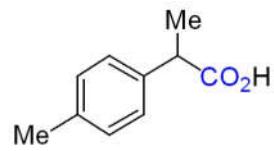


-181.0

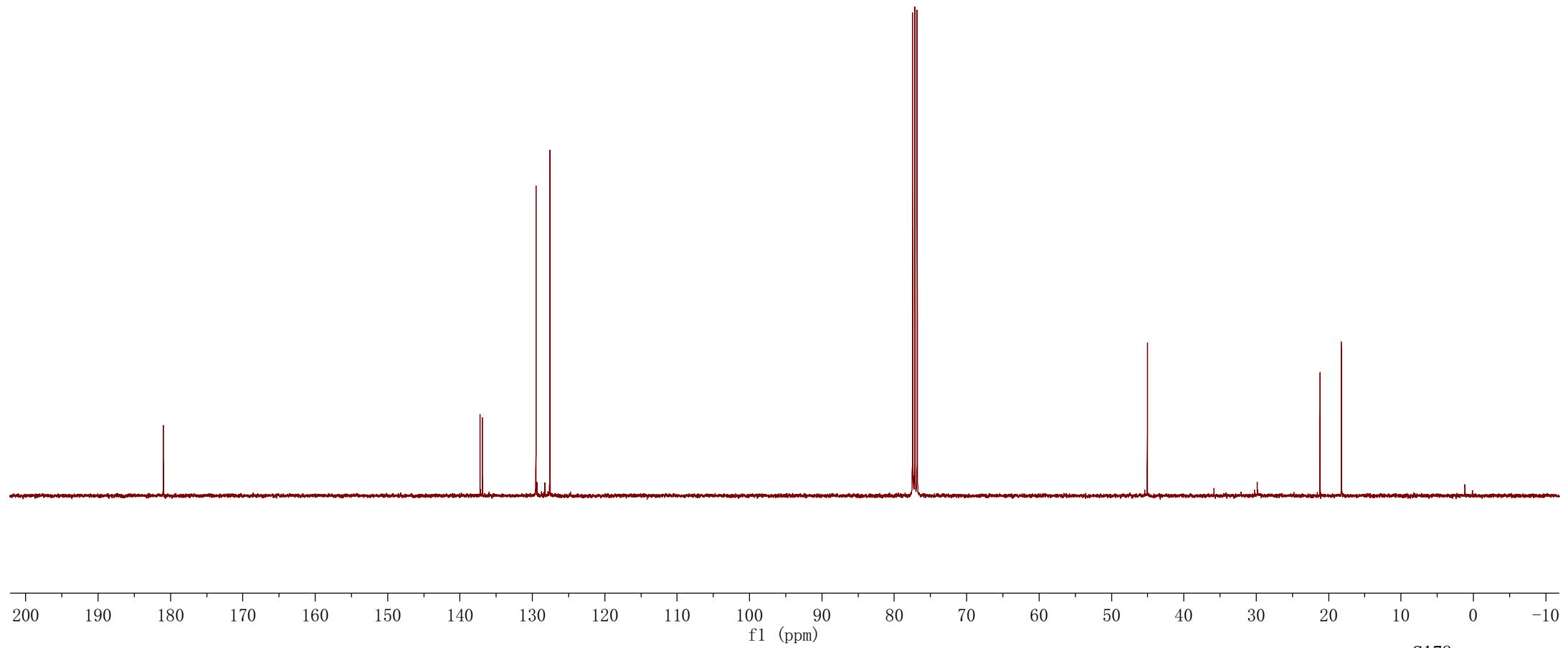
<137.2
<136.9
>129.5
>127.6

-45.0

-21.2
-18.2



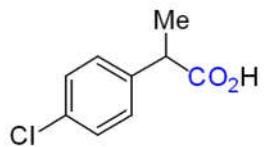
(2y, ¹³C NMR, 100 MHz, CDCl₃)



