

Supporting Information

Metal-Free Chemoselective Hydrogenation of Unsaturated Carbon-Carbon Bonds via Cathodic Reduction

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A. General Methods

Unless otherwise noted, all the chemicals were purchased commercially, and used without further purification. Technical grade petroleum ether (40-60 °C bp.) and ethyl acetate were used for chromatography column. ¹H and ¹³C NMR spectra were recorded on a Bruker Advance III 500 spectrometers using CDCl₃ as solvent with TMS as the internal standard. The chemical shifts are referenced to signals at 7.26 and 77.0 ppm, respectively. Chemical shift (δ) and coupling constants (J) are given in ppm and in Hz, respectively. The peak patterns are indicated as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. The chemical shift signals at 1.28 and 0.92 ppm in ¹H NMR are the signal peaks of impurities. Electrolysis reactions were conducted using a Model MCH-K-300D (36 V) power supply purchased from Meichuang Instruments (Shenzhen) Co., Ltd. Cyclic voltammetry (CV) analysis was performed on Princeton PARSTAT4000 electrochemical workstation, using glassy carbon electrode as working electrode, Pt electrode as counter electrode and Ag/AgCl electrode as a reference electrode. Cyclic voltammogram was recorded at 0.2 V/s scan rate. GC yield and mass spectra were recorded on a Shimadzu GCMS-QP2020 gas chromatograph-mass spectrometer, where *n*-dodecane was used as the internal standard when determining the yield by GC analysis. The data of HRMS was carried out on a high-resolution mass spectrometer (Thermo Scientific Q Exactive-UltiMate3000). TLC was performed by using commercially prepared 100-400 mesh silica gel plates and visualization was affected at 254 nm. Deuterium incorporation was determined by integration of the residual formyl proton in ¹H NMR.

B. General Procedures

General Experimental Procedure for Preparation of Desired Products 2: A mixture of **1** (0.5 mmol), ⁿBu₄NHSO₄ (1 equiv) in 4 mL DMSO was added to an electrolytic cell (30 mL). The electrolytic cell was equipped with graphite rod (ϕ 6 mm) as anode and graphite rod (ϕ 6 mm) as cathode. The solution was electrolyzed at ambient temperature under a constant current (10 mA) for 5-6 h. After electrolysis, the mixture was poured into water and extracted with ethyl acetate twice. The combined organic layer was washed with brine (10 mL) and dried over MgSO₄, filtered and concentrated. The resulting mixture was purified by silica gel column

chromatography to afford **2**.

General Experimental Procedure for Preparation of Desired Products 4: A mixture of **3** (0.5 mmol), $n\text{Bu}_4\text{NHSO}_4$ (1 equiv) in 4 mL DMSO was added to an electrolytic cell (30 mL). The electrolytic cell was equipped with graphite rod (ϕ 6 mm) as anode and graphite rod (ϕ 6 mm) as cathode. The solution was electrolyzed at ambient temperature under a constant current (5 mA) for 10 h. After electrolysis, the mixture was poured into water and extracted with ethyl acetate twice. The combined organic layer was washed with brine (10 mL) and dried over MgSO_4 , filtered and concentrated. The resulting mixture was purified by silica gel column chromatography to afford **4**.

General Experimental Procedure for Preparation of Desired Products 6: A mixture of **5** (0.5 mmol), $n\text{Bu}_4\text{NHSO}_4$ (1 equiv) in 4 mL DMSO was added to an electrolytic cell (30 mL). The electrolytic cell was equipped with graphite rod (ϕ 6 mm) as anode and graphite rod (ϕ 6 mm) as cathode. The solution was electrolyzed at ambient temperature under a constant current (10 mA) for 6-10 h. After electrolysis, the mixture was poured into water and extracted with ethyl acetate twice. The combined organic layer was washed with brine (10 mL) and dried over MgSO_4 , filtered and concentrated. The resulting mixture was purified by silica gel column chromatography to afford **6**.

Procedure for the Gram-scale Synthesis of 2a and 6c: A mixture of **1a** (6 mmol) or **5c** (6 mmol), $n\text{Bu}_4\text{NHSO}_4$ (1 equiv) in 48 mL DMSO was added to a three-necked flask (100 mL). The solution was electrolyzed in the three-necked flask equipped with graphite rod (ϕ 15 mm) as anode and graphite rod (ϕ 15 mm) as cathode at ambient temperature under a constant current (50 mA) for 14 h. After electrolysis, the mixture was poured into water and extracted with ethyl acetate twice. The combined organic layer was washed with brine (10 mL) and dried over MgSO_4 , filtered and concentrated. The resulting mixture was purified by silica gel column chromatography to afford **2a** (0.77 g, 5.1 mmol) and **6c** (0.68 g, 5.2 mmol).

General Experimental Procedure for Preparation of $n\text{Bu}_4\text{NDSO}_4$: $n\text{Bu}_4\text{NOH}$ solution (40 wt% in MeOH, 648.68 mg) was added into a round bottom flask for concentration and drying. After adding D_2O (1 mL) to dissolve the $n\text{Bu}_4\text{NOH}$ solid, D_2SO_4 (96%-98% in D_2O , 100.09 mg)

was added for reaction. After stirring the mixture for 20 min, drying and removing D₂O to give the desired product ⁿBu₄NDSO₄.

C. Cyclic Voltammograms

In order to analyze the reaction process in depth, we implemented cyclic voltammetry (CV) experiment and recorded cyclic voltammetry (CV) diagram shown below (Figure 1). With 0.1 M ⁿBu₄NHSO₄ solution in DMSO as electrolyte solution, the reduction peaks of cinnamionitrile (**5c**) were recorded at -0.272 V and -0.216 V vs Ag/AgCl, respectively, which indicated that two reduction processes appeared in the reaction.

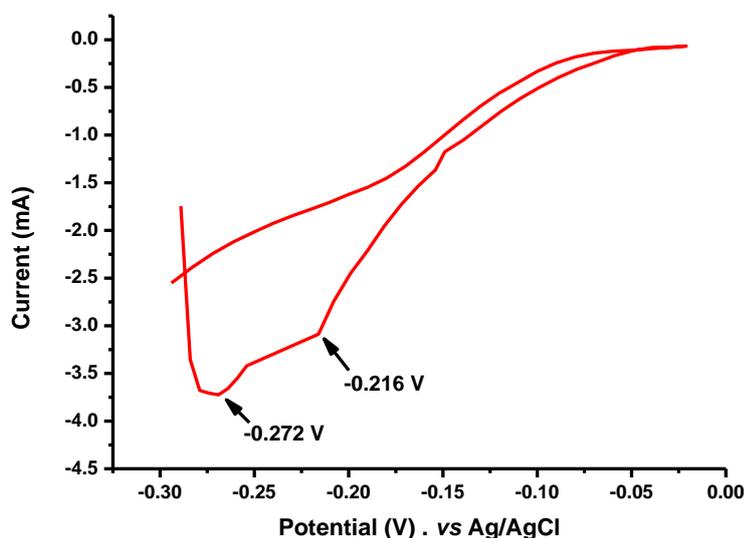
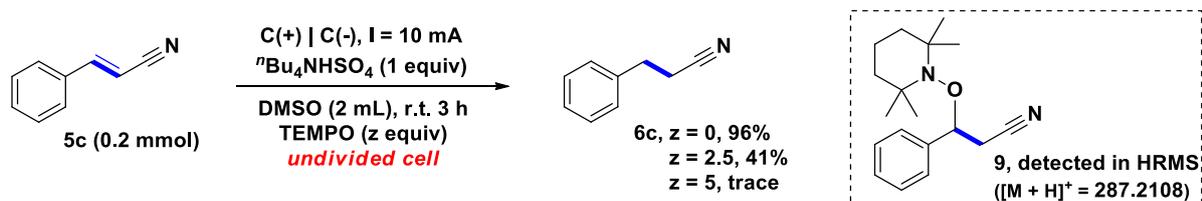


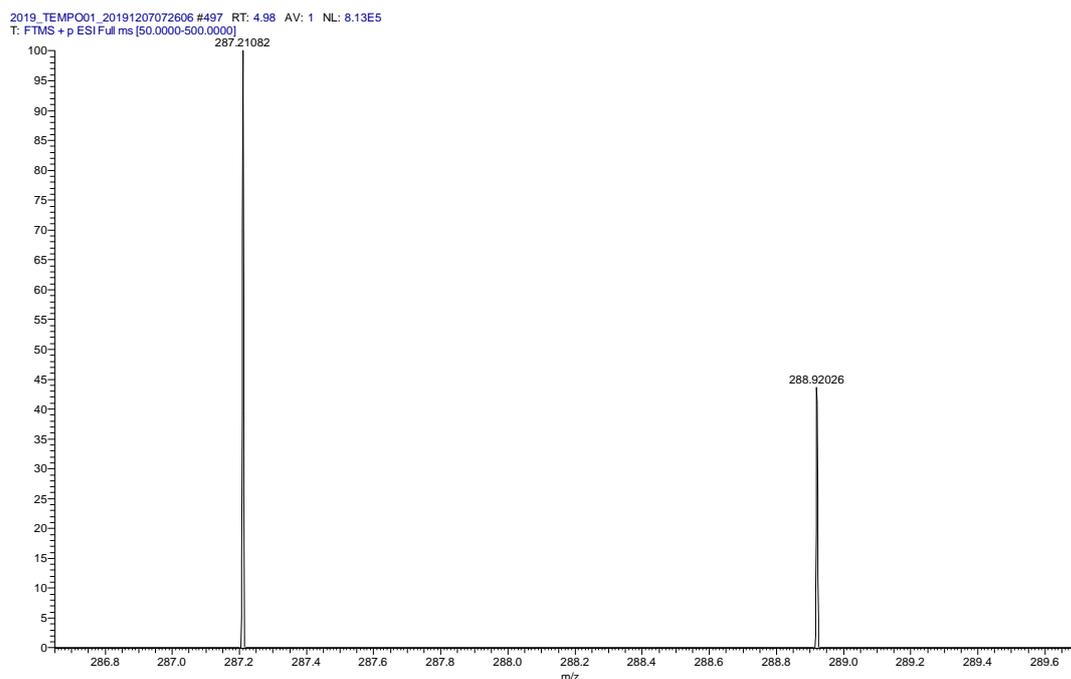
Figure 1. Cyclic voltammogram of 0.1 M ⁿBu₄NHSO₄ solution in DMSO with the addition of cinnamionitrile **1a** (0.1 M) at room temperature. The voltammogram was obtained at a scan rate of 0.2 V/s with glassy carbon electrode as working electrode, Pt electrode as counter electrode and Ag/AgCl electrode as a reference electrode.

D. Free Radical Trapping Experiments

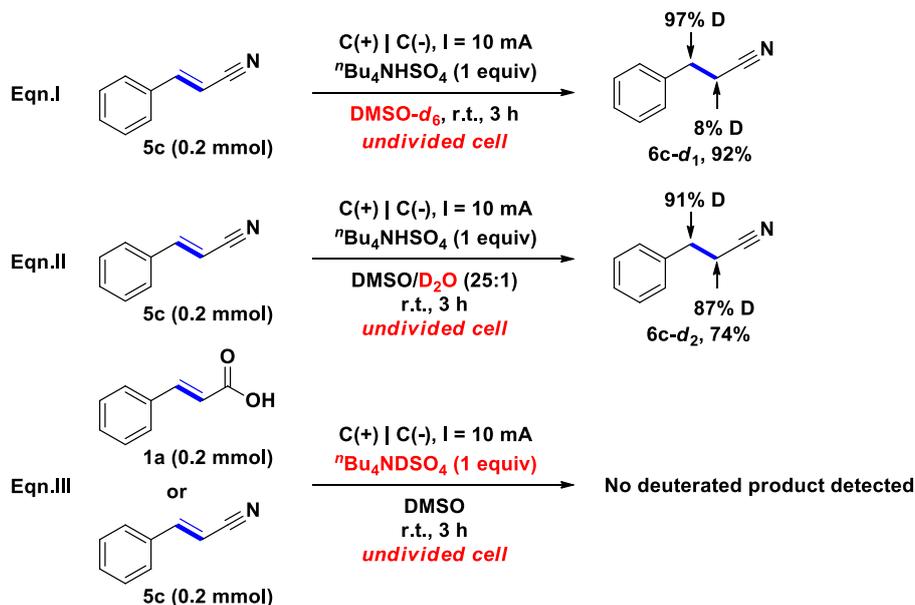


Scheme S1. Free radical trapping experiments. Reaction conditions: (I) cinnamitrile **5c** (0.2 mmol), $n\text{Bu}_4\text{NHSO}_4$ (1 equiv), DMSO (2 mL). The amount of TEMPO is 0 eqviu, 2.5 eqviu and 5 eqviu, respectively. The electrolysis was conducted in an undivided cell at room temperature under air for 3 h. The yields of product **6c** was determined by GC yields with *n*-dodecane as the internal standard, and the free radical trapping product **9** was confirmed by HRMS analysis: HRMS (ESI) (*m/z*): calcd for $\text{C}_{18}\text{H}_{27}\text{N}_2\text{O}$ $[\text{M} + \text{H}]^+$: 287.2118, found: 287.2108.

HRMS analysis of **9**:

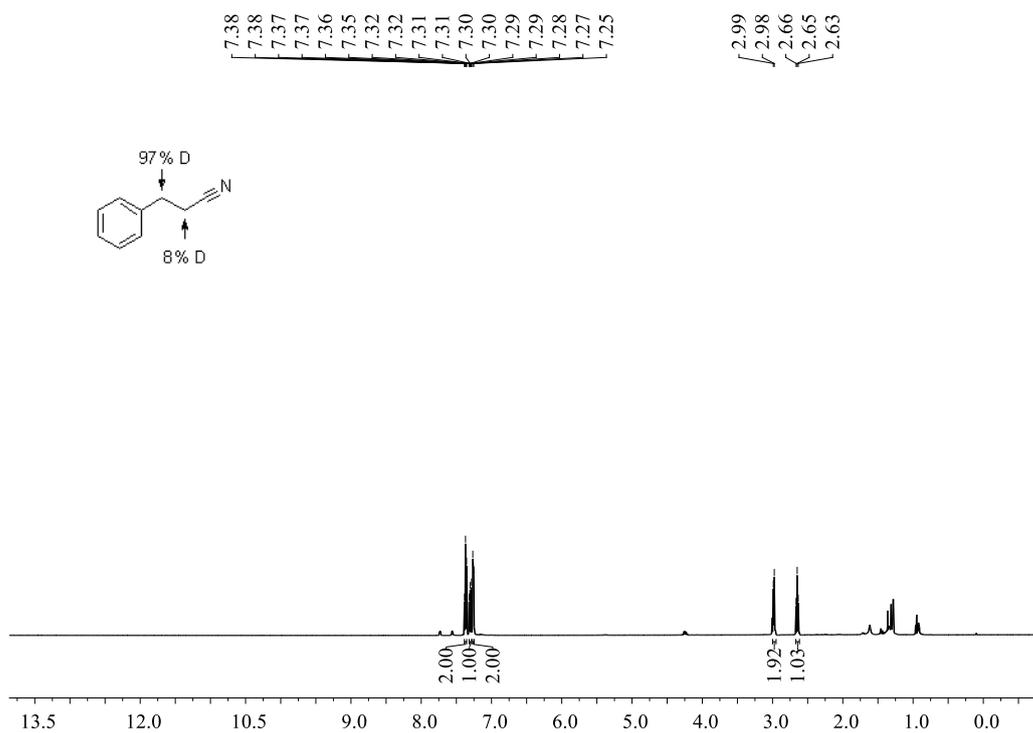


E. Deuterium-labeling Experiments

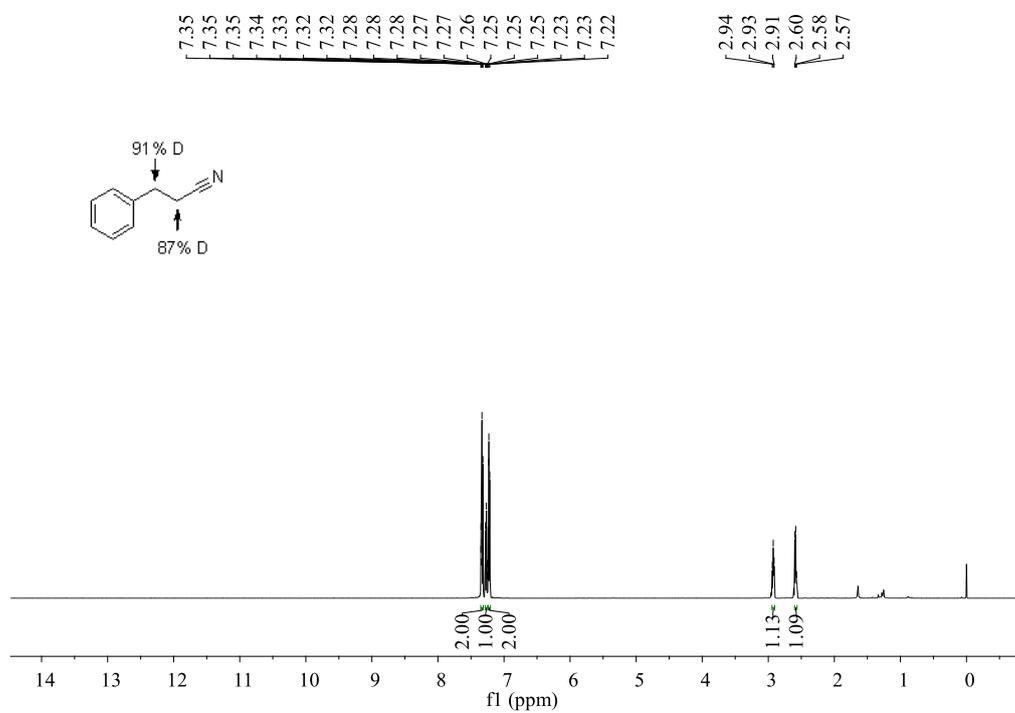


Scheme S2. Deuterium-labeling experiments. Reaction conditions: (I) cinnamitrile **5c** (0.2 mmol), *n*Bu₄NHSO₄ (1 equiv), DMSO-*d*₆ (2 mL). (II) cinnamitrile **5c** (0.2 mmol), *n*Bu₄NHSO₄ (1 equiv), dry DMSO/D₂O (25/1 v/v, 2 mL). (III) **1a** (0.2 mmol) or **5c** (0.2 mmol), *n*Bu₄NDSO₄ (1 equiv), DMSO (2 mL). The electrolysis was conducted in an undivided cell at room temperature under air for 3 h. **6c-*d*₁** was isolated in 92% yield with 8% D-incorporation at α position and 97% D-incorporation at β position as revealed by ¹H NMR. **6c-*d*₂** was isolated in 74% yield with 87% D-incorporation at α position and 91% D-incorporation at β position as revealed by ¹H NMR.

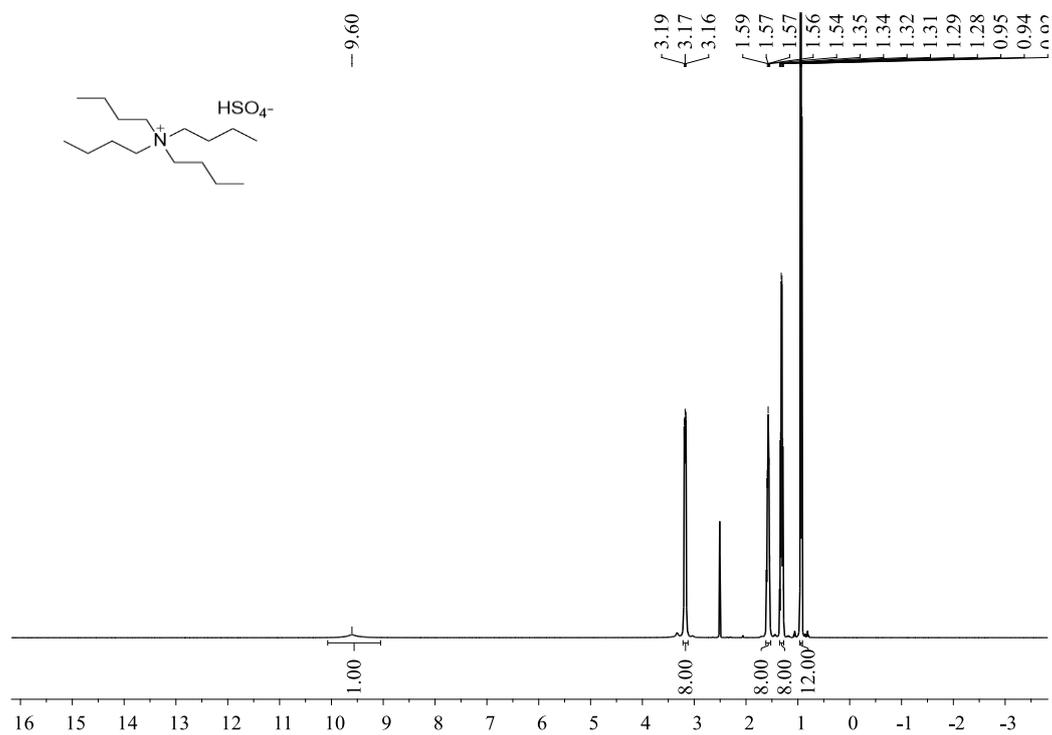
¹H NMR (500 MHz, CDCl₃) spectrum of 6c-d₁:



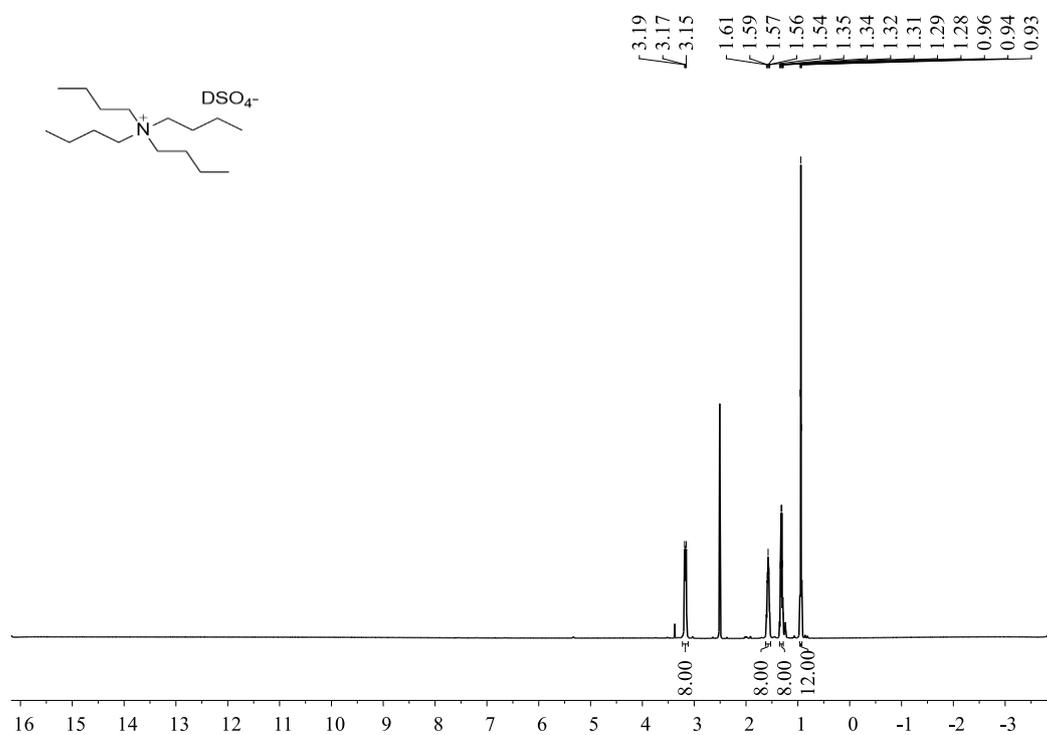
¹H NMR (500 MHz, CDCl₃) spectrum of 6c-d₂:



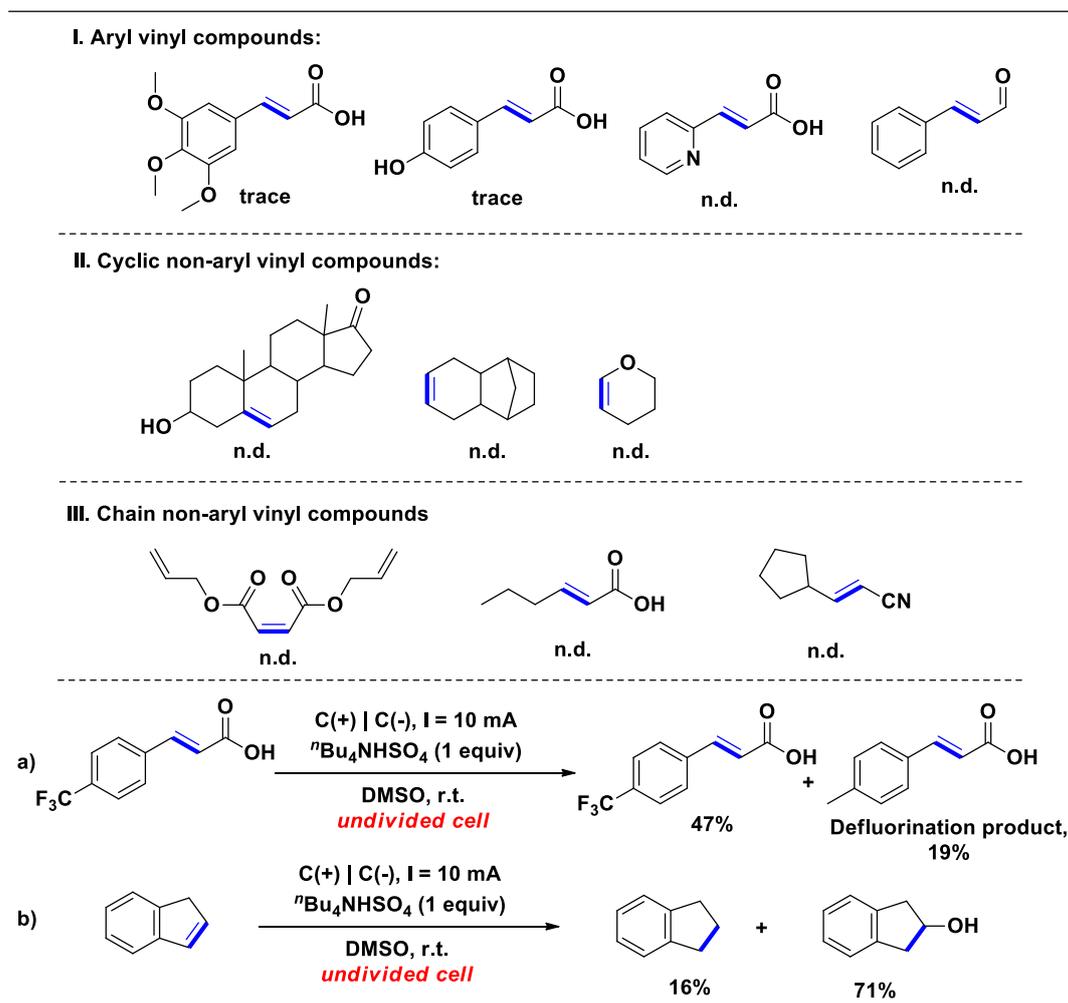
^1H NMR (500 MHz, CDCl_3) spectrum of $n\text{Bu}_4\text{NHSO}_4$



^1H NMR (500 MHz, CDCl_3) spectrum of $n\text{Bu}_4\text{NDSO}_4$



F. Other Olefin Compounds Examined

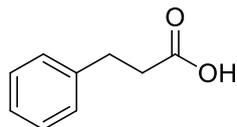


Scheme S3. Examination of olefin compounds.

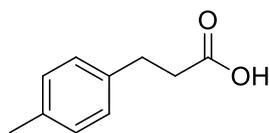
In order to expand the scope of the substrates, we examined different types of olefin compounds (Scheme S3). We continue to try other cinnamic acid and cinnamaldehyde substrates, only a trace of desired products could be detected in the electrochemical hydrogenation of 3,4,5-trimethoxycinnamic acid and *cis*-4-hydroxycinnamic acid (I). No expected products were detected when using 3-(pyridin-2-yl) acrylic acid and cinnamaldehyde as reaction substrates (I). Subsequently, we also tried some cyclic non-aryl vinyl substrates, such as dehydroepiandrosterone, tricyclo [6.2.1.0 (2,7)] undeca-4-ene and 3,4-dihydro-2*H*-pyran, but none of the expected products were detected in the reaction (II). Then, some chain non-aryl vinyl, such as diallyl maleate, *trans*-2-hexenoic acid and 3-cyclopentylacrylonitrile, also proved to be unsuitable (III). The electrochemical hydrogenation of 4-(trifluoromethyl)cinnamic acid under standard conditions produces the

expected product in 47% yield and the defluorinated product in 19% yield, which made the product difficult to separate (a). In addition, when indene was used as a substrate, the expected product was detected with a GC yield of 16%. Meanwhile, we could also detect 2,3-dihydro-1*H*-inden-2-ol with a GC yield of 71% (b).

