

Supplementary Information

Photoinduced C(sp³)-H chlorination of amides with tetrabutyl ammonium chloride

Yanshuo Zhu and Wei Yu*

*State Key Laboratory of Applied Organic Chemistry, College of Chemistry and
Chemical Engineering, Lanzhou University
Lanzhou 730000, China
Email: yuwei@lzu.edu.cn*

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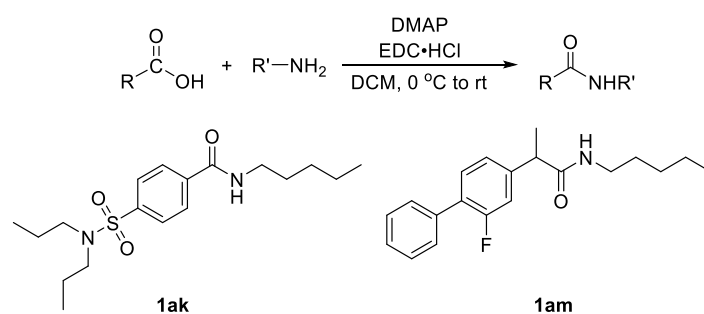
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1. General methods

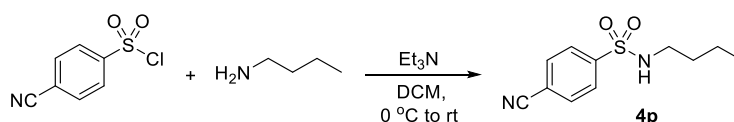
The ^1H and ^{13}C NMR spectra were recorded on a Bruker AVANCE III-400 MHz or an INOVA600 MHz spectrometer with CDCl_3 as the solvent. In CDCl_3 , the chemical shifts in ^1H NMR spectra were determined with $\text{Si}(\text{CH}_3)_4$ as the internal standard ($\delta = 0.00$ ppm); the chemical shifts in ^{13}C NMR spectra were determined based on the chemical shift of CDCl_3 ($\delta = 77.00$ ppm). The coupling constant (s) (J value) are reported in Hz (s = singlet, d = doublet, t = triplet, q = quadruplet, m = multiplet or unresolved, br = broad signal). The high resolution mass spectra (HRMS) were measured on a Thermo Scientific ORBITRAP ELITE by ESI. The Fourier transformation infrared spectra (FT-IR) were measured on a FT-IR spectrometer. Melting points (m.p.) were measured on an XT-4 melting point apparatus and are uncorrected. The compact fluorescent lamp used (45 W Household CFL bulb) as the light source was purchased from Scatter Lighting co. ltd. Common glass tubes were used as the reaction vessel for irradiation, and the distance from the light source was about 1.5 cm. Thin layer chromatography (TLC) analyses were performed using Merck silica gel 60 F254 plates and visualized under UV, by phosphomolybdic acid or iodine stain. Flash column chromatography (FCC) was conducted on silica gel (200–300 mesh). Acetonitrile and other solvents were treated before use following the standard procedures.

2. Experimental procedures

Preparation of substrates **1ak**, **1am** and **4p**



Amides **1ak** and **1am** were prepared following the known procedure.¹



Sulfonamide **4p** was prepared following the reported method.²

General procedure for the $\text{C}(\text{sp}^3)\text{-H}$ chlorination of amides

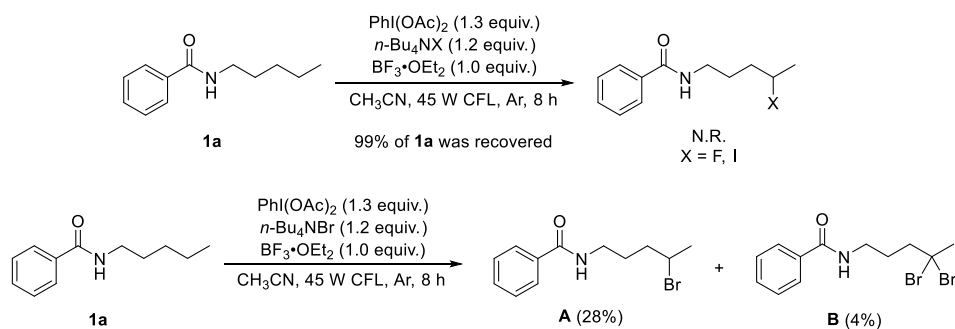
A flame-dried 15 mL glass tube equipped with a magnetic stirring bar and a rubber

stopper was charged with the amide (0.2 mmol), $\text{PhI}(\text{OAc})_2$ (for amides: 83.7 mg, 0.26 mmol, 1.3 equiv.; for sulfonamides: 77.3 mg, 0.24 mmol), $n\text{-Bu}_4\text{NCl}$ (66.7 mg, 0.24 mmol, 1.2 equiv.), $\text{BF}_3\cdot\text{OEt}_2$ (27.0 μL , 0.20 mmol, 1.0 equiv.) and 2 mL of CH_3CN . The tube was evacuated and backfilled with argon for three times. The mixture was irradiated under stirring with a 45 W household CFL lamp (at a distance of 1.5 cm) at ambient temperature ($<35^\circ\text{C}$ in most cases; a small electric fan was used to dissipate heat emitted by the lamp) for an appropriate period of time (8.0 h in general for amides; 3.0 h in general sulfonamides). Once the reaction was complete as indicated by TLC, the mixture was concentrated under reduced pressure on a rotary evaporator, and the residual was subjected to silica gel column chromatography (eluent: petroleum ether (PE) PE and ethyl acetate (EA)) to afford the product.

Gram scale preparation of 2a and 5b:

A flame-dried 100 mL round bottomed flask equipped with a magnetic stirring bar and a rubber stopper was charged with **1a** (1.5 g, 8.0 mmol), $\text{PhI}(\text{OAc})_2$ (3.4 g, 10.4 mmol, 1.3 equiv.), $n\text{-Bu}_4\text{NCl}$ (2.7 g, 9.6 mmol, 1.2 equiv.), $\text{BF}_3\cdot\text{OEt}_2$ (1.1 mL, 8 mmol, 1.0 equiv.) and 40 mL CH_3CN . The solution was irradiated with a 45 W household CFL lamp (at a distance of 1.5 cm) under an argon atmosphere (argon balloon) for 8 h. The reaction mixture was then poured into an aqueous solution of $\text{Na}_2\text{S}_2\text{O}_3$ (20%, 15 mL), and the product was extracted with CH_2Cl_2 (3 \times 15 mL). The combined organic phases were dried over anhydrous Na_2SO_4 , concentrated in vacuo, and purified by flash column chromatography on silica gel (PE/EA = 6:1 to 5:1) to give **2a** in a yield of 83% (1.50 g).

A flame-dried 100 mL round bottomed flask equipped with a magnetic stirring bar and a rubber stopper was charged with **4b** (1.5 g, 6.0 mmol, 1.0 equiv.), $\text{PhI}(\text{OAc})_2$ (2.3 g, 7.2 mmol, 1.2 equiv.), $n\text{-Bu}_4\text{NCl}$ (2.0 g, 9.6 mmol, 1.2 equiv.), $\text{BF}_3\cdot\text{OEt}_2$ (0.8 mL, 6 mmol, 1.0 equiv.) and 40 mL of CH_3CN . The solution was irradiated with a 45 W household CFL lamp (at a distance of 1.5 cm) under an argon atmosphere (argon balloon) for 3 h. The reaction mixture was then poured into an aqueous solution of $\text{Na}_2\text{S}_2\text{O}_3$ (20%, 15 mL), and the product was extracted with CH_2Cl_2 (3 \times 15 mL). The combined organic phases were dried over anhydrous Na_2SO_4 , concentrated in vacuo, and purified by flash column chromatography on silica gel (PE/EA = 12:1 to 10:1) to give **5b** in a yield of 70 % (1.15 g).



Scheme S1. Reaction of **1a** under the condition of other ammonium salts.

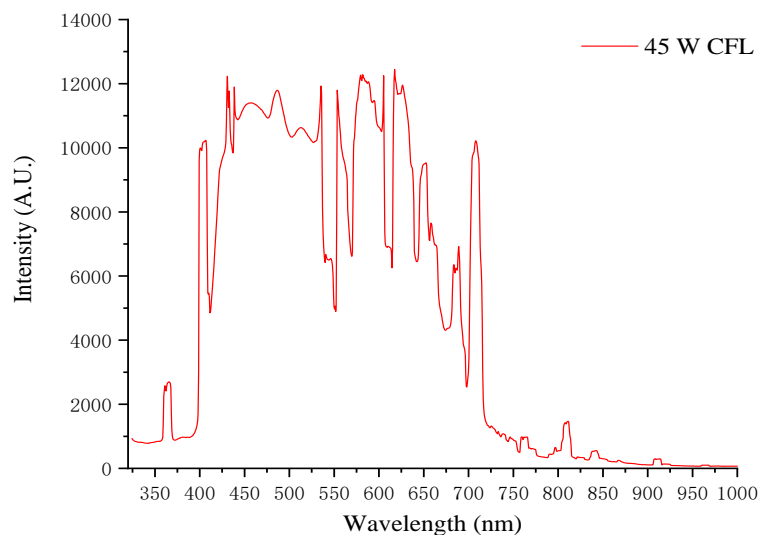
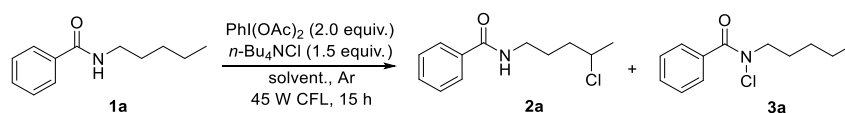


Figure S1. Emission spectrum of the 45 W CFL.

3. Optimization of reaction conditions

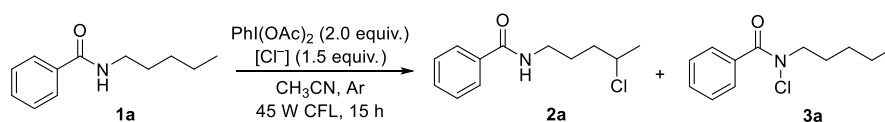
Table S1. Screening of solvent^a



Entry	Solvent	Time (h)	2a/3a Yield (%) ^b
1	CH ₃ CN	15	55/37
2	EtOH	15	0/0
3	EtOAc	15	31/0
4	AcOH	15	0/0
5	H ₂ O	15	0/0
6	CH ₃ CN/H ₂ O (1:1)	15	9/0

^a The reaction was performed under an argon atmosphere on 0.2 mmol scale in 2.0 mL solvent.

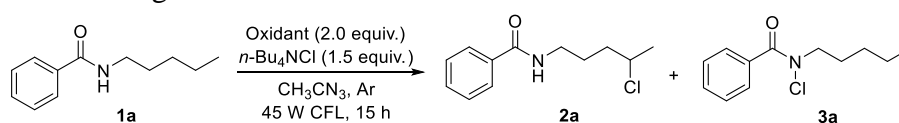
^b Isolated yield after silica gel column chromatography.

Table S2. Screening of nucleophilic chlorine source^a

Entry	[Cl ⁻]	Time (h)	2a/3a Yield (%) ^b
1	NaCl	15	0/0
2	KCl	15	0/0
3	NH ₄ Cl	15	0/0

^a The reaction was performed under an argon atmosphere on 0.2 mmol scale in 2.0 mL CH₃CN.

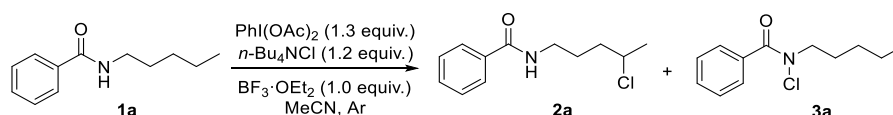
^b Isolated yield.

Table S3. Screening of oxidants^a

Entry	Oxidant (equiv.)	Time (h)	2a/3a Yield (%) ^b
1	PhI(OAc) ₂ (2.0)	15	55/37
2	PhI(OCOCF ₃) ₂ (2.0)	15	25/33
3	PhI(OH)(OTs) (2.0)	15	11/24
4	PhIO (2.0)	15	0/0
5	BI-OH (2.0)	15	0/0
6 ^c	BI-Cl (2.0)	15	0/0
7 ^c	PhICl ₂	15	0/7
8	Oxone (2.0)	15	trace/27
9	K ₂ S ₂ O ₈ (2.0)	15	0/0
10	DTBP (2.0)	15	0/0
11	TBHP (2.0)	15	0/0

^a The reaction was performed under an argon atmosphere on 0.2 mmol scale in 2.0 mL CH₃CN.

^b Isolated yield. ^c Without *n*-Bu₄NCl. Oxone: potassium peroxomonosulfate; DTBP: *di-tert*-butyl peroxide; TBHP: *tert*-butyl hydroperoxide; BI-OH: 1-hydroxy-1λ³-benzo[*d*][1,2]iodaoxol-3(*1H*)-one; BI-Cl: 1-chloro-1λ³-benzo[*d*][1,2]iodaoxol-3(*1H*)-one.

Table S4. Screening of the light source^a

Entry	Light Source	Time (h)	2a/3a Yield (%) ^b
1	45 W CFL	8	91/0
2	10 W blue LEDs	24	73/16

^a The reaction was performed under an argon atmosphere on 0.2 mmol scale in 2.0 mL CH₃CN.

^b Isolated yield.

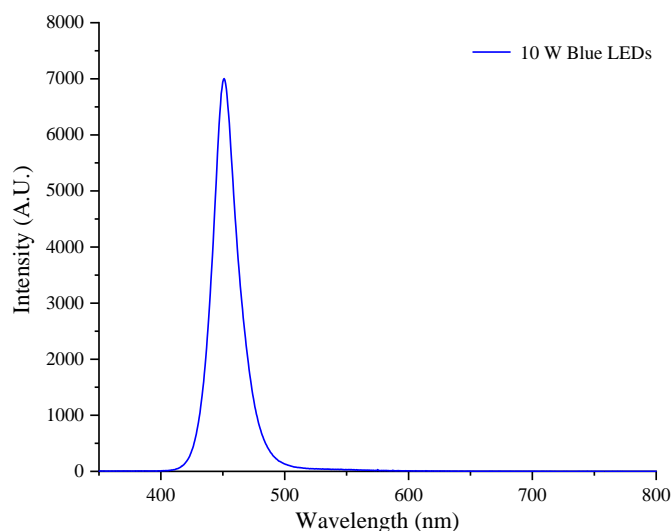
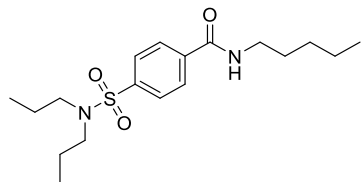


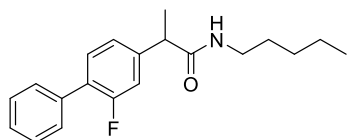
Figure S2. Emission spectrum of the 10 W blue LEDs.

4. Characterization data



4-(*N,N*-Dipropylsulfamoyl)-*N*-pentylbenzamide (**1ak**)

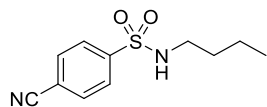
White solid obtained by column chromatography (PE/EA = 8:1); 1.6 g, 65% yield (7.0 mmol scale). m.p. 110–111 °C; R_f = 0.24 (PE/EA = 5:1). ^1H NMR (400 MHz, CDCl_3) δ 7.87 (d, J = 8.3 Hz, 2H), 7.78–7.74 (m, 2H), 6.69 (br, 1H), 3.44 (q, J = 8.0 Hz, 2H), 3.22–2.91 (t, J = 6.0 Hz, 4H), 1.67–1.49 (m, 6H), 1.40–1.32 (m, 4H), 0.91–0.84 (m, 9H). ^{13}C NMR (100 MHz, CDCl_3) δ 166.2, 142.3, 138.5, 127.7, 127.0, 49.9, 40.2, 29.1, 29.1, 22.3, 21.8, 13.9, 11.1. IR (KBr, cm^{-1}) ν 3319, 2964, 2933, 2874, 1639, 1549, 1468, 1327, 1147, 1090, 1000, 852, 778, 739, 663, 604, 560, 451. HRMS (ESI-TOF) calcd for $\text{C}_{18}\text{H}_{31}\text{N}_2\text{O}_3\text{S}$ $[\text{M}+\text{H}]^+$ 355.2050, found 355.2049.



2-(2-Fluoro-[1,1'-biphenyl]-4-yl)-*N*-pentylpropanamide (**1am**)

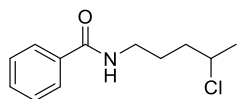
White solid obtained by column chromatography (PE/EA = 8:1); 1.0 g, 78% yield (4.1 mmol scale). m.p. 82–84 °C; R_f = 0.25 (PE/EA = 5:1). ^1H NMR (400 MHz, CDCl_3) δ 7.52 (d, J = 7.9 Hz, 2H), 7.44–7.33 (m, 4H), 7.15 (t, J = 8.5 Hz, 2H), 5.96 (s, 1H), 3.59 (q, J = 7.2 Hz, 1H), 3.21 (q, J = 6.7 Hz, 2H), 1.53 (d, J = 7.2 Hz, 3H), 1.48–1.41 (m, 2H), 1.32–1.17 (m, 4H), 0.85 (t, J = 7.0 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 173.4, 160.8, 158.4, 142.9 (d, J = 7.4 Hz, 1C), 135.3, 130.8 (d, J = 3.9 Hz, 1C), 128.8

(d, $J = 2.8$ Hz, 1C), 128.3, 127.5 (t, $J = 7.0$ Hz, 1C), 123.5 (d, $J = 3.3$ Hz, 1C), 115.1 (d, $J = 23.4$ Hz, 1C), 46.4, 39.6, 29.1, 28.9, 22.2, 18.4, 13.9. IR (KBr, cm^{-1}) ν 3294, 3078, 2931, 2871, 1651, 1557, 1418, 1371, 1230, 1132, 1074 1011, 928, 766, 697, 573, 458. HRMS (ESI-TOF) calcd for $\text{C}_{20}\text{H}_{25}\text{FNO}$ $[\text{M}+\text{H}]^+$ 314.1915, found 314.1913.



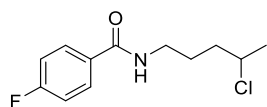
***N*-Butyl-4-cyanobenzenesulfonamide (4p)**

White solid obtained by column chromatography (PE/EA = 5:1); 1.8 g, 90% yield (8.4 mmol scale). m.p. 107–108 °C; $R_f = 0.18$ (PE/EA = 5:1). ^1H NMR (400 MHz, CDCl_3) δ 8.02 (d, $J = 8.4$ Hz, 2H), 7.85 (d, $J = 8.3$ Hz, 2H), 5.26 (t, $J = 6.0$ Hz, 1H), 2.98 (q, $J = 6.7$ Hz, 2H), 1.49–1.42 (m, 2H), 1.34–1.25 (m, 1H), 0.85 (t, $J = 7.4$ Hz, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 144.2, 132.9, 127.6, 117.3, 16.1, 42.9, 31.4, 19.5, 13.4. IR (KBr, cm^{-1}) ν 3265, 2957, 2860, 1435, 1324, 1157, 1089, 845, 568, 515. HRMS (ESI-TOF) calcd for $\text{C}_{11}\text{H}_{15}\text{N}_2\text{O}_2\text{S}$ $[\text{M}+\text{H}]^+$ 239.0849, found 239.0846.



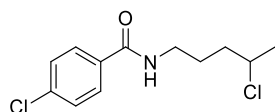
***N*-(4-Chloropentyl)benzamide (2a)³**

Colorless oil obtained by column chromatography (PE/EA = 6:1 to 4:1); 41 mg, 91% yield; reaction time = 6 h. $R_f = 0.35$ (PE/EA = 3:1). ^1H NMR (400 MHz, CDCl_3) δ 7.81–7.78 (d, $J = 7.0$ Hz, 2H), 7.45 (t, $J = 7.3$ Hz, 1H), 7.36 (t, $J = 7.5$ Hz, 2H), 7.20 (s, 1H), 4.04–3.96 (m, 1H), 3.40 (q, $J = 5.3$ Hz, 2H), 1.84–1.65 (m, 4H), 1.46 (d, $J = 6.5$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 167.7, 134.4, 131.2, 128.3, 126.8, 58.3, 39.3, 37.3, 26.7, 25.2.



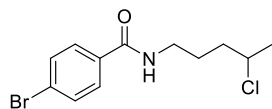
***N*-(4-Chloropentyl)-4-fluorobenzamide (2b)³**

Colorless oil obtained by column chromatography (PE/EA = 5:1 to 4:1); 44 mg, 90% yield; reaction time = 8 h. $R_f = 0.32$ (PE/EA = 3:1). ^1H NMR (400 MHz, CDCl_3) δ 7.81–7.77 (m, 2H), 7.09 (t, $J = 8.6$ Hz, 2H), 6.67 (s, 1H), 4.10–4.03 (m, 1H), 3.46 (d, $J = 6.2$ Hz, 2H), 1.86–1.71 (m, 4H), 1.52 (d, $J = 6.6$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 166.6, 164.6 (d, $J = 251.8$ Hz, 1C), 130.7 (d, $J = 3.1$ Hz, 1C), 129.2 (d, $J = 8.9$ Hz, 1C), 115.5 (d, $J = 21.8$ Hz, 1C), 58.3, 39.5, 37.4, 26.8, 25.3.



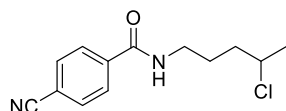
4-Chloro-*N*-(4-chloropentyl)benzamide (2c)³

White solid obtained by column chromatography (PE/EA = 5:1 to 4:1); 45 mg, 87% yield; reaction time = 8 h. $R_f = 0.34$ (PE/EA = 3:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.71 (d, $J = 8.6$ Hz, 2H), 7.38 (d, $J = 8.6$ Hz, 2H), 6.74 (t, $J = 5.9$ Hz, 1H), 4.08–4.00 (m, 1H), 3.43 (q, $J = 5.3$ Hz, 2H), 1.87–1.68 (m, 4H), 1.50 (d, $J = 6.5$ Hz, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 166.6, 137.5, 132.9, 128.7, 128.3, 58.3, 39.5, 37.4, 26.7, 25.3.



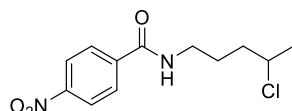
4-bromo-*N*-(4-chloropentyl)benzamide (2d)³

White solid obtained by column chromatography (PE/EA = 5:1 to 4:1); 53 mg, 87% yield; reaction time = 8 h. $R_f = 0.34$ (PE/EA = 3:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.64 (d, $J = 8.4$ Hz, 2H), 7.54 (d, $J = 8.4$ Hz, 2H), 6.51 (s, 1H), 4.09–4.02 (m, 1H), 3.47–3.42 (m, 2H), 1.86–1.71 (m, 4H), 1.51 (d, $J = 6.5$ Hz, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 166.7, 133.4, 131.7, 128.5, 126.0, 58.3, 39.5, 37.5, 26.8, 25.4.



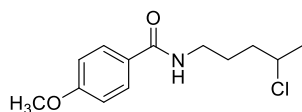
***N*-(4-Chloropentyl)-4-cyanobenzamide (2e)**³

White solid obtained by column chromatography (PE/EA = 4:1 to 3:1); 40 mg, 80% yield; reaction time = 8 h. $R_f = 0.15$ (PE/EA = 3:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.89 (d, $J = 8.3$ Hz, 2H), 7.73 (d, $J = 8.3$ Hz, 2H), 6.67 (s, $J = 6.0$ Hz, 1H), 4.12–4.03 (m, 1H), 3.48 (q, $J = 6.4$ Hz, 2H), 1.89–1.74 (m, 4H), 1.52 (d, $J = 6.6$ Hz, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 165.8, 138.5, 132.3, 127.6, 117.9, 114.8, 58.2, 39.6, 37.4, 26.6, 25.3.



***N*-(4-Chloropentyl)-4-nitrobenzamide (2f)**³

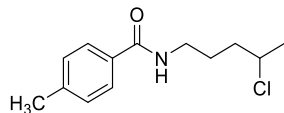
White solid obtained by column chromatography (PE/EA = 4:1 to 3:1); 24 mg, 51% yield; reaction time = 8 h. $R_f = 0.15$ (PE/EA = 3:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.29 (d, $J = 8.8$ Hz, 2H), 7.94 (d, $J = 8.9$ Hz, 2H), 6.41 (s, 1H), 4.13–4.05 (m, 1H), 3.52 (q, $J = 5.3$ Hz, 2H), 1.91–1.75 (m, 4H), 1.54 (d, $J = 6.5$ Hz, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 165.6, 149.5, 140.1, 128.1, 123.8, 58.3, 39.8, 37.4, 26.7, 25.4.



***N*-(4-Chloropentyl)-4-methoxybenzamide (2g)**³

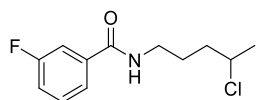
White solid obtained by column chromatography (PE/EA = 4:1 to 3:1); 42 mg, 82% yield; reaction time = 8 h. $R_f = 0.13$ (PE/EA = 3:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ

7.74 (d, $J = 8.8$ Hz, 2H), 6.92 (d, $J = 8.8$ Hz, 2H), 6.24 (s, 1H), 4.11–4.02 (m, 1H), 3.85 (s, 3H), 3.52–3.42 (m, 2H), 1.86–1.72 (m, 4H), 1.52 (d, $J = 6.6$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 167.1, 162.1, 128.6, 126.8, 113.7, 58.4, 55.4, 39.3, 37.5, 27.0, 25.4.



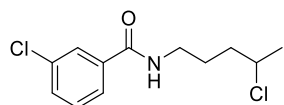
***N*-(4-Chloropentyl)-4-methylbenzamide (2h)³**

White solid obtained by column chromatography (PE/EA = 6:1 to 5:1); 43 mg, 91% yield; reaction time = 8 h. $R_f = 0.36$ (PE/EA = 3:1). ^1H NMR (400 MHz, CDCl_3) δ 7.68 (d, $J = 7.8$ Hz, 2H), 7.20 (d, $J = 7.8$ Hz, 2H), 6.55 (s, 1H), 4.08–4.01 (m, 1H), 3.49–3.41 (m, 2H), 2.38 (s, 3H), 1.86–1.68 (m, 4H), 1.50 (d, $J = 6.5$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 167.5, 141.7, 131.6, 129.1, 126.8, 58.3, 39.2, 37.5, 26.9, 25.3, 21.3.



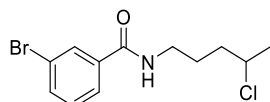
***N*-(4-Chloropentyl)-3-fluorobenzamide (2i)³**

Colorless oil obtained by column chromatography (PE/EA = 5:1); 42 mg, 86% yield; reaction time = 8 h. $R_f = 0.30$ (PE/EA = 3:1). ^1H NMR (400 MHz, CDCl_3) δ 7.52 (dd, $J_1 = 13.8$, $J_2 = 8.5$ Hz, 2H), 7.41–7.35 (m, 1H), 7.18 (t, $J = 8.3$ Hz, 1H), 6.56 (br, 1H), 4.10–4.03 (m, 1H), 3.49–3.42 (m, 2H), 1.86–1.73 (m, 4H), 1.51 (d, $J = 6.6$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 166.4, 162.7 (d, $J = 248.1$ Hz, 1C), 136.8 (d, $J = 6.7$ Hz, 1C), 130.2 (d, $J = 7.9$ Hz, 1C), 122.3, 118.4 (d, $J = 21.3$ Hz, 1C), 114.3 (d, $J = 22.9$ Hz, 1C), 58.3, 39.5, 37.4, 26.8, 25.3.



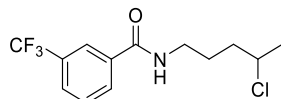
3-Chloro-*N*-(4-chloropentyl)benzamide (2j)³

Colorless oil obtained by column chromatography (PE/EA = 5:1); 48 mg, 92% yield; reaction time = 8 h. $R_f = 0.33$ (PE/EA = 3:1). ^1H NMR (400 MHz, CDCl_3) δ 7.76 (s, 1H), 7.64 (d, $J = 7.9$ Hz, 1H), 7.44 (d, $J = 8.0$ Hz, 1H), 7.33 (t, $J = 7.9$ Hz, 1H), 6.86 (s, 1H), 4.07–4.00 (m, 1H), 3.44 (q, $J = 6.3$ Hz, 2H), 1.87–1.70 (m, 4H), 1.50 (d, $J = 6.5$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 166.4, 136.3, 134.5, 131.3, 129.8, 127.2, 125.0, 58.3, 39.5, 37.4, 26.7, 25.3.



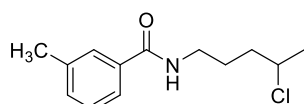
3-Bromo-*N*-(4-chloropentyl)benzamide (2k)³

Colorless oil obtained by column chromatography (PE/EA = 5:1); 55 mg, 92% yield; reaction time = 8 h. $R_f = 0.33$ (PE/EA = 3:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.91 (s, 1H), 7.69 (d, $J = 7.8$ Hz, 1H), 7.61 (d, $J = 7.9$ Hz, 1H), 7.30 (t, $J = 16.0$ Hz, 1H), 6.47 (s, 1H), 4.11–4.03 (m, 1H), 3.51–3.43 (m, 2H), 1.87–1.72 (m, 4H), 1.52 (d, $J = 6.5$ Hz, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 166.3, 136.5, 134.3, 130.1, 130.0, 125.5, 122.6, 58.3, 39.6, 37.4, 26.7, 25.3.



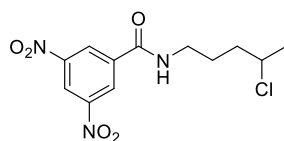
***N*-(4-Chloropentyl)-3-(trifluoromethyl)benzamide (2l)³**

Colorless oil obtained by column chromatography (PE/EA = 5:1); 51 mg, 87% yield; reaction time = 8 h. $R_f = 0.32$ (PE/EA = 3:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.03 (s, 1H), 7.96 (d, $J = 7.7$ Hz, 1H), 7.74 (d, $J = 7.8$ Hz, 1H), 7.55 (t, $J = 7.8$ Hz, 1H), 6.66 (s, 1H), 4.10–4.03 (m, 1H), 3.49 (q, $J = 6.7$ Hz, 3H), 1.89–1.73 (m, 4H), 1.52 (d, $J = 6.5$ Hz, 2H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 166.2, 135.3, 131.0 (d, $J = 33.0$ Hz, 1C), 129.7 (d, $J = 104.0$ Hz, 1C), 128.0 (d, $J = 3.6$ Hz, 1C), 125.0, 123.9 (d, $J = 3.9$ Hz, 1C), 122.3, 58.3, 39.6, 37.4, 26.8, 25.3.



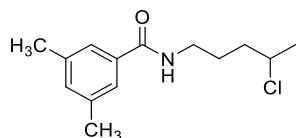
***N*-(4-Chloropentyl)-3-methylbenzamide (2m)³**

Colorless oil obtained by column chromatography (PE/EA = 6:1 to 5:1); 43 mg, 90% yield; reaction time = 8 h. $R_f = 0.35$ (PE/EA = 3:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.60 (s, 1H), 7.56–7.54 (m, 1H), 7.29 (d, $J = 5.0$ Hz, 2H), 6.56 (s, 1H), 4.10–4.01 (m, 1H), 3.50–3.41 (m, 2H), 2.36 (s, 3H), 1.85–1.70 (m, 4H), 1.50 (d, $J = 6.6$ Hz, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 167.8, 138.3, 134.5, 132.0, 128.3, 127.6, 123.8, 58.3, 39.3, 37.4, 26.8, 25.3, 21.2.



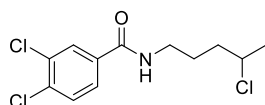
***N*-(4-Chloropentyl)-3,5-dinitrobenzamide (2n)³**

White solid obtained by column chromatography (PE/EA = 4:1 to 3:1); 22 mg, 35% yield; reaction time = 8 h. $R_f = 0.13$ (PE/EA = 3:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 9.16 (t, $J = 2.1$ Hz, 1H), 9.00 (d, $J = 2.1$ Hz, 2H), 6.92 (t, $J = 6.0$ Hz, 1H), 4.13–4.05 (m, 1H), 3.59 (q, $J = 6.3$ Hz, 2H), 1.96–1.77 (m, 4H), 1.54 (d, $J = 6.5$ Hz, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 162.9, 148.6, 137.8, 127.2, 121.1, 58.2, 40.2, 37.4, 26.6, 25.4.



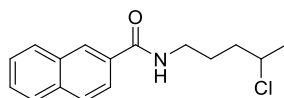
***N*-(4-Chloropentyl)-3,5-dimethylbenzamide (2o)³**

Colorless oil obtained by column chromatography (PE/EA = 6:1); 43 mg, 85% yield; reaction time = 8 h. $R_f = 0.36$ (PE/EA = 3:1). ^1H NMR (400 MHz, CDCl_3) δ 7.37 (s, 2H), 7.11 (s, 1H), 6.38 (s, 1H), 4.09–4.02 (m, 1H), 3.50–3.41 (m, 2H), 2.33 (s, 6H), 1.85–1.70 (m, 4H), 1.51 (d, $J = 6.5$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 167.9, 138.1, 134.5, 132.9, 124.6, 58.3, 39.3, 37.5, 26.9, 25.3, 21.1.



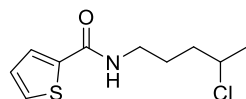
***N*-(4-Chloropentyl)-3,5-dichlorobenzamide (2p)³**

Colorless oil obtained by column chromatography (PE/EA = 4:1 to 3:1); 52 mg, 89% yield; reaction time = 6 h. $R_f = 0.30$ (PE/EA = 3:1). ^1H NMR (400 MHz, CDCl_3) δ 7.85 (d, $J = 2.0$ Hz, 1H), 7.60 (d, $J = 8.4$ Hz, 1H), 7.44 (d, $J = 8.3$ Hz, 1H), 7.24 (s, 1H), 4.05–3.99 (m, 1H), 3.42 (q, $J = 6.1$ Hz, 2H), 1.87–1.70 (m, 4H), 1.50 (d, $J = 6.6$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 165.6, 135.7, 134.3, 132.8, 130.4, 129.1, 126.1, 58.2, 39.6, 37.4, 26.6, 25.3.



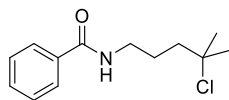
***N*-(4-Chloropentyl)-2-naphthamide (2q)³**

Colorless oil obtained by column chromatography (PE/EA = 5:1); 42 mg, 76% yield; reaction time = 8 h. $R_f = 0.31$ (PE/EA = 3:1). ^1H NMR (400 MHz, CDCl_3) δ 8.28 (s, 1H), 7.85–7.80 (m, 4H), 7.54–7.45 (m, 2H), 6.93 (t, $J = 5.8$ Hz, 1H), 4.04–3.96 (m, 1H), 3.47 (q, $J = 5.8$ Hz, 2H), 1.86–1.69 (m, 4H), 1.46 (d, $J = 6.5$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 167.7, 134.5, 132.4, 131.7, 128.8, 128.2, 127.6, 127.5, 127.3, 126.6, 123.5, 58.4, 39.5, 37.4, 26.8, 25.3.



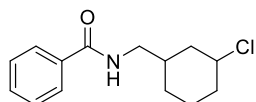
***N*-(4-Chloropentyl)thiophene-2-carboxamide (2r)³**

Colorless oil obtained by column chromatography (PE/EA = 5:1 to 4:1); 35 mg, 81% yield; reaction time = 8 h. $R_f = 0.30$ (PE/EA = 3:1). ^1H NMR (400 MHz, CDCl_3) δ 7.58 (dd, $J_1 = 3.8$ Hz, $J_2 = 1.2$ Hz, 1H), 7.46 (dd, $J_1 = 5.0$ Hz, $J_2 = 1.1$ Hz, 1H), 7.06 (dd, $J_1 = 5.0$ Hz, $J_2 = 3.7$ Hz, 1H), 6.71 (t, $J = 8.0$ Hz, 1H), 4.09–3.99 (m, 1H), 3.49–3.88 (m, 2H), 1.85–1.70 (m, 4H), 1.49 (d, $J = 6.6$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 162.1, 139.0, 129.8, 128.0, 127.6, 58.3, 39.3, 37.4, 26.8, 25.3.



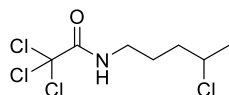
***N*-(4-Chloro-4-methylpentyl)benzamide (2s)⁴**

Colorless oil obtained by column chromatography (PE/EA = 6:1); 44 mg, 92% yield; reaction time = 8 h. $R_f = 0.37$ (PE/EA = 3:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.79 (d, $J = 7.0$ Hz, 2H), 7.48 (t, $J = 7.4$ Hz, 1H), 7.40 (t, $J = 7.5$ Hz, 2H), 6.71 (s, 1H), 3.45 (q, $J = 6.2$ Hz, 2H), 1.85–1.75 (m, 4H), 1.56 (s, 6H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 167.6, 134.5, 131.3, 128.4, 126.8, 70.6, 43.0, 39.8, 32.3, 25.4.



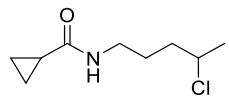
***N*-((3-Chlorocyclohexyl)methyl)benzamide (2t)³**

Colorless oil obtained by column chromatography (PE/EA = 6:1 to 5:1); 31 mg, 51% yield; reaction time = 8 h. $dr = 2:1$. $R_f = 0.25$ (PE/EA = 3:1); $^1\text{H NMR}$ (major) (400 MHz, CDCl_3) δ 7.77 (d, $J = 7.3$ Hz, 2H), 7.49 (t, $J = 7.3$ Hz, 1H), 7.42 (t, $J = 7.4$ Hz, 2H), 6.61 (s, 1H), 4.53–4.50 (m, 1H), 3.40–3.23 (m, 1H), 2.28–2.09 (m, 1H), 2.08–2.01 (m, 1H), 1.98–1.90 (m, 1H), 1.84–1.67 (m, 3H), 1.63–1.49 (m, 1H), 1.54–1.49 (m, 1H), 1.36–1.25 (m, 1H), 1.10–0.88 (m, 1H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 167.7, 134.6, 131.4, 128.5, 126.9, 126.8, 59.2, 59.0, 45.4, 45.0, 41.4, 38.2, 38.1, 37.1, 33.9, 32.4, 29.7, 29.1, 25.2, 19.7.



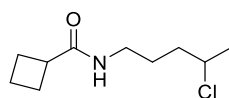
2,2,2-Trichloro-*N*-(4-chloropentyl)acetamide (2u)³

Colorless oil obtained by column chromatography (PE/EA = 50:1 to 30:1); 34 mg, 64% yield; reaction time = 8 h. $R_f = 0.50$ (PE/EA = 5:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 6.76 (s, 1H), 4.18–3.97 (m, 1H), 3.52–3.27 (m, 2H), 2.00–1.68 (m, 4H), 1.53 (d, $J = 6.6$ Hz, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 162.0, 92.5, 58.0, 40.8, 37.1, 26.2, 25.4.



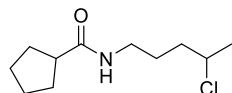
***N*-(4-Chloropentyl)cyclopropanecarboxamide (2v)³**

Light yellow oil obtained by column chromatography (PE/EA = 3:1); 32 mg, 82% yield; reaction time = 8 h. $R_f = 0.18$ (PE/EA = 3:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 6.01 (s, 1H), 4.06–3.98 (m, 1H), 3.26 (q, $J = 5.5, 4.6$ Hz, 2H), 1.76–1.57 (m, 4H), 1.49 (d, $J = 6.5$ Hz, 3H), 1.37–1.30 (m, 1H), 0.94–0.90 (m, 2H), 0.72–0.68 (m, 2H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 173.6, 58.4, 39.0, 37.4, 26.9, 25.3, 14.6, 7.0.



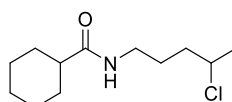
***N*-(4-Chloropentyl)cyclobutanecarboxamide (2w)**³

Colorless oil obtained by column chromatography (PE/EA = 3:1); 32 mg, 78% yield; reaction time = 6 h. $R_f = 0.18$ (PE/EA = 3:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 5.52 (s, 1H), 4.07–3.99 (m, 1H), 3.30–3.19 (m, 2H), 3.01–2.93 (m, 1H), 2.29–2.20 (m, 2H), 2.16–2.08 (m, 2H), 1.96–1.82 (m, 2H), 1.74–1.58 (m, 4H), 1.51 (d, $J = 6.5$ Hz, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 175.0, 58.3, 39.9, 38.7, 37.4, 26.9, 25.4, 25.3, 18.1.



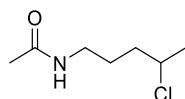
***N*-(4-Chloropentyl)cyclobutanecarboxamide (2x)**³

Colorless oil obtained by column chromatography (PE/EA = 3:1); 33 mg, 75% yield; reaction time = 8 h. $R_f = 0.18$ (PE/EA = 3:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 5.64 (s, 1H), 4.07–3.99 (m, 1H), 3.30–3.19 (m, 2H), 2.52–2.44 (m, 1H), 1.87–1.48 (m, 15H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 176.3, 58.4, 45.8, 38.7, 37.4, 30.4 (2C), 27.0, 25.9, 25.4.



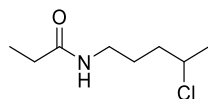
***N*-(4-Chloropentyl)cyclohexanecarboxamide (2y)**³

Colorless oil obtained by column chromatography (PE/EA = 3:1); 35 mg, 76% yield; reaction time = 8 h. $R_f = 0.20$ (PE/EA = 3:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 5.58 (s, 1H), 4.07–3.99 (m, 1H), 3.32–3.21 (m, 2H), 2.09–2.01 (m, 1H), 1.85–1.60 (m, 9H), 1.49 (d, $J = 6.6$ Hz, 3H), 1.45–1.37 (m, 2H), 1.29–1.17 (m, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 176.1, 58.3, 45.6, 38.6, 37.4, 29.7 (2C), 26.9, 25.7, 25.4.



***N*-(4-Chloropentyl)acetamide (2z)**³

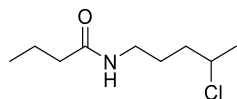
Light yellow oil obtained by column chromatography (PE/EA = 1:1 to 1:3); 29 mg, 89% yield; reaction time = 8 h. $R_f = 0.22$ (PE/EA = 1:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 6.06 (s, 1H), 4.09–4.01 (m, 1H), 3.27 (q, $J = 5.7$ Hz, 2H), 1.99 (s, 3H), 1.77–1.61 (m, 4H), 1.52 (d, $J = 6.6$ Hz, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 170.2, 58.3, 38.9, 37.3, 26.7, 25.3, 23.2.



***N*-(4-Chloropentyl)propionamide (2aa)**³

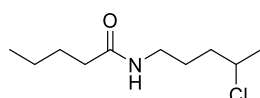
Light yellow oil obtained by column chromatography (PE/EA = 2:1 to 1:1); 30 mg, 84% yield; reaction time = 8 h. $R_f = 0.11$ (PE/EA = 3:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 5.75 (s, 1H), 4.06–3.98 (m, 1H), 3.25 (q, $J = 6.3$, 2H), 2.18 (q, $J = 7.6$ Hz, 2H), 1.74–

1.58 (m, 4H), 1.49 (d, $J = 6.5$ Hz, 3H), 1.13 (t, $J = 7.6$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 173.9, 58.3, 38.8, 37.4, 29.7, 26.8, 25.3, 9.9.



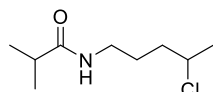
***N*-(4-Chloropentyl)butyramide (2ab)**³

Light yellow oil obtained by column chromatography (PE/EA = 3:1); 32 mg, 84% yield; reaction time = 8 h. $R_f = 0.10$ (PE/EA = 3:1). ^1H NMR (400 MHz, CDCl_3) δ 5.75 (s, 1H), 4.06–3.98 (m, 1H), 3.30–3.21 (m, 2H), 2.13 (t, $J = 7.5$ Hz, 2H), 1.74–1.48 (m, 6H), 1.48 (d, $J = 6.6$ Hz, 3H), 0.92 (t, $J = 7.4$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 173.1, 58.3, 38.7, 38.6, 37.4, 26.9, 25.3, 19.1, 13.7.



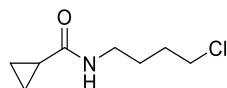
***N*-(4-Chloropentyl)pentanamide (2ac)**³

Light yellow oil obtained by column chromatography (PE/EA = 3:1); 32 mg, 78% yield; reaction time = 8 h. $R_f = 0.15$ (PE/EA = 3:1). ^1H NMR (400 MHz, CDCl_3) δ 5.60 (s, 1H), 4.07–3.99 (m, 1H), 3.29–3.23 (m, 2H), 2.16 (t, $J = 8.0$ Hz, 2H), 1.75–1.56 (m, 6H), 1.50 (d, $J = 6.6$ Hz, 3H), 1.38–1.28 (m, 2H), 0.90 (t, $J = 7.3$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 173.2, 58.3, 38.7, 37.4, 36.5, 27.8, 26.9, 25.4, 22.4, 13.8.



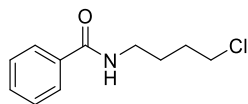
***N*-(4-Chloropentyl)isobutyramide (2ad)**³

Colorless oil obtained by column chromatography (PE/EA = 3:1); 27 mg, 70% yield; reaction time = 8 h. $R_f = 0.20$ (PE/EA = 3:1). ^1H NMR (400 MHz, CDCl_3) δ 5.63 (s, 1H), 4.07–3.99 (m, 1H), 3.32–3.19 (m, 2H), 2.38–2.28 (m, 1H), 1.76–1.59 (m, 4H), 1.46 (d, $J = 6.5$ Hz, 3H), 1.10 (d, $J = 6.9$ Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ 177.0, 58.4, 38.6, 37.4, 35.6, 26.9, 25.4, 19.6 (2C).



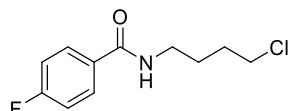
***N*-(4-Chlorobutyl)cyclopropanecarboxamide (2ae)**³

Colorless oil obtained by column chromatography (PE/EA = 3:1); 18 mg, 51% yield; reaction time = 16 h. $R_f = 0.10$ (PE/EA = 3:1). ^1H NMR (400 MHz, CDCl_3) δ 5.89 (s, 1H), 3.55 (t, $J = 6.4$ Hz, 2H), 3.28 (q, $J = 6.7$ Hz, 2H), 1.83–1.76 (m, 2H), 1.69–1.62 (m, 2H), 1.36–1.30 (m, 1H), 0.95–0.91 (m, 2H), 0.73–0.69 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 173.6, 44.6, 38.8, 29.8, 27.1, 14.6, 7.0.



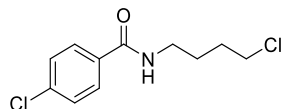
N-(4-chlorobutyl)benzamide (2af)⁵

Colorless oil obtained by column chromatography (PE/EA = 5:1); 28 mg, 66% yield; reaction time = 16 h. $R_f = 0.31$ (PE/EA = 3:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.79–7.76 (m, 2H), 7.49 (t, $J = 7.3$ Hz, 1H), 7.42 (t, $J = 7.4$ Hz, 2H), 6.46 (s, 1H), 3.58 (t, $J = 6.3$ Hz, 2H), 3.48 (q, $J = 5.3$ Hz, 2H), 1.89–1.73 (m, 4H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 167.6, 134.5, 131.4, 128.5, 126.8, 44.6, 39.2, 29.8, 27.0.



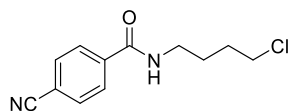
N-(4-Chlorobutyl)-4-fluorobenzamide (2ag)³

White solid obtained by column chromatography (PE/EA = 6:1 to 5:1); 32 mg, 70% yield; reaction time = 16 h. $R_f = 0.22$ (PE/EA = 3:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.81–7.76 (m, 2H), 7.09 (t, $J = 8.6$ Hz, 2H), 6.55 (s, 1H), 3.58 (t, $J = 6.3$ Hz, 2H), 3.46 (q, $J = 5.3$ Hz, 2H), 1.89–1.73 (m, 4H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 166.6, 164.6 (d, $J = 251.9$ Hz, 1C), 130.6 (d, $J = 3.1$ Hz, 1C), 129.2 (d, $J = 8.8$ Hz, 1C), 115.5 (d, $J = 21.9$ Hz, 1C), 44.6, 39.3, 29.8, 27.0.



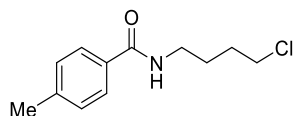
4-Chloro-N-(4-chlorobutyl)benzamide (2ah)³

White solid obtained by column chromatography (PE/EA = 6:1 to 4:1); 36 mg, 73% yield; reaction time = 16 h. $R_f = 0.31$ (PE/EA = 3:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.70 (d, $J = 8.5$ Hz, 2H), 7.37 (d, $J = 8.5$ Hz, 2H), 6.68 (s, 1H), 3.57 (t, $J = 6.2$ Hz, 2H), 3.45 (q, $J = 6.5$ Hz, 2H), 1.87–1.72 (m, 4H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 166.7, 137.6, 132.8, 128.7, 128.3, 44.5, 39.3, 29.8, 26.9.



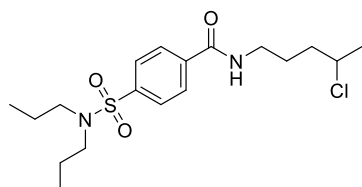
N-(4-Chlorobutyl)-4-cyanobenzamide (2ai)³

White solid obtained by column chromatography (PE/EA = 4:1 to 3:1); 34 mg, 72% yield; reaction time = 16 h. $R_f = 0.19$ (PE/EA = 3:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.88 (d, $J = 8.4$ Hz, 2H), 7.74 (d, $J = 8.4$ Hz, 2H), 6.52 (s, 1H), 3.60 (t, $J = 6.1$ Hz, 2H), 3.51 (q, $J = 6.5$ Hz, 2H), 1.89–1.78 (m, 4H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 165.8, 138.4, 132.4, 127.6, 118.0, 114.9, 44.5, 39.5, 29.7, 26.8.

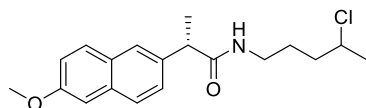


***N*-(4-Chlorobutyl)-4-methylbenzamide (2aj)**³

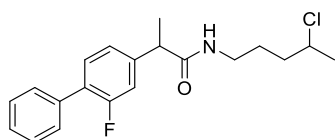
Colorless oil obtained by column chromatography (PE/EA = 6:1 to 5:1); 24 mg, 53% yield; reaction time = 16 h. $R_f = 0.22$ (PE/EA = 3:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.66 (d, $J = 8.2$ Hz, 2H), 7.21 (d, $J = 7.9$ Hz, 2H), 6.38 (s, 1H), 3.57 (t, $J = 6.3$ Hz, 2H), 3.47 (q, $J = 6.6$ Hz, 2H), 2.39 (s, 3H), 1.88–1.82 (m, 2H), 1.80–1.71 (m, 2H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 167.6, 141.8, 131.7, 129.2, 126.8, 44.6, 39.1, 29.9, 27.1, 21.4.

***N*-(4-Chloropentyl)-4-(*N,N*-dipropylsulfamoyl)benzamide (2ak)**

Colorless oil obtained by column chromatography (PE/EA = 5:1); 63 mg, 81% yield; reaction time = 8 h. $R_f = 0.13$ (PE/EA = 3:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.87 (d, $J = 8.4$ Hz, 2H), 7.74 (d, $J = 8.4$ Hz, 2H), 6.95 (t, $J = 5.8$ Hz, 1H), 4.11–4.03 (m, 1H), 3.47 (q, $J = 6.2$ Hz, 2H), 3.06 (t, $J = 8.0$ Hz, 1H, 3H), 1.89–1.73 (m, 4H), 1.58–1.48 (m, 7H), 0.86 (t, $J = 7.4$ Hz, 6H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 166.4, 142.2, 138.3, 127.7, 127.0, 58.3, 49.8, 39.6, 37.4, 26.6, 25.3, 21.8, 11.1. IR (KBr, cm^{-1}) ν 3326, 3073, 2968, 1645, 1543, 1459, 1339, 1148, 1090, 993, 739, 602, 452. HRMS (ESI-TOF) calcd for $\text{C}_{18}\text{H}_{30}\text{ClN}_2\text{O}_3\text{S}$ $[\text{M}+\text{H}]^+$ 389.1660, found 389.1660.

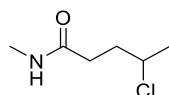
**(2*S*)-*N*-(4-Chloropentyl)-2-(6-methoxynaphthalen-2-yl)propanamide (2al)**³

White solid obtained by column chromatography (PE/EA = 5:1); 44 mg, 66% yield; reaction time = 8 h. $R_f = 0.30$ (PE/EA = 3:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.16 (d, $J = 8.8$ Hz, 1H), 7.71 (d, $J = 9.5$ Hz, 2H), 7.49 (dd, $J_1 = 8.8$ Hz, $J_2 = 1.8$ Hz, 1H), 7.29–7.27 (m, 1H), 5.66 (t, $J = 5.9$ Hz, 1H), 4.01 (s, 3H), 3.69 (q, $J = 7.1$ Hz, 1H), 3.17 (q, $J = 6.9$ Hz, 2H), 1.59 (d, $J = 7.1$ Hz, 2H), 1.42–1.35 (m, 2H), 1.25–1.13 (m, 4H), 0.81 (t, $J = 7.1$ Hz, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 173.8, 152.4, 137.4, 130.9, 129.4, 127.7, 127.4, 126.1, 124.0, 116.6, 113.9, 56.8, 46.7, 39.6, 29.1, 28.8, 22.2, 18.5, 13.8.

***N*-(4-Chloropentyl)-2-(2-fluoro-[1,1'-biphenyl]-4-yl)propanamide (2am)**

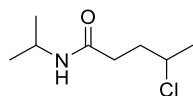
Colorless oil obtained by column chromatography (PE/EA = 5:1); 43 mg, 62% yield; reaction time = 8 h. $R_f = 0.14$ (PE/EA = 3:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.53 (d, J

= 8.0 Hz, 2H), 7.46–7.35 (m, 4H), 7.16–7.12 (m, 2H), 5.64 (s, 1H), 4.02–3.94 (m, 1H), 3.57 (q, $J = 7.1$ Hz, 1H), 3.32–3.21 (m, 2H), 1.71–1.58 (m, 4H), 1.54 (d, $J = 7.1$ Hz, 3H), 1.46 (dd, $J_1 = 6.5$, $J_2 = 3.1$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 173.5, 161.0, 158.47, 142.7 (t, $J = 4.0$ Hz, 1C), 135.3, 131.0 (d, $J = 4.0$ Hz, 1C), 128.9 (d, $J = 2.9$ Hz, 1C), 128.4, 127.8 (d, $J = 40.0$ Hz, 1C), 123.5 (d, $J = 3.3$ Hz, 1C), 115.2 (d, $J = 23.6$ Hz, 1C), 58.2 (d, $J = 4.3$ Hz, 1C), 46.6, 39.0 (d, $J = 7.6$ Hz, 1C), 37.3 (d, $J = 6.7$ Hz, 1C), 26.7, 25.3, 18.4 (d, $J = 2.6$ Hz, 1C). IR (KBr, cm^{-1}) ν 3295, 3077, 2971, 2932, 1645, 1553, 1418, 1292, 1230, 1132, 928, 767, 698. HRMS (ESI-TOF) calcd for $\text{C}_{20}\text{H}_{24}\text{ClFNO}$ $[\text{M}+\text{H}]^+$ 348.1525, found 348.1524.



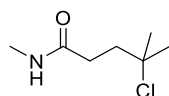
4-Chloro-*N*-methylpentanamide (2an)⁶

Colorless oil obtained by column chromatography (PE/EA = 2:1 to 1:1); 18 mg, 60% yield; reaction time = 16 h. $R_f = 0.25$ (PE/EA = 1:1). ^1H NMR (400 MHz, CDCl_3) δ 5.92 (s, 1H), 4.08–4.00 (m, 1H), 2.78 (d, $J = 4.7$ Hz, 3H), 2.43–2.28 (m, 2H), 2.19–2.11 (m, 1H), 1.91–1.82 (m, 1H), 1.50 (d, $J = 6.6$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 172.6, 58.4, 35.6, 33.3, 26.3, 25.4.



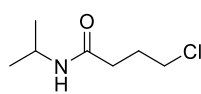
4-Chloro-*N*-isopropylpentanamide (2ao)³

Colorless oil obtained by column chromatography (PE/EA = 6:1 to 5:1); 22 mg, 62% yield; reaction time = 16 h. $R_f = 0.50$ (PE/EA = 1:1). ^1H NMR (400 MHz, CDCl_3) δ 5.60 (s, 1H), 4.08–3.99 (m, 2H), 2.37–2.22 (m, 2H), 2.17–2.07 (m, 1H), 1.90–1.81 (m, 1H), 1.49 (d, $J = 6.6$ Hz, 3H), 1.11 (d, $J = 6.6$ Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ 170.9, 58.3, 41.2, 35.7, 33.6, 25.4, 22.7, 22.6.



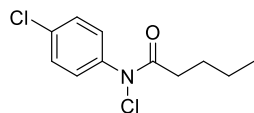
4-Chloro-*N*,4-dimethylpentanamide (2ap)⁶

Colorless oil obtained by column chromatography (PE/EA = 2:1 to 1:1); 24 mg, 73% yield; reaction time = 16 h. $R_f = 0.32$ (PE/EA = 1:1). ^1H NMR (400 MHz, CDCl_3) δ 5.92 (s, 1H), 2.78 (d, $J = 4.8$ Hz, 3H), 2.39 (t, $J = 8.0$ Hz, 2H), 2.07 (t, $J = 8.0$ Hz, 2H), 1.55 (s, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ 173.0, 70.4, 41.0, 32.3, 26.3, 22.3.



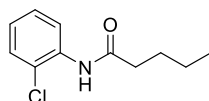
4-Chloro-*N*-isopropylbutanamide (2aq)³

Colorless oil obtained by column chromatography (PE/EA = 3:1); 10 mg, 31% yield; reaction time = 16 h. $R_f = 0.50$ (PE/EA = 1:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 5.66 (s, 1H), 4.08–3.99 (m, 1H), 3.56 (t, $J = 6.2$ Hz, 2H), 2.28 (t, $J = 7.2$ Hz, 2H), 2.10–2.03 (m, 2H), 1.11 (d, $J = 6.6$ Hz, 6H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 170.7, 44.5, 41.2, 33.3, 28.1, 22.6.



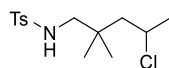
***N*-Chloro-*N*-(4-chlorophenyl)pentanamide (3ar-1)**

Colorless oil obtained by column chromatography (PE/EA = 80:1); 10 mg, 20% yield. $R_f = 0.79$ (PE/EA = 5:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.42 (d, $J = 8.8$ Hz, 2H), 7.34 (d, $J = 8.7$ Hz, 2H), 2.29 (s, 2H), 1.66–1.58 (m, 2H), 1.33–1.24 (m, 2H), 0.86 (t, $J = 7.4$ Hz, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 171.0, 143.1, 129.7, 129.4, 128.2, 33.9, 27.5, 22.0, 13.5. IR (KBr, cm^{-1}) ν 3341, 2959, 2872, 1694, 1592, 1489, 1274, 1182, 1090, 697, 526. HRMS (ESI-TOF) calcd for $\text{C}_{12}\text{H}_{14}\text{Cl}_2\text{NO}$ $[\text{M}+\text{H}]^+$ 246.0447, found 246.0444.



