Supporting Information

One Pot Preparation of α-Dithioacetal/α-Diselenoacetal Amides via Cascade Dual-C-S/C-Se Bond Formation and C-C Bond Cleavage of 3-Oxo-Butanamides

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General Information

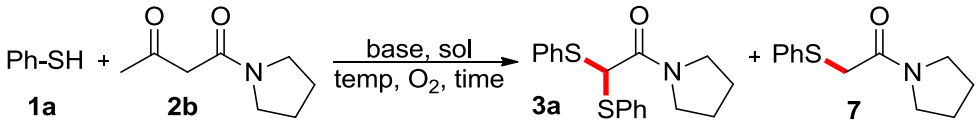
All reactions were carried out in a dry solvent under argon atmosphere unless otherwise noted. NMR spectra were recorded on Bruker 400 MHz (400 MHz for ^{1}H NMR and 100 MHz for ^{13}C NMR) spectrometers. Proton chemical shifts are reported relative to a residual solvent peak ($CDCl_3$ at 7.26 ppm, CD_3COCD_3 at 2.05 ppm, CD_3SOCD_3 at 2.50 ppm). Carbon chemical shifts are reported relative to a residual solvent peak ($CDCl_3$ at 77.3 ppm, CD_3COCD_3 at 29.8 ppm, CD_3SOCD_3 at 39.5 ppm). The following abbreviations were used to designate multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, m = multiplet, br = broad. Fourier transform infrared spectra (FT-IR) were recorded on an Agilent Cary 630 FT-IR instrument. High-resolution mass spectra (HRMS) were measured on a Brucker Daltonics Apex II 47e Specification (for HRMS).

Unless otherwise noted, all reagents were obtained from commercial suppliers and used without further purification. Substrates **2f, 2g, 2k**, **2l**, **2m** are commercially available. 3-oxo-butanamides **2a-2e**[1], **2h**[2], **2i**[3], **2j**[4], **3ab**[6], **7**[7] were prepared according to the literatures procedures.

Optimization of Reaction Conditions

A test tube equipped with a magnetic stir bar was charged with thiophenol **1a** (0.25 mmol, 2.5 equiv), 1-(pyrrolidin-1-yl) butane-1,3-dione **2b** (0.10 mmol, 1.0 equiv), base (0.30 mmol, 3.0 equiv) and solvent (1.0 mL) under oxygen atmosphere. The resulting mixture was stirred for 5 min at room temperature, and then heated at indicated temperature for 24 h. The reaction solution was cooled to ambient temperature, quenched by saturated $NaHCO_3$ aqueous solution, and extracted with ethyl acetate (3*25mL). The combined organic extracts were concentrated and the resulting residue was purified by column chromatography on silica gel (hexane / EtOAc = 1:1) to give **3a** as colorless oil.

Table S1. Optimization of Reaction Conditions[a].

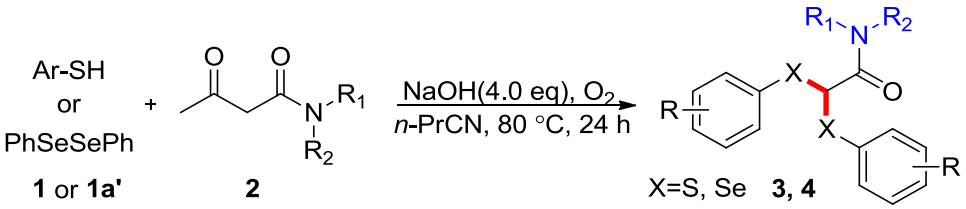


Entry	Base	Sol.	Temp./ °C	Yield[b] **3a** /%	Yield[b] **7** /%	**3a**/**7**
1	NaOH	*n*-PrCN	110	40	23	1.7:1
2	NaOH	DMSO	110	19	36	0.5:1
3	NaOH	DCE	110	N.R.	N.R.	
4	NaOH	toluene	110	6	14	0.4:1
5	NaOH	DMF	110	35	28	1.3:1
6	NaOH	*n*-PrCN	80	63	26	2.4:1
7	NaOH	*n*-PrCN	50	19	37	0.5:1
8	K_2CO_3	*n*-PrCN	80	14	21	0.7:1
9	K_3PO_4	*n*-PrCN	80	11	70	0.2:1
10	DBU	*n*-PrCN	80	7	17	0.4:1
11	AcOK	*n*-PrCN	80	5	9	0.6:1
12	/	*n*-PrCN	80	N.R.	N.R.	
13[c]	NaOH	*n*-PrCN	80	21	57	0.4:1
14[d]	NaOH	*n*-PrCN	80	95	2	47.5:1
15[e]	NaOH	*n*-PrCN	80	67	19	3.5:1
16[f]	NaOH	*n*-PrCN	80	91	3	30:1
17[g]	NaOH	*n*-PrCN	80	N.R.	N.R.	

[a]Reaction conditions: **1a** (0.25 mmol, 2.5 equiv), **2b** (0.10 mmol, 1.0 equiv), base (0.30 mmol, 3.0 equiv), solvent (1.0 mL), O_2 (1 atm), 24 h. [b]Isolated yields. [c]NaOH was used 0.20 mmol (2.0 equiv). [d]NaOH was used 0.40 mmol (4.0 equiv). [e]NaOH was used 0.50 mmol (5.0 equiv). [f]Air atmosphere. [g]Ar atmosphere. *n*-PrCN = Butanenitrile. N.R. = no results.

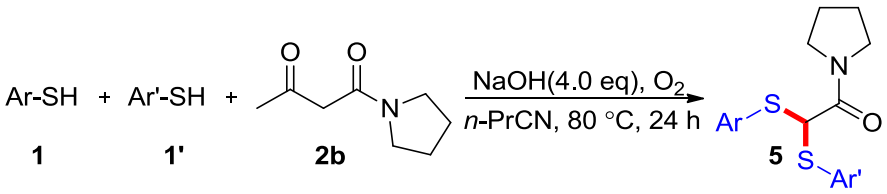
General procedure

1. The procedure for the thiols **1** or diselenide **1a'** bissulfenylation/bisselenenylation of the *α*-C-H bond of the corresponding amides **2**.



A test tube equipped with a magnetic stir bar was charged with thiols **1** (0.25 mmol, 2.5 equiv) or PhSePhSe **1a'** (0.125 mmol, 1.25 equiv), 3-oxo-butanamides **2** (0.10 mmol, 1.0 equiv), NaOH (0.40 mmol, 4.0 equiv) and *n*-PrCN (1.0 mL) under O_2 atmosphere. The resulting mixture was stirred for 5 min at room temperature, and then heated at 80 ℃ for 24 h. The reaction solution was cooled to ambient temperature, quenched by saturated $NaHCO_3$ aqueous solution, and extracted with ethyl acetate (3*25mL), the combined organic extracts were concentrated and the resulting residue was purified by column chromatography on silica gel (hexane / EtOAc = 2 : 1) to provide the corresponding product **3** or **4**.

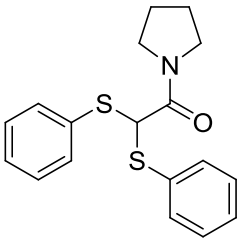
2. The proceduce for the thiols **1** and **1'** hetero-bissulfenylation of the *α*-C-H bond amide **2b**.



A test tube equipped with a magnetic stir bar was charged with thiol **1** (0.20 mmol, 1.0 equiv), thiol **1'** (0.20 mmol, 1.0 equiv), 1-(pyrrolidin-1-yl) butane-1,3-dione **2b** (0.20 mmol, 1.0 equiv), NaOH (0.80 mmol, 4.0 equiv) and *n*-PrCN (2.0 mL) under O_2 atmosphere. The resulting mixture was stirred for 5 min at room temperature, and then heated at 80 ℃ for 24 h. The reaction solution was cooled to ambient temperature, quenched by saturated $NaHCO_3$ aqueous solution, and extracted with ethyl acetate (3*25mL), the combined organic extracts were concentrated and the resulting residue was purified by column chromatography on silica gel (hexane / EtOAc = 2 : 1) to provide the desired product **5**.

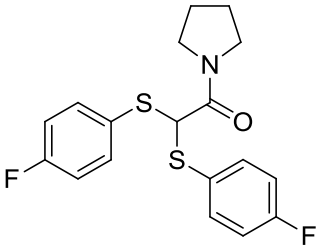
Characterization data of products

2,2-bis(phenylthio)-1-(pyrrolidin-1-yl)ethanone (3a)



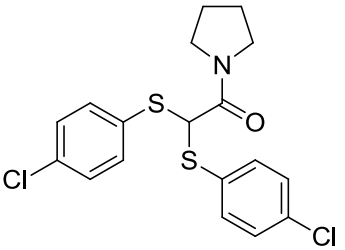
30.3 mg, 95%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.54 (dd, *J* = 4.2, 3.3 Hz, 4H), 7.34 (dd, *J* = 4.9, 1.8 Hz, 6H), 5.37 (s, 1H), 3.48 (t, *J* = 6.6 Hz, 2H), 3.33 (t, *J* = 6.7 Hz, 2H), 1.81 (ddd, *J* = 21.5, 13.7, 7.3 Hz, 4H); ^{13}C NMR (100 MHz, Acetone) δ 165.51, 134.13, 134.04, 129.71, 128.97, 59.80, 47.25, 46.91, 26.72, 24.69; IR (KBr, ν / cm^{-1}) 2982, 1737, 1649, 1435, 1243, 1046, 736, 691; HRMS (ESI^{+}) Calcd for $C_{18}H_{19}NNaOS_2^{+}$ (M+Na^{+}) 352.0800, Found 352.0800.

2,2-bis((4-fluorophenyl)thio)-1-(pyrrolidin-1-yl)ethanone (3b)



33.2 mg, 91%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.62 – 7.56 (m, 4H), 7.16 – 7.10 (m, 4H), 5.32 (s, 1H), 3.57 (t, *J* = 6.7 Hz, 2H), 3.34 (t, *J* = 6.8 Hz, 2H), 1.91 (dd, *J* = 12.9, 6.4 Hz, 2H), 1.84 (dd, *J* = 13.4, 6.4 Hz, 2H); ^{13}C NMR (100 MHz, Acetone) δ 165.27, 165.13, 162.67, 137.38, 137.29, 129.06, 129.02, 116.76, 116.54, 61.21, 47.29, 46.95, 26.77, 24.78; IR (KBr, ν / cm^{-1}) 2976, 1644, 1590, 1489, 1426, 1224, 1156, 837; HRMS (ESI^{+}) Calcd for $C_{18}H_{17}F_2NNaOS_2^{+}$ (M+Na^{+}) 388.0612, Found 388.0619.

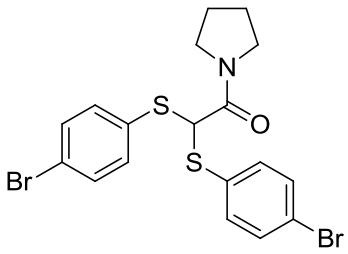
2,2-bis((4-chlorophenyl)thio)-1-(pyrrolidin-1-yl)ethanone (3c)



31.8 mg, 80%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.56 – 7.51 (m, 4H), 7.37 (d, *J* = 8.5 Hz, 4H), 5.47 (s, 1H), 3.64 (t, *J* = 6.7 Hz, 2H), 3.35 (t, *J* = 6.9 Hz, 2H), 1.96 – 1.90 (m, 2H), 1.88 – 1.81 (m, 2H); ^{13}C NMR (100 MHz, Acetone) δ 165.00, 135.99, 134.85, 132.42, 129.74, 60.07, 47.34, 47.03, 26.78, 24.78; IR (KBr, ν / cm^{-1}) 2973, 1642, 1476, 1426, 1094, 1014, 811; HRMS (ESI^{+}) Calcd for $C_{18}H_{17}Cl_2NNaOS_2^{+}$ (M+Na^{+}) 420.0021, Found 420.0017.

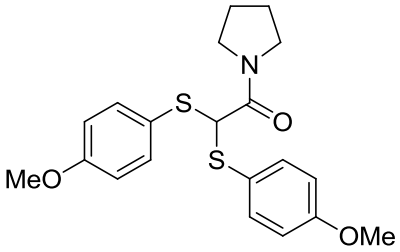
2,2-bis((4-bromophenyl)thio)-1-(pyrrolidin-1-yl)ethanone (3d)

36.4 mg, 75%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.55 – 7.50 (m, 4H), 7.49 – 7.44 (m, 4H), 5.49 (s, 1H), 3.65 (t, *J* = 6.7 Hz, 2H), 3.35 (t, *J* = 6.9 Hz, 2H), 1.98 – 1.92 (m, 2H), 1.88 – 1.82 (m, 2H); ^{13}C NMR (100 MHz, DMSO) δ 163.73, 134.95, 131.71, 131.36, 121.72, 57.91, 46.31, 46.16, 25.62, 23.70; IR (KBr, ν / cm^{-1}) 2952, 1640, 1472, 1426, 1089, 1008, 809; HRMS (ESI^{+}) Calcd for $C_{18}H_{17}Br_2NNaOS_2^{+}$ (M+Na^{+}) 507.9011, Found 507.9005.



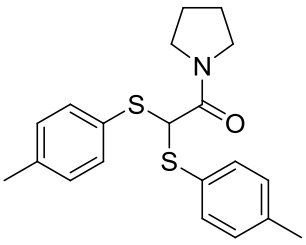
2,2-bis((4-methoxyphenyl)thio)-1-(pyrrolidin-1-yl)ethanone (3e)

35.0 mg, 90%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.46 (d, *J* = 8.8 Hz, 4H), 6.91 (d, *J* = 8.8 Hz, 4H), 5.05 (s, 1H), 3.81 (s, 6H), 3.42 (t, *J* = 6.5 Hz, 2H), 3.32 (t, *J* = 6.7 Hz, 2H), 1.87 – 1.76 (m, 4H); ^{13}C NMR (100 MHz, Acetone) δ 165.93, 161.28, 137.11, 124.35, 115.25, 62.16, 55.66, 47.21, 46.82, 26.76, 24.76; IR (KBr, ν / cm^{-1}) 2971, 1644, 1591, 1492, 1423, 1249, 1030, 833; HRMS (ESI^{+}) Calcd for $C_{20}H_{23}NNaO_3S_2^{+}$ (M+Na^{+}) 412.1012, Found 412.1010.



1-(pyrrolidin-1-yl)-2,2-bis(p-tolylthio)ethanone (3f)

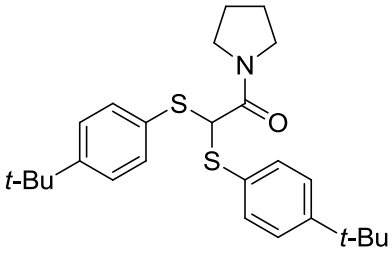
24.3 mg, 68%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.40 (d, *J* = 8.1 Hz, 4H), 7.16 (d, *J* = 7.9 Hz, 4H), 5.21 (s, 1H), 3.47 (t, *J* = 6.6 Hz, 2H), 3.33 (t, *J* = 6.7 Hz, 2H), 2.32 (s, 6H), 1.89 – 1.77 (m, 4H); ^{13}C NMR (100 MHz, Acetone) δ 165.77, 139.22, 134.63, 130.57, 130.43, 60.79, 47.26, 46.90, 26.76, 24.76, 21.10; IR (KBr, ν / cm^{-1}) 2971, 1648, 1491, 1422, 814; 833; HRMS (ESI^{+}) Calcd for $C_{20}H_{23}NNaOS_2^{+}$ (M+Na^{+}) 380.1113, Found 380.1116.



2,2-bis((4-(tert-butyl)phenyl)thio)-1-(pyrrolidin-1-yl)ethanone (3g)

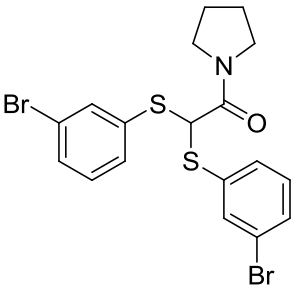
37.9 mg, 86%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.45 (d, J = 8.5 Hz, 4H), 7.39 (d, J = 8.5 Hz, 4H), 5.22 (s, 1H), 3.40 – 3.29 (m, 4H), 1.85 – 1.73 (m, 4H), 1.31 (s, 18H); ^{13}C NMR (100 MHz, Acetone) δ 165.90, 152.24, 134.11, 130.87, 126.75, 60.22, 47.25, 46.87, 35.13,

31.47, 26.73, 24.72; IR (KBr, ν / cm^{-1}) 2965, 1739, 1649, 1489, 1422, 833; HRMS (ESI^+) Calcd for $C_{26}H_{35}NNaOS_2^+$ ($M+Na^+$) 464.2052, Found 464.2050.



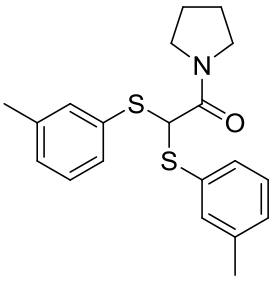
2,2-bis((3-bromophenyl)thio)-1-(pyrrolidin-1-yl)ethanone (3h)

29.6 mg, 61%, colorless oil; ^1H NMR (400 MHz, Acetone) δ 7.68 (t, *J* = 1.6 Hz, 2H), 7.54 – 7.48 (m, 4H), 7.30 (t, *J* = 7.9 Hz, 2H), 5.61 (s, 1H), 3.65 (t, *J* = 6.7 Hz, 2H), 3.37 (t, *J* = 6.8 Hz, 2H), 1.97 – 1.91 (m, 2H), 1.86 (dd, *J* = 13.5, 6.4 Hz, 2H); ^{13}C NMR (100 MHz, Acetone) δ 164.85, 136.19, 135.99, 132.85, 132.00, 131.38, 122.80, 59.48, 47.38, 47.09, 26.79, 24.78; IR (KBr, ν / cm^{-1}) 2968, 1640, 1584, 1469, 1412, 811, 689; HRMS (ESI^+) Calcd for $C_{18}H_{17}Br_2NNaOS_2^+$ ($M+Na^+$) 507.9011, Found 507.9005.



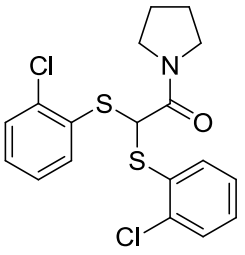
1-(pyrrolidin-1-yl)-2,2-bis(m-tolylthio)ethanone (3i)

27.5 mg, 77%, colorless oil; ^1H NMR (400 MHz, Acetone) δ 7.32 (d, *J* = 8.3 Hz, 4H), 7.22 (t, *J* = 7.5 Hz, 2H), 7.14 (d, *J* = 7.5 Hz, 2H), 5.32 (s, 1H), 3.47 (t, *J* = 6.5 Hz, 2H), 3.34 (t, *J* = 6.7 Hz, 2H), 2.30 (s, 6H), 1.88 – 1.77 (m, 4H); ^{13}C NMR (100 MHz, Acetone) δ 165.74, 139.45, 134.49, 134.02, 131.00, 129.73, 129.57, 60.05, 47.32, 46.94, 26.77, 24.76, 21.20; IR (KBr, ν / cm^{-1}) 2973, 1648, 1593, 1474, 1422, 779, 691; HRMS (ESI^+) Calcd for $C_{20}H_{23}NNaOS_2^+$ ($M+Na^+$) 380.1113, Found 380.1112.



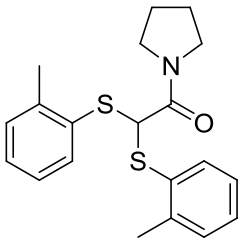
2,2-bis((2-chlorophenyl)thio)-1-(pyrrolidin-1-yl)ethanone (3j)

33.0 mg, 83%, colorless oil; ^1H NMR (400 MHz, Acetone) δ 7.75 – 7.70 (m, 2H), 7.49 – 7.45 (m, 2H), 7.36 – 7.31 (m, 4H), 5.57 (s, 1H), 3.55 (t, *J* = 6.7 Hz, 2H), 3.35 (t, *J* = 6.8 Hz, 2H), 1.93 – 1.86 (m, 2H), 1.82 (dd, *J* = 13.4, 6.4 Hz, 2H); ^{13}C NMR (100 MHz, Acetone) δ 164.73, 137.17, 135.71, 132.75, 130.64, 130.50, 128.33, 56.87, 47.40, 47.11, 26.77, 24.75; IR (KBr, ν / cm^{-1}) 2974, 1646, 1452, 1426, 1034, 749; HRMS (ESI^+) Calcd for



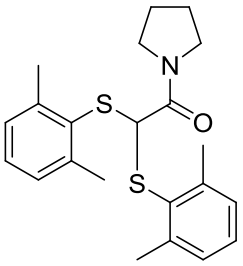
$C_{18}H_{17}Cl_2NNaOS_2^+$ (M+Na^+) 420.0021, Found 420.0031.

1-(pyrrolidin-1-yl)-2,2-bis(o-tolylthio)ethanone (3k)



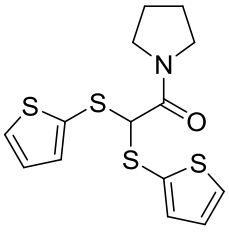
30.7 mg, 86%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.55 (d, *J* = 7.4 Hz, 2H), 7.24 (d, *J* = 5.6 Hz, 3H), 7.19 (dd, *J* = 13.8, 6.1 Hz, 3H), 5.17 (s, 1H), 3.35 – 3.27 (m, 4H), 2.36 (s, 6H), 1.78 (dd, *J* = 9.1, 5.7 Hz, 4H); ^{13}C NMR (100 MHz, Acetone) δ 166.04, 141.48, 134.84, 133.58, 131.17, 129.26, 127.34, 58.59, 47.26, 46.99, 26.77, 24.70, 20.78; IR (KBr, ν / cm^{-1}) 2973, 1644, 1422, 751; HRMS (ESI$^+$) Calcd for $C_{20}H_{23}NNaOS_2^+$ (M+Na^+) 380.1113, Found 380.1106.

2,2-bis((2,6-dimethylphenyl)thio)-1-(pyrrolidin-1-yl)ethanone (3l)



24.3 mg, 63%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.19 (dd, *J* = 8.7, 6.0 Hz, 2H), 7.14 (d, *J* = 6.8 Hz, 4H), 4.46 (s, 1H), 3.32 (t, *J* = 6.9 Hz, 2H), 2.66 (s, 2H), 2.38 (s, 12H), 1.73 – 1.66 (m, 2H), 1.60 – 1.54 (m, 2H); ^{13}C NMR (100 MHz, Acetone) δ 166.43, 143.74, 131.83, 129.47, 128.27, 58.08, 54.08, 46.17, 25.95, 23.82, 20.99; IR (KBr, ν / cm^{-1}) 2974, 1643, 1461, 1418, 775; HRMS (ESI$^+$) Calcd for $C_{22}H_{27}NNaOS_2^+$ (M+Na^+) 408.1426, Found 408.1422.

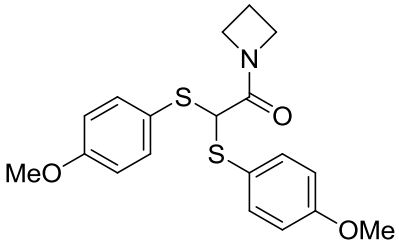
1-(pyrrolidin-1-yl)-2,2-bis(thiophen-2-ylthio)ethanone (3m)



28.3 mg, 83%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.64 (d, *J* = 5.3 Hz, 2H), 7.33 – 7.27 (m, 2H), 7.09 (dd, *J* = 5.3, 3.6 Hz, 2H), 5.05 (s, 1H), 3.51 (t, *J* = 6.6 Hz, 2H), 3.35 (t, *J* = 6.8 Hz, 2H), 1.94 – 1.88 (m, 2H), 1.87 – 1.81 (m, 2H); ^{13}C NMR (100 MHz, Acetone) δ 164.80, 137.42, 132.64, 130.82, 128.55, 65.41, 47.32, 46.93, 26.75, 24.76; IR (KBr, ν / cm^{-1}) 3083, 2972, 1702, 1648, 1426, 1402, 1249, 850, 710; HRMS (ESI$^+$) Calcd for $C_{14}H_{15}NNaOS_4^+$ (M+Na^+) 363.9929, Found 363.9937.

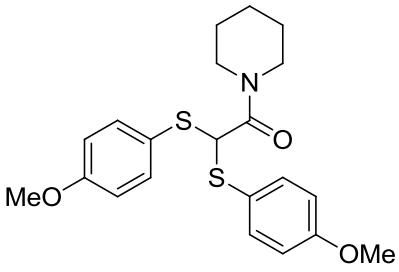
1-(azetidin-1-yl)-2,2-bis((4-methoxyphenyl)thio)ethanone (4a)

30.0 mg, 80%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.47 (d, *J* = 8.5 Hz, 4H), 6.93 (d, *J* = 8.7 Hz, 4H), 4.76 (s, 1H), 4.02 (t, *J* = 7.6 Hz, 2H), 3.91 – 3.86 (m, 2H), 3.81 (s, 6H), 2.20 – 2.13 (m, 2H); ^{13}C NMR (100 MHz, Acetone) δ 167.12, 161.29, 136.95, 124.31, 115.31, 59.00, 55.67, 51.30, 48.80, 15.84; IR (KBr, ν / cm^{-1}) 2958, 1651, 1592, 1029, 1433, 1251, 833, 734; HRMS (ESI^{+}) Calcd for $C_{19}H_{21}NNaO_3S_2^{+}$ (M+Na^{+}) 398.0855, Found 398.0850.



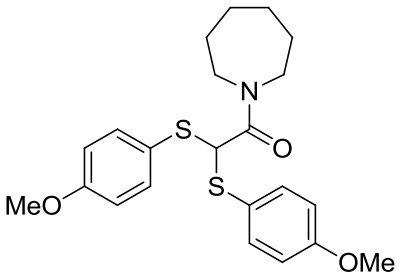
2,2-bis((4-methoxyphenyl)thio)-1-(piperidin-1-yl)ethanone (4b)

35.1 mg, 87%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.46 (d, *J* = 8.6 Hz, 4H), 6.91 (d, *J* = 8.6 Hz, 4H), 5.27 (s, 1H), 3.80 (s, 6H), 3.47 (d, *J* = 3.8 Hz, 4H), 1.62 (d, *J* = 4.9 Hz, 2H), 1.51 (dd, *J* = 11.7, 5.3 Hz, 4H; ^{13}C NMR (100 MHz, Acetone) δ 166.06, 161.21, 137.02, 124.37, 115.23, 61.36, 55.63, 48.02, 43.69, 26.97, 26.32, 24.94; IR (KBr, ν / cm^{-1}) 2941, 1644, 1592, 1495, 1254, 1029, 833, 724; HRMS (ESI^{+}) Calcd for $C_{21}H_{25}NNaO_3S_2^{+}$ (M+Na^{+}) 426.1168, Found 426.1161.



1-(azepan-1-yl)-2,2-bis((4-methoxyphenyl)thio)ethanone (4c)

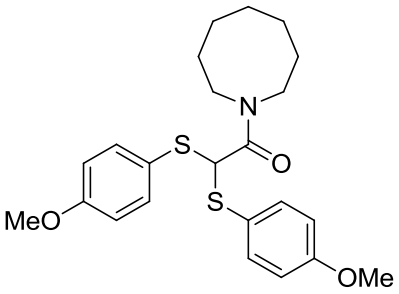
37.1 mg, 89%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.48 – 7.43 (m, 4H), 6.93 – 6.88 (m, 4H), 5.15 (s, 1H), 3.80 (s, 6H), 3.44 (dd, *J* = 12.3, 6.6 Hz, 4H), 1.63 (d, *J* = 4.7 Hz, 4H), 1.50 (d, *J* = 2.8 Hz, 4H); ^{13}C NMR (100 MHz, Acetone) δ 167.49, 161.27, 137.13, 124.45, 115.24, 61.03, 55.64, 48.74, 46.75, 28.22, 27.66, 27.07; IR (KBr, ν / cm^{-1}) 2933, 1638, 1592, 1493, 1249, 833, 736; HRMS (ESI^{+}) Calcd for $C_{22}H_{27}NNaO_3S_2^{+}$ (M+Na^{+}) 440.1325, Found 440.1331.