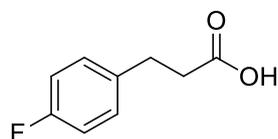
G. Characterization Data for Products



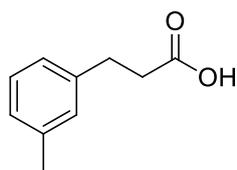
3-Phenylpropanoic acid (2a). Yellow oil, yield: 68 mg (91%). $R_f = 0.85$ (dichloromethane→ethyl acetate). $^1\text{H NMR}$ (500 MHz, CDCl_3 , ppm) $\delta = 7.33$ (m, 2H), 7.25 (dd, $J = 7.2, 5.2$ Hz, 3H), 2.99 (t, $J = 7.8$ Hz, 2H), 2.72 (m, 2H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3 , ppm) $\delta = 178.8, 140.2, 128.6, 128.3, 126.4, 35.6, 30.6$. HRMS (ESI) (m/z): calcd for $\text{C}_9\text{H}_{11}\text{O}_2$ $[\text{M}+\text{H}]^+$: 151.0754, found: 151.0753.



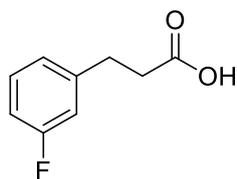
3-(*p*-Tolyl)propanoic acid (2b). White solid, yield: 47 mg (57%), mp 92-93 °C. $R_f = 0.82$ (dichloromethane→ethyl acetate). $^1\text{H NMR}$ (500 MHz, CDCl_3 , ppm) $\delta = 7.13$ (s, 4H), 2.94 (t, $J = 7.8$ Hz, 2H), 2.69 (t, $J = 7.8$ Hz, 2H), 2.34 (s, 3H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3 , ppm) $\delta = 178.5, 137.1, 135.9, 129.2, 128.2, 35.7, 30.2, 21.0$. HRMS (ESI) (m/z): calcd for $\text{C}_{10}\text{H}_{13}\text{O}_2$ $[\text{M}+\text{H}]^+$: 165.0910, found: 165.0907.



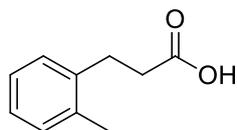
3-(4-Fluorophenyl)propanoic acid (2c). Yellow solid, yield: 62 mg (74%), mp 85-86 °C. $R_f = 0.76$ (dichloromethane→ethyl acetate). $^1\text{H NMR}$ (500 MHz, CDCl_3 , ppm) $\delta = 7.19$ (m, 2H), 7.01 (ddd, $J = 9.6, 5.9, 2.6$ Hz, 2H), 2.96 (t, $J = 7.7$ Hz, 2H), 2.69 (t, $J = 7.7$ Hz, 2H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3 , ppm) $\delta = 179.2, 161.6$ (d, $J = 244.3$ Hz), 135.8 (d, $J = 2.9$ Hz), 129.8 (d, $J = 7.6$ Hz), 115.4 (d, $J = 21.3$ Hz), 35.8, 29.8. HRMS (ESI) (m/z): calcd for $\text{C}_9\text{H}_9\text{FO}_2$ $[\text{M}+\text{H}]^+$: 169.0659, found: 169.0660.



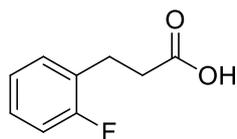
3-(*m*-Tolyl)propanoic acid (2d). Yellow oil, yield: 70 mg (86%). $R_f = 0.83$ (dichloromethane→ethyl acetate). $^1\text{H NMR}$ (500 MHz, CDCl_3 , ppm) $\delta = 7.22$ (t, $J = 7.8$ Hz, 1H), 7.05 (m, 3H), 2.96 (t, $J = 7.9$ Hz, 2H), 2.71 (m, 2H), 2.36 (s, 3H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3 , ppm) $\delta = 178.9, 140.2, 138.2, 129.1, 128.5, 127.1, 125.3, 35.6, 30.5, 21.4$. HRMS (ESI) (m/z): calcd for $\text{C}_{10}\text{H}_{13}\text{O}_2$ $[\text{M}+\text{H}]^+$: 165.0910, found: 165.0908.



3-(3-Fluorophenyl)propanoic acid (2e). Yellow oil, yield: 81 mg (96%). $R_f = 0.73$ (dichloromethane→ethyl acetate). $^1\text{H NMR}$ (500 MHz, CDCl_3 , ppm) $\delta = 7.27$ (dt, $J = 5.6, 3.2$ Hz, 1H), 7.01 (m, 1H), 6.93 (ddd, $J = 8.8, 1.6, 1.1$ Hz, 2H), 2.98 (t, $J = 7.7$ Hz, 2H), 2.71 (t, $J = 7.7$ Hz, 2H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3 , ppm) $\delta = 178.6, 162.9$ (d, $J = 245.7$ Hz), 142.7 (d, $J = 7.4$ Hz), 130.0 (d, $J = 8.3$ Hz), 123.9 (d, $J = 2.8$ Hz), 115.2 (d, $J = 21.2$ Hz), 113.3 (d, $J = 21.1$ Hz), 35.2, 30.2. HRMS (ESI) (m/z): calcd for $\text{C}_9\text{H}_9\text{FO}_2$ $[\text{M}+\text{H}]^+$: 169.0659, found: 169.0659.

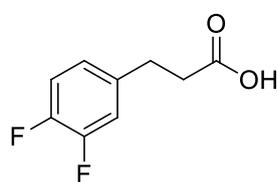


3-(*o*-Tolyl)propanoic acid (2f). Yellow solid, yield: 58 mg (71%), mp 100-101 °C. $R_f = 0.83$ (dichloromethane→ethyl acetate). $^1\text{H NMR}$ (500 MHz, $\text{DMSO}-d_6$, ppm) $\delta = 12.20$ (s, 1H), 7.12 (m, 4H), 2.81 (m, 2H), 2.49 (dd, $J = 9.3, 6.4$ Hz, 2H), 2.27 (s, 3H). $^{13}\text{C NMR}$ (126 MHz, d_6 -DMSO, ppm) $\delta = 174.4, 139.4, 136.1, 130.4, 128.7, 126.5, 126.4, 34.4, 28.2, 19.3$. HRMS (ESI) (m/z): calcd for $\text{C}_{10}\text{H}_{13}\text{O}_2$ $[\text{M}+\text{H}]^+$: 165.0910, found: 165.0907.



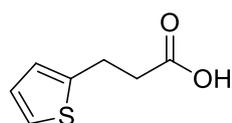
3-(2-Fluorophenyl)propanoic acid (2g). Yellow oil, yield: 77 mg (92%).

$R_f = 0.73$ (dichloromethane→ethyl acetate). $^1\text{H NMR}$ (500 MHz, d_6 -DMSO, ppm) $\delta = 7.23$ (m, 2H), 7.07 (m, 2H), 3.01 (t, $J = 7.8$ Hz, 2H), 2.72 (t, $J = 7.8$ Hz, 2H). $^{13}\text{C NMR}$ (126 MHz, d_6 -DMSO, ppm) $\delta = 178.5$, 161.2 (d, $J = 245.4$ Hz), 130.6 (d, $J = 4.8$ Hz), 128.2 (d, $J = 8.0$ Hz), 127.0 (d, $J = 15.6$ Hz), 124.1 (d, $J = 3.6$ Hz), 115.4 (d, $J = 22.0$ Hz), 34.1 (d, $J = 1.2$ Hz), 24.3 (d, $J = 2.7$ Hz). HRMS (ESI) (m/z): calcd for $\text{C}_9\text{H}_9\text{FO}_2$ $[\text{M}+\text{H}]^+$: 169.0659, found: 169.0653.



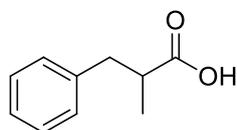
3-(3,4-Difluorophenyl)propanoic acid (2h). Yellow oil, yield: 72 mg

(78%). $R_f = 0.72$ (dichloromethane→ethyl acetate). $^1\text{H NMR}$ (500 MHz, CDCl_3 , ppm) $\delta = 7.09$ (dd, $J = 8.4, 1.9$ Hz, 1H), 7.04 (ddd, $J = 11.1, 7.6, 2.1$ Hz, 1H), 6.94 (m, 1H), 2.93 (t, $J = 7.6$ Hz, 2H), 2.68 (t, $J = 7.6$ Hz, 2H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3 , ppm) $\delta = 178.6$, 150.6 (dd, $J = 142.4, 12.7$ Hz), 148.6 (dd, $J = 141.0, 12.7$ Hz), 137.1 (d, $J = 5.2$ Hz), 124.2 (dd, $J = 6.2, 3.6$ Hz), 117.3 (d, $J = 8.2$ Hz), 117.2 (d, $J = 8.0$ Hz), 35.3, 29.7. HRMS (ESI) (m/z): calcd for $\text{C}_9\text{H}_9\text{F}_2\text{O}_2$ $[\text{M}+\text{H}]^+$: 187.0565, found: 187.0565.



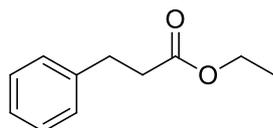
3-(Thiophen-2-yl)propanoic acid (2i). Brown oil, yield: 26 mg (34%). $R_f =$

0.78 (petroleum ether/ethyl acetate = 10:1). $^1\text{H NMR}$ (500 MHz, CDCl_3 , ppm) $\delta = 7.17$ (dd, $J = 5.1, 0.7$ Hz, 1H), 6.95 (dd, $J = 5.1, 3.5$ Hz, 1H), 6.87 (m, 1H), 3.20 (t, $J = 7.6$ Hz, 2H), 2.78 (t, $J = 7.6$ Hz, 2H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3 , ppm) $\delta = 178.6$, 142.7, 126.9, 124.8, 123.7, 35.9, 24.8. HRMS (ESI) (m/z): calcd for $\text{C}_7\text{H}_9\text{O}_2\text{S}$ $[\text{M}+\text{H}]^+$: 157.0317, found: 157.0314.

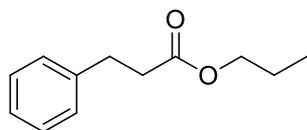


2-Methyl-3-phenylpropanoic acid (2j). Yellow oil, yield: 74 mg (90%). $R_f =$

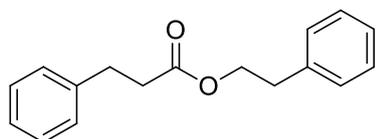
= 0.84 (dichloromethane→ethyl acetate). ^1H NMR (500 MHz, CDCl_3 , ppm) δ = 7.33 (t, J = 7.4 Hz, 2H), 7.27 (m, 1H), 7.23 (d, J = 7.3 Hz, 2H), 3.12 (dd, J = 13.5, 6.4 Hz, 1H), 2.81 (dd, J = 14.2, 7.2 Hz, 1H), 2.71 (dd, J = 13.3, 7.9 Hz, 1H), 1.22 (d, J = 6.9 Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3 , ppm) δ = 182.5, 139.1, 129.0, 128.5, 126.5, 41.3, 39.3, 16.5. HRMS (ESI) (m/z): calcd for $\text{C}_{10}\text{H}_{13}\text{O}_2$ $[\text{M}+\text{H}]^+$: 165.0910, found: 165.0909.



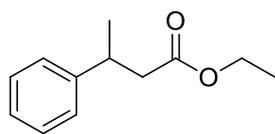
Ethyl 3-phenylpropanoate (2k). Colorless oil, yield: 72 mg (81%). R_f = 0.78 (petroleum ether/ethyl acetate = 15:1). ^1H NMR (500 MHz, CDCl_3 , ppm) δ = 7.33 (m, 2H), 7.24 (m, 3H), 4.17 (q, J = 7.1 Hz, 2H), 3.00 (t, J = 7.8 Hz, 2H), 2.67 (m, 2H), 1.28 (t, J = 7.2 Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3 , ppm) δ = 172.9, 140.6, 128.5, 128.3, 126.3, 60.4, 36.0, 31.0, 14.2. HRMS (ESI) (m/z): calcd for $\text{C}_{11}\text{H}_{15}\text{O}_2$ $[\text{M}+\text{H}]^+$: 179.1066, found: 179.1063.



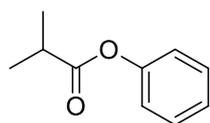
Propyl 3-phenylpropanoate (2l). Yellow oil, yield: 79 mg (82%). R_f = 0.78 (petroleum ether/ethyl acetate = 15:1). ^1H NMR (500 MHz, CDCl_3 , ppm) δ = 7.32 (m, 2H), 7.23 (m, 3H), 4.07 (t, J = 6.7 Hz, 2H), 2.99 (t, J = 7.8 Hz, 2H), 2.67 (m, 2H), 1.66 (dd, J = 14.2, 6.9 Hz, 2H), 0.95 (t, J = 7.4 Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3 , ppm) δ = 173.0, 140.6, 128.5, 128.3, 126.2, 66.1, 35.9, 31.0, 22.0, 10.4. HRMS (ESI) (m/z): calcd for $\text{C}_{12}\text{H}_{17}\text{O}_2$ $[\text{M}+\text{H}]^+$: 193.1223, found: 193.1218.



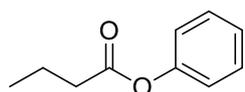
Phenethyl 3-phenylpropanoate (2m). Yellow oil, yield: 109 mg (86%). R_f = 0.74 (petroleum ether/ethyl acetate = 15:1). ^1H NMR (500 MHz, CDCl_3 , ppm) δ = 7.38 (m, 4H), 7.29 (m, 6H), 4.38 (t, J = 7.1 Hz, 2H), 3.00 (dt, J = 9.2, 7.4 Hz, 4H), 2.70 (m, 2H). ^{13}C NMR (126 MHz, CDCl_3 , ppm) δ = 172.9, 140.6, 137.9, 129.0, 128.6, 128.4, 126.6, 126.3, 65.0, 36.0, 35.2, 31.0. HRMS (ESI) (m/z): calcd for $\text{C}_{17}\text{H}_{19}\text{O}_2$ $[\text{M}+\text{H}]^+$: 255.1380, found: 255.1372.



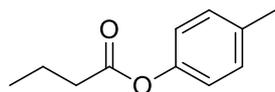
Ethyl 3-phenylbutanoate (2n). Yellow oil, yield: 36 mg (38%). $R_f = 0.78$ (petroleum ether/ethyl acetate = 15:1). $^1\text{H NMR}$ (500 MHz, CDCl_3 , ppm) $\delta = 7.33$ (m, 2H), 7.25 (ddd, $J = 14.5, 7.8, 1.3$ Hz, 3H), 4.12 (q, $J = 7.1$ Hz, 2H), 3.33 (h, $J = 7.1$ Hz, 1H), 2.61 (ddd, $J = 23.2, 15.0, 8.1$ Hz, 2H), 1.35 (d, $J = 7.0$ Hz, 3H), 1.22 (t, $J = 7.1$ Hz, 3H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3 , ppm) $\delta = 172.4, 145.8, 128.5, 126.8, 126.4, 60.2, 43.0, 36.6, 21.8, 14.2$. HRMS (ESI) (m/z): calcd for $\text{C}_{12}\text{H}_{17}\text{O}_2$ $[\text{M}+\text{H}]^+$: 193.1223, found: 193.1217.



Phenyl isobutyrate (2o). Yellow oil, yield: 20 mg (24%). $R_f = 0.77$ (petroleum ether/ethyl acetate = 15:1). $^1\text{H NMR}$ (500 MHz, CDCl_3 , ppm) $\delta = 7.41$ (t, $J = 8.0$ Hz, 2H), 7.25 (m, 1H), 7.11 (m, 2H), 2.84 (dt, $J = 14.0, 7.0$ Hz, 1H), 1.36 (s, 3H), 1.35 (s, 3H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3 , ppm) $\delta = 175.6, 150.9, 129.4, 125.7, 121.5, 34.2, 19.0$. HRMS (ESI) (m/z): calcd for $\text{C}_{10}\text{H}_{13}\text{O}_2$ $[\text{M}+\text{H}]^+$: 165.0910, found: 165.0905.

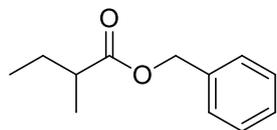


Phenyl butyrate (2p).¹ Yellow oil, yield: 22 mg (27%). $R_f = 0.78$ (petroleum ether/ethyl acetate = 15:1). $^1\text{H NMR}$ (500 MHz, CDCl_3 , ppm) $\delta = 7.40$ (m, 2H), 7.25 (t, $J = 7.4$ Hz, 1H), 7.11 (m, 2H), 2.57 (t, $J = 7.4$ Hz, 2H), 1.83 (dt, $J = 14.8, 7.4$ Hz, 2H), 1.08 (t, $J = 7.4$ Hz, 3H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3 , ppm) $\delta = 172.2, 150.8, 129.4, 125.7, 121.6, 36.3, 18.5, 13.6$.

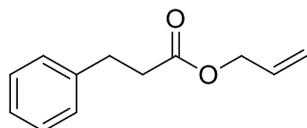


***p*-Tolyl butyrate (2q).**¹ Yellow oil, yield: 77 mg (87%). $R_f = 0.74$ (petroleum ether/ethyl acetate = 15:1). $^1\text{H NMR}$ (500 MHz, CDCl_3 , ppm) $\delta = 7.20$ (d, $J = 8.2$ Hz, 2H), 7.00 (m, 2H), 2.56 (t, $J = 7.4$ Hz, 2H), 2.38 (s, 3H), 1.82 (dt, $J = 14.8, 7.4$ Hz, 2H),

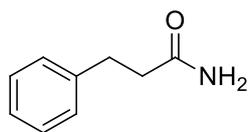
1.08 (t, $J = 7.4$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3 , ppm) $\delta = 172.4, 148.5, 135.3, 129.9, 121.3, 36.2, 20.9, 18.5, 13.7$.



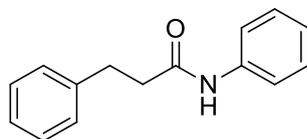
Benzyl 2-methylbutanoate (2r).² Yellow oil, yield: 86 mg (90%). $R_f = 0.75$ (petroleum ether/ethyl acetate = 15:1). ^1H NMR (500 MHz, CDCl_3 , ppm) $\delta = 7.37$ (m, 5H), 5.15 (s, 2H), 2.46 (dd, $J = 13.9, 6.9$ Hz, 1H), 1.73 (m, 1H), 1.52 (m, 1H), 1.20 (d, $J = 7.0$ Hz, 3H), 0.93 (t, $J = 7.5$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3 , ppm) $\delta = 176.6, 136.3, 128.5, 128.1, 128.1, 66.0, 41.1, 26.8, 16.6, 11.6$.



Allyl 3-phenylpropanoate (2p). Yellow oil, yield: 65 mg (68%). $R_f = 0.76$ (petroleum ether/ethyl acetate = 15:1). ^1H NMR (500 MHz, CDCl_3 , ppm) $\delta = 7.32$ (m, 2H), 7.23 (m, 3H), 5.92 (ddt, $J = 16.2, 10.6, 5.7$ Hz, 1H), 5.28 (ddd, $J = 13.8, 11.5, 1.3$ Hz, 2H), 4.61 (d, $J = 5.7$ Hz, 2H), 3.00 (t, $J = 7.8$ Hz, 2H), 2.69 (t, $J = 7.9$ Hz, 2H). ^{13}C NMR (126 MHz, CDCl_3 , ppm) $\delta = 172.6, 140.5, 132.2, 128.5, 128.3, 126.3, 118.2, 65.2, 35.9, 30.9$. HRMS (ESI) (m/z): calcd for $\text{C}_{12}\text{H}_{15}\text{O}_2$ $[\text{M}+\text{H}]^+$: 191.1066, found: 191.1067.

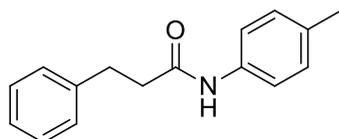


3-Phenylpropanamide (4a). White solid, yield: 67 mg (90%), mp 92-93 $^{\circ}\text{C}$. $R_f = 0.82$ (petroleum ether/ethyl acetate = 10:1). ^1H NMR (500 MHz, CDCl_3 , ppm) $\delta = 7.30$ (dd, $J = 12.1, 4.8$ Hz, 2H), 7.22 (d, $J = 2.7$ Hz, 3H), 6.06 (s, 1H), 5.66 (s, 1H), 2.97 (m, 2H), 2.53 (m, 2H). ^{13}C NMR (126 MHz, CDCl_3 , ppm) $\delta = 174.9, 140.7, 128.6, 128.3, 126.3, 37.5, 31.4$. HRMS (ESI) (m/z): calcd for $\text{C}_9\text{H}_{12}\text{NO}$ $[\text{M}+\text{H}]^+$: 150.0913, found: 150.0908.

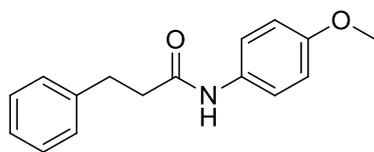


N,3-Diphenylpropanamide (4b). White solid, yield: 65 mg (58%),

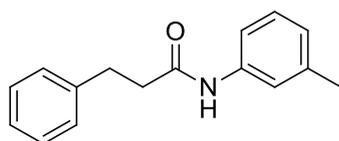
mp 92-93 °C. $R_f = 0.83$ (petroleum ether/ethyl acetate = 10:1). ^1H NMR (500 MHz, CDCl_3 , ppm) $\delta = 8.26$ (s, 1H), 7.54 (d, $J = 7.9$ Hz, 2H), 7.32 (m, 4H), 7.27 (d, $J = 7.2$ Hz, 1H), 7.22 (d, $J = 7.4$ Hz, 2H), 7.14 (t, $J = 7.4$ Hz, 1H), 3.06 (t, $J = 7.8$ Hz, 2H), 2.69 (m, 2H). ^{13}C NMR (126 MHz, CDCl_3 , ppm) $\delta = 171.0, 140.6, 137.9, 129.0, 128.6, 128.4, 126.4, 124.4, 120.2, 39.3, 31.6$. HRMS (ESI) (m/z): calcd for $\text{C}_{15}\text{H}_{16}\text{NO}$ $[\text{M}+\text{H}]^+$: 226.1226, found: 226.1217.



3-Phenyl-*N*-(*p*-tolyl)propanamide (4c). Yellow solid, yield: 71 mg (59%), mp 116-117 °C. $R_f = 0.86$ (petroleum ether/ethyl acetate = 10:1). ^1H NMR (500 MHz, CDCl_3 , ppm) $\delta = 7.52$ (s, 1H), 7.32 (dd, $J = 13.2, 7.6$ Hz, 4H), 7.25 (d, $J = 7.4$ Hz, 3H), 7.11 (d, $J = 8.2$ Hz, 2H), 3.07 (t, $J = 7.6$ Hz, 2H), 2.66 (t, $J = 7.7$ Hz, 2H), 2.32 (s, 3H). ^{13}C NMR (126 MHz, CDCl_3 , ppm) $\delta = 170.3, 140.7, 135.2, 134.0, 129.4, 128.6, 128.4, 126.4, 120.1, 39.4, 31.6, 20.9$. HRMS (ESI) (m/z): calcd for $\text{C}_{16}\text{H}_{18}\text{NO}$ $[\text{M}+\text{H}]^+$: 240.1383, found: 240.1382.

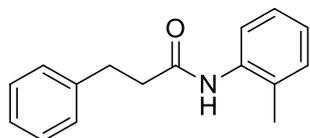


***N*-(4-Methoxyphenyl)-3-phenylpropanamide (4d).** White solid, yield: 68 mg (53%), mp 138-139 °C. $R_f = 0.78$ (petroleum ether/ethyl acetate = 10:1). ^1H NMR (500 MHz, CDCl_3 , ppm) $\delta = 7.54$ (s, 1H), 7.31 (m, 4H), 7.23 (t, $J = 7.3$ Hz, 3H), 6.82 (d, $J = 8.8$ Hz, 2H), 3.78 (s, 3H), 3.04 (t, $J = 7.6$ Hz, 2H), 2.63 (t, $J = 7.7$ Hz, 2H). ^{13}C NMR (126 MHz, CDCl_3 , ppm) $\delta = 170.6, 156.5, 140.7, 130.8, 128.6, 128.4, 126.4, 122.1, 114.1, 55.5, 39.2, 31.7$. HRMS (ESI) (m/z): calcd for $\text{C}_{16}\text{H}_{18}\text{NO}_2$ $[\text{M}+\text{H}]^+$: 256.1332, found: 256.1322.

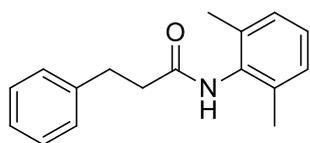


3-Phenyl-*N*-(*m*-tolyl)propanamide (4e). Brown solid, yield: 90 mg (75%), mp 75-76 °C. $R_f = 0.86$ (petroleum ether/ethyl acetate = 10:1). ^1H NMR (500 MHz, CDCl_3 , ppm) $\delta = 7.57$ (s, 1H), 7.35 (s, 1H), 7.31 (t, $J = 7.4$ Hz, 2H), 7.24 (t, $J = 7.1$ Hz, 4H), 7.19 (t, $J = 7.8$ Hz, 1H), 6.94 (d, $J = 7.4$ Hz, 1H), 3.06 (t, $J = 7.7$ Hz, 2H), 2.66 (t, $J = 7.7$ Hz, 2H), 2.32 (s, 3H). ^{13}C NMR (126 MHz, CDCl_3 , ppm) $\delta = 170.7, 140.7, 138.9, 137.8, 128.8,$

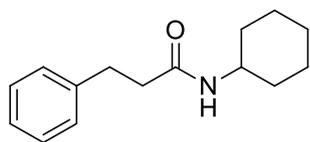
128.6, 128.4, 126.4, 125.2, 120.8, 117.2, 39.3, 31.6, 21.5. HRMS (ESI) (m/z): calcd for $C_{16}H_{18}NO$ $[M+H]^+$: 240.1383, found: 240.1380.



3-Phenyl-N-(o-tolyl)propanamide (4f). White solid, yield: 106 mg (89%), mp 129-130 °C. R_f = 0.88 (petroleum ether/ethyl acetate = 10:1). 1H NMR (500 MHz, $CDCl_3$, ppm) δ = 7.64 (d, J = 7.9 Hz, 1H), 7.32 (d, J = 7.0 Hz, 2H), 7.26 (d, J = 7.0 Hz, 4H), 7.16 (m, 2H), 7.08 (t, J = 7.3 Hz, 1H), 3.06 (t, J = 7.5 Hz, 2H), 2.70 (t, J = 7.6 Hz, 2H), 2.08 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$, ppm) δ = 170.8, 140.7, 135.6, 130.5, 130.0, 128.7, 128.5, 126.6, 126.4, 125.4, 123.9, 39.0, 31.8, 17.6. HRMS (ESI) (m/z): calcd for $C_{16}H_{18}NO$ $[M+H]^+$: 240.1383, found: 240.1381.

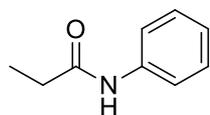


N-(2,6-Dimethylphenyl)-3-phenylpropanamide (4g). White solid, yield: 101 mg (80%), mp 149-150 °C. R_f = 0.83 (petroleum ether/ethyl acetate = 10:1). 1H NMR (500 MHz, $CDCl_3$, ppm) δ = 7.37 (s, 1H), 7.31 (t, J = 7.3 Hz, 2H), 7.23 (t, J = 7.0 Hz, 3H), 7.05 (dd, J = 8.3, 6.6 Hz, 1H), 6.99 (d, J = 7.5 Hz, 2H), 3.01 (t, J = 7.6 Hz, 2H), 2.65 (t, J = 7.6 Hz, 2H), 2.04 (s, 6H). ^{13}C NMR (126 MHz, $CDCl_3$, ppm) δ = 171.0, 140.8, 135.4, 134.0, 128.5, 128.5, 128.5, 128.0, 127.1, 126.3, 37.8, 31.7, 18.3. HRMS (ESI) (m/z): calcd for $C_{17}H_{20}NO$ $[M+H]^+$: 254.1539, found: 254.1538.



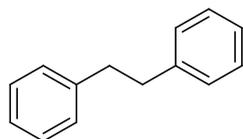
N-Cyclohexyl-3-phenylpropanamide (4h). White solid, yield: 46 mg (40%), mp 115-116 °C. R_f = 0.84 (petroleum ether/ethyl acetate = 10:1). 1H NMR (500 MHz, $CDCl_3$, ppm) δ = 7.28 (m, 2H), 7.20 (dd, J = 5.1, 2.8 Hz, 3H), 5.51 (d, J = 6.8 Hz, 1H), 3.74 (ttd, J = 12.0, 8.1, 3.9 Hz, 1H), 2.96 (t, J = 7.7 Hz, 2H), 2.44 (m, 2H), 1.83 (m, 2H), 1.66 (m, 2H), 1.59 (m, 1H), 1.33 (m, 2H), 1.13 (ddd, J = 12.4, 8.0, 3.5 Hz, 1H), 1.04 (dt, J = 11.8, 8.8 Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$, ppm) δ = 171.2, 141.0, 128.5, 128.4, 126.2, 48.1, 38.7, 33.1,

31.9, 25.5, 24.8. HRMS (ESI) (m/z): calcd for C₁₅H₂₂NO [M+H]⁺: 232.1695, found: 232.1685.