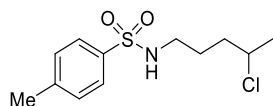
***N*-(2-Chlorophenyl)pentanamide (3ar-2)**

Colorless oil obtained by column chromatography (PE/EA = 80:1); 4 mg, 10% yield. $R_f = 0.78$ (PE/EA = 5:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.37 (d, $J = 8.3$ Hz, 1H), 7.64 (s, 1H), 7.35 (dd, $J_1 = 8.1$ Hz, $J_2 = 1.6$ Hz, 1H), 7.27–7.24 (m, 1H), 7.04–7.00 (m, 1H), 2.43 (t, $J = 7.6$ Hz, 2H), 1.77–1.69 (m, 2H), 1.47–1.38 (m, 2H), 0.96 (t, $J = 7.3$ Hz, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 171.3, 134.6, 128.9, 127.7, 124.4, 122.5, 121.6, 37.7, 27.5, 22.3, 13.7. $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 171.0, 143.1, 129.7, 129.4, 128.2, 33.9, 27.5, 22.0, 13.5. IR (KBr, cm^{-1}) ν 3283, 2960, 1661, 1585, 1527, 1441, 1288, 1188, 1034, 757, 678. HRMS (ESI-TOF) calcd for $\text{C}_{11}\text{H}_{14}\text{Cl}_2\text{NO}$ $[\text{M}+\text{H}]^+$ 212.0837, found 212.0842.



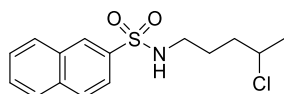
***N*-(4-Chloropentyl)benzenesulfonamide (5a)⁷**

Colorless oil obtained by column chromatography (PE/EA = 12:1 to 8:1); 38 mg, 73% yield; reaction time = 3 h. $R_f = 0.22$ (PE/EA = 5:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.88 (d, $J = 7.1$ Hz, 2H), 7.59 (t, $J = 7.4$ Hz, 1H), 7.53 (t, $J = 7.4$ Hz, 2H), 5.11 (t, $J = 6.1$ Hz, 1H), 4.99–3.91 (m, 1H), 2.97 (q, $J = 6.3$ Hz, 2H), 1.79–1.55 (m, 4H), 1.45 (d, $J = 6.5$ Hz, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 143.2, 136.9, 129.6, 126.9, 54.8, 52.6, 48.8, 34.1, 27.7, 25.9, 25.1, 21.4.



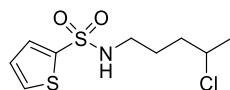
***N*-(4-Chloropentyl)-4-methylbenzenesulfonamide (5b)⁷**

Colorless oil obtained by column chromatography (PE/EA = 12:1 to 8:1); 39 mg, 71% yield; reaction time = 3 h. R_f = 0.23 (PE/EA = 5:1). $^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.76 (d, J = 8.0 Hz, 2H), 7.31 (d, J = 7.9 Hz, 2H), 5.00 (t, J = 6.3 Hz, 1H), 3.98–3.92 (m, 1H), 2.95 (q, J = 6.4 Hz, 2H), 2.43 (s, 3H), 1.76–1.57 (m, 4H), 1.45 (d, J = 6.6 Hz, 3H). $^{13}\text{C NMR}$ (150 MHz, CDCl_3) δ 143.4, 136.8, 129.7, 127.0, 58.0, 42.5, 36.9, 26.6, 25.3, 21.4.



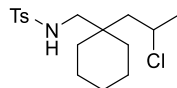
***N*-(4-Chloropentyl)naphthalene-2-sulfonamide (5c)⁷**

White solid obtained by column chromatography (PE/EA = 8:1 to 6:1); 40 mg, 64% yield; reaction time = 3 h. R_f = 0.15 (PE/EA = 5:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.46 (s, 1H), 7.97–7.95 (m, 2H), 7.91–7.86 (m, 2H), 7.66–7.58 (m, 2H), 5.21 (t, J = 6.2 Hz, 1H), 3.96–3.88 (m, 1H), 3.00 (q, J = 6.2 Hz, 2H), 1.75–1.56 (m, 4H), 1.40 (d, J = 6.6 Hz, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 136.5, 134.7, 132.1, 129.5, 129.1, 128.8, 128.4, 127.8, 127.5, 122.2, 58.0, 42.6, 36.9, 26.6, 25.2.



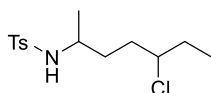
***N*-(4-Chloropentyl)thiophene-2-sulfonamide (5d)⁷**

Colorless oil obtained by column chromatography (PE/EA = 10:1 to 8:1); 36 mg, 67% yield; reaction time = 3 h. R_f = 0.23 (PE/EA = 5:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.63–7.60 (m, 2H), 7.12–7.10 (m, 1H), 4.92 (t, J = 6.3 Hz, 1H), 4.03–3.95 (m, 1H), 3.07 (q, J = 6.3 Hz, 2H), 1.80–1.61 (m, 4H), 1.49 (d, J = 6.6 Hz, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 140.6, 132.1, 131.9, 127.4, 58.0, 42.8, 36.9, 26.5, 25.3.



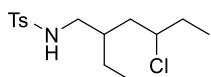
***N*-((1-(2-Chloropropyl)cyclohexyl)methyl)-4-methylbenzenesulfonamide (5e)³**

White solid obtained by column chromatography (PE/EA = 30:1 to 20:1); 47 mg, 70% yield; reaction time = 3 h. R_f = 0.43 (PE/EA = 5:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.74 (d, J = 8.1 Hz, 2H), 7.31 (d, J = 7.9 Hz, 2H), 5.04 (dd, J_1 = 9.4, J_2 = 5.1 Hz, 1H), 4.12–4.05 (m, 1H), 2.98 (dd, J_1 = 12.9, J_2 = 9.4 Hz, 1H), 2.80 (dd, J_1 = 13.0, J_2 = 5.1 Hz, 1H), 2.43 (s, 3H), 1.85 (dd, J_1 = 16.0, J_1 = 9.4 Hz, 1H), 1.69 (d, J = 15.9 Hz, 1H), 1.49 (d, J = 6.6 Hz, 3H), 1.50–1.29 (m, 10H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 143.2, 136.8, 129.6, 126.9, 54.4, 48.4, 45.7, 36.1, 33.9, 33.5, 27.9, 25.8, 21.4, 21.2, 21.0.



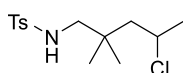
***N*-(5-Chloroheptan-2-yl)-4-methylbenzenesulfonamide (5f)**

Colorless oil obtained by column chromatography (PE/EA = 12:1 to 10:1); 45 mg, 74% yield; dr = 1:1; reaction time = 3 h. R_f = 0.29 (PE/EA = 5:1). ^1H NMR (400 MHz, CDCl_3) δ 7.78 (d, J = 8.2 Hz, 2H), 7.31 (d, J = 8.1 Hz, 2H), 4.78 (dd, J_1 = 16.0, J_2 = 8.5 Hz, 1H), 3.74–3.68 (m, 1H), 3.37–3.24 (m, 1H), 2.43 (s, 3H), 1.74–1.37 (m, 6H), 1.0 (dd, J_1 = 6.6, J_2 = 3.4 Hz, 3H), 0.96 (t, J = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 143.3, 138.1, 129.6, 127.0, 65.5, 65.0, 50.0, 49.2, 34.5, 34.2, 34.1, 33.6 (2C), 31.5, 31.4, 21.8 (2C), 21.5, 10.8 (2C). IR (KBr, cm^{-1}) ν 3284, 2971, 1638, 1431, 1382, 1322, 1161, 1094, 990, 815, 665, 581, 552. HRMS (ESI-TOF) calcd for $\text{C}_{14}\text{H}_{22}\text{ClNO}_2\text{SNa}$ [$\text{M}+\text{Na}$] $^+$ 326.0952, found 326.0957.



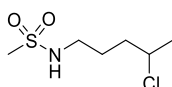
***N*-(4-Chloro-2-ethylhexyl)-4-methylbenzenesulfonamide (5g)**

Colorless oil obtained by column chromatography (PE/EA = 12:1 to 10:1); 46 mg, 72% yield; dr = 1:1; reaction time = 3 h. R_f = 0.34 (PE/EA = 5:1). ^1H NMR (400 MHz, CDCl_3) δ 7.75 (d, J = 8.0 Hz, 2H), 7.29 (d, J = 8.0 Hz, 2H), 5.16–5.07 (m, 1H), 3.85–3.84 (m, 1H), 2.94–2.86 (m, 2H), 2.40 (s, 3H), 1.77–1.50 (m, 5H), 1.44–1.18 (m, 2H), 0.96 (t, J = 8.0 Hz, 3H), 0.79 (q, J = 8.0 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 143.3 (2C), 136.7, 136.6, 129.6 (2C), 127.0 (2C), 63.3 (2C), 45.8, 44.1, 39.9 (2C), 36.4 (2C), 31.9 (2C), 24.6, 22.8, 21.4, 10.9, 10.8, 10.7, 10.2. IR (KBr, cm^{-1}) ν 3284, 2966, 2933, 2878, 1599, 1460, 1325, 1160, 1094, 907, 815, 665, 551. HRMS (ESI-TOF) calcd for $\text{C}_{15}\text{H}_{25}\text{ClNO}_2\text{S}$ [$\text{M}+\text{H}$] $^+$ 318.1289, found 318.1297.



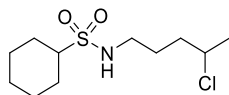
***N*-(4-Chloro-2,2-dimethylpentyl)-4-methylbenzenesulfonamide (5h)³**

Colorless oil obtained by column chromatography (PE/EA = 15:1 to 12:1); 43 mg, 71% yield; reaction time = 3 h. R_f = 0.37 (PE/EA = 5:1). ^1H NMR (400 MHz, CDCl_3) δ 7.75 (d, J = 8.3 Hz, 2H), 7.31 (d, J = 8.0 Hz, 2H), 5.12 (t, J = 7.1 Hz, 1H), 4.10–4.02 (m, 1H), 2.75 (d, J = 6.6 Hz, 2H), 2.42 (s, 3H), 1.81 (dd, J_1 = 15.5, J_2 = 9.1 Hz, 1H), 1.61 (dd, J_1 = 15.5, J_2 = 2.6 Hz, 1H), 1.48 (d, J = 6.6 Hz, 3H), 0.95 (d, J = 11.1 Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ 143.3, 136.8, 129.7, 126.9, 54.9, 52.4, 48.8, 34.1, 27.8, 26.1, 25.1, 21.5.



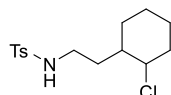
***N*-(4-Chloropentyl)methanesulfonamide (5i)⁷**

Colorless oil obtained by column chromatography (PE/EA = 4:1 to 3:1); 29 mg, 73% yield; reaction time = 3 h. $R_f = 0.20$ (PE/EA = 3:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 4.72 (s, 1H), 4.08–4.01 (m, 1H), 3.15 (q, $J = 6.2$ Hz, 2H), 2.96 (s, 3H), 1.82–1.66 (m, 4H), 1.51 (d, $J = 6.5$ Hz, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 58.1, 42.6, 40.2, 36.9, 27.2, 25.4.



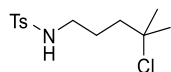
***N*-(4-Chloropentyl)cyclohexanesulfonamide (5j)**⁷

White solid obtained by column chromatography (PE/EA = 10:1 to 8:1); 36 mg, 67% yield; reaction time = 3 h. $R_f = 0.19$ (PE/EA = 5:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 4.57 (t, $J = 6.4$ Hz, 1H), 4.07–3.99 (m, 1H), 3.12 (q, $J = 6.3$ Hz, 2H), 2.84 (t, $J = 12.1$ Hz, 1H), 2.14 (d, $J = 11.0$ Hz, 2H), 1.88 (d, $J = 10.0$ Hz, 2H), 1.80–1.63 (m, 5H), 1.51–1.42 (m, 5H), 1.31–1.15 (m, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 61.1, 58.1, 42.9, 36.9, 27.7, 26.4, 26.4, 25.4, 25.1, 25.0.



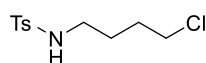
***N*-(2-(2-Chlorocyclohexyl)ethyl)-4-methylbenzenesulfonamide (5k)**⁸

Colorless oil obtained by column chromatography (PE/EA = 12:1 to 10:1); 42 mg, 66% yield; reaction time = 3 h; dr = 1.5:1. $R_f = 0.32$ (PE/EA = 5:1). $^1\text{H NMR}$ (major) (400 MHz, CDCl_3) δ 7.76 (d, $J = 8.3$ Hz, 2H), 7.32 (d, $J = 8.1$ Hz, 2H), 4.96 (t, $J = 6.3$ Hz, 1H), 4.24 (q, $J = 2.8$ Hz, 1H), 3.07–2.89 (m, 2H), 2.43 (s, 3H), 2.20–2.14 (m, 1H), 2.03–1.93 (m, 1H), 1.84–1.78 (m, 1H), 1.75–1.60 (m, 3H), 1.58–1.51 (m, 1H), 1.47–1.35 (m, 1H), 1.39–1.30 (m, 1H), 1.30–1.13 (m, 1H), 0.99–0.90 (m, 1H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 143.4 (2C), 136.6, 136.6, 129.7, 127.1, 127.0, 65.6, 63.9, 43.1, 40.6, 40.2, 38.6, 37.4, 34.1, 33.3, 31.4, 26.1, 26.1, 25.1, 24.9, 21.5, 19.9.



***N*-(4-Chloro-4-methylpentyl)-4-methylbenzenesulfonamide (5l)**⁷

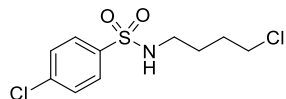
Colorless oil obtained by column chromatography (PE/EA = 15:1); 47 mg, 81% yield; reaction time = 3 h. $R_f = 0.27$ (PE/EA = 5:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.77 (d, $J = 8.3$ Hz, 2H), 7.32 (d, $J = 7.8$ Hz, 2H), 5.04 (t, $J = 6.3$ Hz, 1H), 2.96 (q, $J = 6.2$ Hz, 2H), 2.43 (s, 3H), 1.72–1.63 (m, 4H), 1.50 (s, 6H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 143.4, 136.7, 129.7, 127.0, 70.3, 43.1, 42.6, 32.3, 25.2, 21.5.



***N*-(4-Chlorobutyl)-4-methylbenzenesulfonamide (5m)**⁷

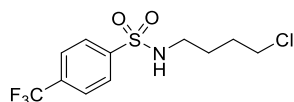
Colorless oil obtained by column chromatography (PE/EA = 10:1 to 8:1); 23 mg, 44% yield; reaction time = 8 h. $R_f = 0.20$ (PE/EA = 5:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ

7.76 (d, $J = 8.3$ Hz, 2H), 7.32 (d, $J = 8.2$ Hz, 2H), 5.03 (t, $J = 6.3$ Hz, 1H), 3.48 (t, $J = 6.4$ Hz, 2H), 2.95 (q, $J = 6.7$ Hz, 2H), 2.43 (s, 3H), 1.82–1.74 (m, 2H), 1.65–1.58 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 143.5, 136.7, 129.7, 127.0, 44.3, 42.3, 29.3, 26.7, 21.5.



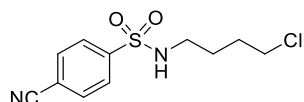
4-Chloro-*N*-(4-chlorobutyl)benzenesulfonamide (5n)³

Colorless oil obtained by column chromatography (PE/EA = 12:1 to 10:1); 13 mg, 23% yield; reaction time = 8 h. $R_f = 0.25$ (PE/EA = 5:1). ^1H NMR (400 MHz, CDCl_3) δ 7.81 (d, $J = 8.5$ Hz, 2H), 7.51 (d, $J = 8.6$ Hz, 2H), 4.53 (t, $J = 6.2$ Hz, 1H), 3.52 (t, $J = 6.4$ Hz, 2H), 3.01 (q, $J = 6.7$ Hz, 2H), 1.83–1.76 (m, 2H), 1.69–1.62 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 139.3, 138.3, 129.5, 128.5, 76.7, 44.2, 42.5, 29.2, 26.9.



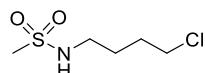
***N*-(4-Chlorobutyl)-4-(trifluoromethyl)benzenesulfonamide (5o)**³

Colorless oil obtained by column chromatography (PE/EA = 12:1 to 10:1); 16 mg, 25% yield; reaction time = 8 h. $R_f = 0.30$ (PE/EA = 5:1). ^1H NMR (400 MHz, CDCl_3) δ 8.02 (d, $J = 8.1$ Hz, 2H), 7.81 (d, $J = 8.1$ Hz, 2H), 5.05 (t, $J = 6.3$ Hz, 1H), 3.51 (t, $J = 6.3$ Hz, 2H), 3.03 (q, $J = 6.7$ Hz, 2H), 1.83–1.76 (m, 2H), 1.70–1.63 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 143.3, 134.4 (d, $J = 33.2$ Hz, 1C), 127.5, 126.4 (q, $J = 3.7$ Hz, 1C), 124.5, 121.8, 44.2, 42.5, 29.2, 26.9.



***N*-(4-Chlorobutyl)-4-cyanobenzenesulfonamide (5p)**

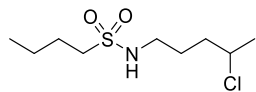
White solid obtained by column chromatography (PE/EA = 6:1 to 5:1); 15 mg, 27% yield; reaction time = 8 h. $R_f = 0.20$ (PE/EA = 5:1). ^1H NMR (400 MHz, CDCl_3) δ 7.99 (d, $J = 8.4$ Hz, 2H), 7.84 (d, $J = 8.2$ Hz, 2H), 4.72 (t, $J = 6.3$ Hz, 1H), 3.53 (t, $J = 6.2$ Hz, 2H), 3.05 (q, $J = 6.6$ Hz, 2H), 1.83–1.76 (m, 2H), 1.71–1.65 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 144.2, 133.0, 127.6, 117.3, 116.5, 44.1, 42.6, 29.2, 27.0. IR (KBr, cm^{-1}) ν 3283, 2956, 1595, 1432, 1332, 1158, 1020, 842, 570, 518. HRMS (ESI-TOF) calcd for $\text{C}_{11}\text{H}_{14}\text{ClN}_2\text{O}_2\text{S}$ $[\text{M}+\text{H}]^+$ 273.0459, found 273.0455.



***N*-(4-Chlorobutyl)methanesulfonamide (5q)**³

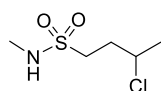
Colorless oil obtained by column chromatography (PE/EA = 3:1); 16 mg, 43% yield; reaction time = 16 h. $R_f = 0.16$ (PE/EA = 3:1). ^1H NMR (400 MHz, CDCl_3) δ 4.66 (s,

1H), 3.58 (t, $J = 6.3$ Hz, 2H), 3.18 (q, $J = 6.6$ Hz, 2H), 2.97 (s, 3H), 1.90–1.84 (m, 2H), 1.78–1.71 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 44.3, 42.5, 40.3, 29.3, 27.4.



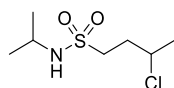
***N*-(4-Chloropentyl)butane-1-sulfonamide (5r)**⁷

Colorless oil obtained by column chromatography (PE/EA = 5:1); 30 mg, 62% yield; reaction time = 3 h. $R_f = 0.17$ (PE/EA = 5:1). ^1H NMR (400 MHz, CDCl_3) δ 4.64 (t, $J = 6.3$ Hz, 1H), 4.07–4.00 (m, 1H), 3.12 (q, $J = 6.3$ Hz, 2H), 3.02–2.98 (m, 2H), 1.84–1.64 (m, 6H), 1.51 (d, $J = 6.6$ Hz, 3H), 1.47–1.39 (m, 2H), 0.94 (t, $J = 7.4$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 58.1, 52.3, 42.6, 36.9, 27.4, 25.6, 25.4, 21.5, 13.6.



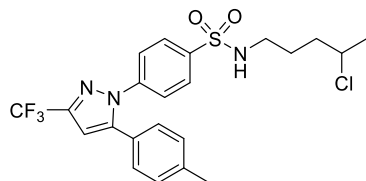
3-Chloro-*N*-methylbutane-1-sulfonamide (5s)⁹

Colorless oil obtained by column chromatography (PE/EA = 5:1); 23 mg, 62% yield; reaction time = 16 h. $R_f = 0.18$ (PE/EA = 3:1). ^1H NMR (600 MHz, CDCl_3) δ 4.44 (s, 1H), 4.19–4.14 (m, 1H), 3.30–3.25 (m, 1H), 3.18–3.13 (m, 1H), 2.81 (d, $J = 5.3$ Hz, 3H), 2.23–2.24 (m, 1H), 2.13–2.06 (m, 1H), 1.56 (d, $J = 6.5$ Hz, 3H). ^{13}C NMR (150 MHz, CDCl_3) δ 56.3, 48.6, 34.1, 29.3, 25.2.



3-Chloro-*N*-isopropylbutane-1-sulfonamide (5t)³

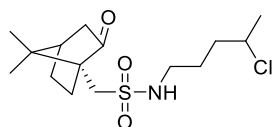
Colorless oil obtained by column chromatography (PE/EA = 6:1); 26 mg, 61% yield; reaction time = 16 h. $R_f = 0.26$ (PE/EA = 3:1). ^1H NMR (400 MHz, CDCl_3) δ 4.23 (d, $J = 7.9$ Hz, 1H), 4.19–4.12 (m, 1H), 3.68–3.59 (m, 1H), 3.31–3.23 (m, 1H), 3.18–3.11 (m, 1H), 2.32–2.23 (m, 1H), 2.15–2.05 (m, 1H), 1.56 (d, $J = 6.6$ Hz, 3H), 1.25 (d, $J = 6.6$ Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ 56.4, 51.1, 46.3, 34.2, 25.3, 24.3, 24.2.



***N*-(4-Chloropentyl)-4-(2-(*p*-tolyl)-4-(trifluoromethyl)cyclopenta-2,4-dien-1-yl)benzenesulfonamide (5u)**⁷

White solid obtained by column chromatography (PE/EA = 12:1 to 10:1); 63 mg, 65% yield; reaction time = 3 h. $R_f = 0.19$ (PE/EA = 5:1). ^1H NMR (400 MHz, CDCl_3) δ 7.84 (t, $J = 8.5$ Hz, 2H), 7.45 (dd, $J_1 = 25.5$ Hz, $J_2 = 8.7$ Hz, 2H), 7.25–7.10 (m, 4H), 6.75 (s, 1H), 4.84 (t, $J = 6.1$ Hz, 1H), 4.02–3.94 (m, 1H), 2.97 (q, $J = 6.3$ Hz, 2H),

2.39 (d, $J = 9.7$ Hz, 3H), 1.77–1.58 (m, 4H), 1.48 (d, $J = 6.5$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 145.2, 144.0 (d, $J = 38.9$ Hz, 1C), 142.5, 141.9 (d, $J = 30.0$ Hz, 1C), 140.1 (d, $J = 71.1$ Hz, 1C), 139.3 (d, $J = 29.5$ Hz, 1C), 129.70 (t, $J = 15.0$ Hz, 1C), 128.6, 128.0 (d, $J = 4.4$ Hz, 1C), 125.6, 125.1, 122.7 (d, $J = 79.5$ Hz, 1C), 119.6, 106.3, 57.9, 42.6, 36.8, 26.6, 25.3, 21.3 (d, $J = 13.6$ Hz, 1C).



***N*-(4-Chloropentyl)-1-((1R,4S)-7,7-dimethyl-2-oxobicyclo[2.2.1]heptan-1-yl)methanesulfonamide (5v)**⁷

Colorless oil obtained by column chromatography (PE/EA = 7:1 to 5:1); 45 mg, 67% yield; dr = 1:1; reaction time = 3 h. $R_f = 0.14$ (PE/EA = 5:1). ^1H NMR (400 MHz, CDCl_3) δ 5.32 (s, 1H), 4.09–4.00 (m, 1H), 3.37 (d, $J = 15.0$ Hz, 2H), 3.18 (s, 1H), 2.90 (d, $J = 15.1$ Hz, 1H), 2.38 (d, $J = 18.6$ Hz, 1H), 2.22–2.11 (m, 2H), 2.06–1.90 (m, 3H), 1.92–1.86 (m, 4H), 1.83–1.68 (m, 4H), 1.51 (d, $J = 6.6$ Hz, 1H), 1.47–1.41 (m, 3H), 0.99 (s, 1H), 0.88 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 217.3, 59.2 (2C), 58.3, 58.1, 49.2 (2C), 48.9, 48.8, 43.1, 43.0, 42.9 (2C), 42.7, 37.1, 36.9, 27.2, 27.0 (2C), 26.7, 26.6, 25.4, 19.9, 19.4.

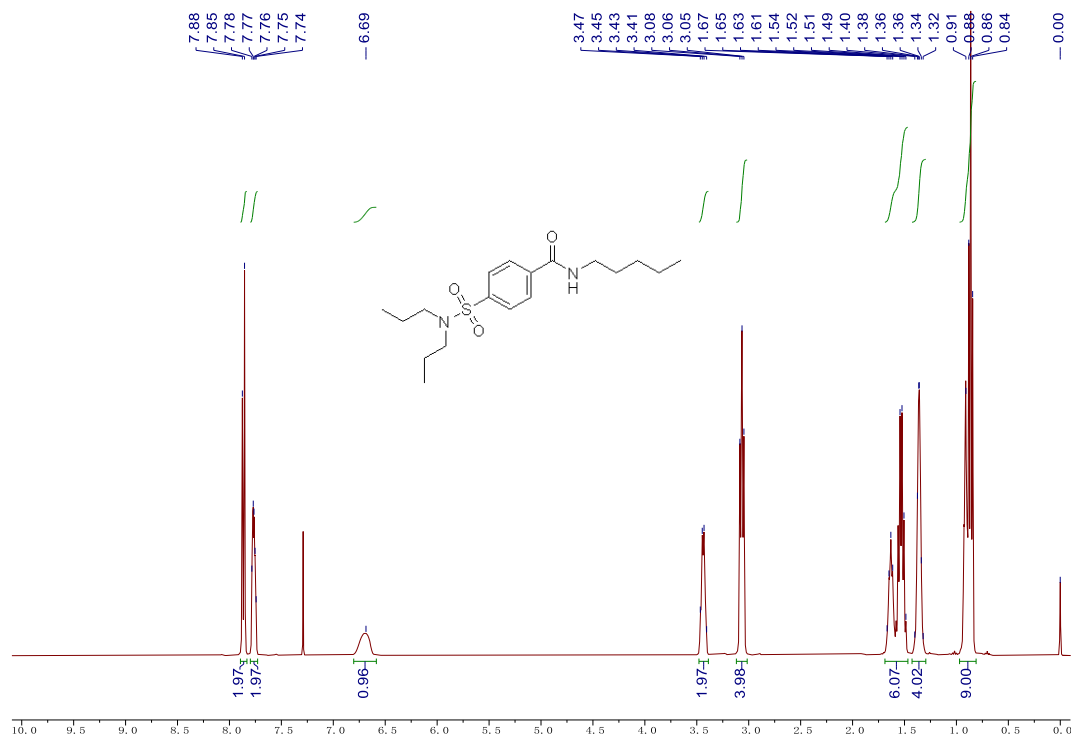
5. References

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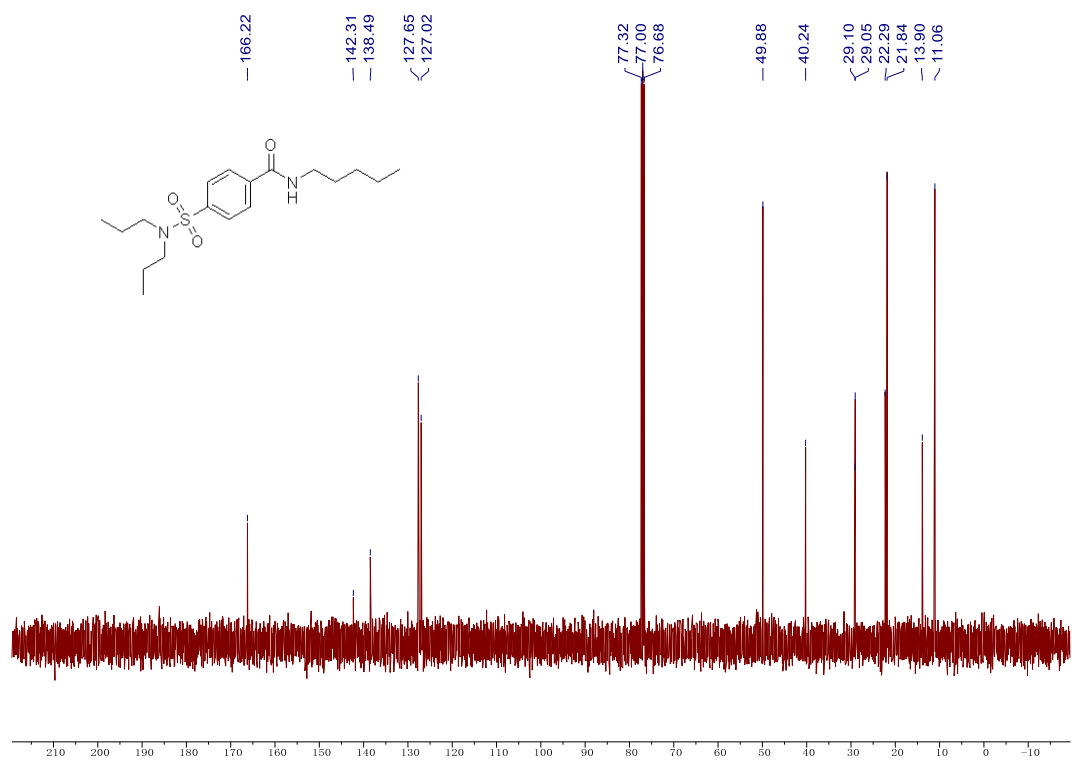
6. ^1H and ^{13}C NMR spectra

1ak

^1H NMR (CDCl_3 , 400 MHz)

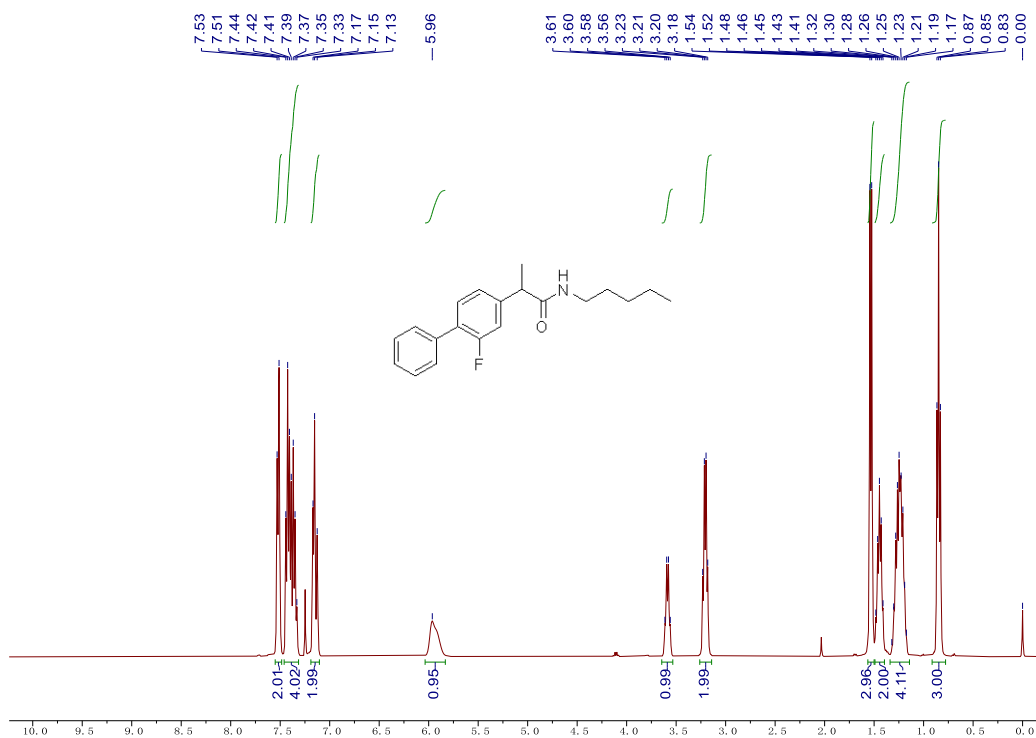


^{13}C NMR (CDCl_3 , 100 MHz)

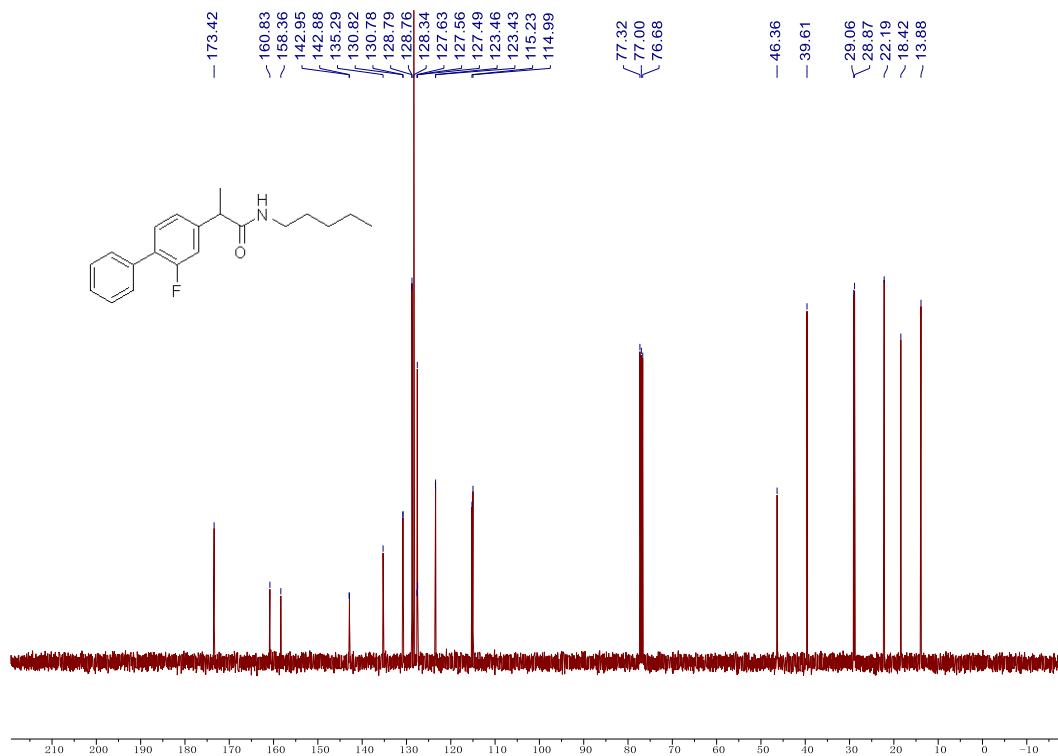


1am

¹H NMR (CDCl₃, 400 MHz)

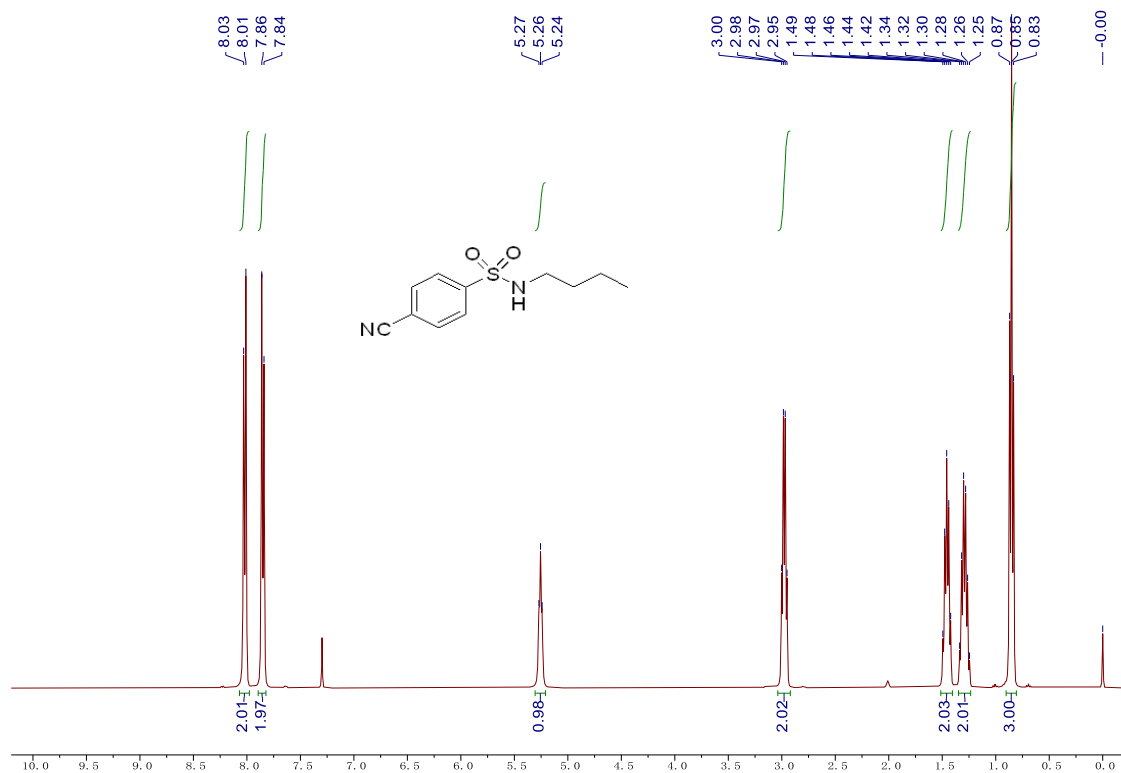


¹³C NMR (CDCl₃, 100 MHz)

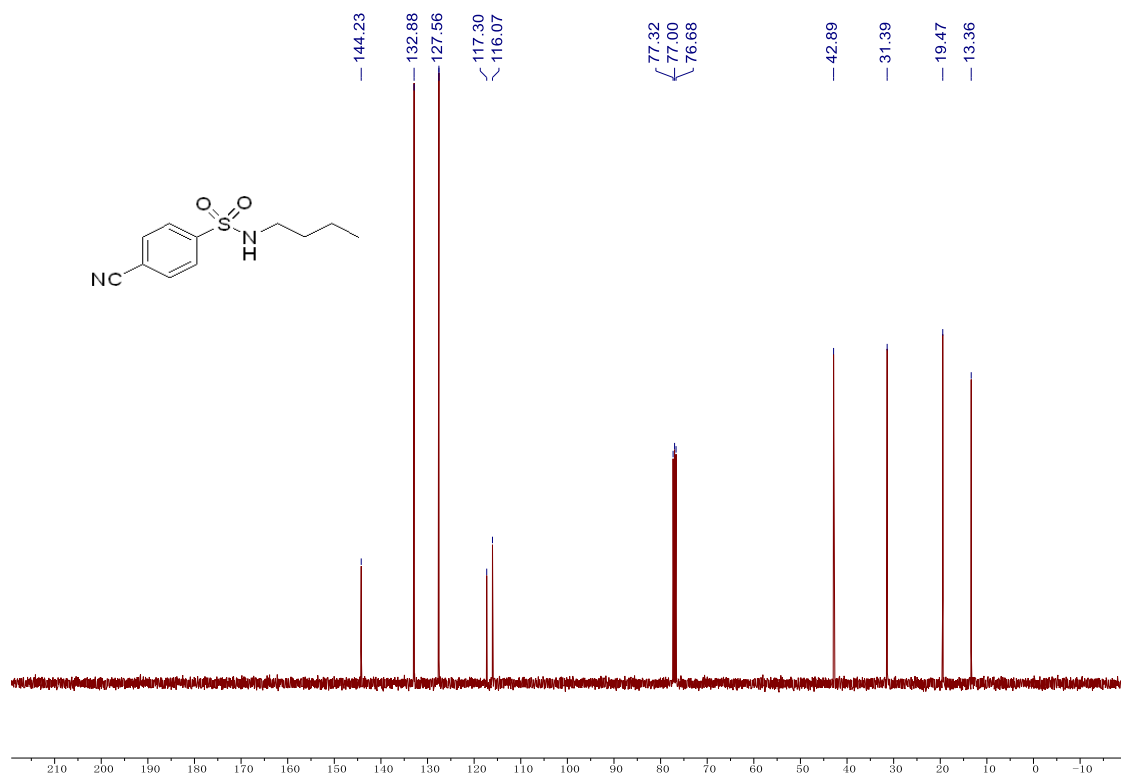


4p

^1H NMR (CDCl_3 , 400 MHz)

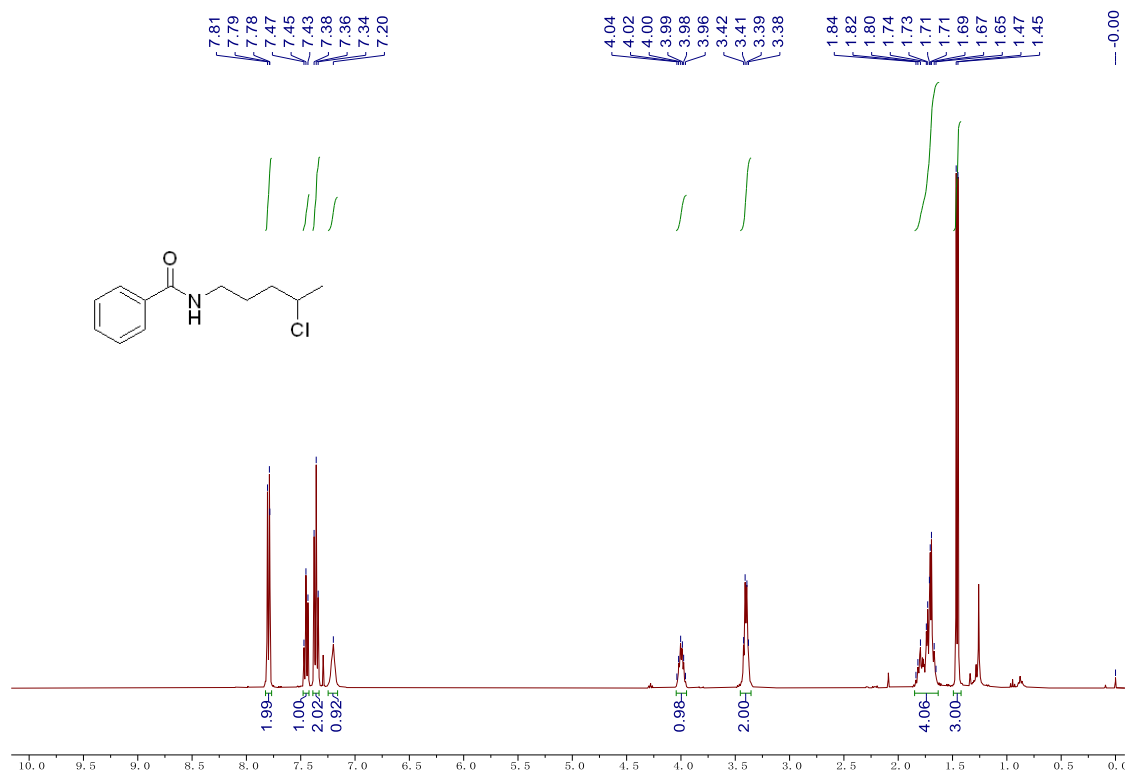


^{13}C NMR (CDCl_3 , 100 MHz)

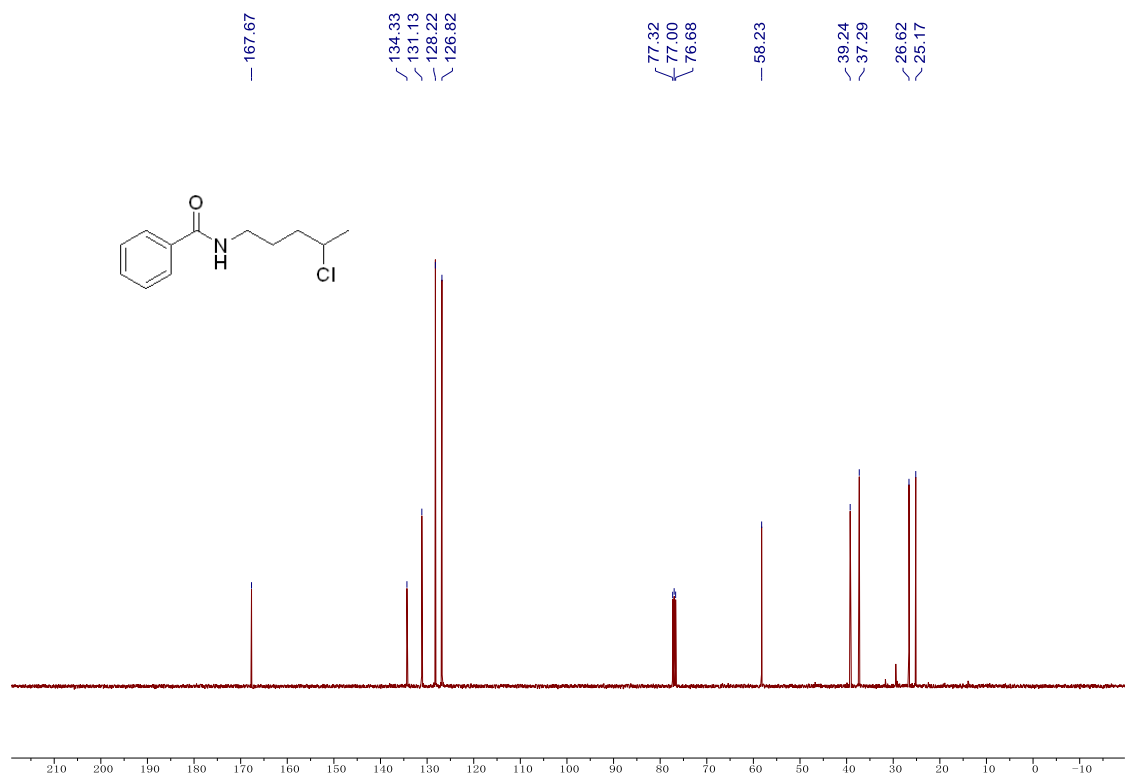


2a

^1H NMR (CDCl_3 , 400 MHz)

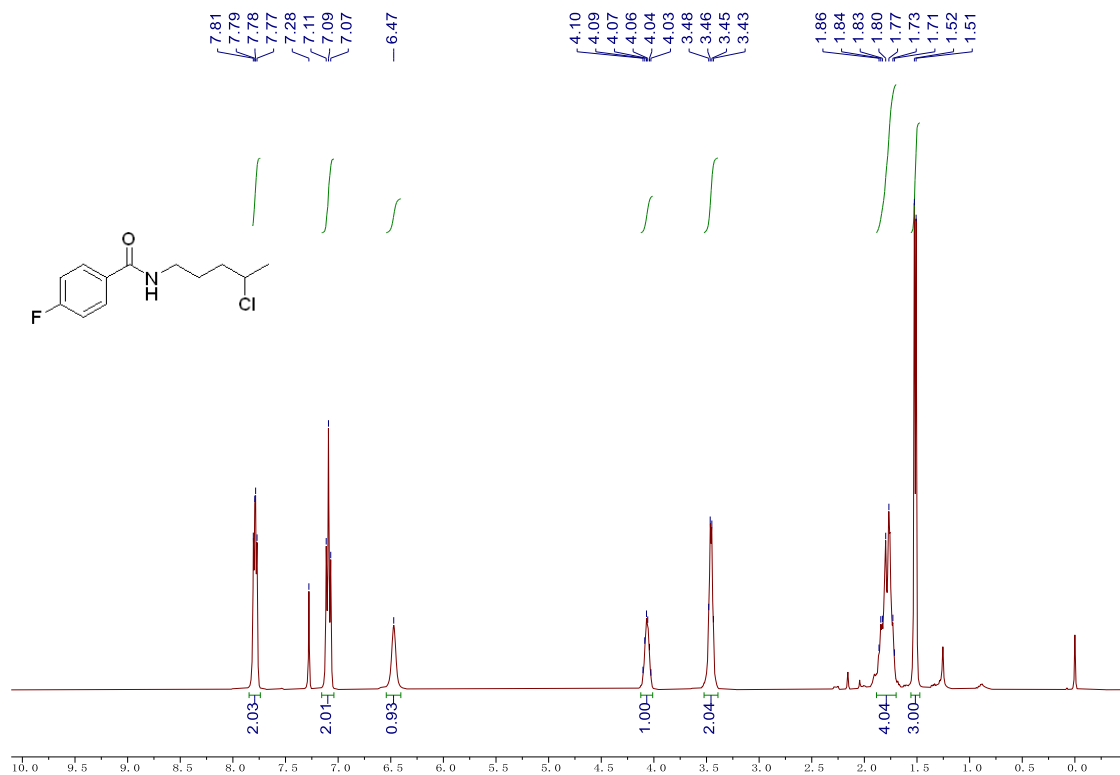


^{13}C NMR (CDCl_3 , 100 MHz)

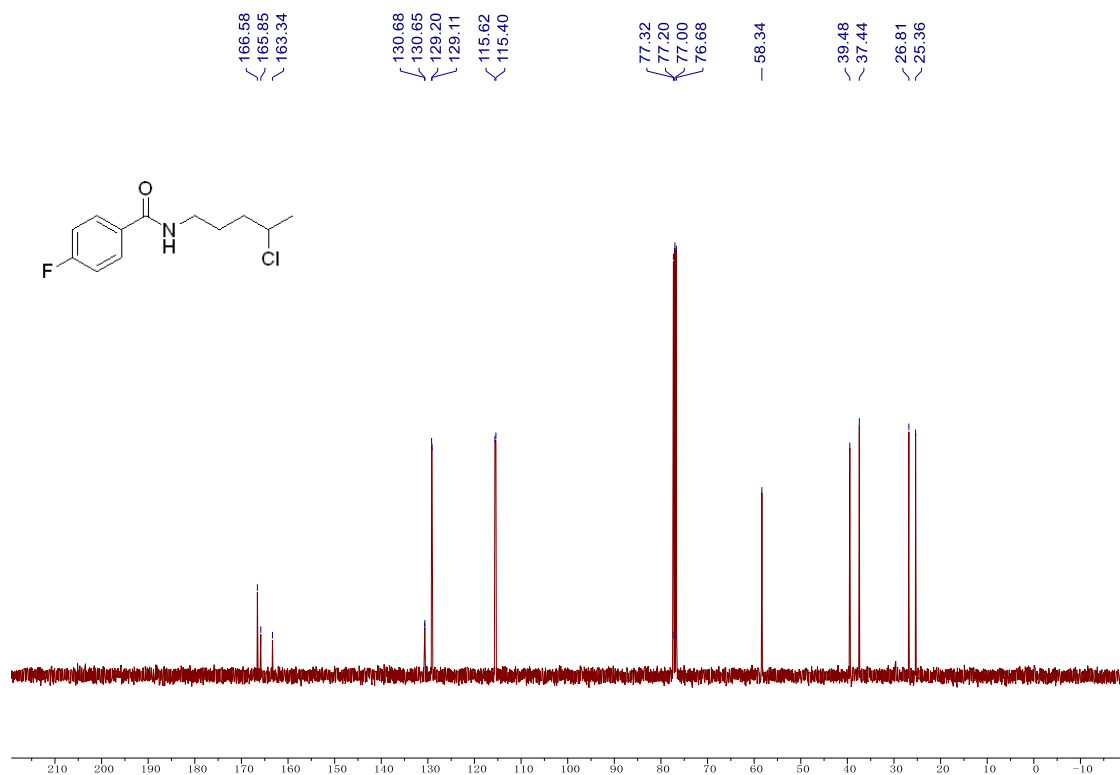


2b

^1H NMR (CDCl_3 , 400 MHz)

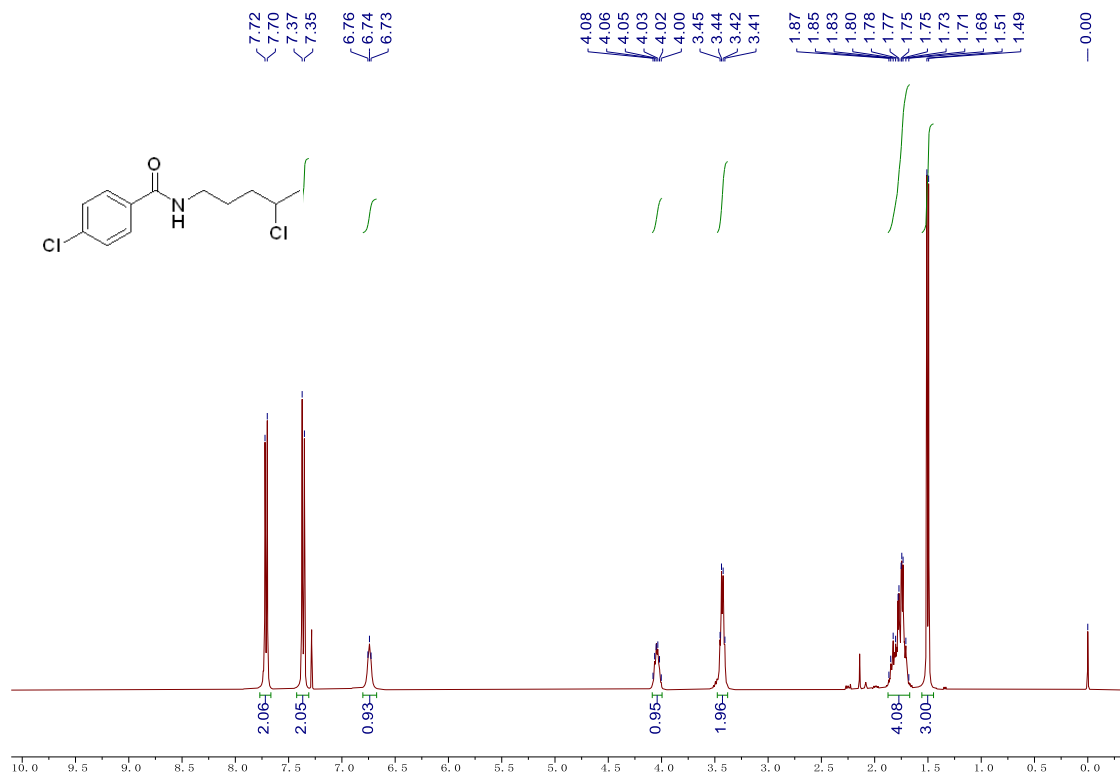


^{13}C NMR (CDCl_3 , 100 MHz)

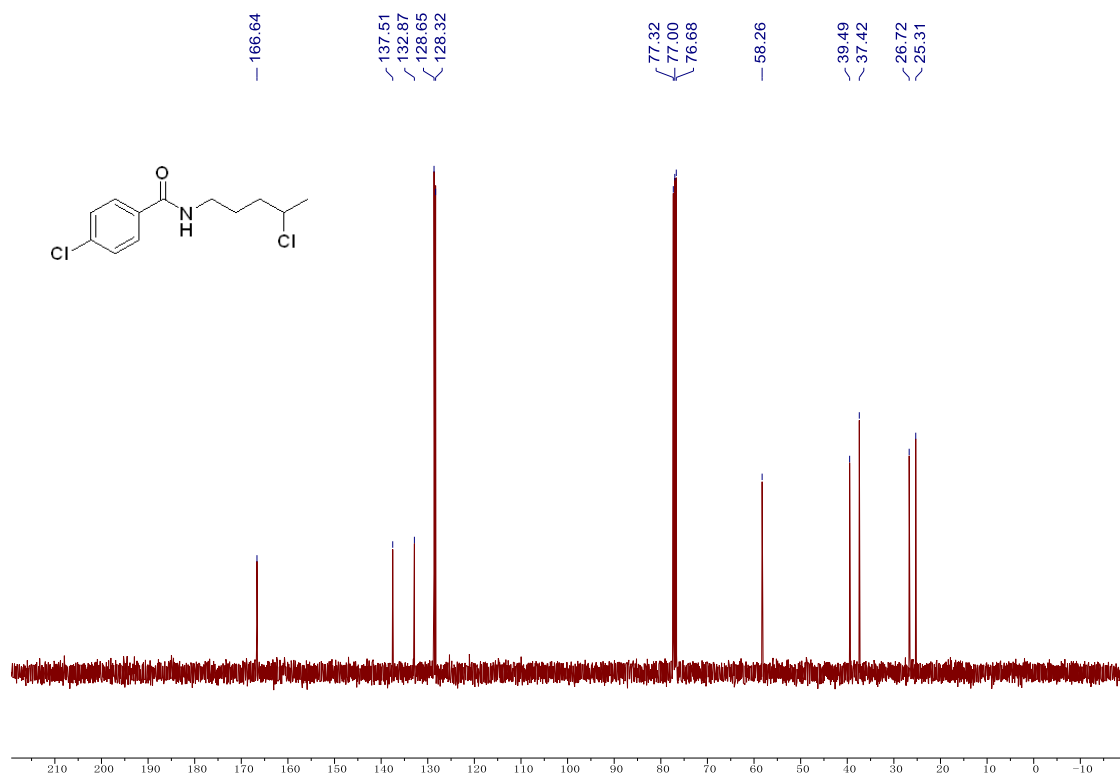


2c

^1H NMR (CDCl_3 , 400 MHz)

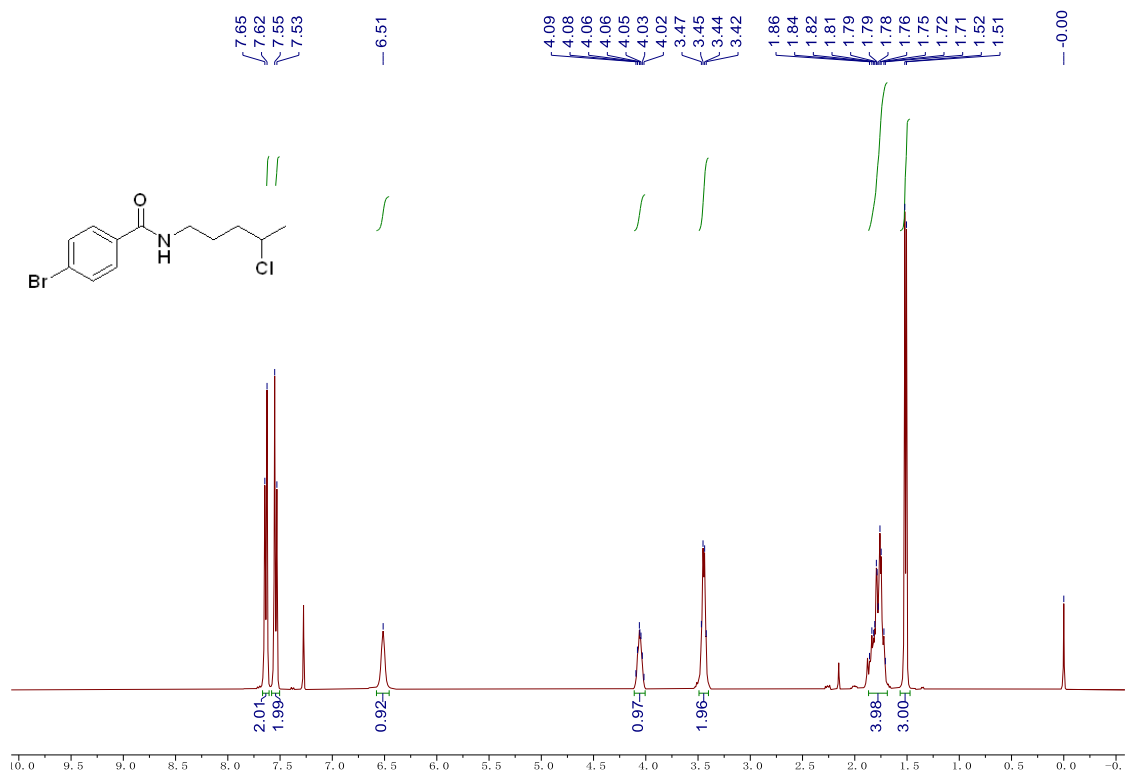


^{13}C NMR (CDCl_3 , 100 MHz)