1-(azocan-1-yl)-2,2-bis((4-methoxyphenyl)thio)ethanone (4d)

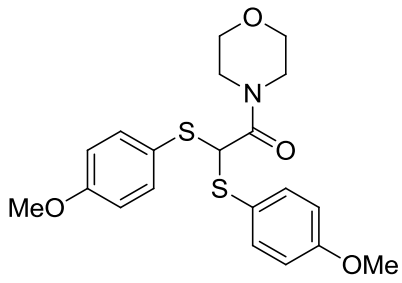
36.6 mg, 85%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.45 (d, *J* = 8.8 Hz, 4H), 6.90 (d, *J* = 8.8 Hz, 4H), 5.12 (s, 1H), 3.81 (s, 6H), 3.39 (dd, *J* = 11.8, 6.0 Hz, 4H), 1.64 (dd, *J* = 10.9, 4.5 Hz, 4H), 1.55 (d, *J* = 3.4 Hz, 2H), 1.46 (s, 4H); ^{13}C NMR (100 MHz, Acetone) δ 167.64,

161.31, 137.12, 124.54, 115.28, 61.22, 55.66, 49.95, 48.17, 28.52, 27.53, 26.76, 26.27, 25.67; IR (KBr, ν / cm^{-1}) 2928, 1642, 1592, 1493, 1249, 1031, 833, 799; HRMS (ESI^+) Calcd for $C_{23}H_{29}NNaO_3S_2^+$ ($M+Na^+$) 454.1481, Found 454.1486.



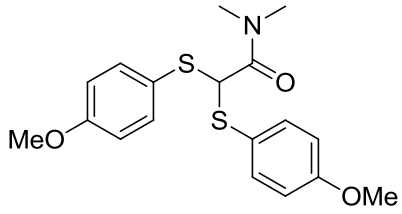
2,2-bis((4-methoxyphenyl)thio)-1-morpholinoethanone (4e)

31.6 mg, 78%, colorless oil; ^1H NMR (400 MHz, Acetone) δ 7.48 (d, *J* = 8.7 Hz, 4H), 6.92 (d, *J* = 8.7 Hz, 4H), 5.31 (s, 1H), 3.81 (s, 6H), 3.57 (s, 2H), 3.54 (s, 4H), 3.48 (s, 2H); ^{13}C NMR (100 MHz, Acetone) δ 166.09, 160.88, 136.73, 123.55, 114.84, 66.71, 66.60, 60.17, 60.04, 55.21, 47.14, 42.70; IR (KBr, ν / cm^{-1}) 2963, 1642, 1591, 1493, 1249, 1180, 829; HRMS (ESI^+) Calcd for $C_{20}H_{23}NNaO_4S_2^+$ ($M+Na^+$) 428.0961, Found 428.0956.



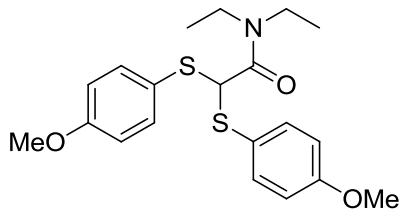
2,2-bis((4-methoxyphenyl)thio)-N,N-dimethylacetamide (4f)

29.4 mg, 81%, colorless oil; ^1H NMR (400 MHz, Acetone) δ 7.45 (d, *J* = 8.7 Hz, 4H), 6.91 (d, *J* = 8.7 Hz, 4H), 5.26 (s, 1H), 3.81 (s, 6H), 3.03 (s, 3H), 2.86 (s, 3H); ^{13}C NMR (100 MHz, Acetone) δ 167.68, 161.30, 137.21, 124.28, 115.24, 61.23, 55.64, 37.90, 35.96; IR (KBr, ν / cm^{-1}) 2940, 1644, 1593, 1493, 1243, 1023, 833, 798; HRMS (ESI^+) Calcd for $C_{18}H_{21}NNaO_3S_2^+$ ($M+Na^+$) 386.0855, Found 386.0849.

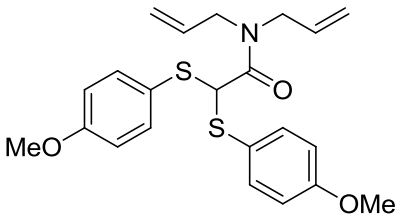


N,N-diethyl-2,2-bis((4-methoxyphenyl)thio)acetamide (4g)

33.6 mg, 86%, colorless oil; ^1H NMR (400 MHz, Acetone) δ 7.47 (d, *J* = 8.7 Hz, 4H), 6.91 (d, *J* = 8.7 Hz, 4H), 5.08 (s, 1H), 3.81 (s, 6H), 3.30 (dd, *J* = 6.9, 4.9 Hz, 4H), 1.09 (t, *J* = 7.1 Hz, 3H), 1.03 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, Acetone) δ 167.17, 161.32, 137.21, 124.37, 115.26, 60.84, 55.65, 42.93, 41.18, 14.94, 13.17; IR (KBr, ν / cm^{-1}) 2973, 1646, 1593, 1495, 1250, 1030, 833; HRMS (ESI^+) Calcd for $C_{20}H_{25}NNaO_3S_2^+$ ($M+Na^+$) 414.1168, Found 414.1164.

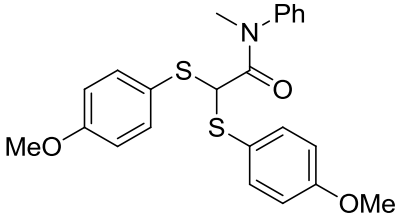


N,N-diallyl-2,2-bis((4-methoxyphenyl)thio)acetamide (4h)



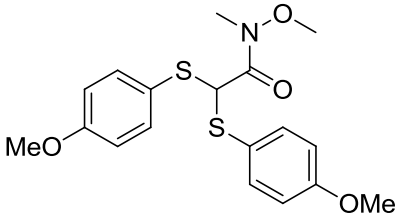
36.9 mg, 89%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.45 (d, *J* = 8.7 Hz, 4H), 6.91 (d, *J* = 8.7 Hz, 4H), 5.77 (qdd, *J* = 11.8, 10.1, 5.4 Hz, 2H), 5.23 – 5.08 (m, 4H), 5.03 (s, 1H), 3.94 – 3.86 (m, 4H), 3.81 (s, 6H); ^{13}C NMR (100 MHz, Acetone) δ 168.10, 161.39, 137.34, 134.63, 134.13, 124.05, 117.71, 116.71, 115.27, 61.03, 55.65, 50.09, 49.00; IR (KBr, ν / cm^{-1}) 2941, 1653, 1593, 1494, 1250, 1030, 831; HRMS (ESI^{+}) Calcd for $C_{22}H_{25}NNaO_3S_2^{+}$ (M+Na^{+}) 438.1168, Found 438.1171.

2,2-bis((4-methoxyphenyl)thio)-N-methyl-N-phenylacetamide (4i)



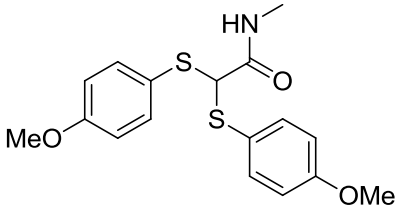
33.6 mg, 79%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.33 (d, *J* = 7.4 Hz, 3H), 7.24 (d, *J* = 8.6 Hz, 4H), 6.98 (d, *J* = 6.6 Hz, 2H), 6.84 (d, *J* = 8.6 Hz, 4H), 4.62 (s, 1H), 3.79 (s, 6H), 3.19 (s, 3H); ^{13}C NMR (100 MHz, Acetone) δ 168.23, 161.27, 143.97, 136.82, 130.39, 128.79, 128.00, 124.41, 115.26, 60.70, 55.65, 37.56; IR (KBr, ν / cm^{-1}) 2941, 1661, 1593, 1495, 1288, 1251, 1030, 833,701; HRMS (ESI^{+}) Calcd for $C_{23}H_{23}NNaO_3S_2^{+}$ (M+Na^{+}) 448.1012, Found 448.1009.

N-methoxy-2,2-bis((4-methoxyphenyl)thio)-N-methylacetamide (4j)



25.4 mg, 67%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.45 (d, *J* = 8.7 Hz, 4H), 6.91 (d, *J* = 8.7 Hz, 4H), 5.23 (s, 1H), 3.81 (s, 6H), 3.61 (s, 3H), 3.13 (s, 3H); ^{13}C NMR (100 MHz, Acetone) δ 169.43, 161.37, 137.02, 124.40, 115.35, 68.28, 61.99, 59.06, 55.66; IR (KBr, ν / cm^{-1}) 2969, 1739, 1592, 1493, 1247, 1030, 799, 680; HRMS (ESI^{+}) Calcd for $C_{18}H_{21}NNaO_4S_2^{+}$ (M+Na^{+}) 402.0804, Found 402.0809.

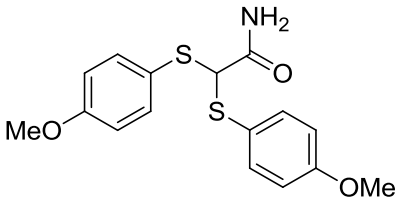
2,2-bis((4-methoxyphenyl)thio)-N-methylacetamide (4k)



22.3 mg, 64%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.42 (d, *J* = 8.8 Hz, 4H), 7.21 (s, 1H), 6.91 (d, *J* = 8.8 Hz,

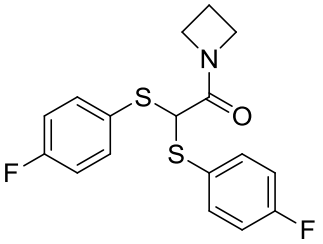
4H), 4.74 (s, 1H), 3.80 (s, 6H), 2.68 (d, *J* = 4.7 Hz, 3H); ^{13}C NMR (100 MHz, Acetone) δ 168.60, 161.21, 136.46, 124.67, 115.34, 62.23, 55.65, 26.49; IR (KBr, ν / cm^{-1}) 3472, 2952, 1649, 1591, 1495, 1247, 827; HRMS (ESI$^+$) Calcd for $C_{17}H_{19}NNaO_3S_2^+$ (M+Na$^+$) 372.0699, Found 372.0701.

2,2-bis((4-methoxyphenyl)thio)acetamide (4l)



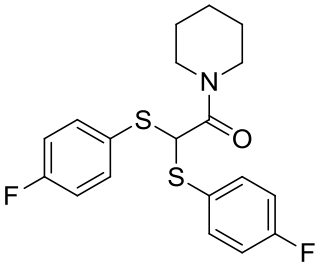
10.4 mg, 31%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.45 (d, *J* = 8.8 Hz, 4H), 6.91 (d, *J* = 8.8 Hz, 4H), 4.77 (s, 1H), 3.81 (s, 6H); ^{13}C NMR (100 MHz, DMSO) δ 169.14, 159.57, 135.05, 123.44, 114.58, 60.43, 55.23; IR (KBr, ν / cm^{-1}) 3401, 1655, 1051, 1026, 826, 764; HRMS (ESI$^+$) Calcd for $C_{16}H_{17}NNaO_3S_2^+$ (M+Na$^+$) 358.0542, Found 358.0538.

1-(azetidin-1-yl)-2,2-bis((4-fluorophenyl)thio)ethanone (4m)



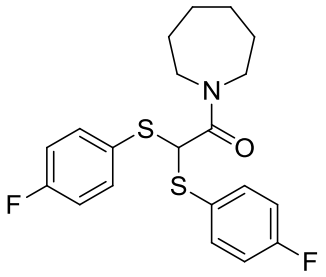
25.6 mg, 73%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.63 – 7.56 (m, 4H), 7.15 (t, *J* = 8.8 Hz, 4H), 5.06 (s, 1H), 4.19 (t, *J* = 7.6 Hz, 2H), 3.92 (t, *J* = 7.7 Hz, 2H), 2.28 – 2.20 (m, 2H); ^{13}C NMR (100 MHz, Acetone) δ 166.48, 165.13, 162.68, 137.18, 137.10, 129.10, 129.07, 116.86, 116.64, 57.96, 51.53, 48.98, 15.94; IR (KBr, ν / cm^{-1}) 2958, 1649, 1590, 1489, 1224, 835, 718; HRMS (ESI$^+$) Calcd for $C_{17}H_{15}F_2NNaOS_2^+$ (M+Na$^+$) 374.0455, Found 374.0461.

2,2-bis((4-fluorophenyl)thio)-1-(piperidin-1-yl)ethanone (4n)



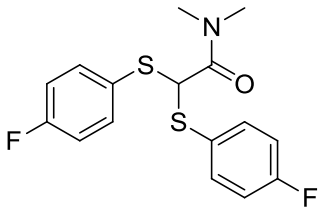
34.5 mg, 91%, colorless oil;^{1}H NMR (400 MHz, Acetone) δ 7.63 – 7.56 (m, 4H), 7.13 (t, *J* = 8.8 Hz, 4H), 5.58 (s, 1H), 3.58 – 3.53 (m, 2H), 3.49 – 3.44 (m, 2H), 1.65 – 1.56 (m, 4H), 1.50 (d, *J* = 4.1 Hz, 2H); ^{13}C NMR (100 MHz, Acetone) δ 165.45, 165.06, 162.61, 137.28, 137.20, 129.08, 129.05, 116.71, 116.50, 60.14, 48.02, 43.75, 27.00, 26.33, 24.89; IR (KBr, ν / cm^{-1}) 2943, 1638, 1590, 1489, 1221, 835; HRMS (ESI$^+$) Calcd for $C_{19}H_{19}F_2NNaOS_2^+$ (M+Na$^+$) 402.0768, Found 402.0760.

1-(azepan-1-yl)-2,2-bis((4-fluorophenyl)thio)ethanone (4o)



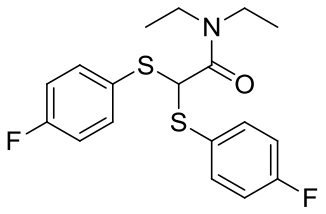
34.9 mg, 89%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.59 (dd, *J* = 8.6, 5.5 Hz, 4H), 7.12 (t, *J* = 8.8 Hz, 4H), 5.44 (s, 1H), 3.57 (t, *J* = 6.1 Hz, 2H), 3.47 – 3.42 (m, 2H), 1.70 (dd, *J* = 10.8, 5.2 Hz, 2H), 1.62 (dd, *J* = 10.9, 5.3 Hz, 2H), 1.52 (dd, *J* = 5.8, 3.3 Hz, 4H); ^{13}C NMR (100 MHz, Acetone) δ 166.83, 165.07, 162.61, 137.34, 137.26, 129.11, 129.07, 116.72, 116.50, 59.83, 48.82, 46.80, 28.16, 27.78, 27.03; IR (KBr, ν / cm^{-1}) 2932, 1642, 1590, 1489, 1228, 1090, 837; HRMS (ESI^+) Calcd for $C_{20}H_{21}F_2NNaOS_2^+$ ($M+Na^+$) 416.0925, Found 416.0917.

2,2-bis((4-fluorophenyl)thio)-N,N-dimethylacetamide (4p)



27.5 mg, 81%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.61 – 7.56 (m, 4H), 7.13 (t, *J* = 8.8 Hz, 4H), 5.55 (s, 1H), 3.13 (s, 3H), 2.88 (s, 3H); ^{13}C NMR (100 MHz, Acetone) δ 167.01, 165.09, 162.64, 137.45, 137.36, 128.91, 128.88, 116.71, 116.49, 60.21, 37.93, 36.03; IR (KBr, ν / cm^{-1}) 2935, 1648, 1590, 1489, 1224, 834; HRMS (ESI^+) Calcd for $C_{16}H_{15}F_2NNaOS_2^+$ ($M+Na^+$) 362.0455, Found 362.050.

N,N-diethyl-2,2-bis((4-fluorophenyl)thio)acetamide (4q)

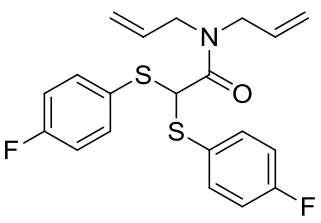


31.2 mg, 85%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.61 (dd, *J* = 8.5, 5.5 Hz, 4H), 7.13 (t, *J* = 8.8 Hz, 4H), 5.38 (s, 1H), 3.41 (q, *J* = 7.1 Hz, 2H), 3.31 (q, *J* = 7.1 Hz, 2H), 1.15 (t, *J* = 7.1 Hz, 3H), 1.03 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, Acetone) δ 166.43, 165.13, 162.67, 137.47, 137.38, 129.02, 128.99, 116.73, 116.51, 59.69, 43.03, 41.23, 14.91, 13.14; IR (KBr, ν / cm^{-1}) 2976, 1644, 1590, 1491, 1223, 837; HRMS (ESI^+) Calcd for $C_{18}H_{19}F_2NNaOS_2^+$ ($M+Na^+$) 390.0768, Found 390.0762.

N,N-diallyl-2,2-bis((4-fluorophenyl)thio)acetamide (4r)

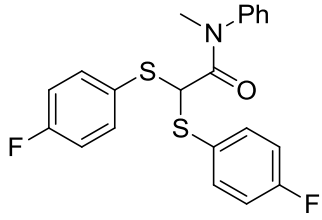
34.4 mg, 88%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.62 – 7.53 (m, 4H), 7.14 (t, *J* = 8.8 Hz, 4H), 5.87 (ddd, *J* = 22.0, 10.1, 4.9 Hz, 1H), 5.74 (ddt, *J* = 16.2, 10.3, 6.0 Hz, 1H), 5.30

(s, 1H), 5.21 – 5.09 (m, 4H), 4.01 (d, *J* = 4.7 Hz, 2H), 3.93 (d, *J* = 5.9 Hz, 2H); ^{13}C NMR (100 MHz, Acetone) δ 167.45, 165.24, 162.78, 137.63, 137.54, 134.68, 133.96, 128.78, 128.75, 117.89, 116.88, 116.81, 116.59, 59.88, 50.29, 49.07; IR (KBr, ν / cm^{-1}) 2983, 1655, 1590, 1489, 1226, 1157, 835; HRMS (ESI$^+$) Calcd for $C_{20}H_{19}F_2NNaOS_2^+$ (M+Na$^+$) 414.0768, Found 414.0773.



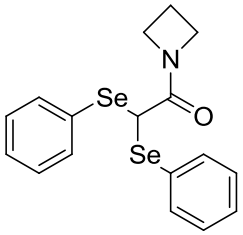
2,2-bis((4-fluorophenyl)thio)-N-methyl-N-phenylacetamide (4s)

34.9 mg, 87%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.41 – 7.32 (m, 7H), 7.13 (d, *J* = 7.1 Hz, 2H), 7.06 (t, *J* = 8.7 Hz, 4H), 4.76 (s, 1H), 3.22 (s, 3H); ^{13}C NMR (100 MHz, Acetone) δ 167.64, 165.15, 162.69, 143.89, 137.11, 137.03, 130.62, 129.30, 129.27, 129.09, 128.09, 116.92, 116.70, 60.25, 37.66; IR (KBr, ν / cm^{-1}) 3066, 1661, 1590, 1491, 1230, 835, 701; HRMS (ESI$^+$) Calcd for $C_{21}H_{17}F_2NNaOS_2^+$ (M+Na$^+$) 424.0612, Found 424.0601.



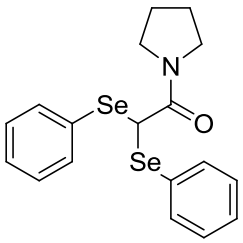
1-(azetidin-1-yl)-2,2-bis(phenylselanyl)ethanone (4t)

34.9 mg, 85%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.64 (dd, *J* = 7.8, 1.4 Hz, 4H), 7.39 – 7.33 (m, 6H), 5.07 (s, 1H), 3.86 (d, *J* = 7.6 Hz, 2H), 3.83 (d, *J* = 7.7 Hz, 2H), 2.12 – 2.06 (m, 2H); ^{13}C NMR (100 MHz, Acetone) δ 168.61, 135.73, 130.43, 129.93, 129.33, 51.24, 48.84, 39.75, 15.61; IR (KBr, ν / cm^{-1}) 2954, 1646, 1435, 1023, 738, 691; HRMS (ESI$^+$) Calcd for $C_{17}H_{17}NNaOSe_2^+$ (M+Na$^+$) 433.9533, Found 433.9529.

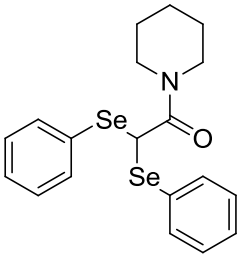


2,2-bis(phenylselanyl)-1-(pyrrolidin-1-yl)ethanone (4u)

38.2 mg, 90%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.63 (dd, *J* = 7.9, 1.3 Hz, 4H), 7.40 – 7.29 (m, 6H), 5.36 (s, 1H), 3.30 (t, *J* = 6.8 Hz, 2H), 3.24 (t, *J* = 6.6 Hz, 2H), 1.73 (d, *J* = 3.0 Hz, 4H); ^{13}C NMR (100 MHz, Acetone) δ 166.59, 135.10, 129.55, 129.04, 128.45, 46.62, 45.99, 42.67, 25.84, 23.94; IR (KBr, ν / cm^{-1}) 2973, 1638, 1437, 1247, 740, 670; HRMS (ESI$^+$) Calcd for $C_{18}H_{19}NNaOSe_2^+$ (M+Na$^+$) 447.9689, Found 447.9683.

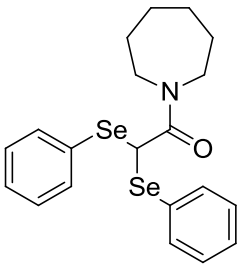


2,2-bis(phenylselanyl)-1-(piperidin-1-yl)ethanone (4v)



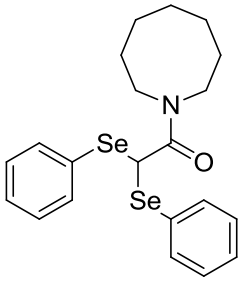
38.2 mg, 87%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.61 (dd, *J* = 8.0, 1.4 Hz, 4H), 7.38 – 7.29 (m, 6H), 5.65 (s, 1H), 3.45 (s, 2H), 3.34 (s, 2H), 1.57 (dd, *J* = 11.0, 5.6 Hz, 2H), 1.44 (s, 4H); ^{13}C NMR (100 MHz, Acetone) δ 167.67, 135.92, 130.47, 129.82, 129.21, 48.29, 43.76, 42.90, 26.85, 26.27, 24.86; IR (KBr, ν / cm^{-1}) 2939, 1627, 1437, 1258, 1021, 738, 690; HRMS (ESI^{+}) Calcd for $C_{19}H_{21}NNaOSe_2^{+}$ ($M+Na^{+}$) 461.9846, Found 461.9843.

1-(azepan-1-yl)-2,2-bis(phenylselanyl)ethanone (4w)



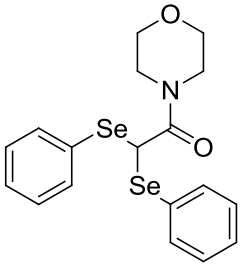
37.1 mg, 82%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.10 (d, *J* = 6.4 Hz, 4H), 6.92 – 6.85 (m, 6H), 5.11 (s, 1H), 2.87 (d, *J* = 3.7 Hz, 4H), 1.08 (s, 4H), 0.97 (s, 4H); ^{13}C NMR (100 MHz, DMSO) δ 168.34, 135.08, 129.56, 128.87, 60.23, 48.43, 46.19, 28.88, 27.53, 27.14, 26.38; IR (KBr, ν / cm^{-1}) 2956, 1645, 1590, 1489, 1226, 1090, 833; HRMS (ESI^{+}) Calcd for $C_{20}H_{23}NNaOSe_2^{+}$ ($M+Na^{+}$) 476.0002, Found 476.0011.

1-(azocan-1-yl)-2,2-bis(phenylselanyl)ethanone (4x)



39.2 mg, 84%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.60 (d, *J* = 6.8 Hz, 4H), 7.33 (dt, *J* = 14.3, 7.0 Hz, 6H), 5.45 (s, 1H), 3.40 – 3.35 (m, 2H), 3.28 – 3.23 (m, 2H), 1.66 (s, 2H), 1.55 (dd, *J* = 12.3, 7.1 Hz, 4H), 1.49 (s, 4H); ^{13}C NMR (100 MHz, Acetone) δ 169.22, 135.98, 130.48, 129.88, 129.33, 50.20, 48.10, 43.04, 28.34, 27.56, 26.76, 26.23, 25.64; IR (KBr, ν / cm^{-1}) 2942, 1651, 1590, 1489, 1228, 1075, 840; HRMS (ESI^{+}) Calcd for $C_{21}H_{25}NNaOSe_2^{+}$ ($M+Na^{+}$) 490.0159, Found 490.0155.

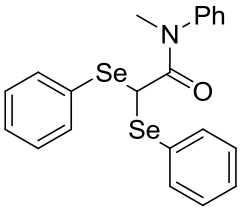
1-morpholino-2,2-bis(phenylselanyl)ethanone (4y)



34.0 mg, 77%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.64 (d, *J* = 6.7 Hz, 4H), 7.35 (dt, *J* = 14.1, 6.8 Hz, 6H), 5.67 (s, 1H), 3.54 (s, 2H), 3.43 (d, *J* = 20.9 Hz, 6H); ^{13}C NMR (100 MHz, Acetone) δ 168.17, 136.05, 130.18, 129.89, 129.36, 67.07, 66.86, 47.76, 43.20, 41.89; IR

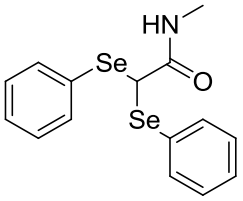
(KBr, ν / cm^{-1}) 2965, 1633, 1437, 1277, 1116, 1032, 740, 691; HRMS (ESI^+) Calcd for $C_{18}H_{19}NNaO_2Se_2^+$ ($M+Na^+$) 463.9638, Found 463.9646.

N-methyl-N-phenyl-2,2-bis(phenylselanyl)acetamide (4z)



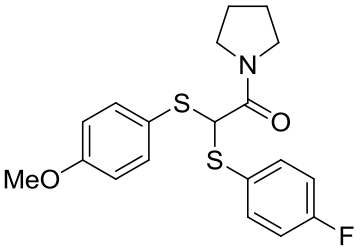
41.0 mg, 89%, colorless oil; ^1H NMR (400 MHz, Acetone) δ 7.37 (d, *J* = 7.3 Hz, 4H), 7.32 (d, *J* = 7.3 Hz, 2H), 7.25 (dd, *J* = 14.5, 6.6 Hz, 7H), 6.94 – 6.90 (m, 2H), 4.83 (s, 1H), 3.18 (s, 3H); ^{13}C NMR (100 MHz, Acetone) δ 169.66, 144.17, 135.64, 130.42, 130.38, 129.92, 129.37, 128.75, 127.77, 42.40, 37.67; IR (KBr, ν / cm^{-1}) 3058, 2937, 1653, 1497, 740, 691; HRMS (ESI^+) Calcd for $C_{21}H_{19}NNaOSe_2^+$ ($M+Na^+$) 483.9689, Found 483.9688.

N-methyl-2,2-bis(phenylselanyl)acetamide (4aa)



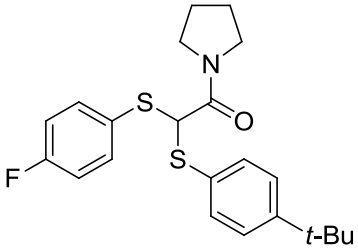
30.0 mg, 78%, colorless oil; ^1H NMR (400 MHz, DMSO) δ 8.08 (d, *J* = 4.2 Hz, 1H), 7.56 (dd, *J* = 6.2, 2.8 Hz, 4H), 7.40 – 7.34 (m, 6H), 5.19 (s, 1H), 2.58 (d, *J* = 4.6 Hz, 3H); ^{13}C NMR (100 MHz, DMSO) δ 169.12, 133.52, 129.83, 129.19, 128.13, 41.83, 25.92; IR (KBr, ν / cm^{-1}) 3439, 1661, 1008, 824, 762; HRMS (ESI^+) Calcd for $C_{15}H_{15}NNaOSe_2^+$ ($M+Na^+$) 407.9376, Found 407.9379.