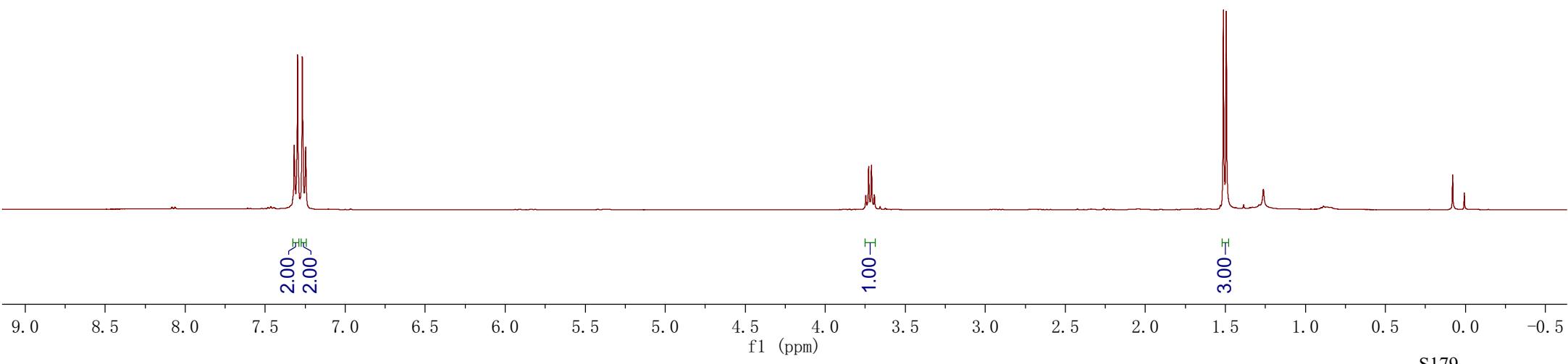
7.319
7.297
7.268
7.246

3.747
3.729
3.711
3.693

1.512
1.494



(**2z**, ^1H NMR, 400 MHz, CDCl_3)