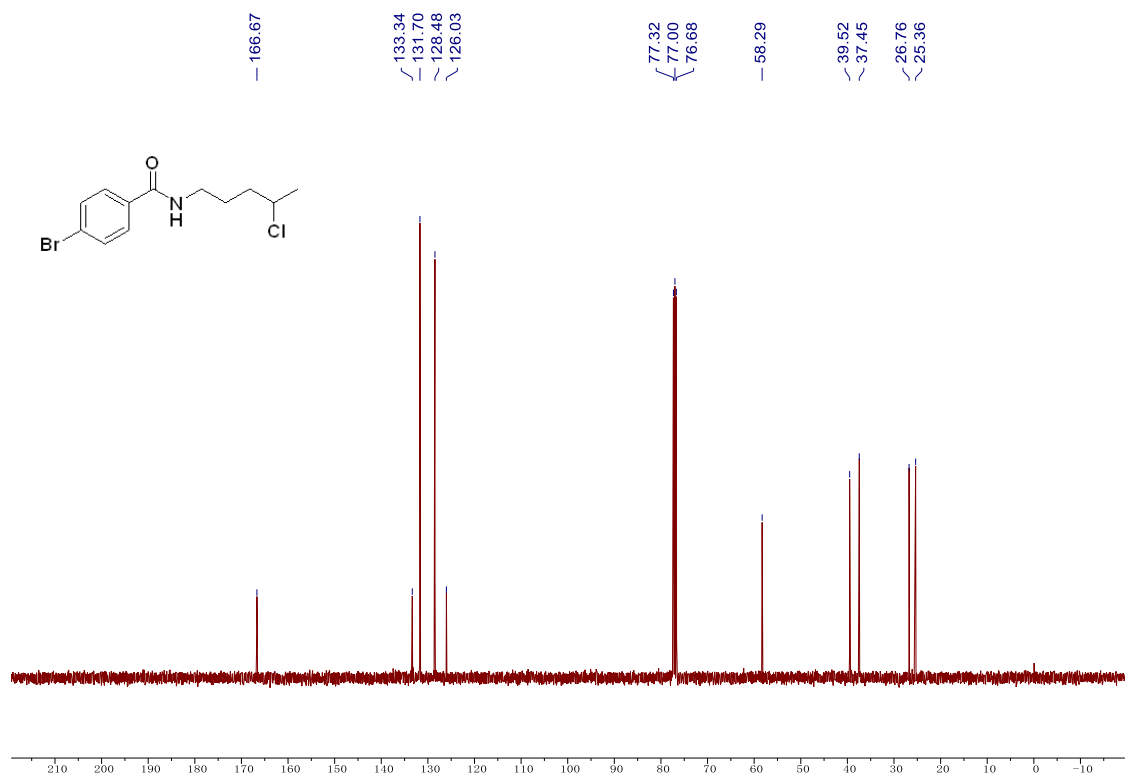


2d

^1H NMR (CDCl_3 , 400 MHz)

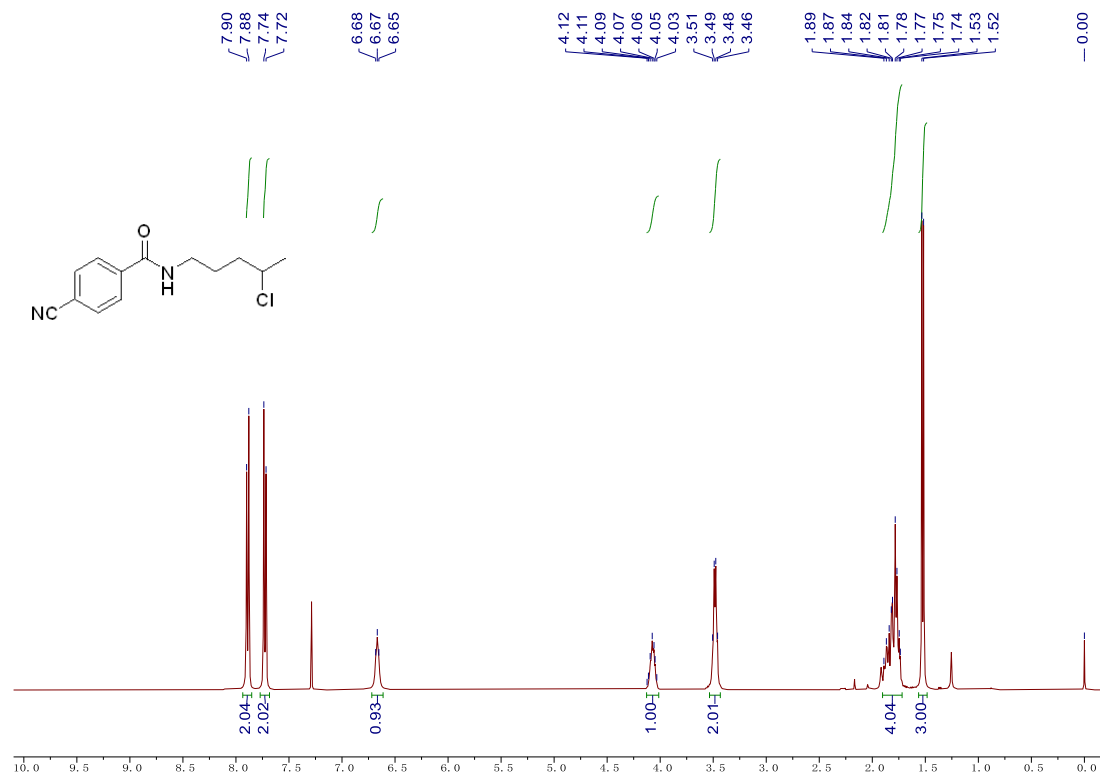


^{13}C NMR (CDCl_3 , 100 MHz)

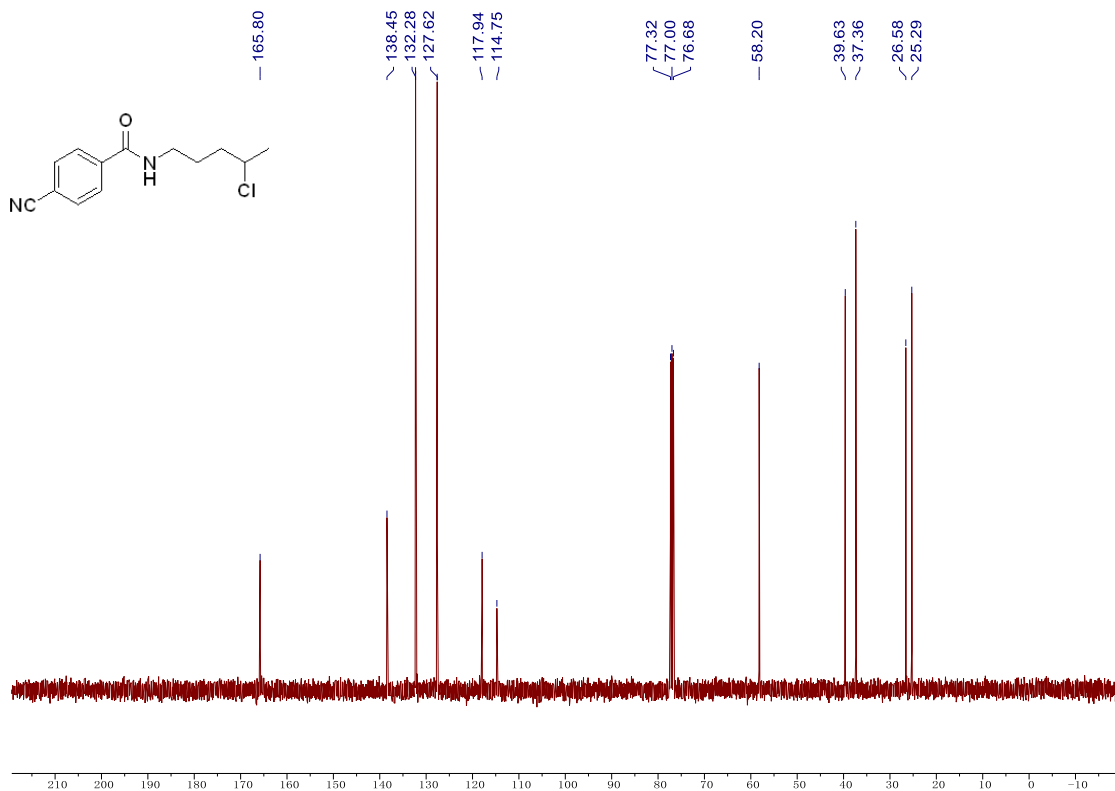


2e

¹H NMR (CDCl₃, 400 MHz)

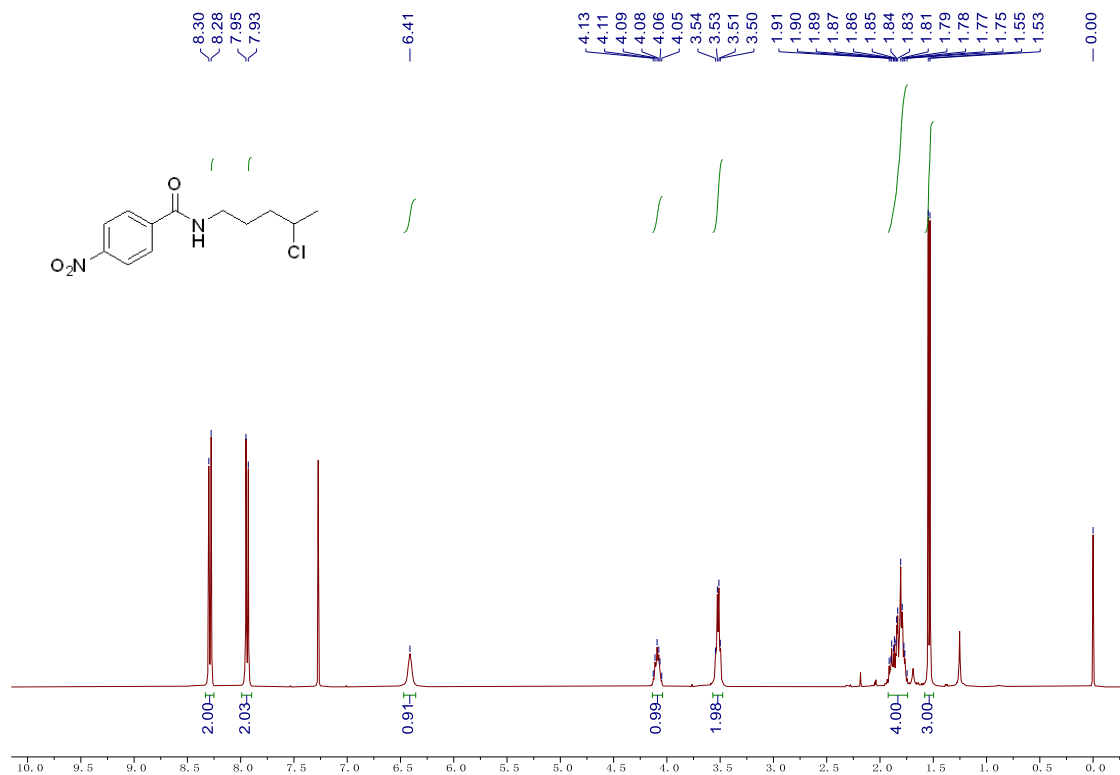


¹³C NMR (CDCl₃, 100 MHz)

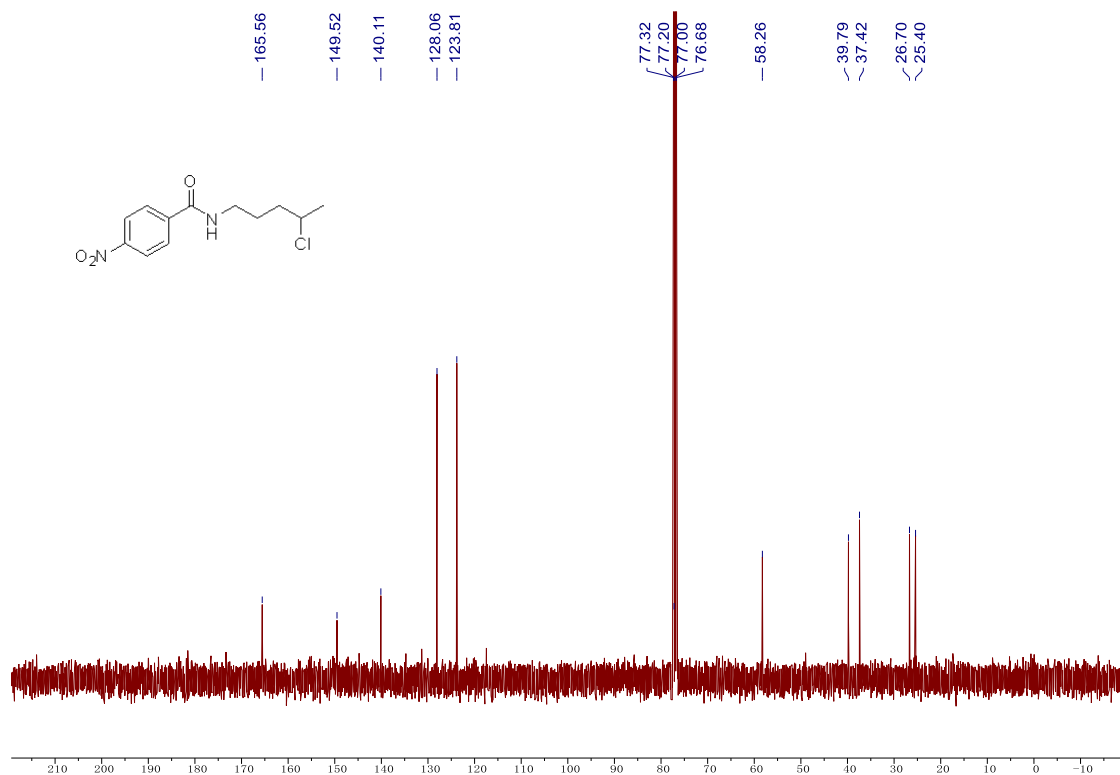


2f

^1H NMR (CDCl_3 , 400 MHz)

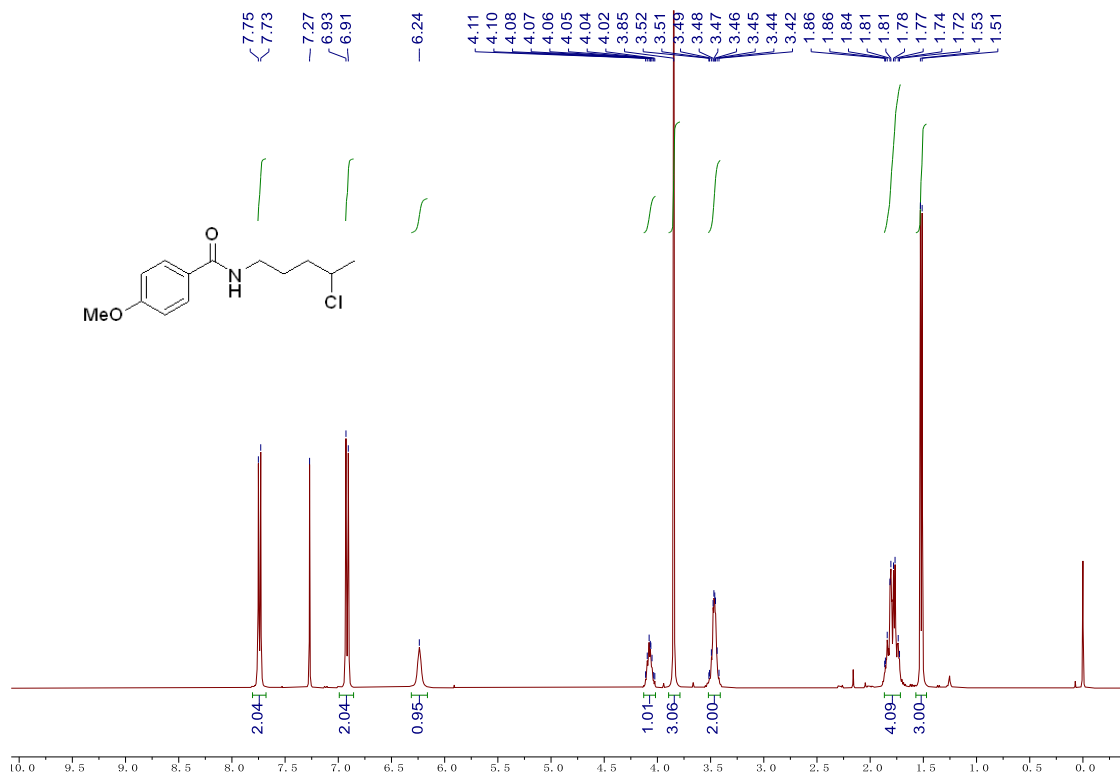


^{13}C NMR (CDCl_3 , 100 MHz)

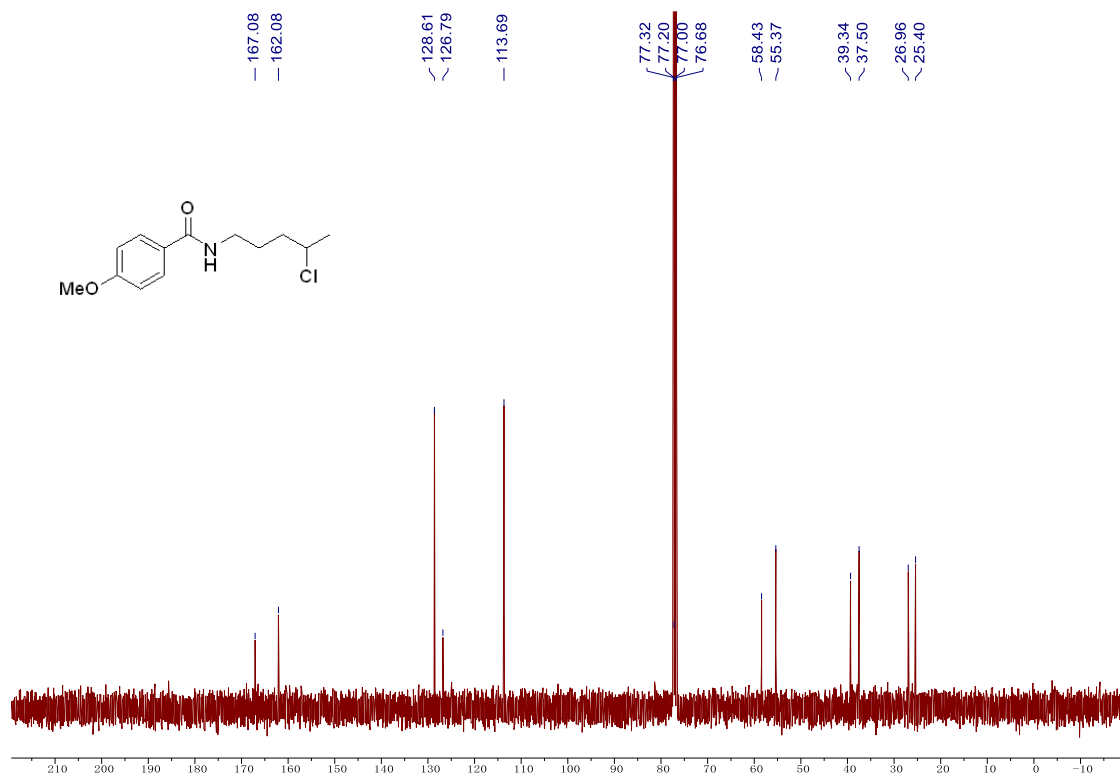


2g

^1H NMR (CDCl_3 , 400 MHz)

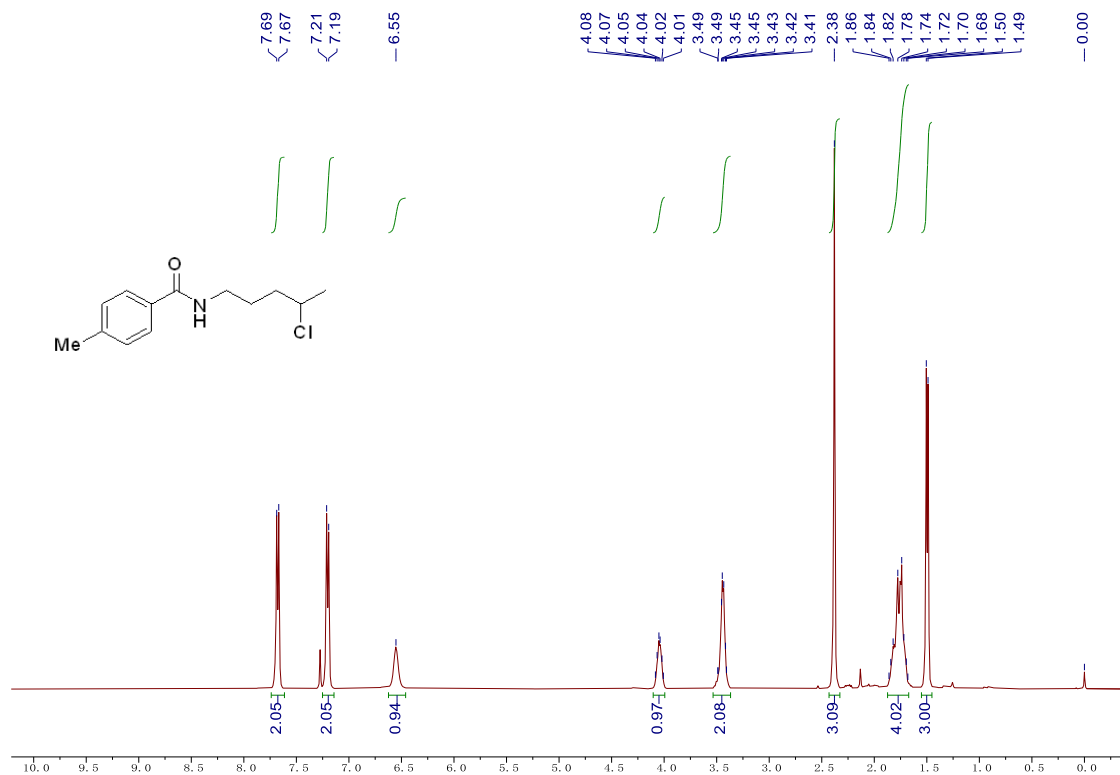


^{13}C NMR (CDCl_3 , 100 MHz)

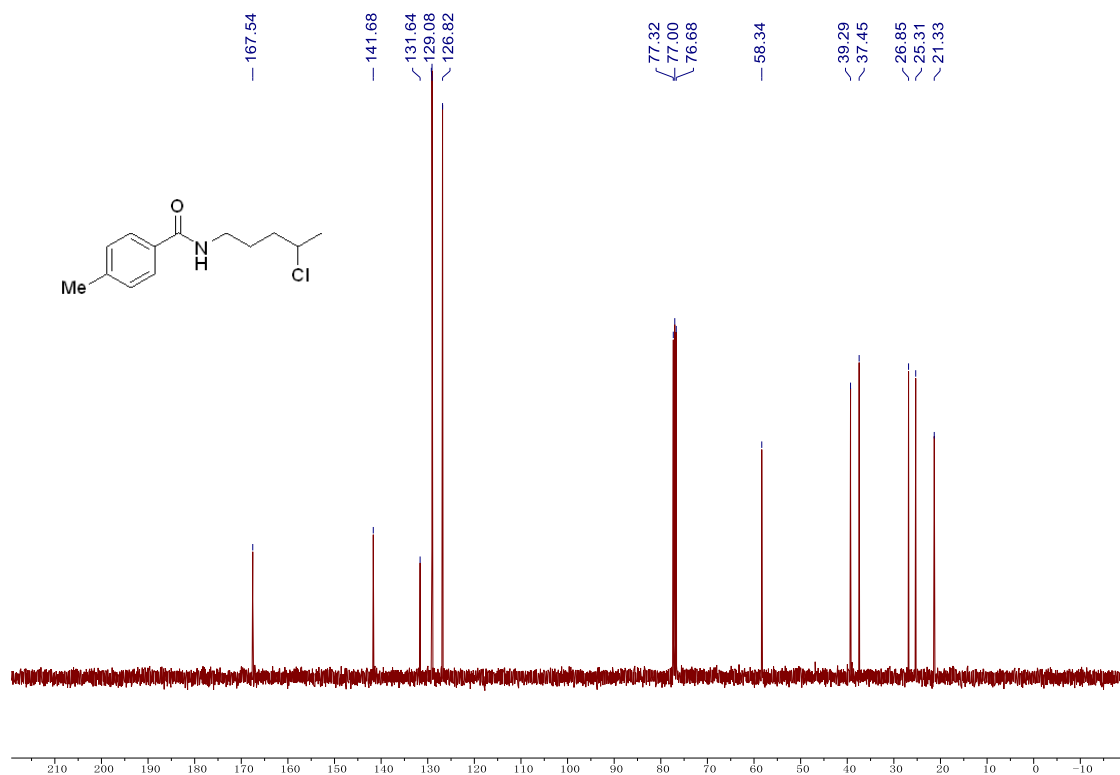


2h

^1H NMR (CDCl_3 , 400 MHz)

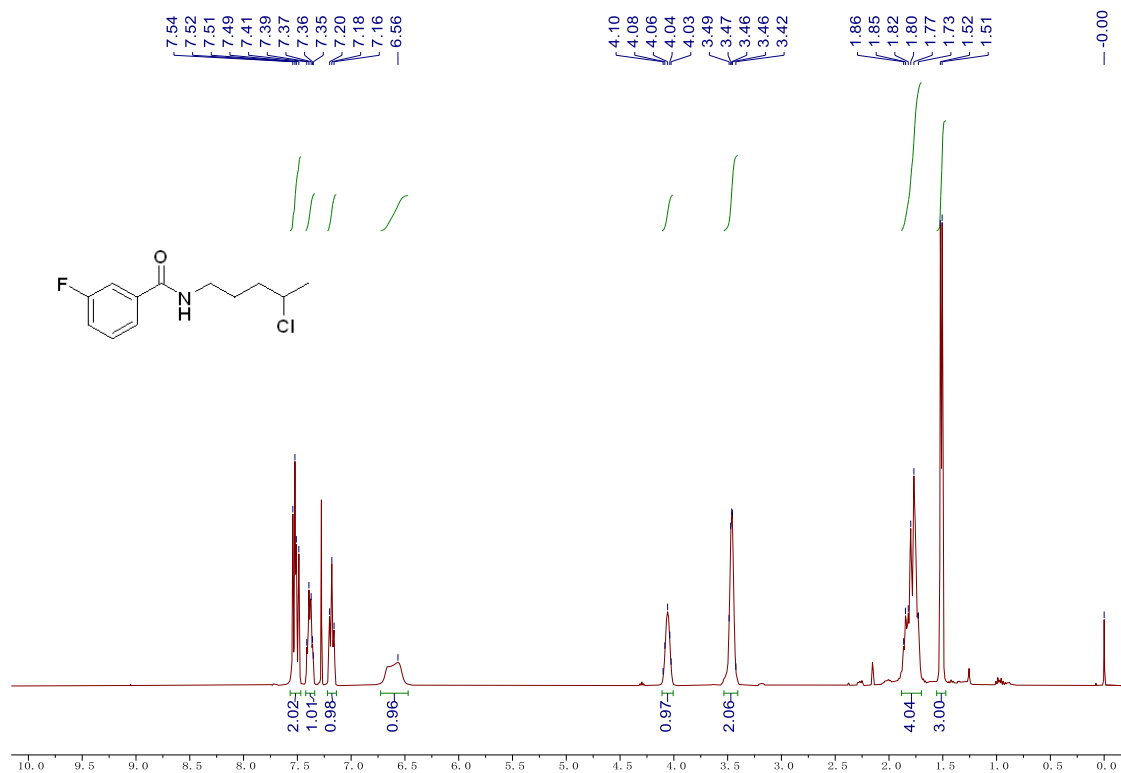


^{13}C NMR (CDCl_3 , 100 MHz)

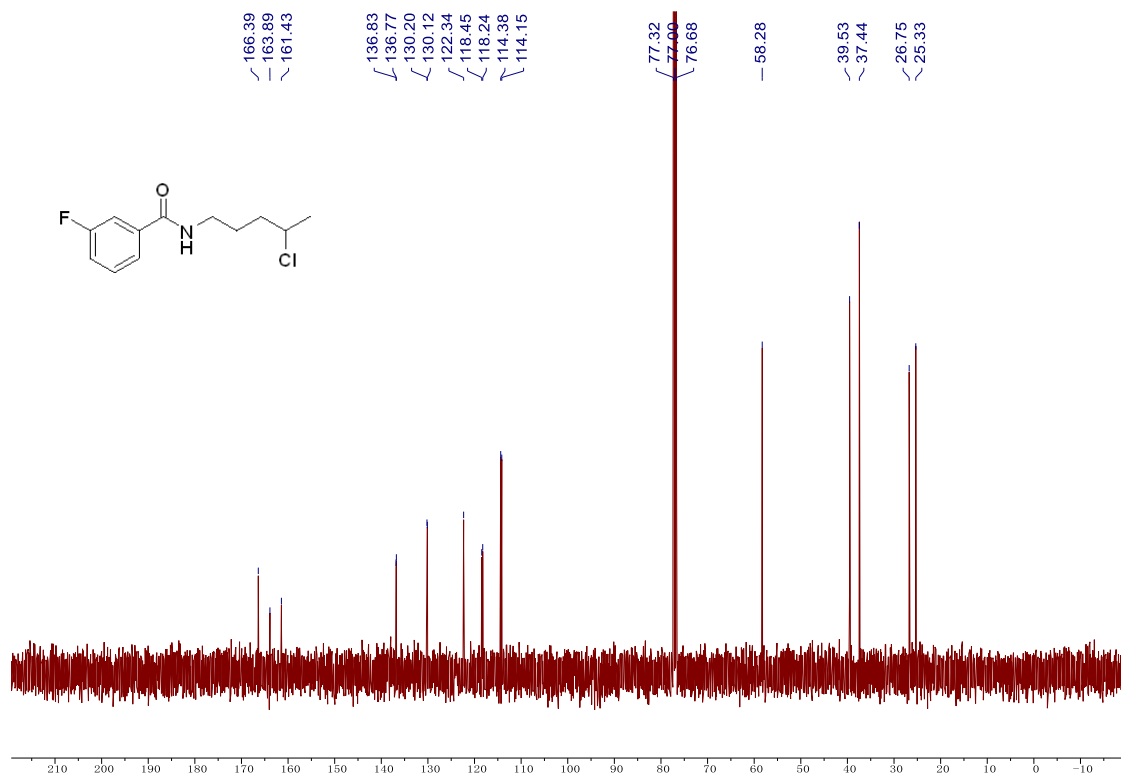


2i

^1H NMR (CDCl_3 , 400 MHz)

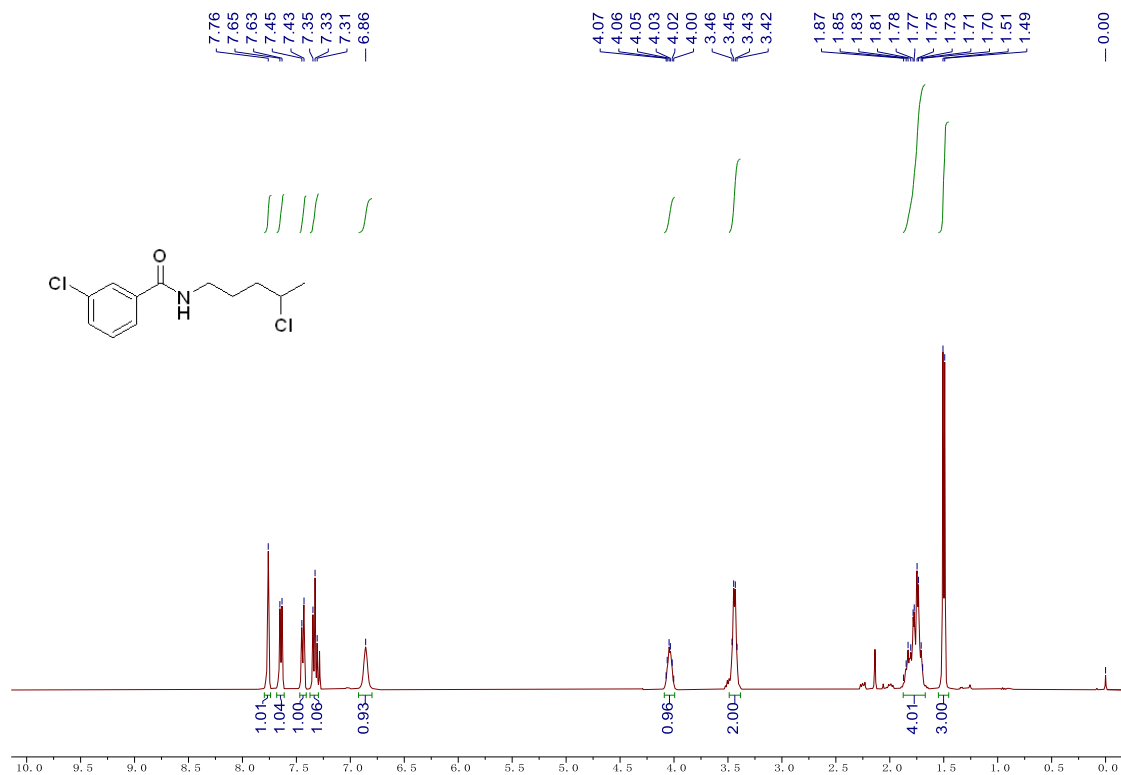


^{13}C NMR (CDCl_3 , 100 MHz)

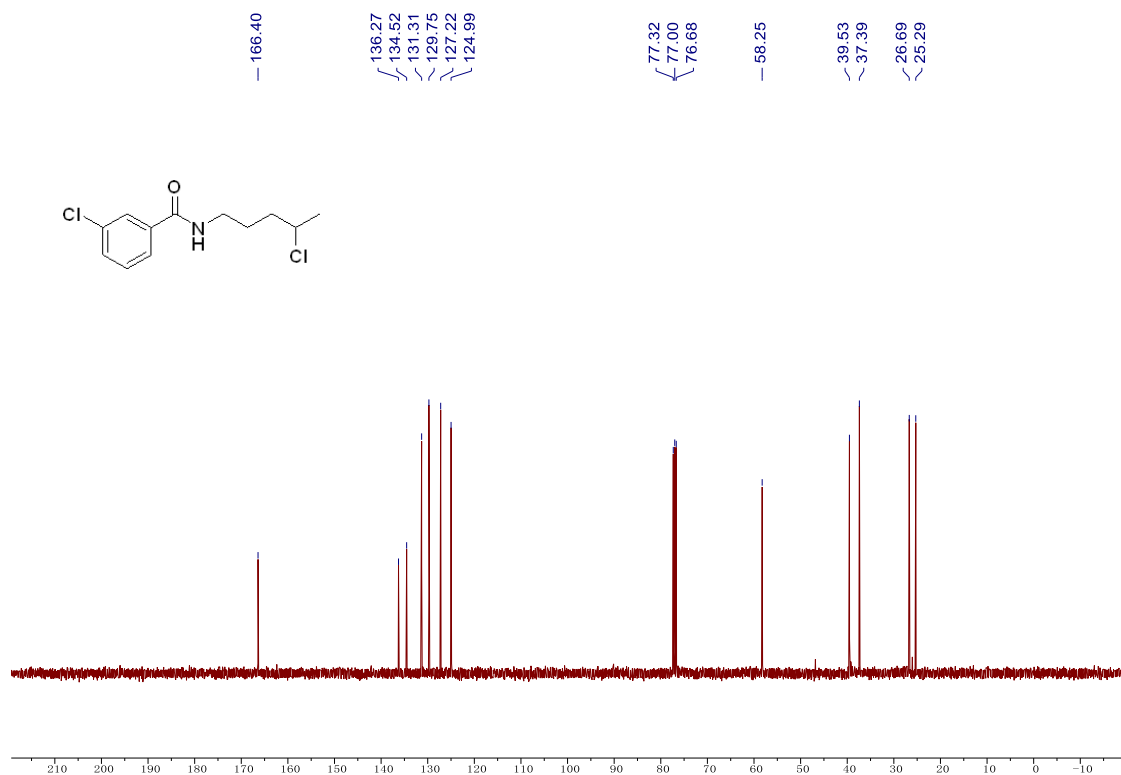


2j

^1H NMR (CDCl_3 , 400 MHz)

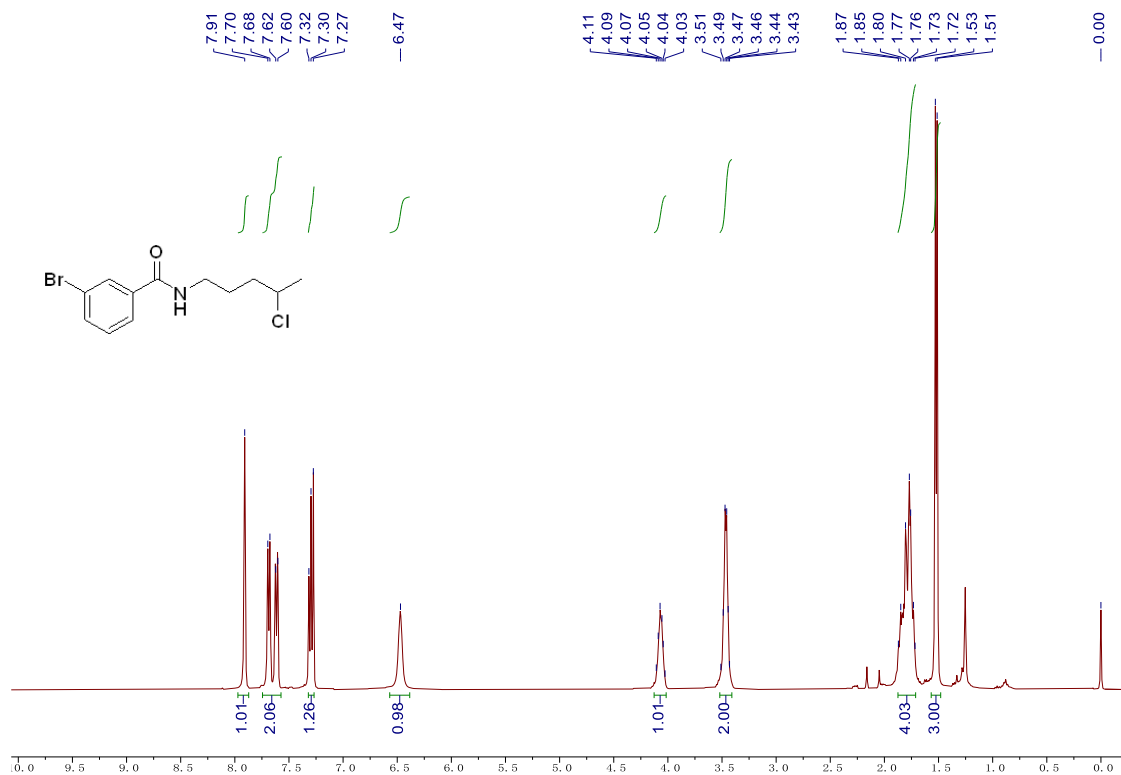


^{13}C NMR (CDCl_3 , 100 MHz)

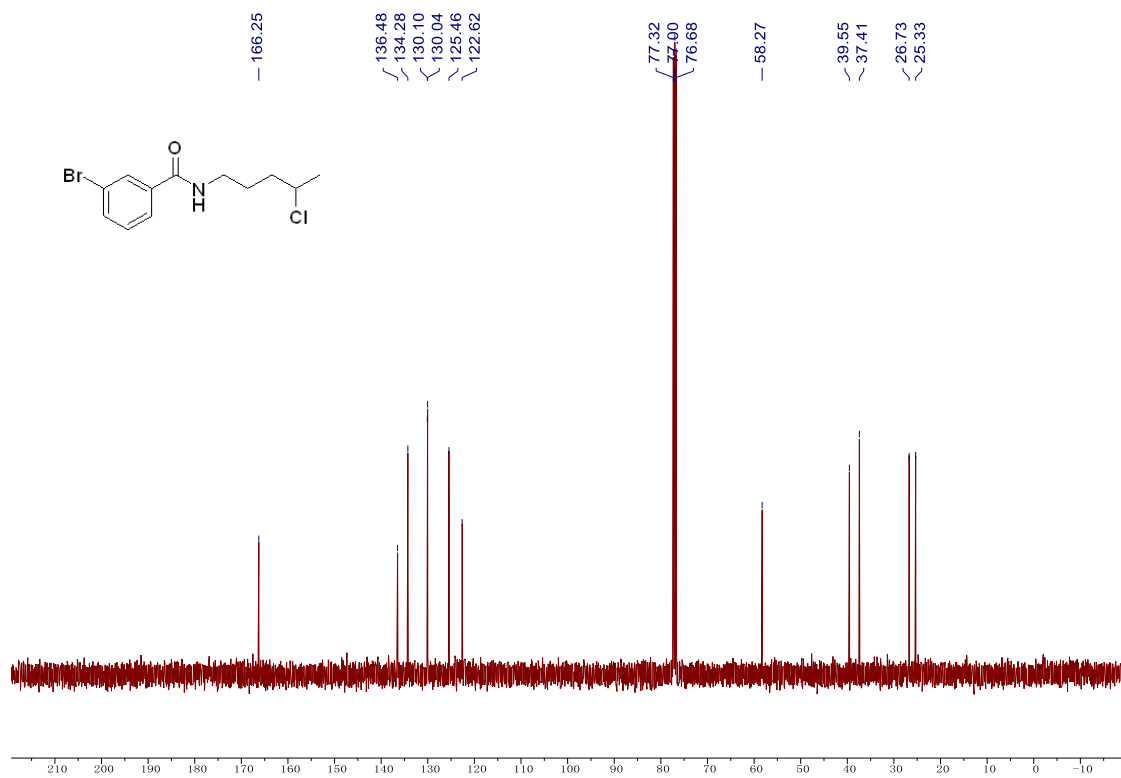


2k

^1H NMR (CDCl_3 , 400 MHz)

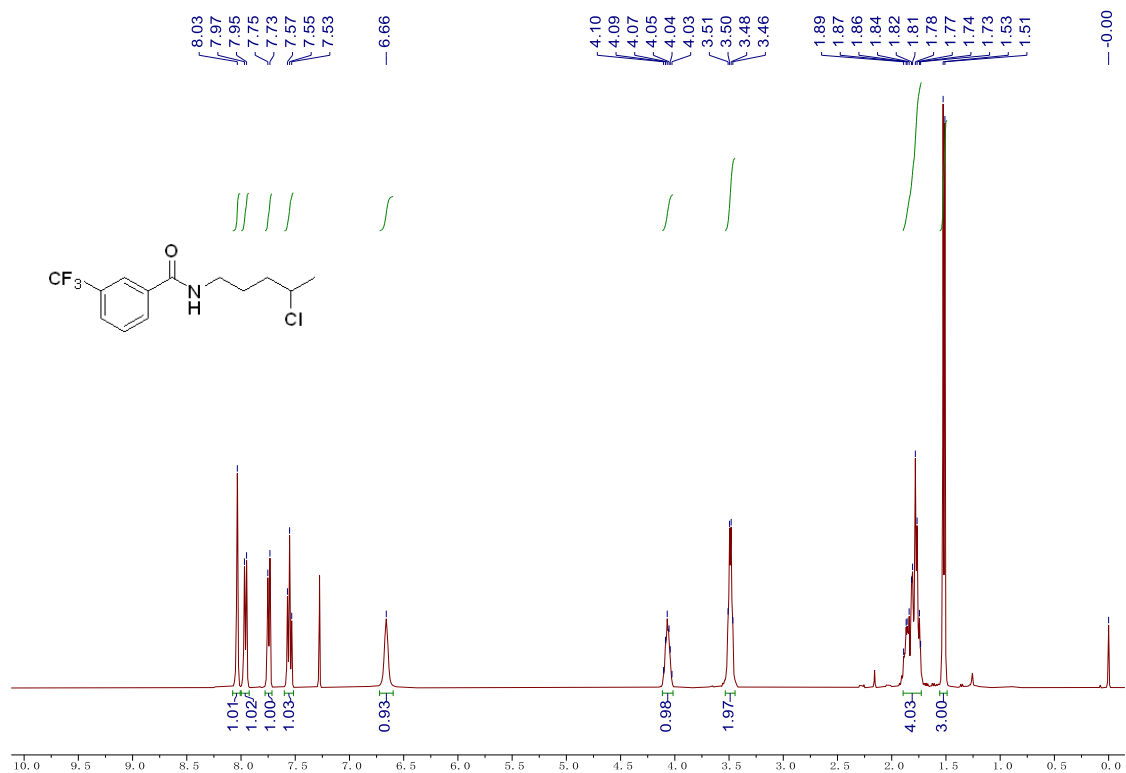


^{13}C NMR (CDCl_3 , 100 MHz)

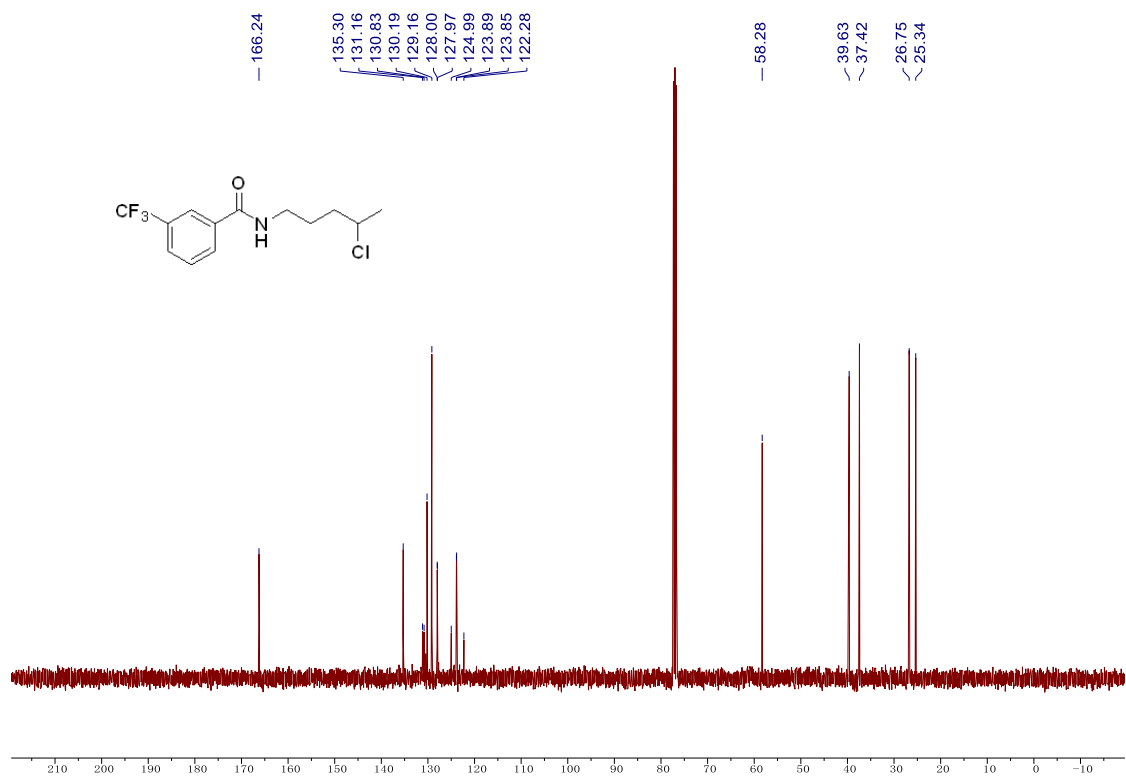


21

¹H NMR (CDCl₃, 400 MHz)

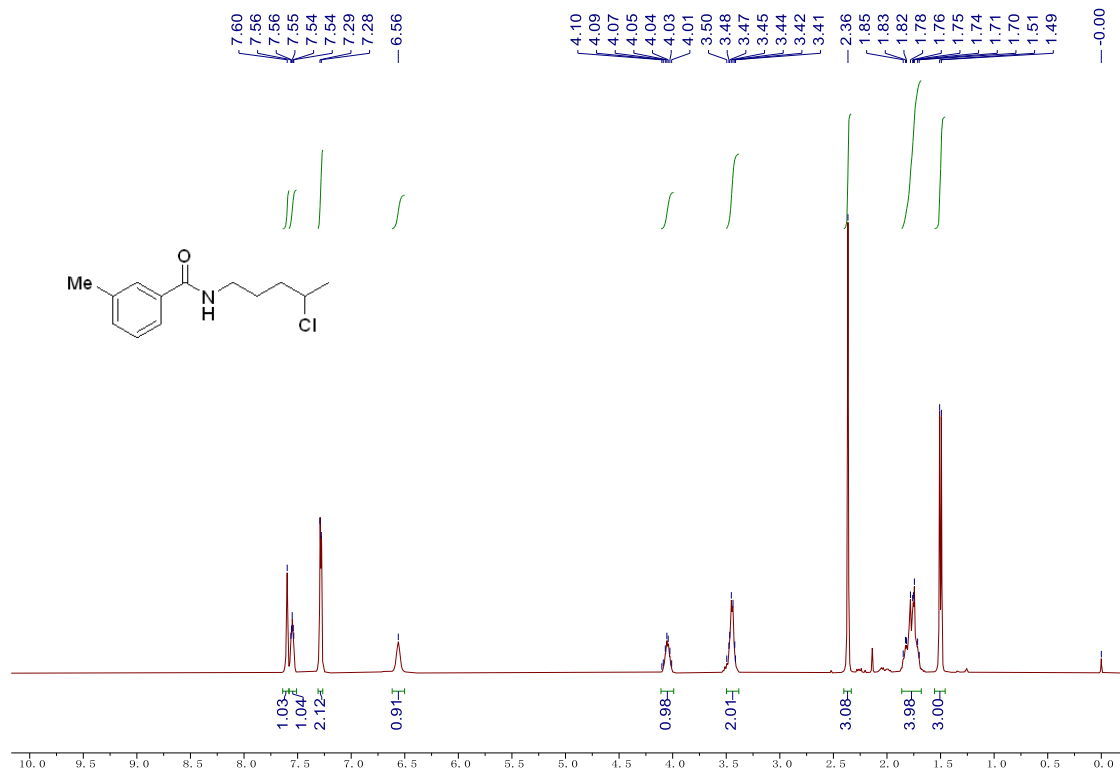


¹³C NMR (CDCl₃, 100 MHz)

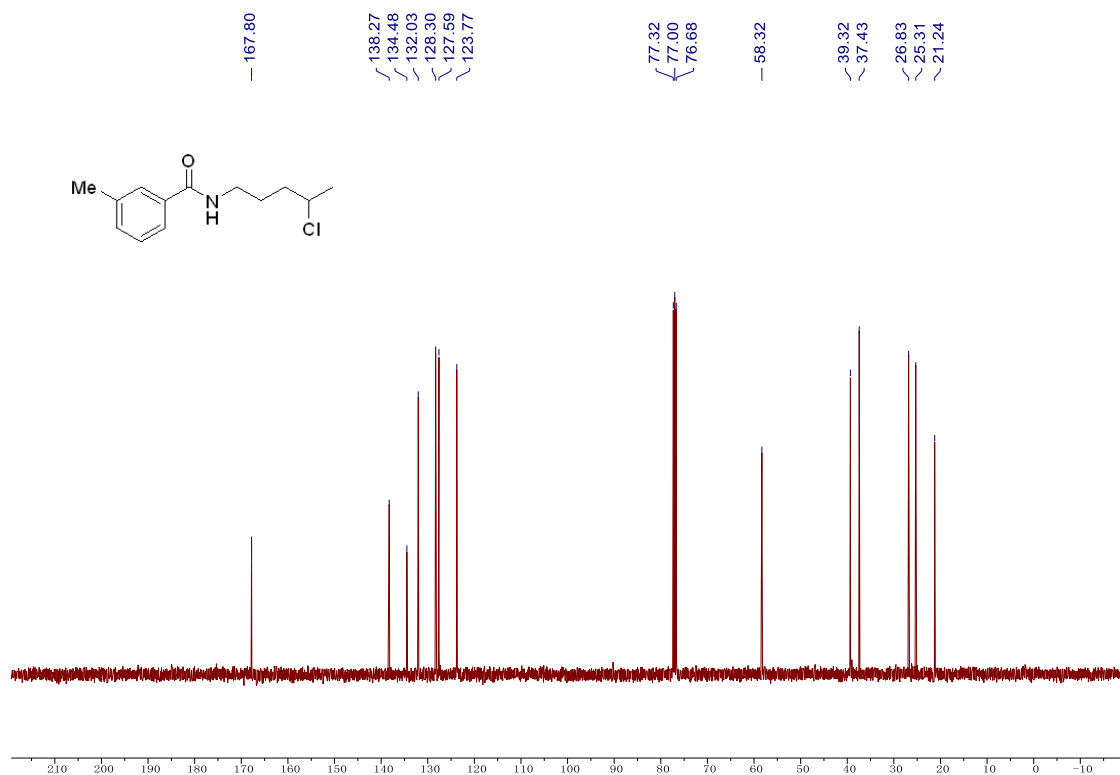


2m

^1H NMR (CDCl_3 , 400 MHz)

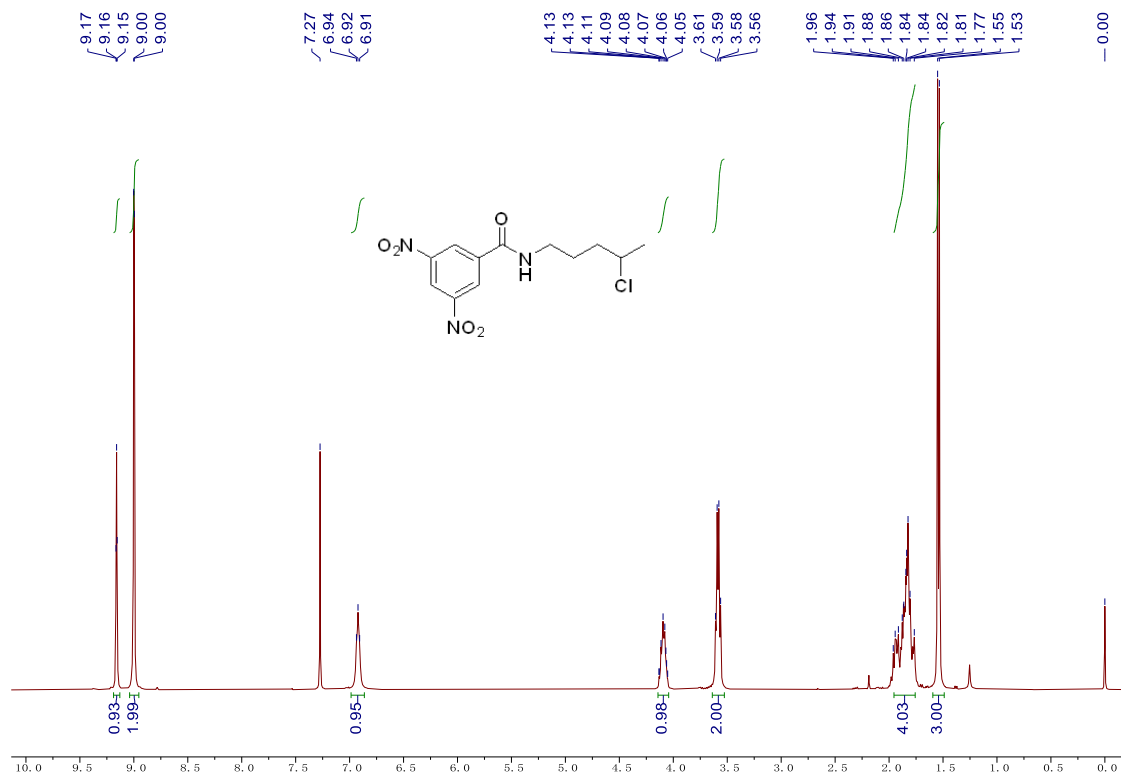


^{13}C NMR (CDCl_3 , 100 MHz)

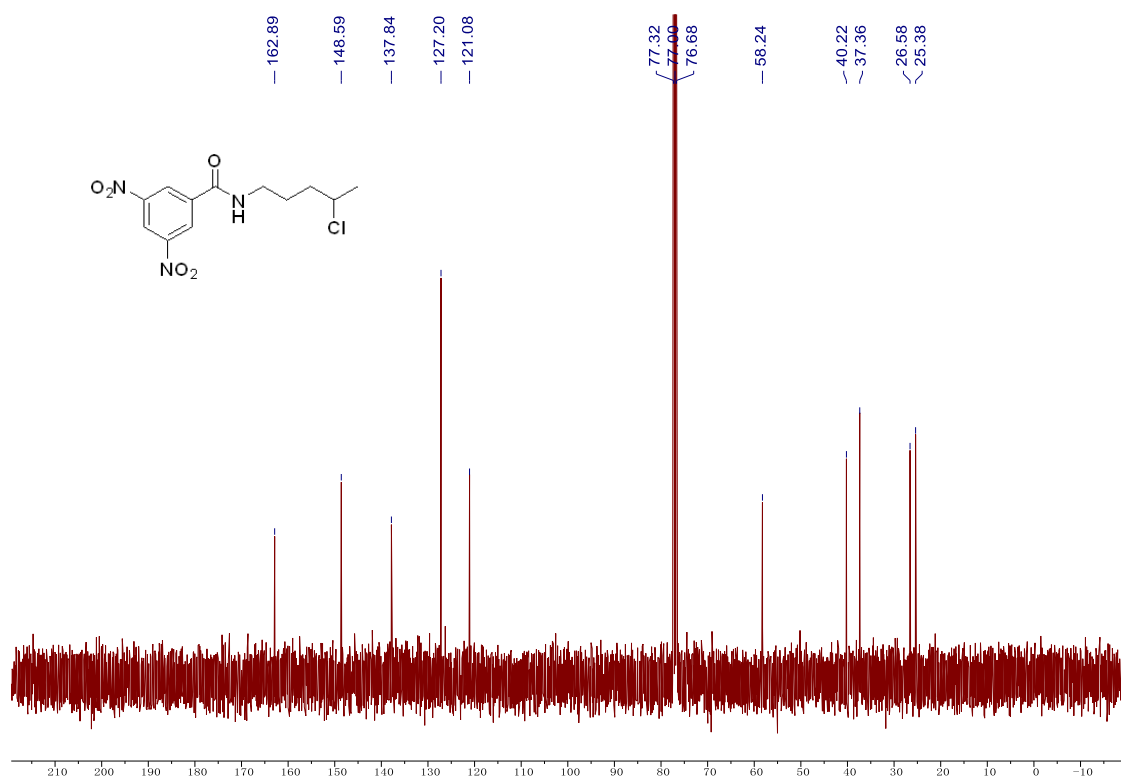


2n

¹H NMR (CDCl₃, 400 MHz)