2-((4-fluorophenyl)thio)-2-((4-methoxyphenyl)thio)-1-(pyrrolidin-1-yl)ethanone (5a)



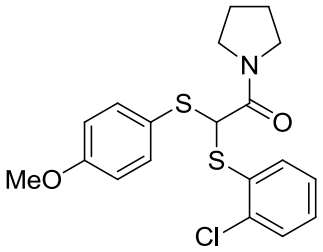
24.1 mg, 32%, colorless oil; ^1H NMR (400 MHz, Acetone) δ 7.62 – 7.54 (m, 2H), 7.46 (d, *J* = 8.8 Hz, 2H), 7.12 (t, *J* = 8.8 Hz, 2H), 6.91 (d, *J* = 8.8 Hz, 2H), 5.19 (s, 1H), 3.81 (s, 3H), 3.50 (t, *J* = 6.6 Hz, 2H), 3.33 (t, *J* = 6.8 Hz, 2H), 1.88 (dt, *J* = 12.7, 6.4 Hz, 2H), 1.80 (dd, *J* = 13.2, 6.7 Hz, 2H); ^{13}C NMR (100 MHz, Acetone) δ 165.56, 164.91, 162.46, 161.41, 137.39, 136.97, 136.89, 129.64, 129.60, 123.64, 116.70, 116.48, 115.25, 61.62, 55.65, 47.23, 46.87, 26.75, 24.75; IR (KBr, ν / cm^{-1}) 2973, 1644, 1590, 1493, 1251, 1029, 835; HRMS (ESI^+) Calcd for $C_{19}H_{20}FNNaO_2S_2^+$ ($M+Na^+$) 400.0812, Found 400.0821.

2-((4-(tert-butyl)phenyl)thio)-2-((4-fluorophenyl)thio)-1-(pyrrolidin-1-yl)ethanone (5b)



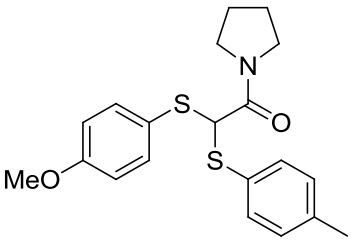
17.7 mg, 22%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.59 – 7.55 (m, 2H), 7.45 (d, *J* = 8.5 Hz, 2H), 7.39 (d, *J* = 8.5 Hz, 2H), 7.14 – 7.08 (m, 2H), 5.29 (s, 1H), 3.56 – 3.44 (m, 2H), 3.34 (t, *J* = 6.8 Hz, 2H), 1.90 – 1.84 (m, 2H), 1.84 – 1.78 (m, 2H), 1.31 (s, 9H); ^{13}C NMR (100 MHz, Acetone) δ 165.55, 165.07, 162.62, 152.26, 137.34, 137.26, 134.19, 130.56, 129.28, 129.24, 126.78, 116.70, 116.48, 60.85, 47.28, 46.91, 35.13, 31.45, 26.76, 24.77; IR (KBr, ν / cm^{-1}) 2965, 1648, 1590, 1489, 1224, 837; HRMS (ESI^{+}) Calcd for $C_{22}H_{26}FNNaOS_2^{+}$ (M+Na^{+}) 426.1332, Found 426.1339.

2-((2-chlorophenyl)thio)-2-((4-methoxyphenyl)thio)-1-(pyrrolidin-1-yl)ethanone (5c)



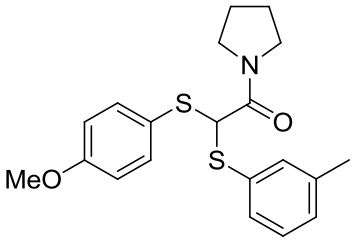
11.8 mg, 15%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.69 (dd, *J* = 7.4, 1.7 Hz, 1H), 7.46 (d, *J* = 8.7 Hz, 3H), 7.32 (dq, *J* = 7.4, 5.8 Hz, 2H), 6.90 (d, *J* = 8.7 Hz, 2H), 5.35 (s, 1H), 3.81 (s, 3H), 3.53 (td, *J* = 6.6, 3.8 Hz, 2H), 3.34 (td, *J* = 6.6, 3.8 Hz, 2H), 1.92 – 1.86 (m, 2H), 1.84 – 1.79 (m, 2H); ^{13}C NMR (100 MHz, Acetone) δ 165.23, 161.68, 137.79, 135.92, 134.19, 134.06, 130.55, 129.50, 128.30, 122.95, 115.27, 58.94, 55.67, 47.31, 46.96, 26.78, 24.77; IR (KBr, ν / cm^{-1}) 2971, 1646, 1592, 1493, 1426, 1250, 1034, 833, 751; HRMS (ESI^{+}) Calcd for $C_{19}H_{20}ClNNaO_2S_2^{+}$ (M+Na^{+}) 416.0516, Found 416.0509.

2-((4-methoxyphenyl)thio)-1-(pyrrolidin-1-yl)-2-(p-tolylthio)ethanone (5d)



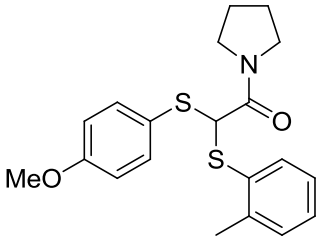
14.9 mg, 20%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.45 (d, *J* = 8.7 Hz, 2H), 7.40 (d, *J* = 8.1 Hz, 2H), 7.16 (d, *J* = 7.9 Hz, 2H), 6.90 (d, *J* = 8.7 Hz, 2H), 5.13 (s, 1H), 3.81 (s, 3H), 3.45 (t, *J* = 6.5 Hz, 2H), 3.33 (t, *J* = 6.7 Hz, 2H), 2.32 (s, 3H), 1.87 – 1.78 (m, 4H); ^{13}C NMR (100 MHz, Acetone) δ 165.85, 161.42, 139.00, 137.41, 134.30, 130.91, 130.44, 123.98, 115.23, 61.40, 55.66, 47.23, 46.86, 26.76, 24.76, 21.07; IR (KBr, ν / cm^{-1}) 2973, 1646, 1592, 1493, 1249, 1031, 835; HRMS (ESI^{+}) Calcd for $C_{20}H_{23}NNaO_2S_2^{+}$ (M+Na^{+}) 396.1062, Found 396.1057.

2-((4-methoxyphenyl)thio)-1-(pyrrolidin-1-yl)-2-(m-tolylthio)ethanone (5e)



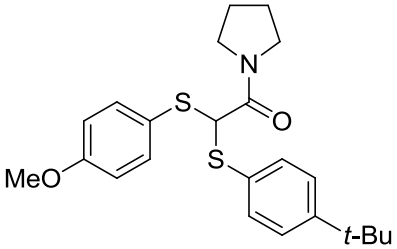
13.4 mg, 18%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.46 (d, *J* = 8.8 Hz, 2H), 7.30 (d, *J* = 12.0 Hz, 2H), 7.22 (t, *J* = 7.6 Hz, 1H), 7.12 (d, *J* = 7.5 Hz, 1H), 6.91 (d, *J* = 8.8 Hz, 2H), 5.20 (s, 1H), 3.81 (s, 3H), 3.46 (t, *J* = 6.6 Hz, 2H), 3.33 (t, *J* = 6.7 Hz, 2H), 2.30 (s, 3H), 1.89 – 1.83 (m, 2H), 1.83 – 1.77 (m, 2H); ^{13}C NMR (100 MHz, Acetone) δ 165.82, 161.49, 139.46, 137.58, 134.58, 133.91, 130.41, 129.59, 129.40, 123.82, 115.24, 60.96, 55.66, 47.27, 46.88, 26.77, 24.77, 21.20; IR (KBr, ν / cm^{-1}) 2961, 1703, 1648, 1593, 1249, 1030, 736; HRMS (ESI^{+}) Calcd for $C_{20}H_{23}NNaO_2S_2^{+}$ (M+Na^{+}) 396.1062, Found 396.1064.

2-((4-methoxyphenyl)thio)-1-(pyrrolidin-1-yl)-2-(o-tolylthio)ethanone (5f)



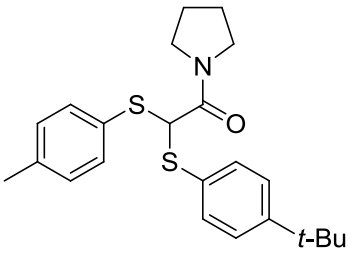
14,2 mg, 19%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.54 (d, *J* = 7.1 Hz, 1H), 7.46 (d, *J* = 8.8 Hz, 2H), 7.26 – 7.14 (m, 3H), 6.91 (d, *J* = 8.8 Hz, 2H), 5.12 (s, 1H), 3.81 (s, 3H), 3.46 – 3.38 (m, 1H), 3.37 – 3.24 (m, 3H), 2.36 (s, 3H), 1.87 – 1.71 (m, 4H); ^{13}C NMR (100 MHz, Acetone) δ 165.93, 161.45, 140.99, 137.28, 134.26, 133.93, 131.10, 128.84, 127.29, 124.00, 115.27, 60.03, 55.67, 47.22, 46.88, 26.74, 24.72, 20.77; IR (KBr, ν / cm^{-1}) 2967, 1646, 1592, 1493, 1249, 1031, 833, 751; HRMS (ESI^{+}) Calcd for $C_{20}H_{23}NNaO_2S_2^{+}$ (M+Na^{+}) 396.1062, Found 396.1061.

2-((4-(tert-butyl)phenyl)thio)-2-((4-methoxyphenyl)thio)-1-(pyrrolidin-1-yl)ethanone (5g)



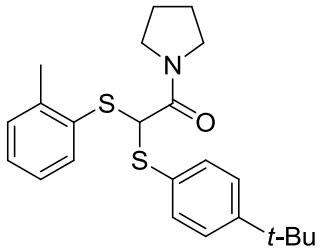
21.6 mg, 26%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.45 (d, *J* = 8.7 Hz, 4H), 7.39 (d, *J* = 8.5 Hz, 2H), 6.90 (d, *J* = 8.8 Hz, 2H), 5.15 (s, 1H), 3.81 (s, 3H), 3.42 (dt, *J* = 8.9, 6.5 Hz, 2H), 3.33 (t, *J* = 6.6 Hz, 2H), 1.86 – 1.78 (m, 4H), 1.31 (s, 9H); ^{13}C NMR (100 MHz, Acetone) δ 165.90, 161.40, 151.99, 137.37, 133.82, 131.15, 126.74, 123.97, 115.22, 61.17, 55.65, 47.24, 46.85, 35.10, 31.46, 26.74, 24.75; IR (KBr, ν / cm^{-1}) 2963, 1646, 1592, 1493, 1249, 835, 799; HRMS (ESI^{+}) Calcd for $C_{23}H_{29}NNaO_2S_2^{+}$ (M+Na^{+}) 438.1532, Found 438.1528.

2-((4-(tert-butyl)phenyl)thio)-1-(pyrrolidin-1-yl)-2-(p-tolylthio)ethanone (5h)



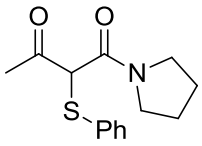
17.6 mg, 22%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.45 (d, *J* = 8.5 Hz, 2H), 7.43 – 7.35 (m, 4H), 7.15 (d, *J* = 7.9 Hz, 2H), 5.22 (s, 1H), 3.48 – 3.30 (m, 4H), 2.31 (s, 3H), 1.87 – 1.75 (m, 4H), 1.31 (s, 9H); ^{13}C NMR (100 MHz, Acetone) δ 165.78, 152.17, 139.16, 134.56, 134.16, 130.80, 130.53, 130.40, 126.71, 60.57, 47.23, 46.86, 35.10, 31.46, 26.73, 24.73, 21.11; IR (KBr, ν / cm^{-1}) 2965, 1648, 1491, 1422, 1120, 835; HRMS (ESI^{+}) Calcd for $C_{23}H_{29}NNaOS_2^{+}$ (M+Na^{+}) 422.1583, Found 422.1581.

2-((4-(tert-butyl)phenyl)thio)-1-(pyrrolidin-1-yl)-2-(o-tolylthio)ethanone (5i)



23.1 mg, 29%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.53 (d, *J* = 7.6 Hz, 1H), 7.46 (d, *J* = 8.4 Hz, 2H), 7.39 (d, *J* = 8.4 Hz, 2H), 7.23 – 7.15 (m, 3H), 5.21 (s, 1H), 3.32 (dt, *J* = 8.6, 6.2 Hz, 4H), 2.35 (s, 3H), 1.81 – 1.75 (m, 4H), 1.31 (s, 9H); ^{13}C NMR (100 MHz, Acetone) δ 165.93, 152.35, 141.34, 134.74, 134.15, 133.63, 131.11, 130.78, 129.10, 127.29, 126.77, 59.44, 47.24, 46.91, 35.13, 31.44, 26.73, 24.71, 20.81; IR (KBr, ν / cm^{-1}) 2963, 1648, 1420, 1269, 1120, 833, 744; HRMS (ESI^{+}) Calcd for $C_{23}H_{29}NNaOS_2^{+}$ (M+Na^{+}) 422.1583, Found 422.1579.

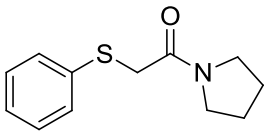
2-(phenylthio)-1-(pyrrolidin-1-yl)butane-1,3-dione (3ab)



19.7 mg, 75%, colorless oil; ^{1}H NMR (400 MHz, Acetone) δ 7.47 (d, *J* = 6.7 Hz, 2H), 7.36 – 7.28 (m, 3H), 4.88 (s, 1H), 3.51 (ddd, *J* = 17.0, 10.0, 5.1 Hz, 2H), 3.39 (t, *J* = 6.8 Hz, 2H), 2.29 (s, 3H), 1.93 – 1.80 (m, 4H); ^{13}C NMR (100 MHz, Acetone) δ 200.55, 164.17, 134.28, 132.56, 129.90, 128.50, 63.41, 47.52, 46.93, 26.71, 26.58, 24.74; IR (KBr, ν / cm^{-1}) 2973, 1709, 1644, 1424, 1170, 746, 691; HRMS (ESI^{+}) Calcd for $C_{14}H_{17}NNaO_2S^{+}$ (M+Na^{+}) 286.0872, Found 286.0870.

2-(phenylthio)-1-(pyrrolidin-1-yl)ethanone (7)

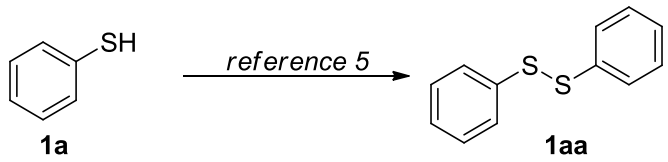
21.0 mg, 95%, colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 – 7.43 (m, 2H), 7.29 (t, *J* = 7.3 Hz, 2H), 7.21 (t, *J* = 7.3 Hz, 1H), 3.68 (s, 2H), 3.46 (dt, *J* = 11.2, 6.8 Hz, 4H), 1.93 (dt, *J* = 12.7,



6.6 Hz, 2H), 1.84 (dt, J = 13.4, 6.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.97, 135.49, 130.38, 129.08, 126.95, 47.02, 46.26, 38.03, 26.28, 24.42; IR (KBr, ν / cm^{-1}) 3434, 2971, 2948, 2878, 1630, 1452, 1438, 911, 727, 686; HRMS (ESI^+) Calcd for $C_{12}H_{15}NNaOS^+$ ($M+Na^+$) 244.0767, Found 244.0761.

Scheme S1. Mechanism study

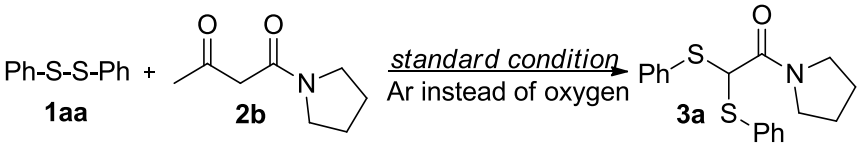
(a) Reaction in eq 1



(1)

According to previous reports[5], thiophenol 1a could be oxidized to 1,2-diphenyldisulfane **1aa** by oxygen under O_2 atmosphere.

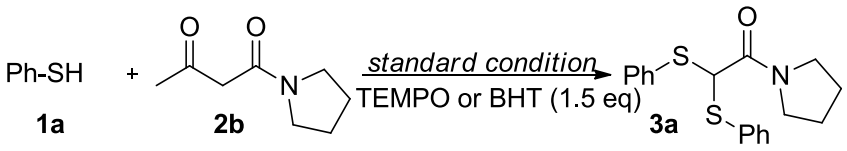
(b) Reaction in eq 2



(2)

A test tube equipped with a magnetic stir bar was charged with 1,2-diphenyldisulfane **1aa** (0.25 mmol), 1-(pyrrolidin-1-yl) butane-1,3-dione **2b** (0.10 mmol), NaOH (0.40 mmol) and *n*-PrCN (1 mL) under O_2 atmosphere. The resulting mixture was stirred for 5 min at room temperature, and then heated at 80 ℃ for 24 h. The reaction solution was cooled to ambient temperature, then quenched by saturated $NaHCO_3$ aqueous solution, and extracted with ethyl acetate (3*25mL), the combined organic extracts were concentrated and the resulting residue was purified by column chromatography on silica gel (hexane / EtOAc = 2:1) to give **3a** as colorless oil (yield: 89%).

(c) Reaction in eq 3

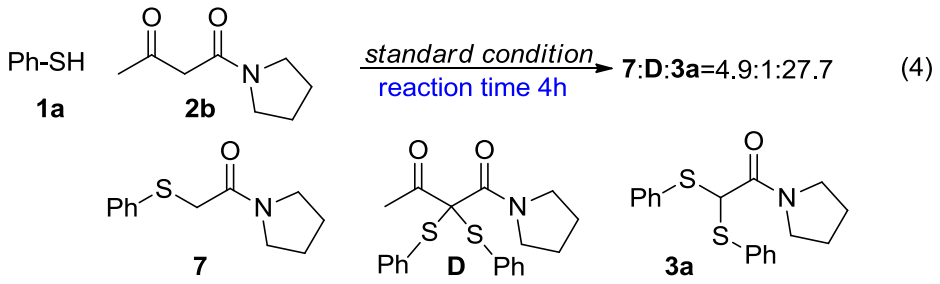


(3)

A test tube equipped with a magnetic stir bar was charged with thiophenol **1a** (0.25 mmol), 1-(pyrrolidin-1-yl) butane-1,3-dione **2b** (0.10 mmol), NaOH (0.40 mmol), TEMPO or BHT (0.15 mmol) and *n*-PrCN (1 mL) under O_2 atmosphere. The resulting mixture was stirred for 5 min at room temperature, and then heated at 80 ℃ for 24 h. The reaction solution was cooled to ambient temperature, then quenched by saturated $NaHCO_3$ aqueous solution, and extracted with

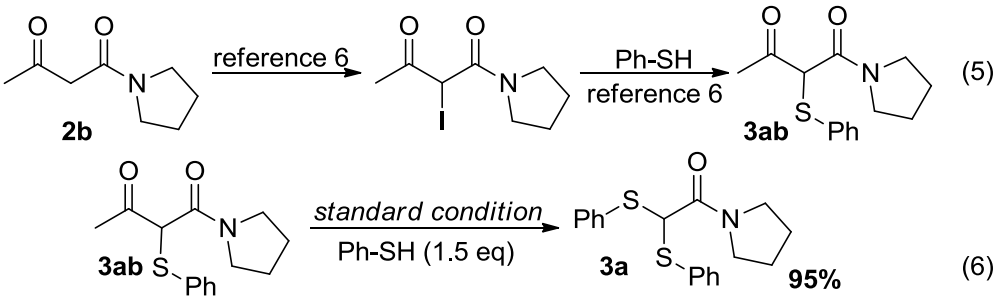
ethyl acetate (3*25mL), the combined organic extracts were concentrated and the resulting residue was purified by column chromatography on silica gel (hexane / EtOAc = 2:1) to give **3a** as colorless oil (yield: 81%).

(d) Reaction in eq 4



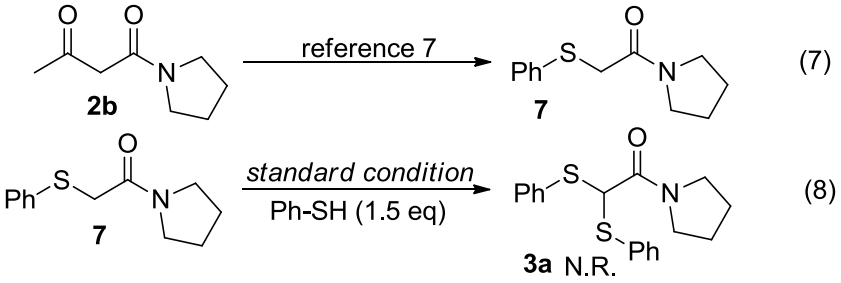
A test tube equipped with a magnetic stir bar was charged with thiophenol **1a** (0.25 mmol), 1-(pyrrolidin-1-yl) butane-1,3-dione **2b** (0.10 mmol), NaOH (0.40 mmol) and *n*-PrCN (1 mL) under O_2 atmosphere (**eq 4**). The resulting mixture was stirred for 5 min at room temperature, and then heated at 80 ℃ for 4 h. The reaction mixture was detected by LC-MS and the ratio of **7**:**D**:**3a** was 4.9:1:27.7.

(e) Reaction in eq 5 and 6



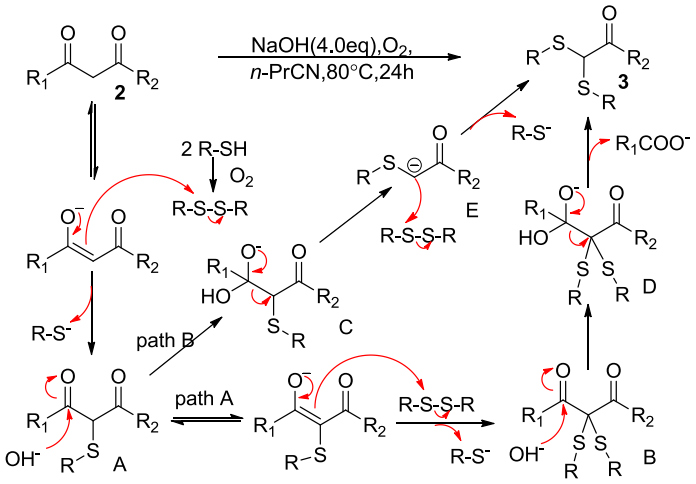
According to the previous report, we prepared 3ab[6] (**eq 5**). A test tube equipped with a magnetic stir bar was charged with thiophenol **1a** (0.15 mmol), 2-(phenylthio)-1-(pyrrolidin-1-yl)butane-1,3-dione **3ab** (0.10 mmol), NaOH (0.30 mmol), and *n*-PrCN (1 mL) under O_2 atmosphere (**eq 6**). The resulting mixture was stirred for 5 min at room temperature, and then heated at 80 ℃ for 24 h. The reaction solution was cooled to ambient temperature, then quenched by saturated $NaHCO_3$ aqueous solution, and extracted with ethyl acetate (3*25mL), the combined organic extracts were concentrated and the resulting residue was purified by column chromatography on silica gel (hexane / EtOAc = 2:1) to give **3a** as colorless oil (yield: 95%).

(f) Reaction in eq 7 and 8



According to our previous report, we prepared **7**[7] (**eq 7**). A test tube equipped with a magnetic stir bar was charged with thiophenol **1a** (0.15 mmol), 2-(phenylthio)-1-(pyrrolidin-1-yl)ethanone **7** (0.10 mmol), NaOH (0.30 mmol), and *n*-PrCN (1 mL) under O_2 atmosphere (**eq 8**). The resulting mixture was stirred for 5 min at room temperature, and then heated at 80 ℃ for 24 h. The reaction solution was cooled to ambient temperature. However, no desired product **3a** was detected.

A proposed mechanism.



The one way was that PhSH was oxidized to disulfide by oxygen, which reacted with enolate by twice nucleophilic attack to generate intermediate **D**. Next, OH-, as a nucleophilic specie, attacked the relatively electron deficient carbonyl group of intermediate **D**. As a consequence, the acetoxy was eliminated and released carboxylic anion. Another way, disulfide reacted with enolate to generate intermediate **A**, Whose carbonyl group was attacked by OH-, then forming carbanion intermediate **E**. Without being quenched, intermediate **E** was transformed into the desired product.