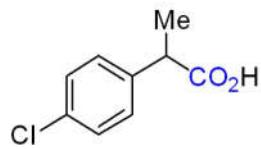


—180.340

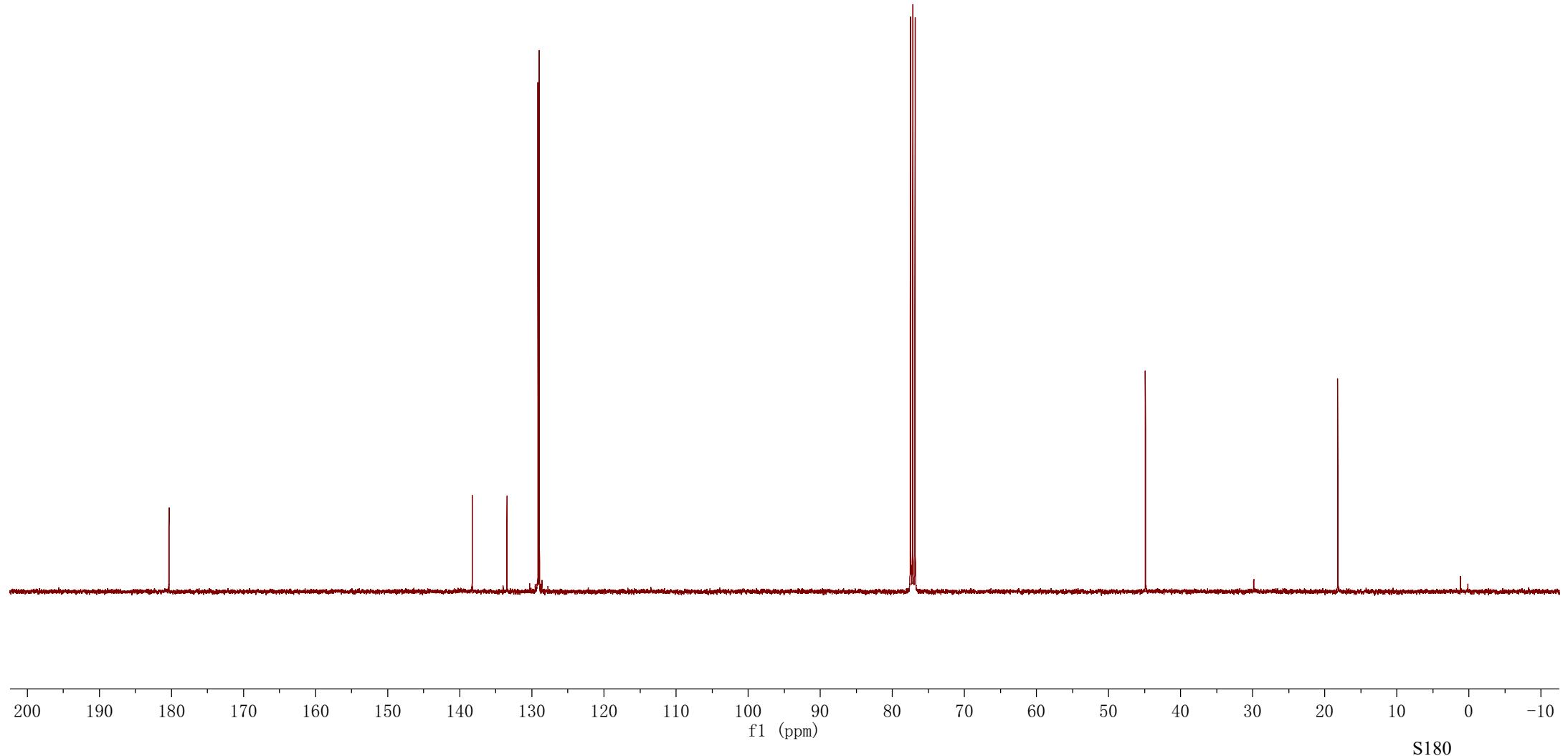
—138.286
✓ 133.465
✓ 129.146
✓ 128.974

—44.878

—18.198

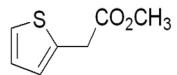


(**2z**, ^{13}C NMR, 100 MHz, CDCl_3)

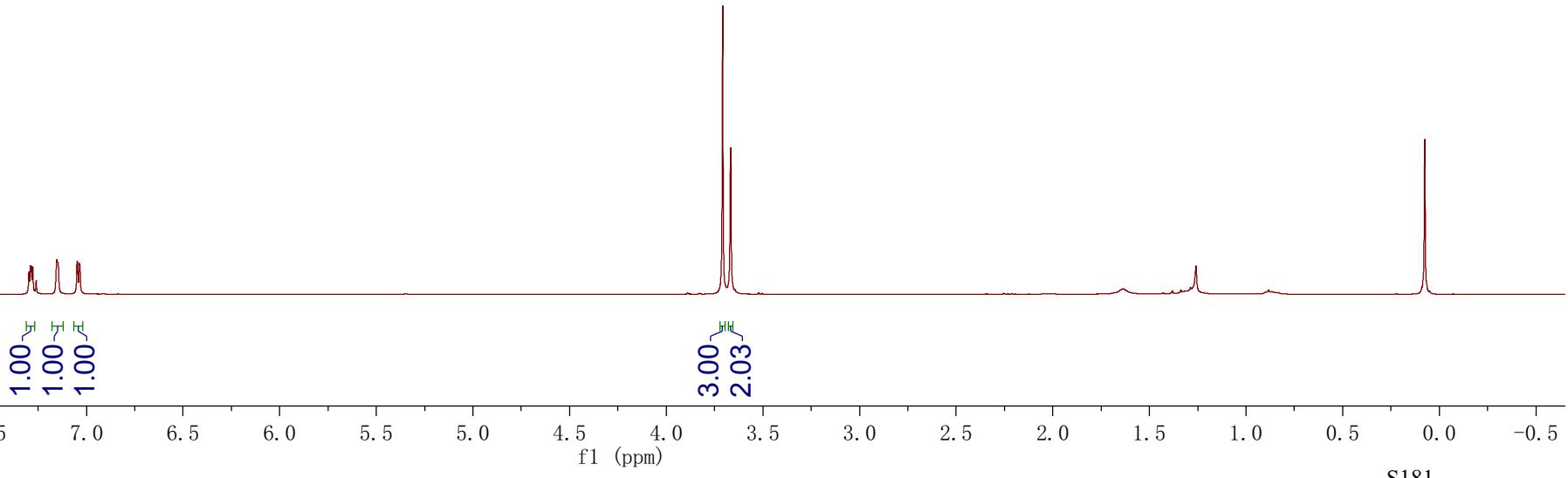


7.297
7.290
7.285
7.278
7.153
7.148
7.048
7.036

3.708
3.667



(**2a'**, ^1H NMR, 400 MHz, DMSO- d_6)

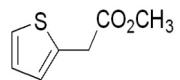


-171.710

~133.661
~128.601
~125.875
~123.007

-52.176

-35.776



(2a', ¹³C NMR, 100 MHz, DMSO-*d*₆)