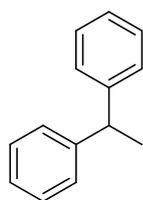
N-Phenylpropionamide (4i). Brown solid, yield: 19 mg (25%), mp 104-105

°C. R_f = 0.77 (petroleum ether/ethyl acetate = 10:1). ¹H NMR (500 MHz, CDCl₃, ppm) δ = 7.90 (s, 1H), 7.55 (d, *J* = 7.7 Hz, 2H), 7.30 (t, *J* = 7.9 Hz, 2H), 7.10 (t, *J* = 7.4 Hz, 1H), 2.40 (q, *J* = 7.6 Hz, 2H), 1.24 (t, *J* = 7.6 Hz, 3H). ¹³C NMR (126 MHz, CDCl₃, ppm) δ = 172.6, 138.1, 128.9, 124.2, 120.1, 30.6, 9.8. HRMS (ESI) (m/z): calcd for C₉H₁₂NO [M+H]⁺: 150.0913, found: 151.0908.



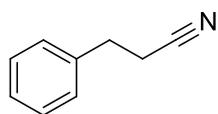
1,2-Diphenylethane (6a). Yellow oil, yield: 86 mg (94%). R_f = 0.94

(petroleum ether). ¹H NMR (500 MHz, CDCl₃, ppm) δ = 7.34 (t, *J* = 7.5 Hz, 4H), 7.25 (t, *J* = 7.5 Hz, 6H), 2.98 (s, 4H). ¹³C NMR (126 MHz, CDCl₃, ppm) δ = 141.8, 128.5, 128.4, 126.0, 38.0. HRMS (ESI) (m/z): calcd for C₁₄H₁₅ [M+H]⁺: 183.1168, found: 183.1174.



Ethane-1,1-diylidibenzene (6b). Yellow oil, yield: 70 mg (77%). R_f = 0.93

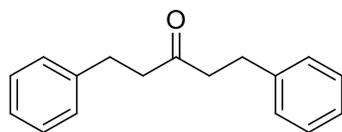
(petroleum ether). ¹H NMR (500 MHz, CDCl₃, ppm) δ = 7.35 (t, *J* = 7.5 Hz, 4H), 7.29 (m, 4H), 7.24 (m, 2H), 4.22 (q, *J* = 7.2 Hz, 1H), 1.71 (d, *J* = 7.2 Hz, 3H). ¹³C NMR (126 MHz, CDCl₃, ppm) δ = 146.4, 128.4, 127.7, 126.1, 44.8, 21.9. HRMS (ESI) (m/z): calcd for C₁₄H₁₅ [M+H]⁺: 183.1168, found: 183.1161.



3-Phenylpropanenitrile (6c). Yellow oil, yield: 62 mg (95%). R_f = 0.82

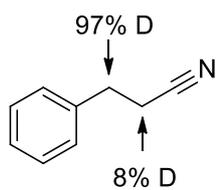
(petroleum ether/ethyl acetate = 10:1). ¹H NMR (500 MHz, CDCl₃, ppm) δ = 7.33 (m, 5H),

2.98 (t, $J = 7.3$ Hz, 2H), 2.63 (t, $J = 7.4$ Hz, 2H). ^{13}C NMR (126 MHz, CDCl_3 , ppm) $\delta = 138.2$, 128.9, 128.3, 127.3, 119.3, 31.6, 19.4. HRMS (ESI) (m/z): calcd for $\text{C}_9\text{H}_{10}\text{N}$ $[\text{M}+\text{H}]^+$: 132.0808, found: 132.0804.



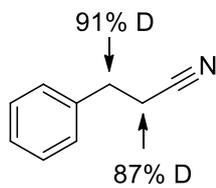
1,5-Diphenylpentan-3-one (6e). Yellow oil, yield: 55 mg (46%).

$R_f = 0.74$ (petroleum ether/ethyl acetate = 10:1). ^1H NMR (500 MHz, CDCl_3 , ppm) $\delta = 7.29$ (d, $J = 6.8$ Hz, 4H), 7.20 (m, 6H), 2.91 (t, $J = 7.6$ Hz, 4H), 2.74 (t, $J = 7.6$ Hz, 4H). ^{13}C NMR (126 MHz, CDCl_3 , ppm) $\delta = 209.2$, 141.0, 128.5, 128.3, 126.1, 44.5, 29.7. HRMS (ESI) (m/z): calcd for $\text{C}_{17}\text{H}_{19}\text{O}$ $[\text{M}+\text{H}]^+$: 239.1430, found: 239.1420.



3-Phenylpropanenitrile- d_1 (6c- d_1). Yellow oil, yield: 24 mg (92%). $R_f =$

0.82 (petroleum ether/ethyl acetate = 10:1). ^1H NMR (500 MHz, CDCl_3 , ppm) $\delta = 7.37$ (m, 2H), 7.30 (m, 1H), 7.26 (d, $J=7.1$, 2H), 2.98 (d, $J=6.7$, 2H), 2.65 (t, $J=7.4$, 1H). ^{13}C NMR (126 MHz, CDCl_3 , ppm) $\delta = 138.0$, 128.9, 128.3, 127.3, 119.1, 31.5 (m), 19.4. HRMS (ESI) (m/z): calcd for $\text{C}_9\text{H}_9\text{DN}$ $[\text{M}+\text{H}]^+$: 133.0870, found: 133.0872.



3-Phenylpropanenitrile- d_2 (6c- d_2). Yellow oil, yield: 20 mg (74%). $R_f = 0.82$

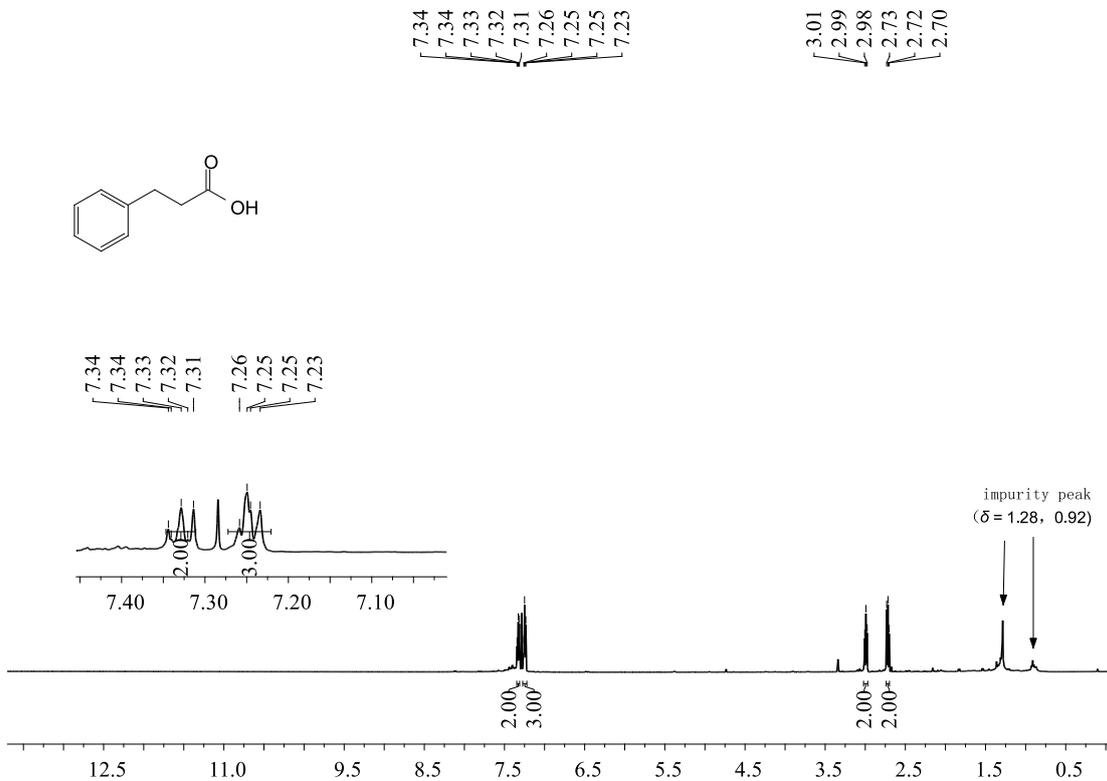
(petroleum ether/ethyl acetate = 10:1). ^1H NMR (500 MHz, CDCl_3 , ppm) $\delta = 7.34$ (m, 2H), 7.27 (m, 1H), 7.23 (m, 2H), 2.93 (m, 1H), 2.58 (m, 1H). ^{13}C NMR (126 MHz, CDCl_3 , ppm) $\delta = 138.1$, 128.9, 128.3, 127.3, 119.2, 31.3 (m), 19.2 (m). HRMS (ESI) (m/z): calcd for $\text{C}_9\text{H}_8\text{D}_2\text{N}$ $[\text{M}+\text{H}]^+$: 134.0933, found: 134.0931.

References

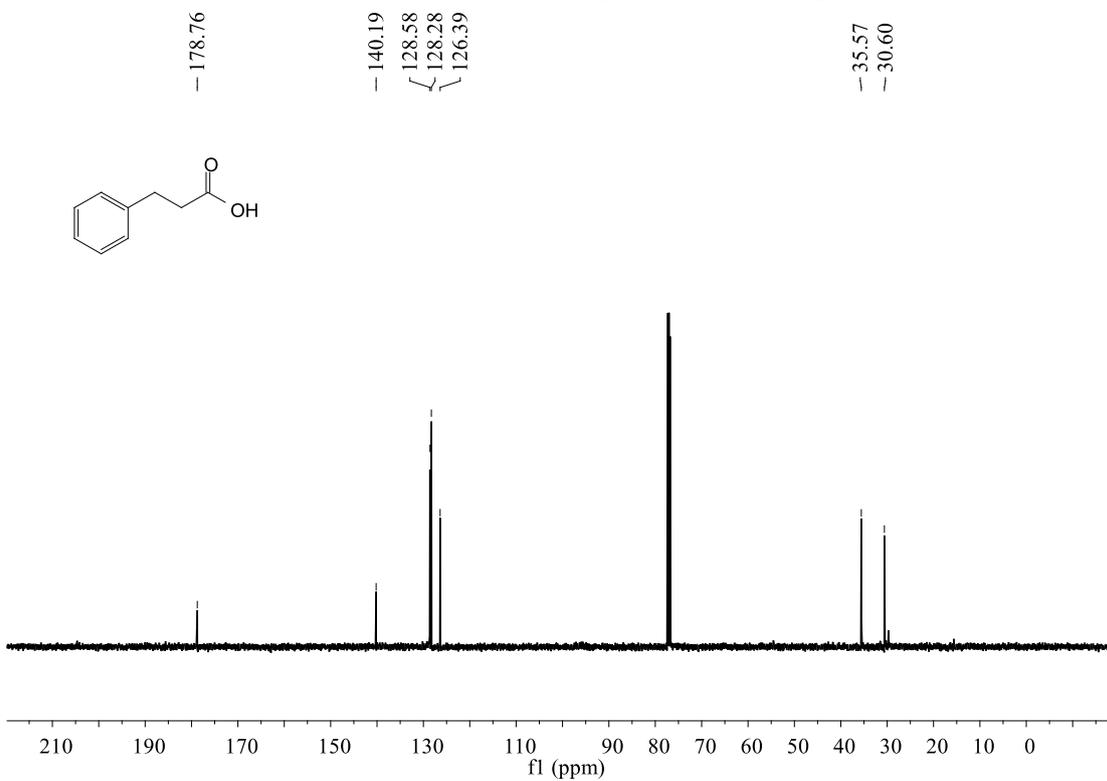
- (1) R. Murashige, Y. Hayashi, S. Ohmori, A. Torii, Y. Aizu, Y. Muto, Y. Murai, Y. Oda, M. Hashimoto, Comparisons of *O*-Acylation and FriedeleCrafts Acylation of Phenols and Acyl Chlorides and Fries Rearrangement of Phenyl Esters in Trifluoromethanesulfonic Acid: Effective Synthesis of Optically Active Homotyrosines. *Tetrahedron* 2011, **67**, 641.
- (2) J.-Q. Li, X. Quan, P. G. Andersson, Highly Enantioselective Iridium-Catalyzed Hydrogenation of α,β -Unsaturated Esters. *Chem. Eur. J.* 2012, **18**, 10609.

H. NMR Spectra

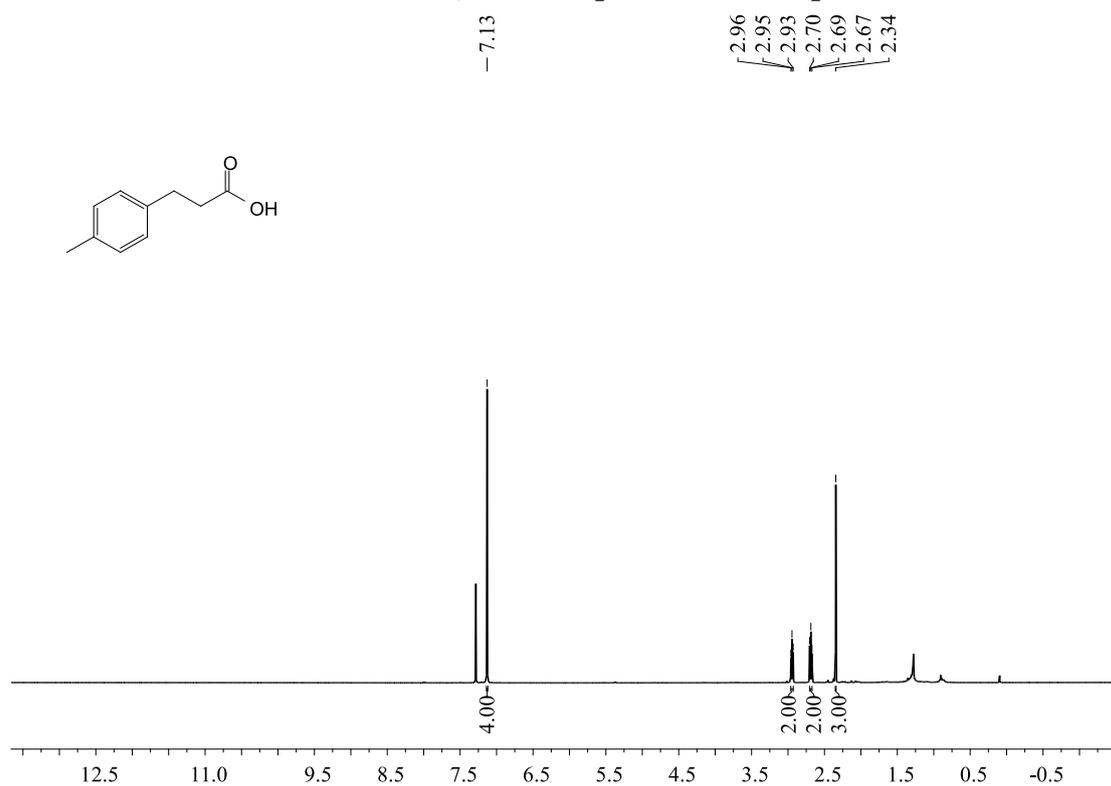
^1H NMR (500 MHz, CDCl_3) spectrum of compound 2a



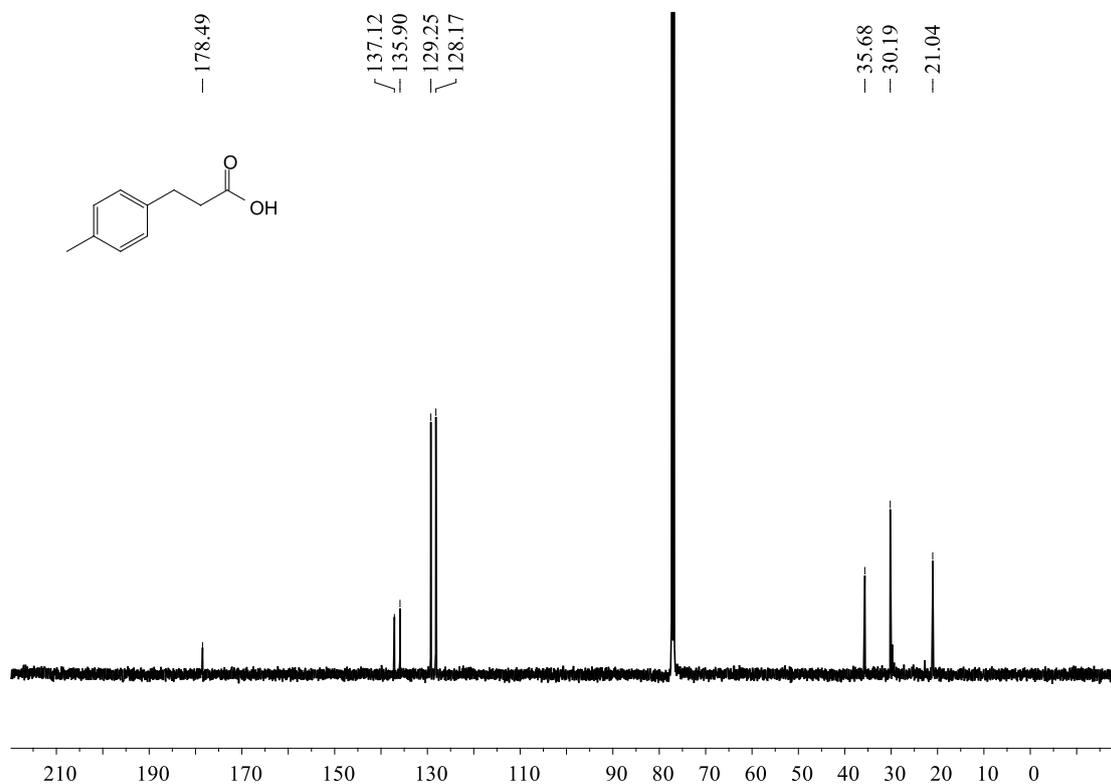
^{13}C NMR (126 MHz, CDCl_3) spectrum of compound 2a



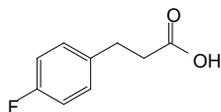
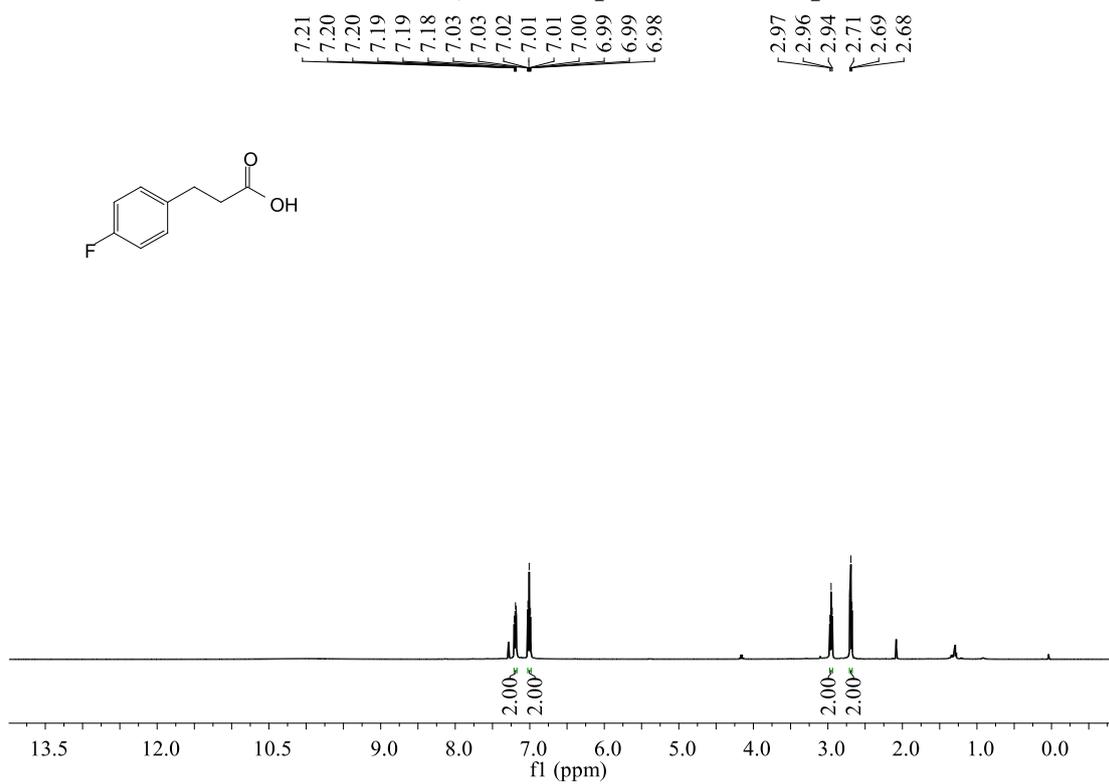
¹H NMR (500 MHz, CDCl₃) spectrum of compound 2b



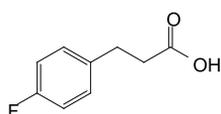
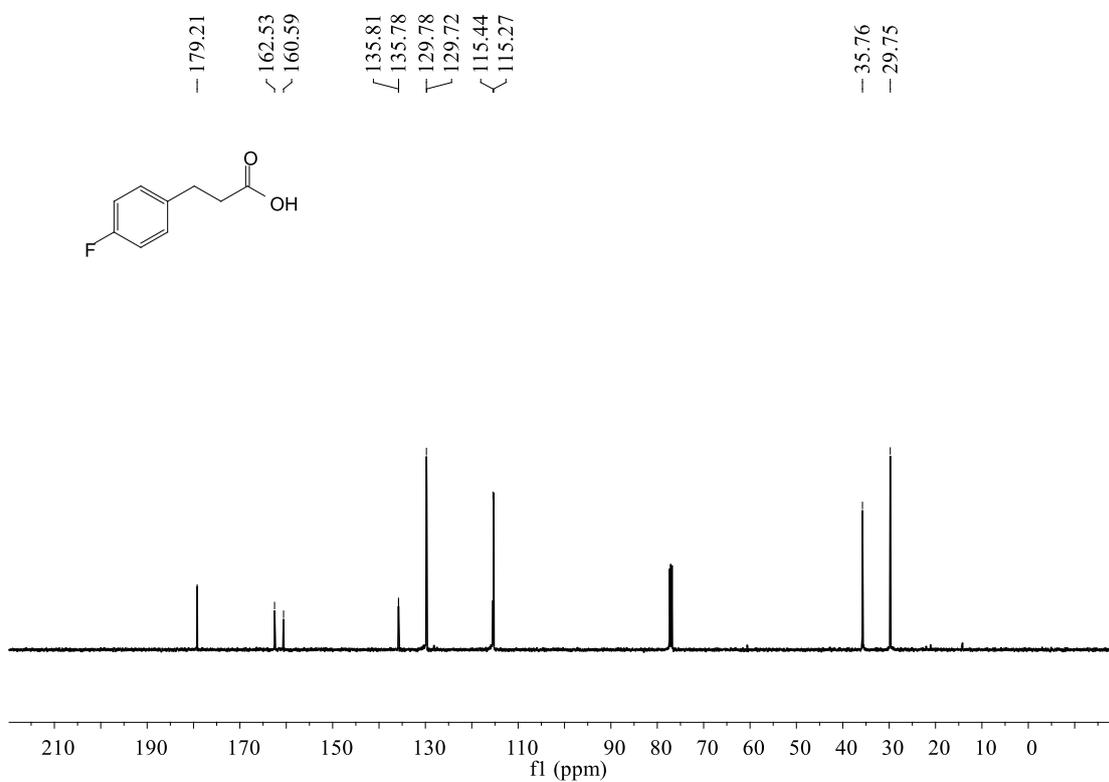
¹³C NMR (126 MHz, CDCl₃) spectrum of compound 2b



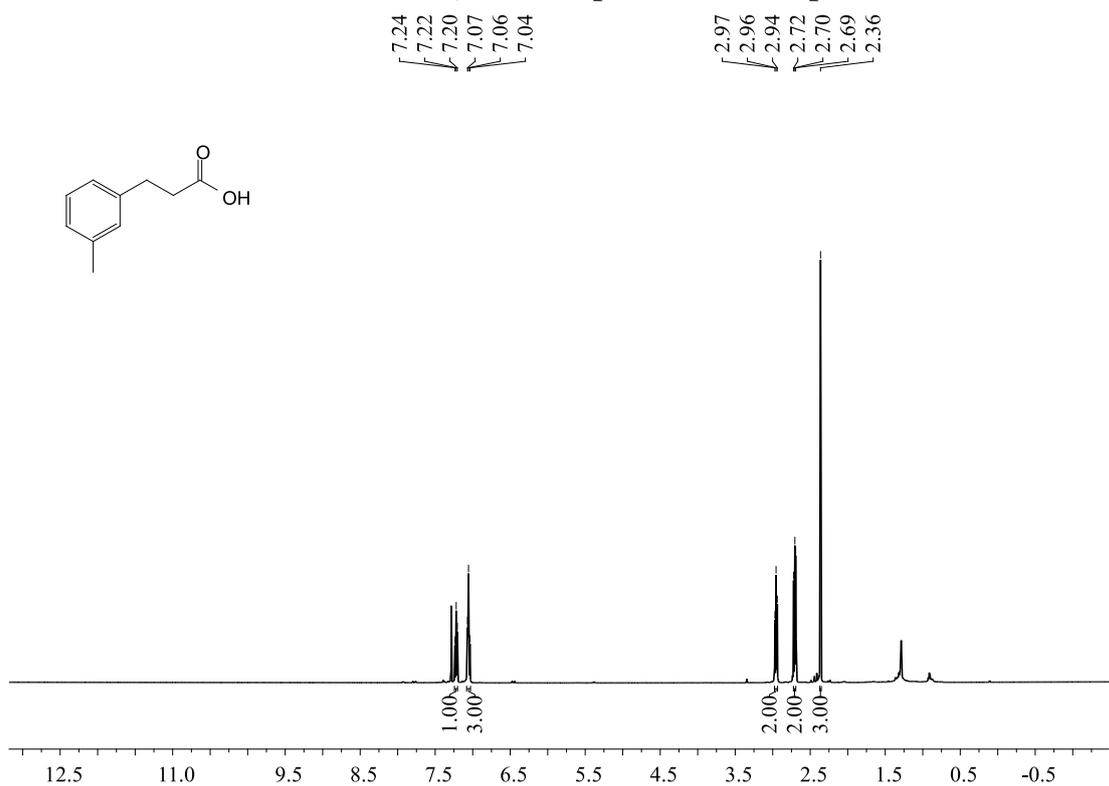
¹H NMR (500 MHz, CDCl₃) spectrum of compound 2c



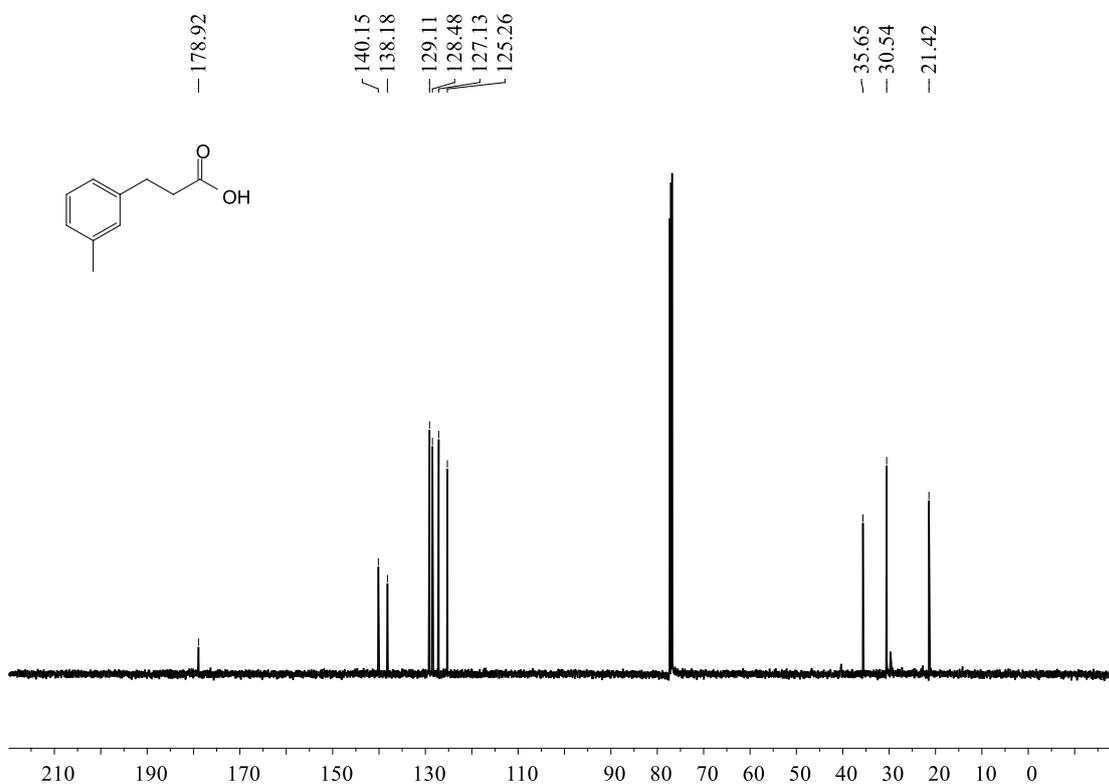
¹³C NMR (126 MHz, CDCl₃) spectrum of compound 2c