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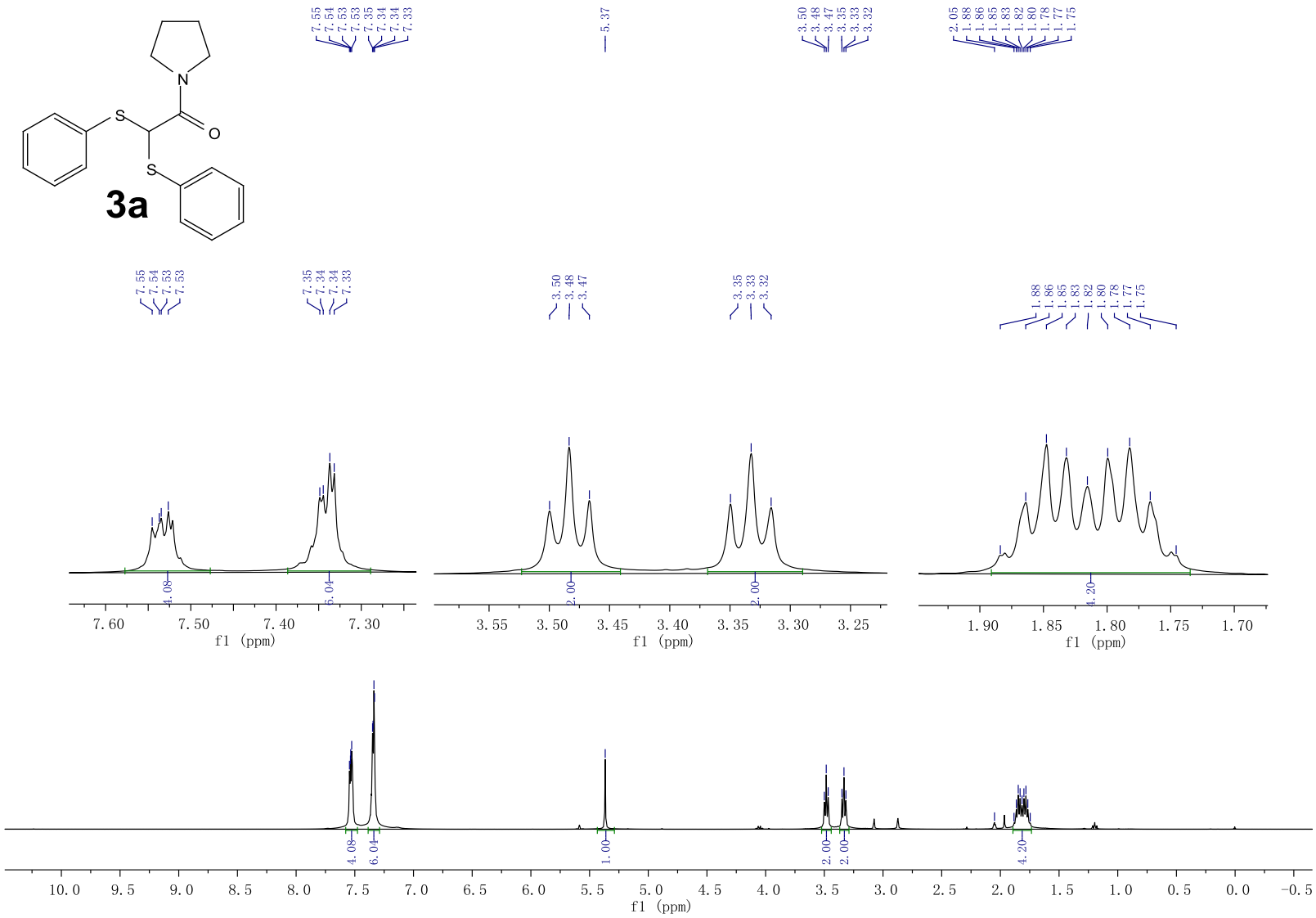
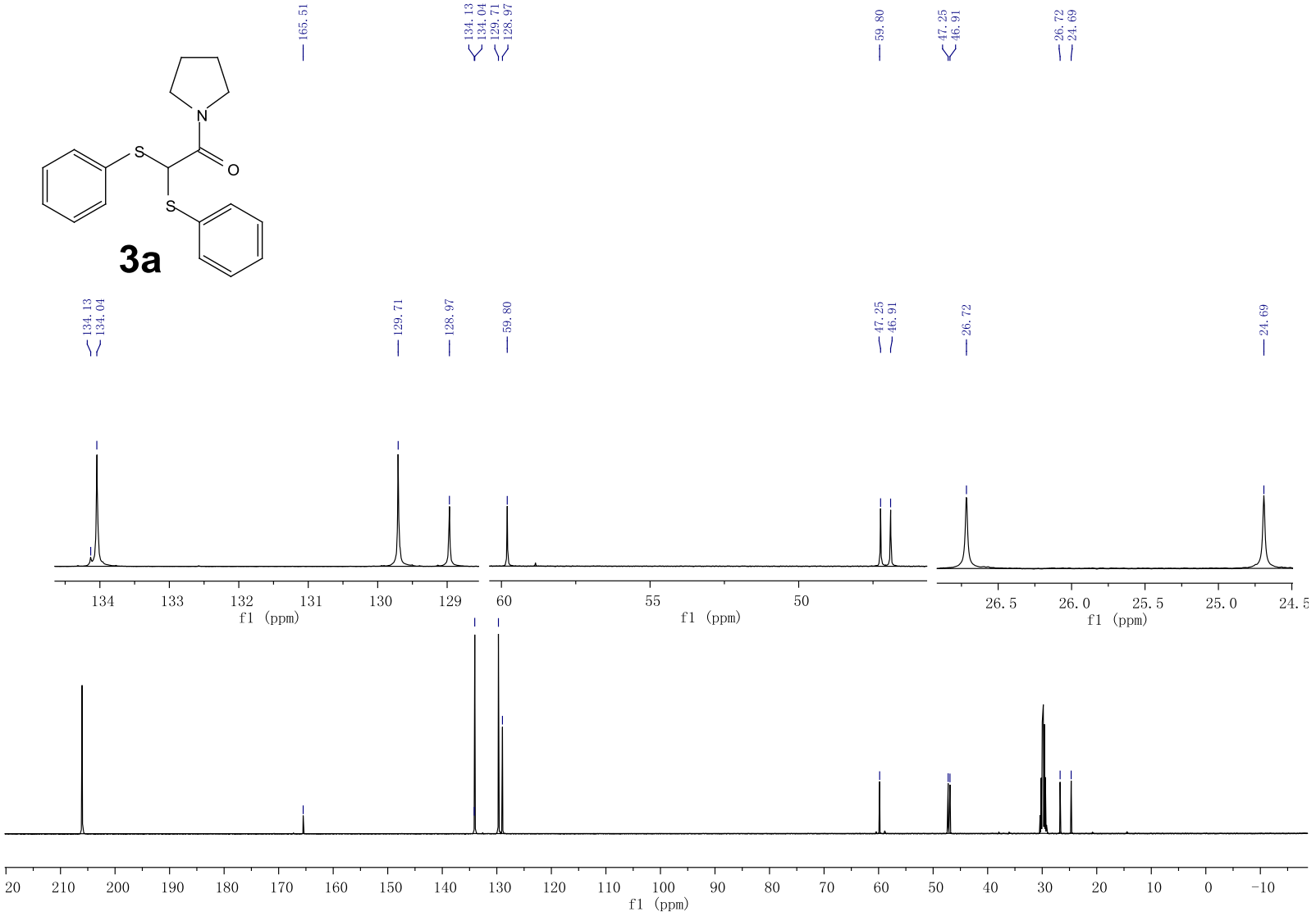
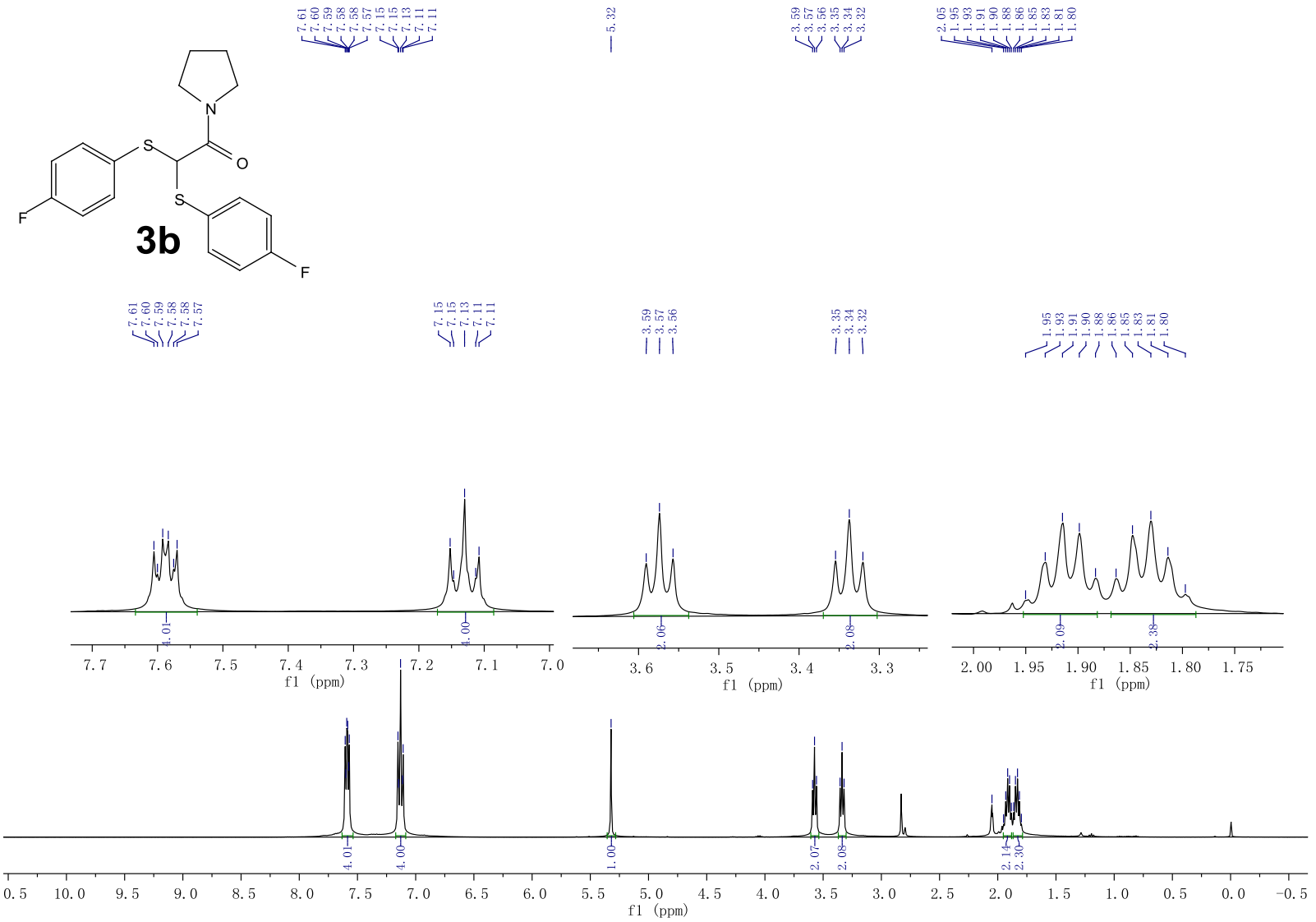


^{1}H NMR (400 MHz, CD_3COCD_3)



^{13}C NMR (100 MHz, CD_3COCD_3)



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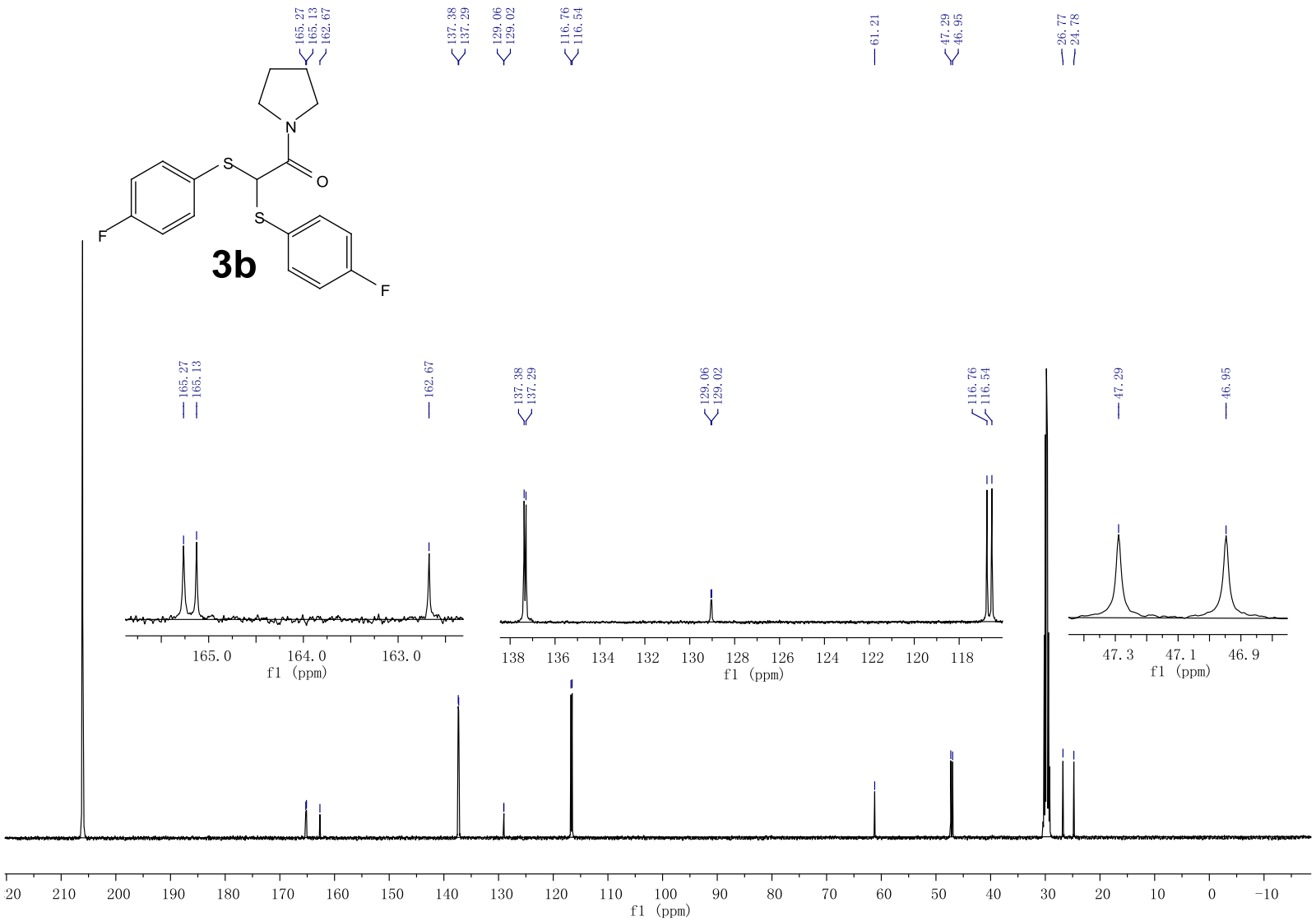

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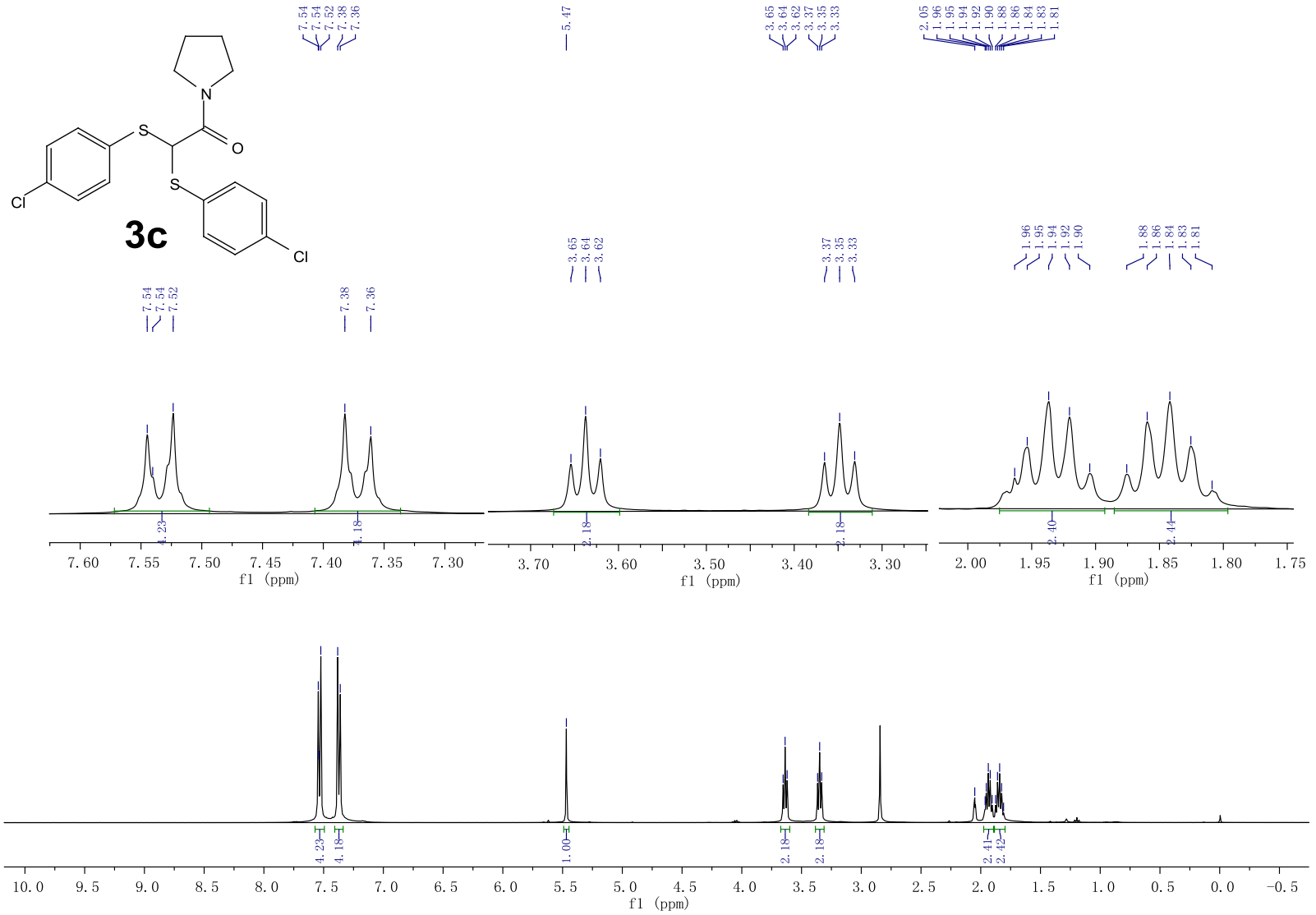

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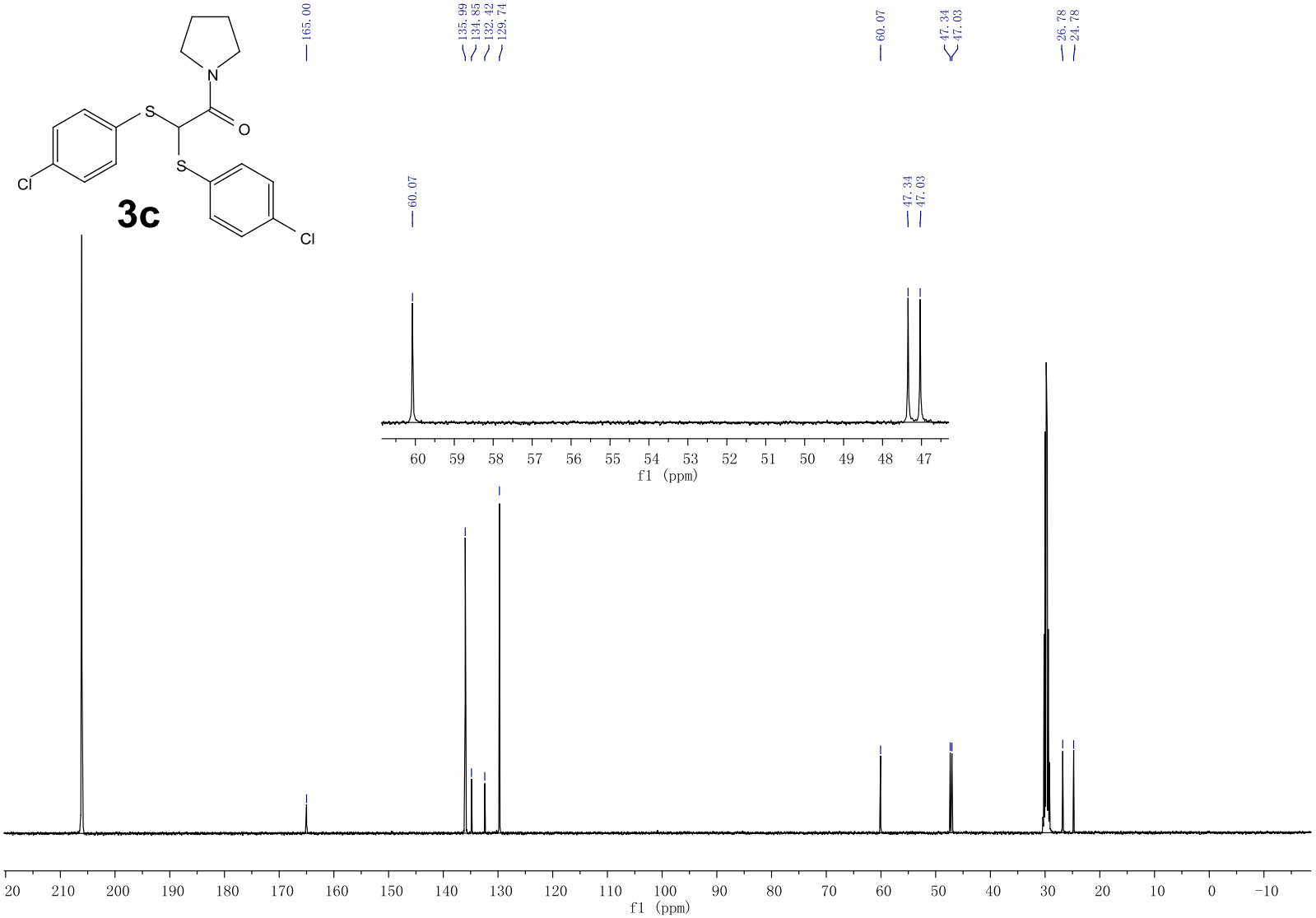

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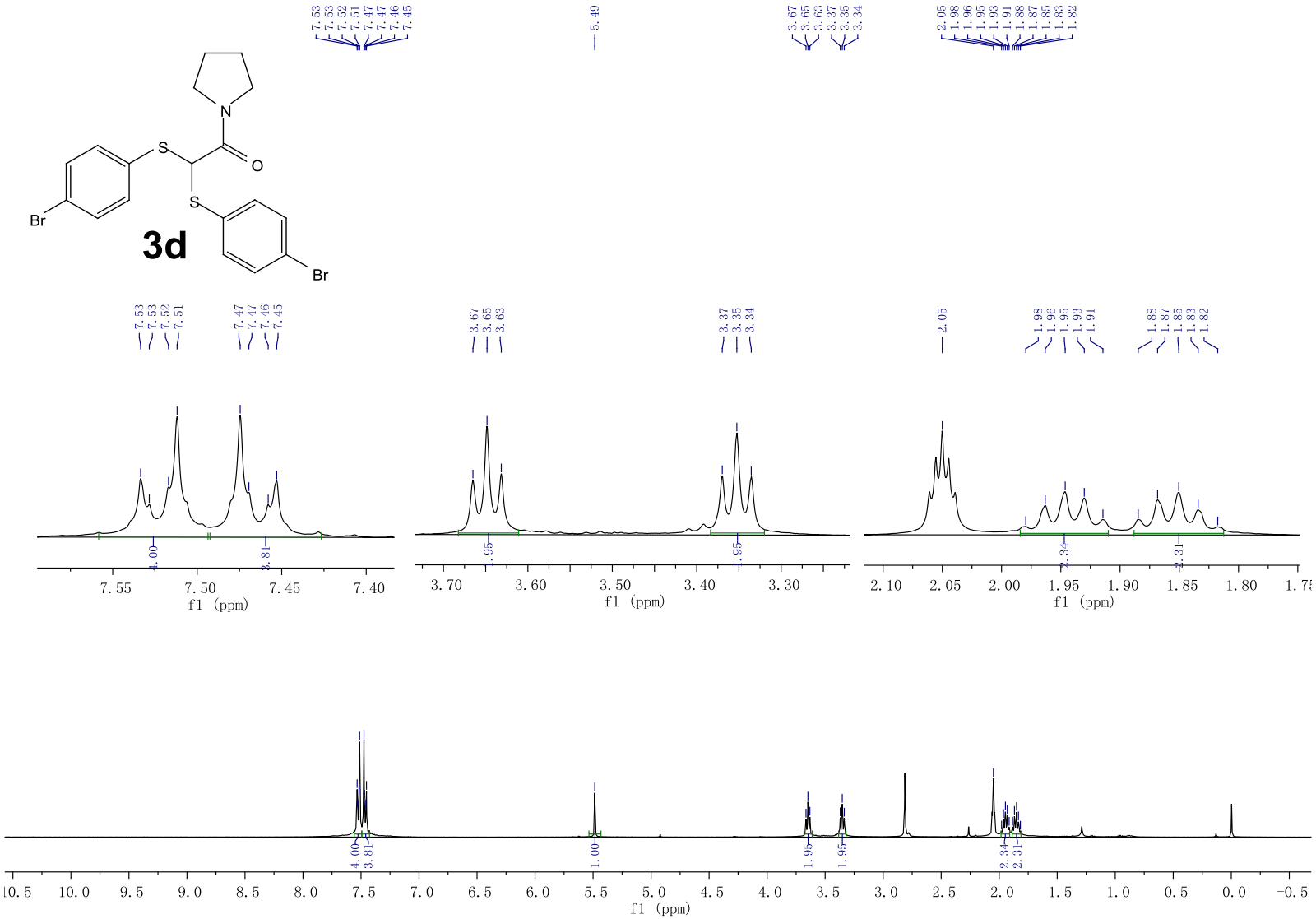

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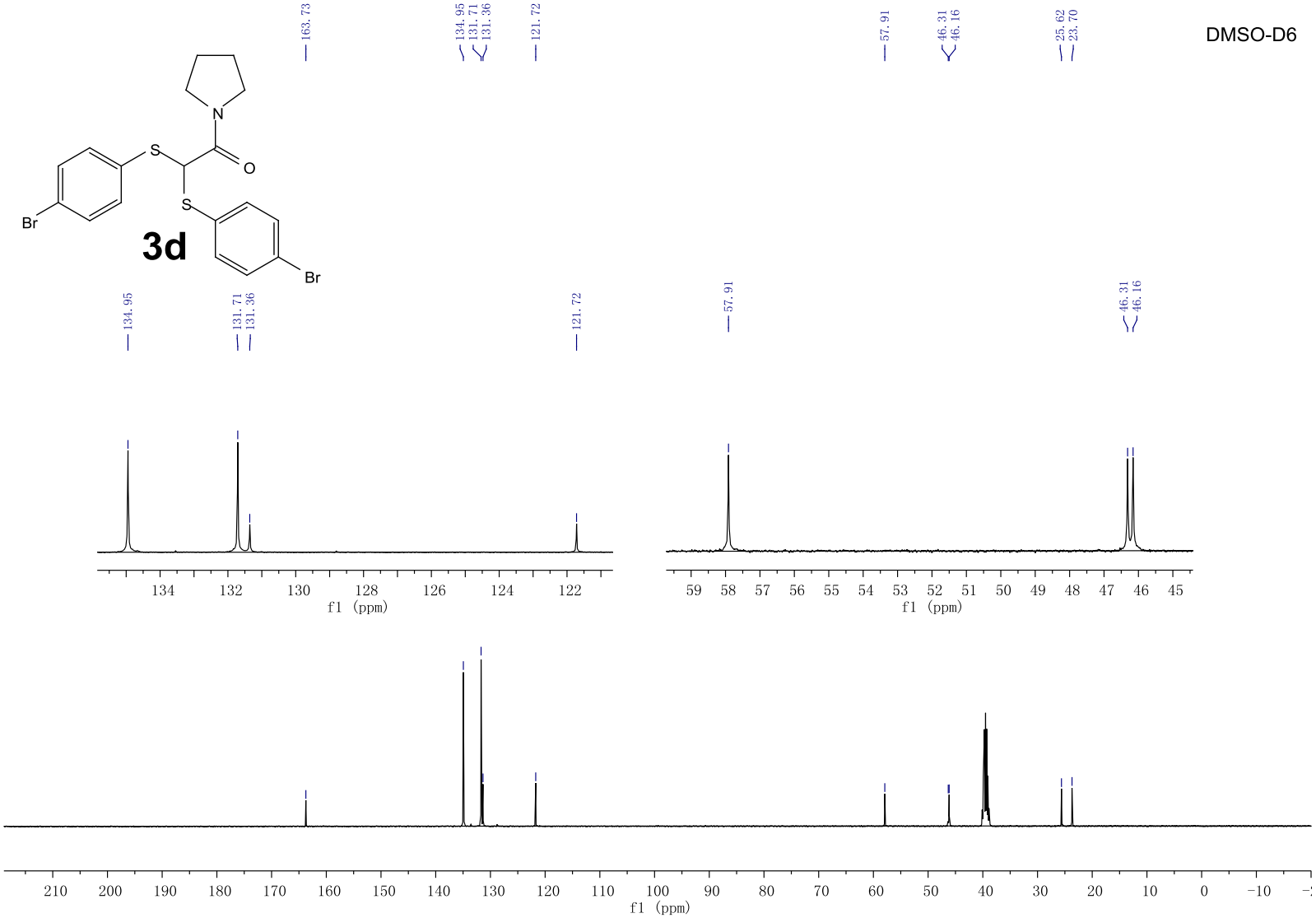