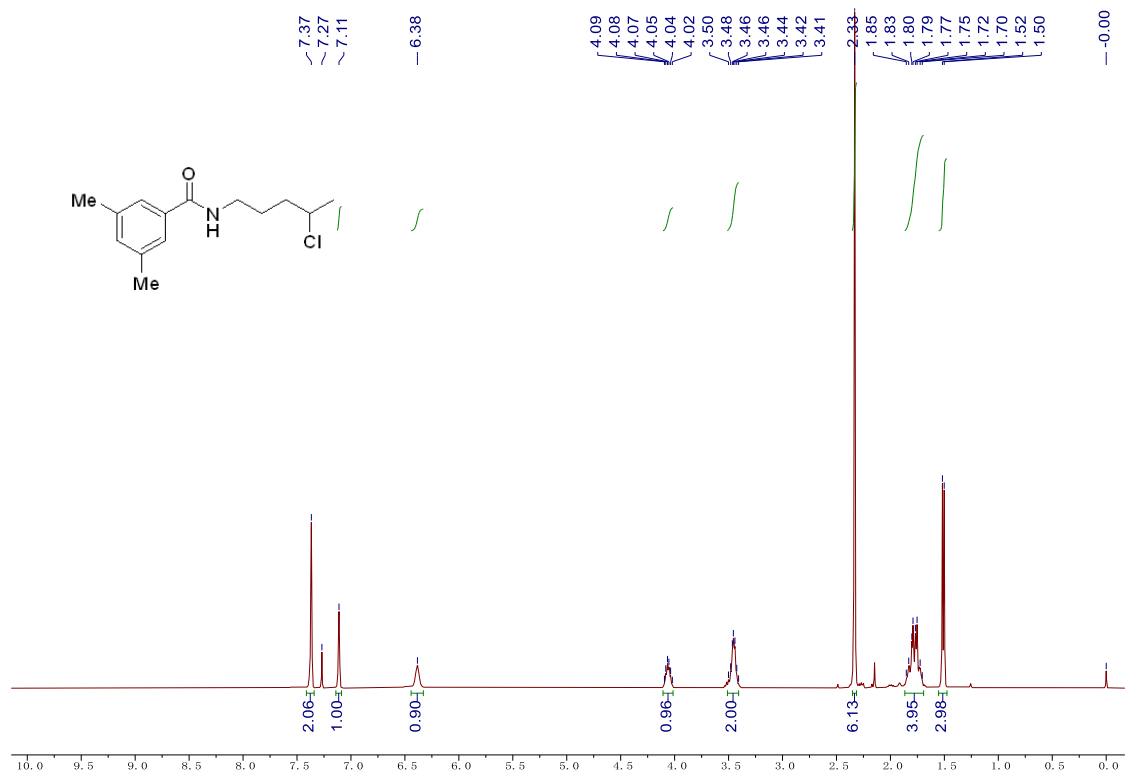


¹³C NMR (CDCl₃, 100 MHz)

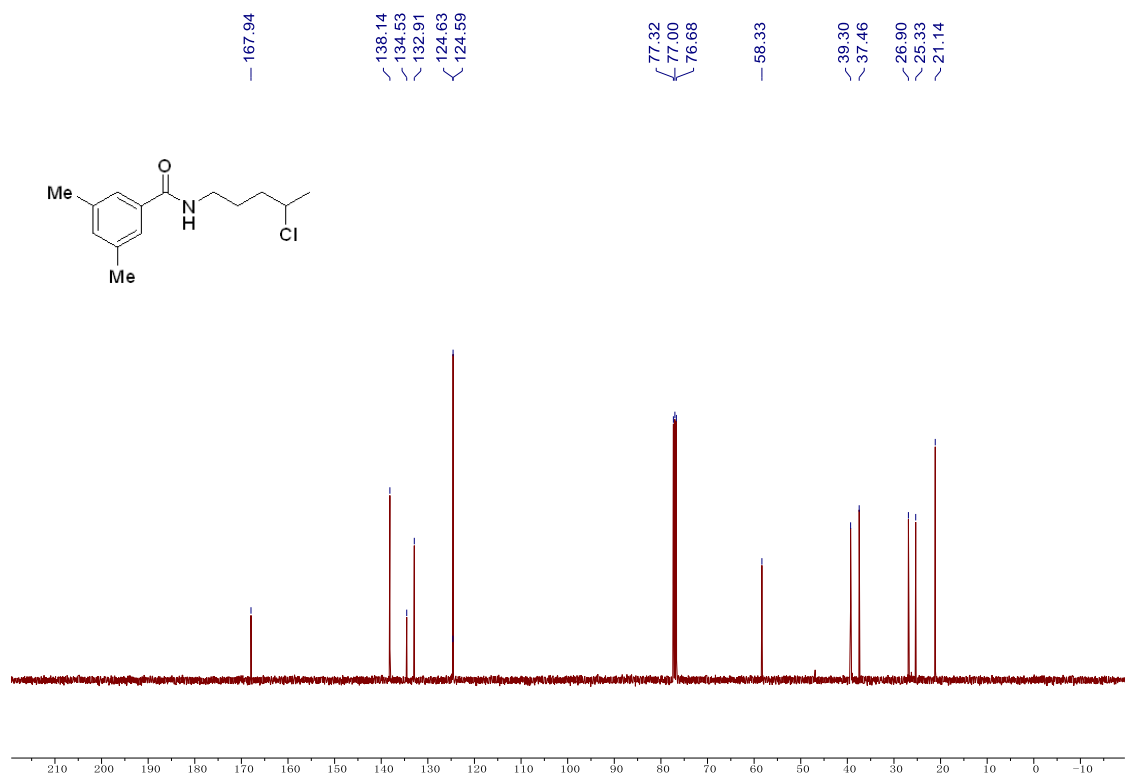


20

^1H NMR (CDCl_3 , 400 MHz)

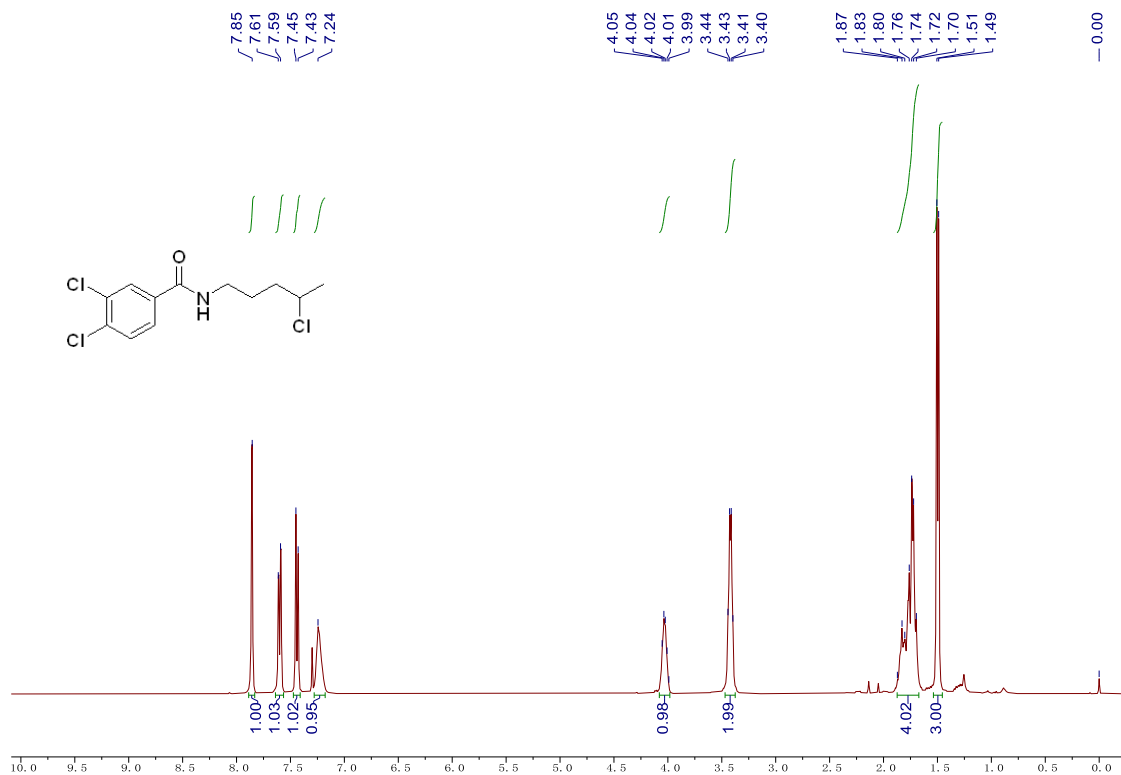


^{13}C NMR (CDCl_3 , 100 MHz)

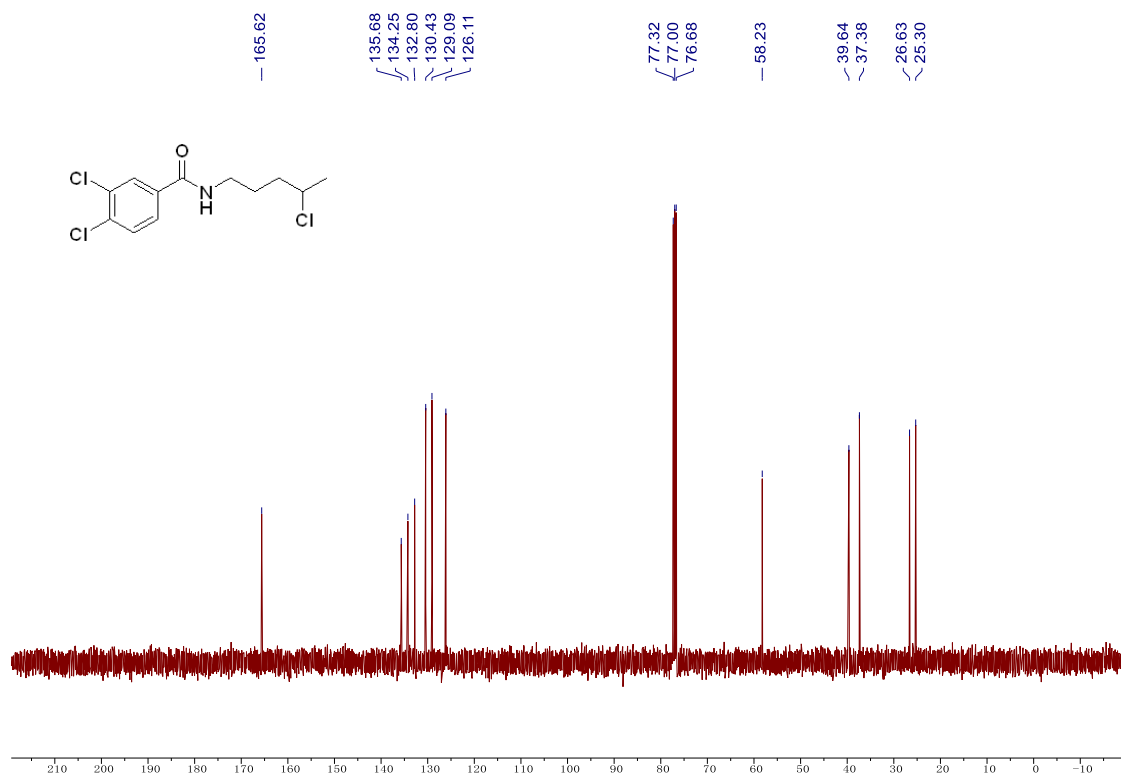


2p

^1H NMR (CDCl_3 , 400 MHz)

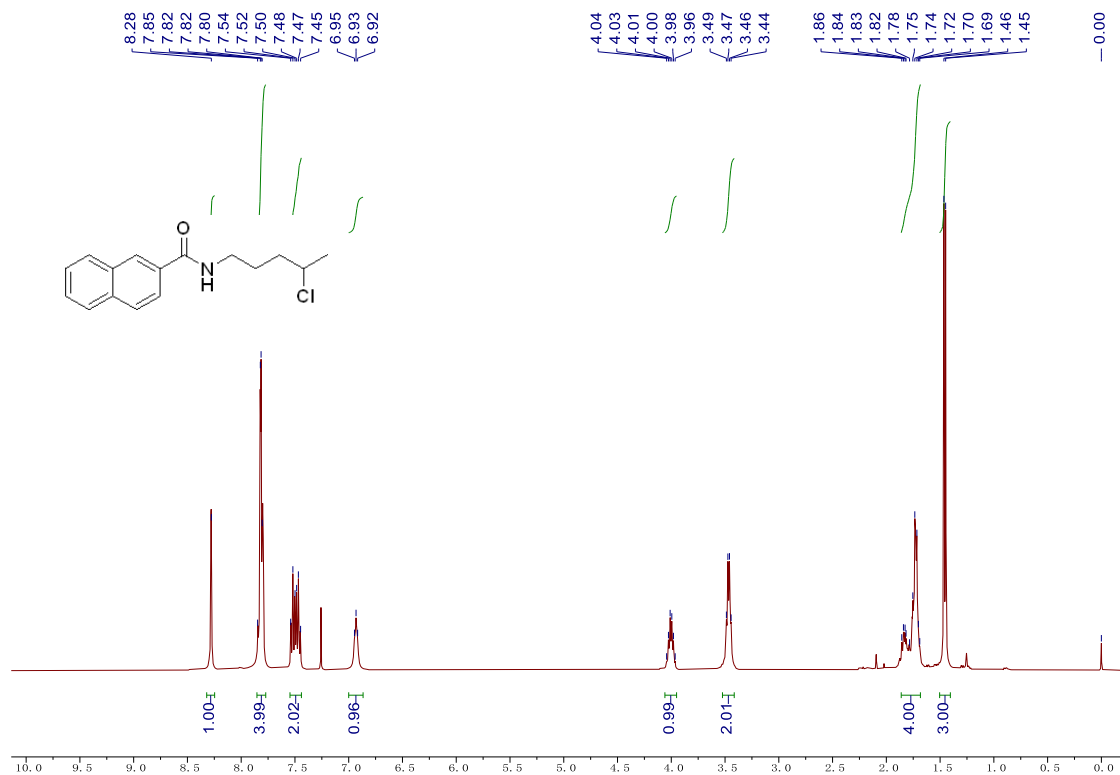


^{13}C NMR (CDCl_3 , 100 MHz)

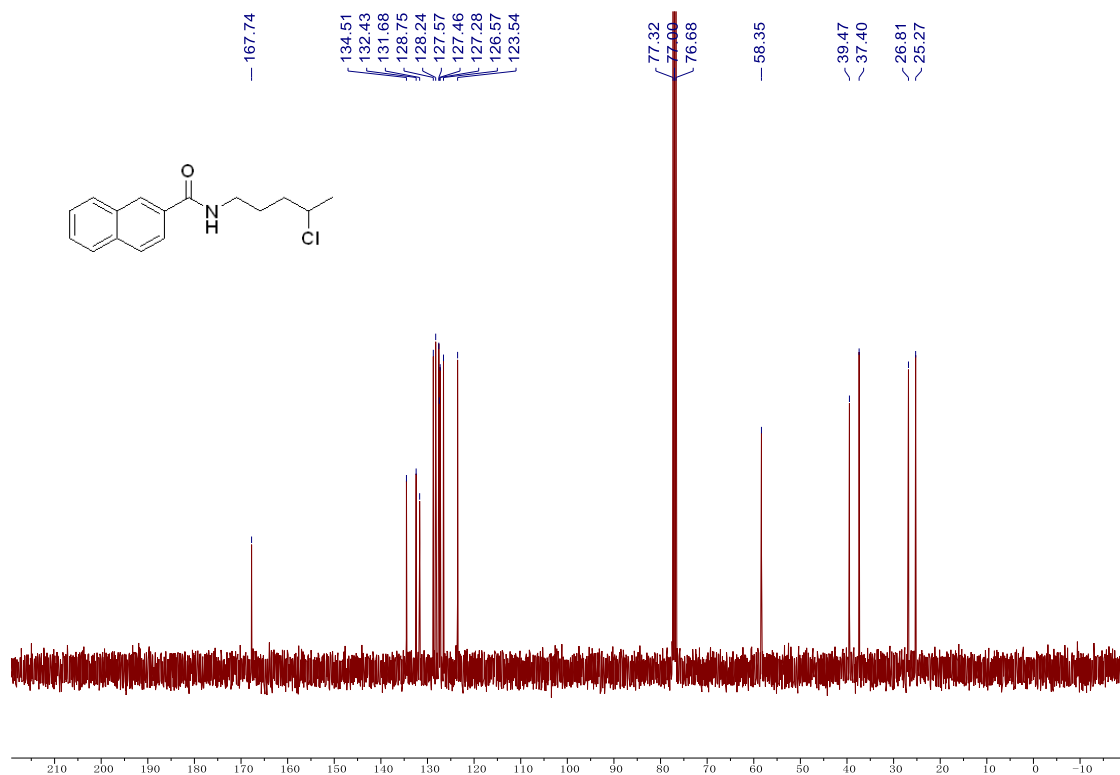


2q

^1H NMR (CDCl_3 , 400 MHz)

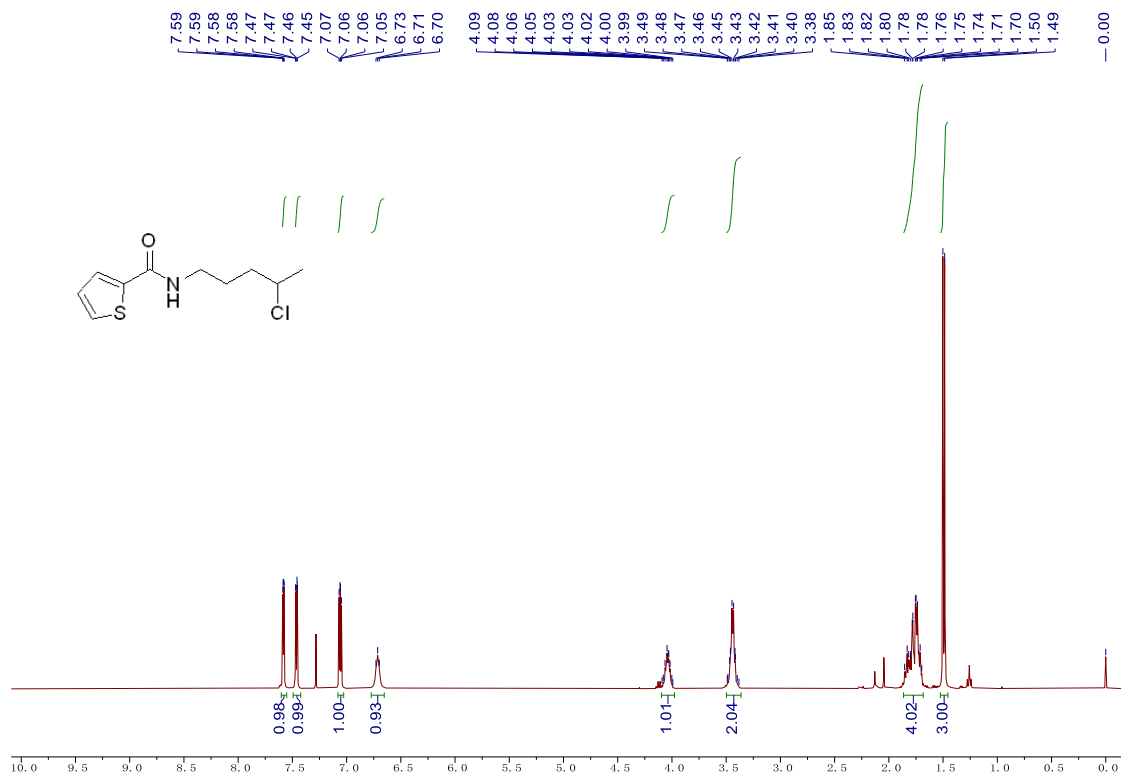


^{13}C NMR (CDCl_3 , 100 MHz)

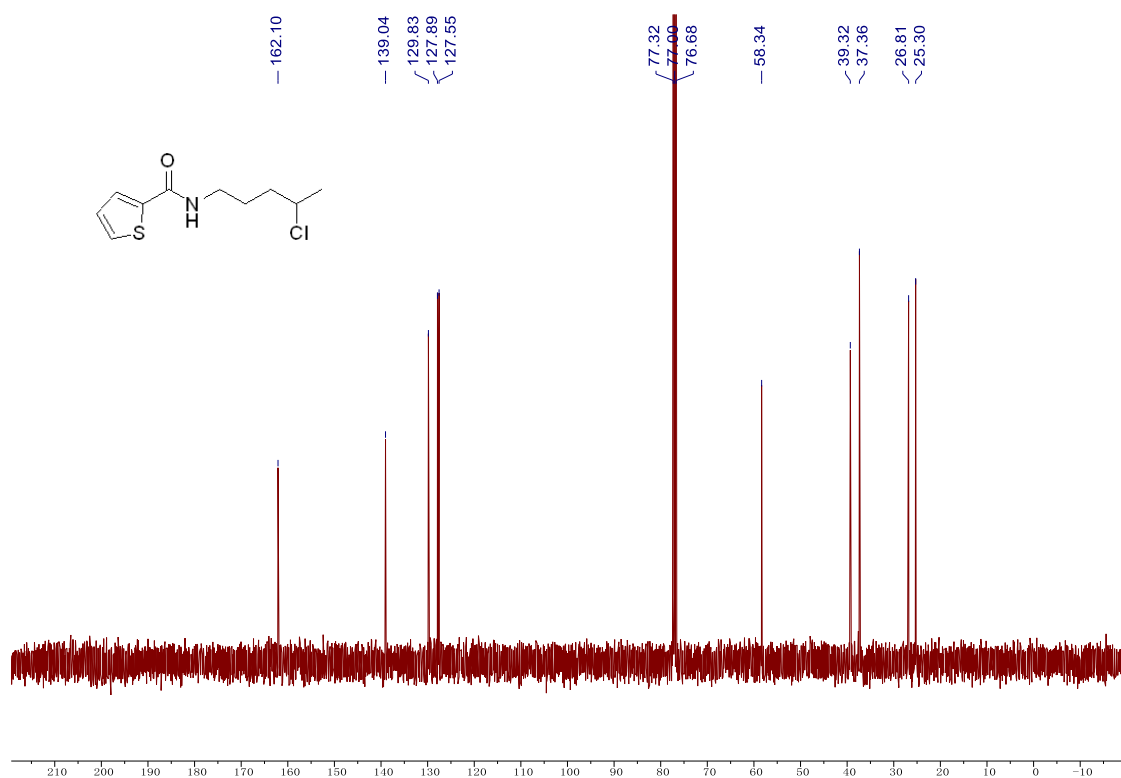


2r

^1H NMR (CDCl_3 , 400 MHz)

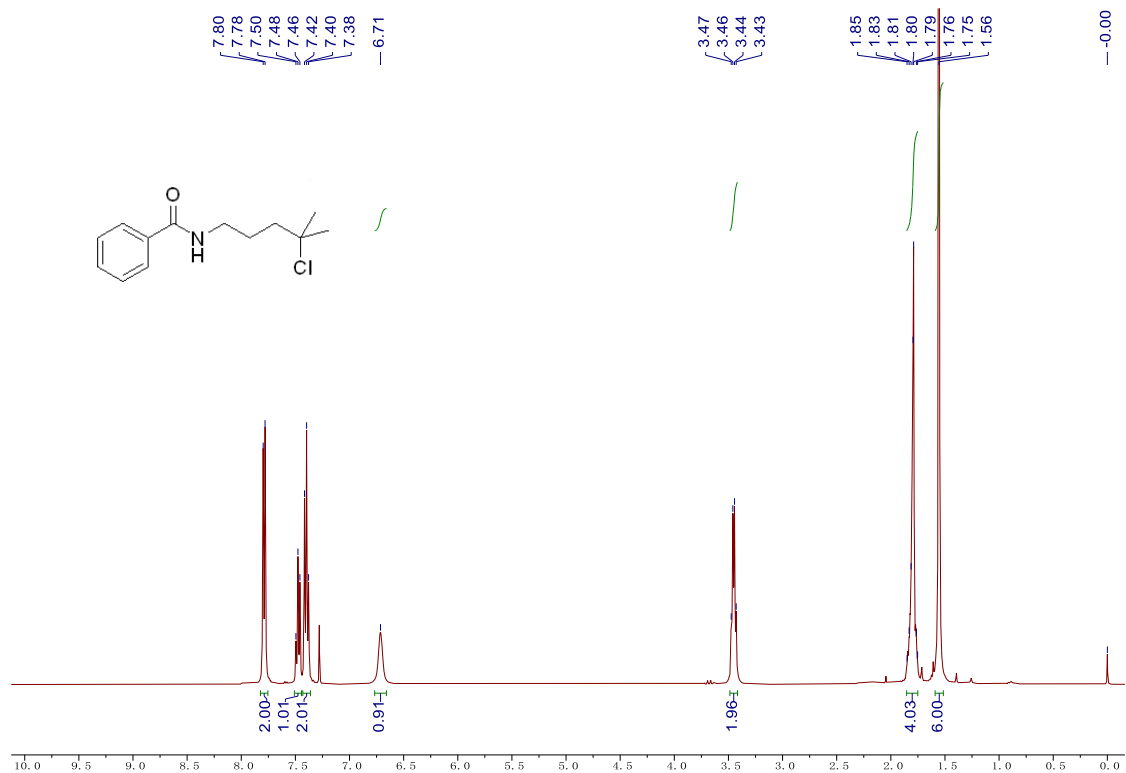


^{13}C NMR (CDCl_3 , 100 MHz)

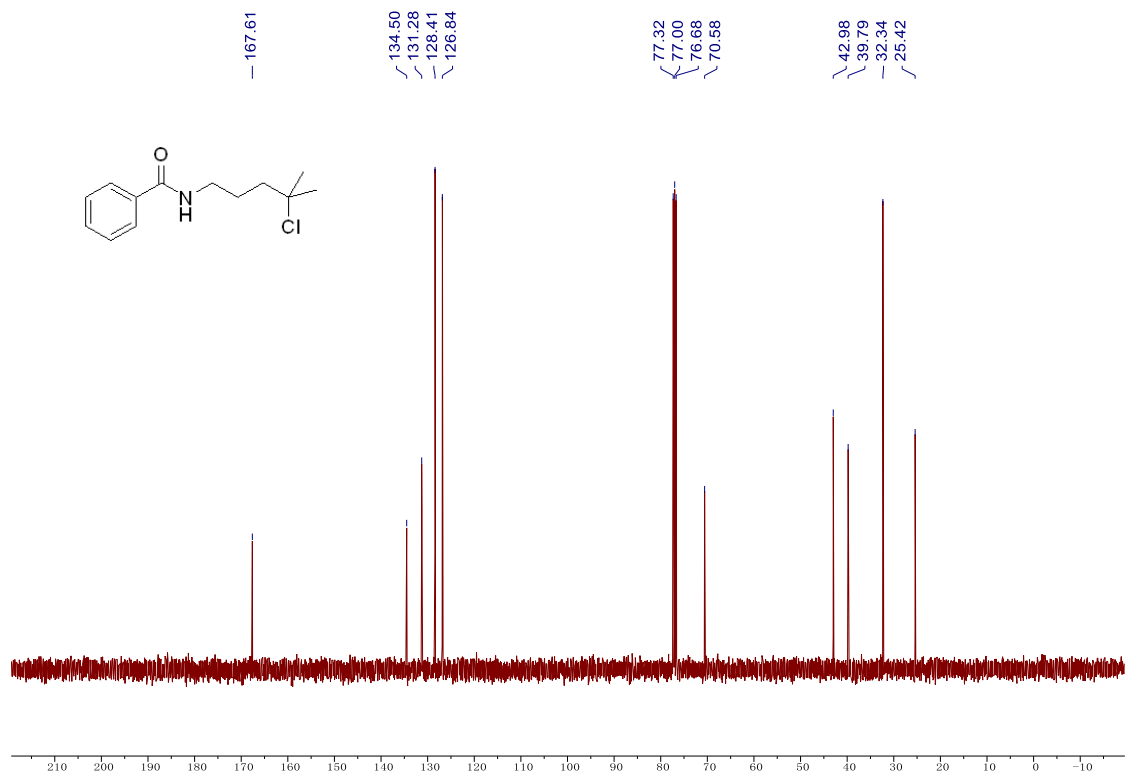


2s

^1H NMR (CDCl_3 , 400 MHz)

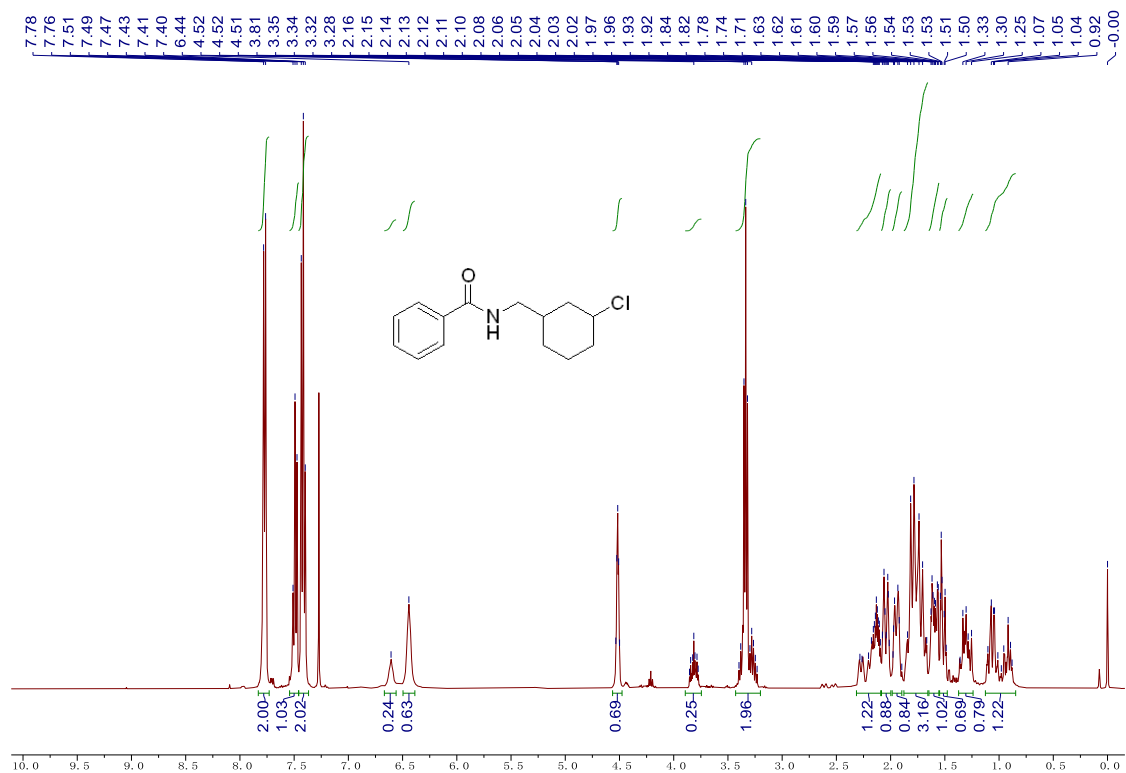


^{13}C NMR (CDCl_3 , 100 MHz)

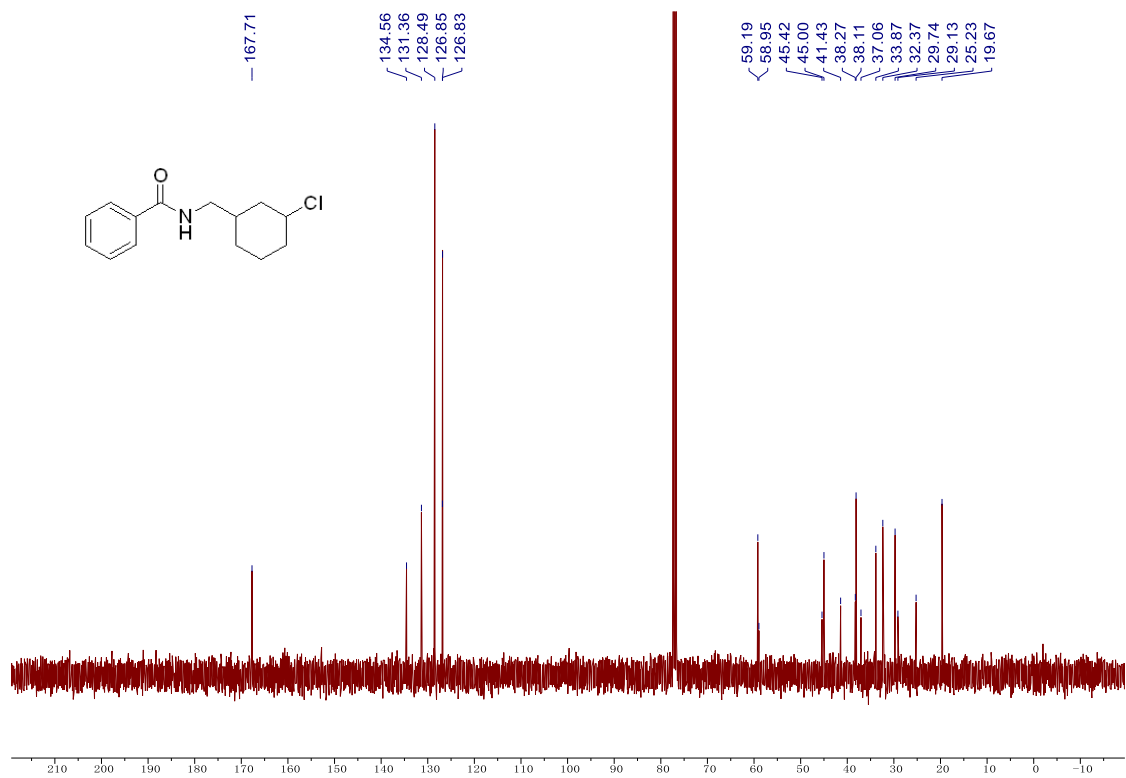


2t

^1H NMR (CDCl_3 , 400 MHz)

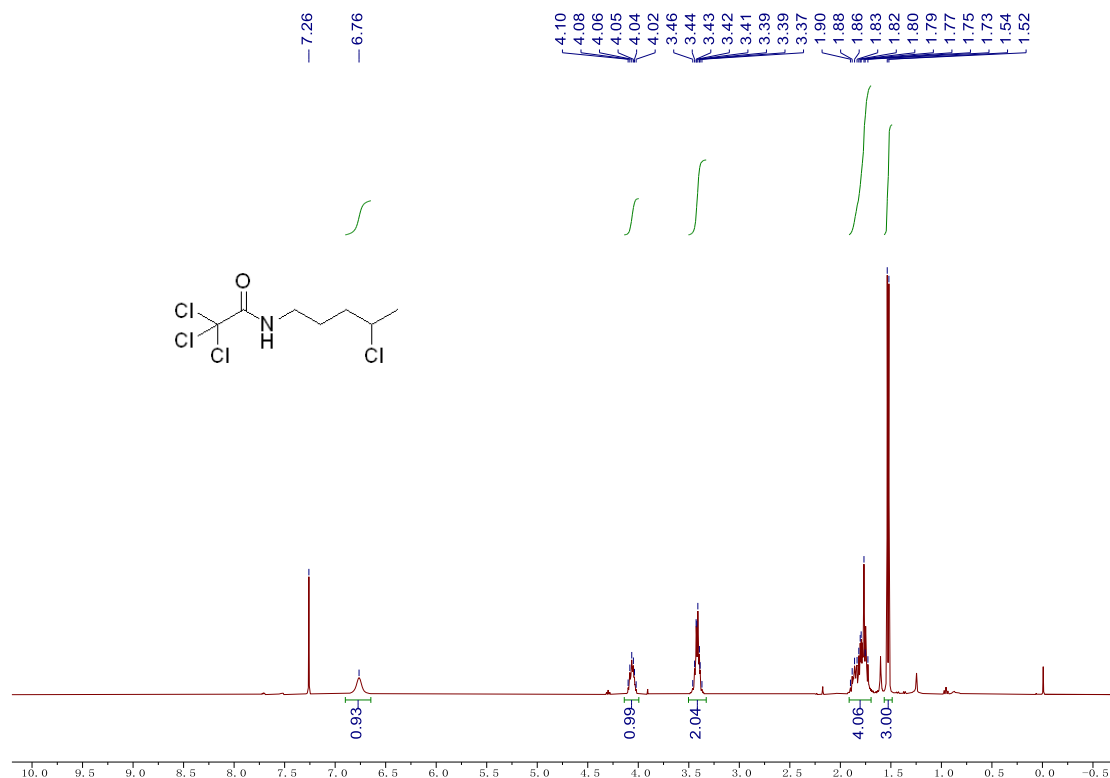


^{13}C NMR (CDCl_3 , 100 MHz)

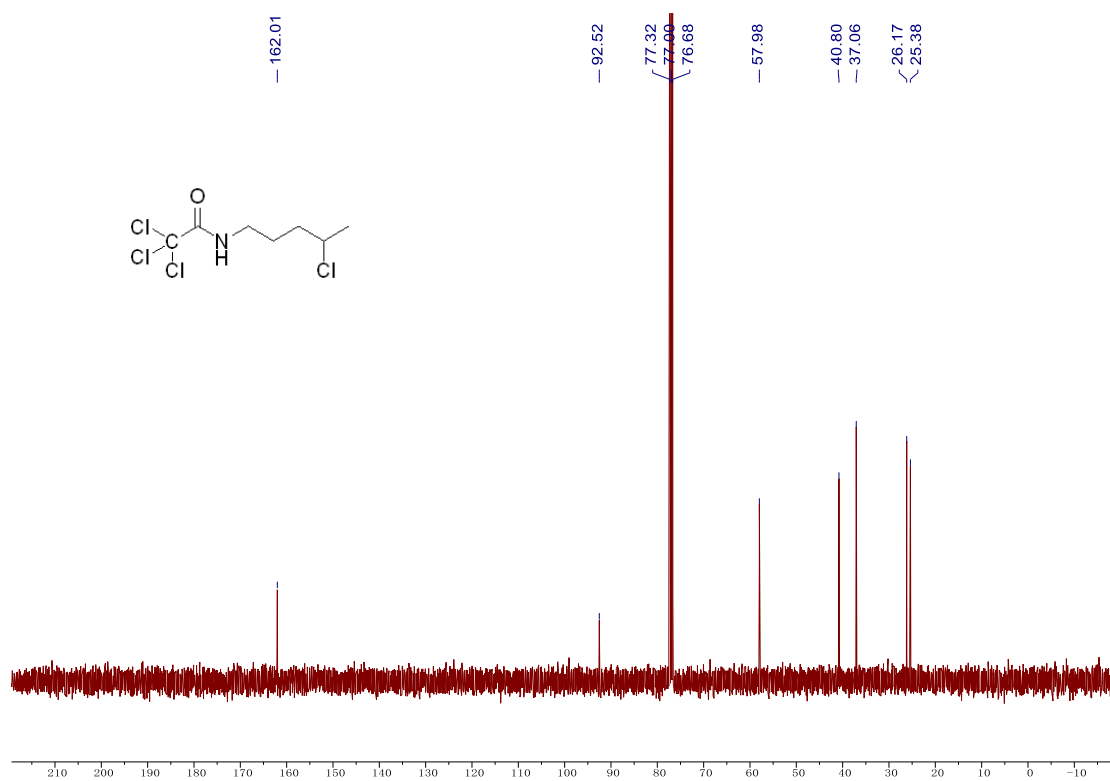


2u

^1H NMR (CDCl_3 , 400 MHz)

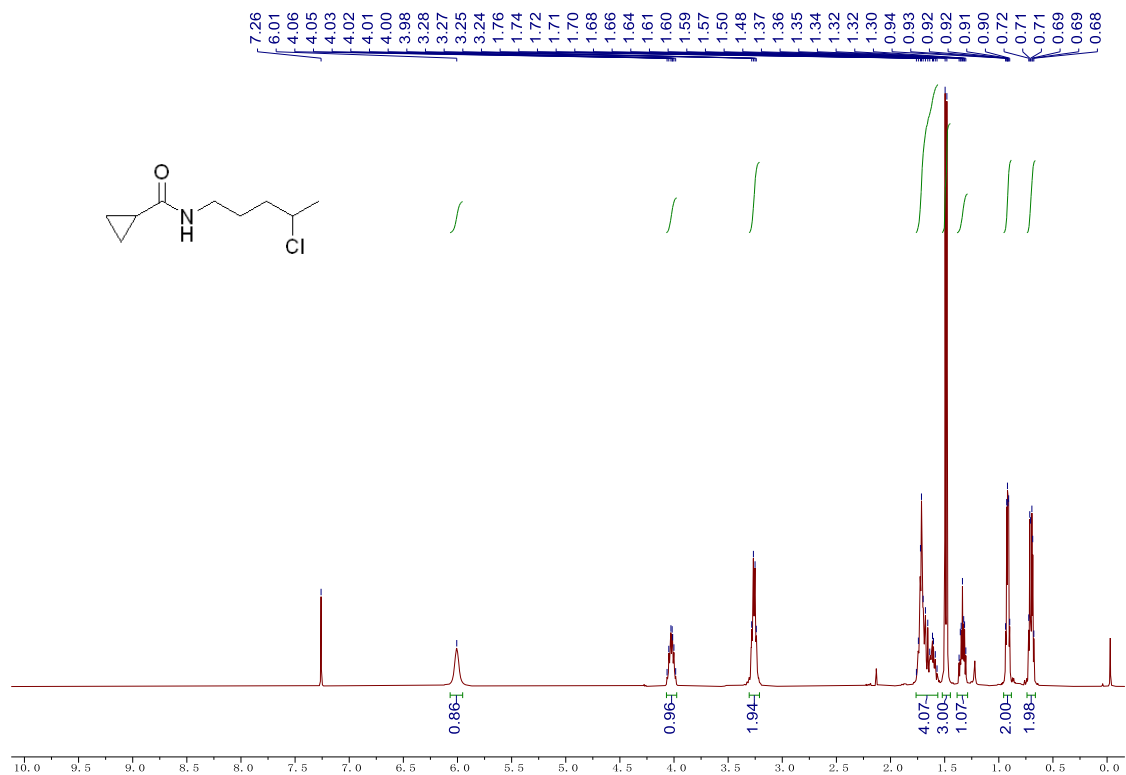


^{13}C NMR (CDCl_3 , 100 MHz)

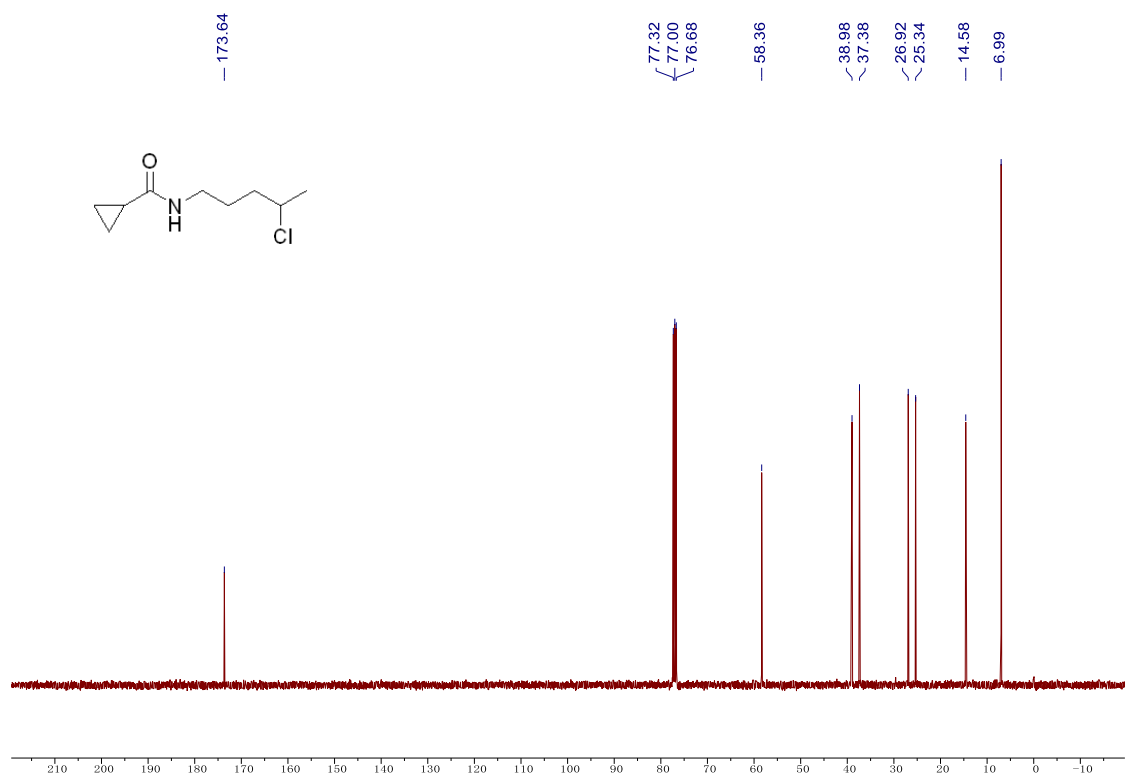


2v

^1H NMR (CDCl_3 , 400 MHz)

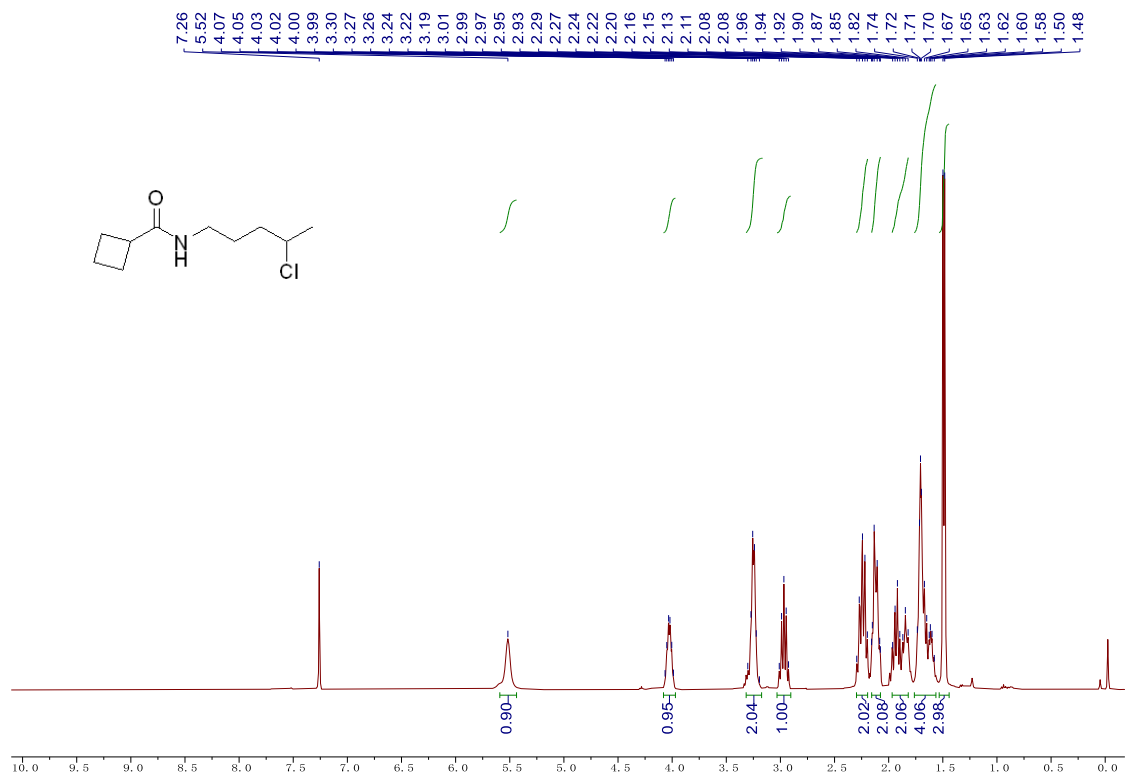


^{13}C NMR (CDCl_3 , 100 MHz)

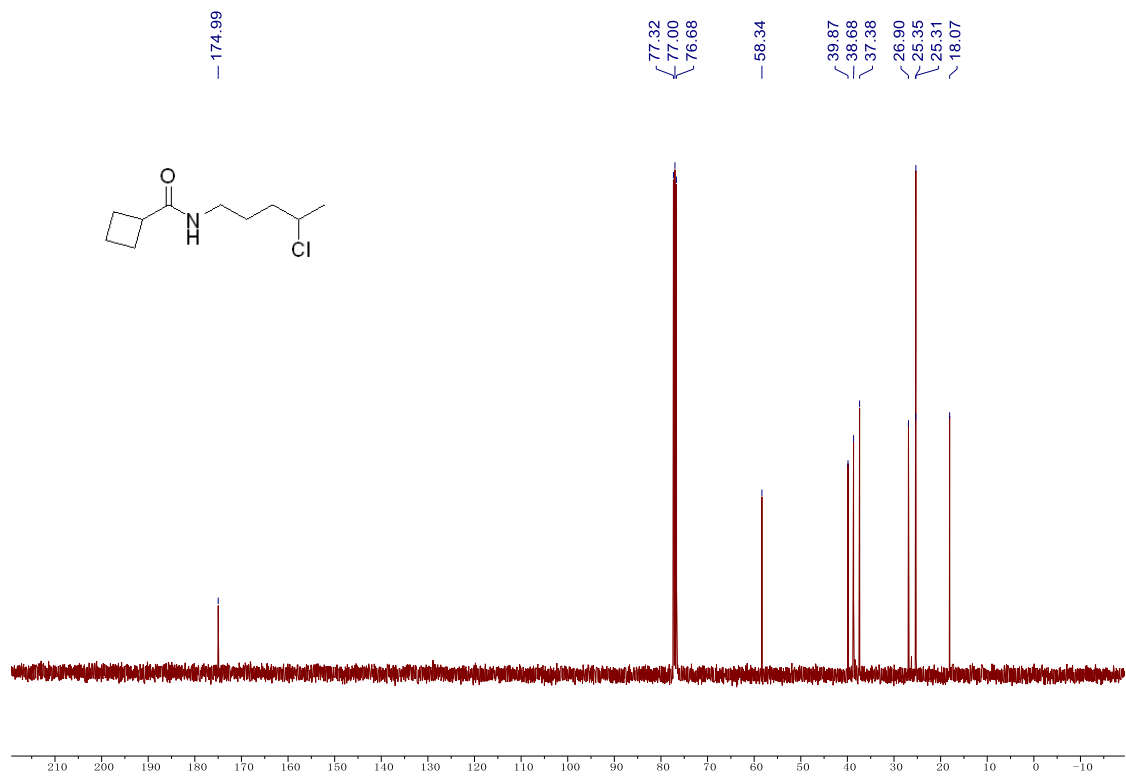


2w

^1H NMR (CDCl_3 , 400 MHz)

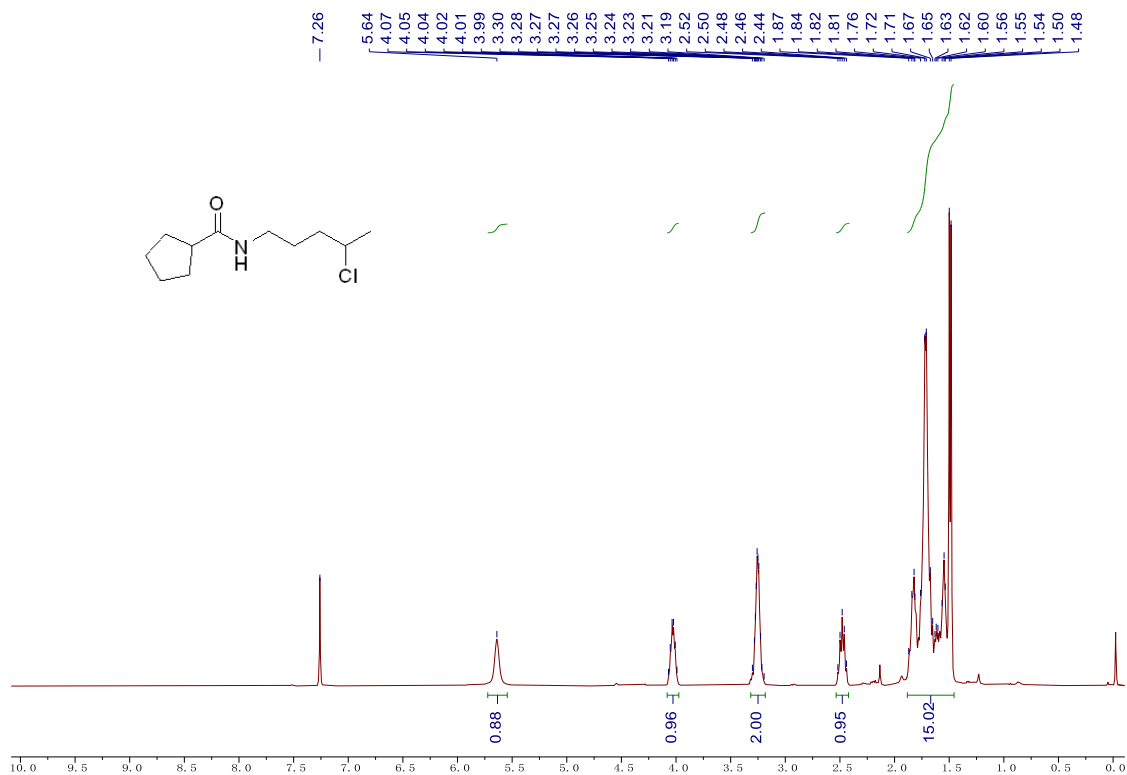


^{13}C NMR (CDCl_3 , 100 MHz)

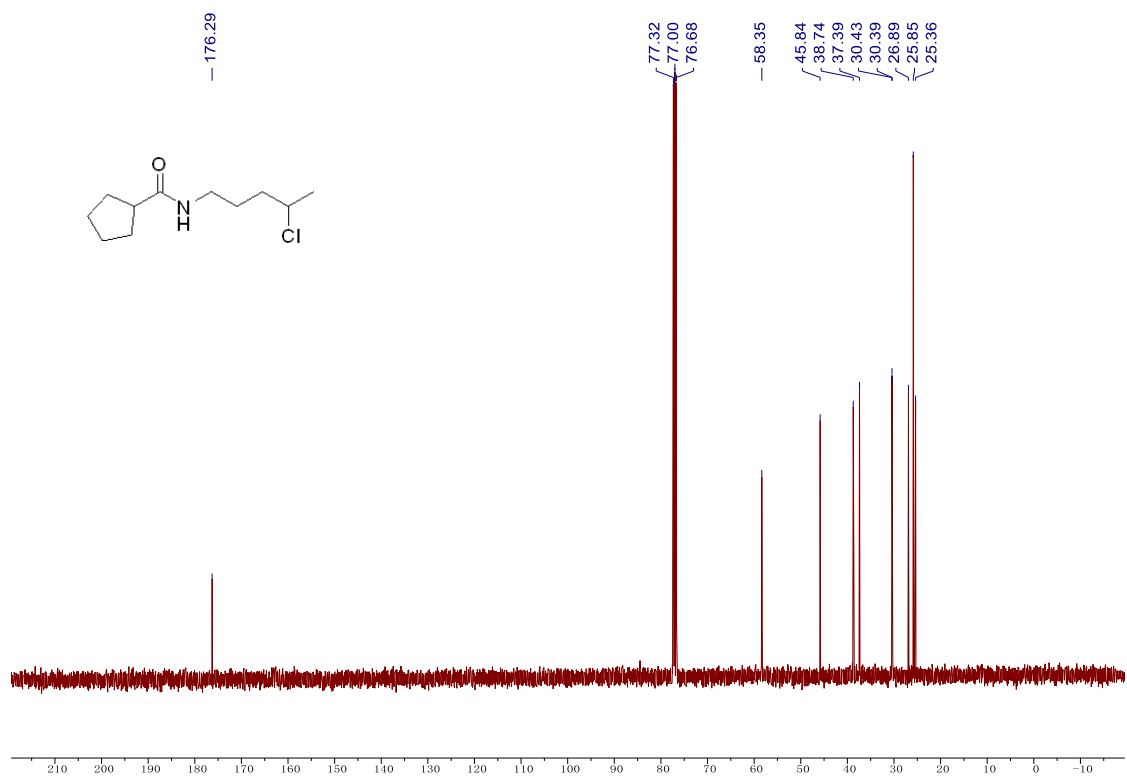


2x

^1H NMR (CDCl_3 , 400 MHz)

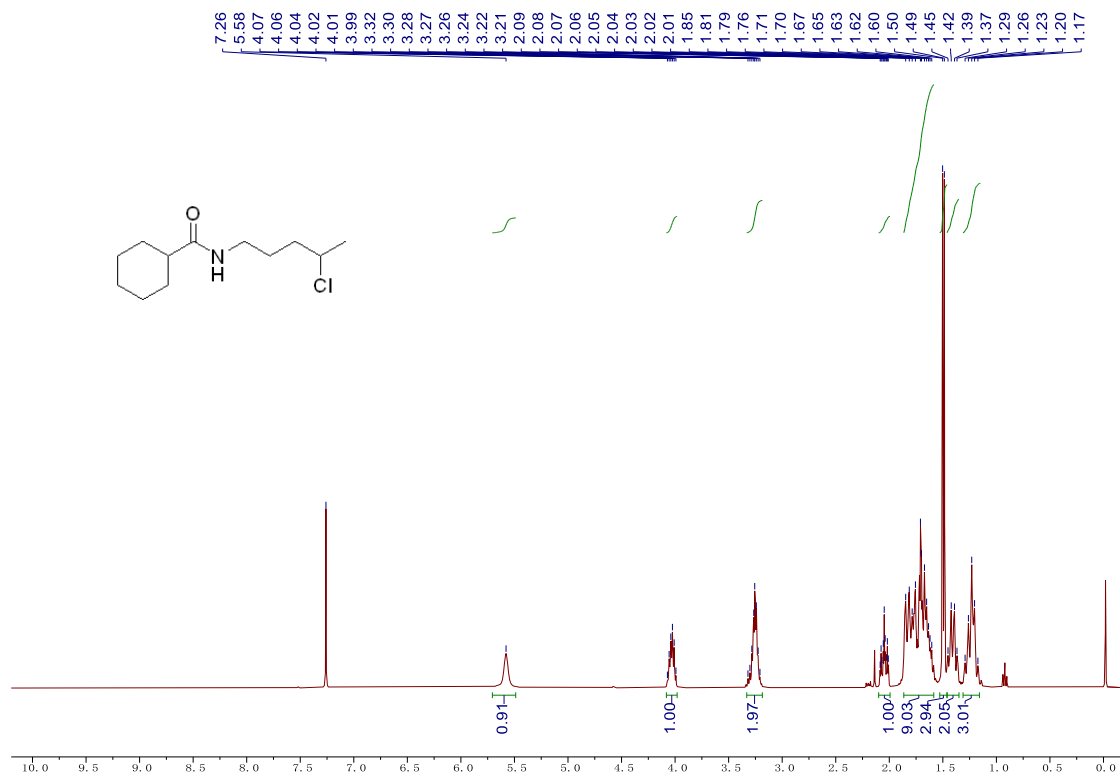


^{13}C NMR (CDCl_3 , 100 MHz)