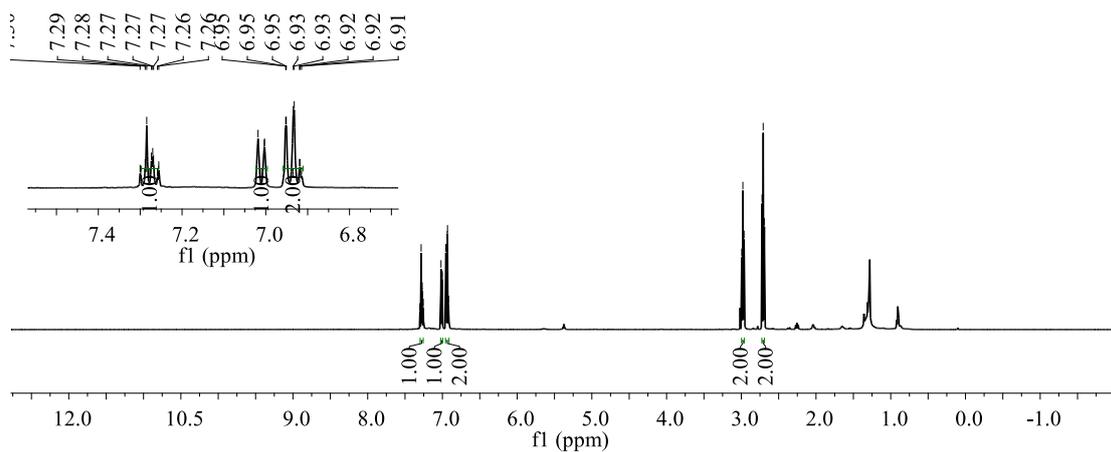
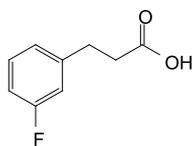
¹H NMR (500 MHz, CDCl₃) spectrum of compound 2d



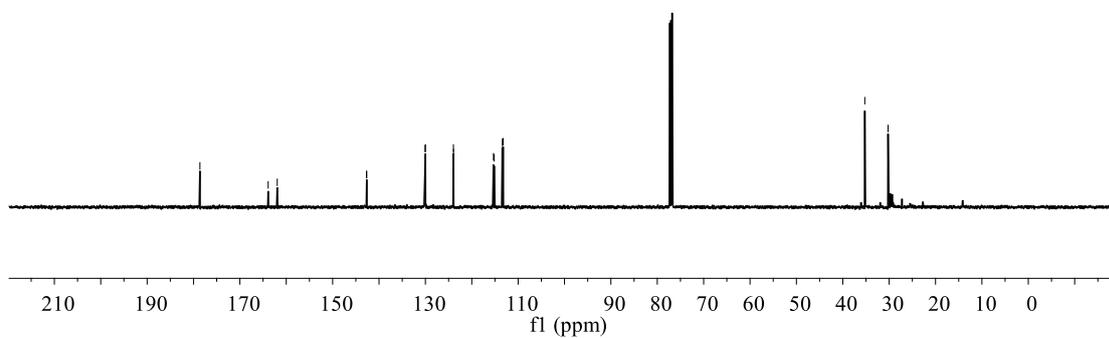
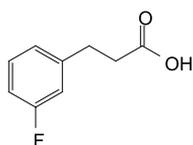
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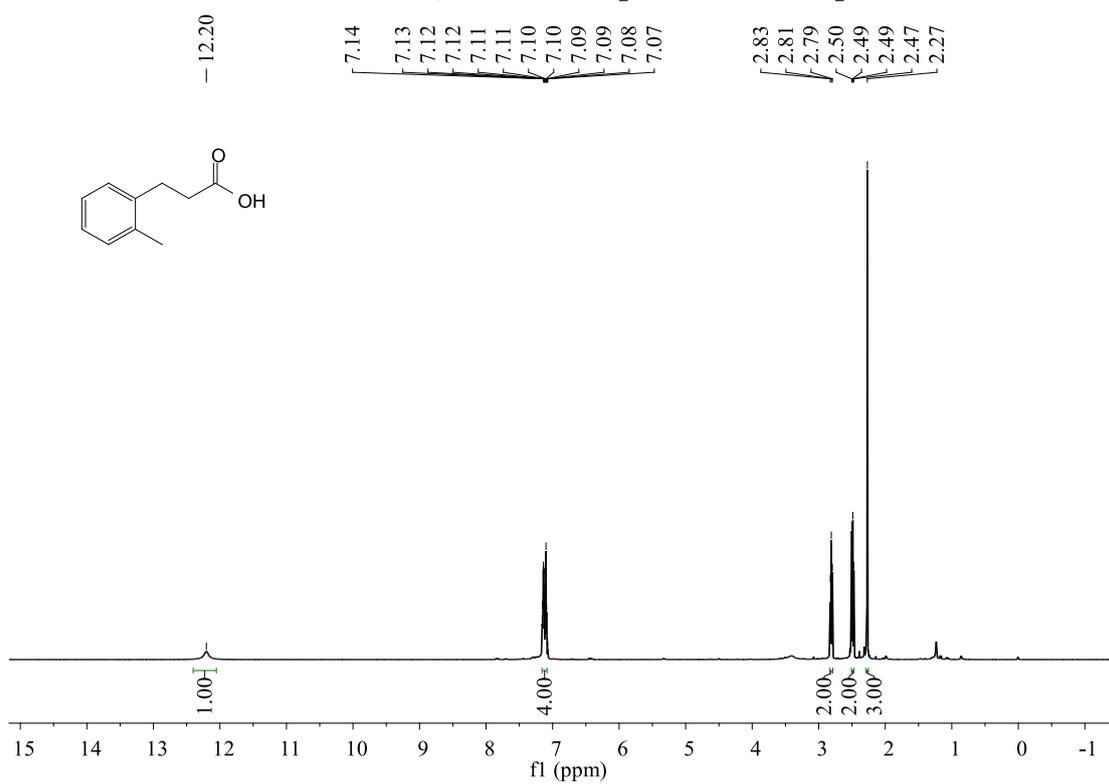
¹H NMR (500 MHz, CDCl₃) spectrum of compound 2e



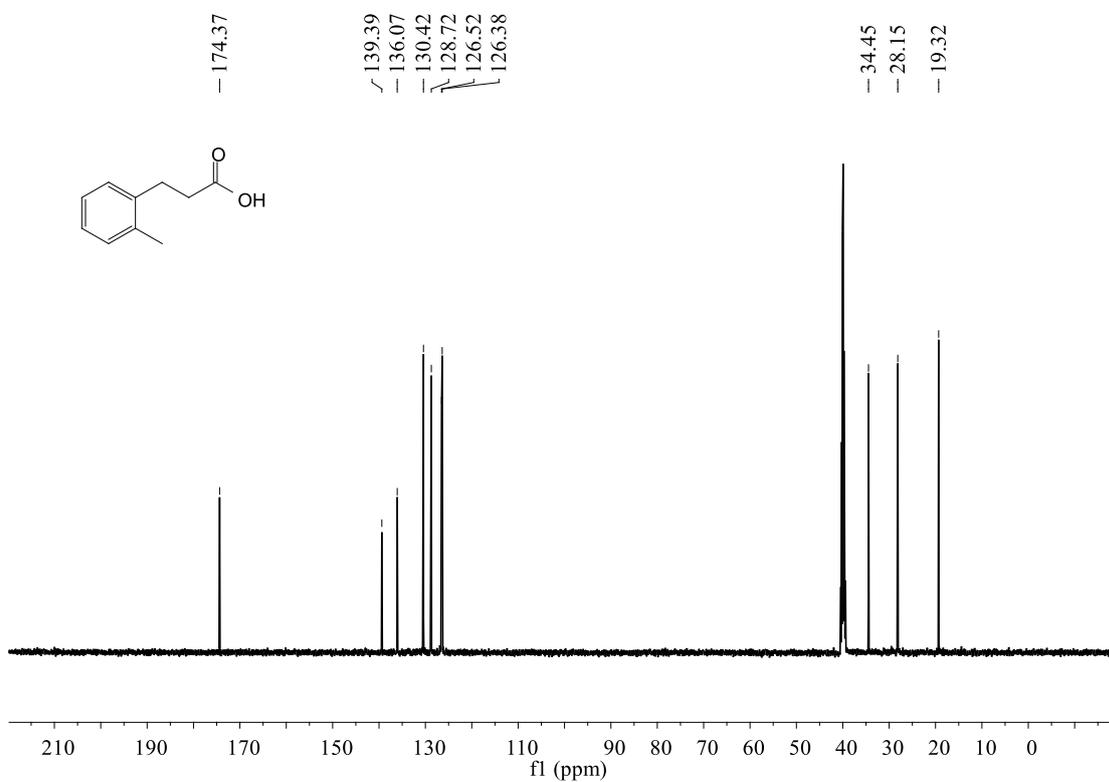
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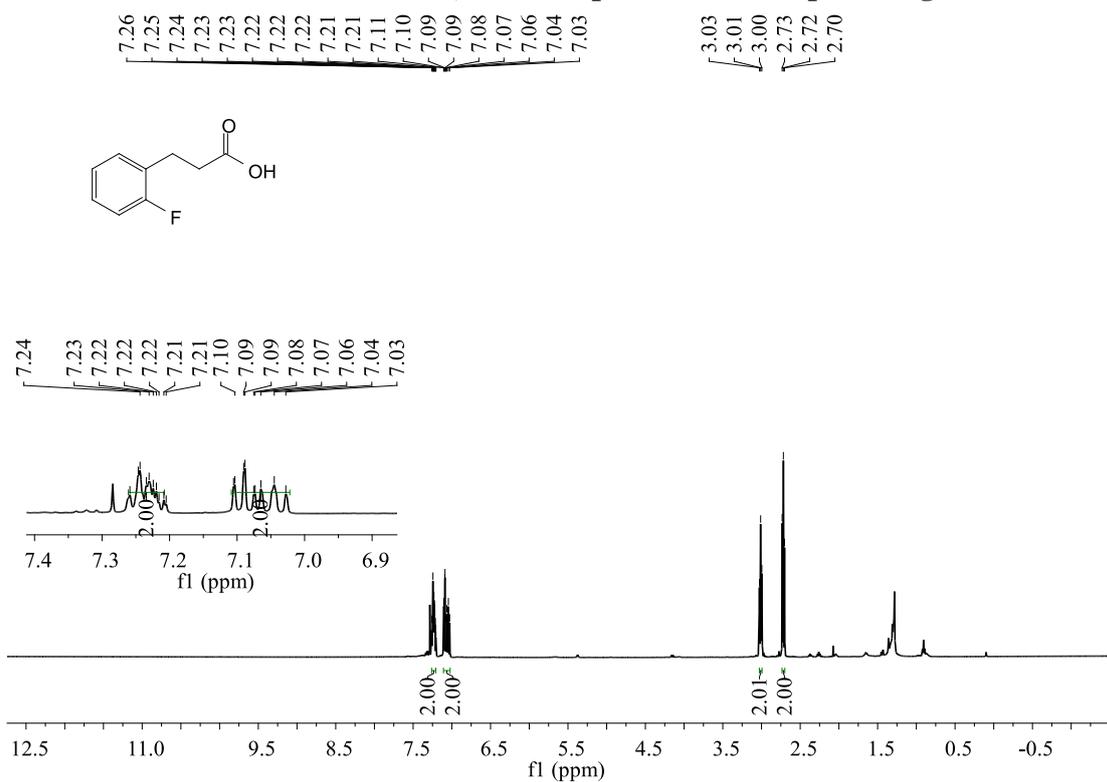
^1H NMR (500 MHz, $\text{DMSO-}d_6$) spectrum of compound 2f



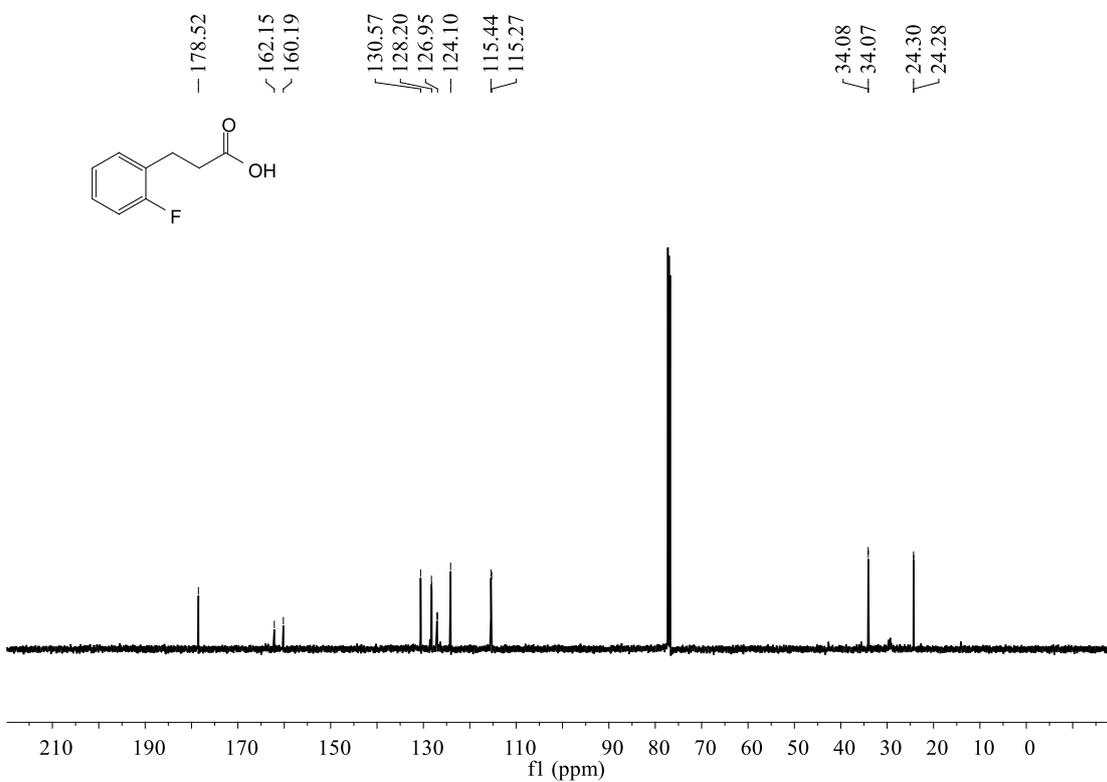
^{13}C NMR (126 MHz, $\text{DMSO-}d_6$) spectrum of compound 2f



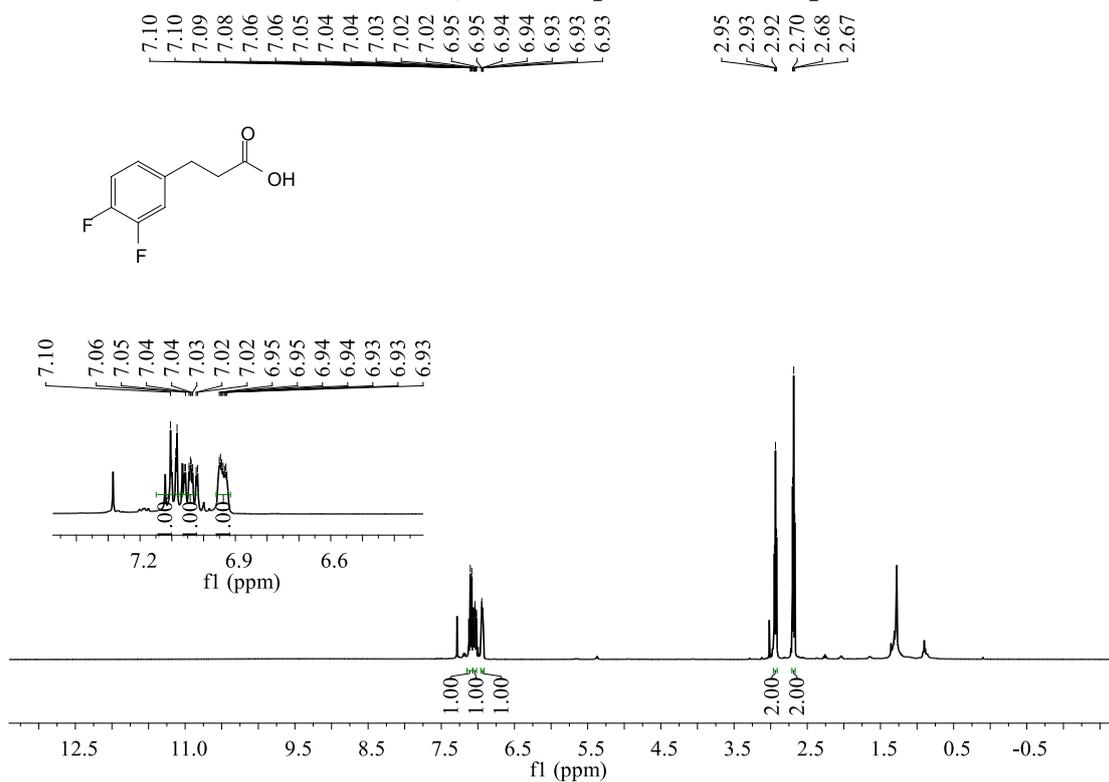
¹H NMR (500 MHz, CDCl₃) spectrum of compound 2g



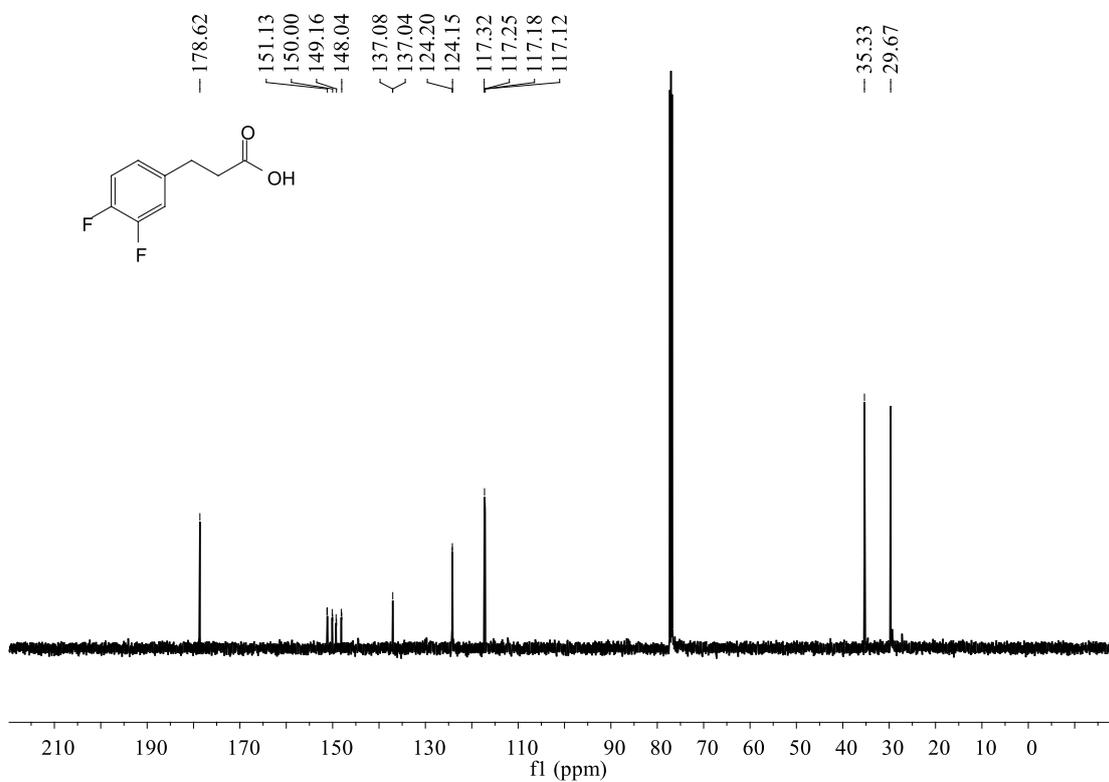
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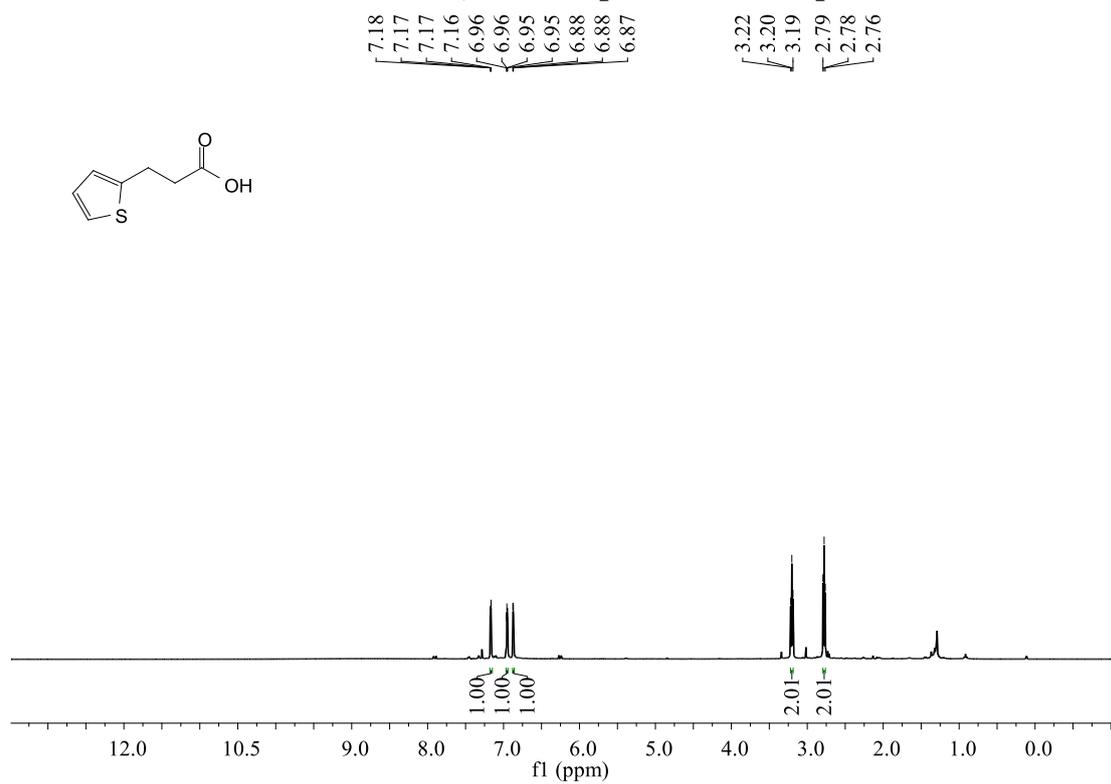
¹H NMR (500 MHz, CDCl₃) spectrum of compound 2h