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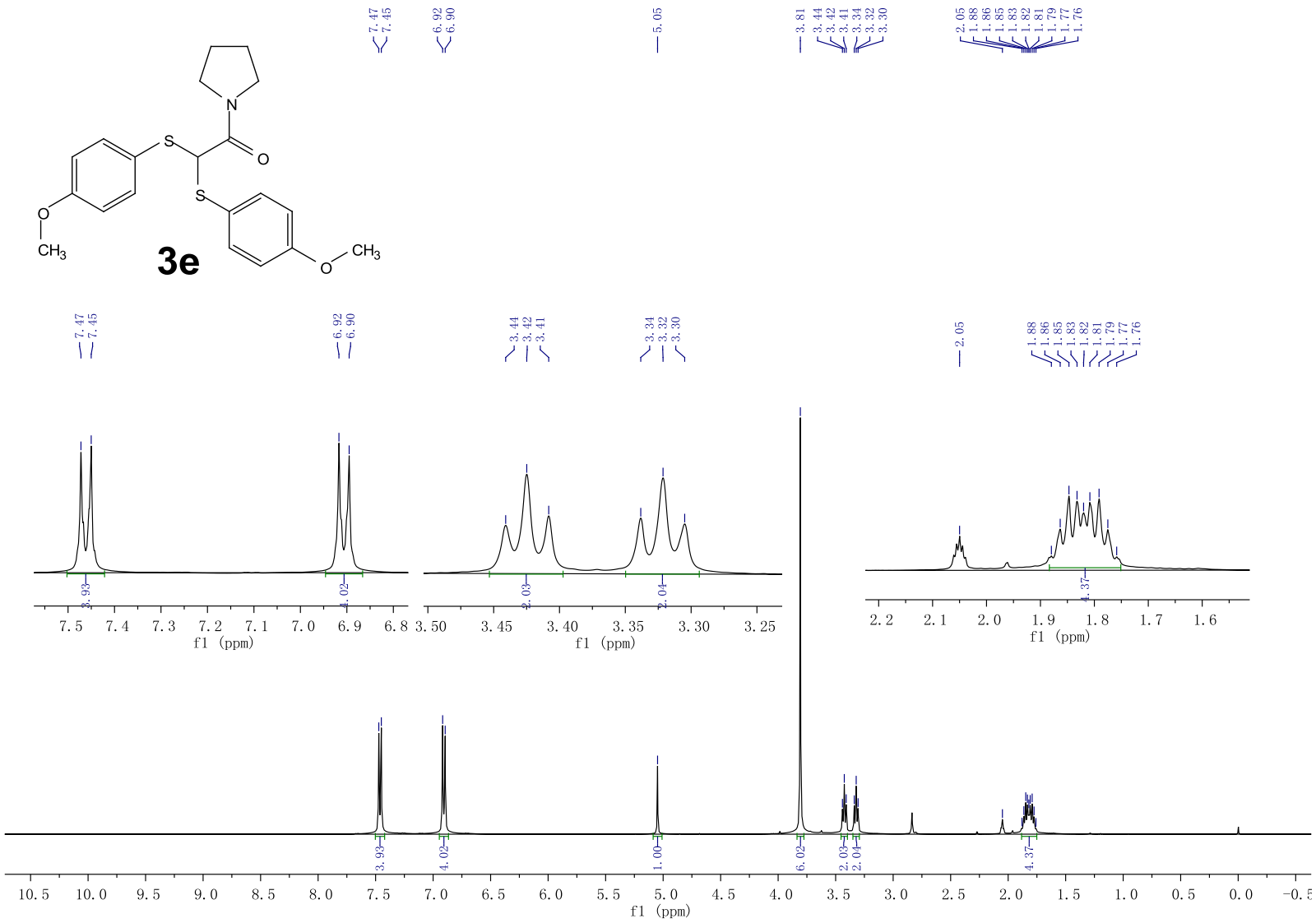

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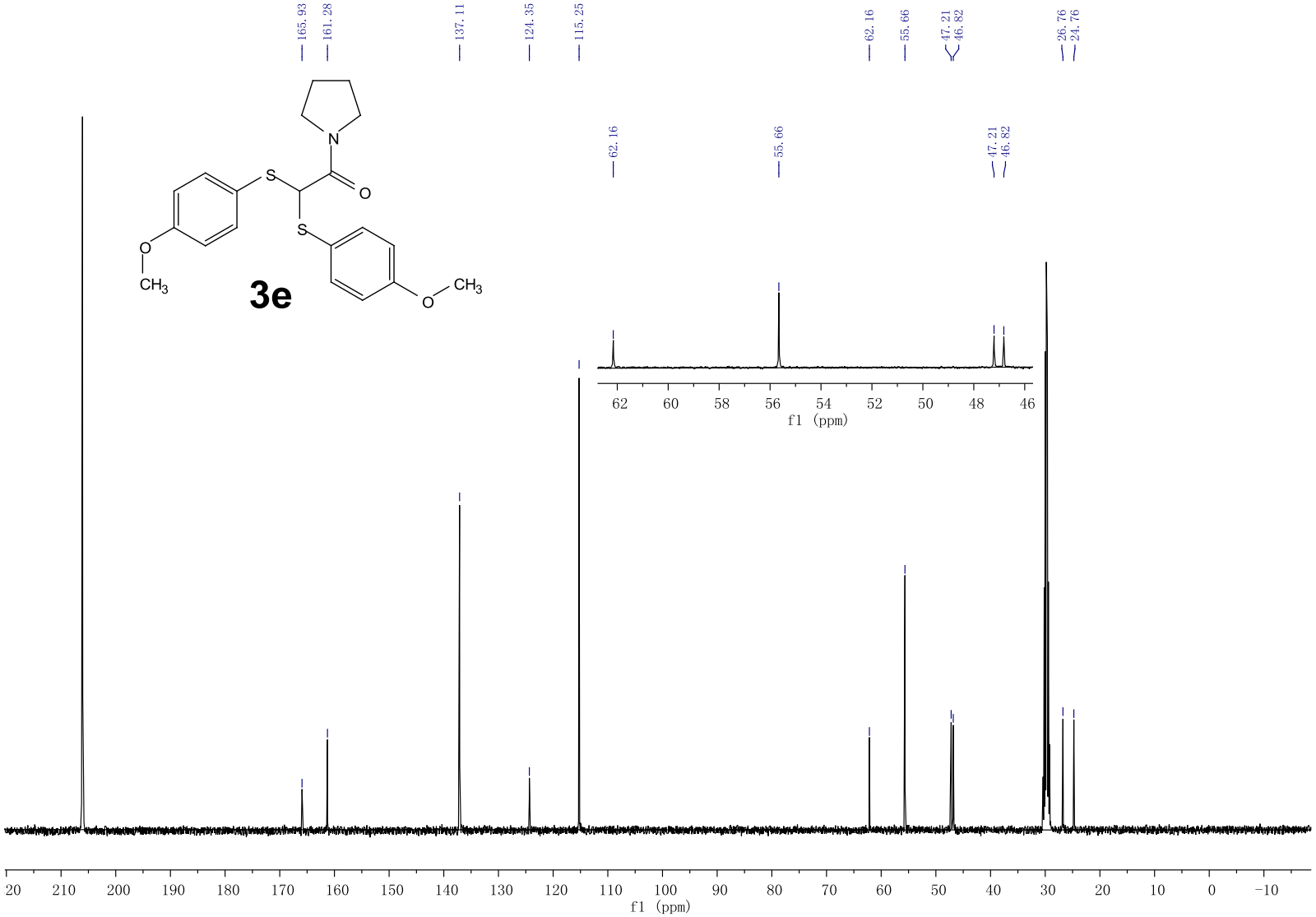

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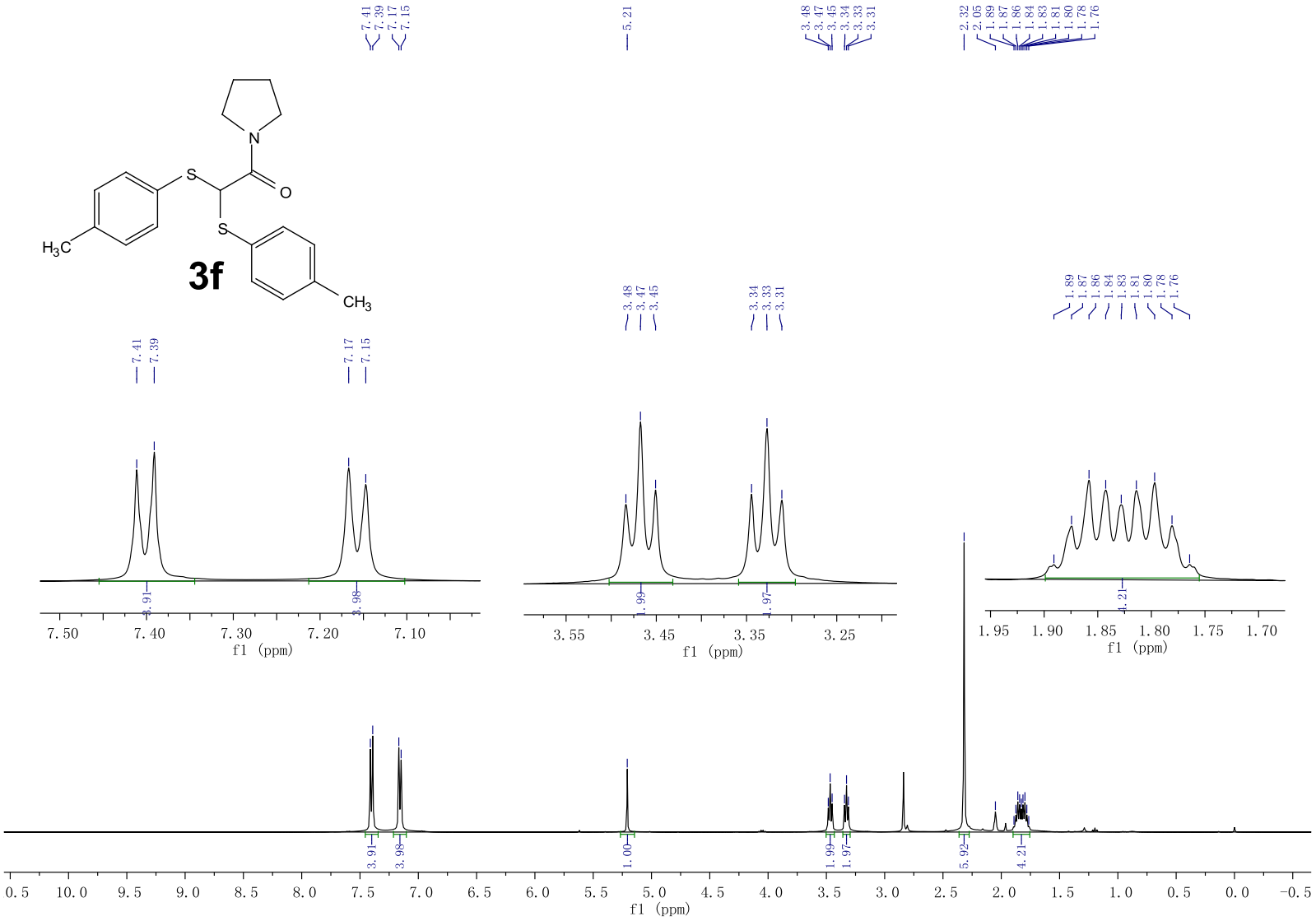

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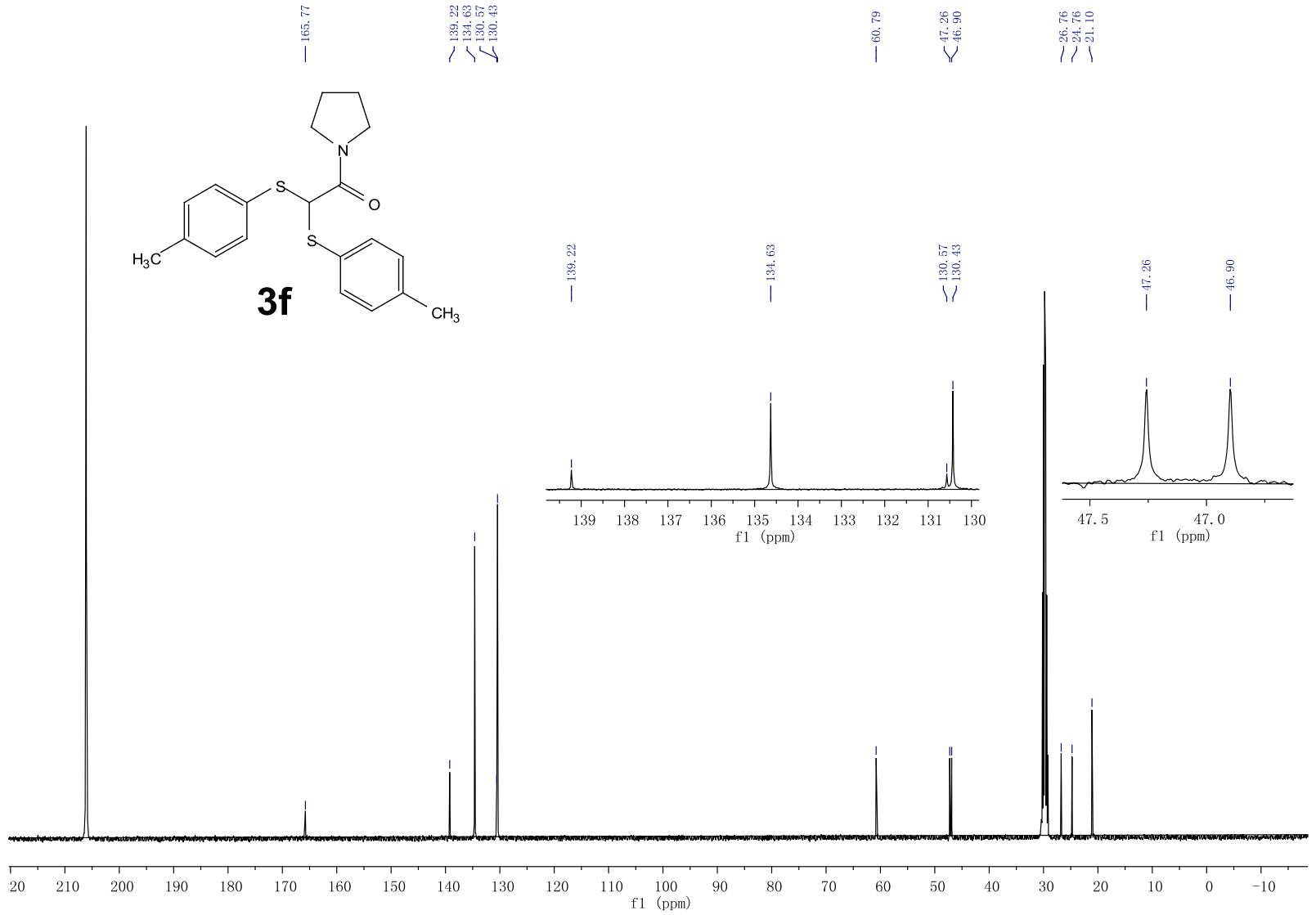
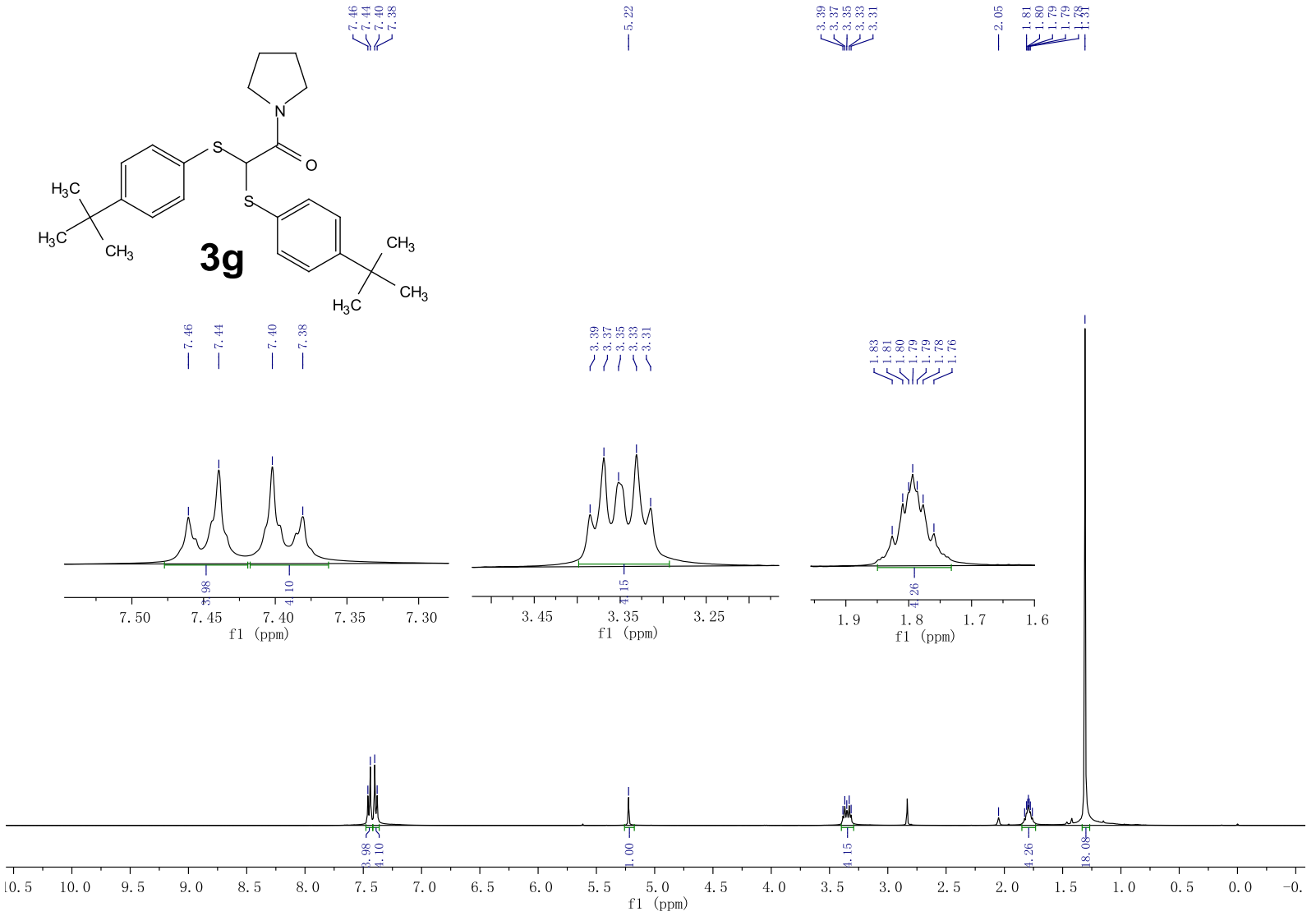


^{13}C NMR (100 MHz, CD_3COCD_3)



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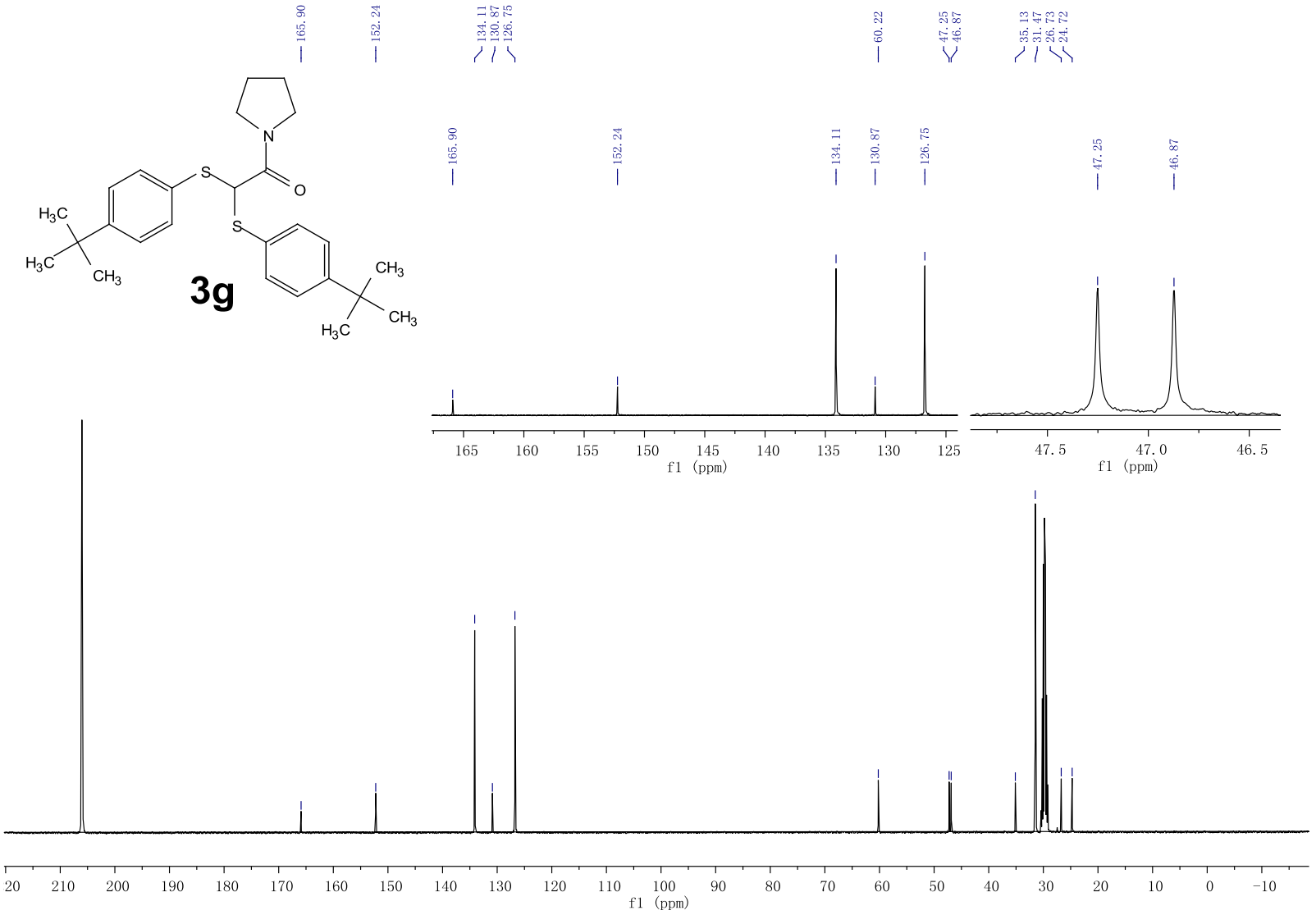

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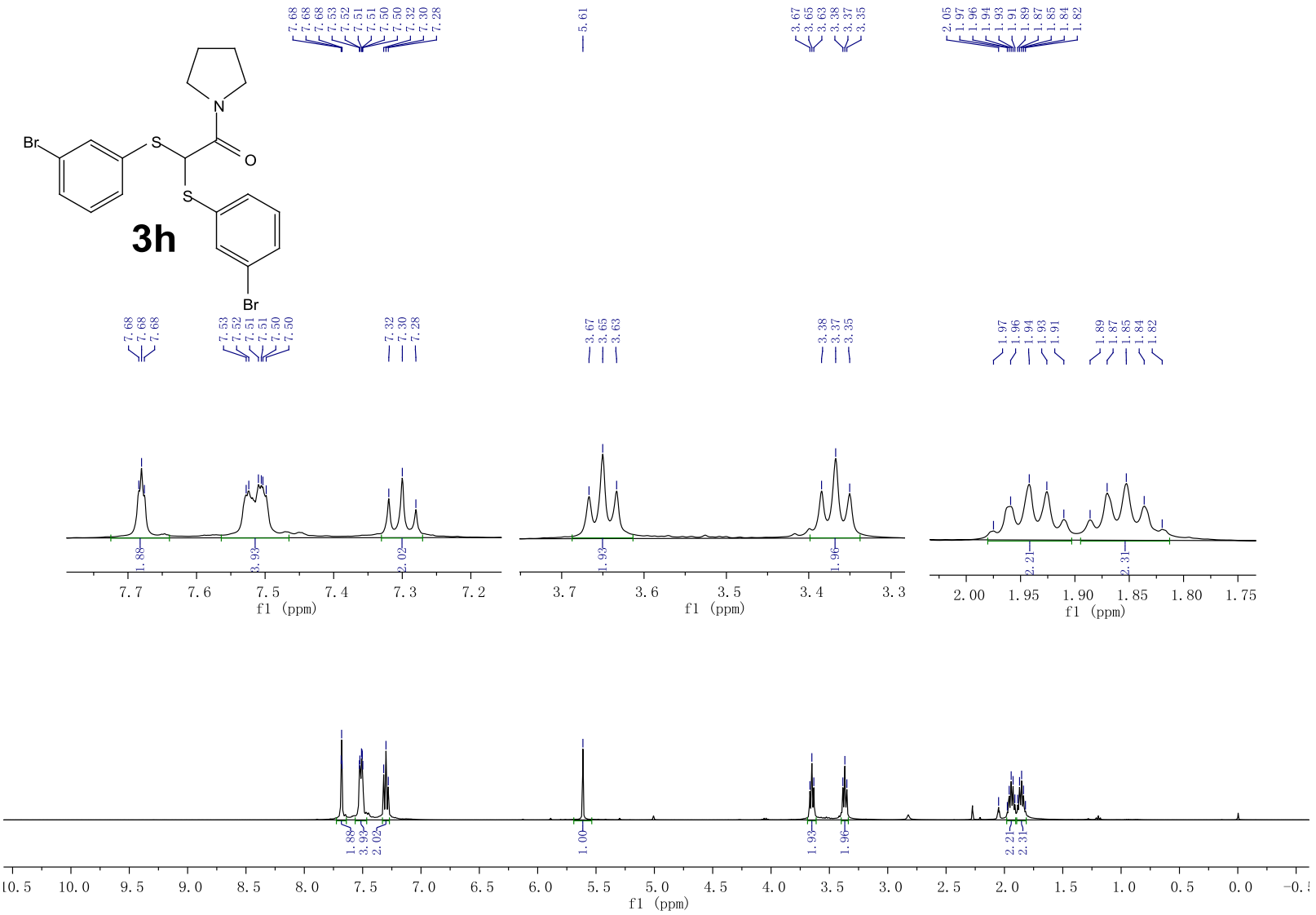

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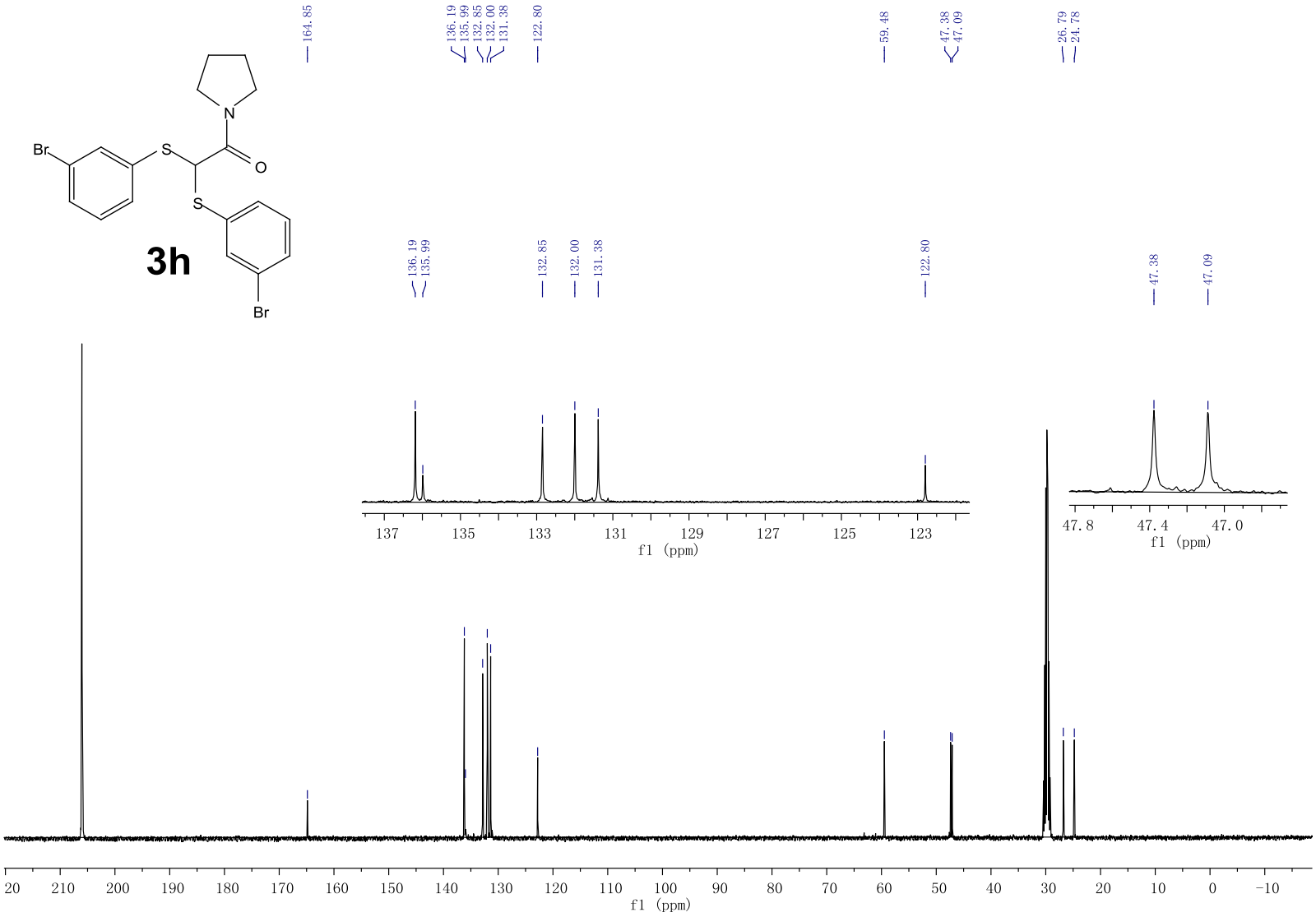