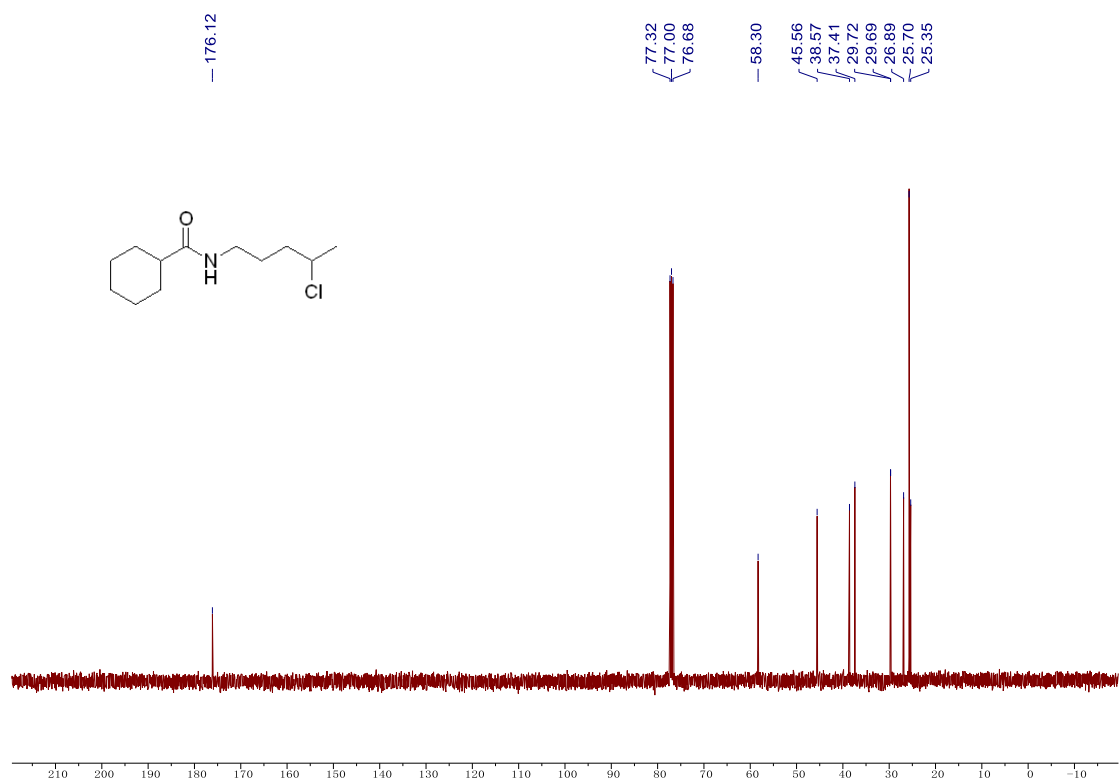


2y

^1H NMR (CDCl_3 , 400 MHz)

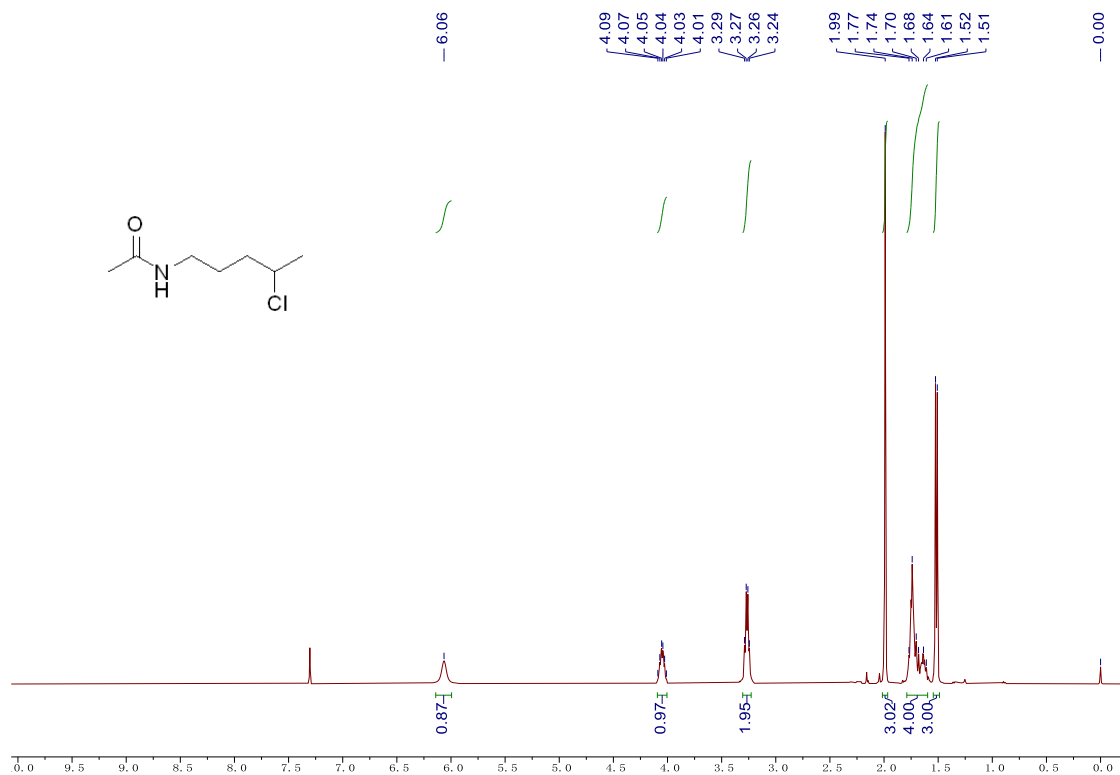


^{13}C NMR (CDCl_3 , 100 MHz)

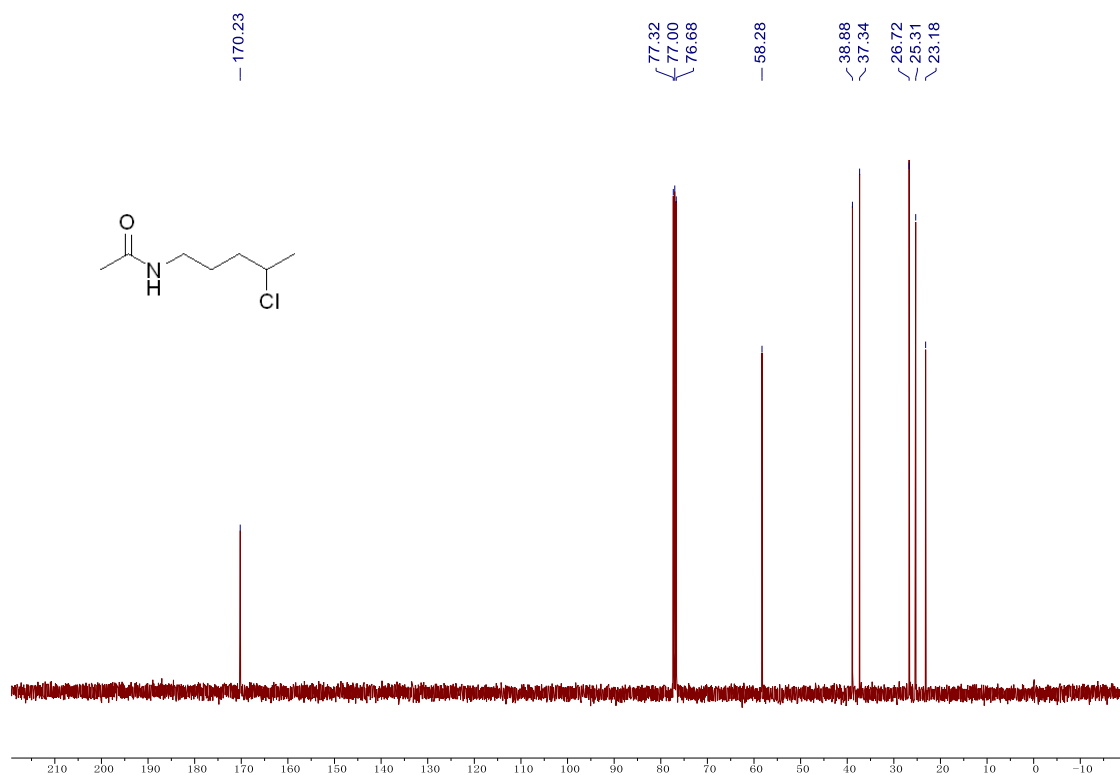


2z

^1H NMR (CDCl_3 , 400 MHz)

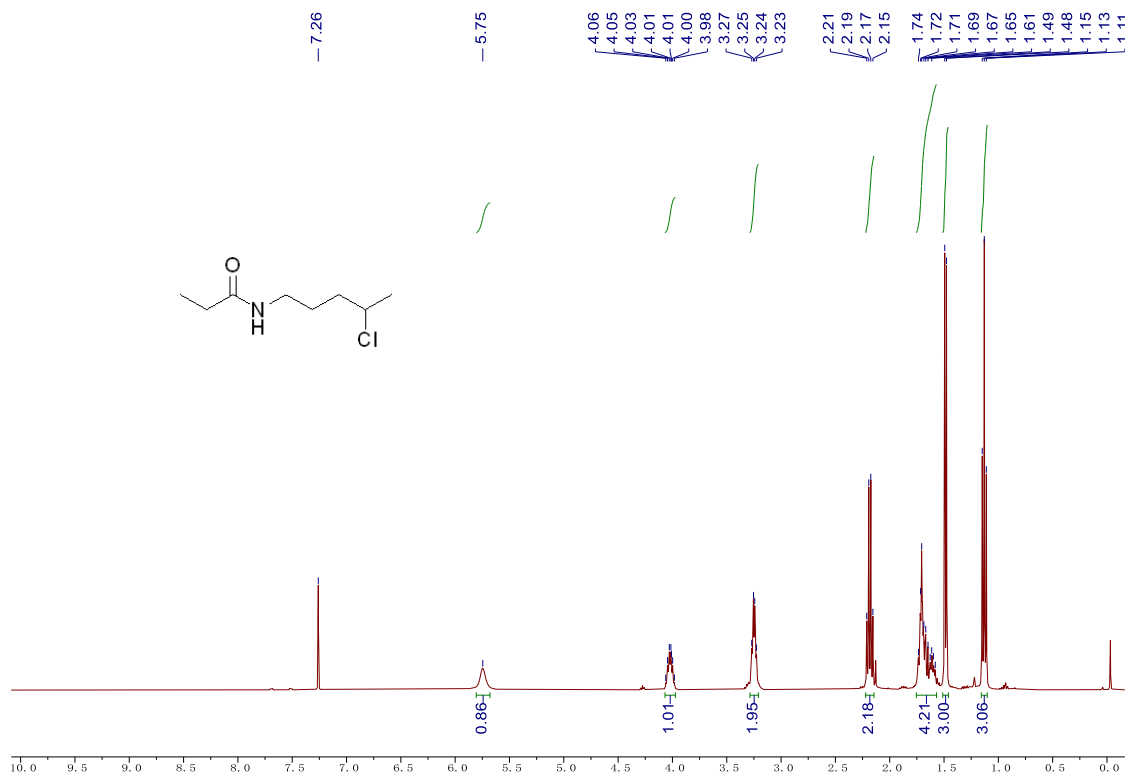


^{13}C NMR (CDCl_3 , 100 MHz)

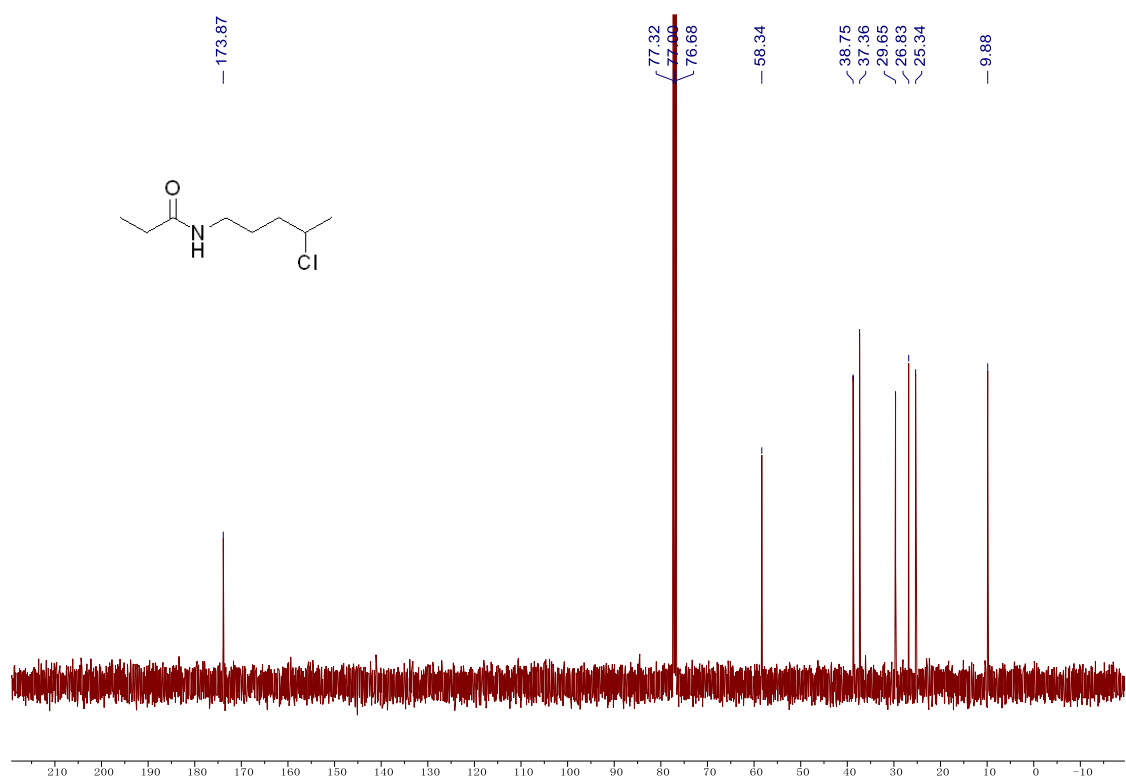


2aa

^1H NMR (CDCl_3 , 400 MHz)

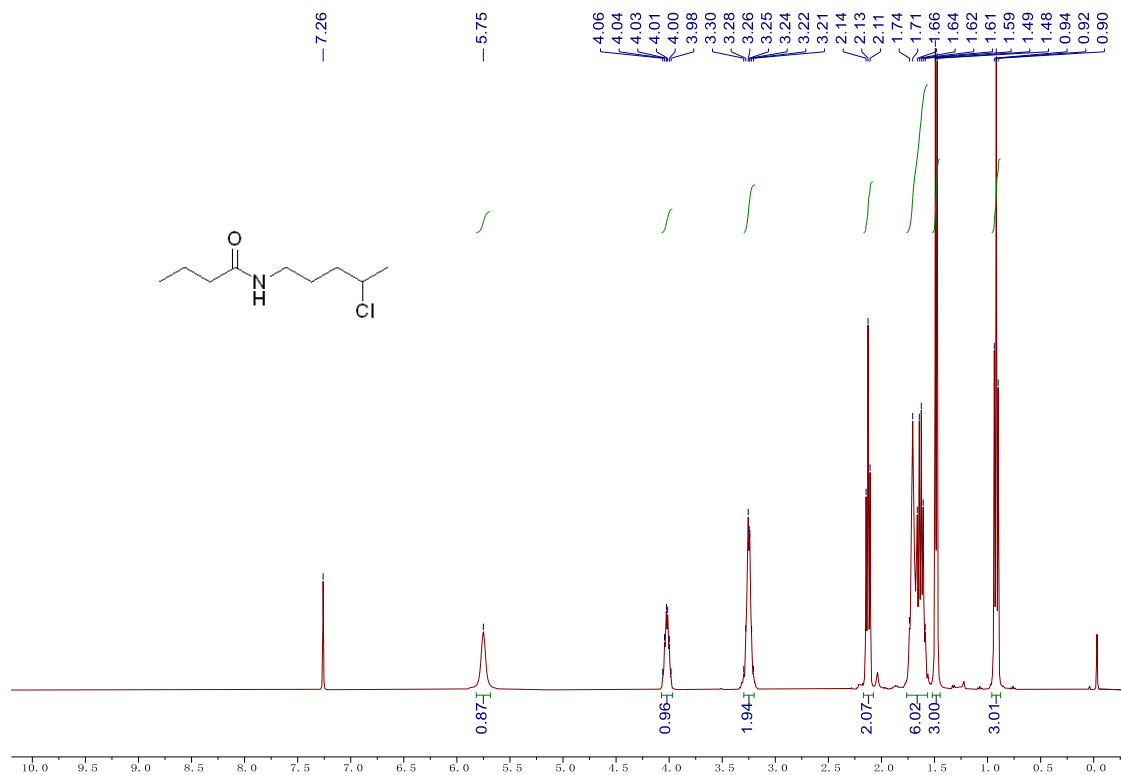


^{13}C NMR (CDCl_3 , 100 MHz)

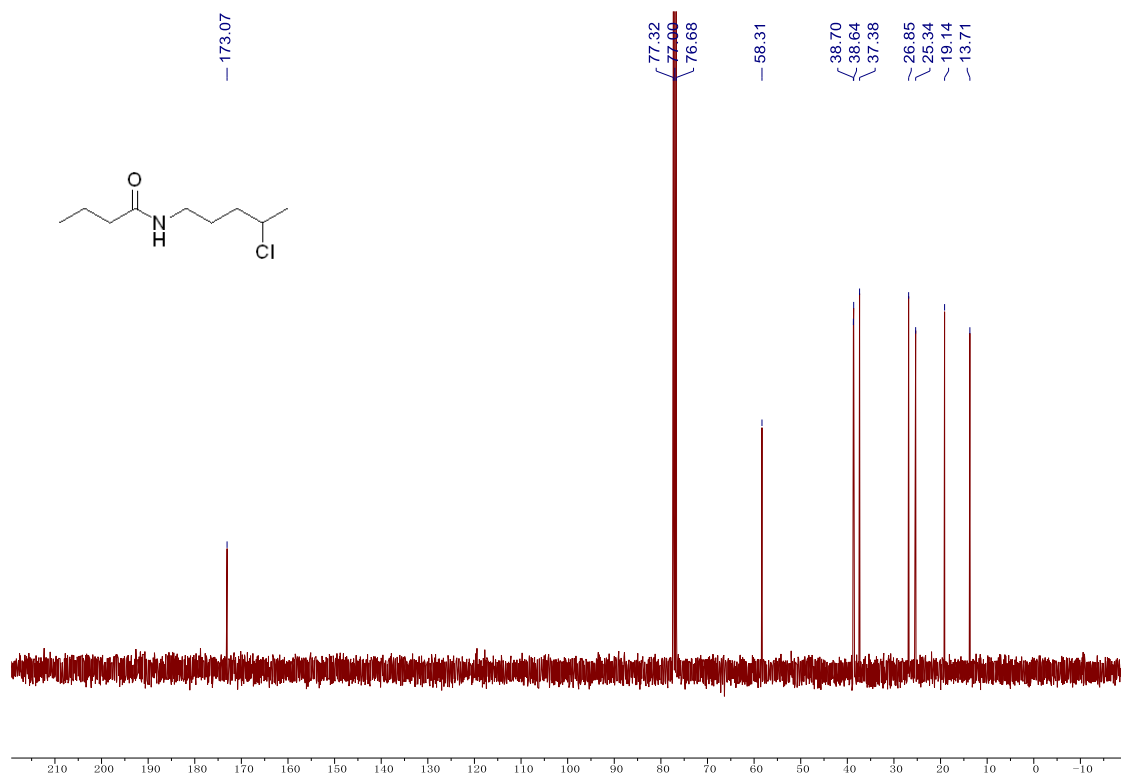


2ab

^1H NMR (CDCl_3 , 400 MHz)

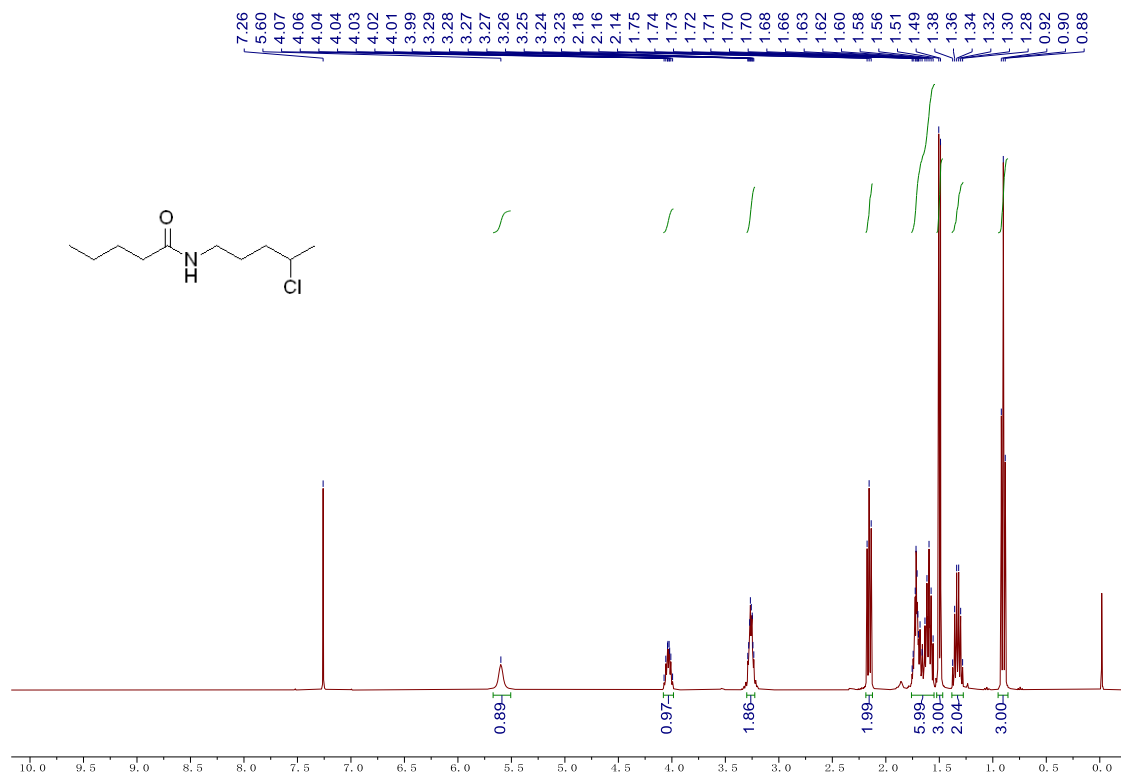


^{13}C NMR (CDCl_3 , 100 MHz)

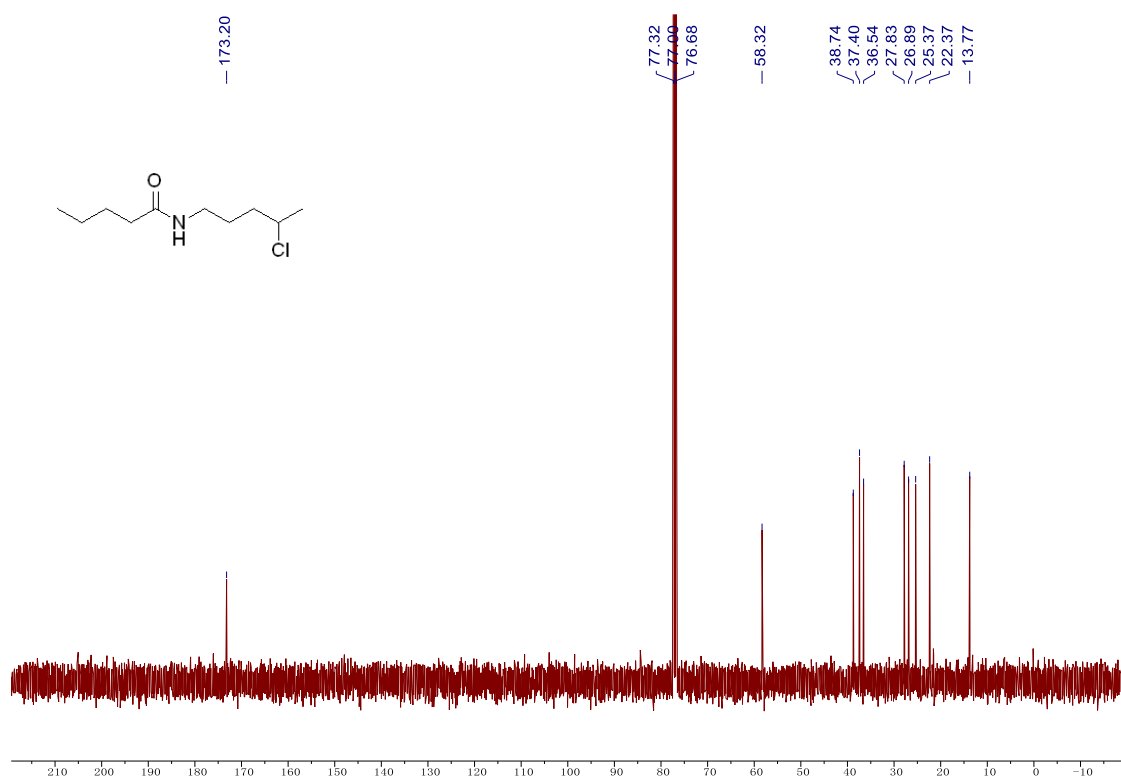


2ac

^1H NMR (CDCl_3 , 400 MHz)

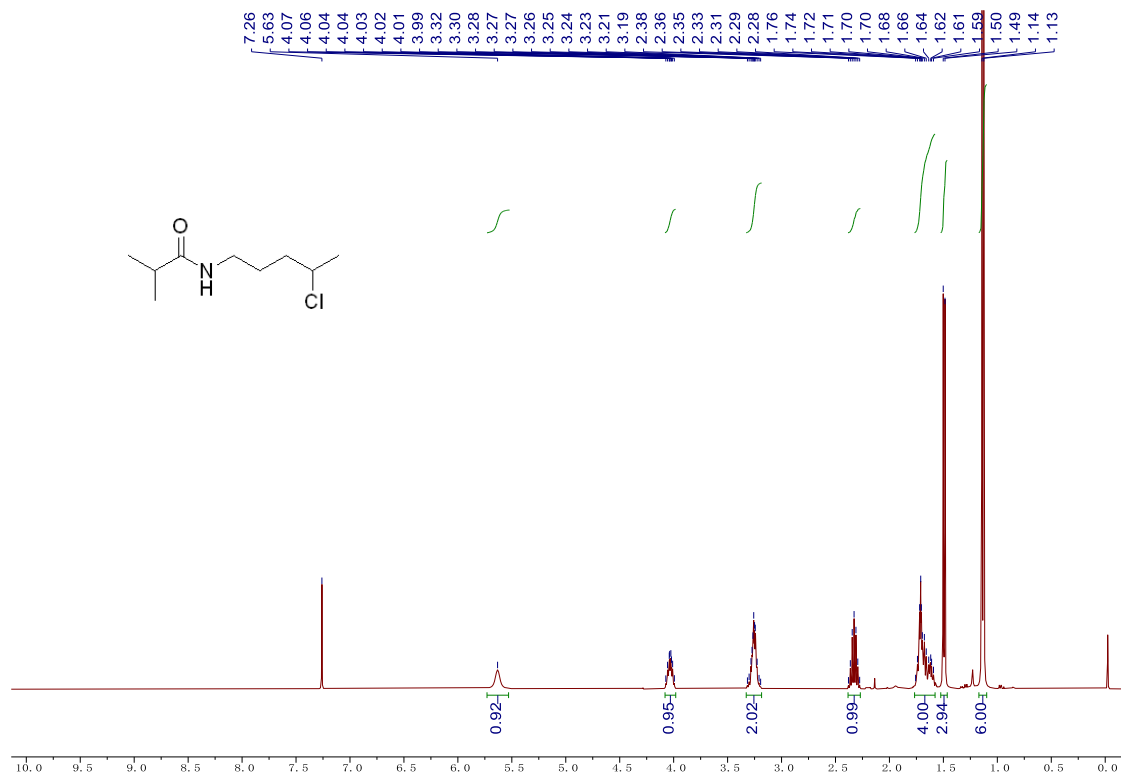


^{13}C NMR (CDCl_3 , 100 MHz)

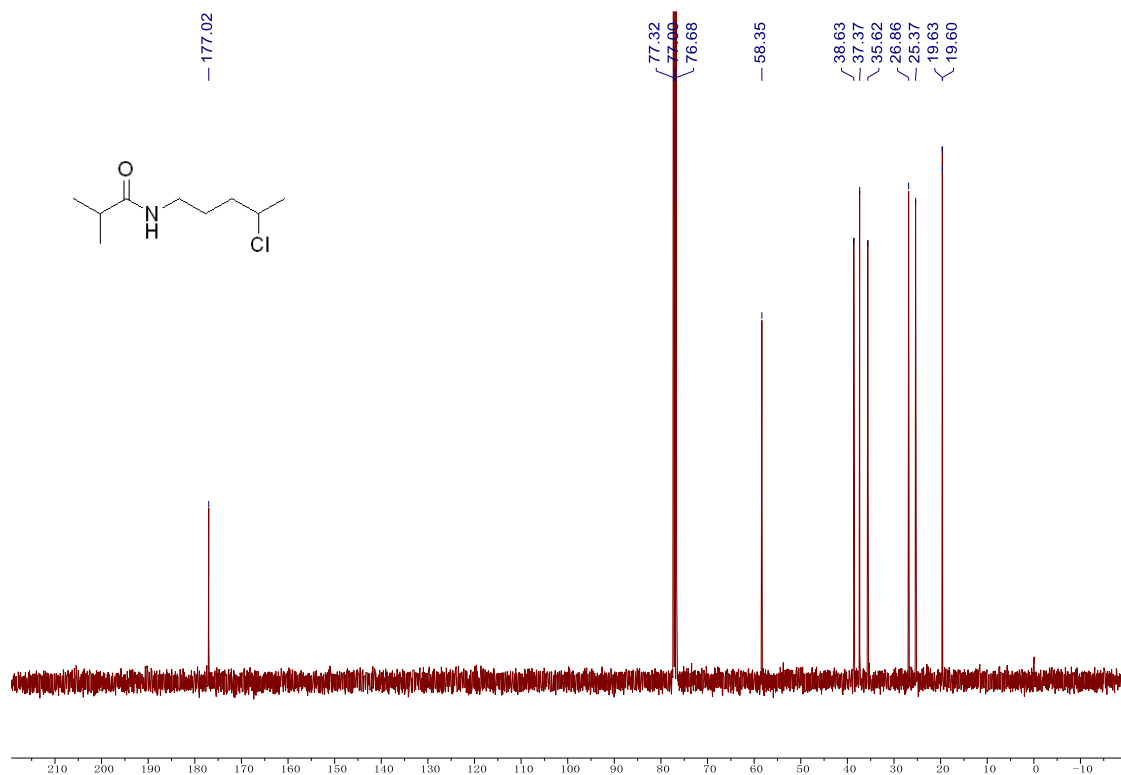


2ad

^1H NMR (CDCl_3 , 400 MHz)

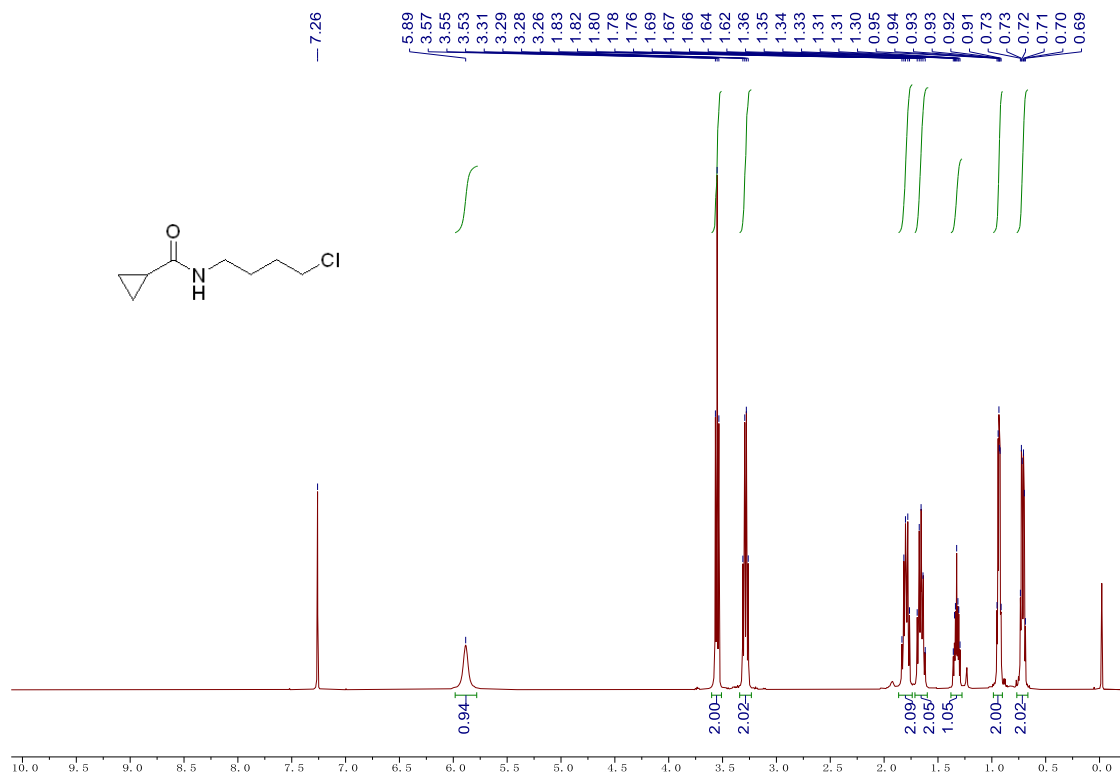


^{13}C NMR (CDCl_3 , 100 MHz)

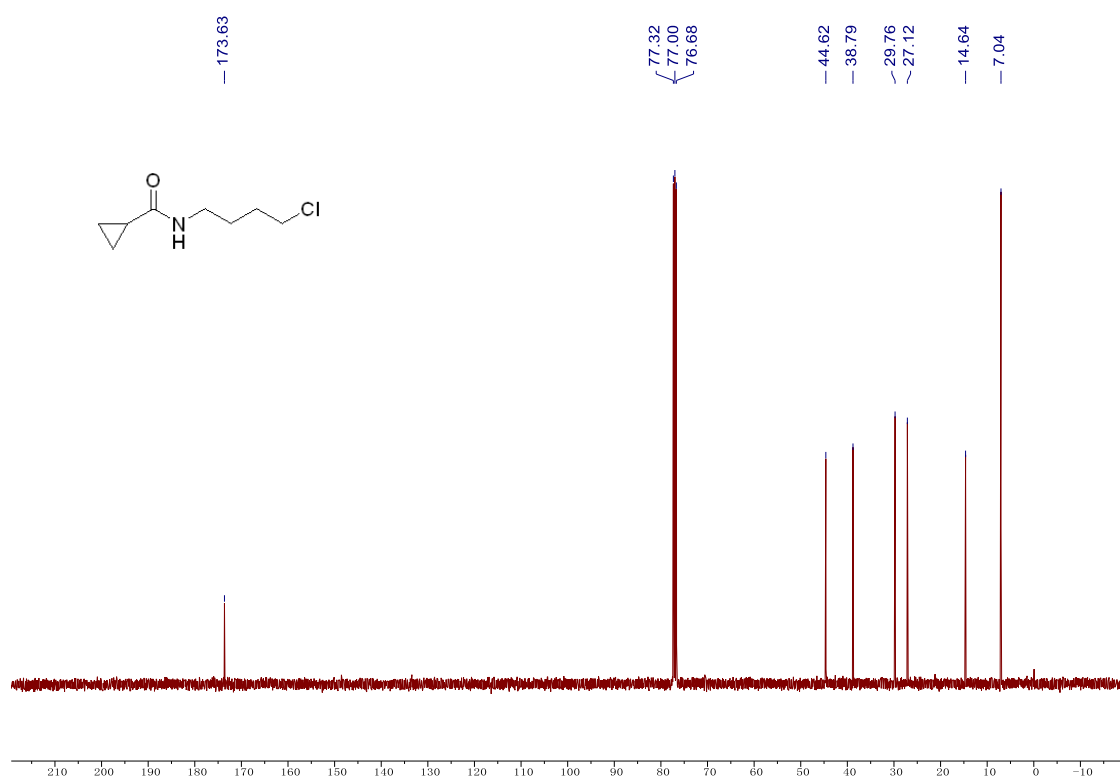


2ae

^1H NMR (CDCl_3 , 400 MHz)

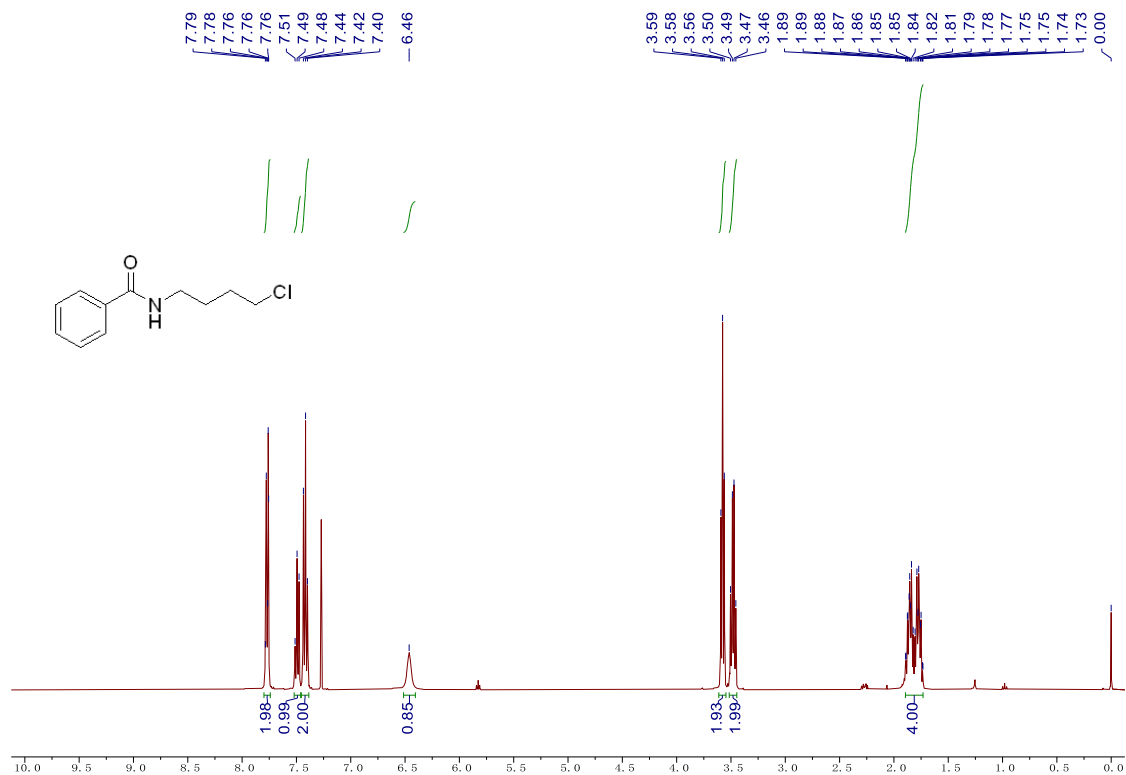


^{13}C NMR (CDCl_3 , 100 MHz)

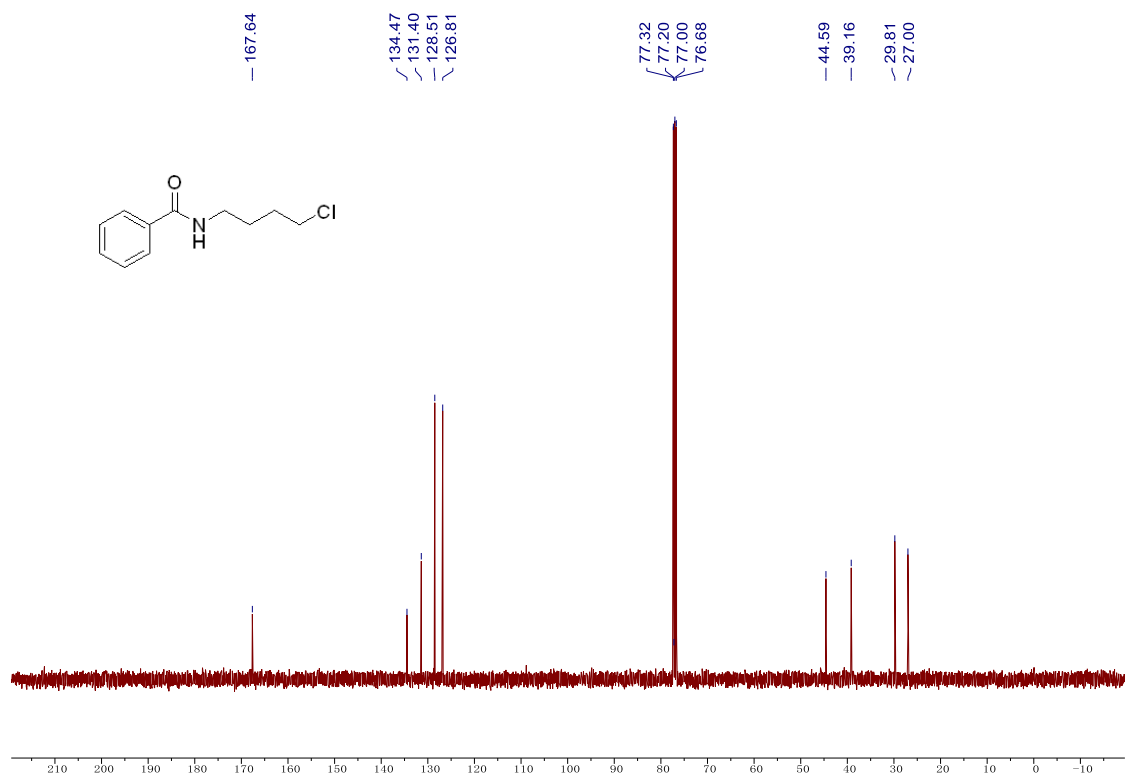


2af

^1H NMR (CDCl_3 , 400 MHz)

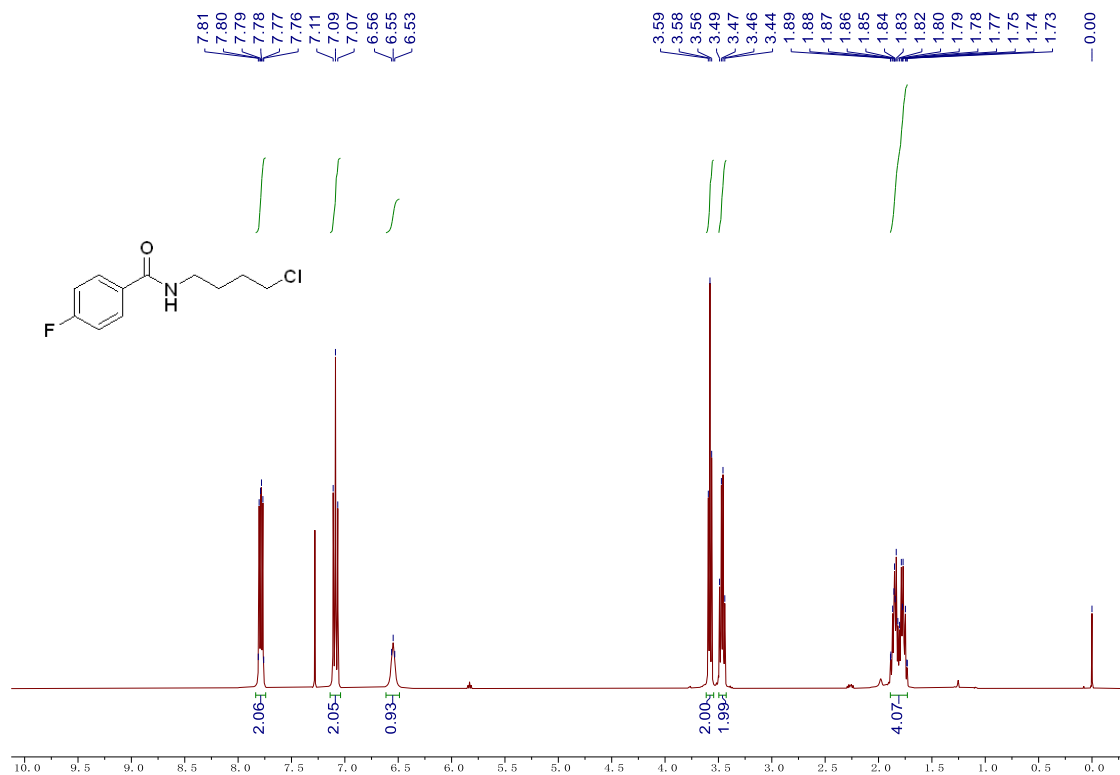


^{13}C NMR (CDCl_3 , 100 MHz)

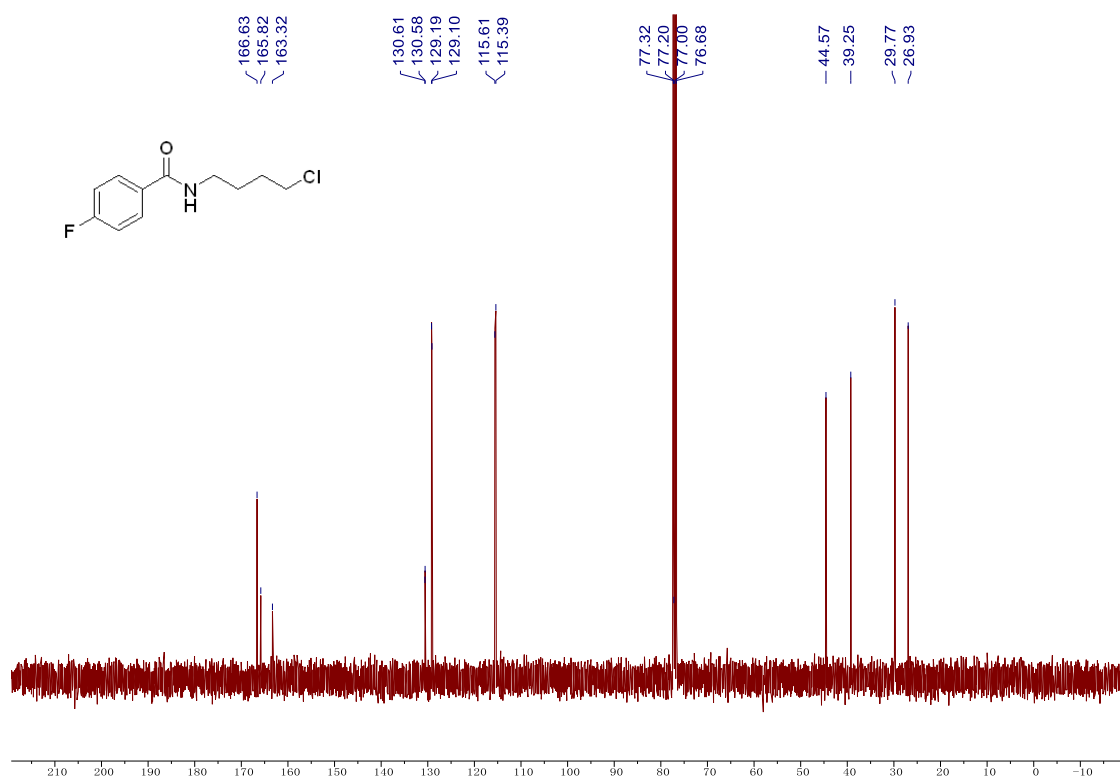


2ag

^1H NMR (CDCl_3 , 400 MHz)

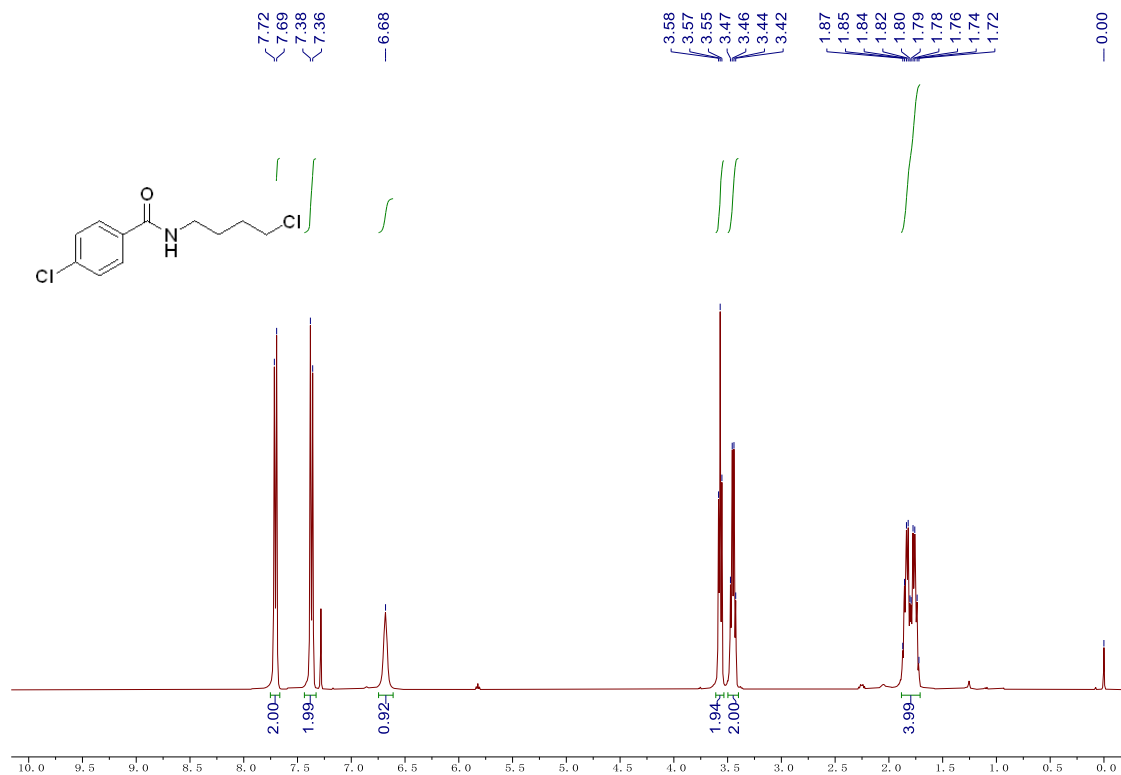


^{13}C NMR (CDCl_3 , 100 MHz)

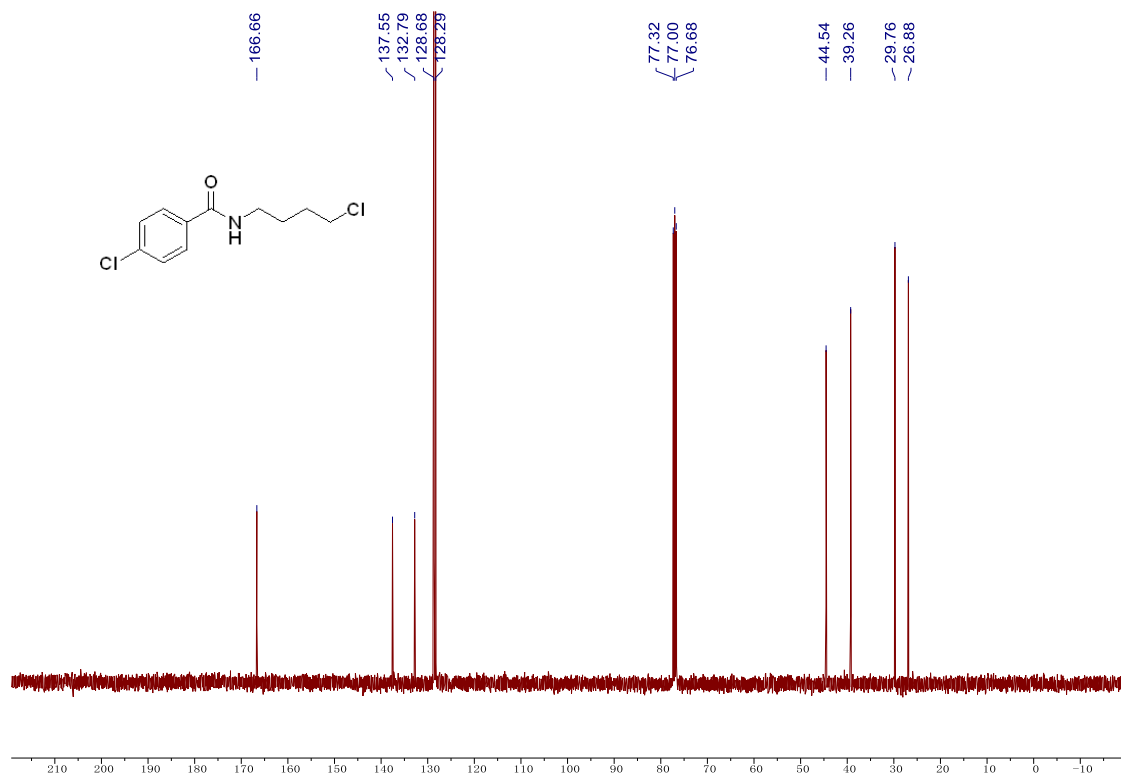


2ah

^1H NMR (CDCl_3 , 400 MHz)

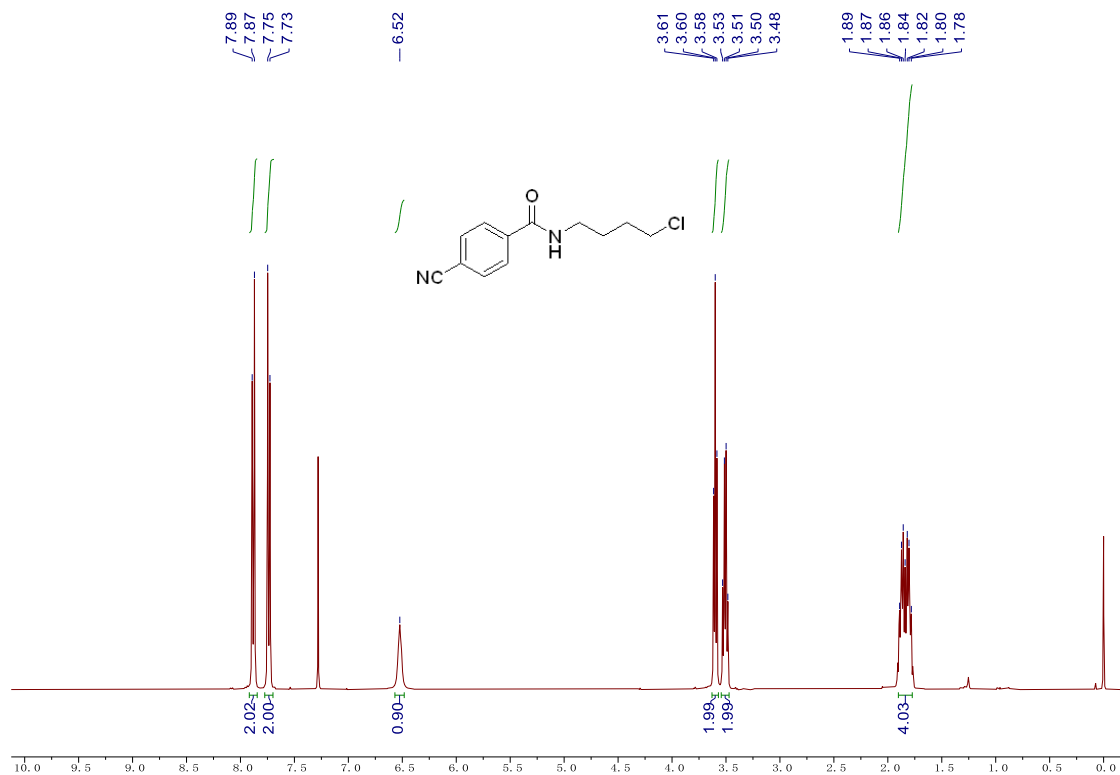


^{13}C NMR (CDCl_3 , 100 MHz)

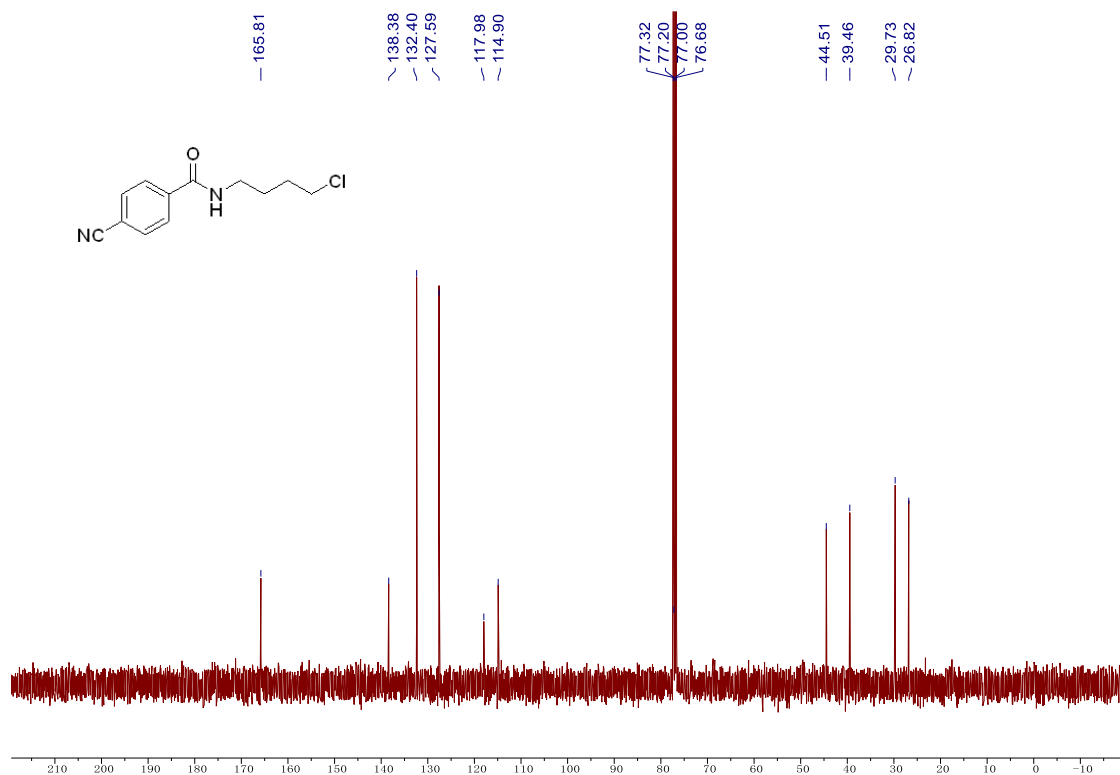


2ai

^1H NMR (CDCl_3 , 400 MHz)