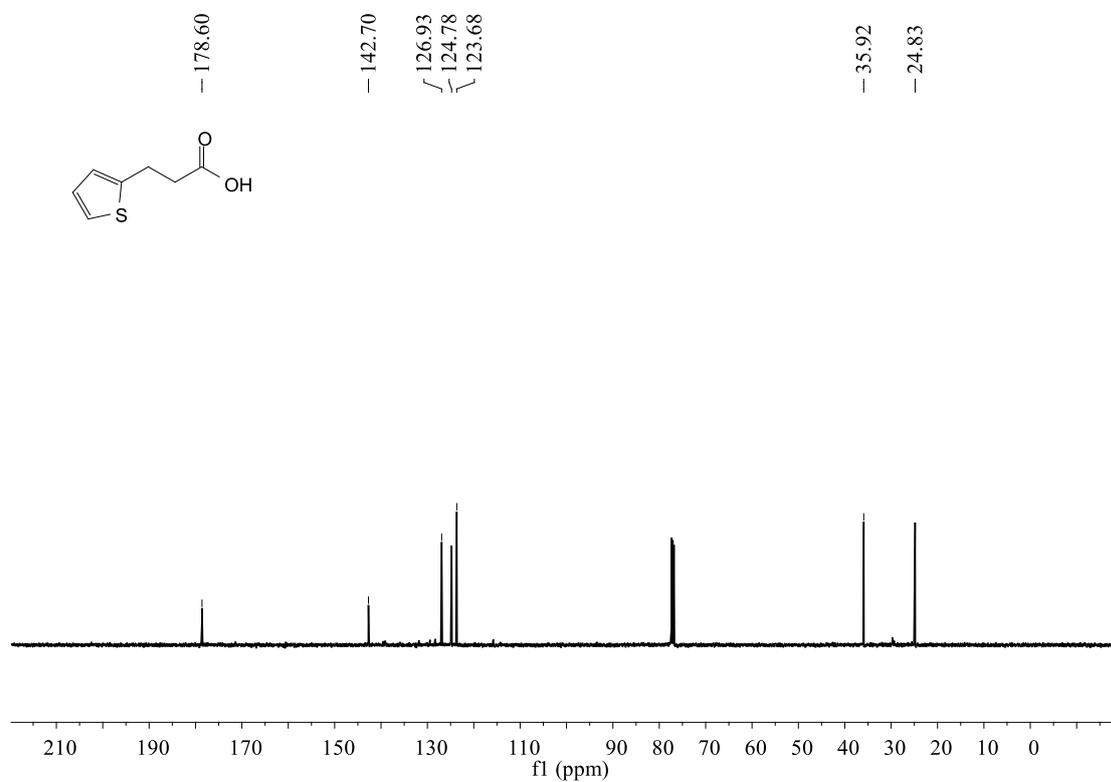
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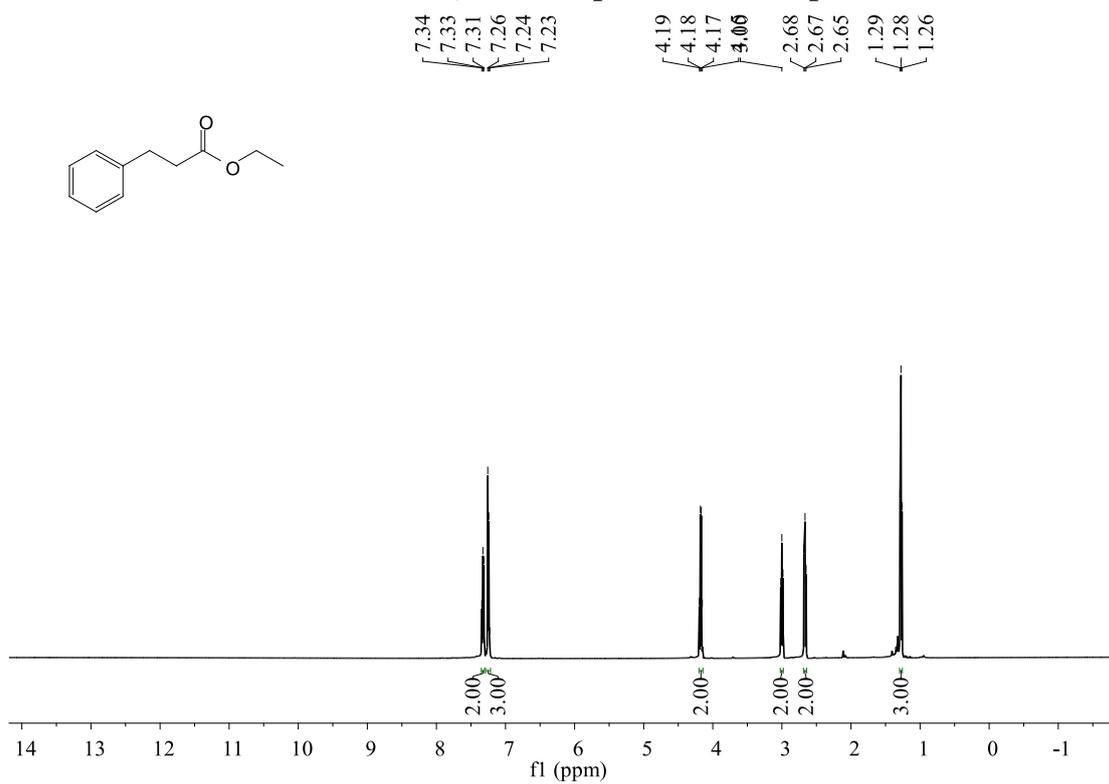
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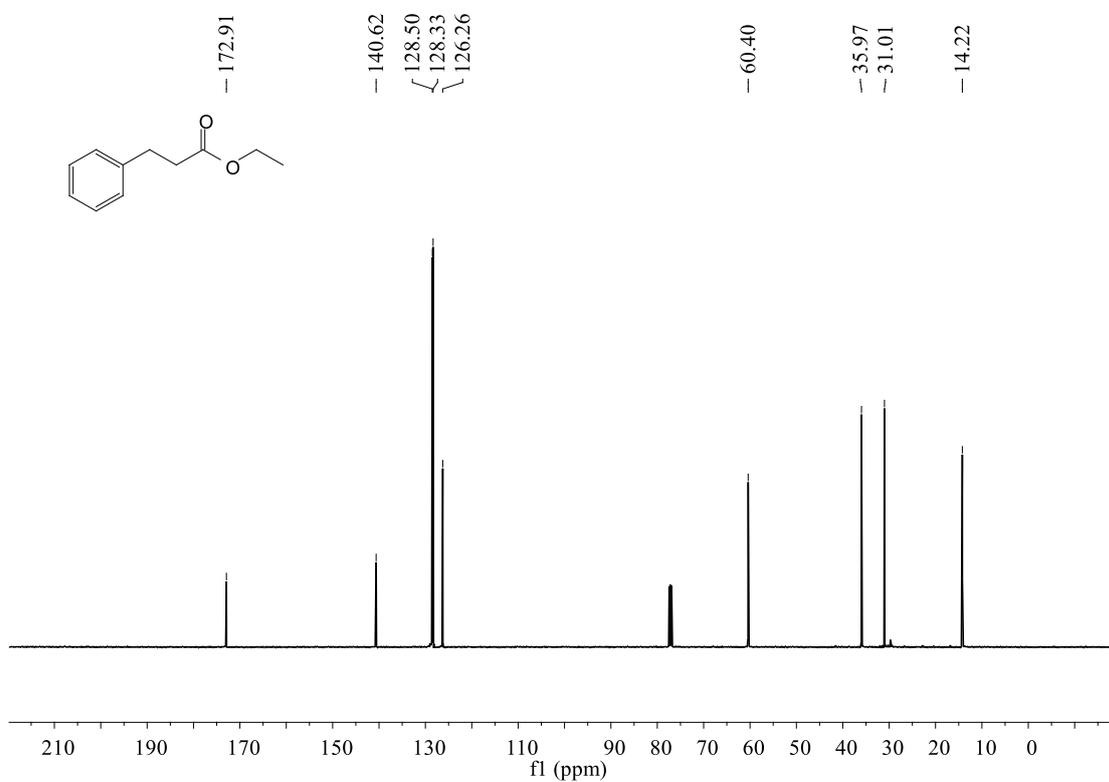
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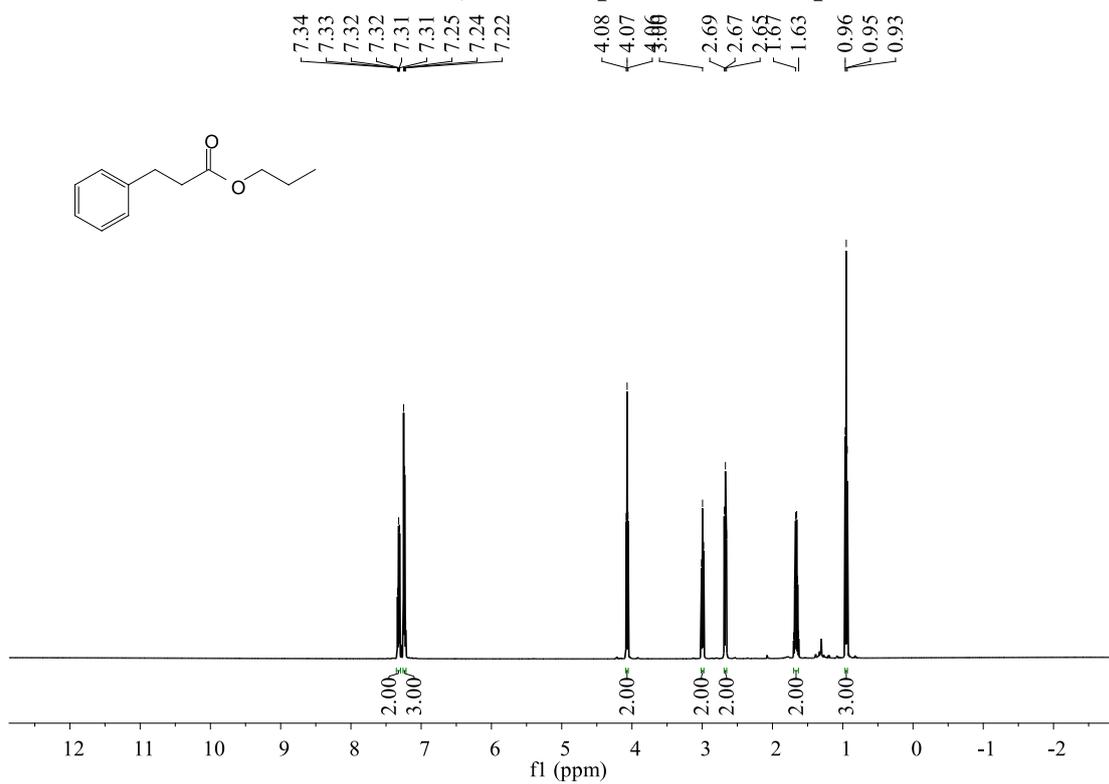
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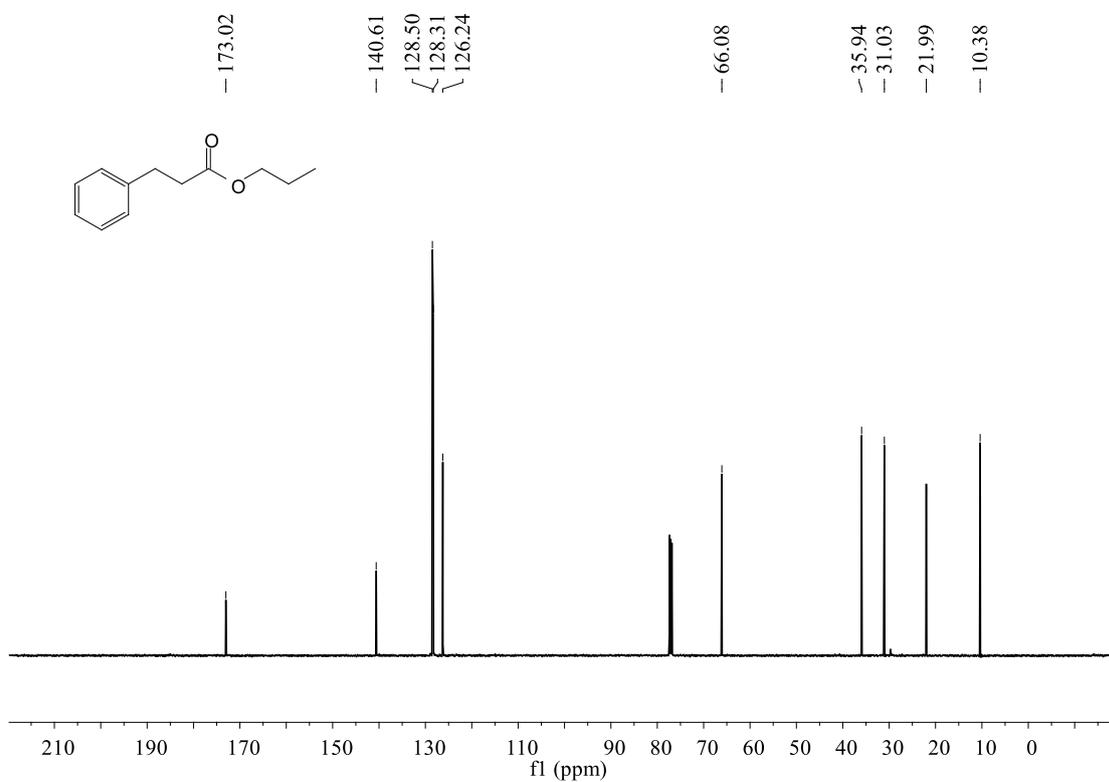
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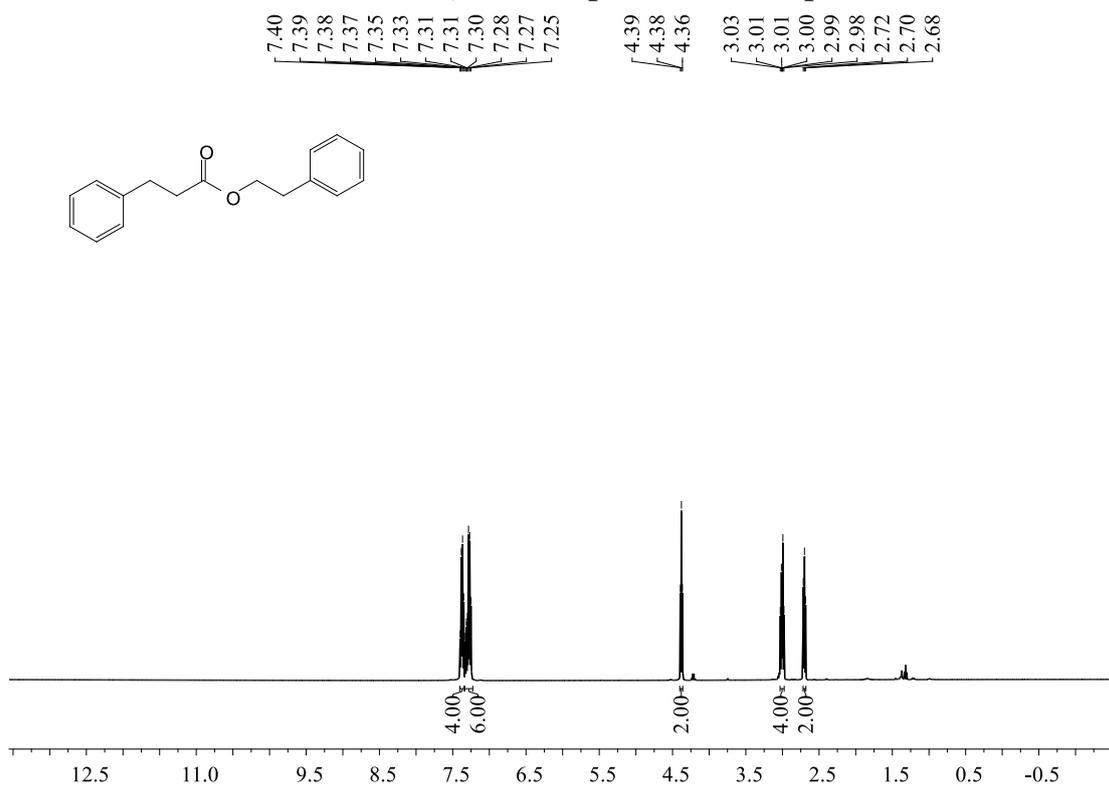
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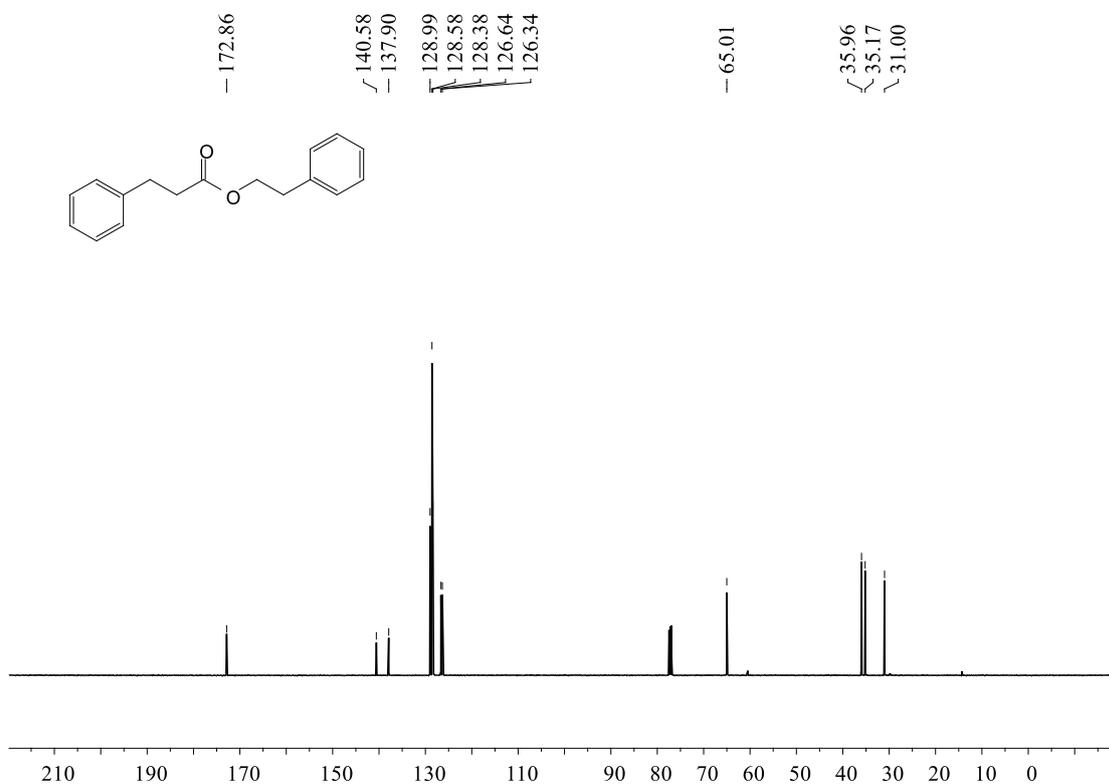
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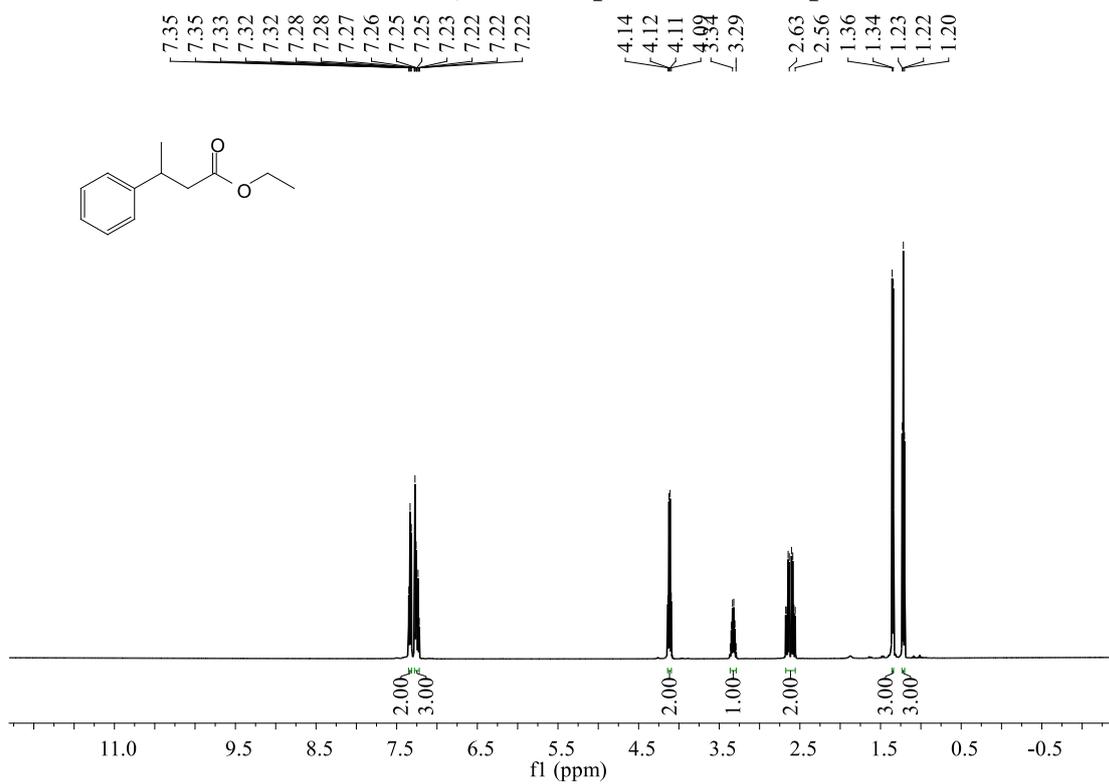
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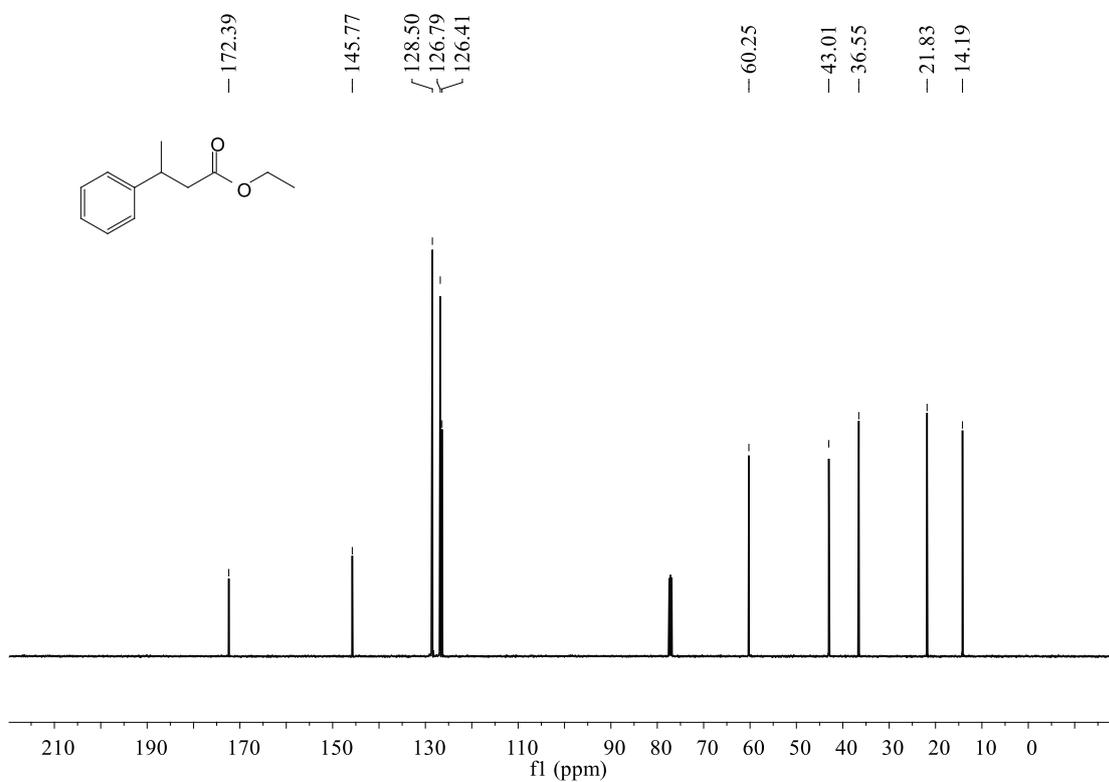
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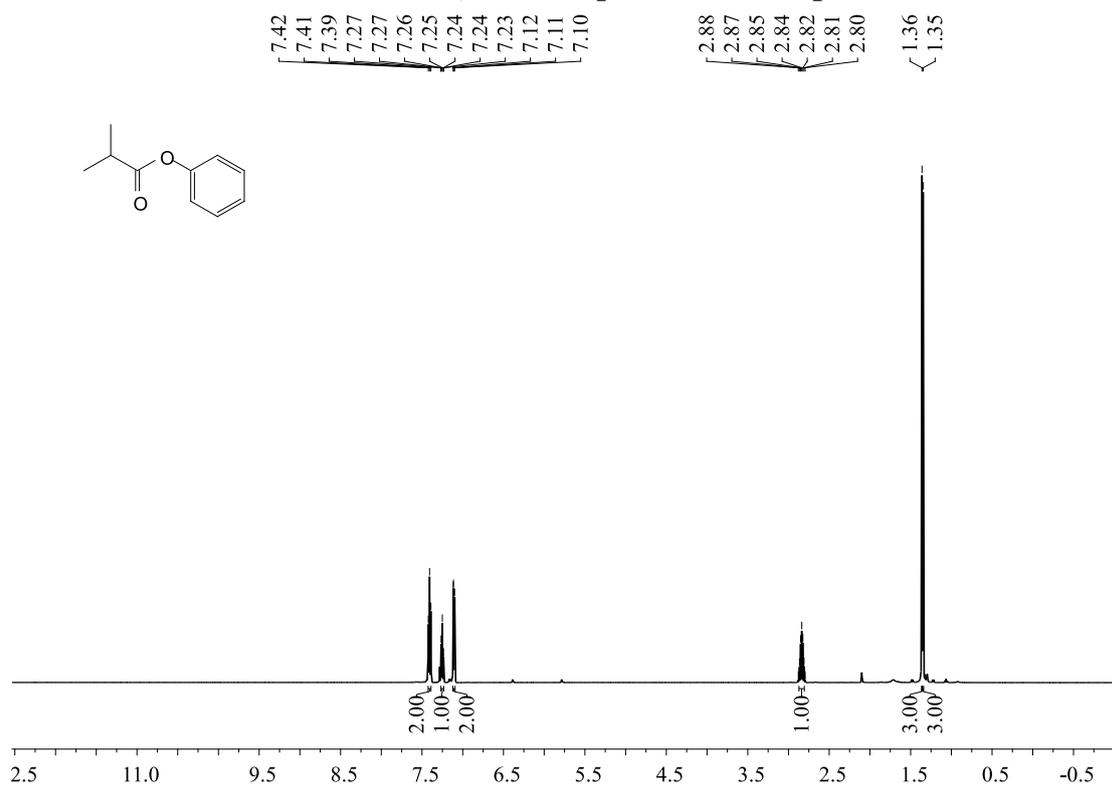
¹H NMR (500 MHz, CDCl₃) spectrum of compound 2n



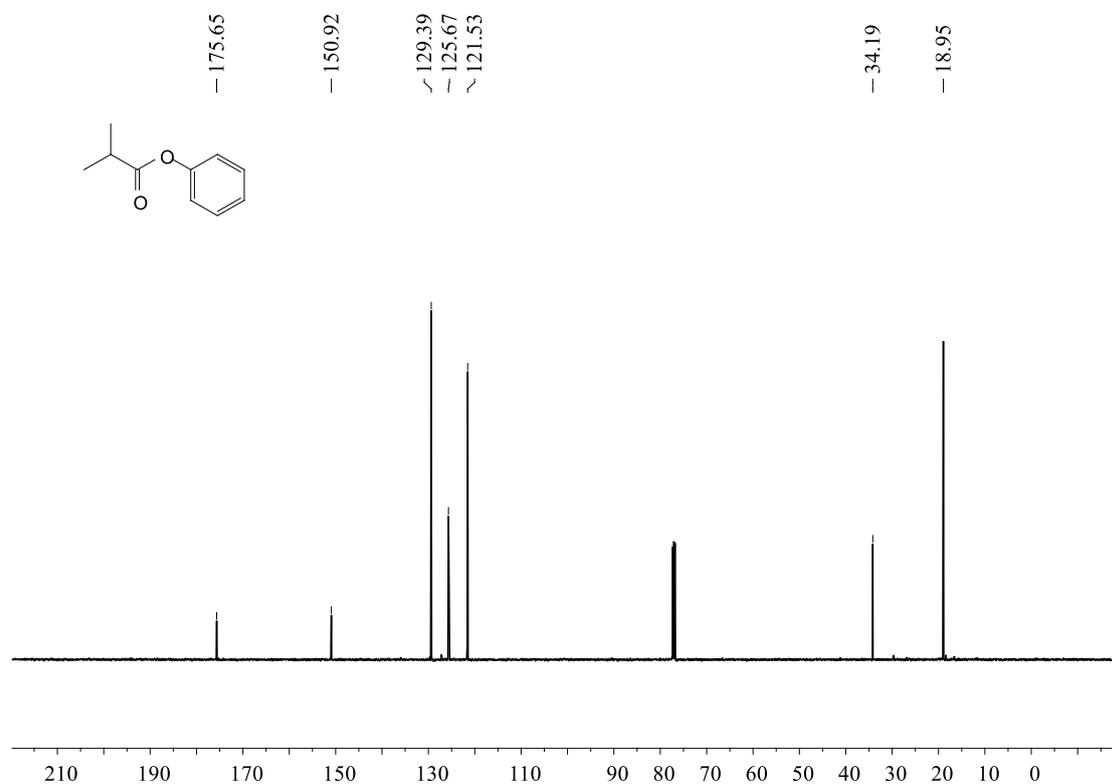
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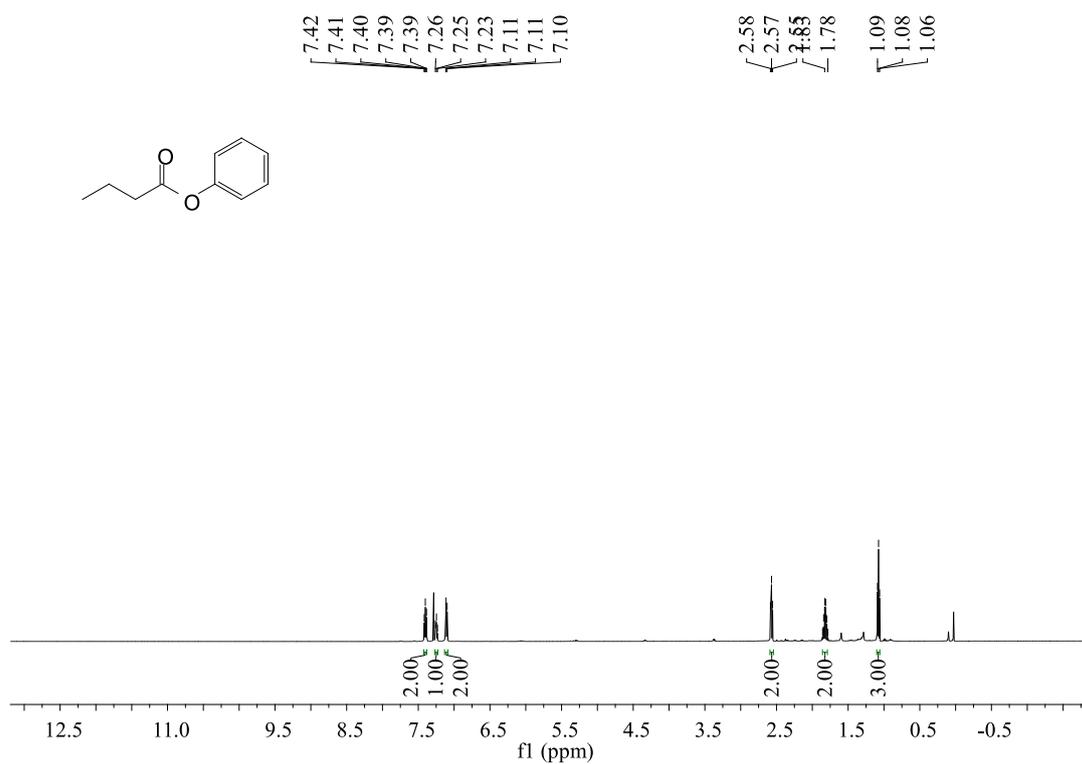
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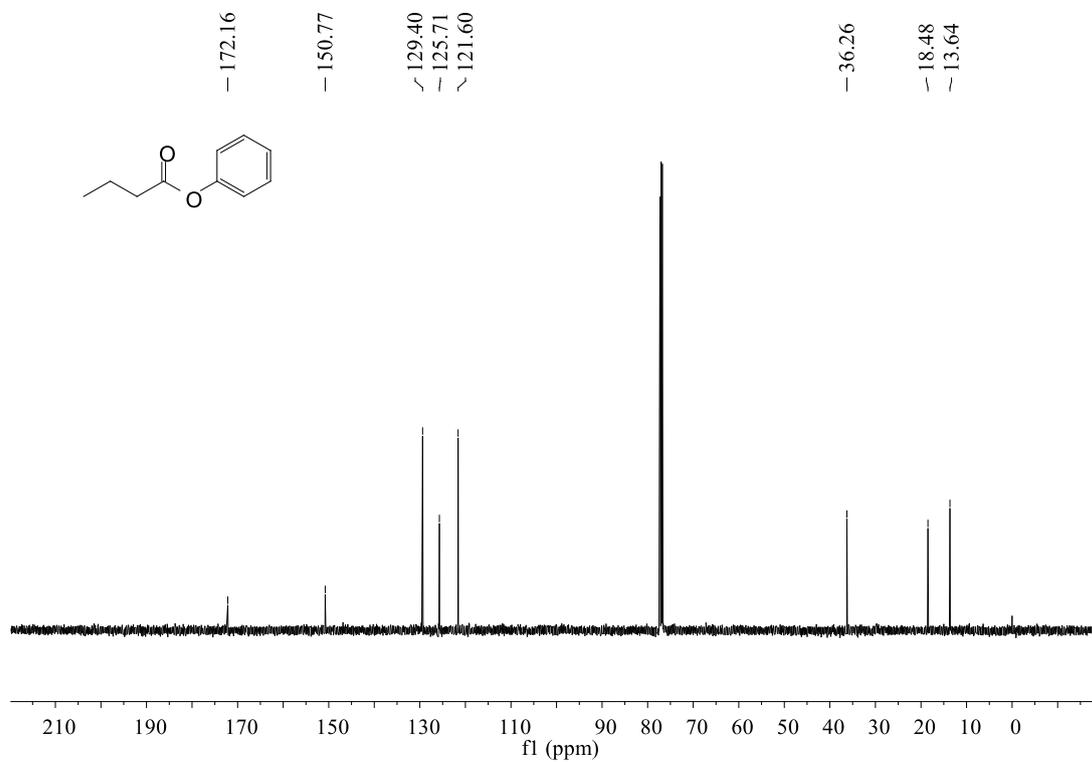
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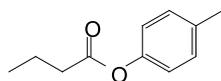
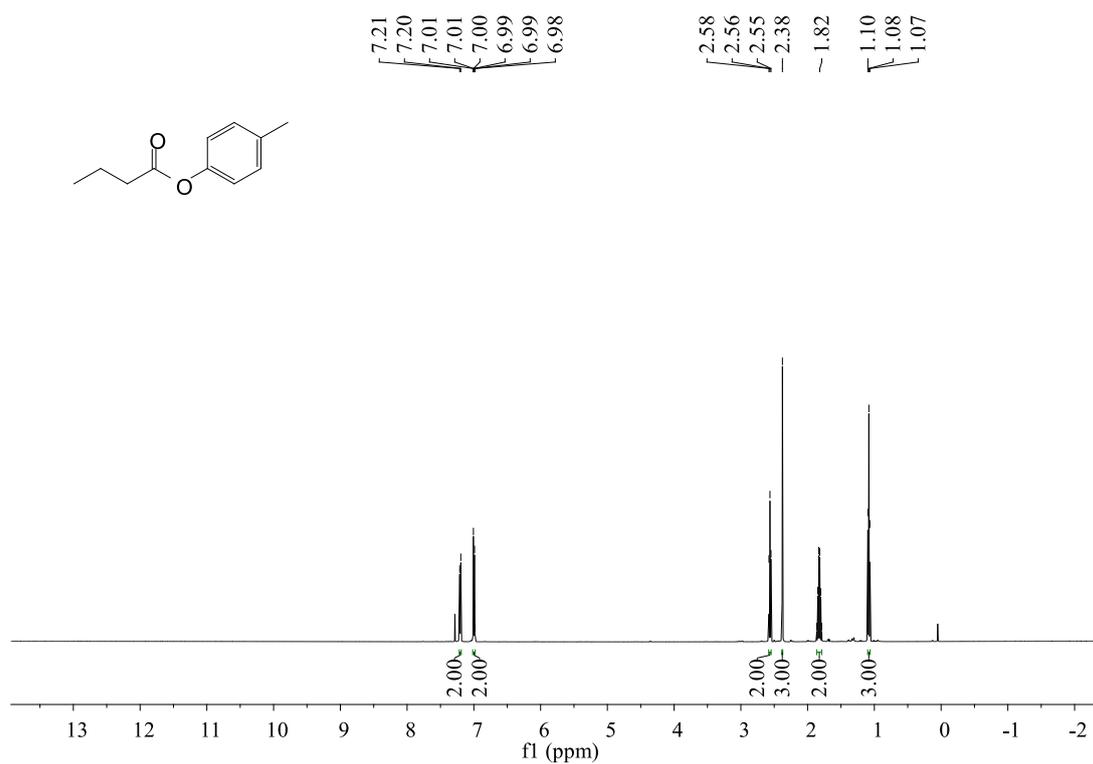
¹H NMR (500 MHz, CDCl₃) spectrum of compound 2p