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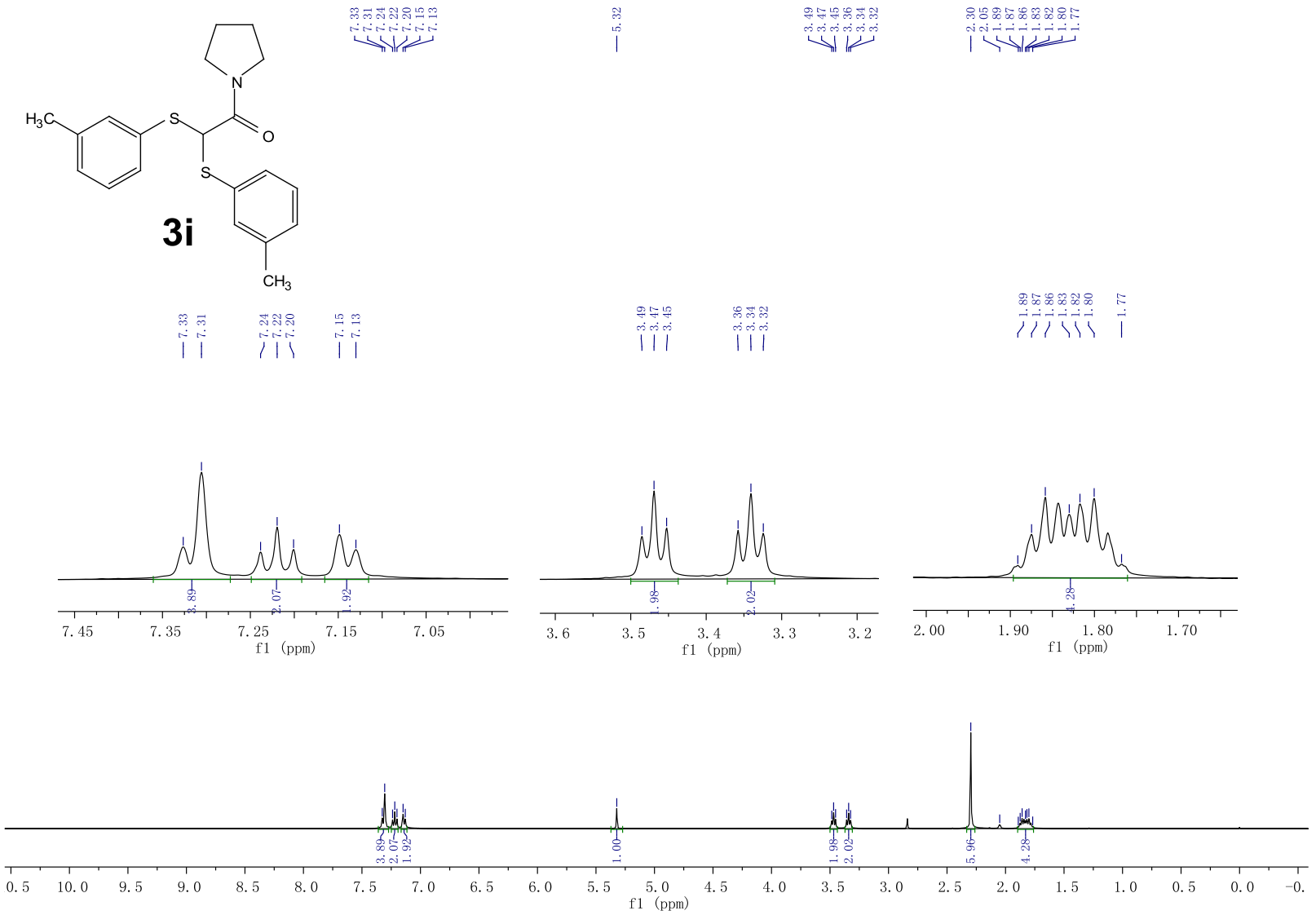

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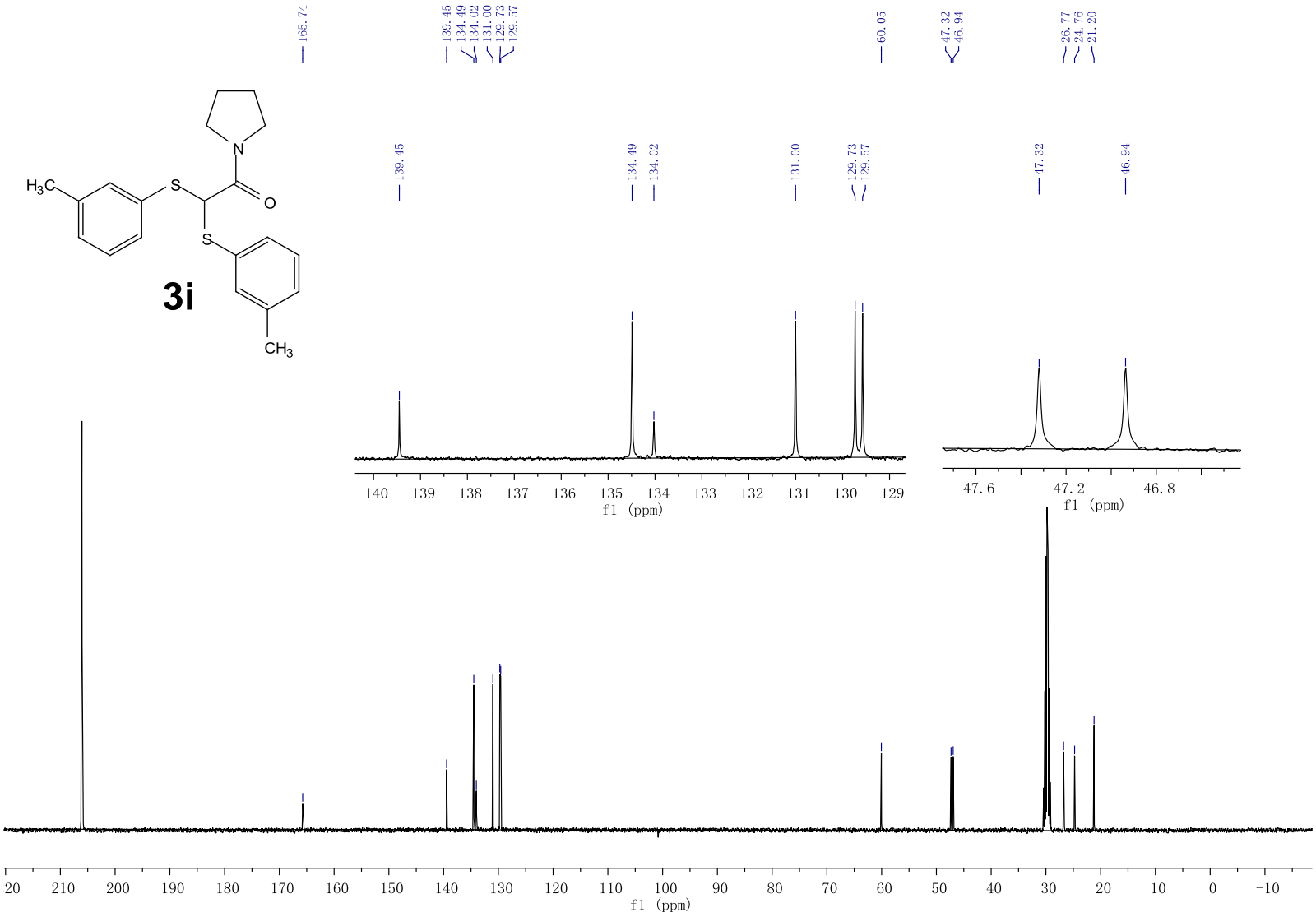

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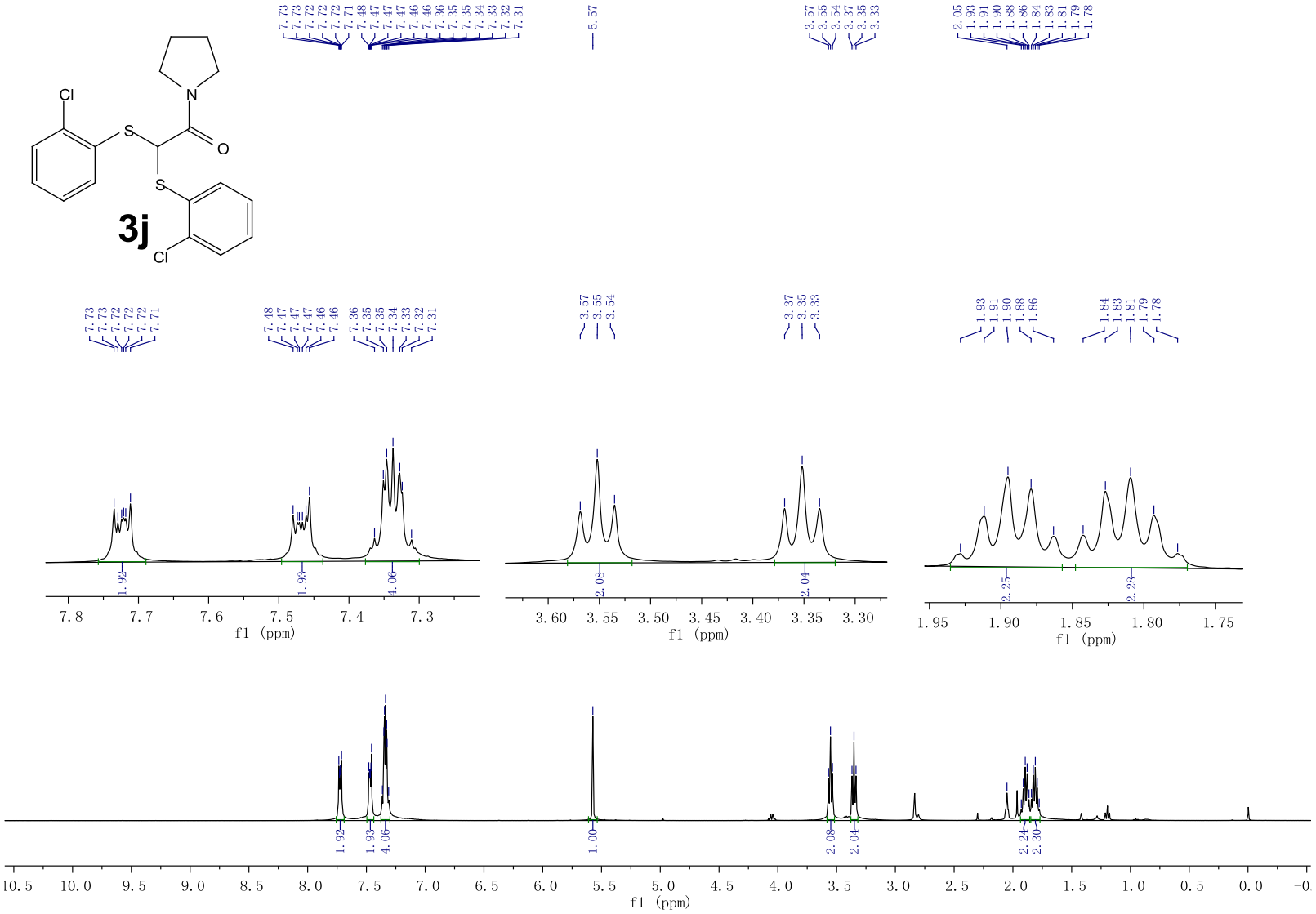

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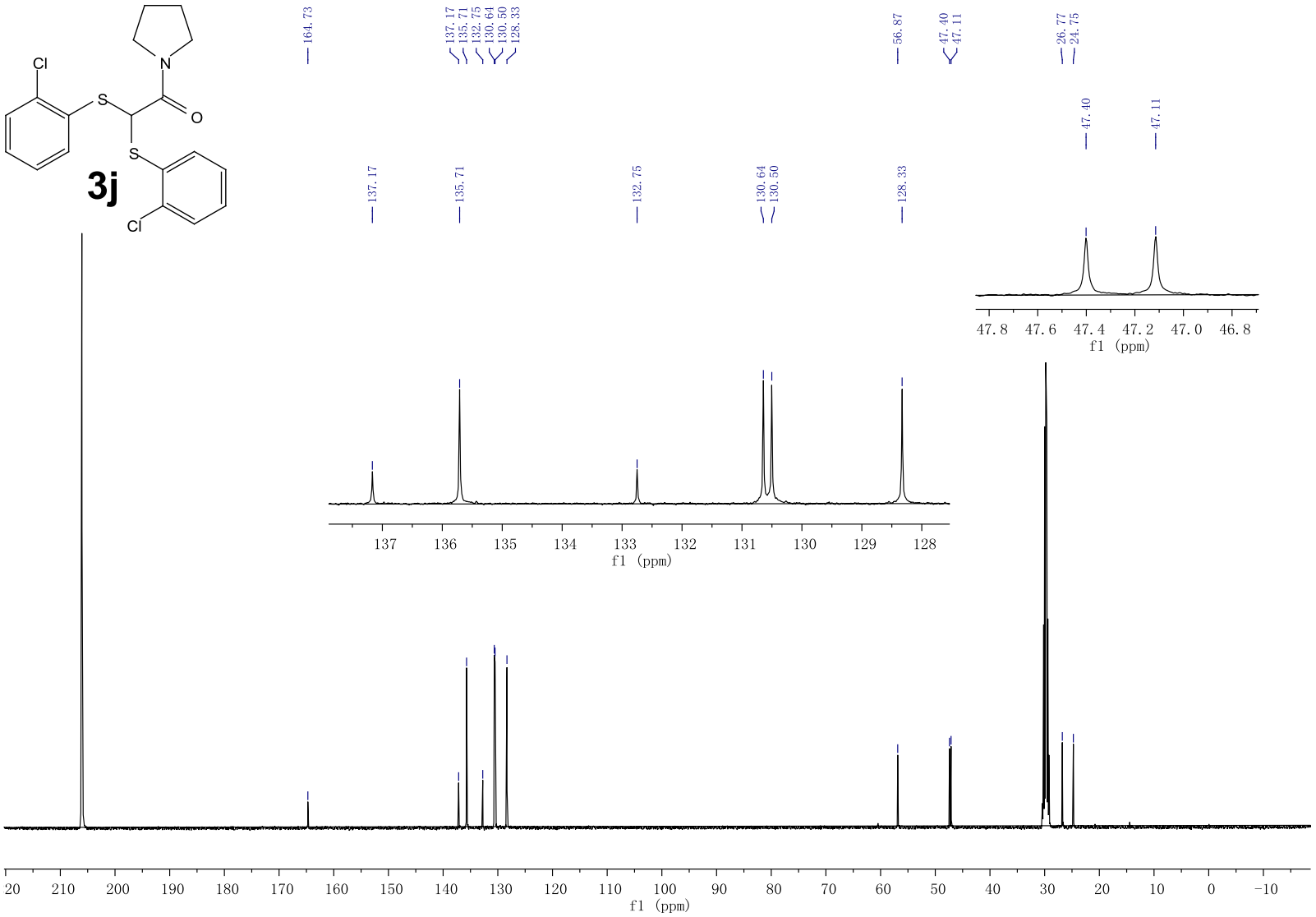

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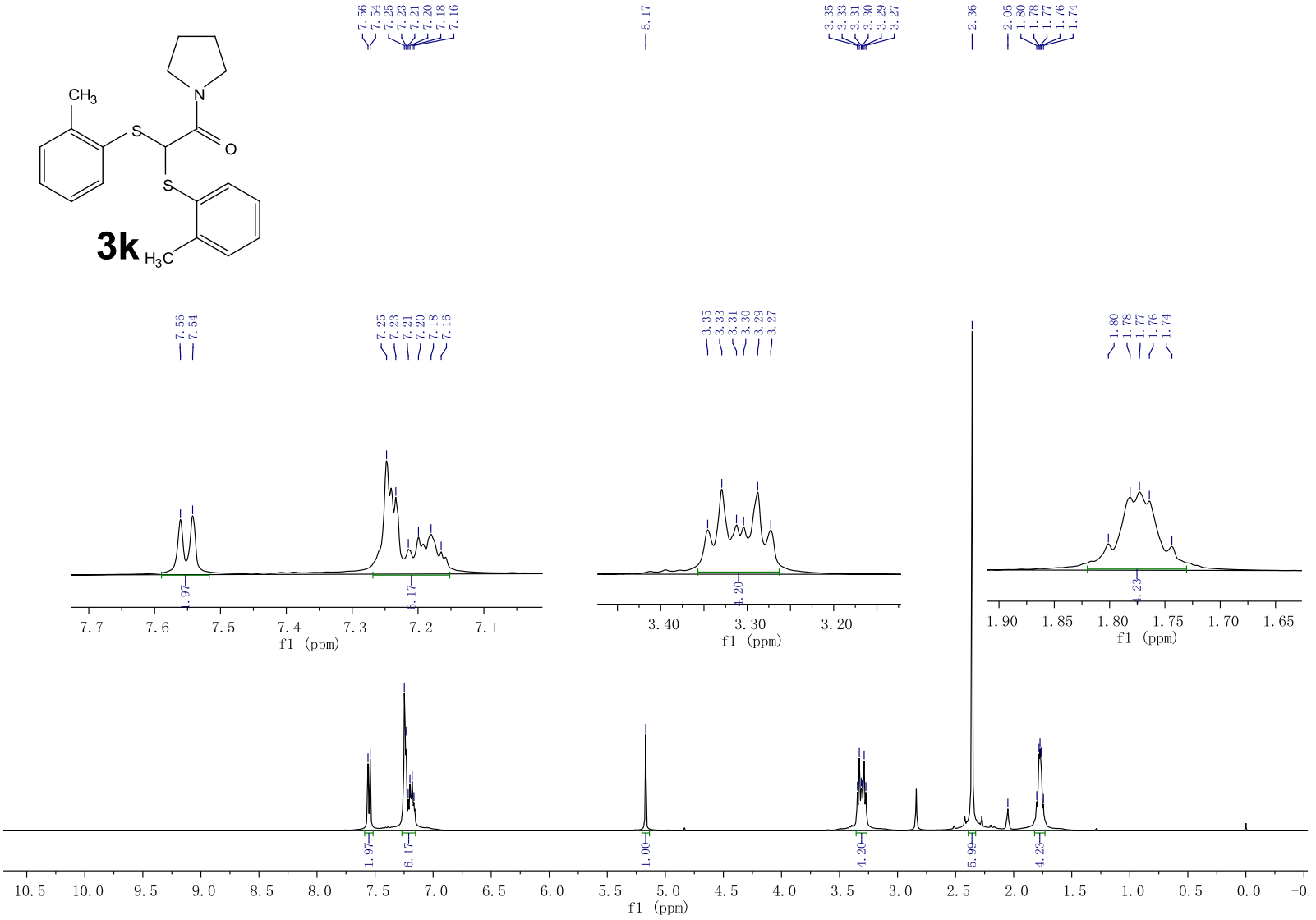

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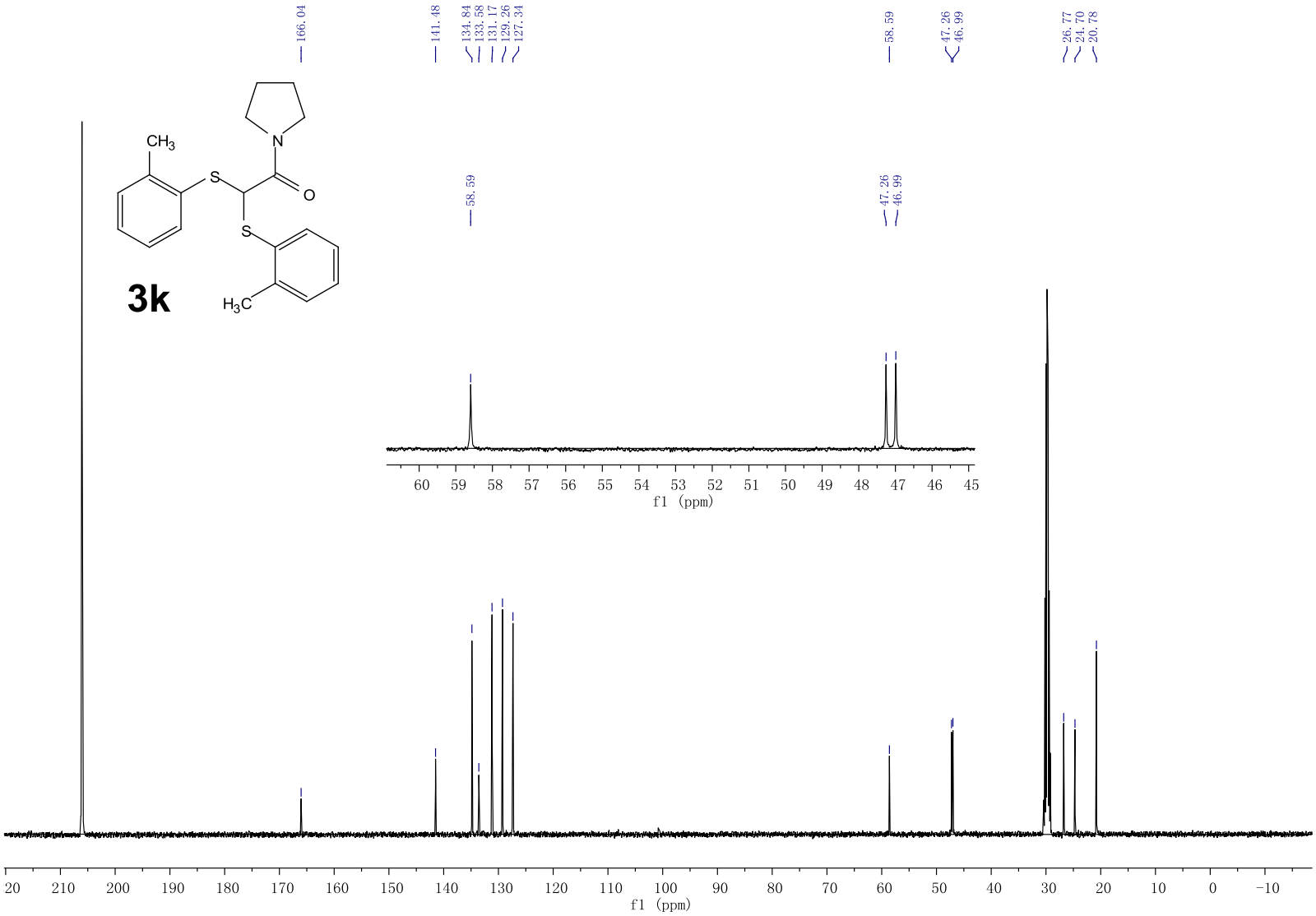