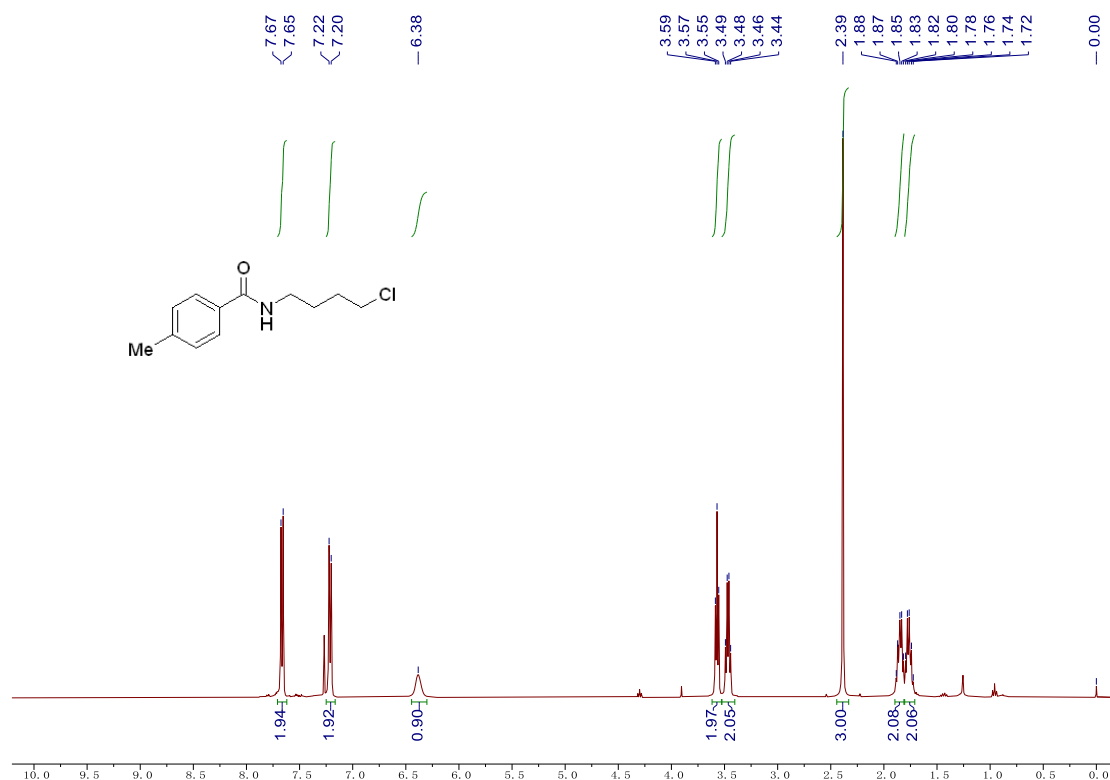


^{13}C NMR (CDCl_3 , 100 MHz)

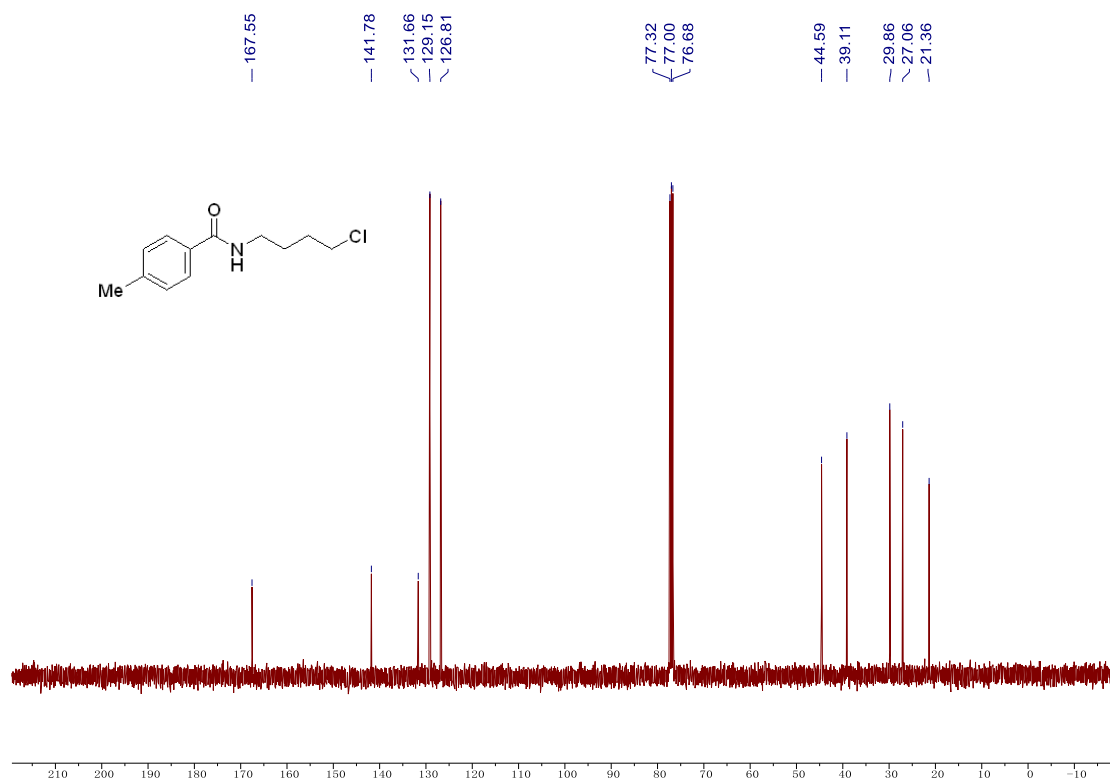


2aj

^1H NMR (CDCl_3 , 400 MHz)

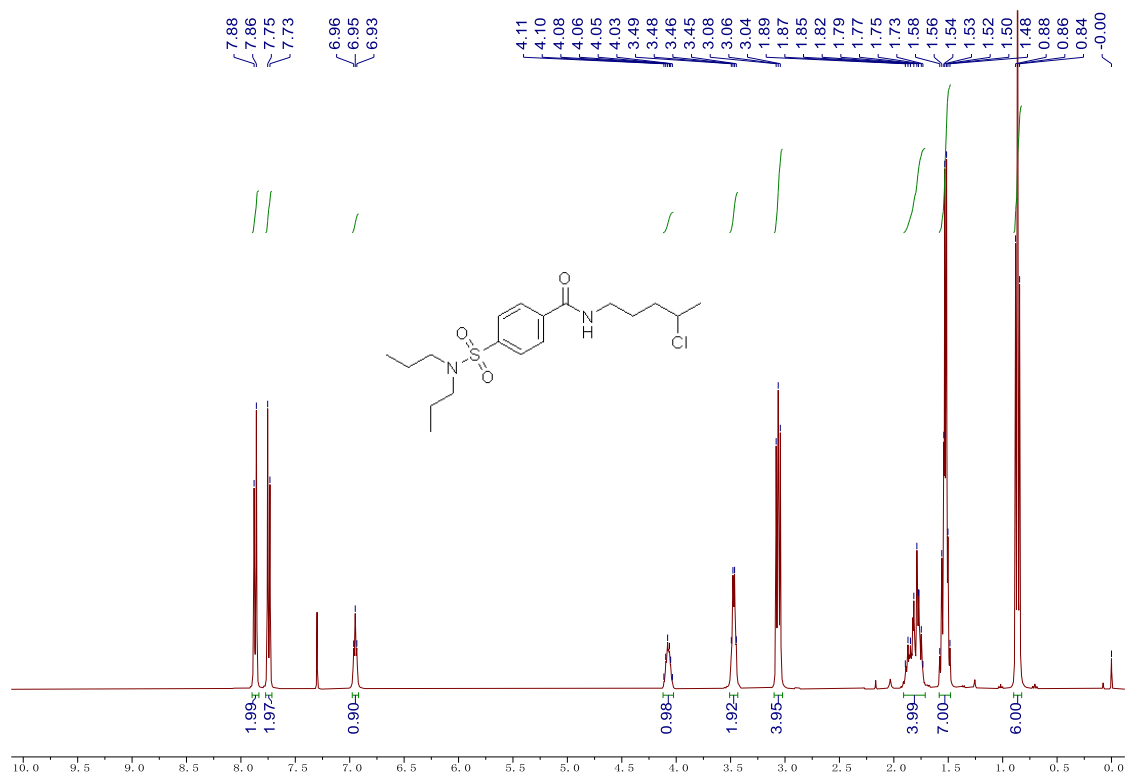


^{13}C NMR (CDCl_3 , 100 MHz)

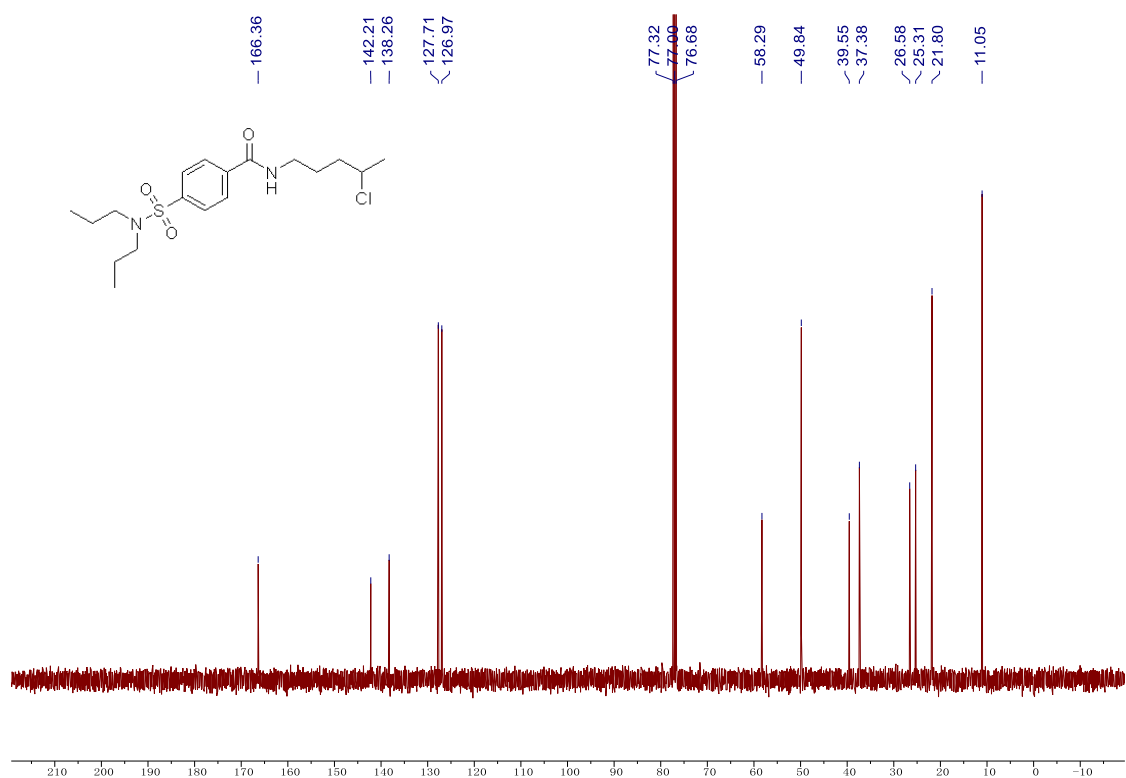


2ak

^1H NMR (CDCl_3 , 400 MHz)

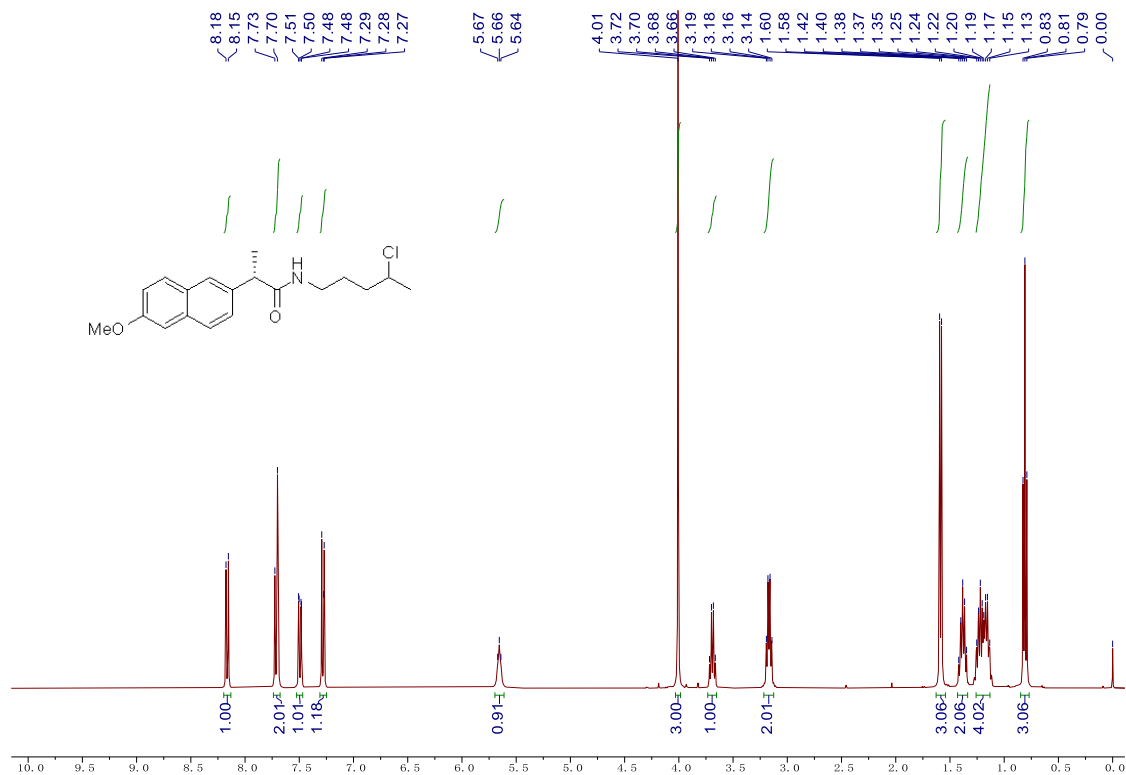


^{13}C NMR (CDCl_3 , 100 MHz)

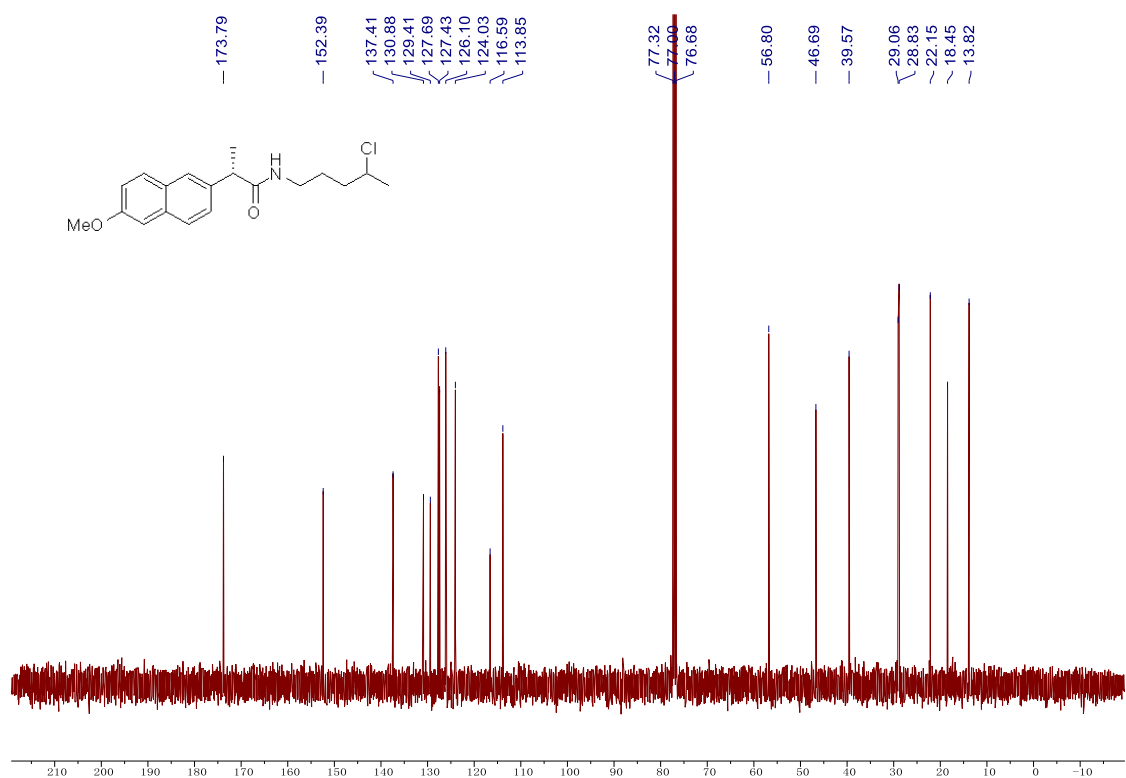


2al

^1H NMR (CDCl_3 , 400 MHz)

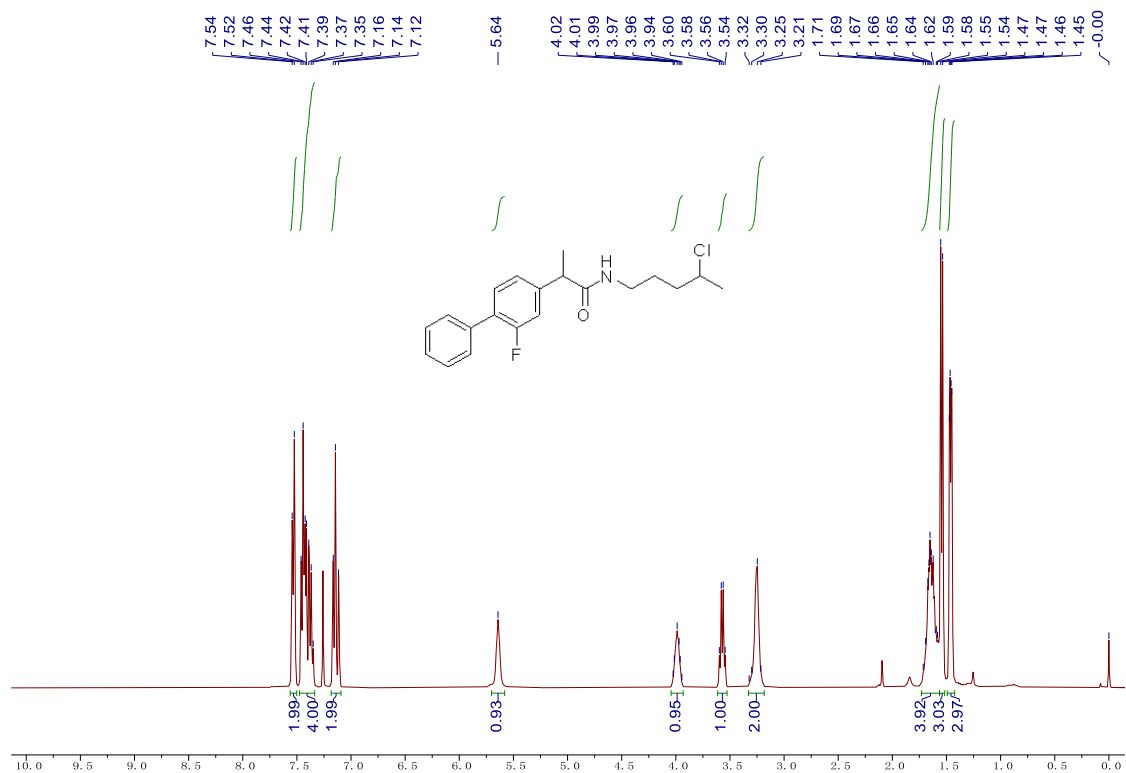


^{13}C NMR (CDCl_3 , 100 MHz)

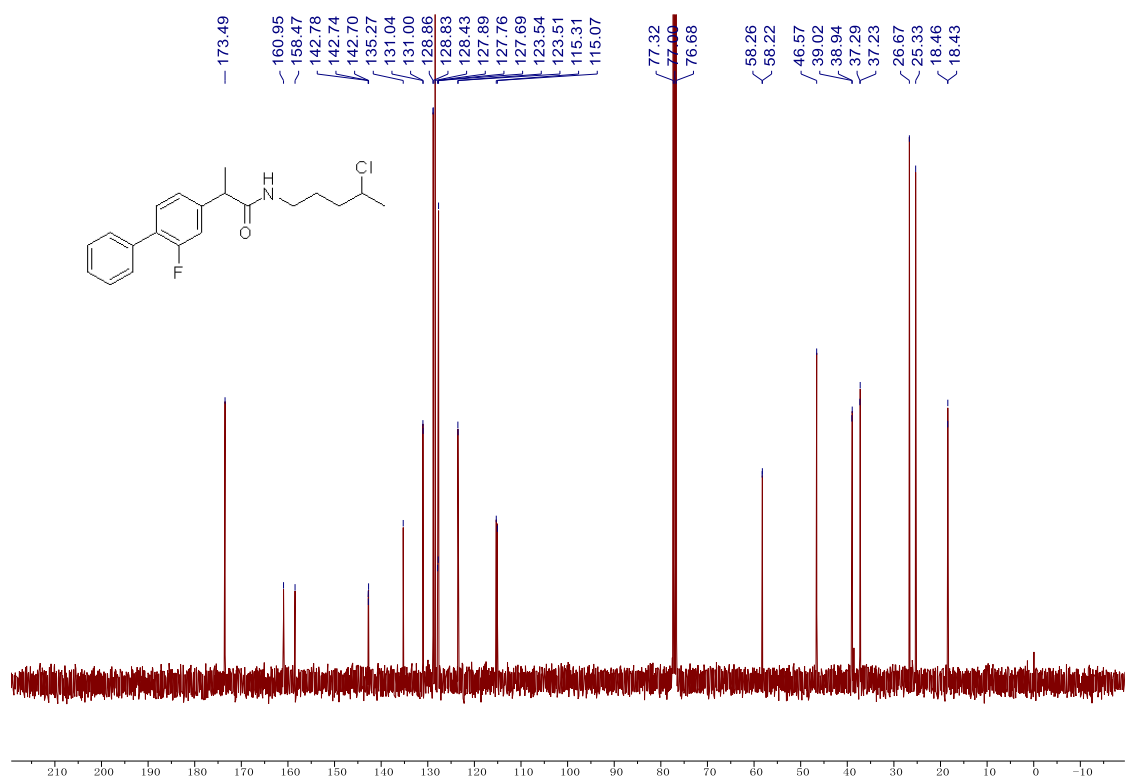


2am

^1H NMR (CDCl_3 , 400 MHz)

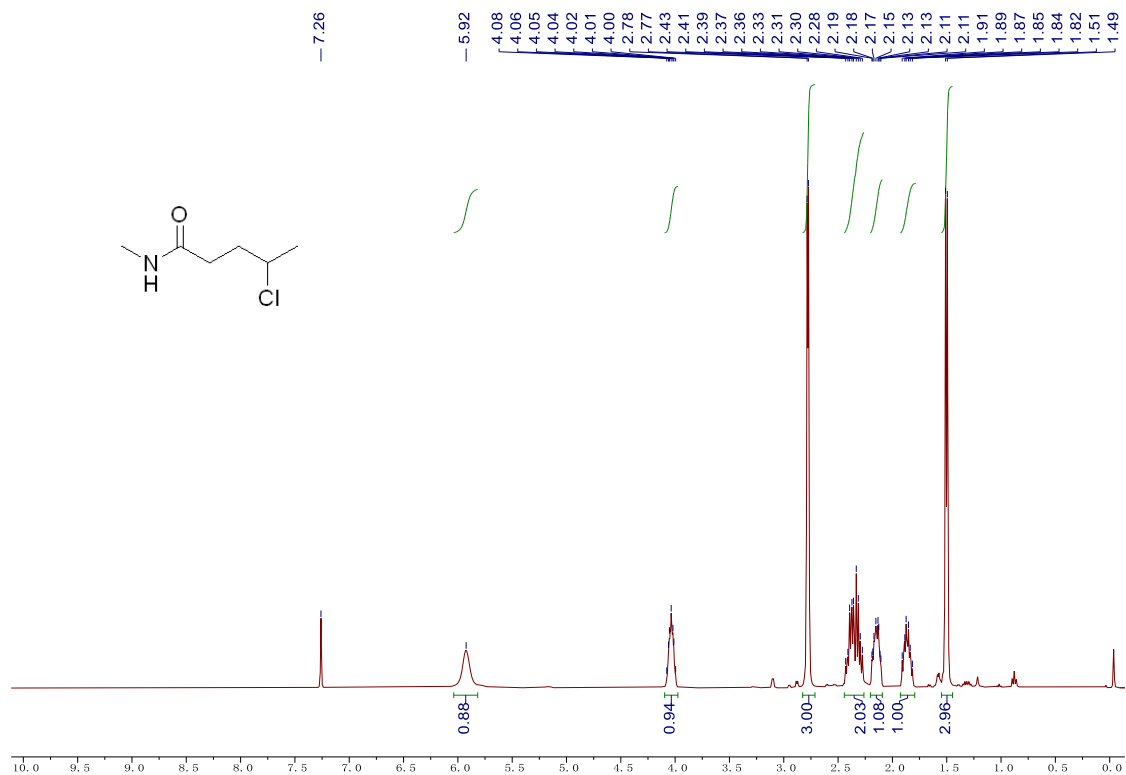


^{13}C NMR (CDCl_3 , 100 MHz)

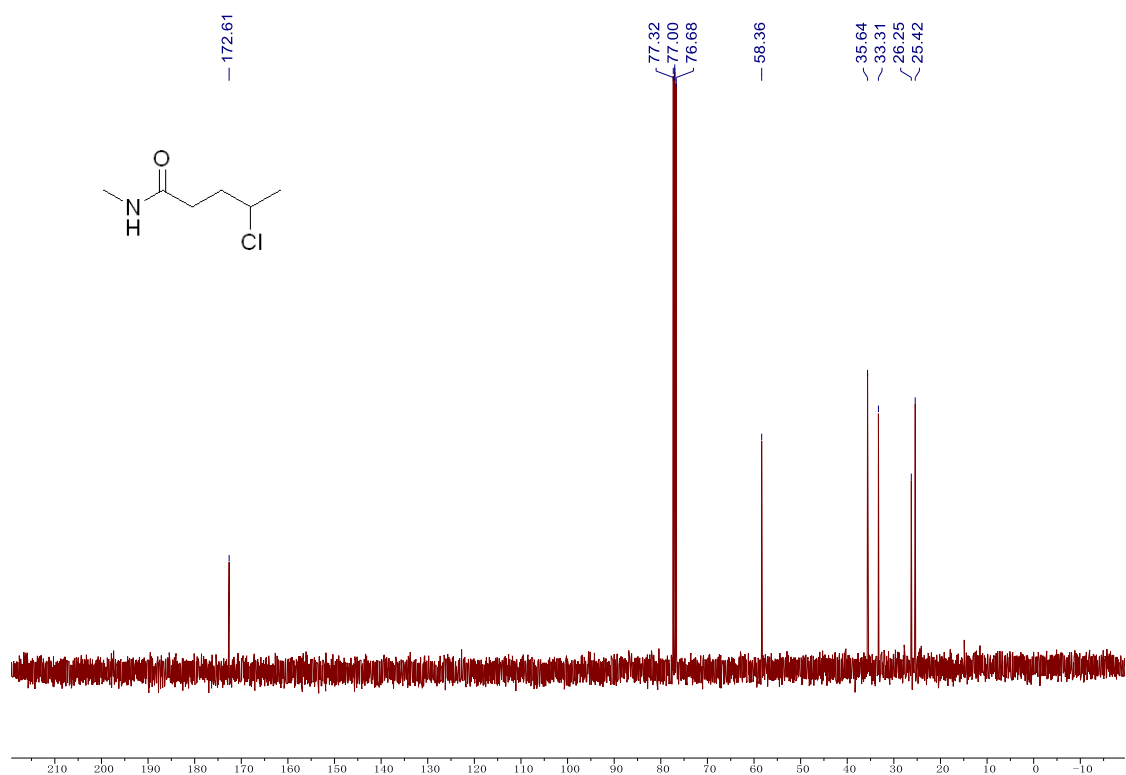


2an

^1H NMR (CDCl_3 , 400 MHz)

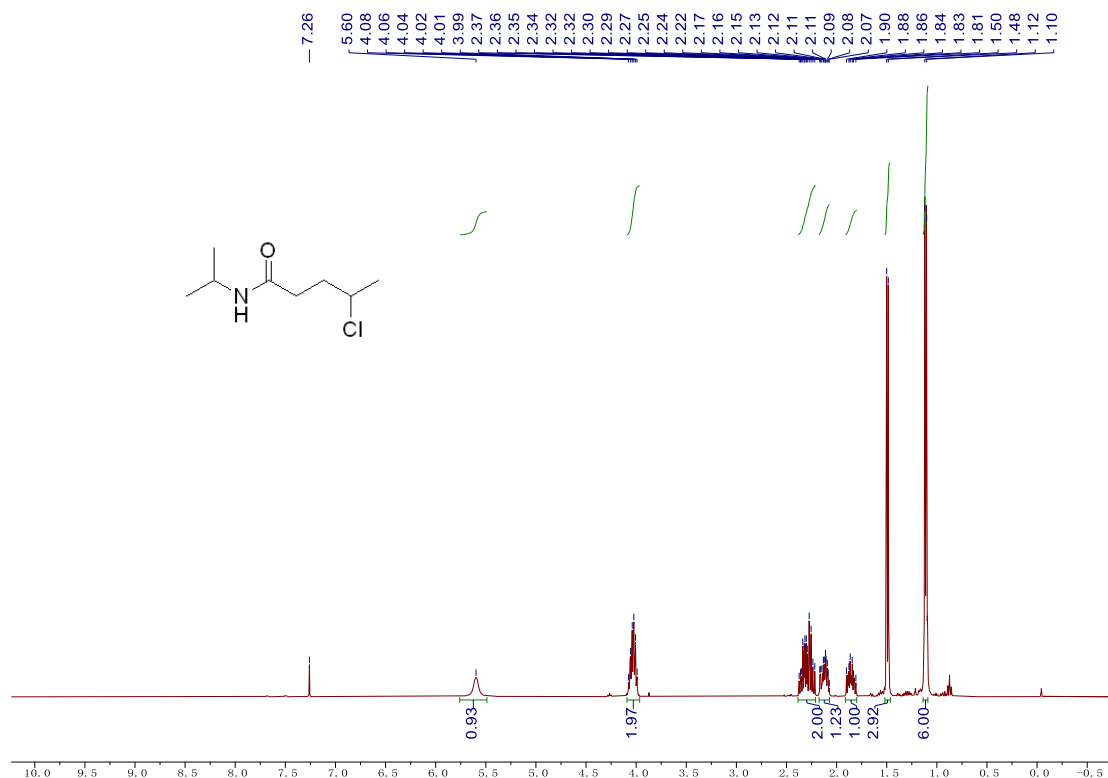


^{13}C NMR (CDCl_3 , 100 MHz)

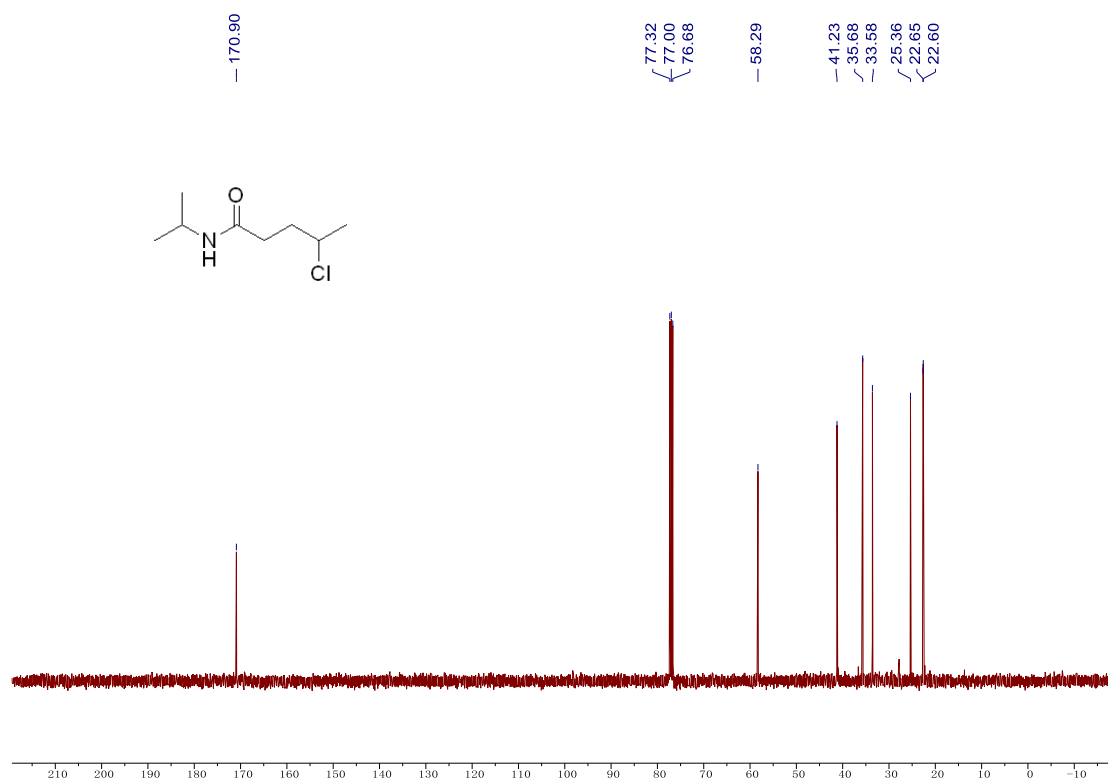


2ao

^1H NMR (CDCl_3 , 400 MHz)

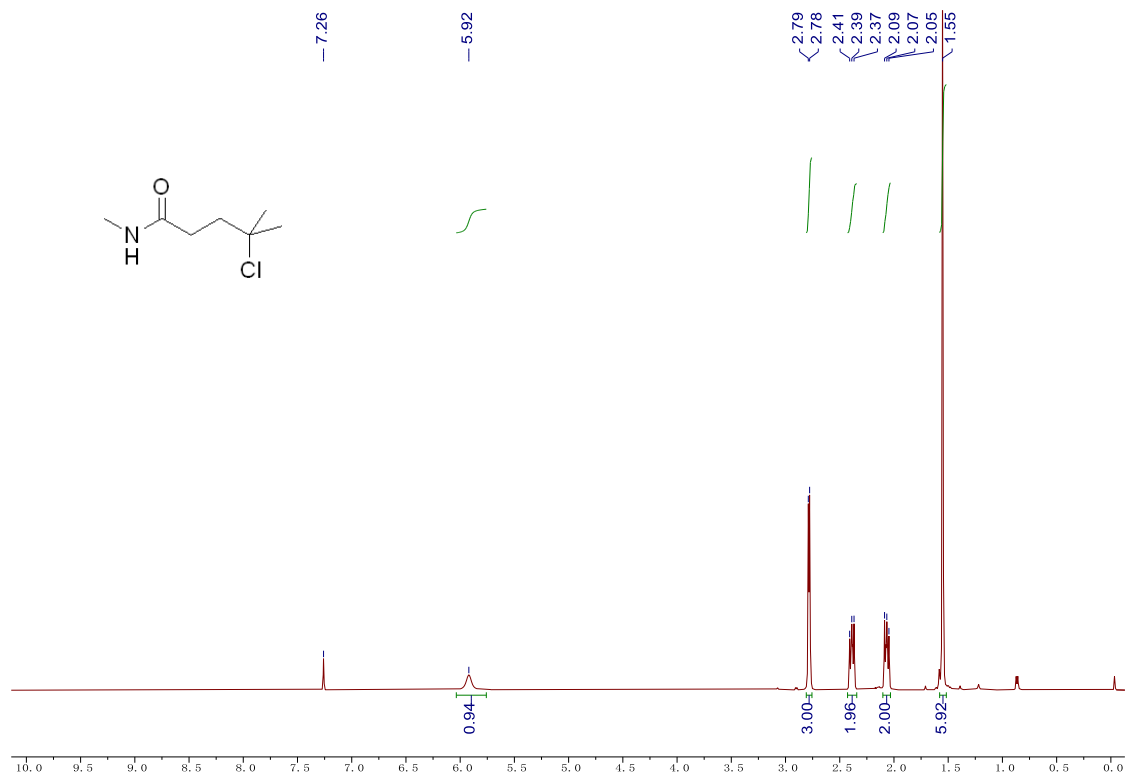


^{13}C NMR (CDCl_3 , 100 MHz)

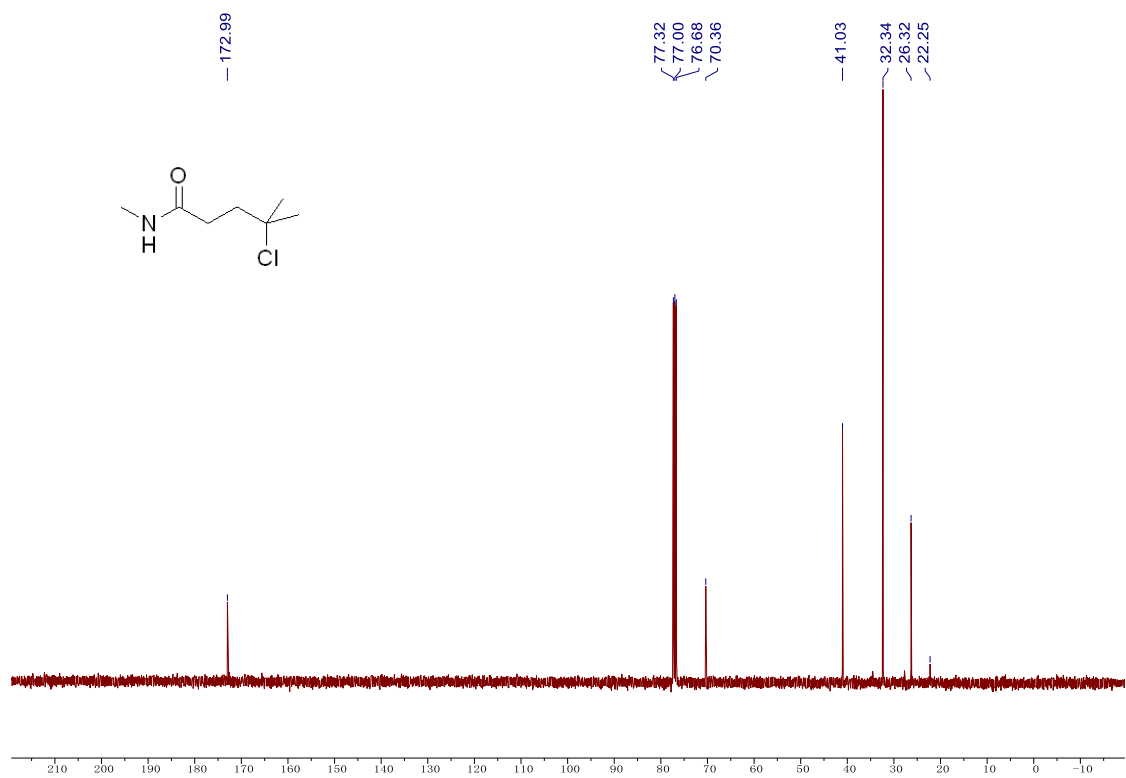


2ap

^1H NMR (CDCl_3 , 400 MHz)

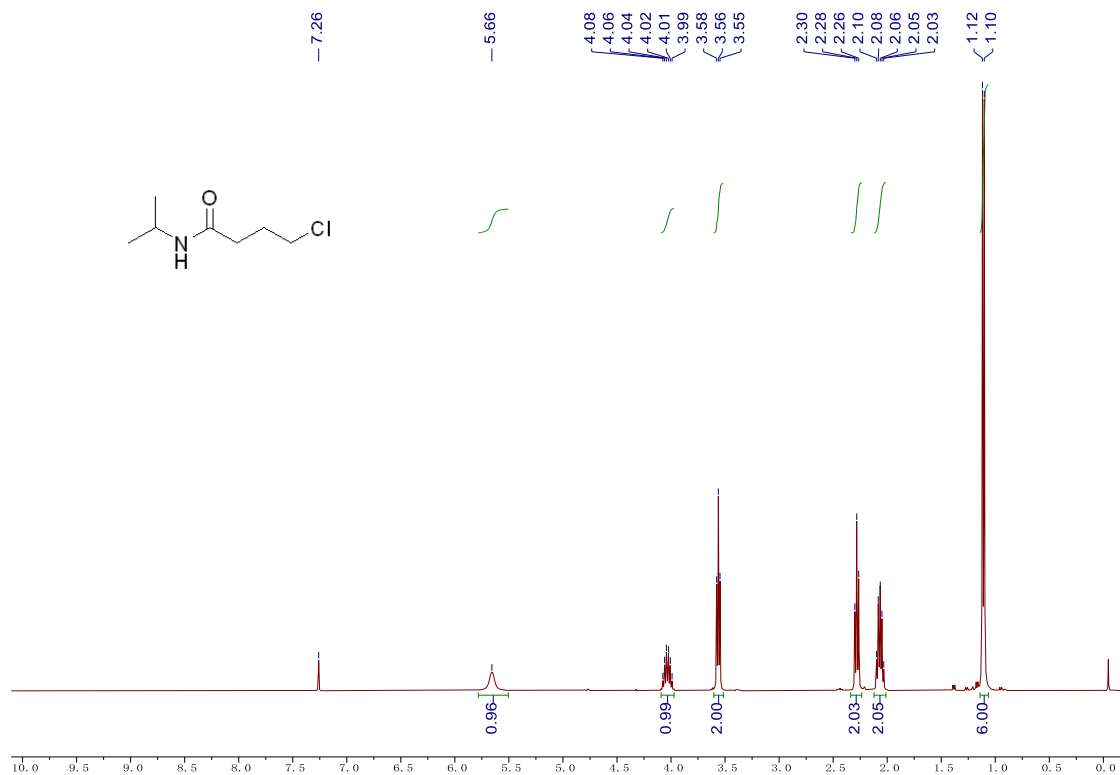


^{13}C NMR (CDCl_3 , 100 MHz)

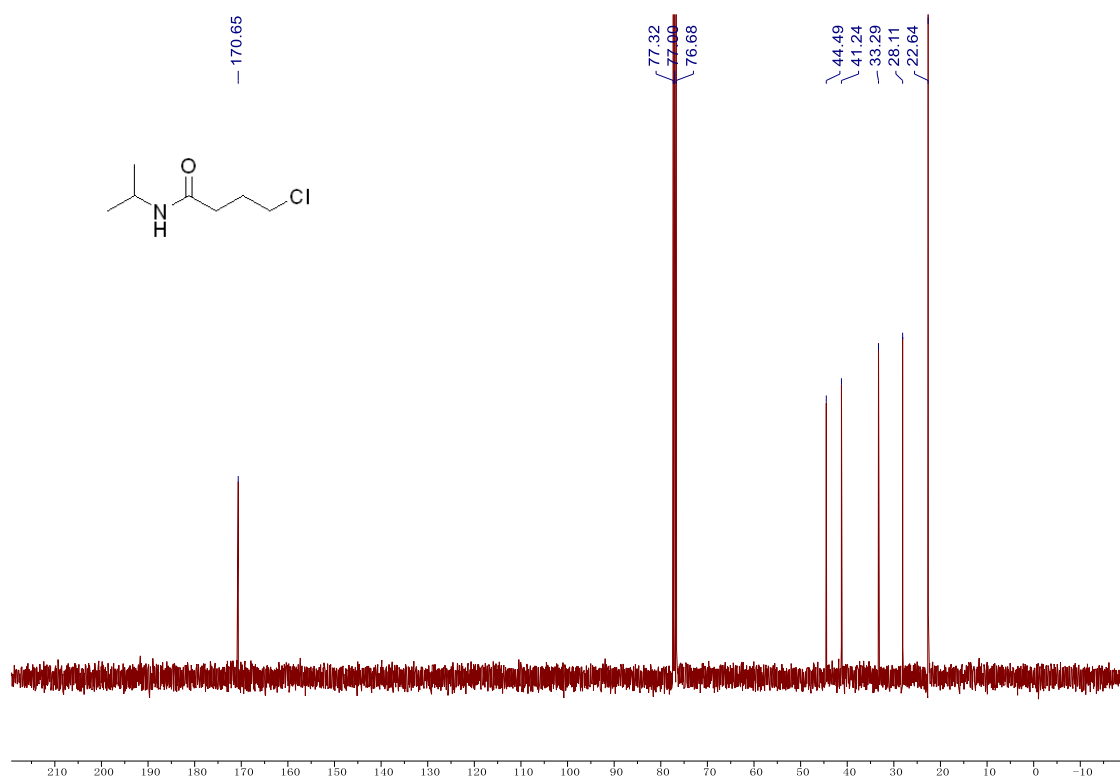


2aq

^1H NMR (CDCl_3 , 400 MHz)

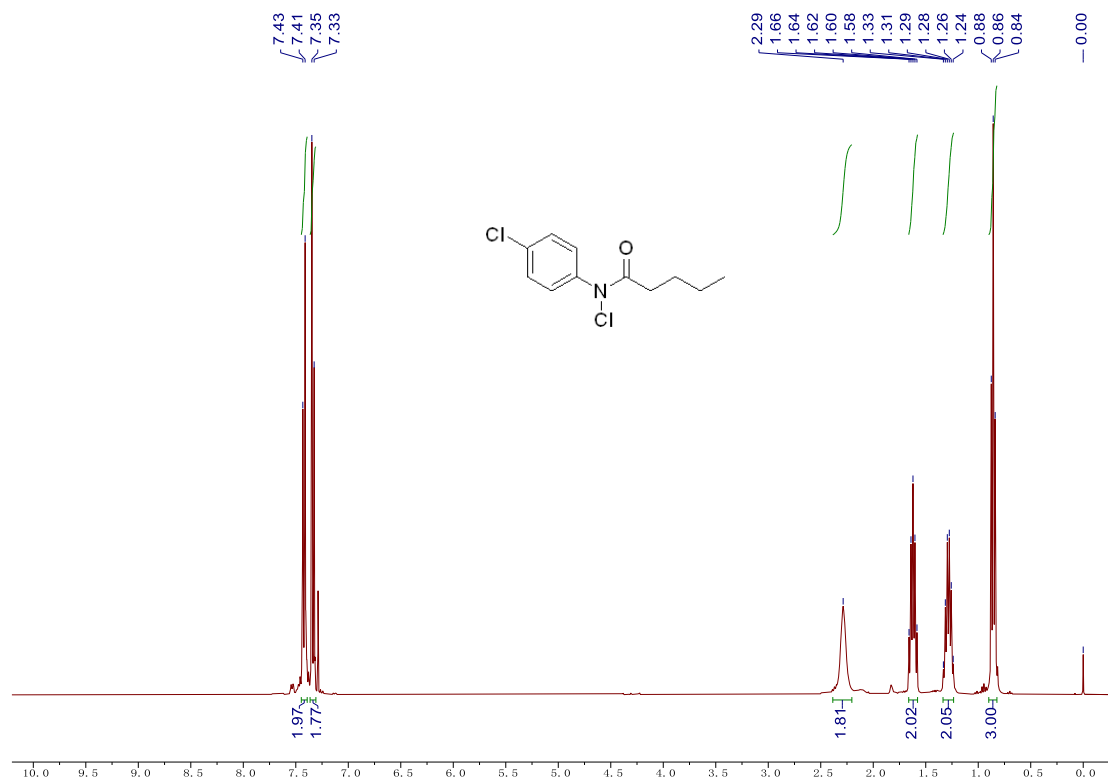


^{13}C NMR (CDCl_3 , 100 MHz)

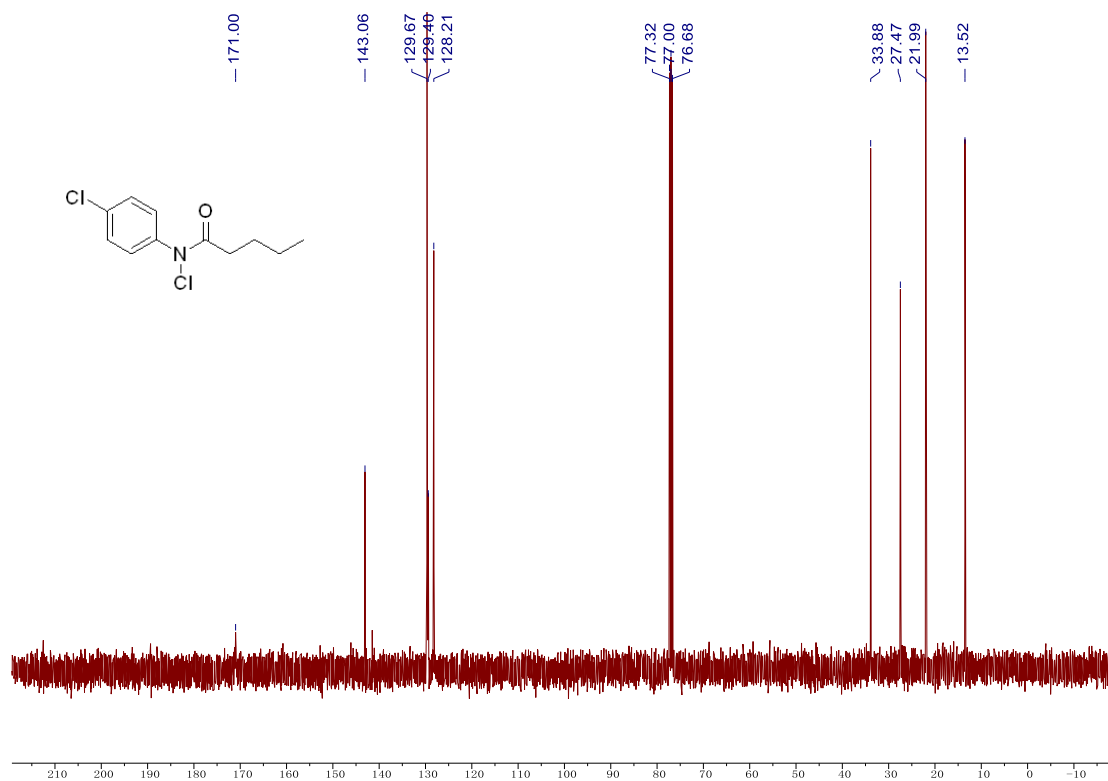


3ar-1

^1H NMR (CDCl_3 , 400 MHz)

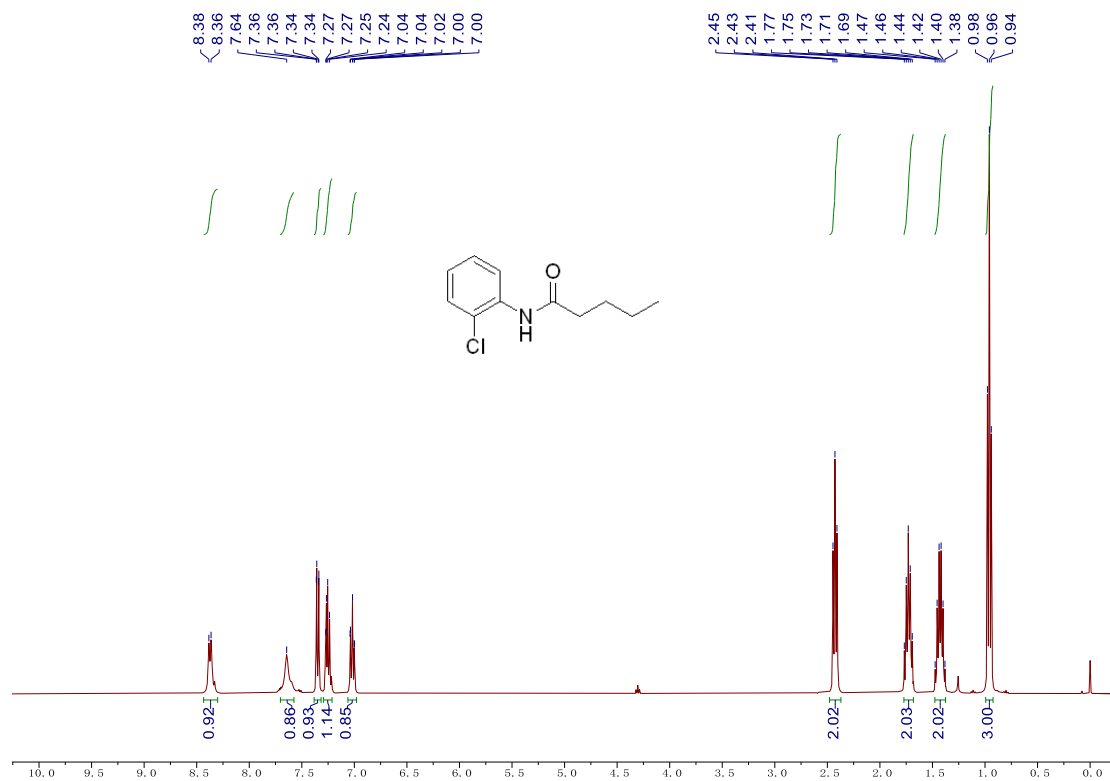


^{13}C NMR (CDCl_3 , 100 MHz)

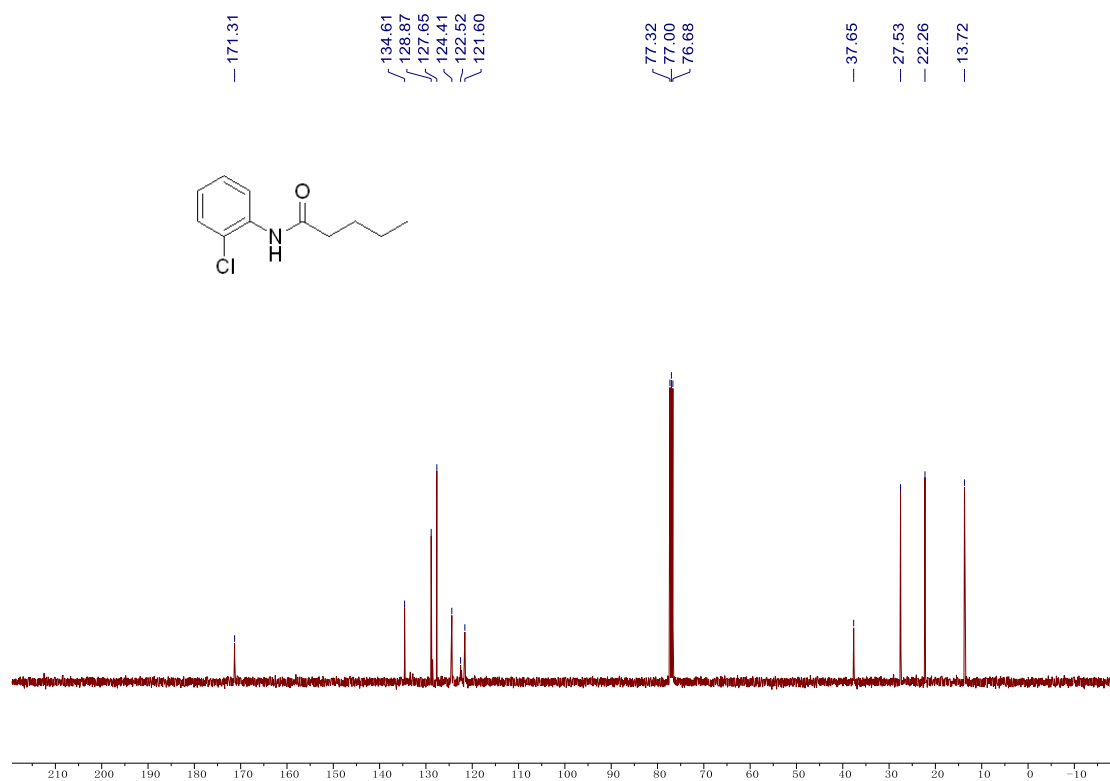


3ar-2

^1H NMR (CDCl_3 , 400 MHz)

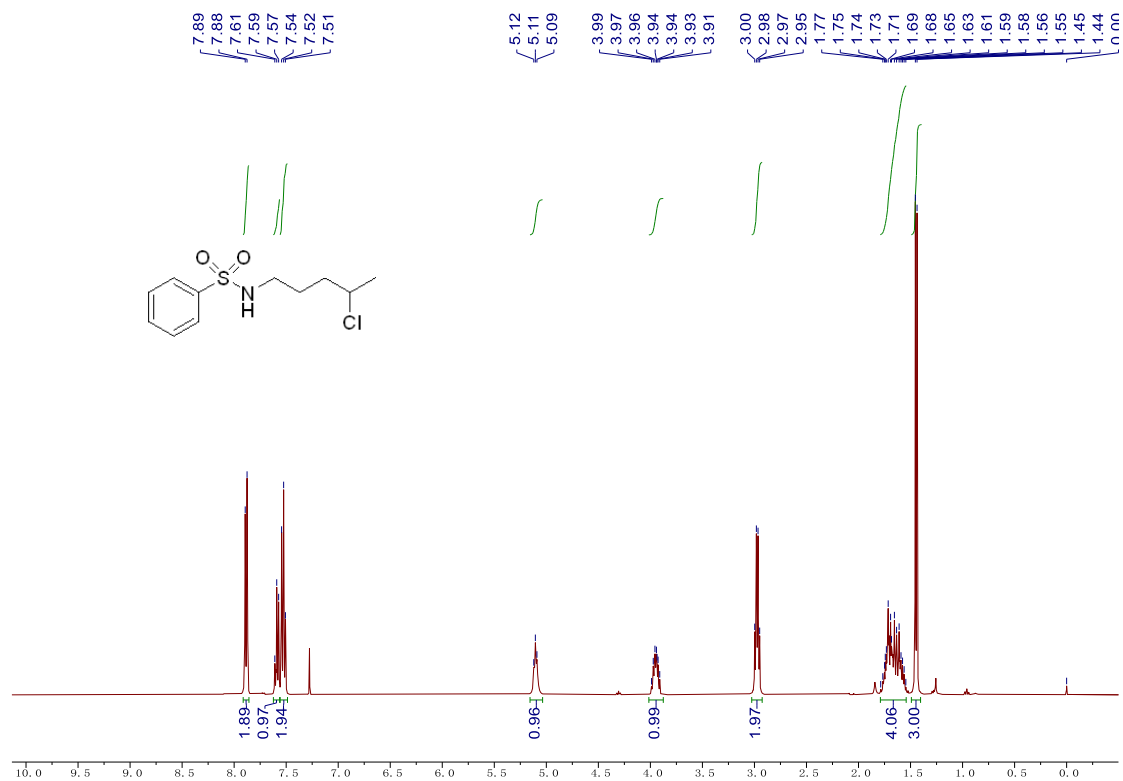


^{13}C NMR (CDCl_3 , 100 MHz)