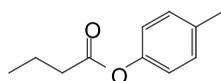
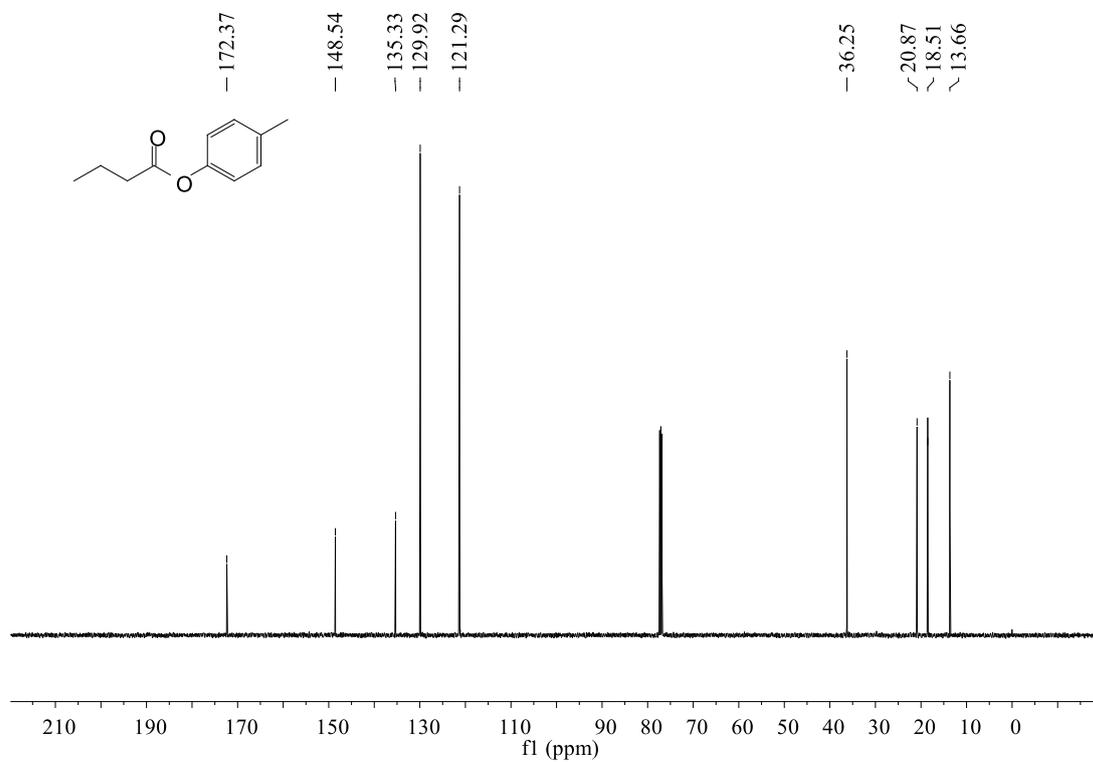
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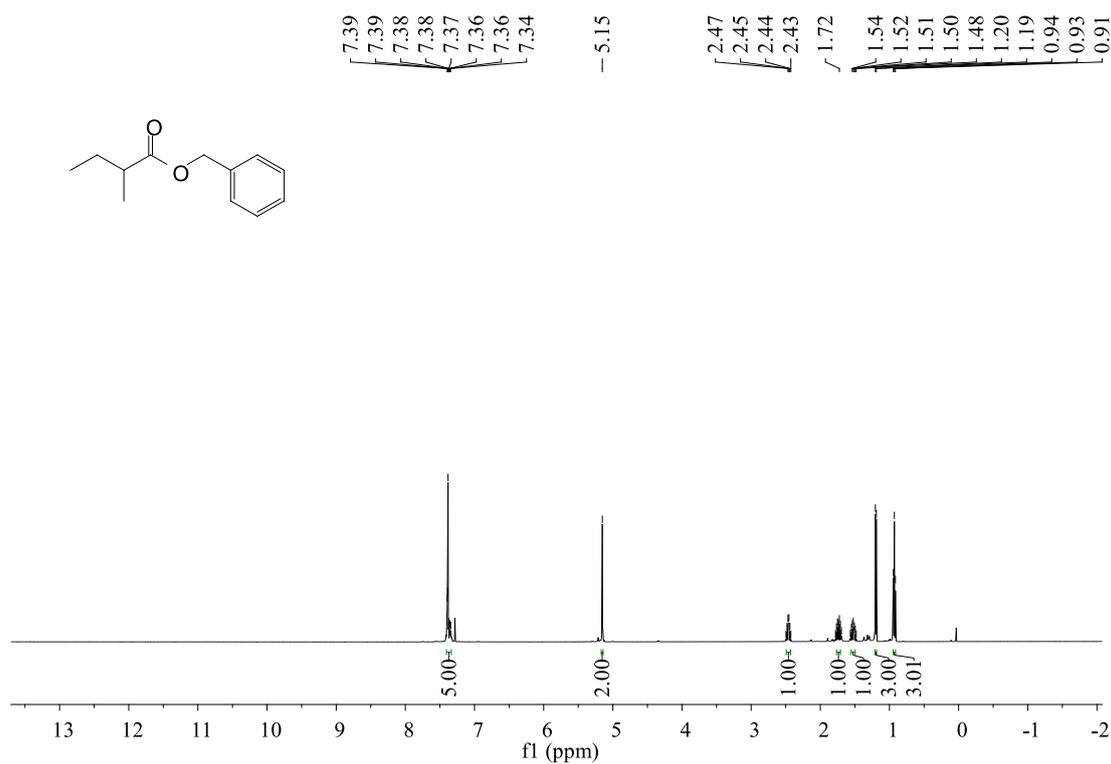
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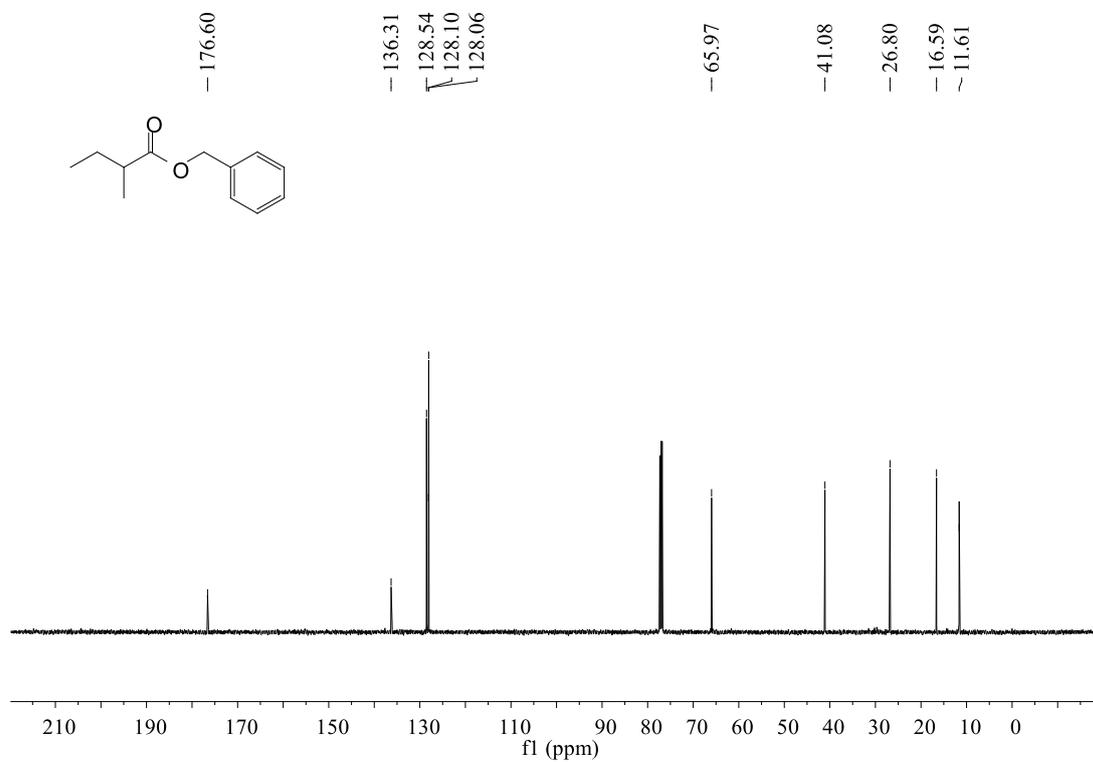
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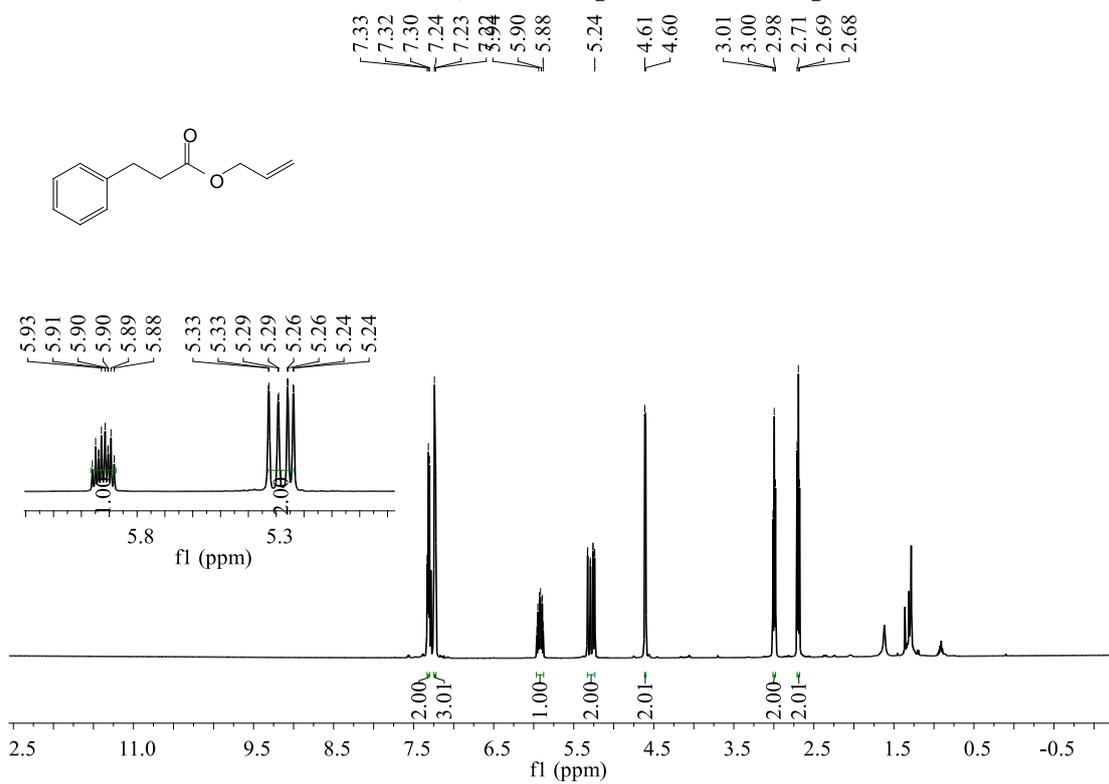
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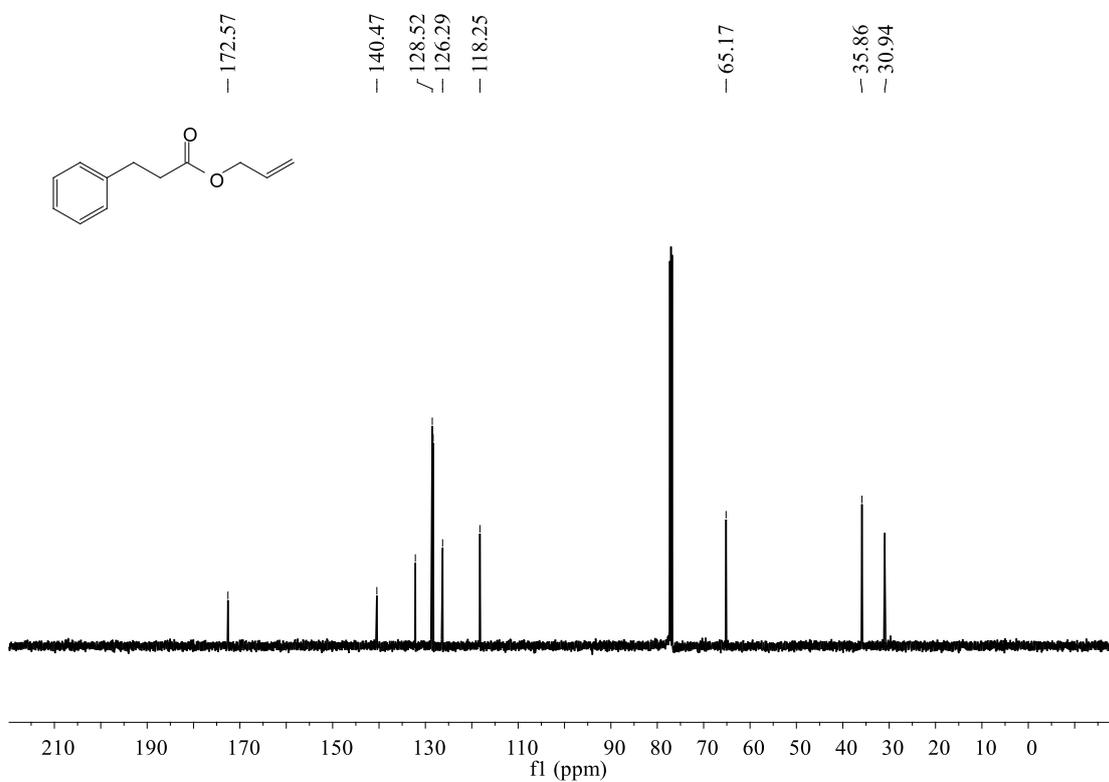
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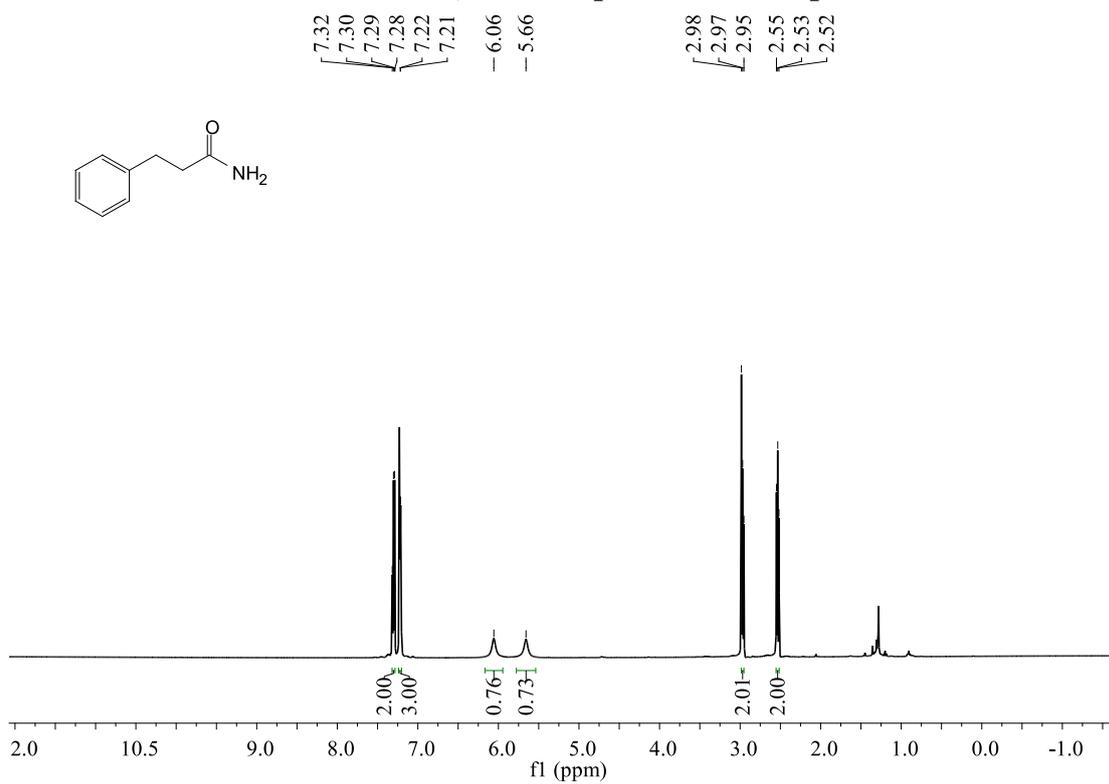
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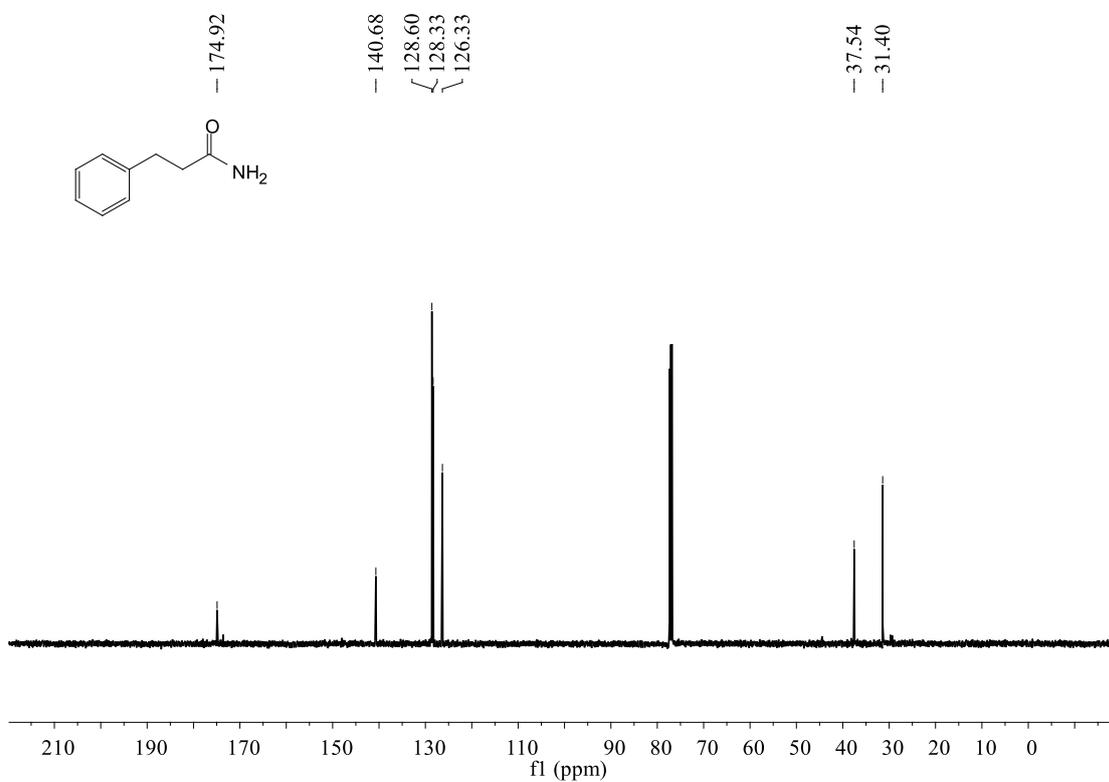
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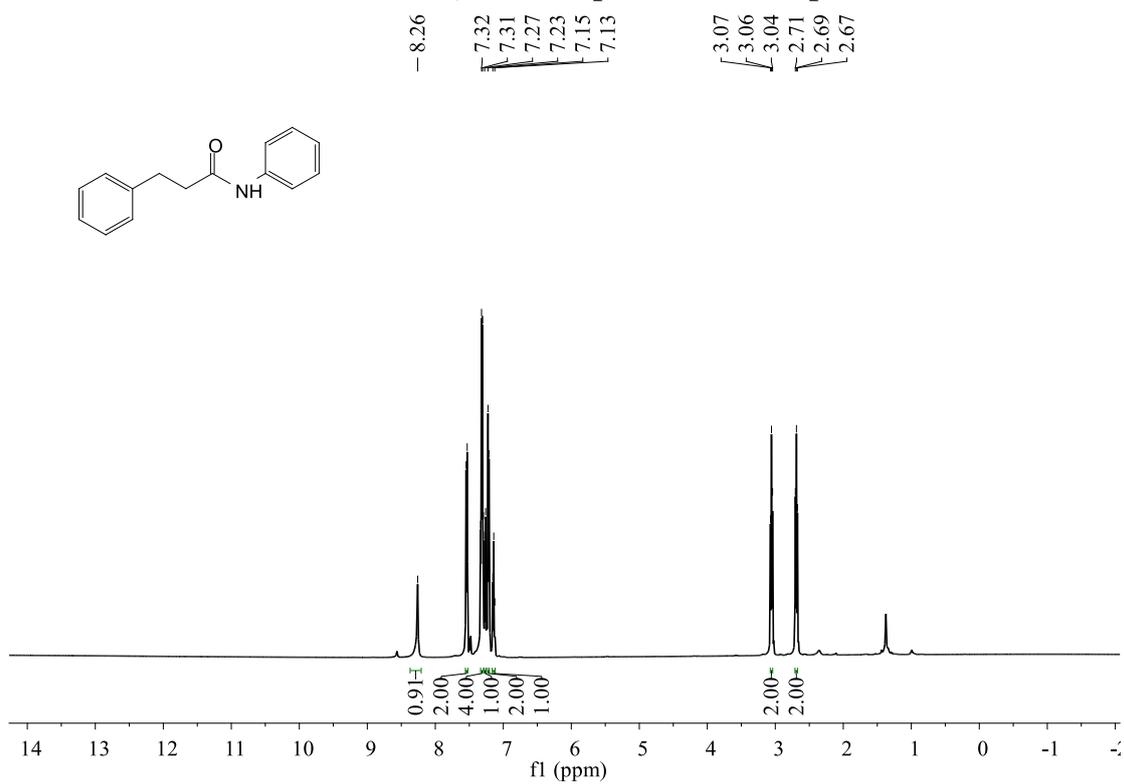
¹H NMR (500 MHz, CDCl₃) spectrum of compound 4a