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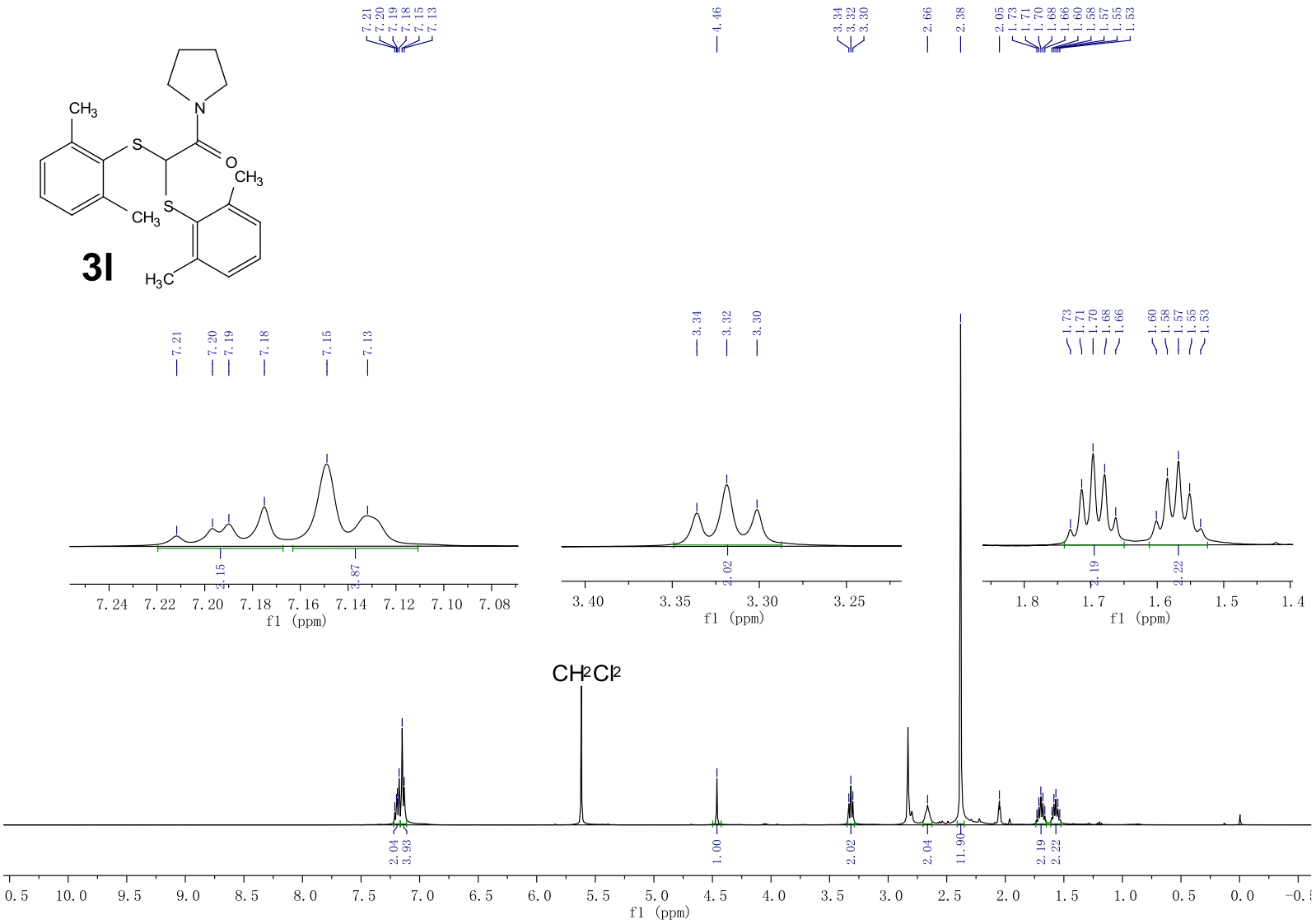
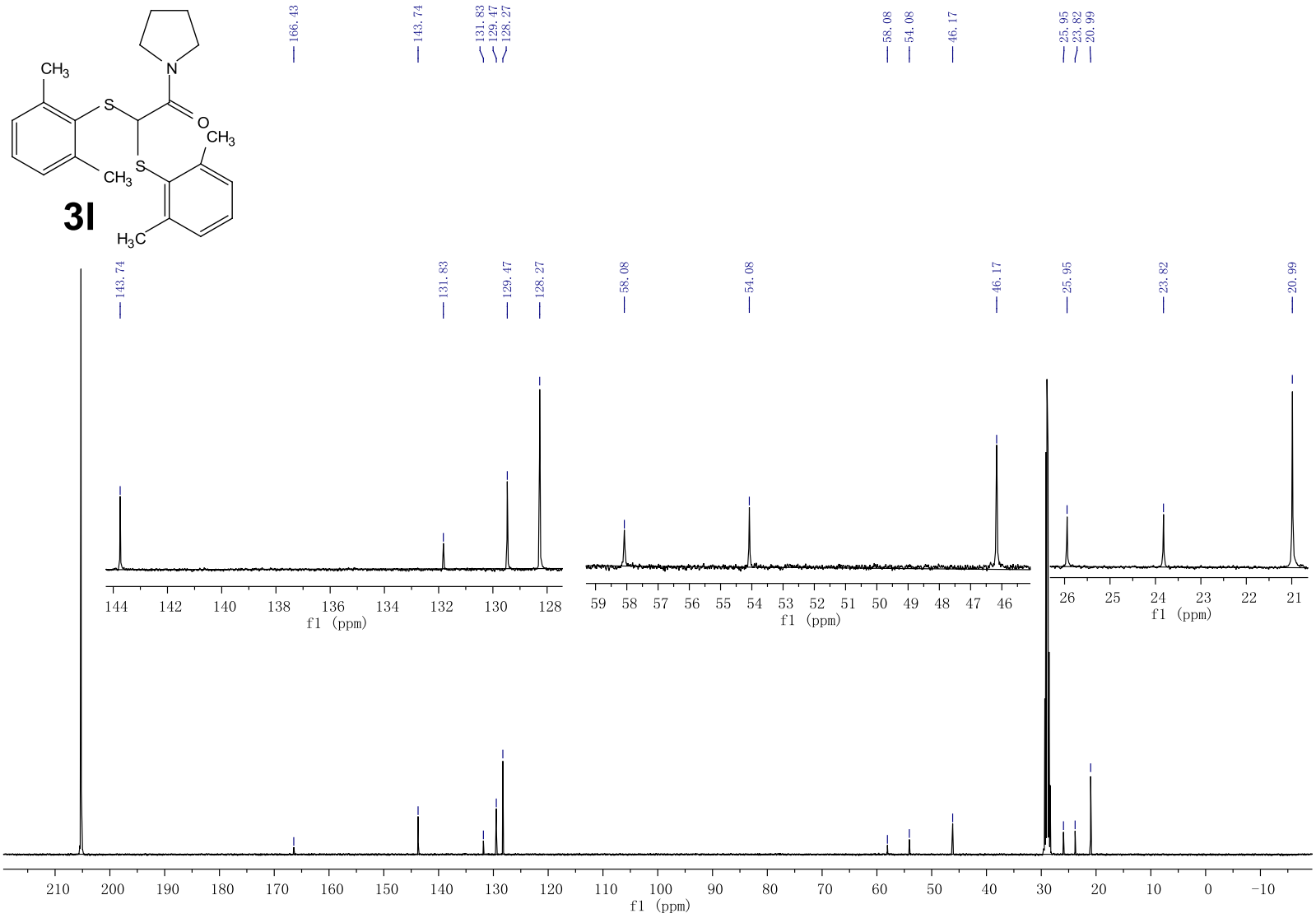
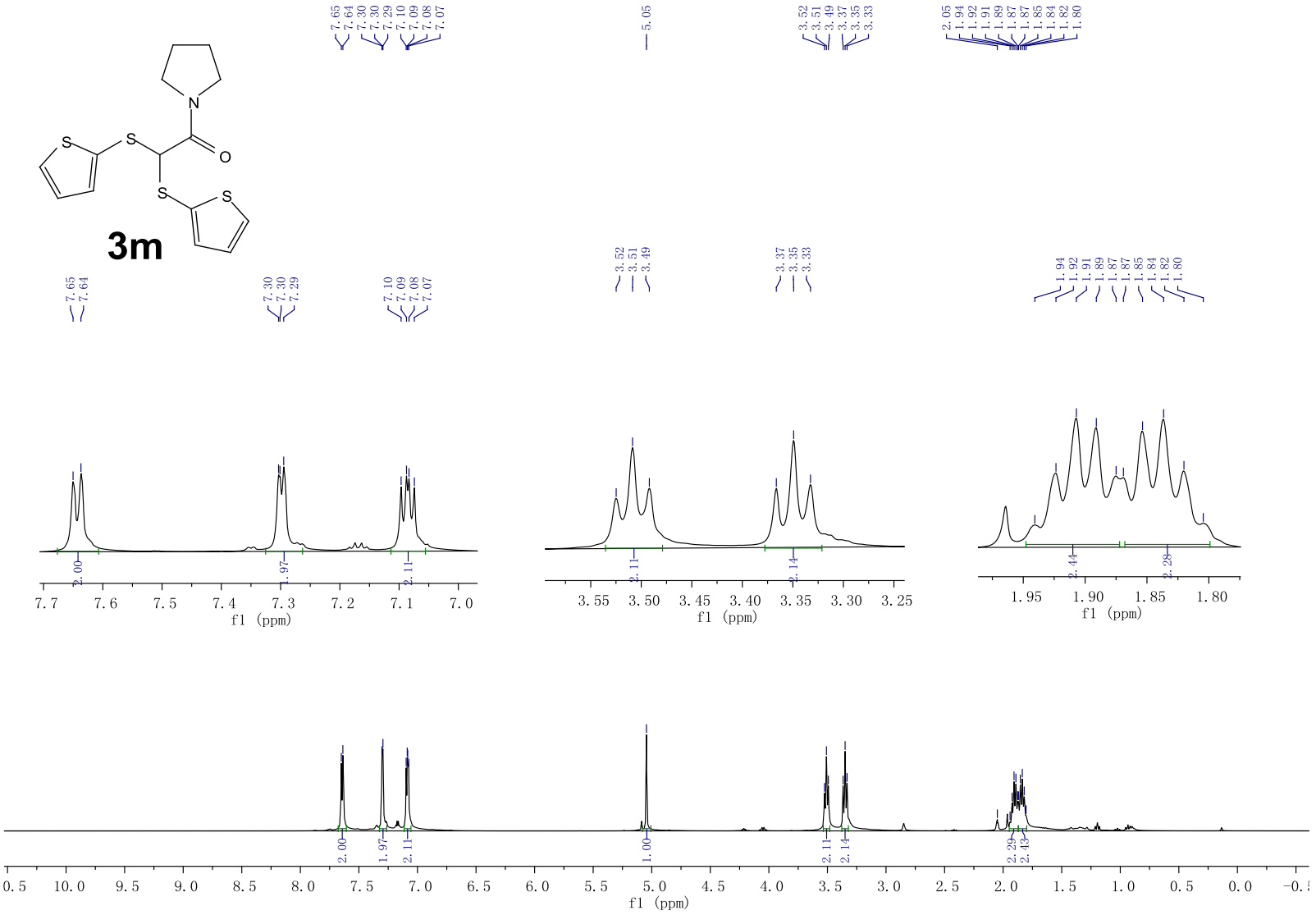


^{1}H NMR (400 MHz, CD_3COCD_3)



^{13}C NMR (100 MHz, CD_3COCD_3)



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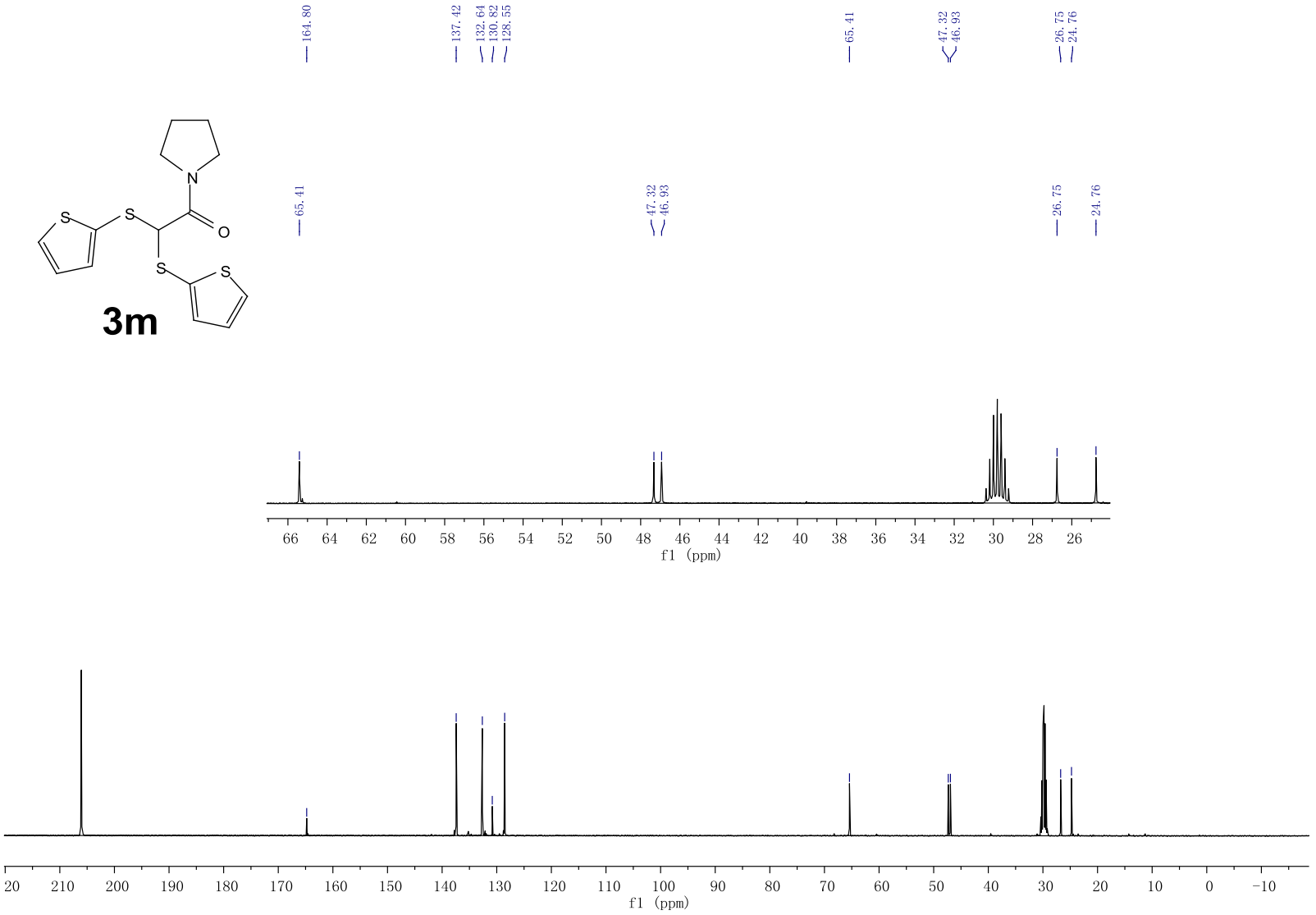
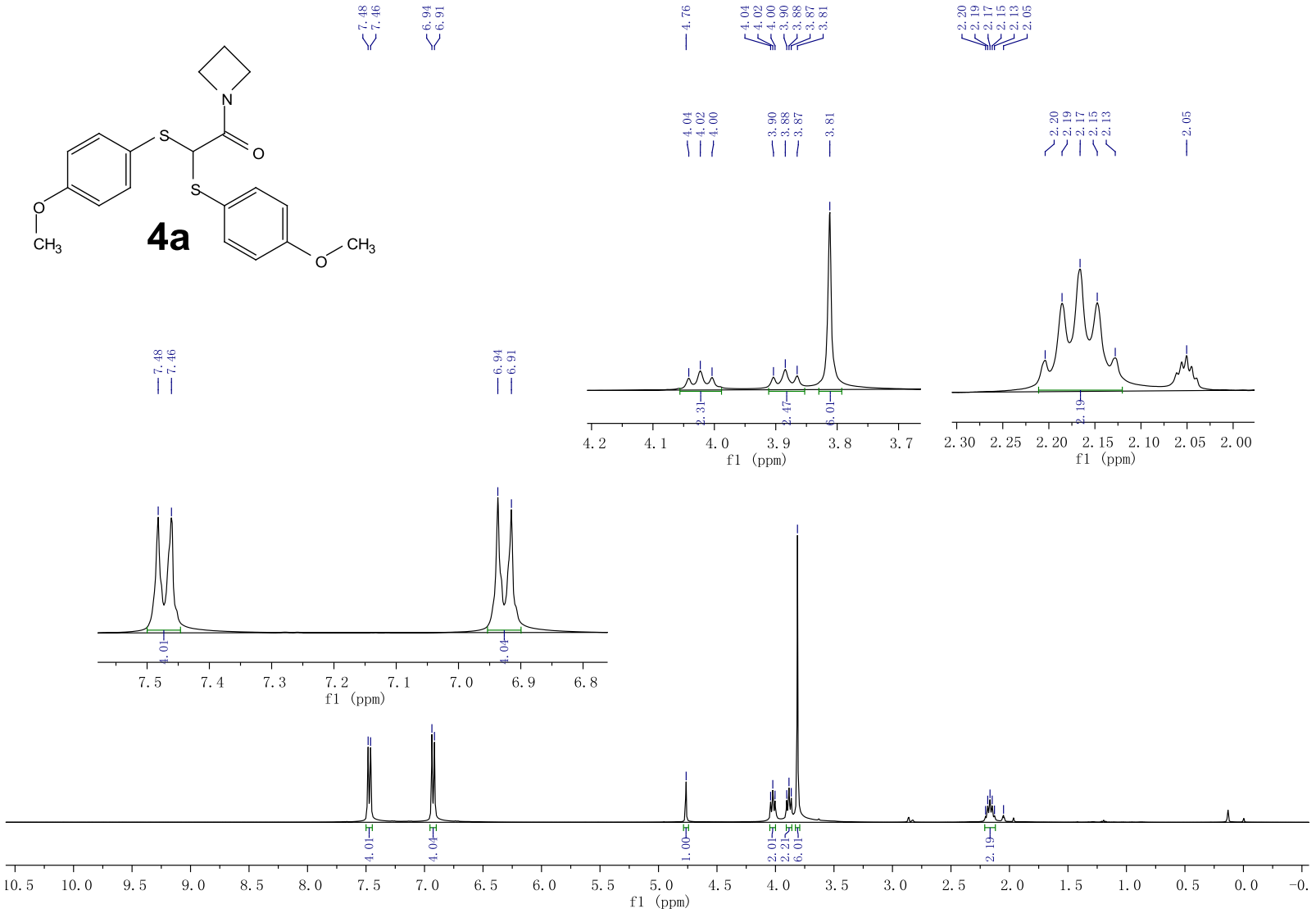


^{13}C NMR (100 MHz, CD_3COCD_3)



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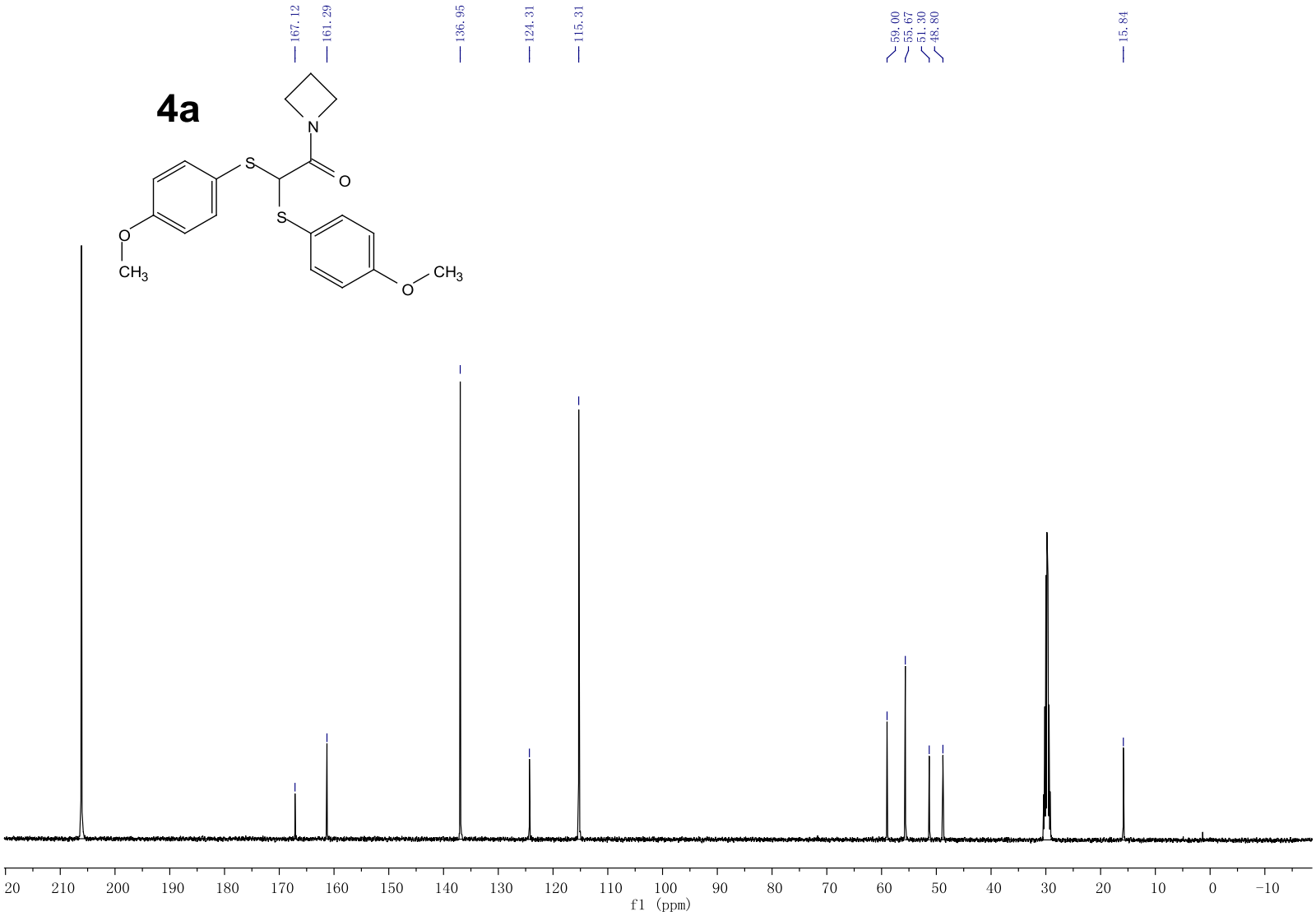
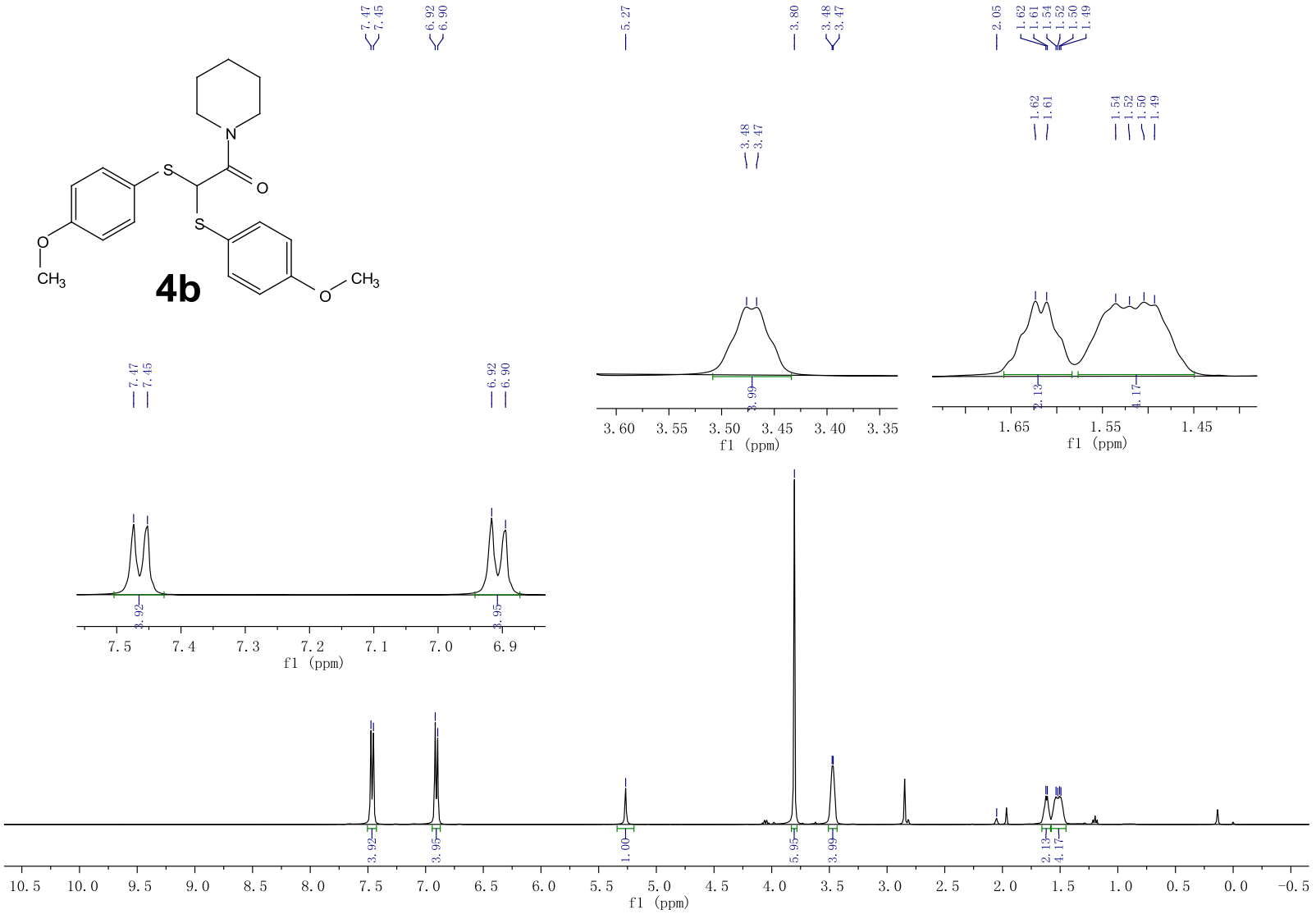
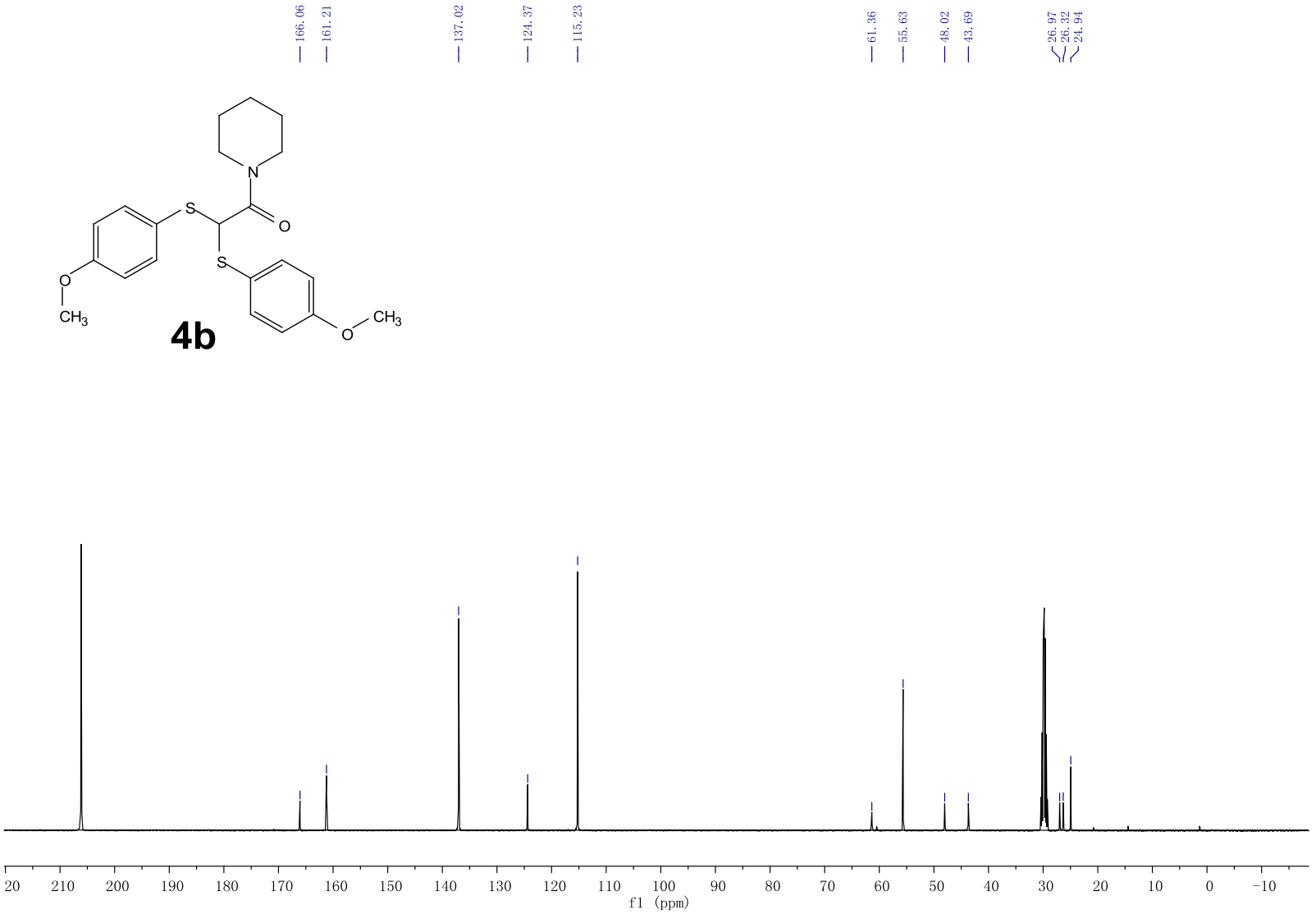


^{13}C NMR (100 MHz, CD_3COCD_3)



^{1}H NMR (400 MHz, CD_3COCD_3)



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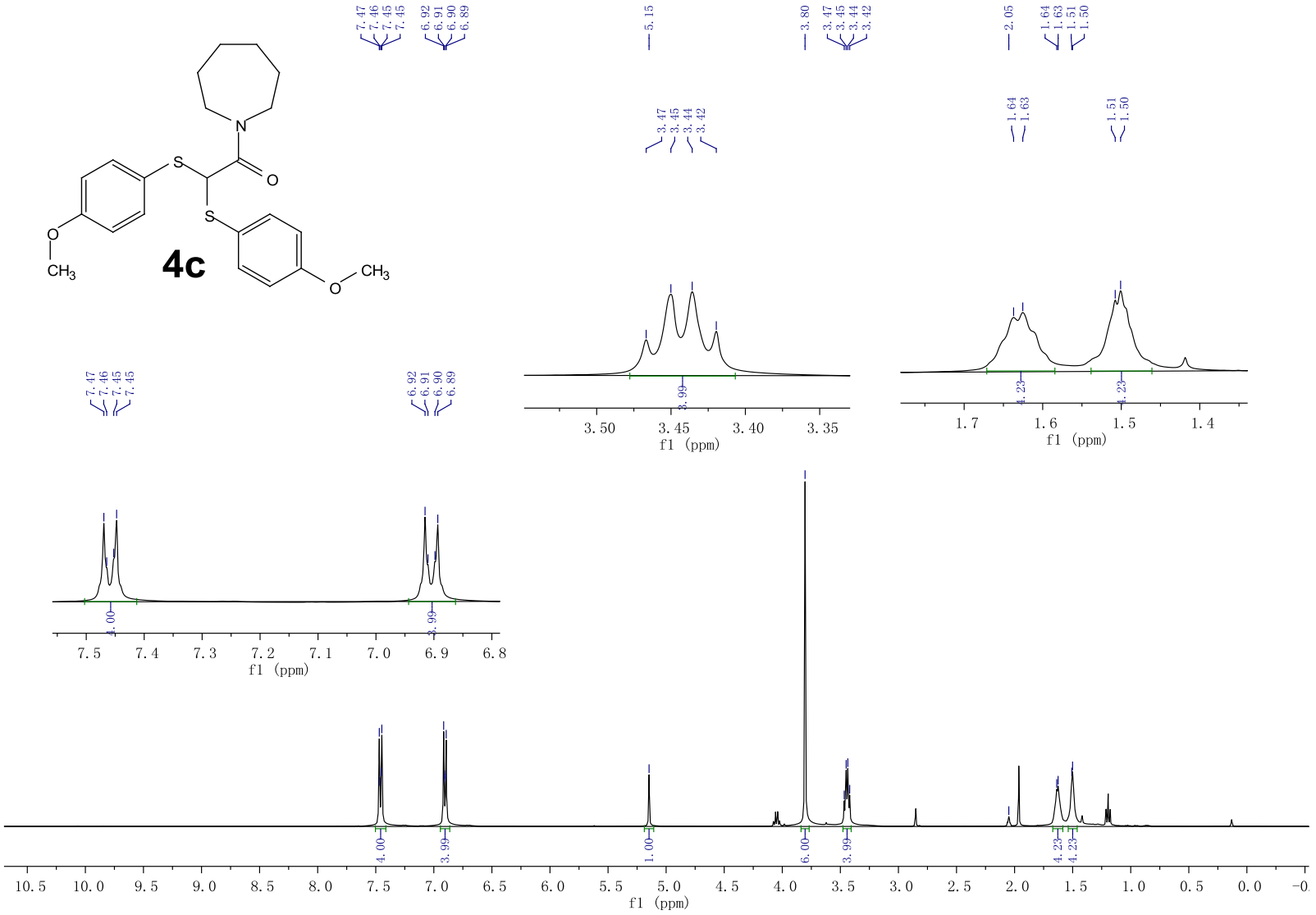