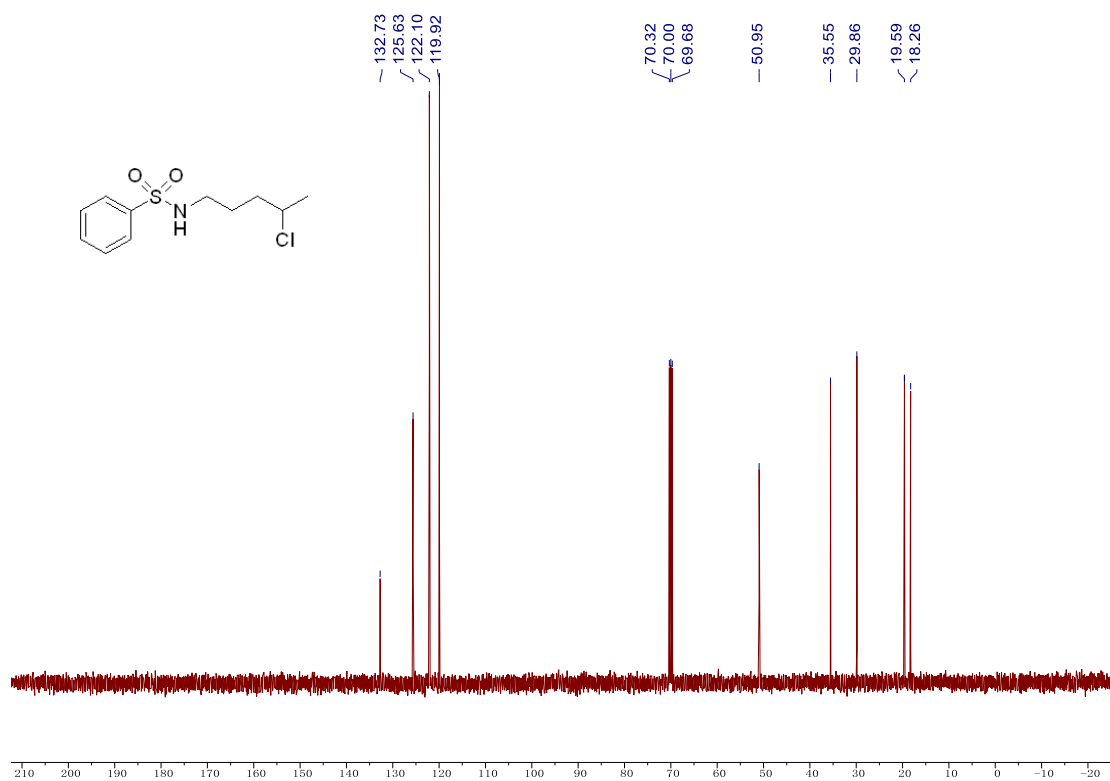


5a

^1H NMR (CDCl_3 , 400 MHz)

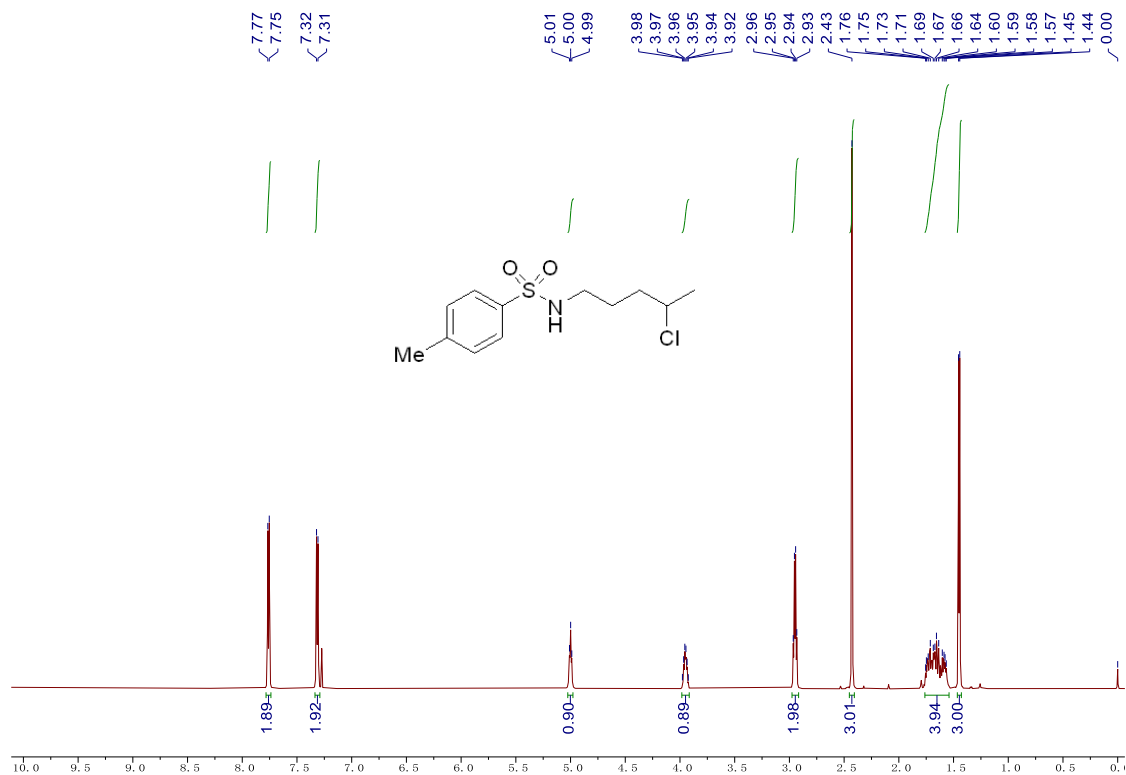


^{13}C NMR (CDCl_3 , 100 MHz)

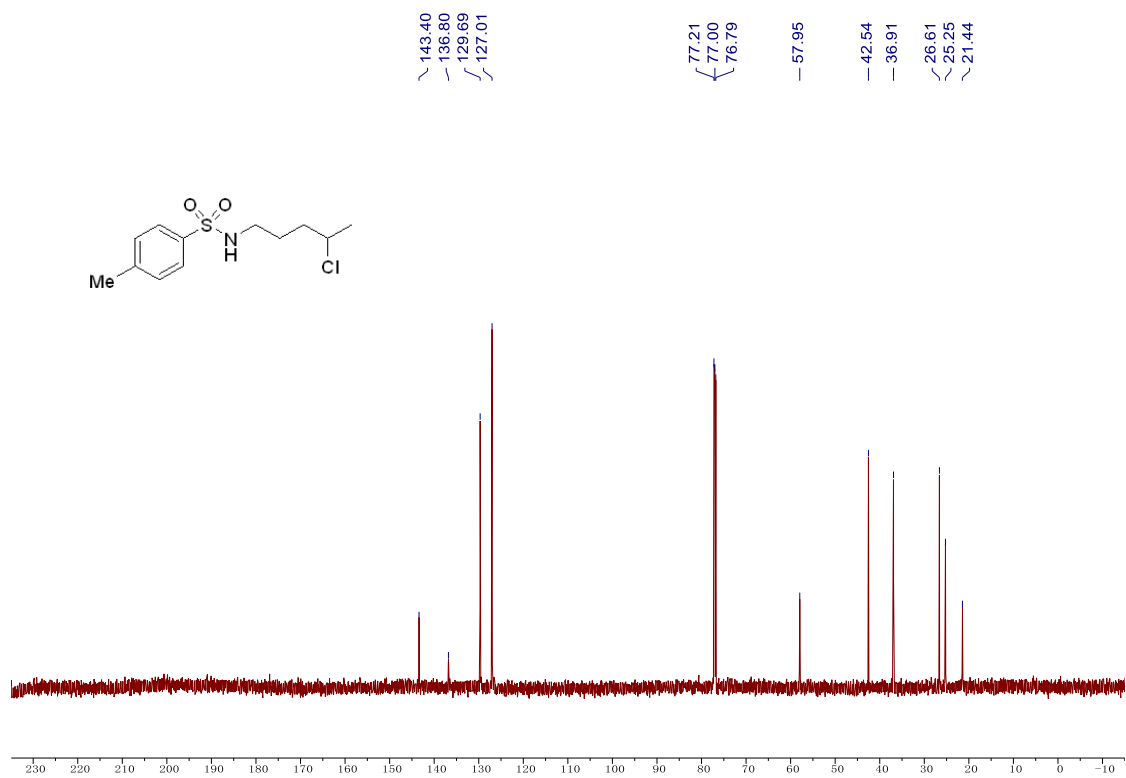


5b

^1H NMR (CDCl_3 , 600 MHz)

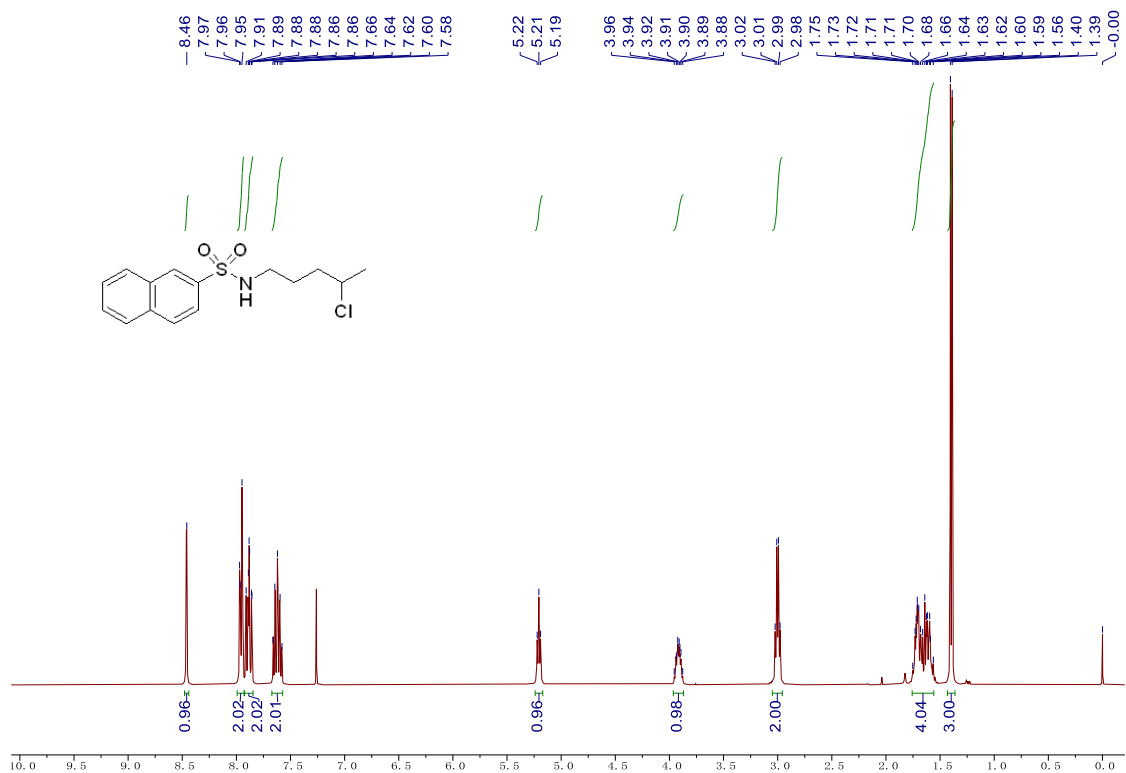


^{13}C NMR (CDCl_3 , 150 MHz)

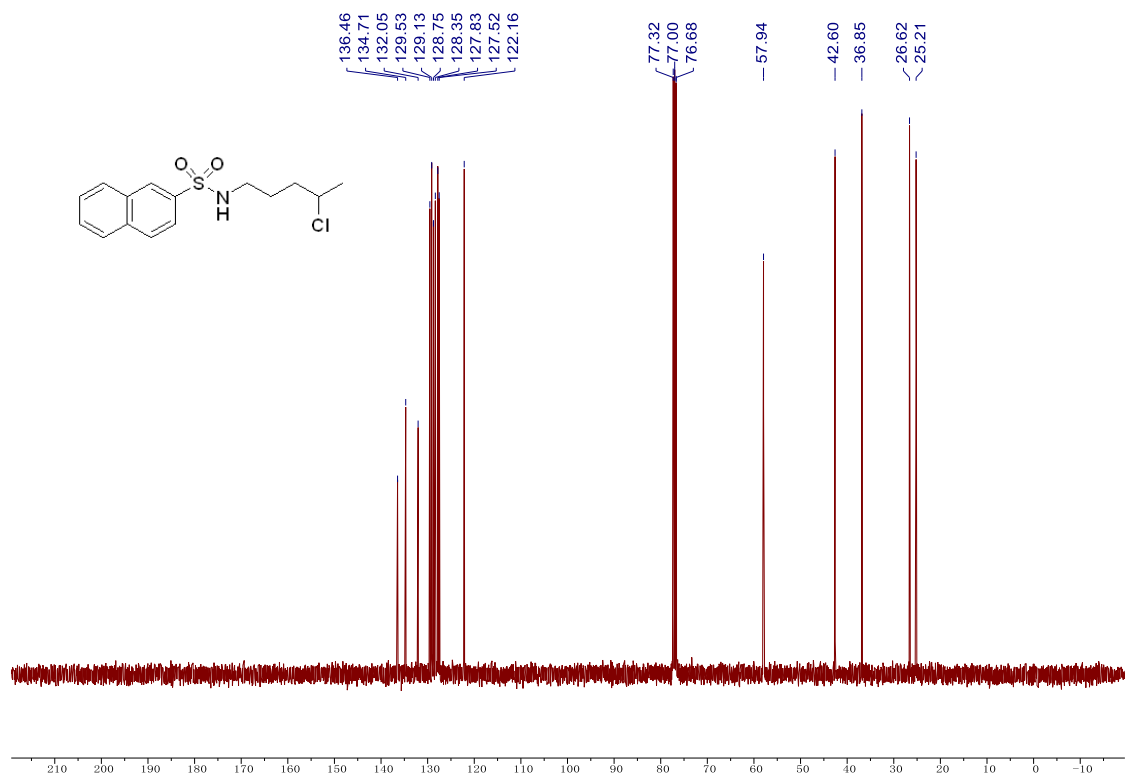


5c

^1H NMR (CDCl_3 , 400 MHz)

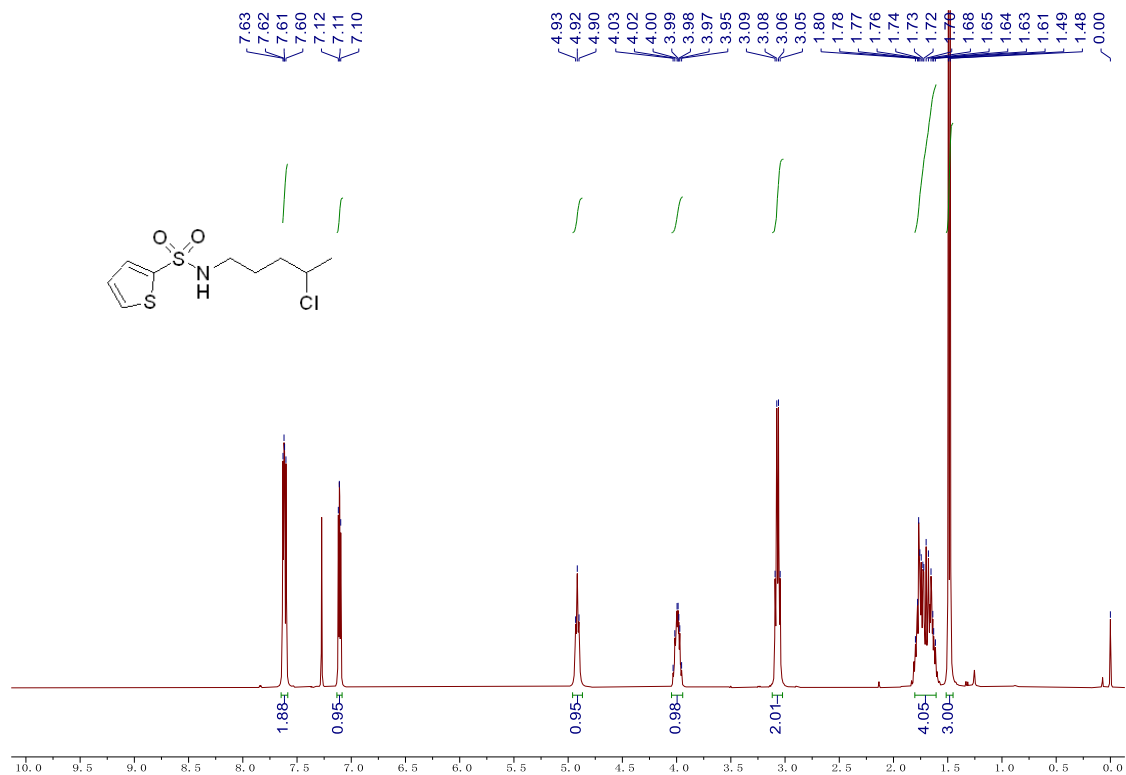


^{13}C NMR (CDCl_3 , 100 MHz)

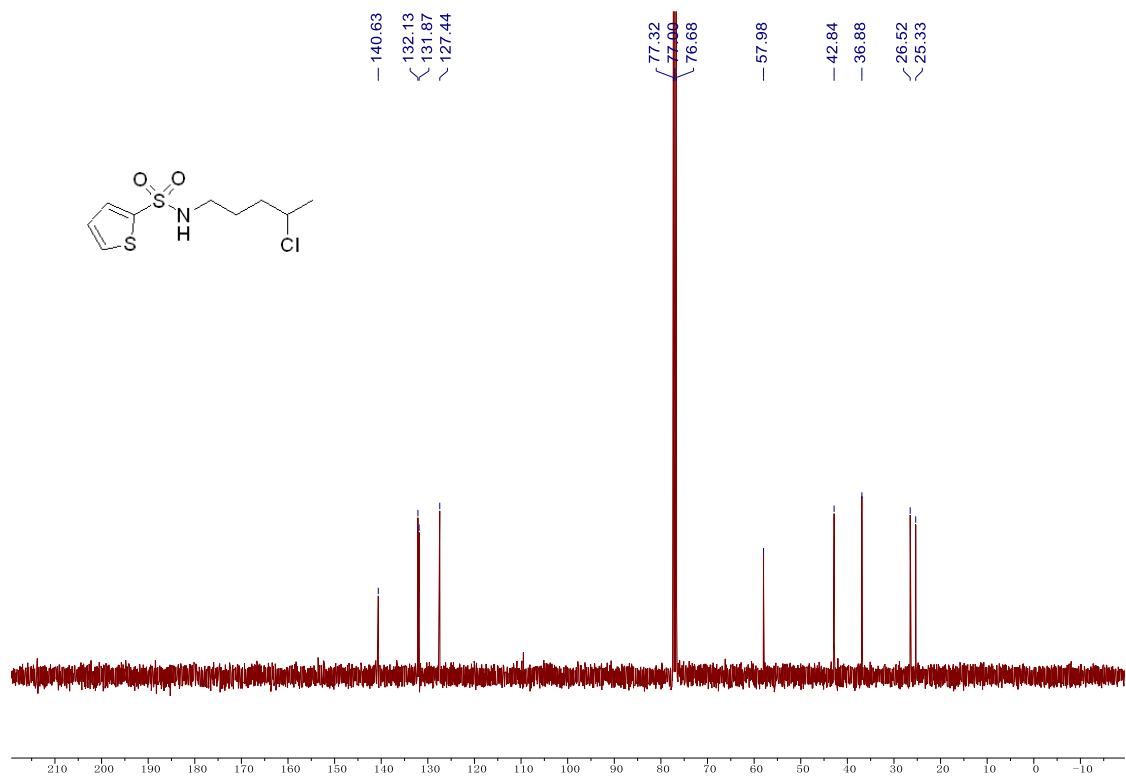


5d

^1H NMR (CDCl_3 , 400 MHz)

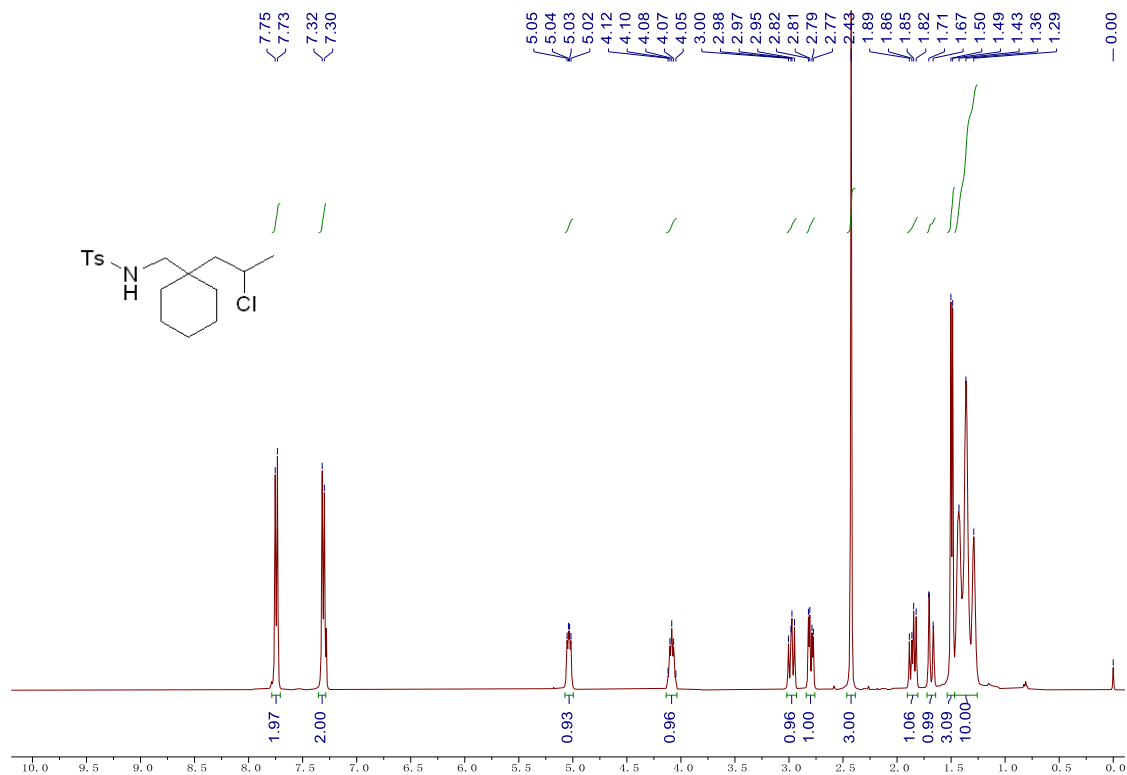


^{13}C NMR (CDCl_3 , 100 MHz)

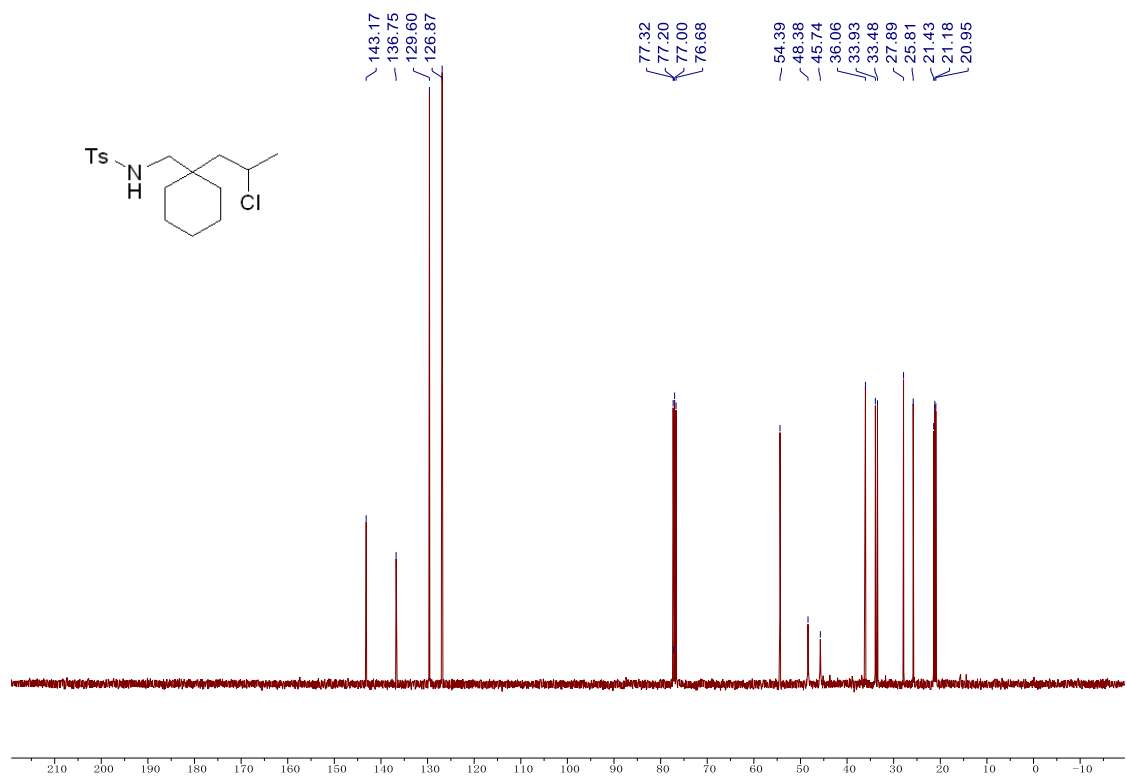


5e

^1H NMR (CDCl_3 , 400 MHz)

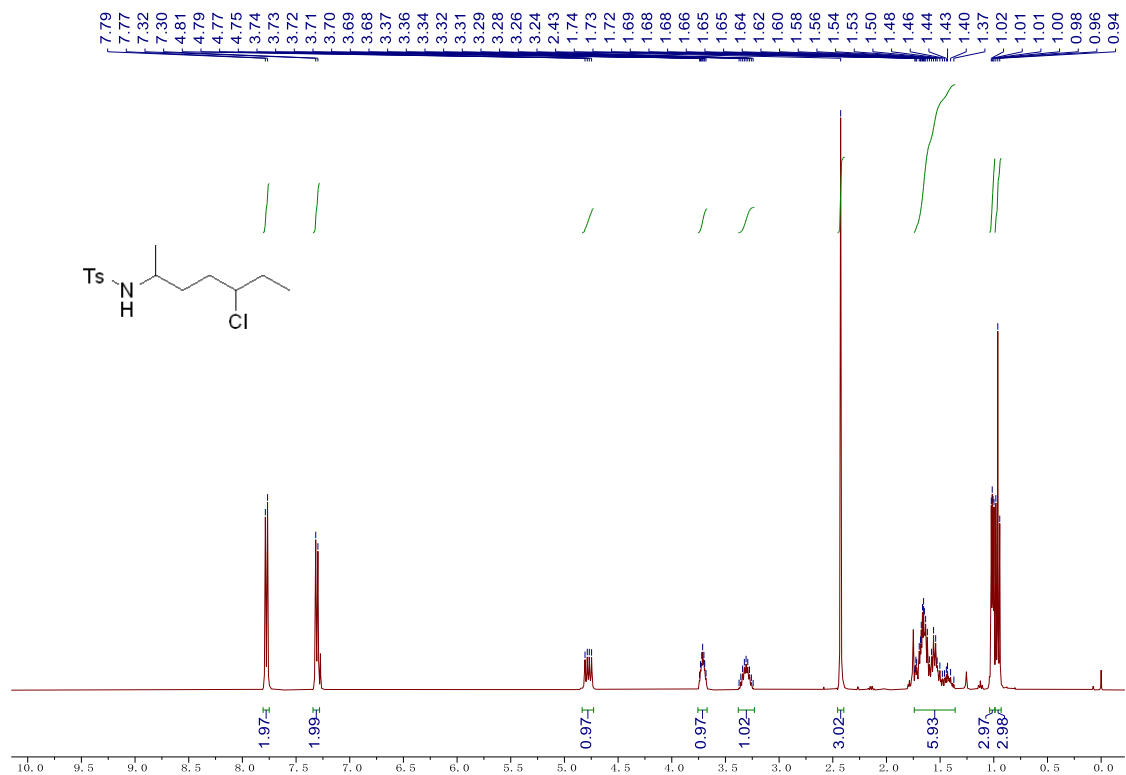


^{13}C NMR (CDCl_3 , 100 MHz)

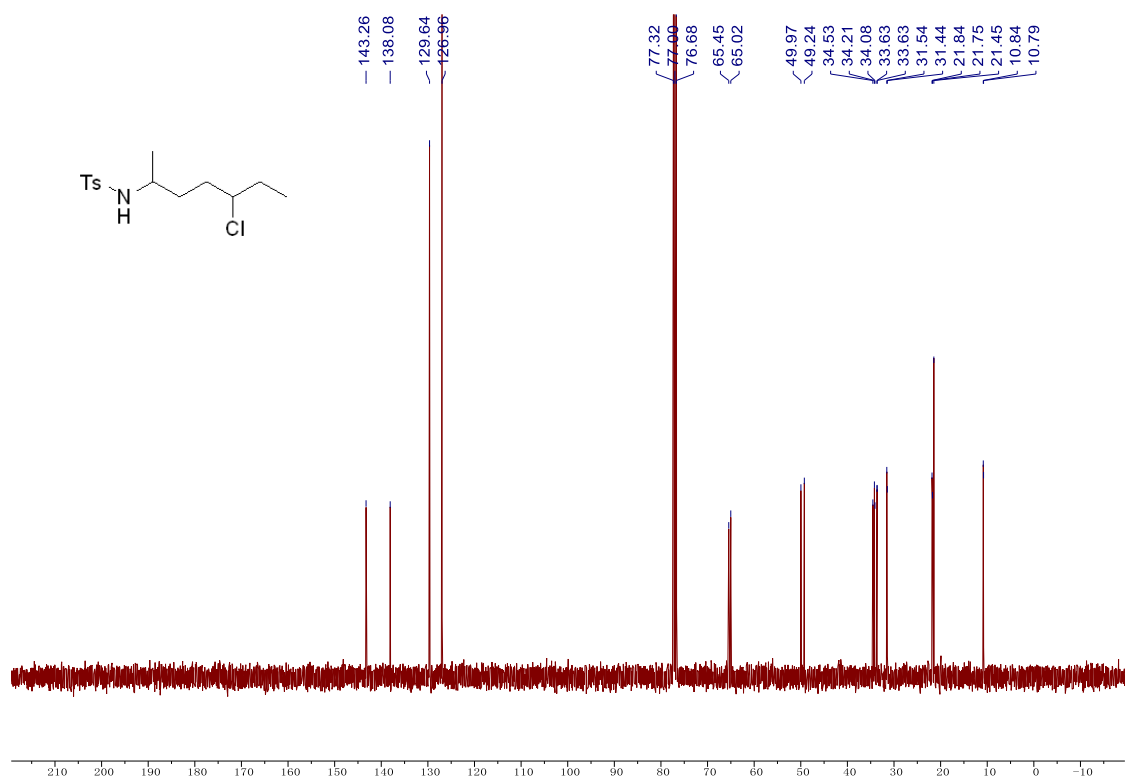


5f

^1H NMR (CDCl_3 , 400 MHz)

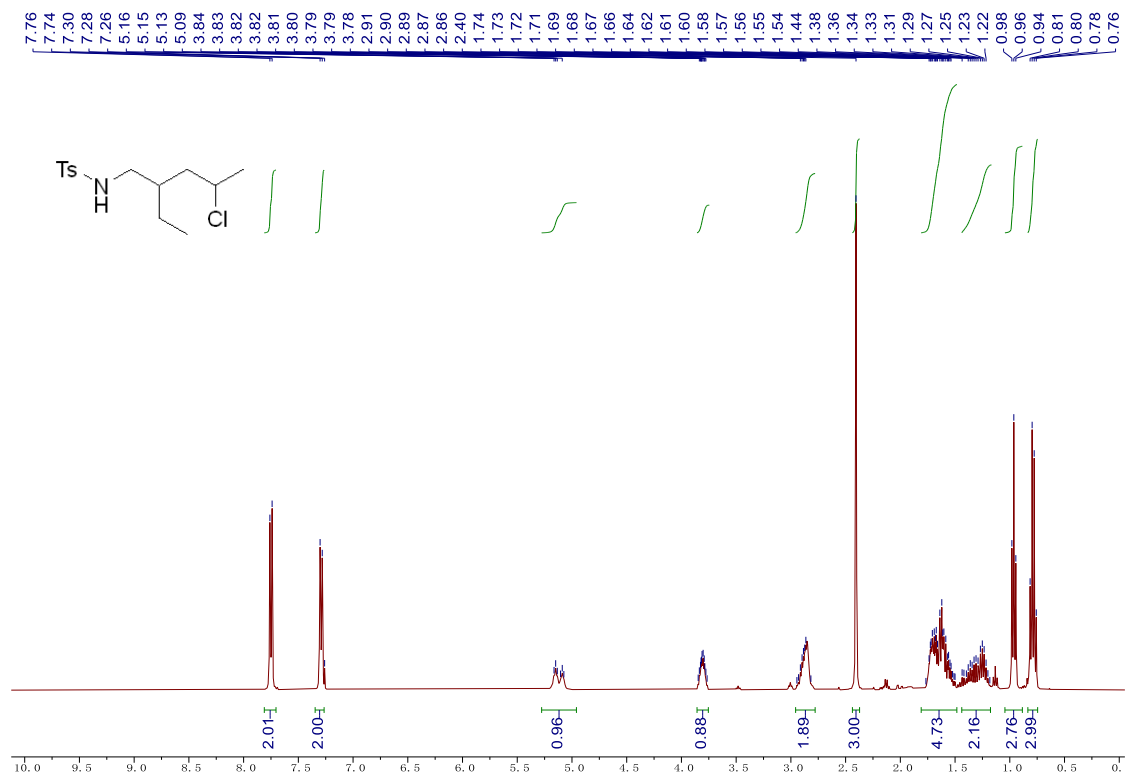


^{13}C NMR (CDCl_3 , 100 MHz)

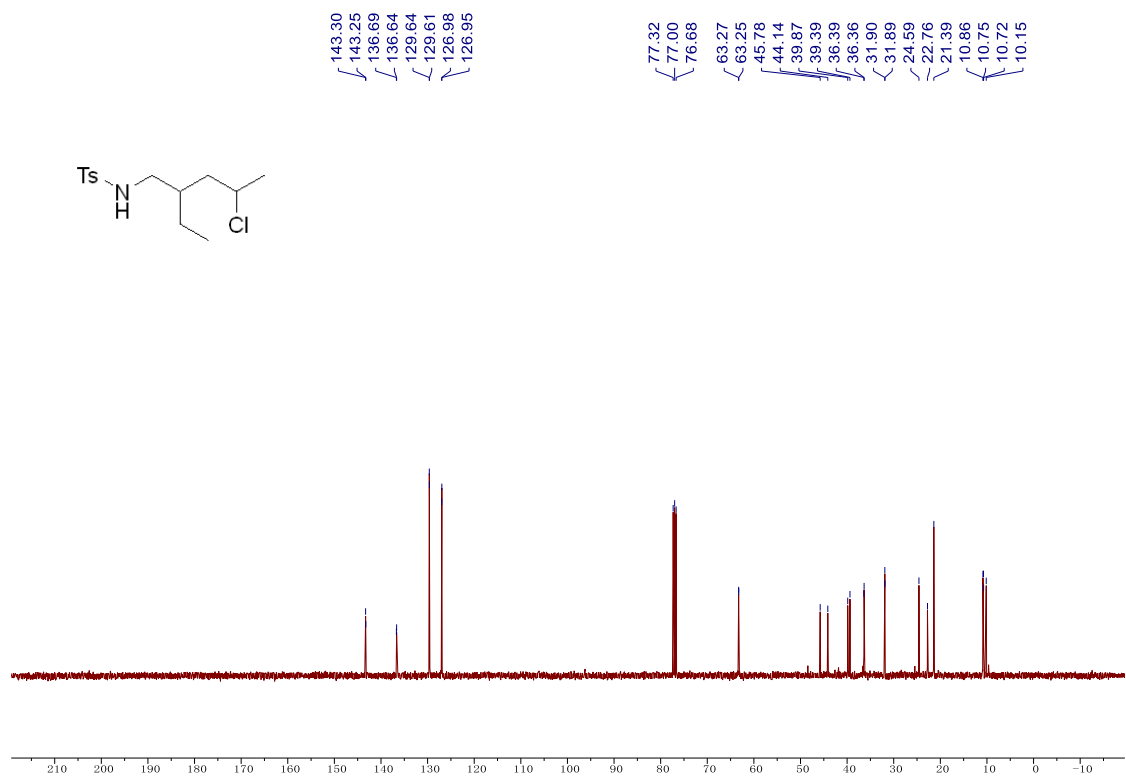


5g

^1H NMR (CDCl_3 , 400 MHz)

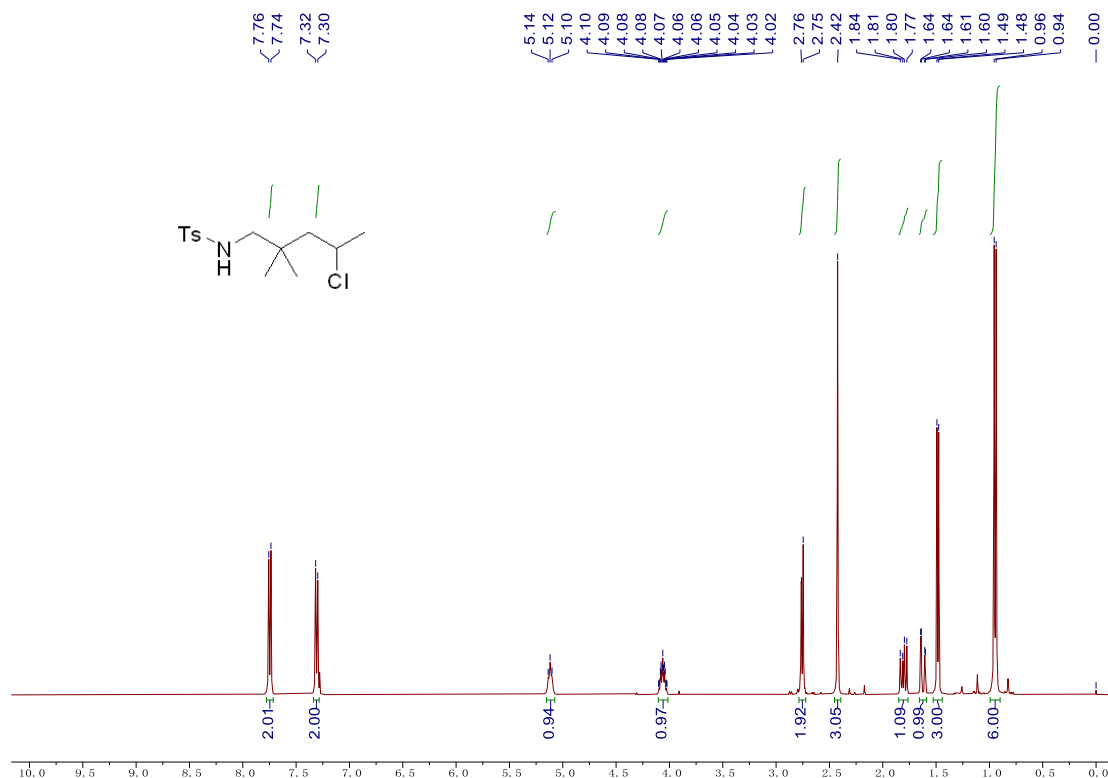


^{13}C NMR (CDCl_3 , 100 MHz)

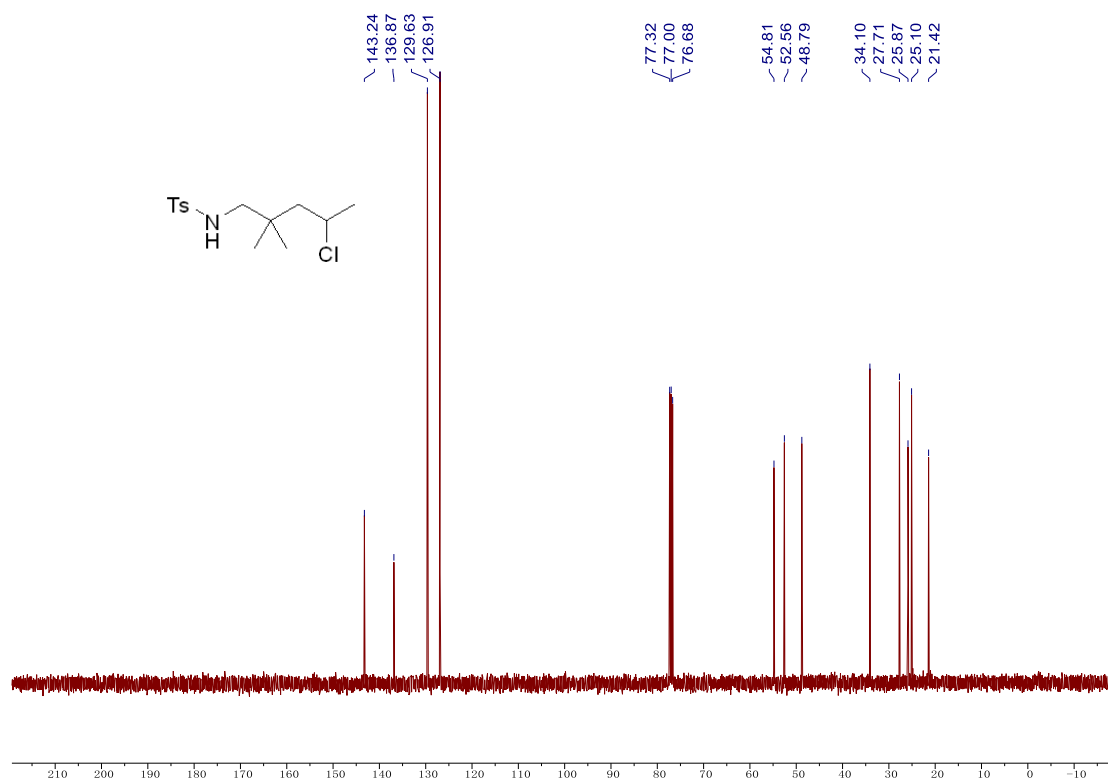


5h

^1H NMR (CDCl_3 , 400 MHz)

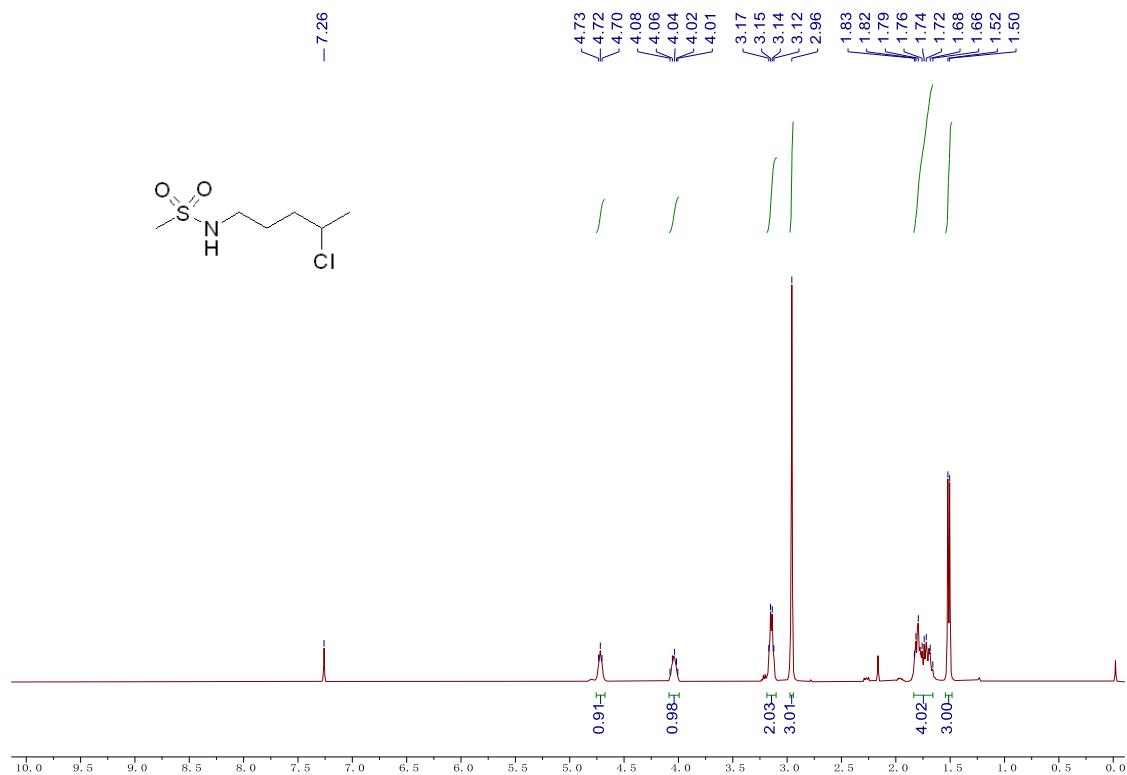


^{13}C NMR (CDCl_3 , 100 MHz)

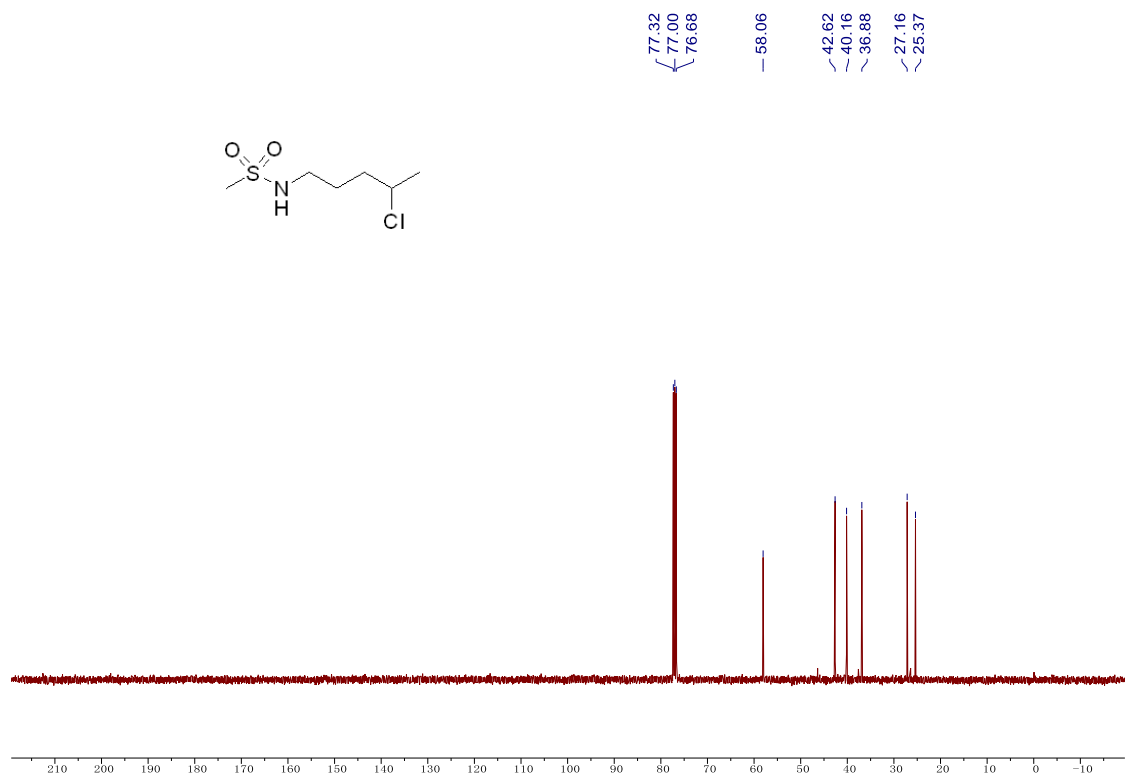


5i

^1H NMR (CDCl_3 , 400 MHz)

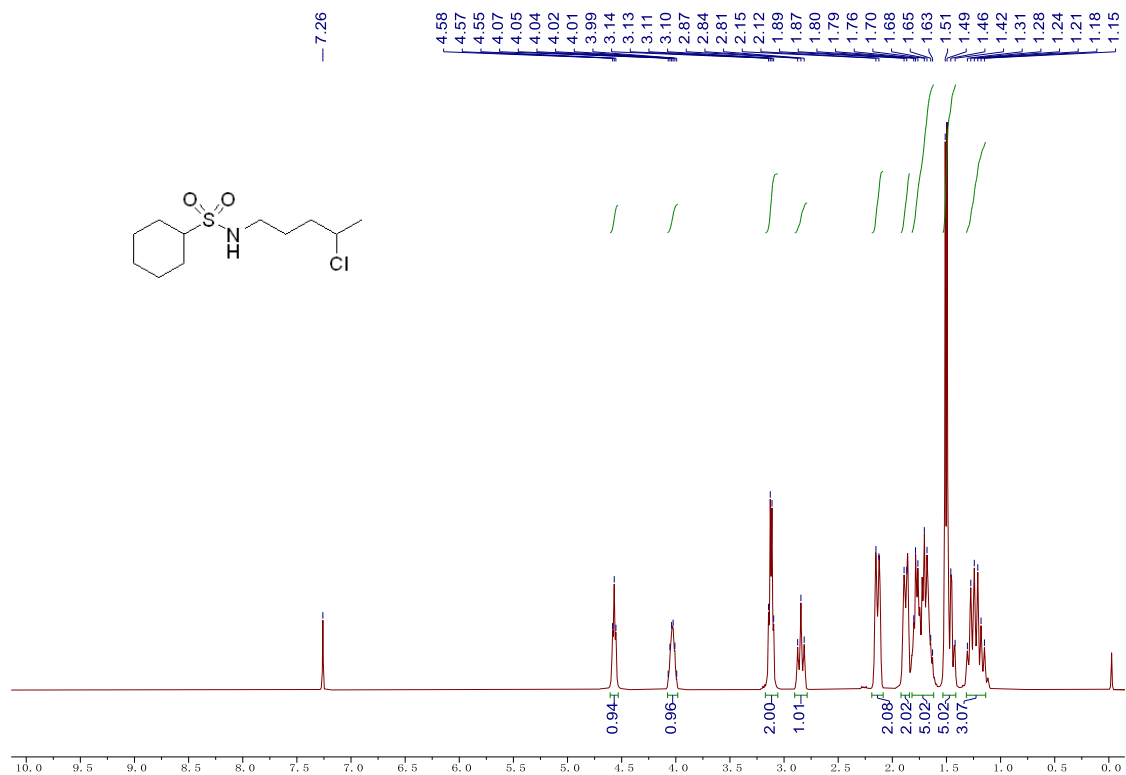


^{13}C NMR (CDCl_3 , 100 MHz)

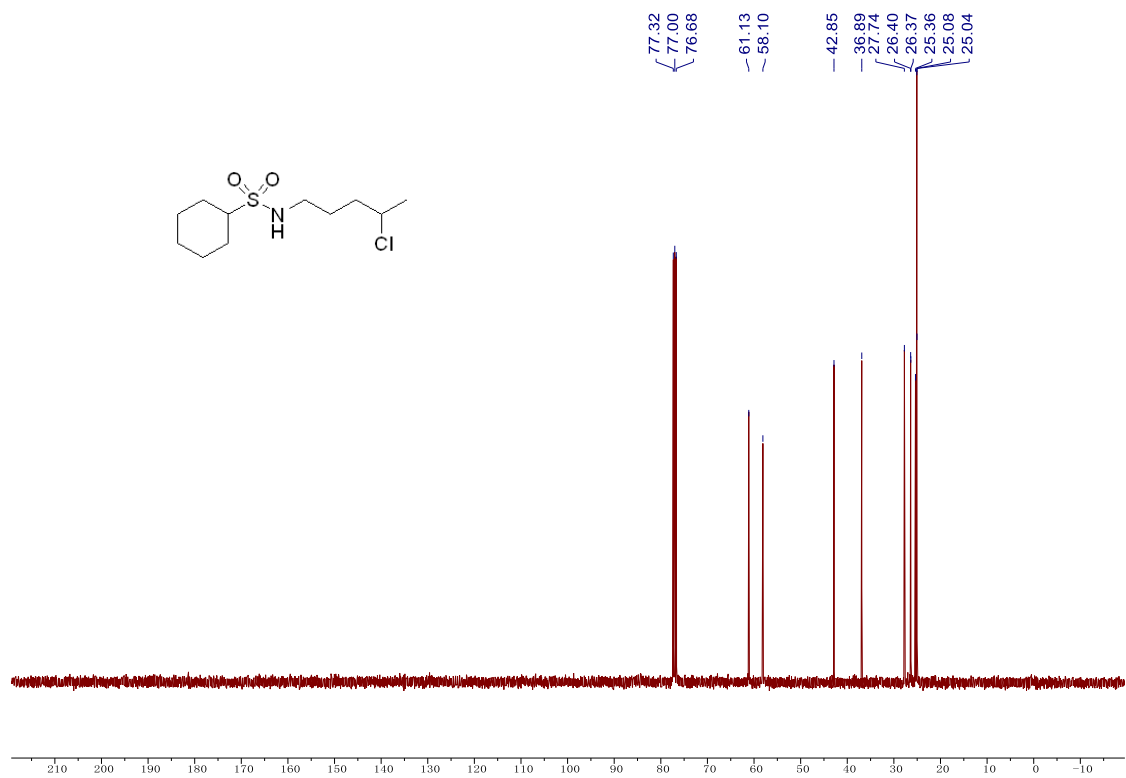


5j

^1H NMR (CDCl_3 , 400 MHz)

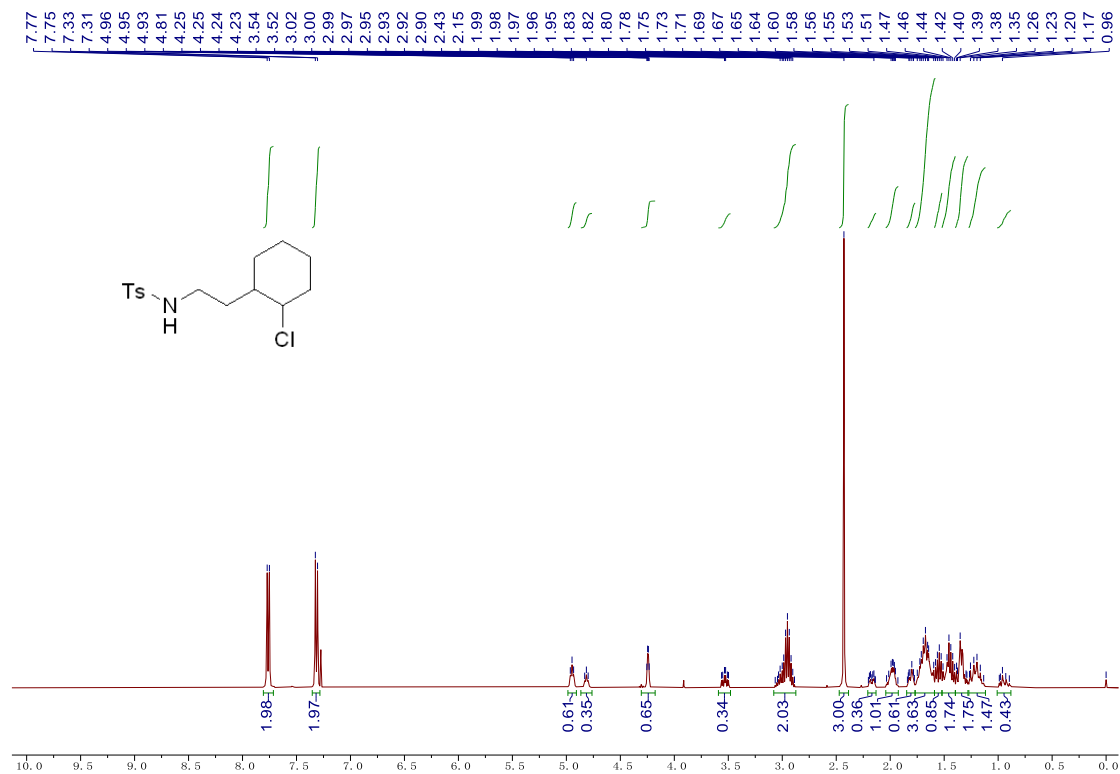


^{13}C NMR (CDCl_3 , 100 MHz)

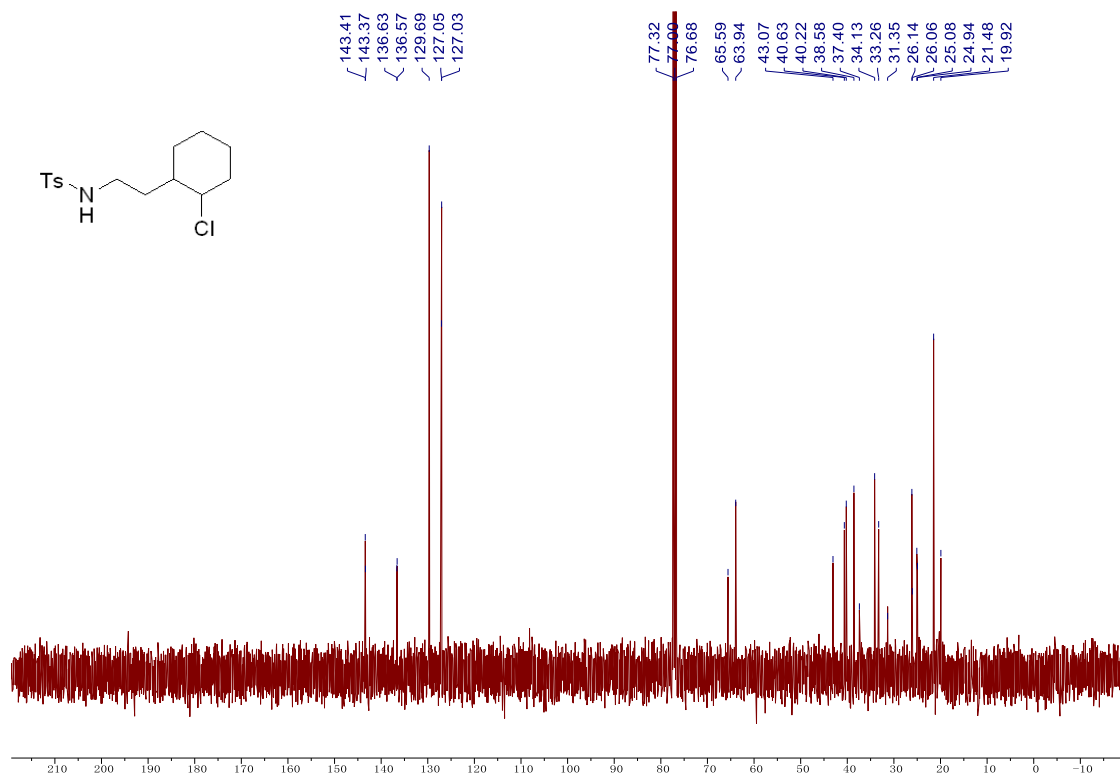


5k

^1H NMR (CDCl_3 , 400 MHz)