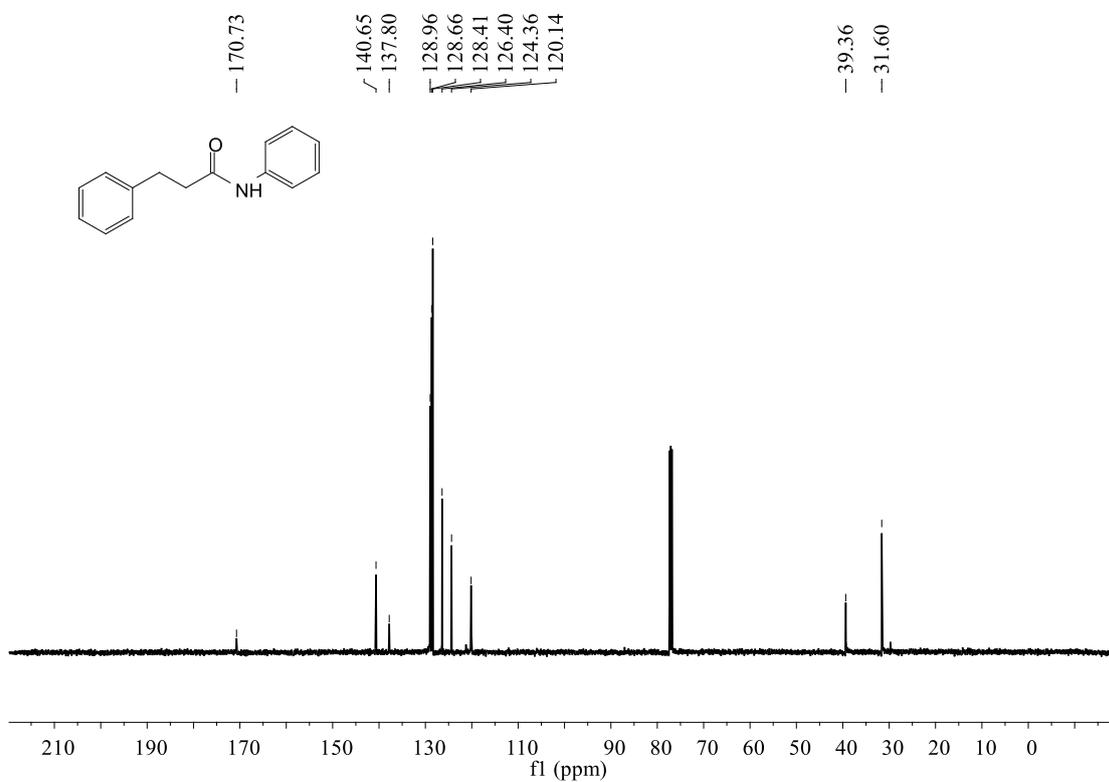
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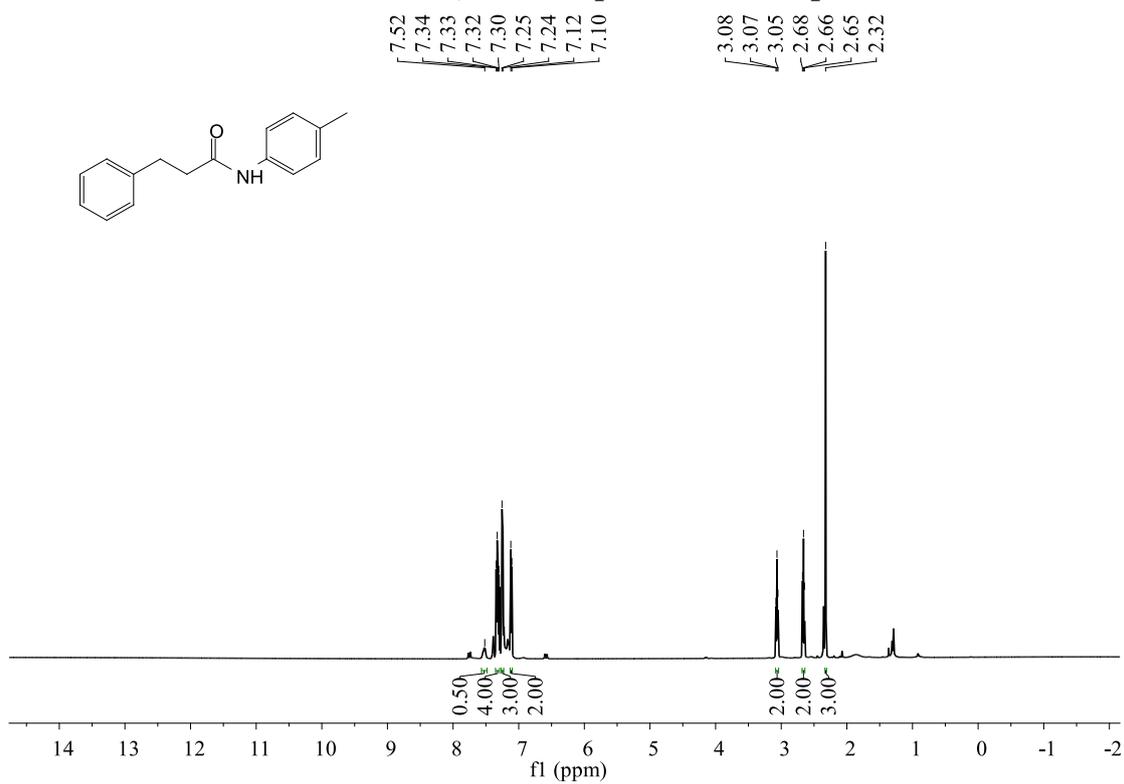
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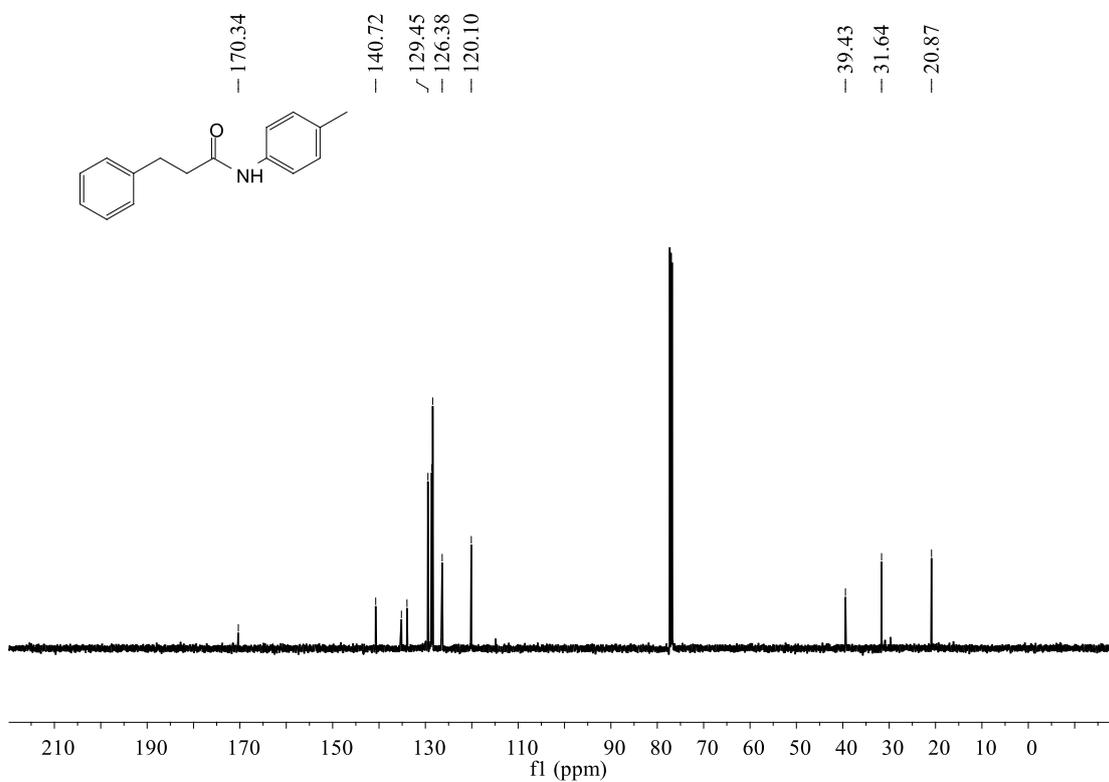
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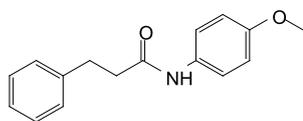
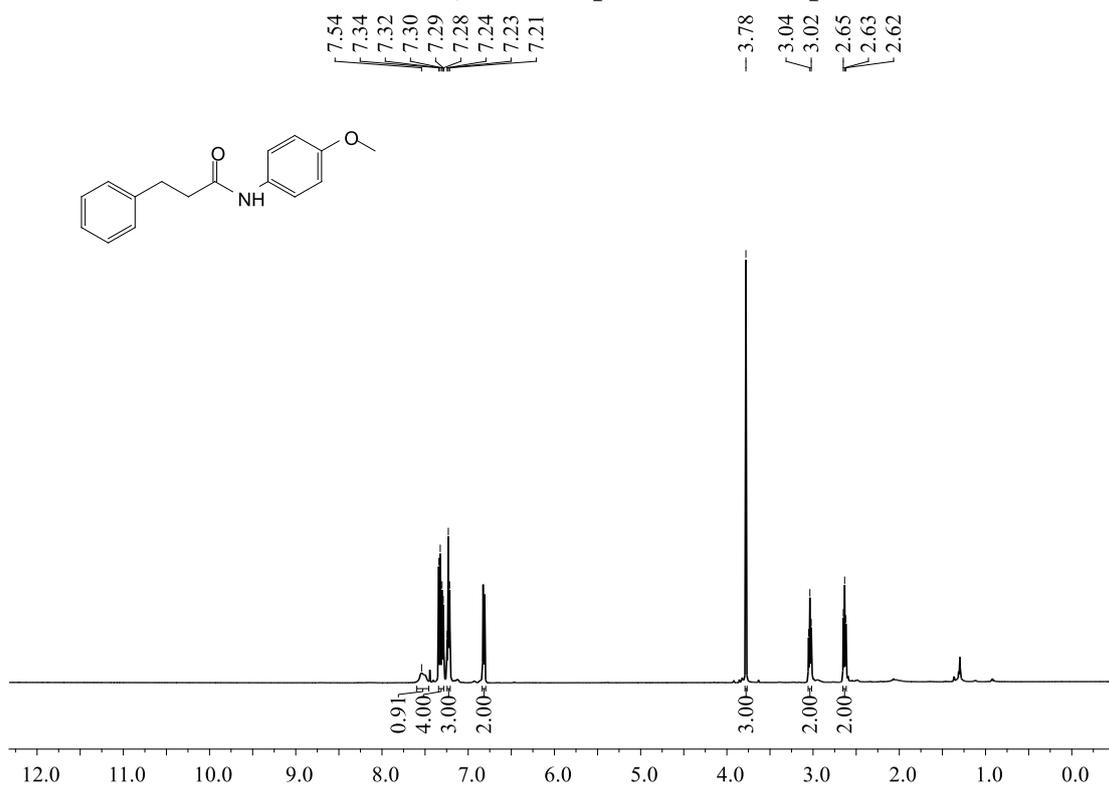
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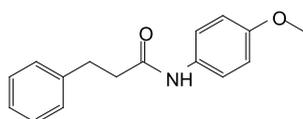
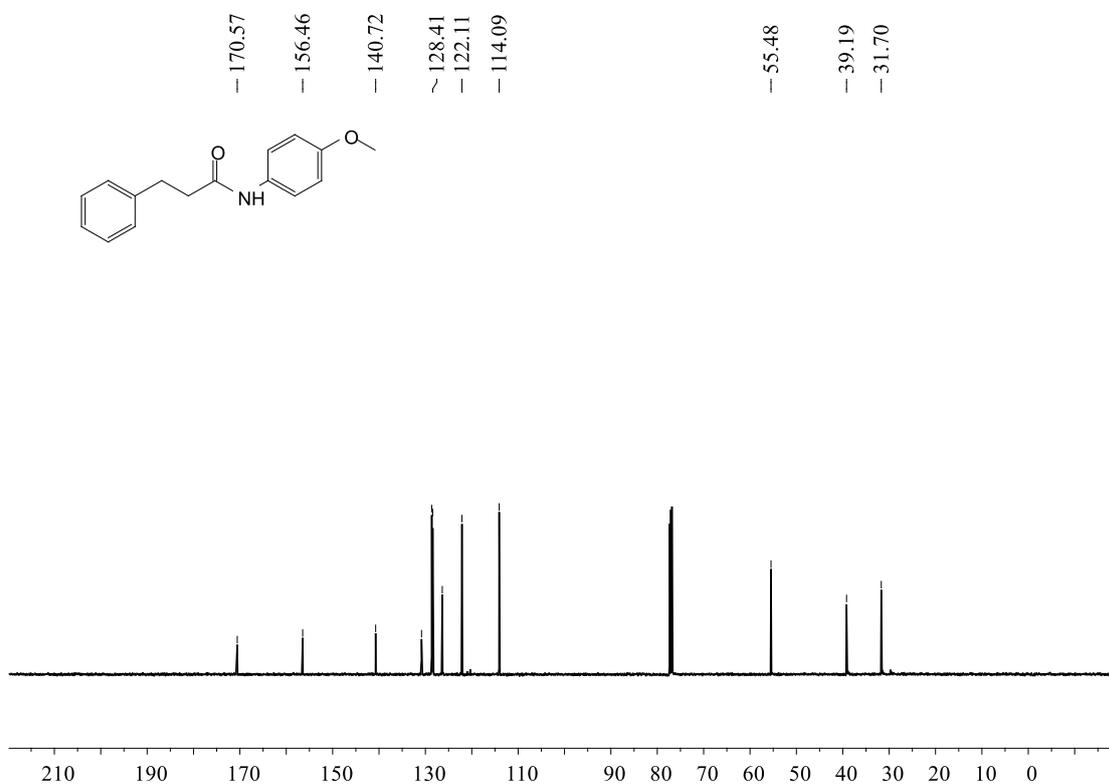
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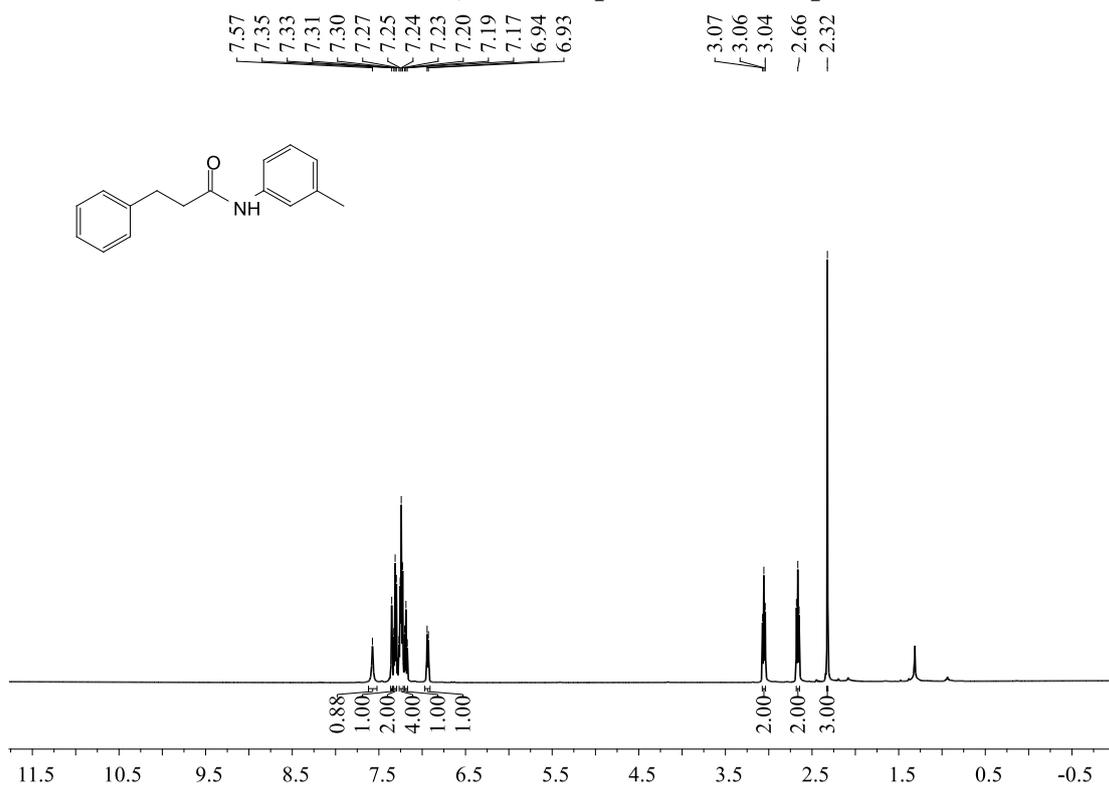
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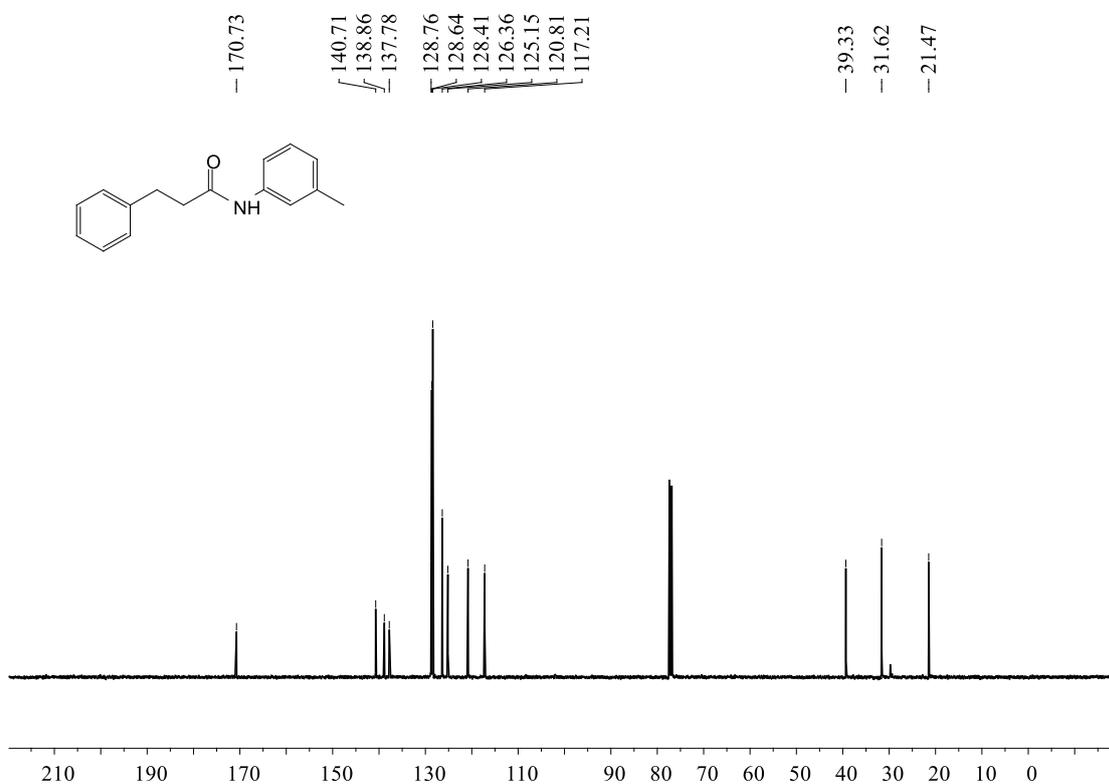
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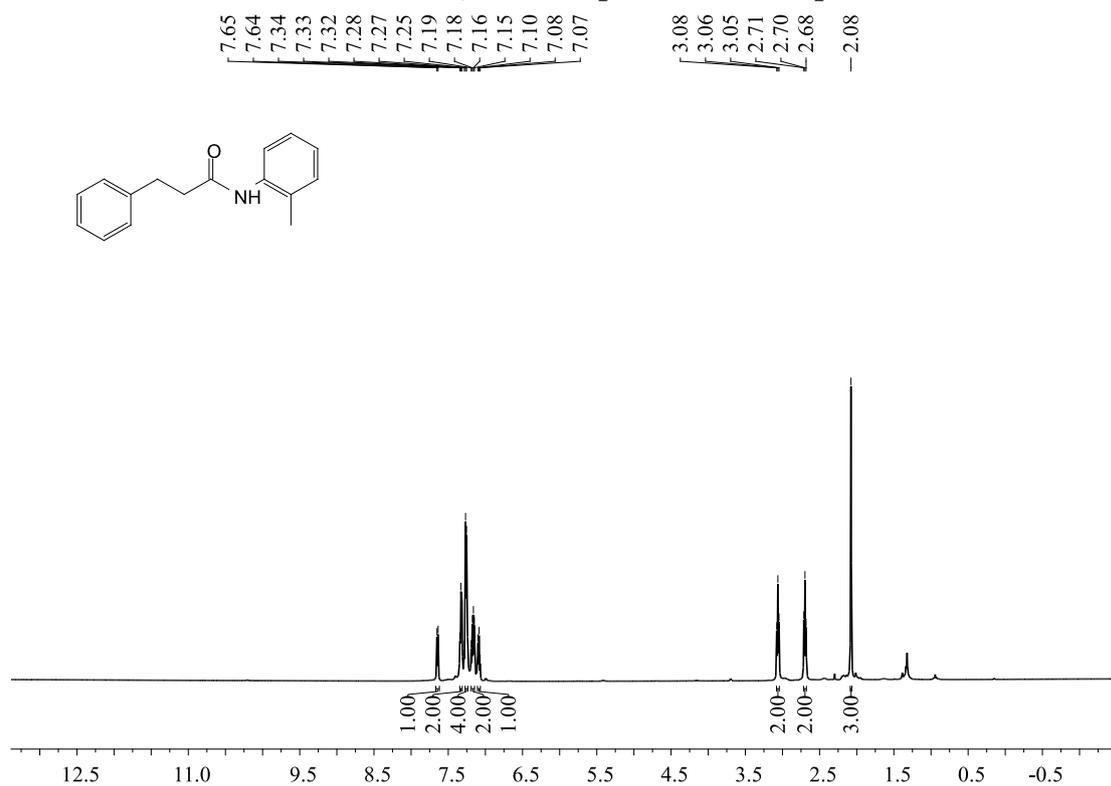
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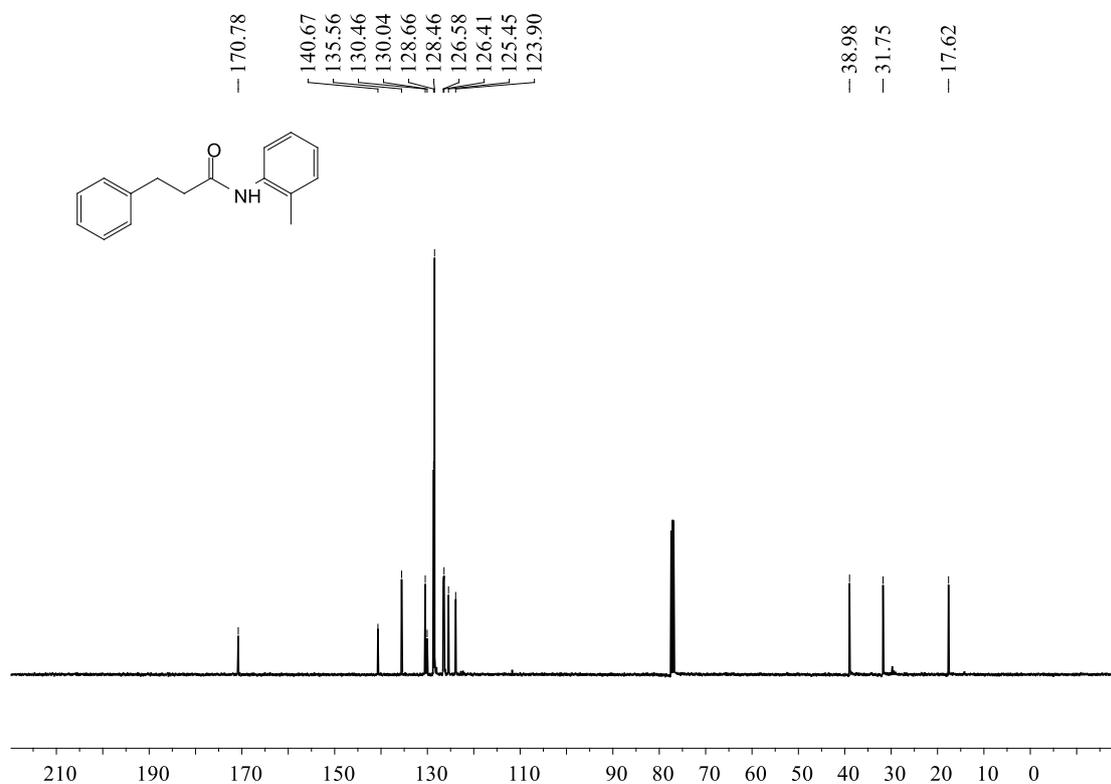
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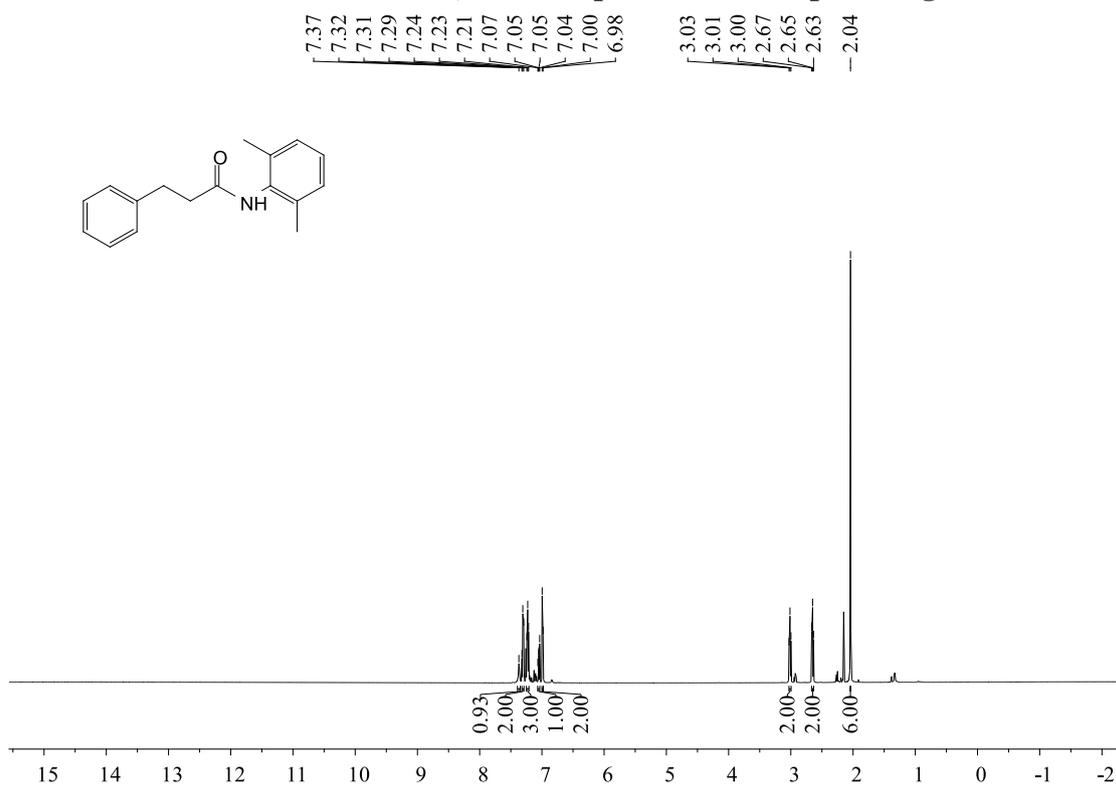
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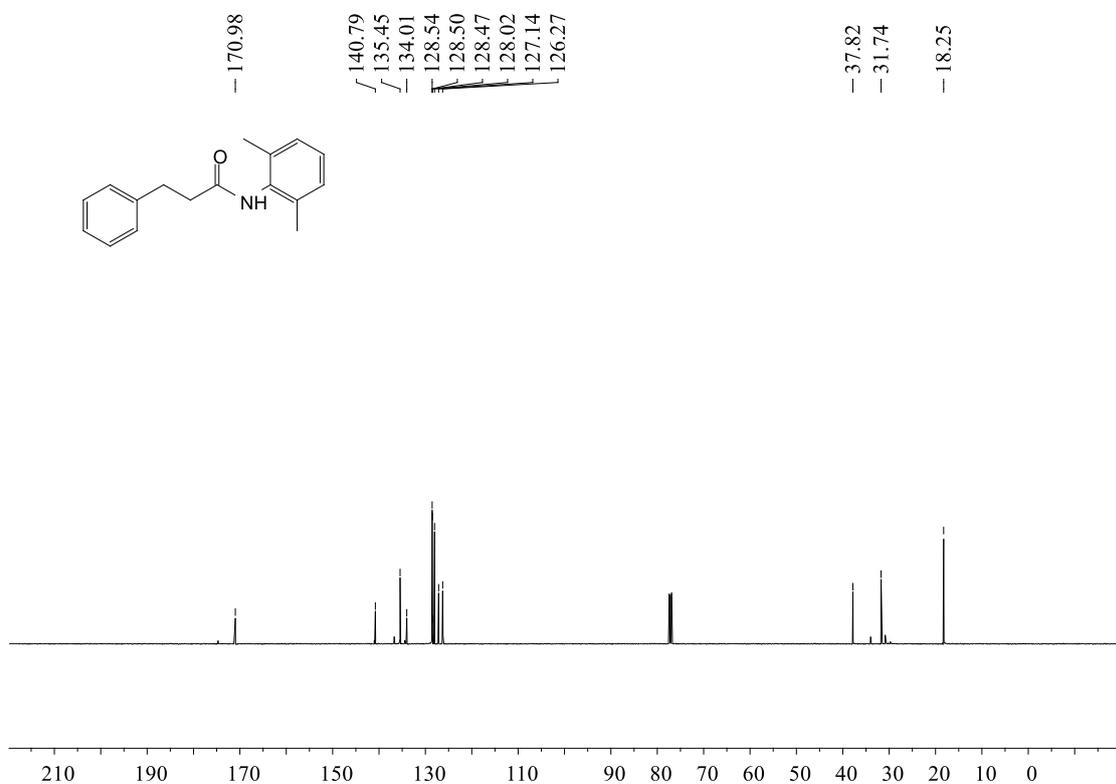
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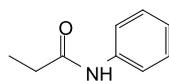
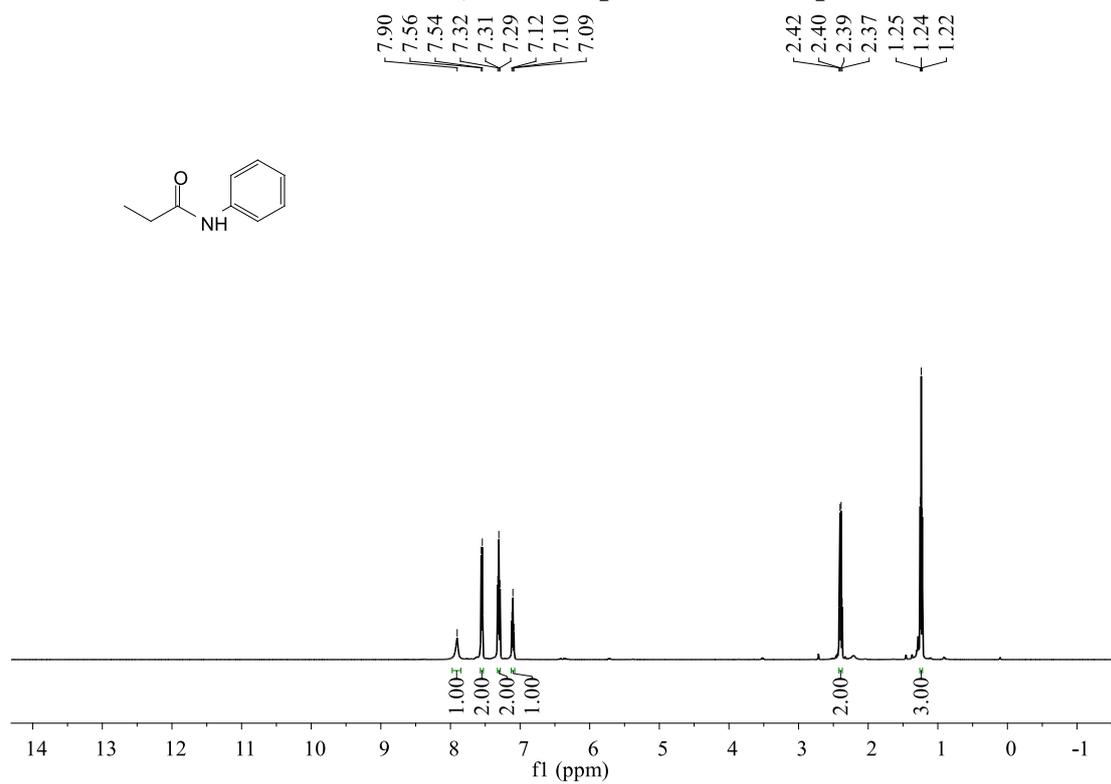
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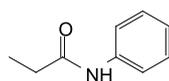
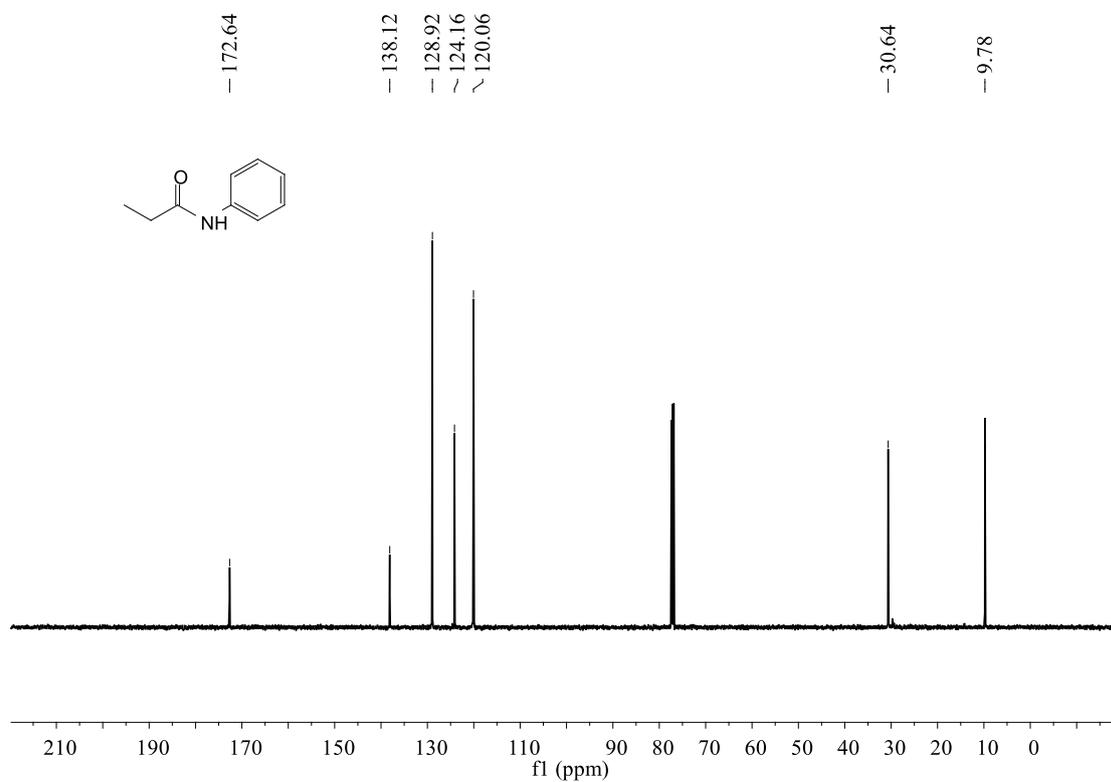
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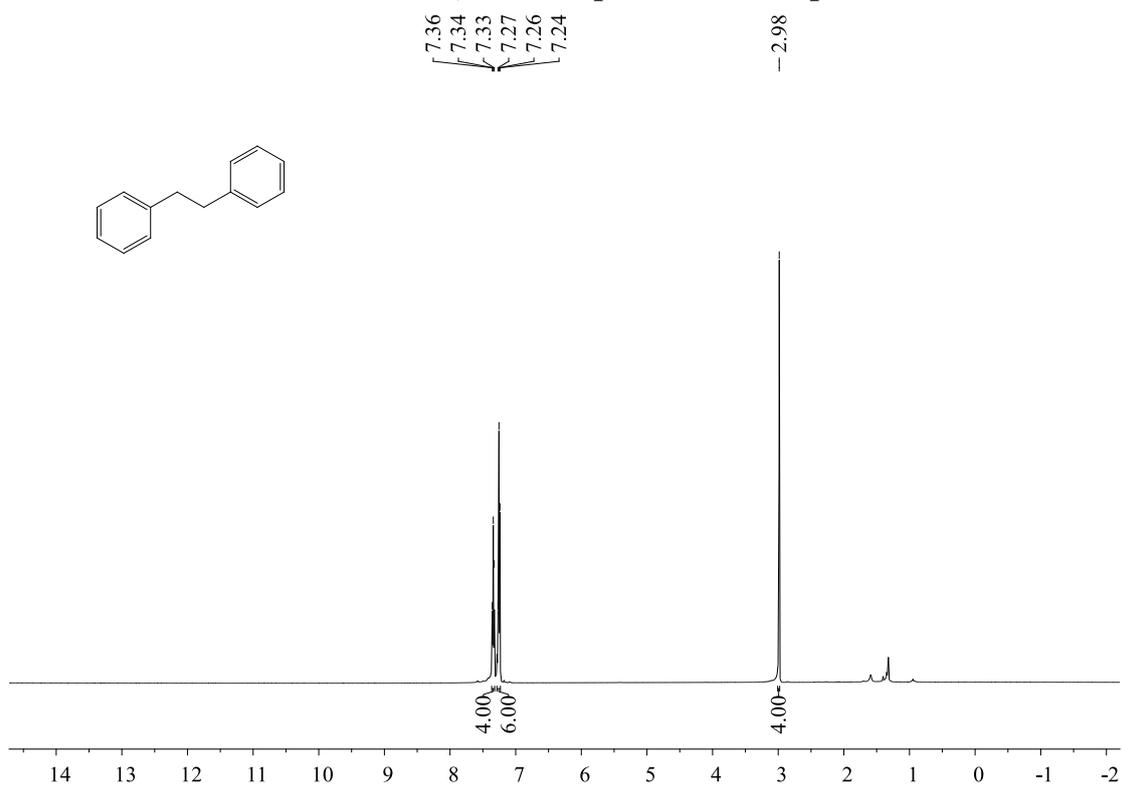
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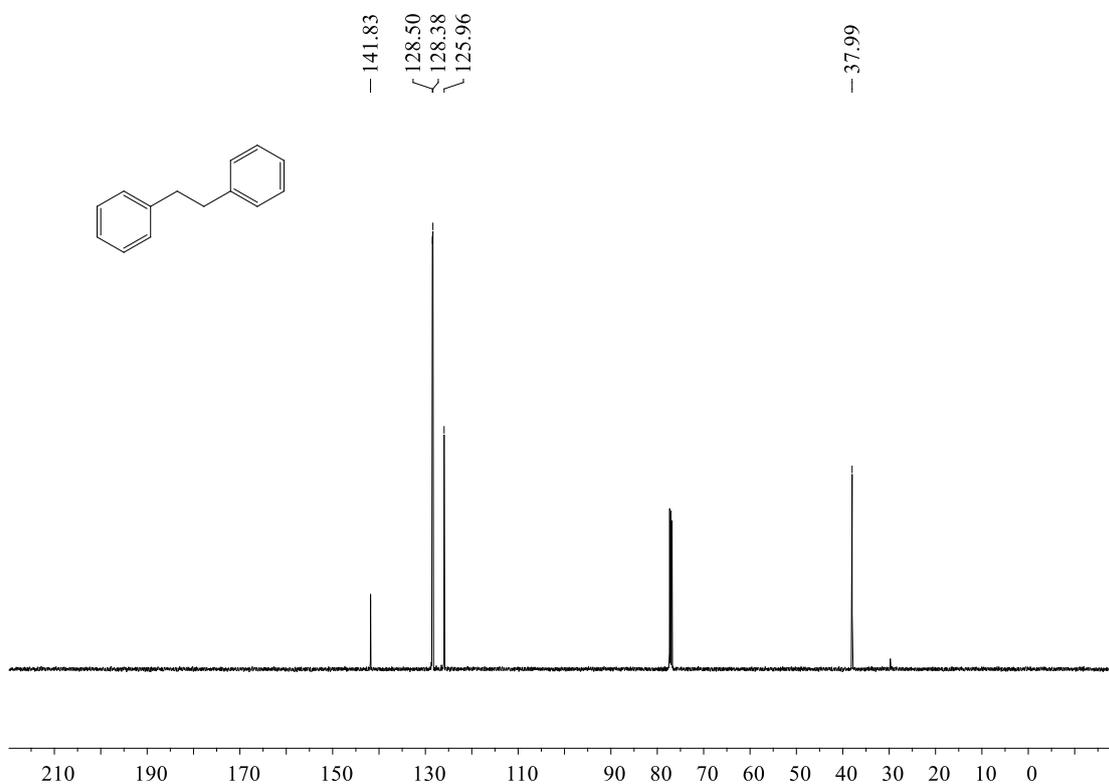
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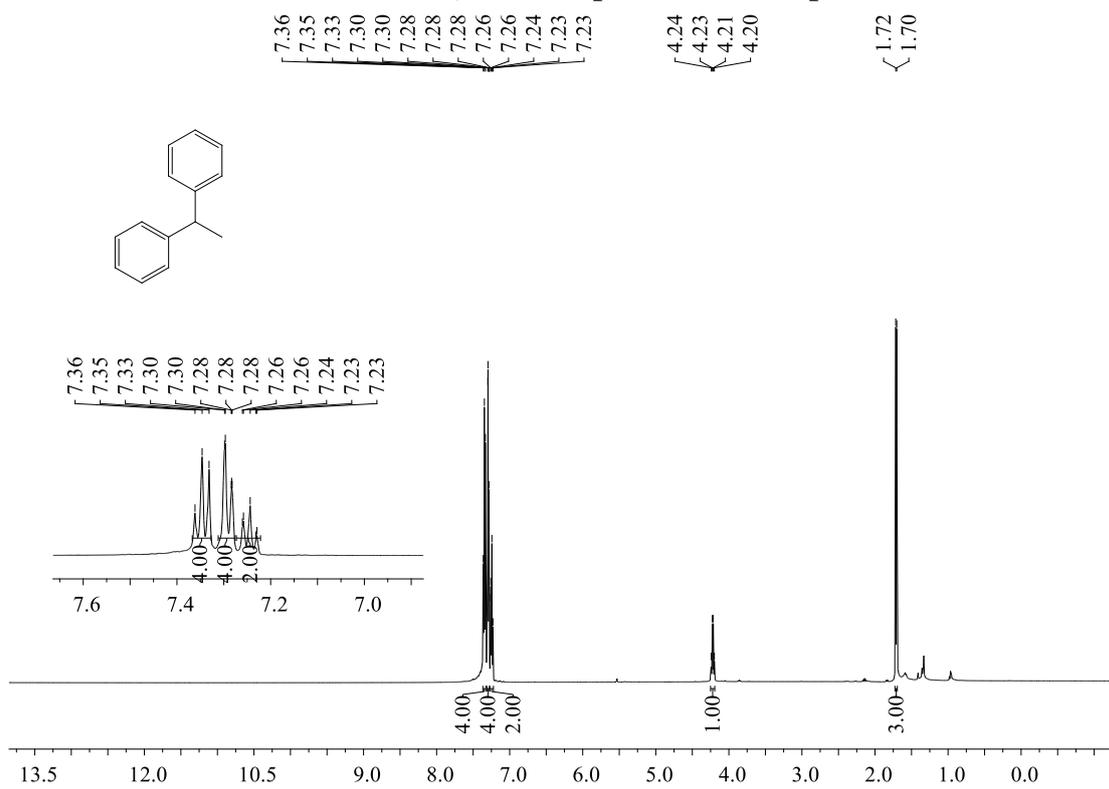
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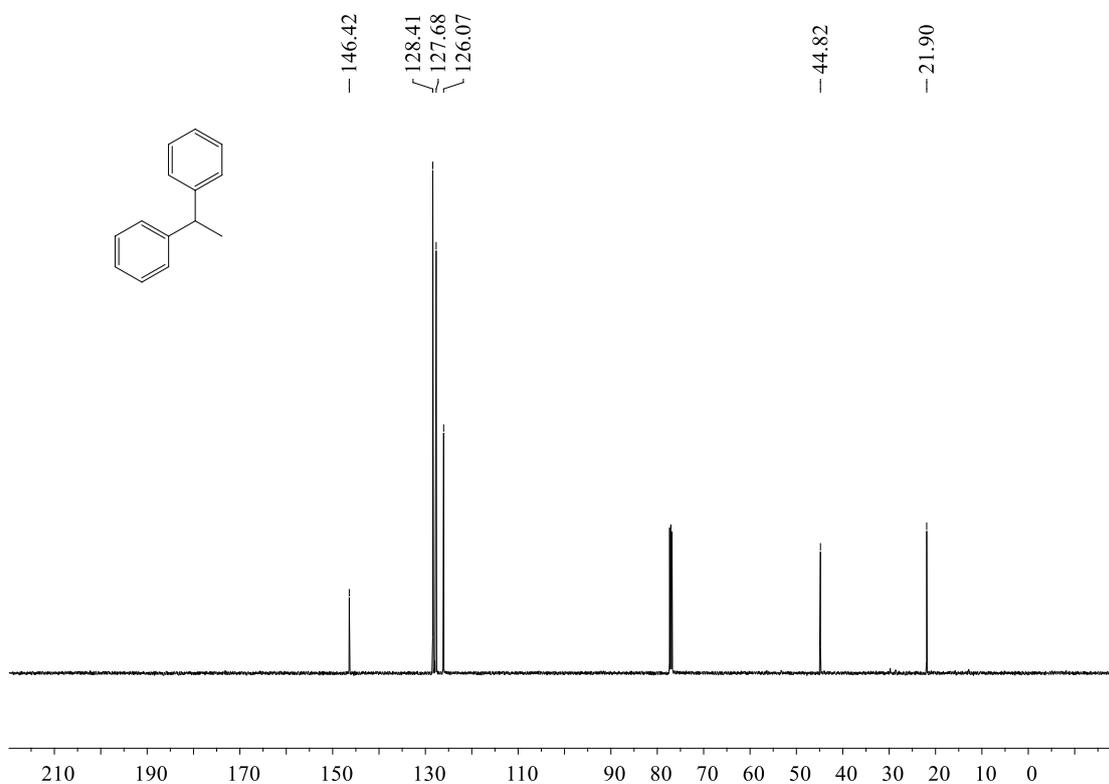
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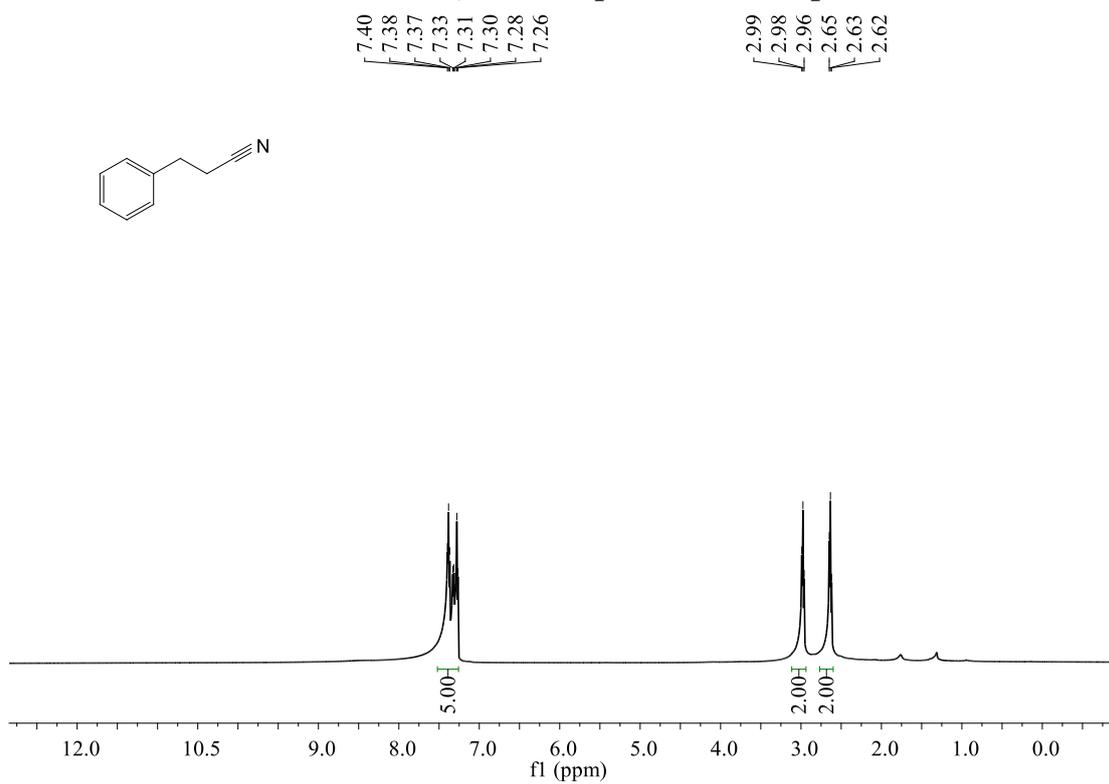
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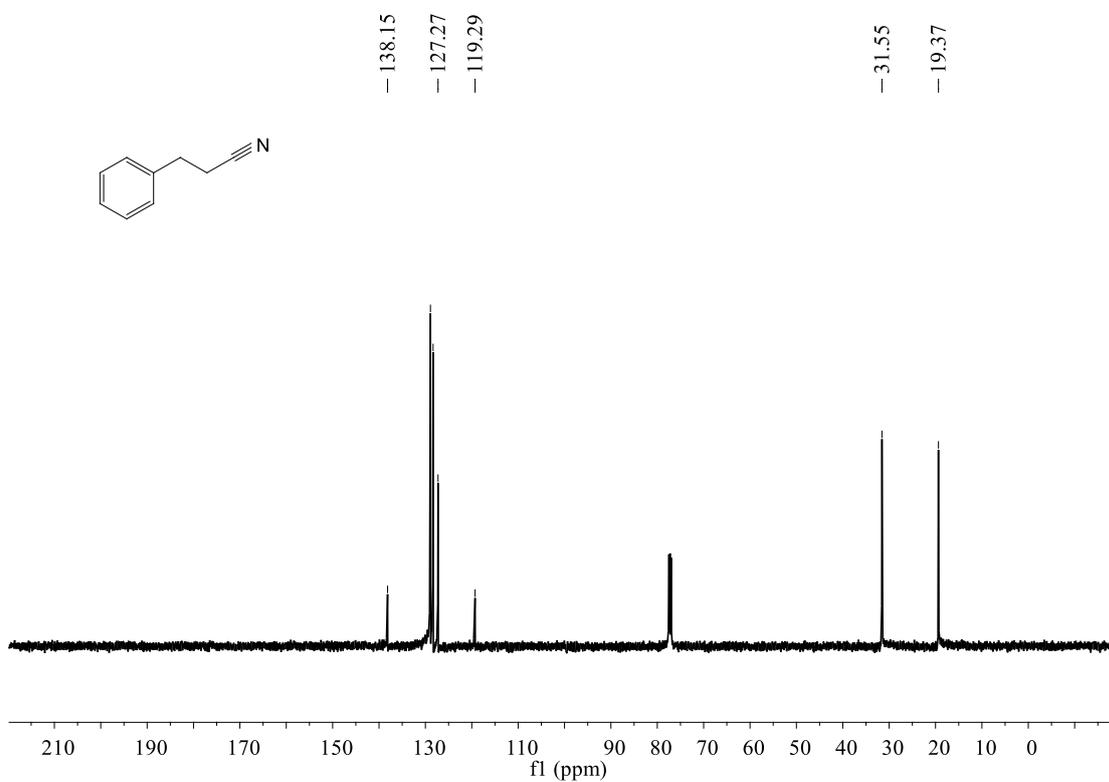
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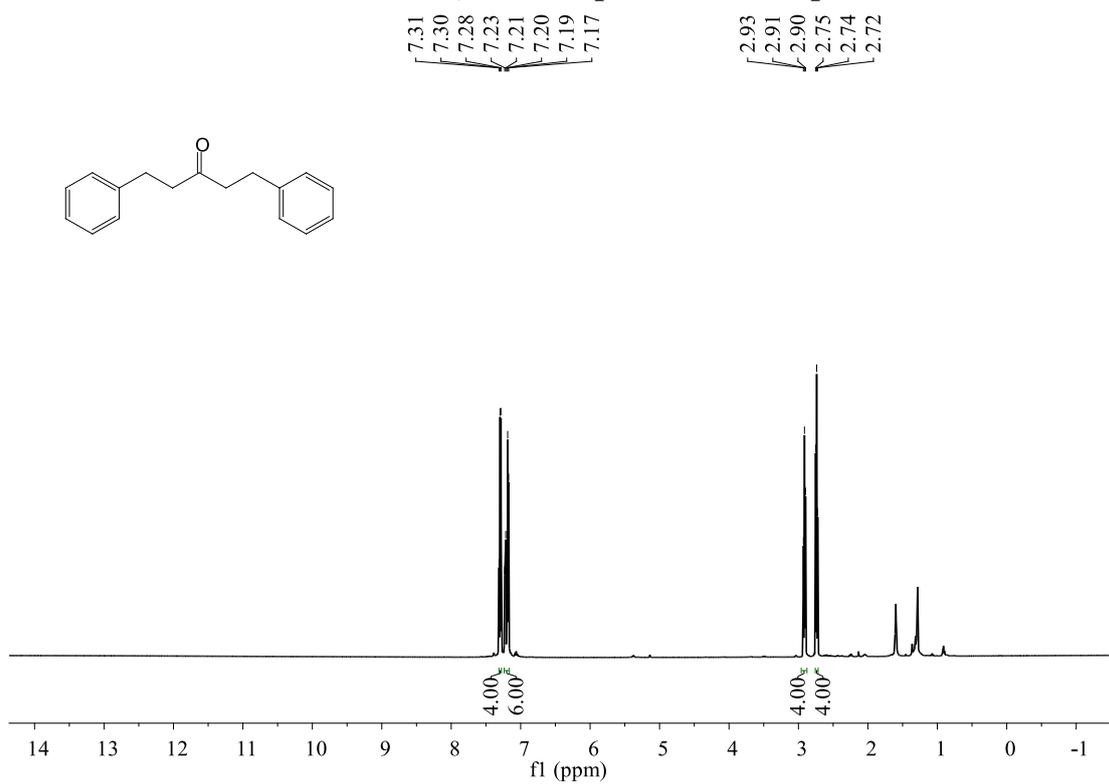
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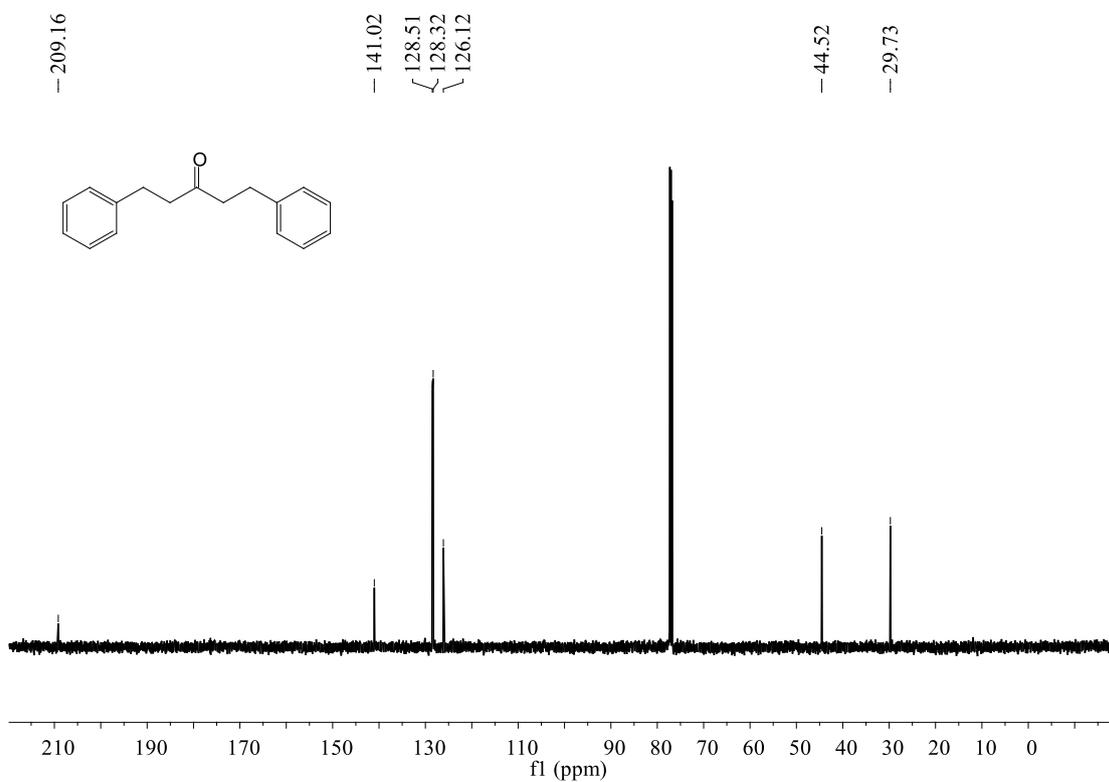
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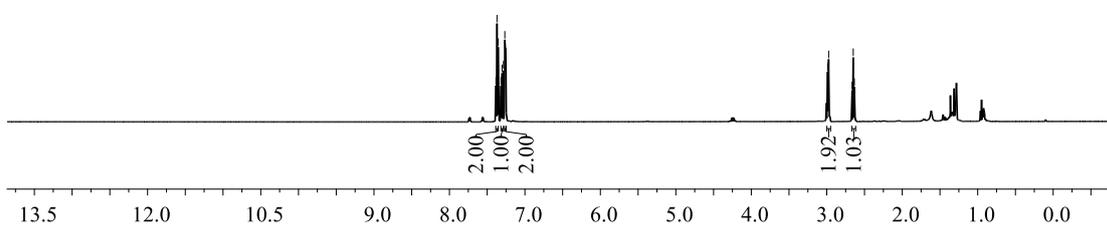
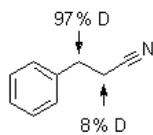


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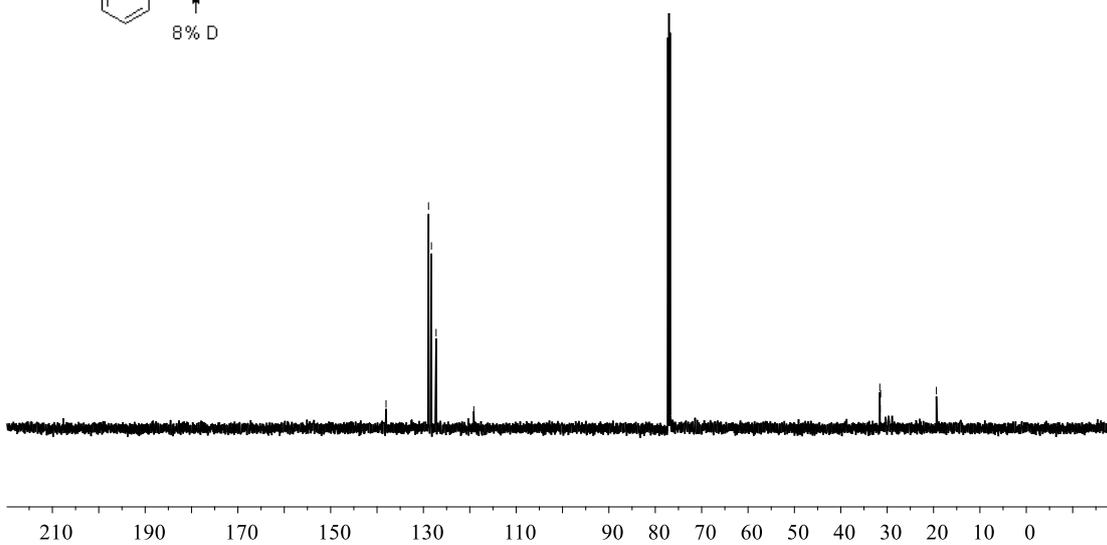
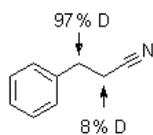
¹H NMR (500 MHz, CDCl₃) spectrum of compound 6c-d₁

7.38
7.38
7.37
7.37
7.36
7.35
7.32
7.32
7.31
7.31
7.30
7.30
7.29
7.29
7.28
7.27
7.25
2.99
2.98
2.66
2.65
2.63

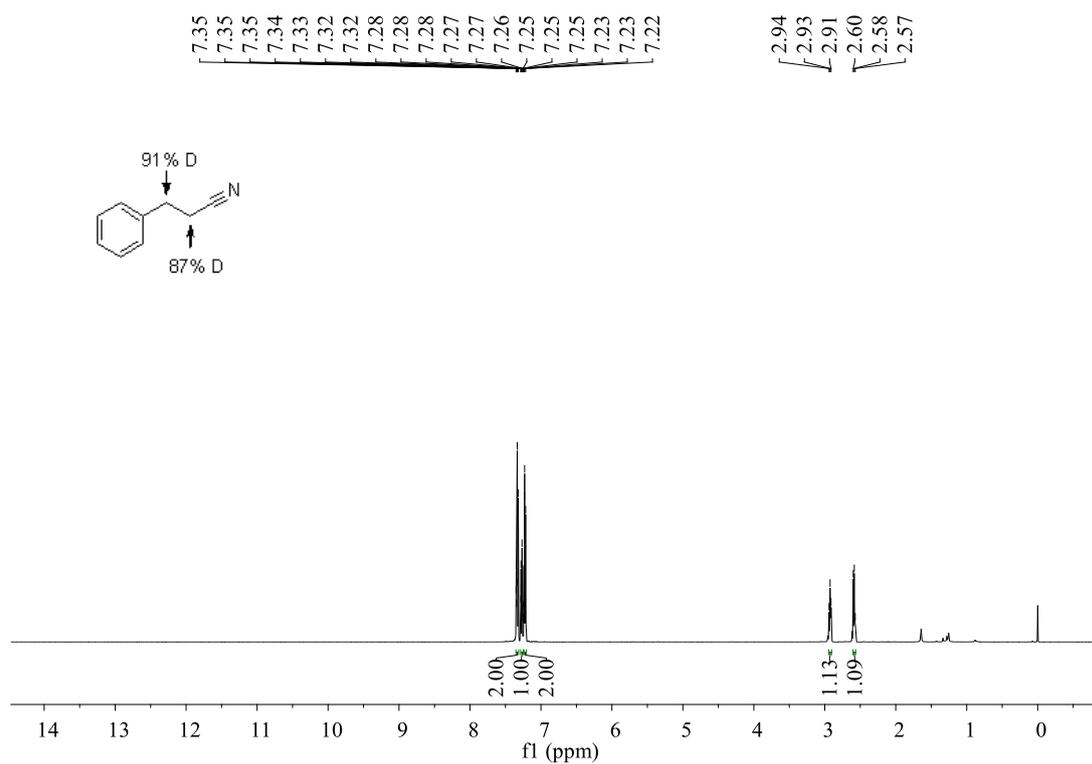


¹³C NMR (126 MHz, CDCl₃) spectrum of compound 6c-d₁

-138.05
-127.26
-119.13
31.59
31.50
31.41
-19.38



¹H NMR (500 MHz, CDCl₃) spectrum of compound 6c-d₂



¹³C NMR (126 MHz, CDCl₃) spectrum of compound 6c-d₂