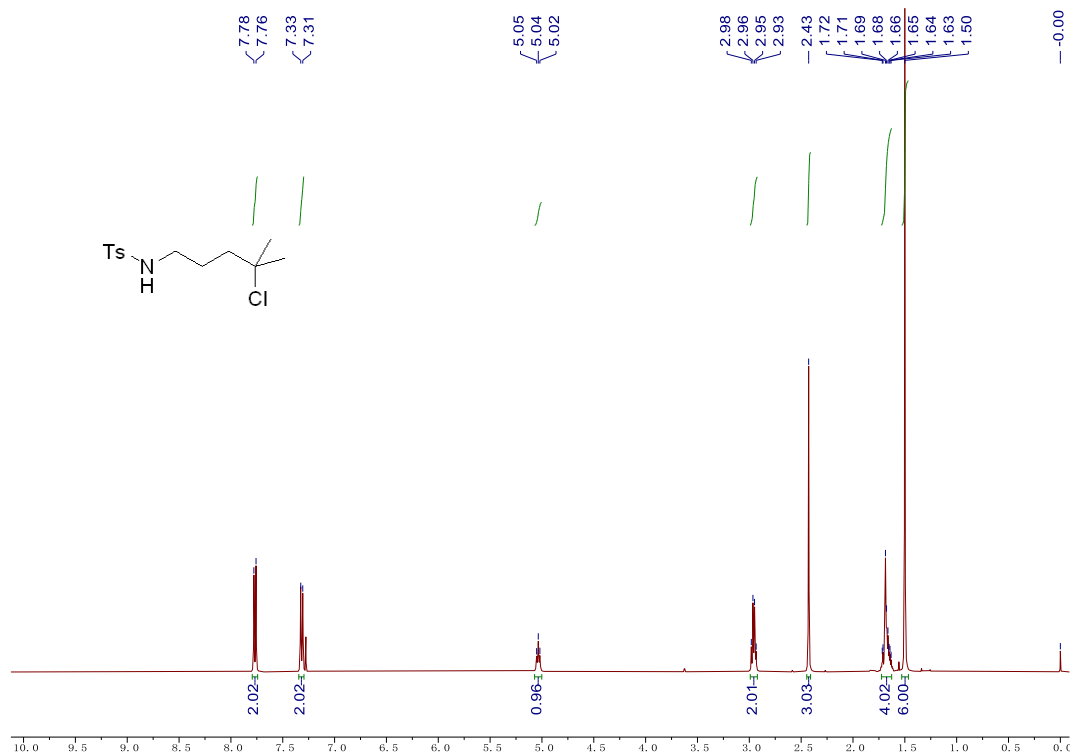


^{13}C NMR (CDCl_3 , 100 MHz)

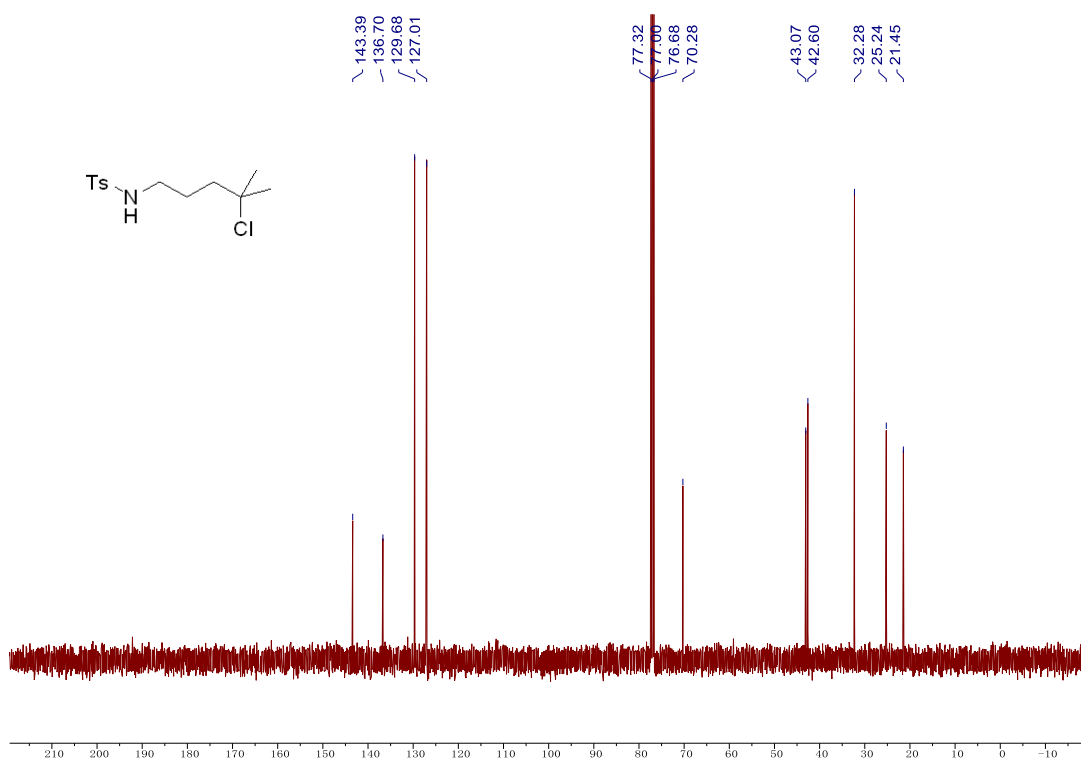


51

^1H NMR (CDCl_3 , 400 MHz)

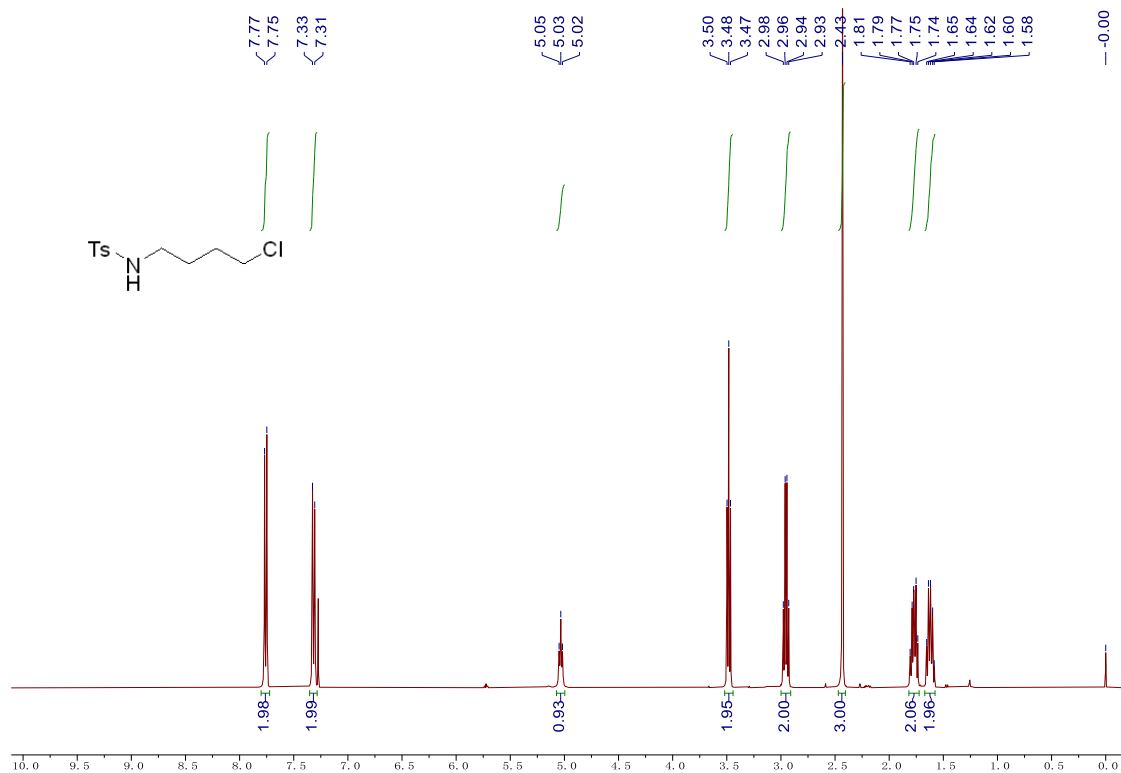


^{13}C NMR (CDCl_3 , 100 MHz)

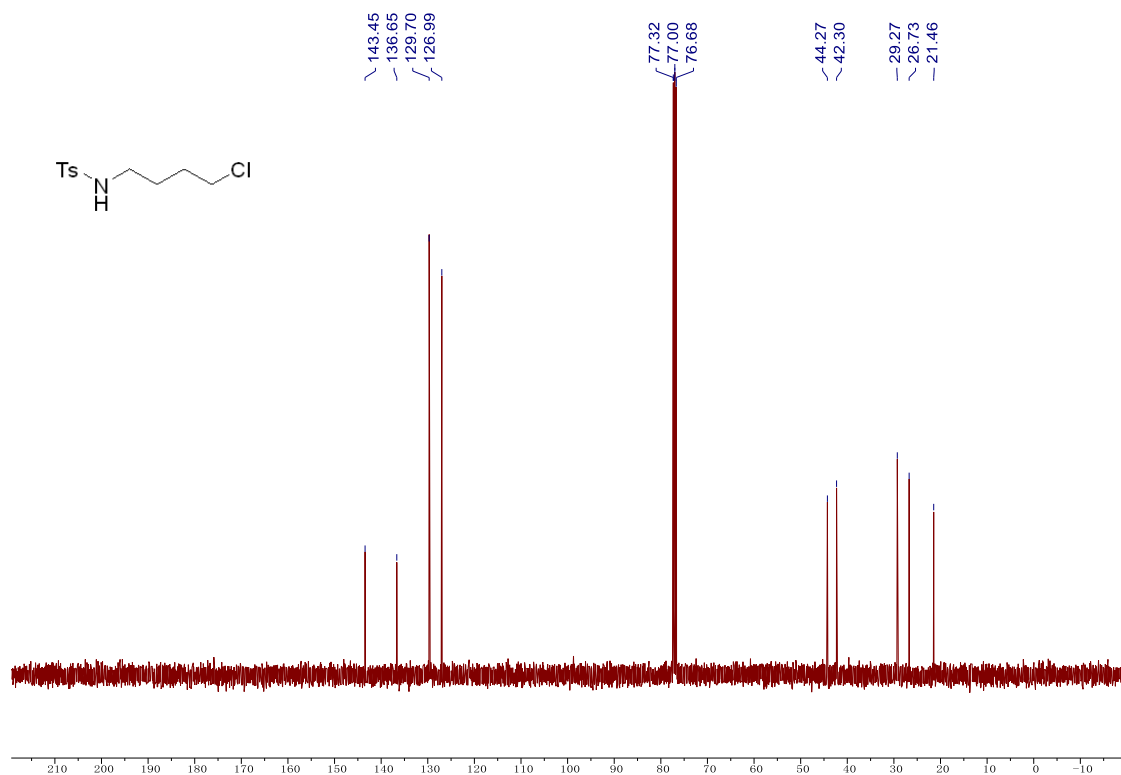


5m

^1H NMR (CDCl_3 , 400 MHz)

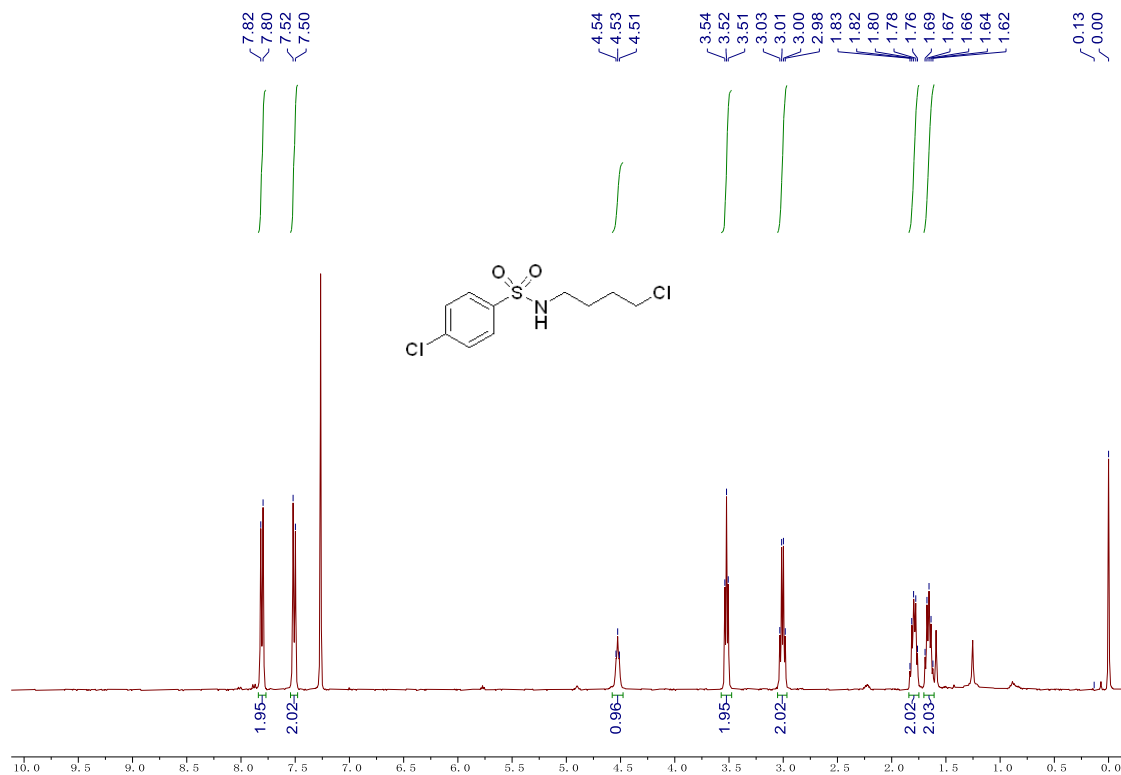


^{13}C NMR (CDCl_3 , 100 MHz)

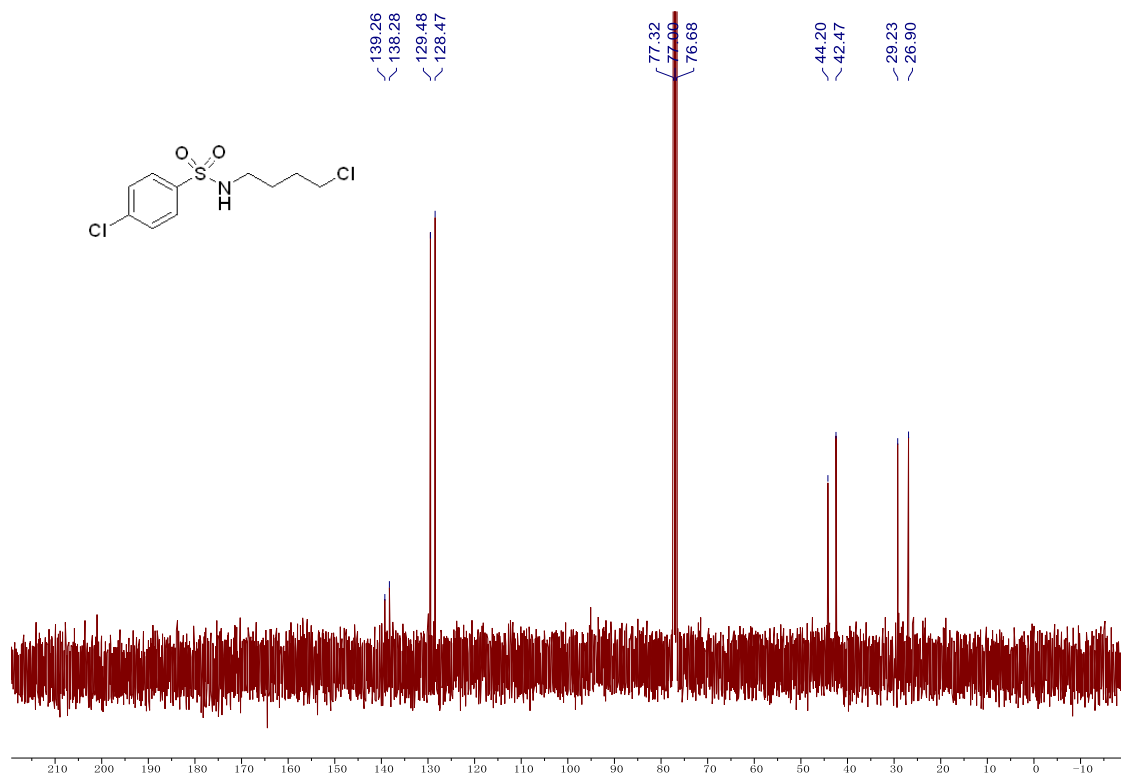


5n

^1H NMR (CDCl_3 , 400 MHz)

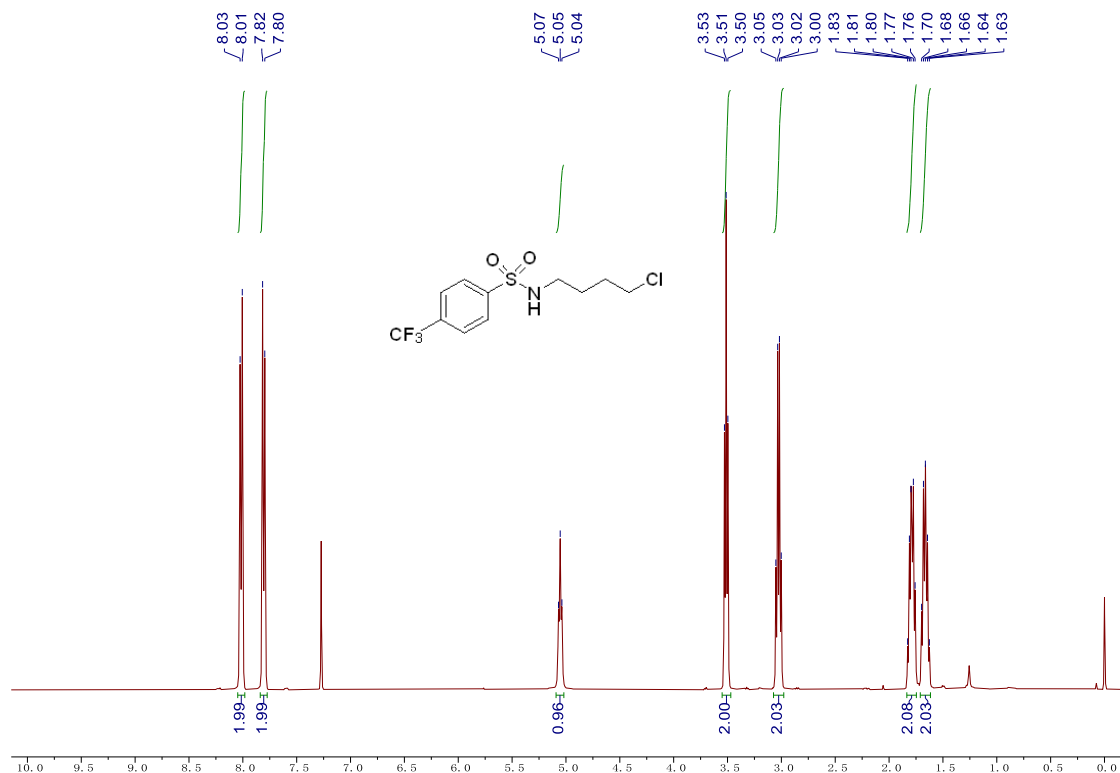


^{13}C NMR (CDCl_3 , 100 MHz)

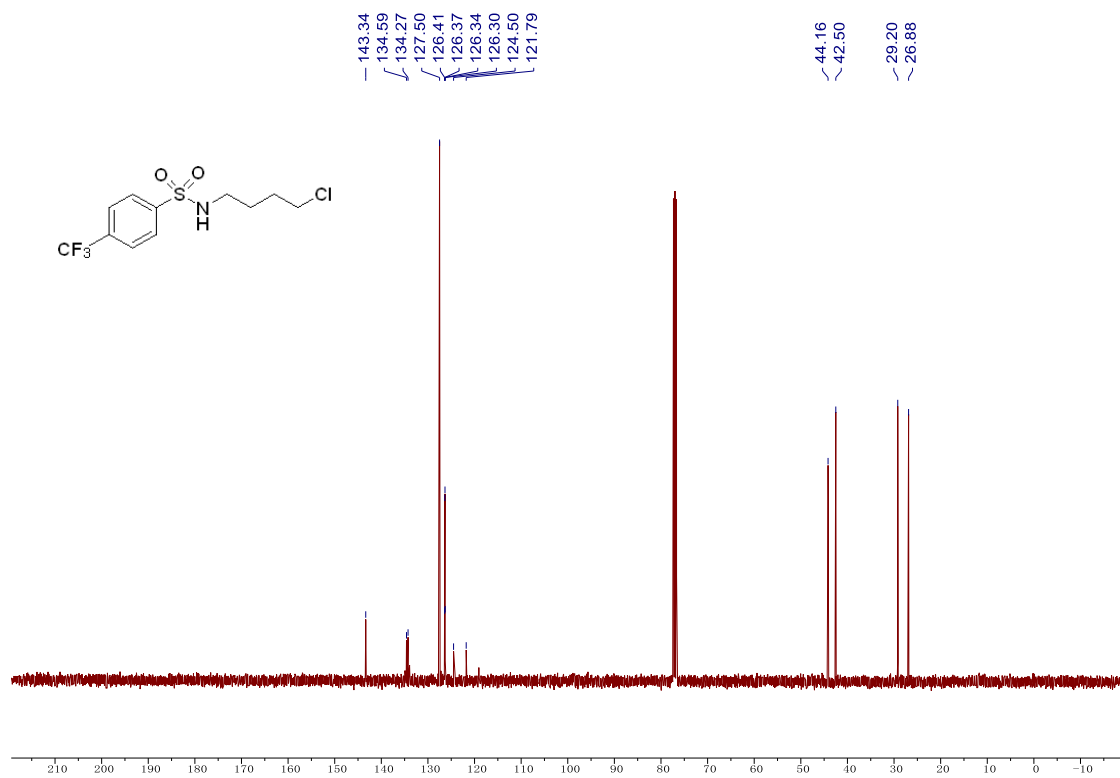


50

^1H NMR (CDCl_3 , 400 MHz)

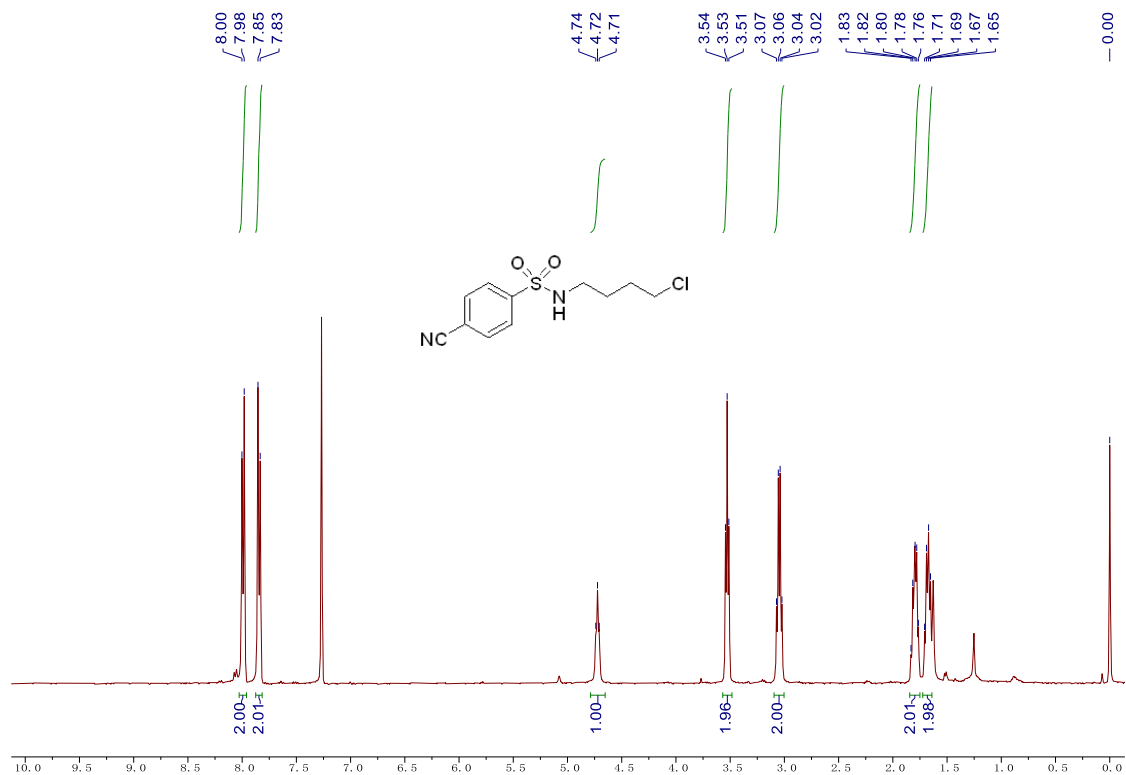


^{13}C NMR (CDCl_3 , 100 MHz)

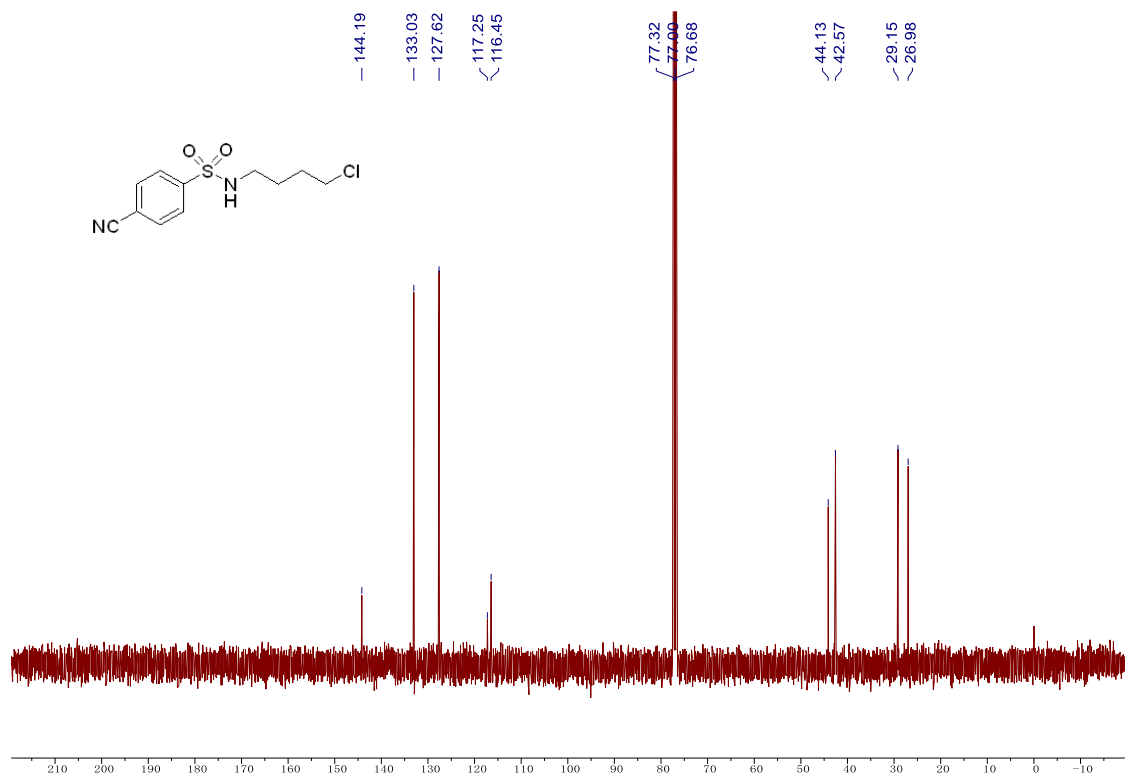


5p

^1H NMR (CDCl_3 , 400 MHz)

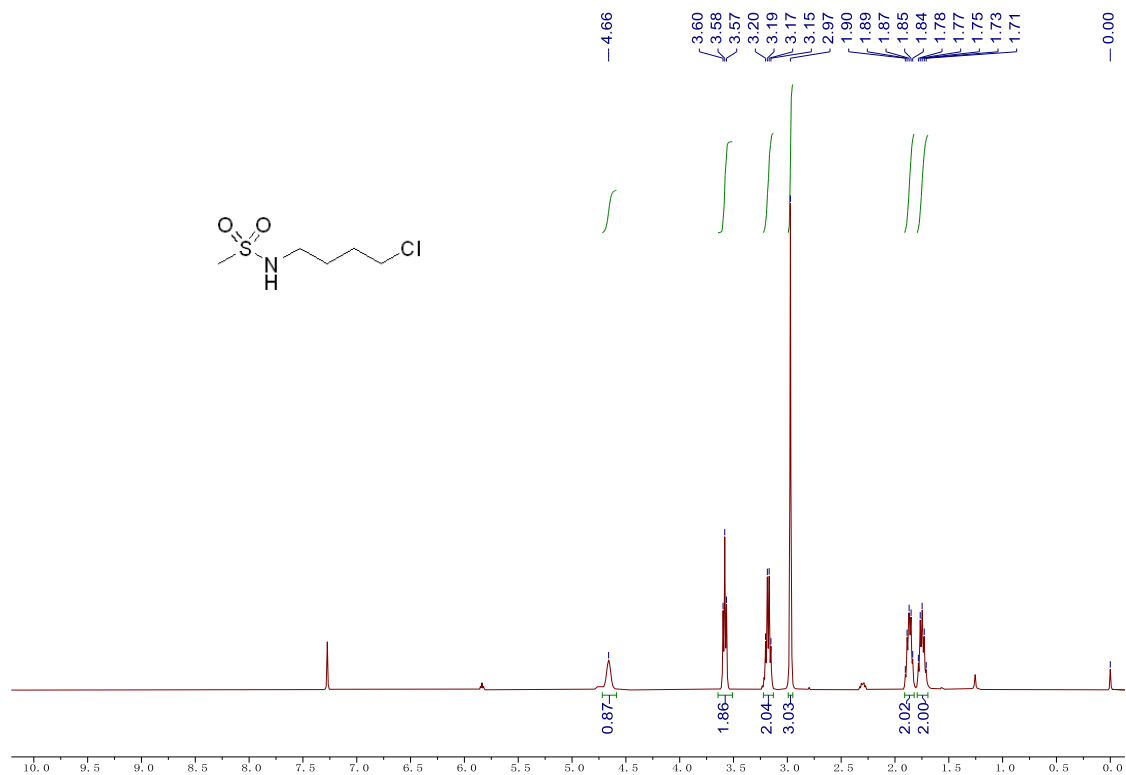


^{13}C NMR (CDCl_3 , 100 MHz)

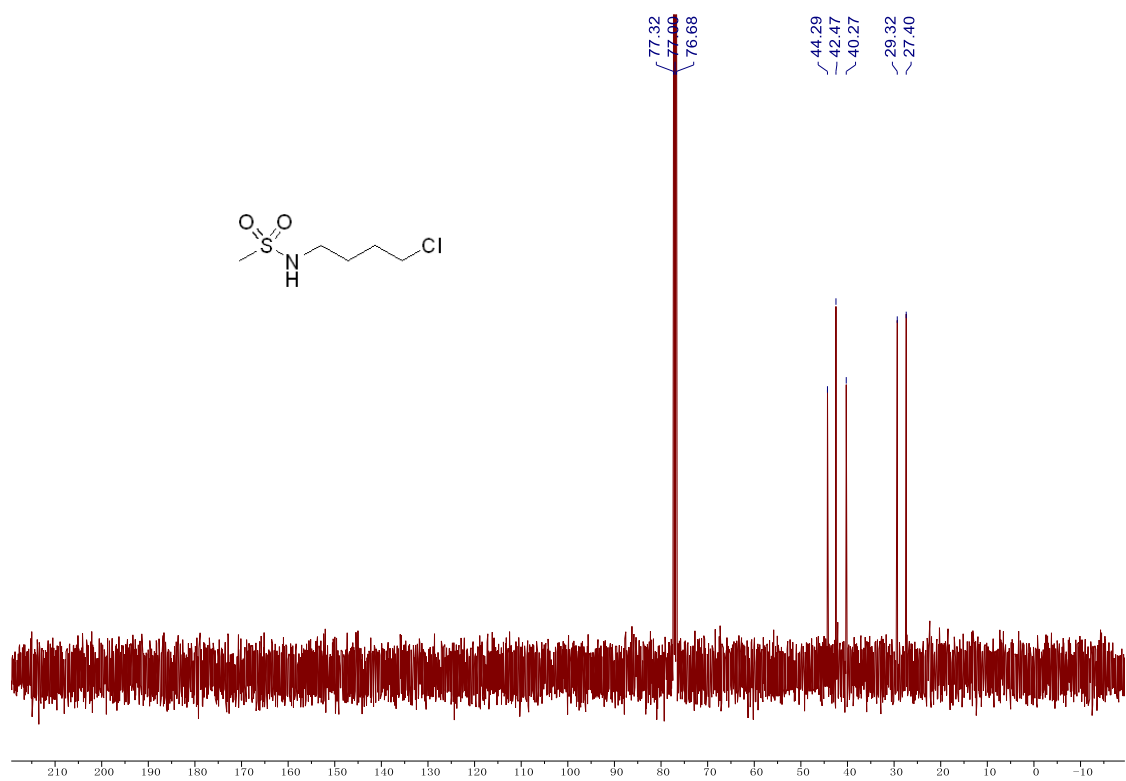


5q

^1H NMR (CDCl_3 , 400 MHz)

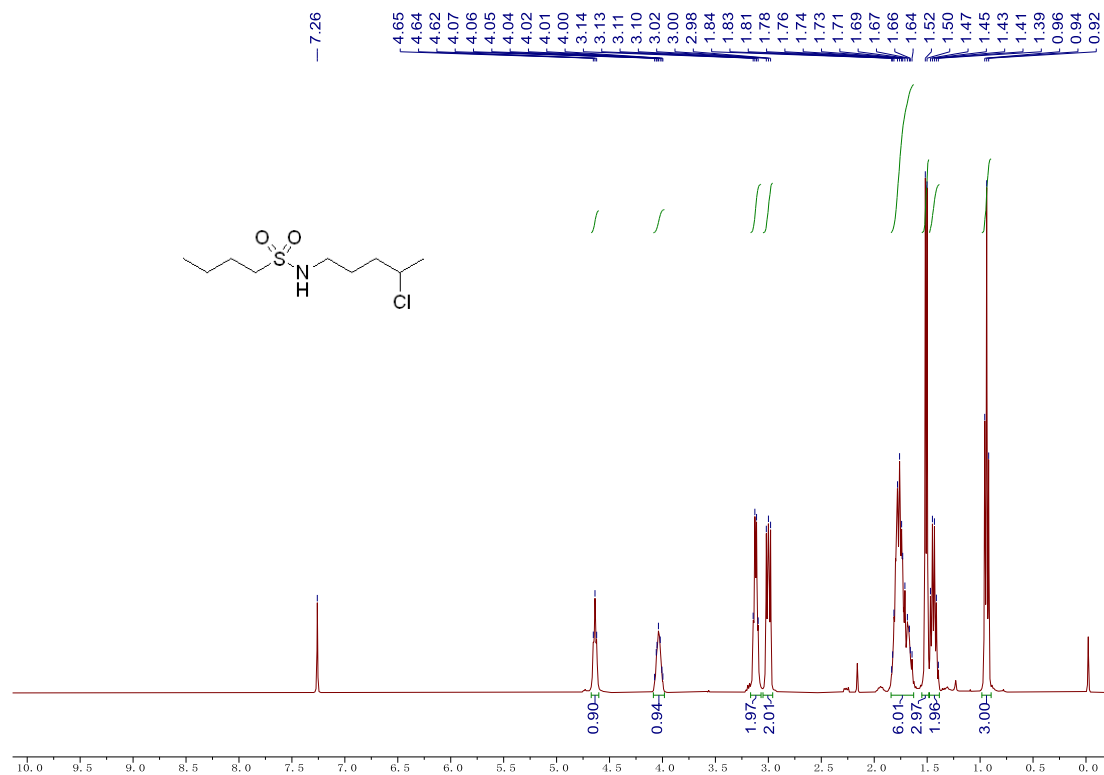


^{13}C NMR (CDCl_3 , 100 MHz)

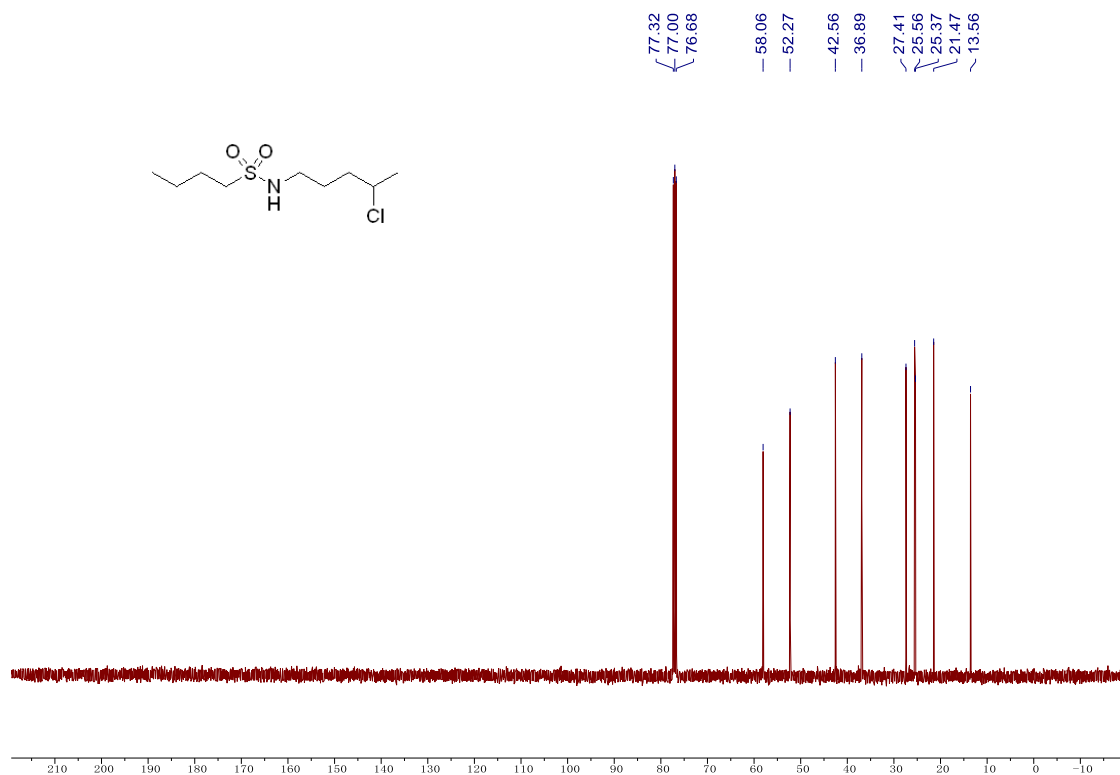


5r

^1H NMR (CDCl_3 , 400 MHz)

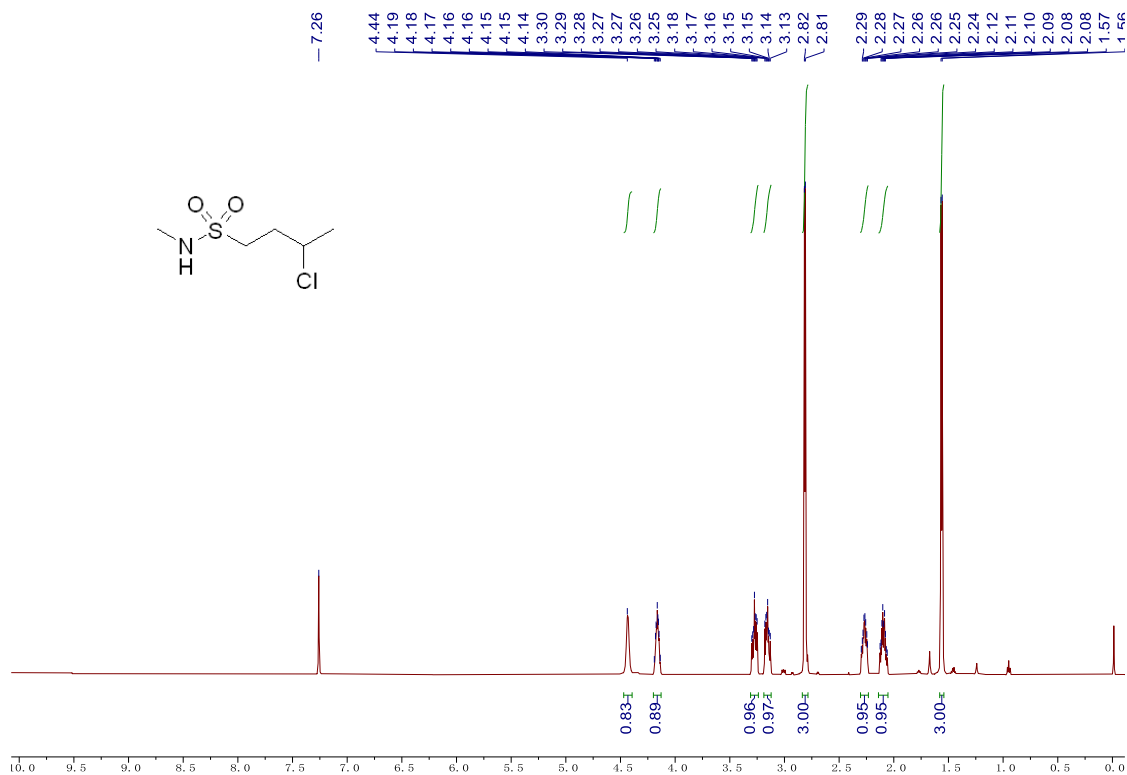


^{13}C NMR (CDCl_3 , 100 MHz)

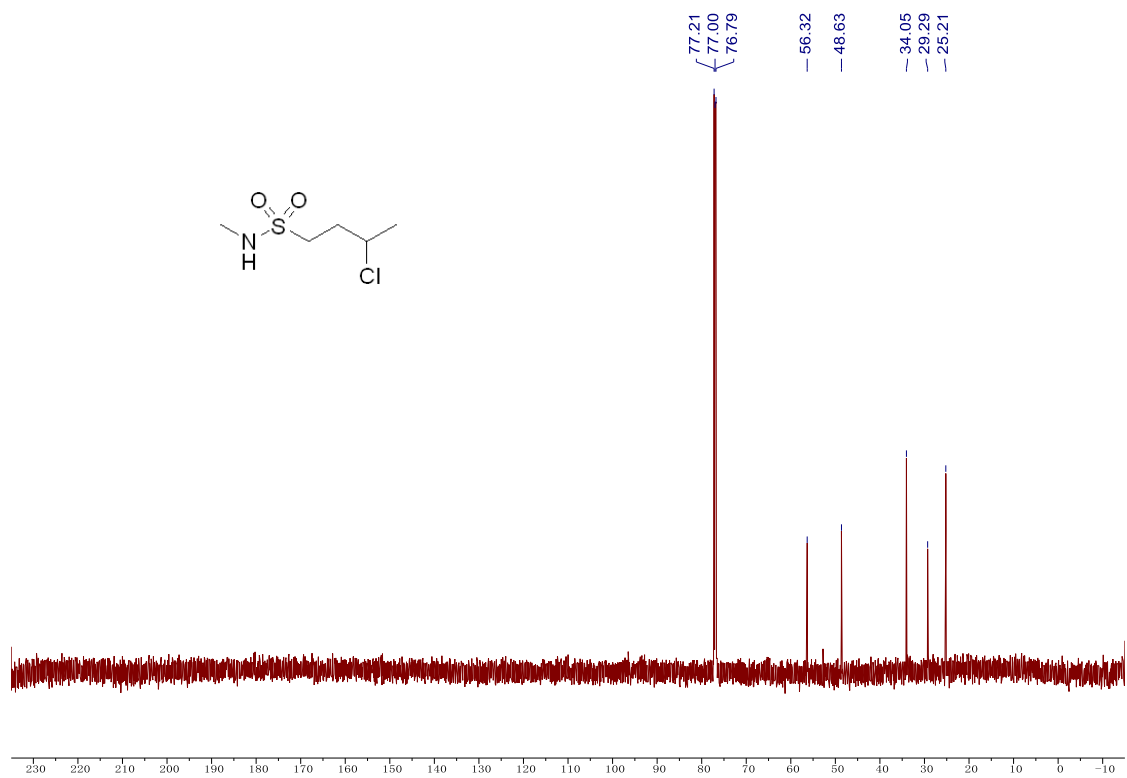


5s

^1H NMR (CDCl_3 , 600 MHz)

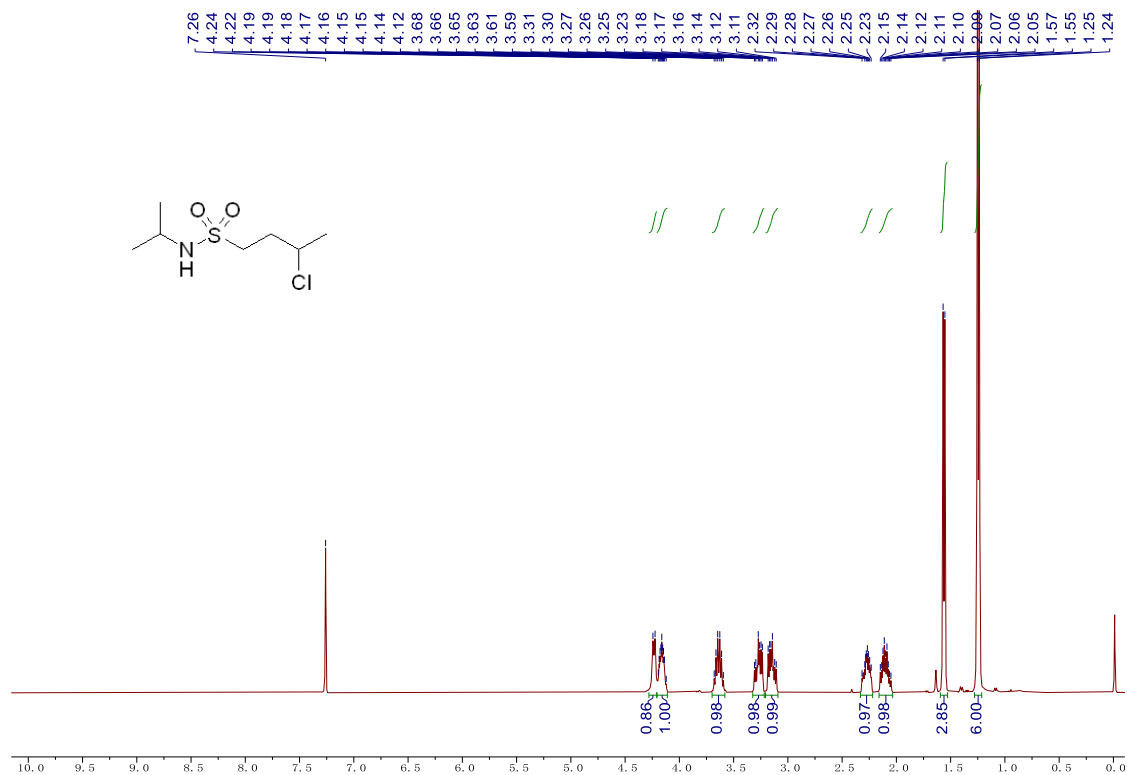


^{13}C NMR (CDCl_3 , 150 MHz)

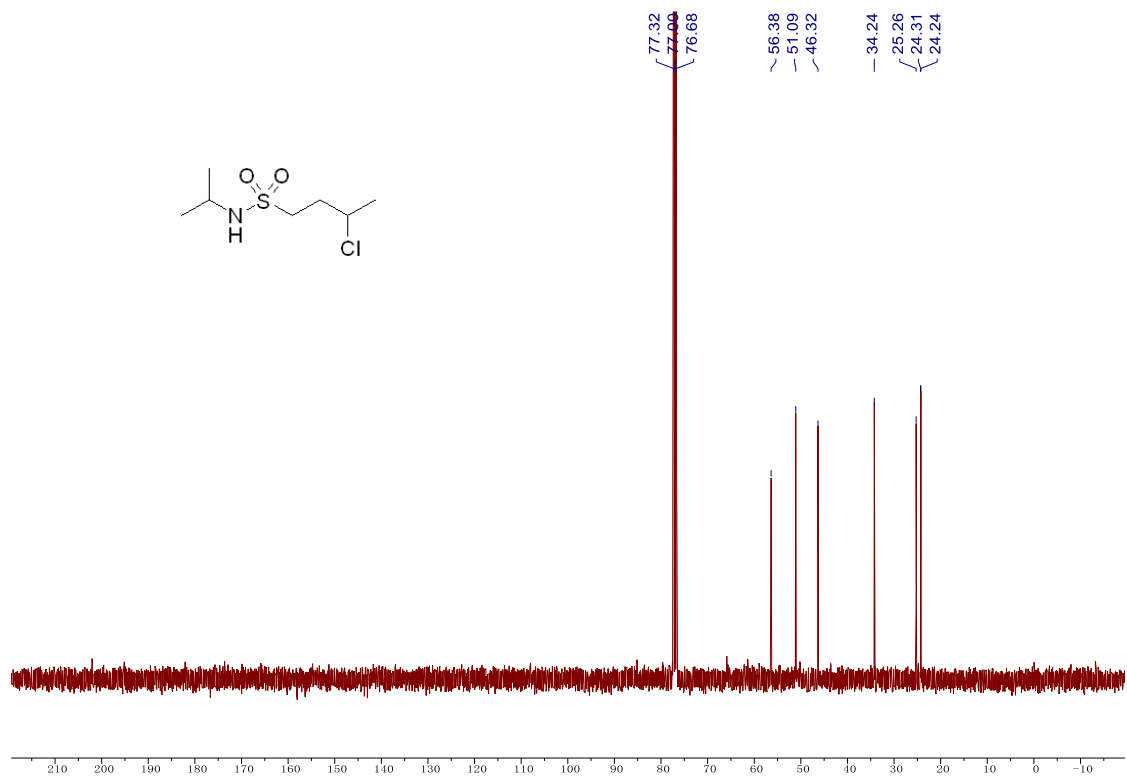


5t

^1H NMR (CDCl_3 , 400 MHz)

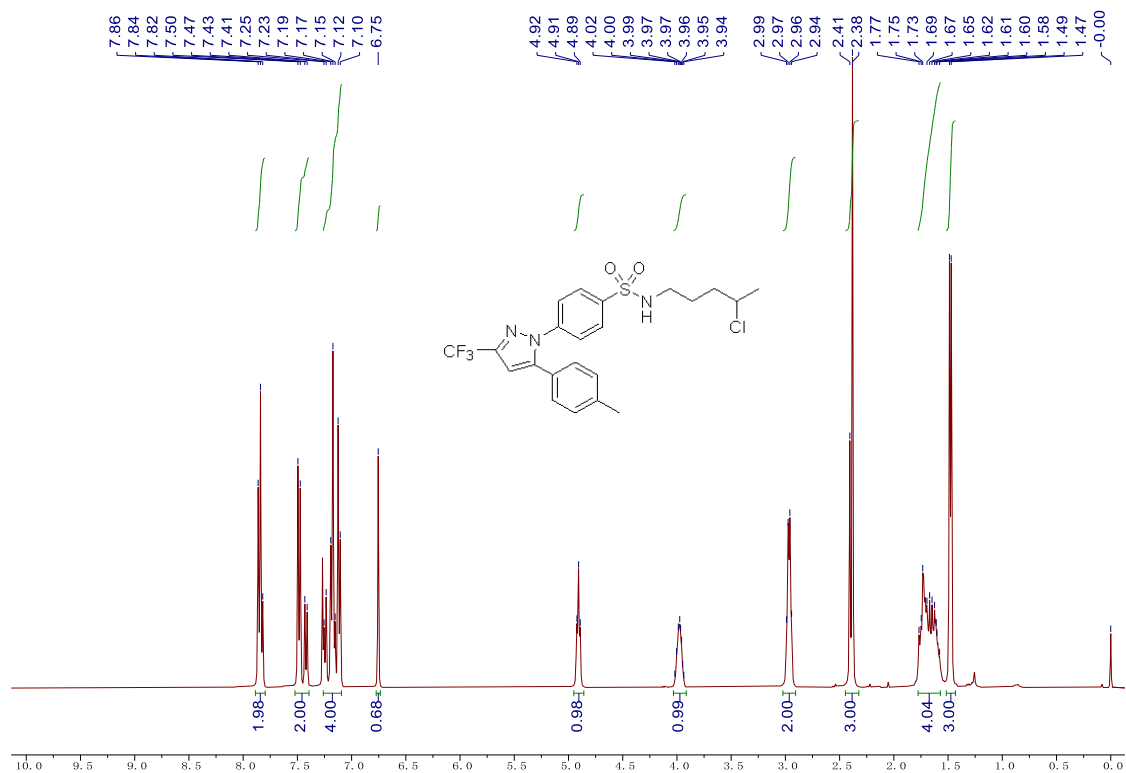


^{13}C NMR (CDCl_3 , 100 MHz)

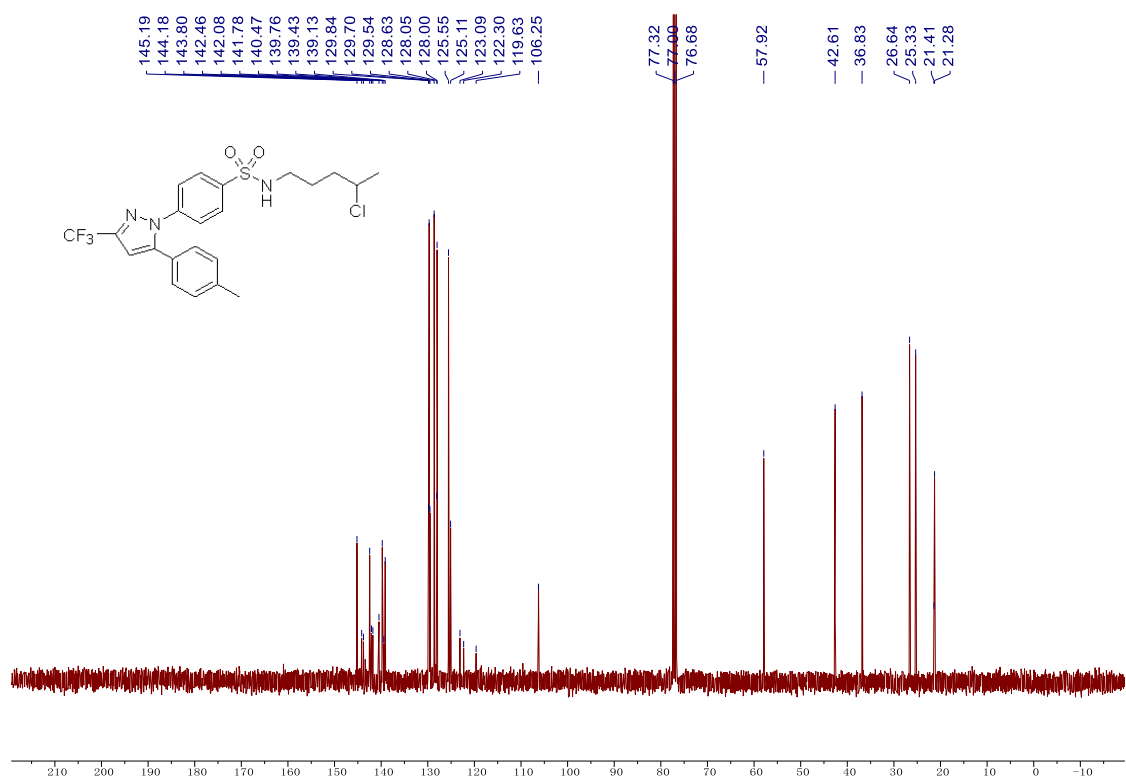


5u

^1H NMR (CDCl_3 , 400 MHz)

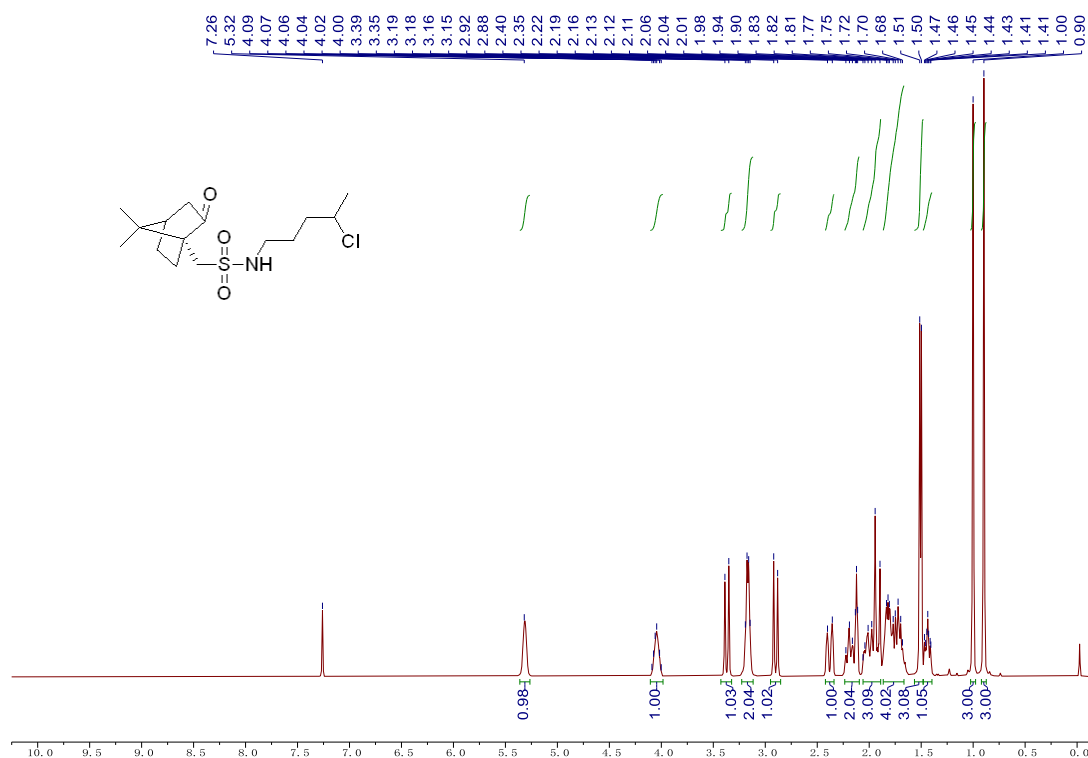


^{13}C NMR (CDCl_3 , 100 MHz)



5v

^1H NMR (CDCl_3 , 400 MHz)



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