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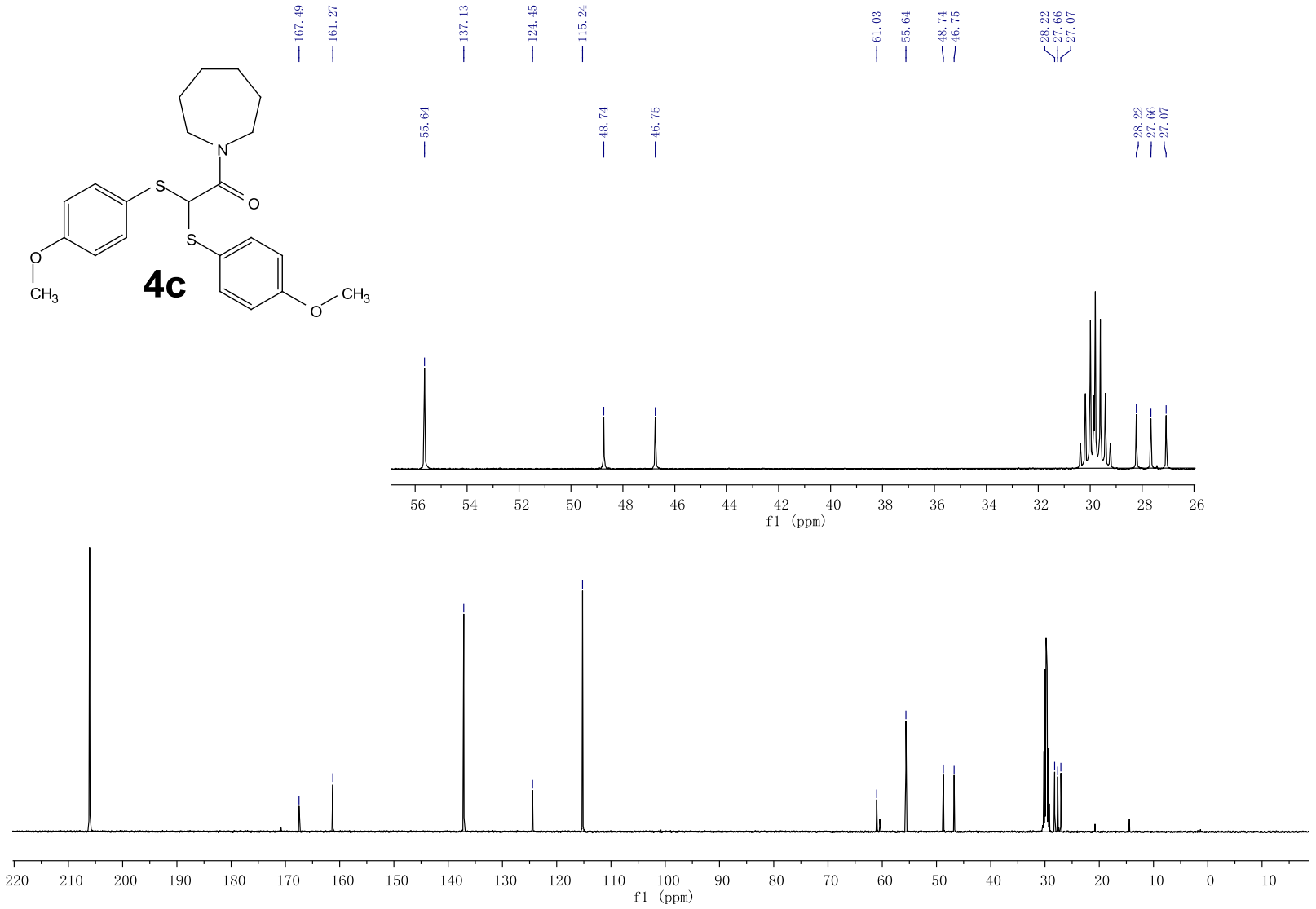
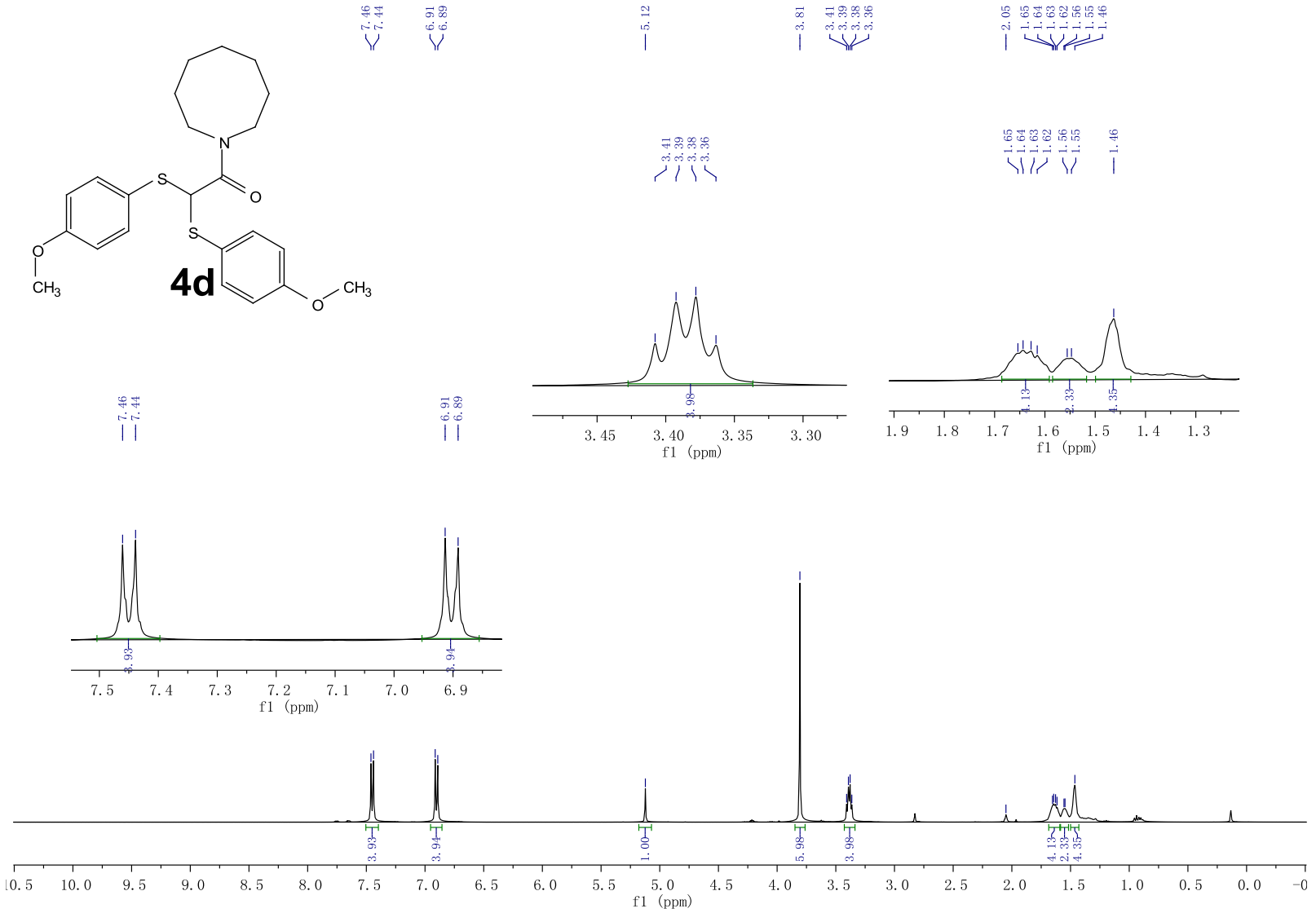


^{13}C NMR (100 MHz, CD_3COCD_3)



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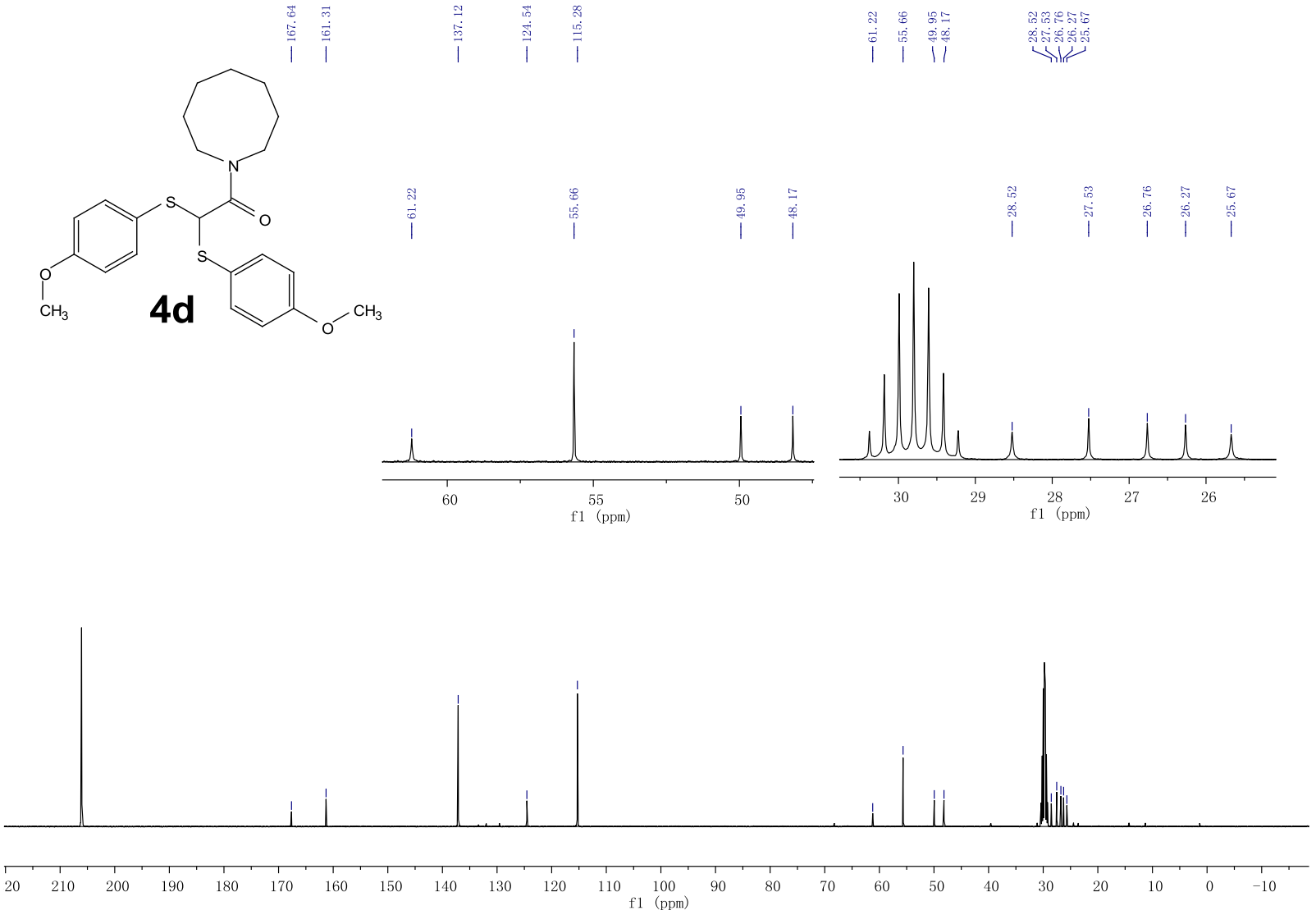

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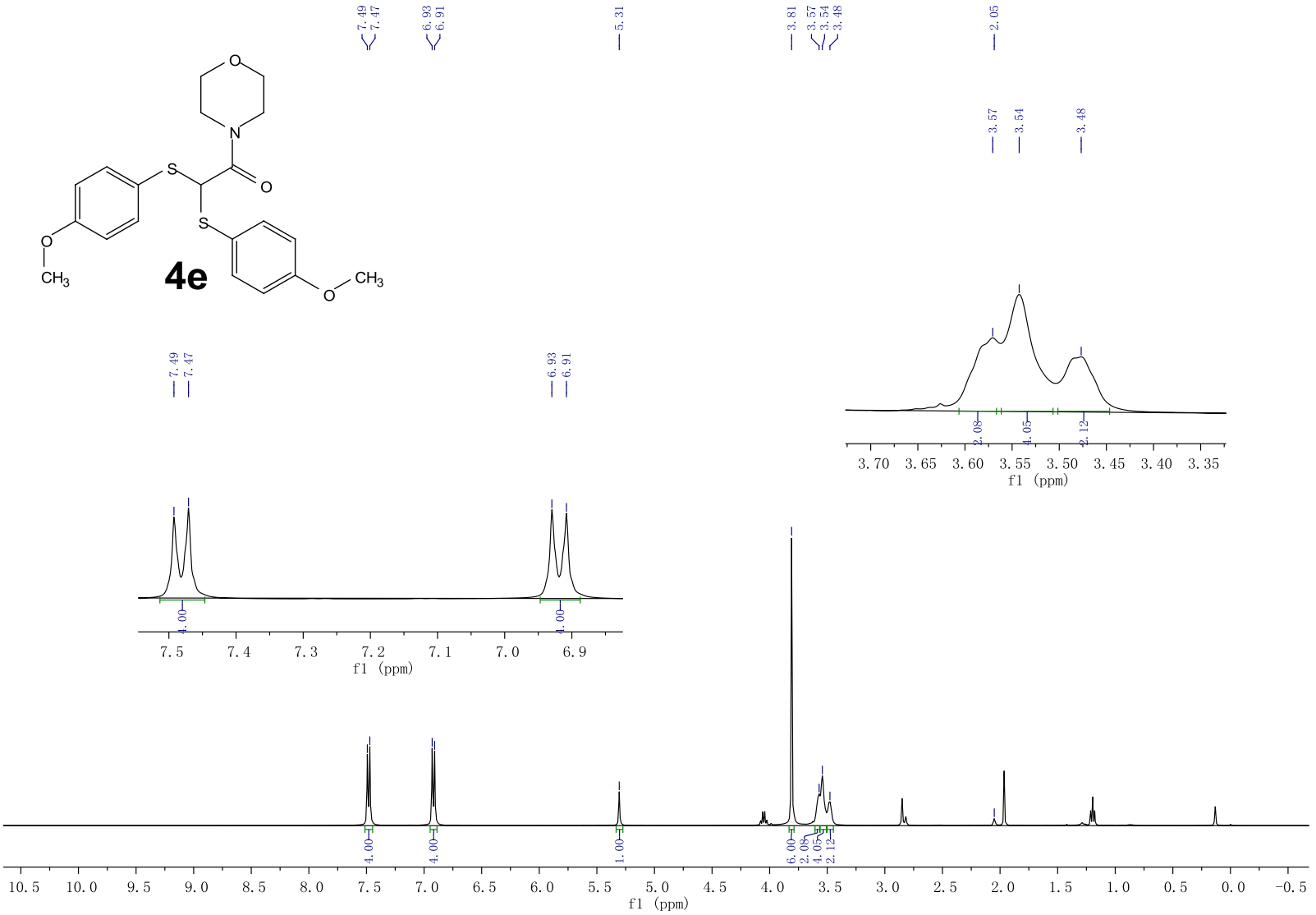

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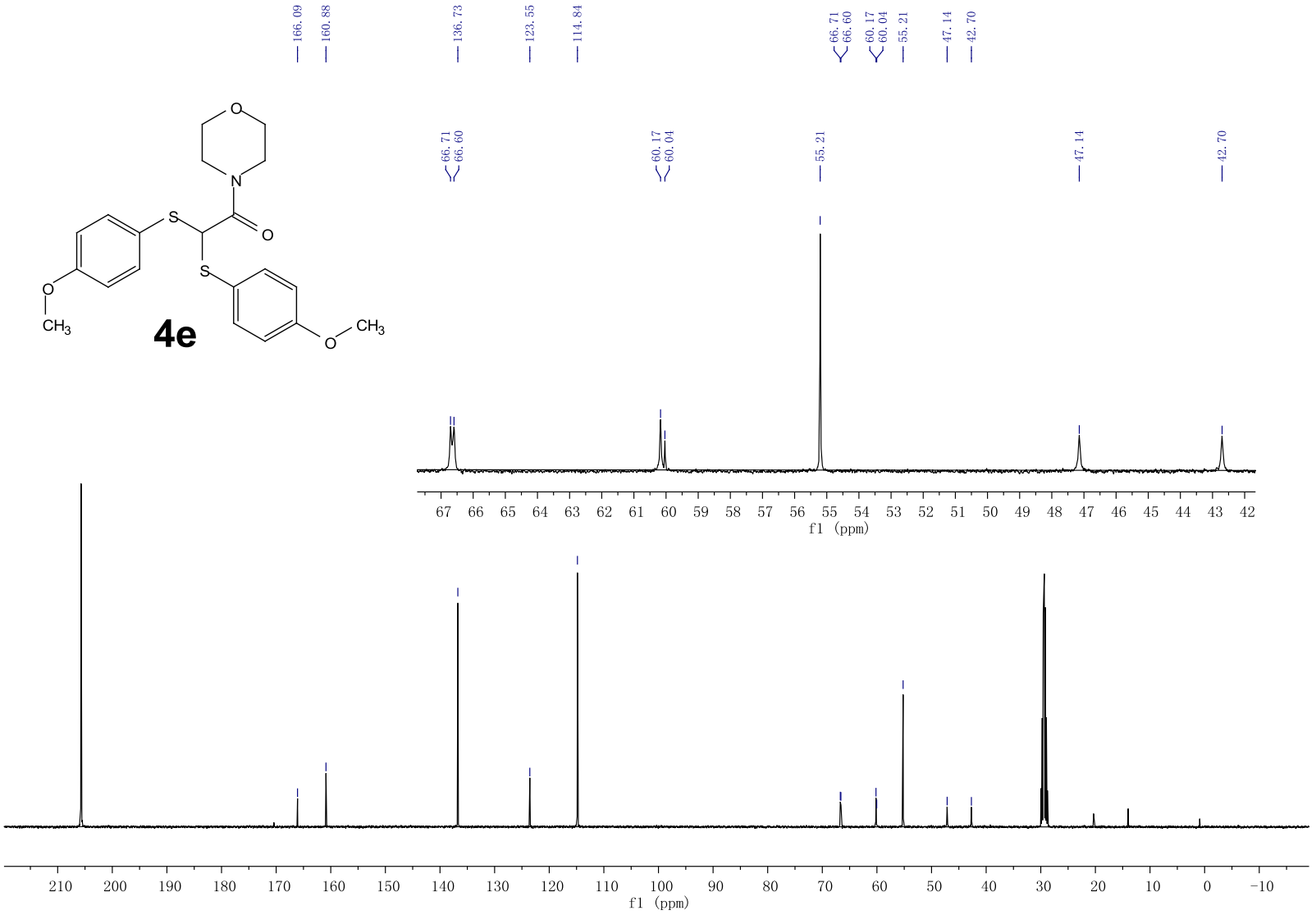

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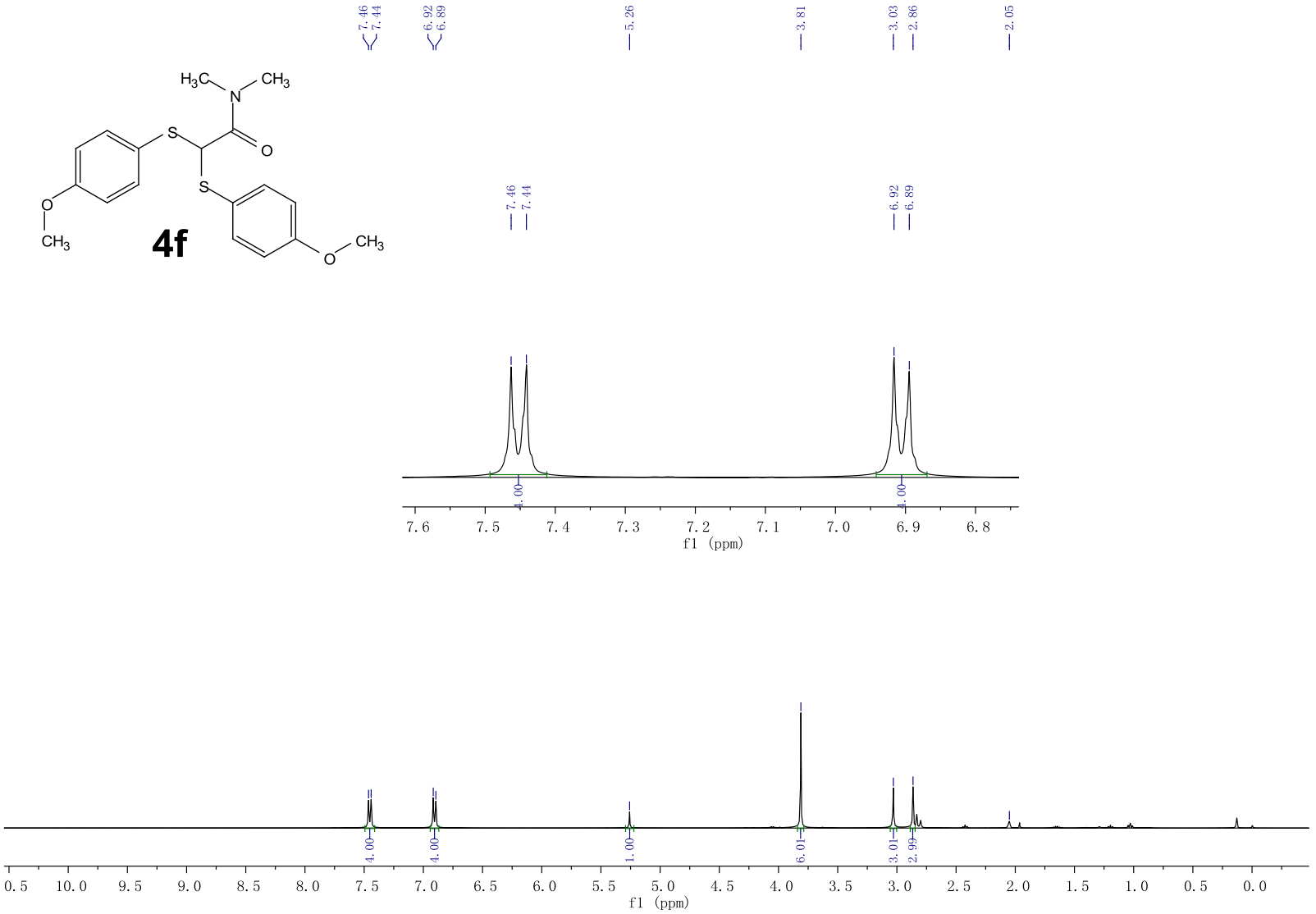

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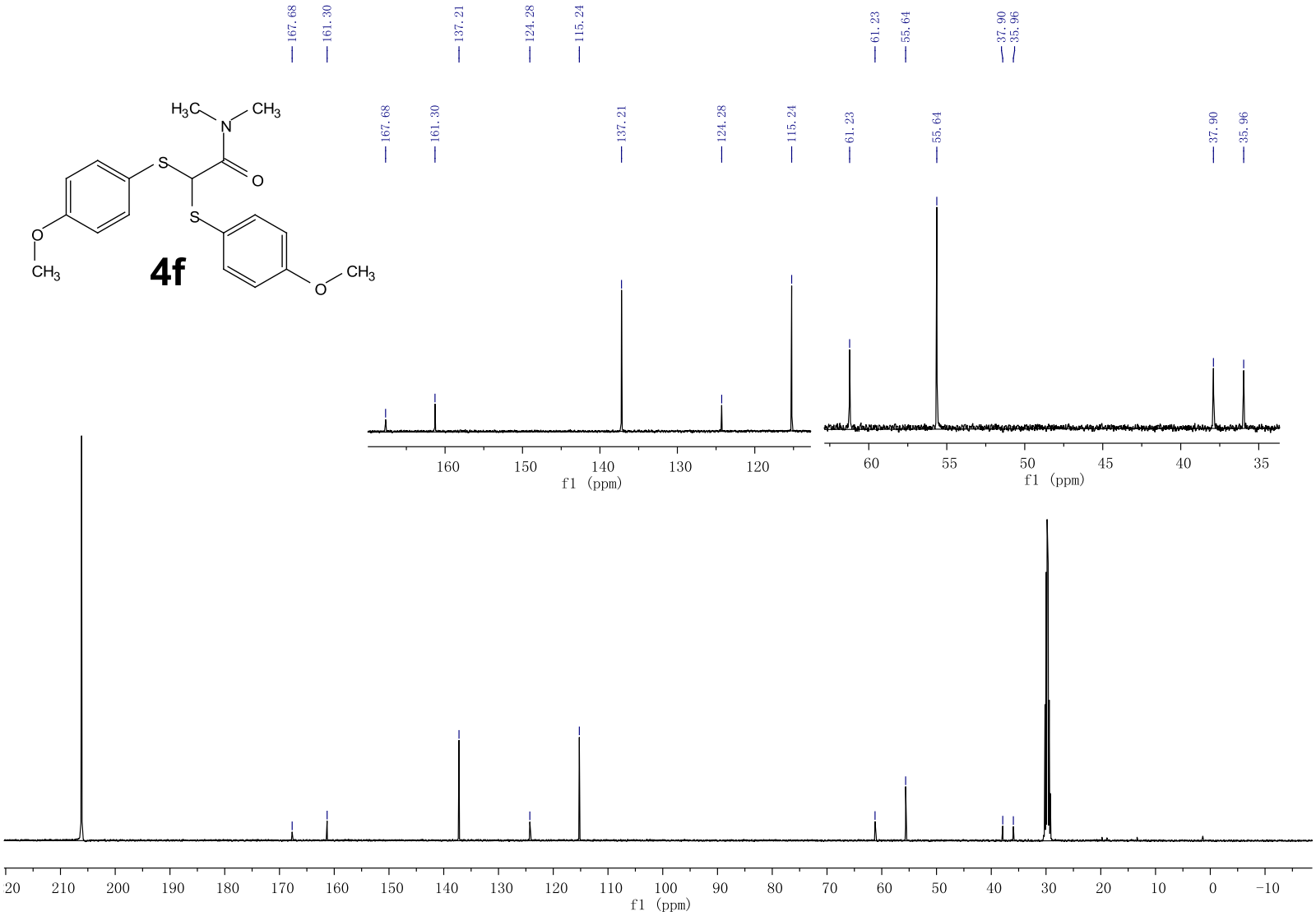

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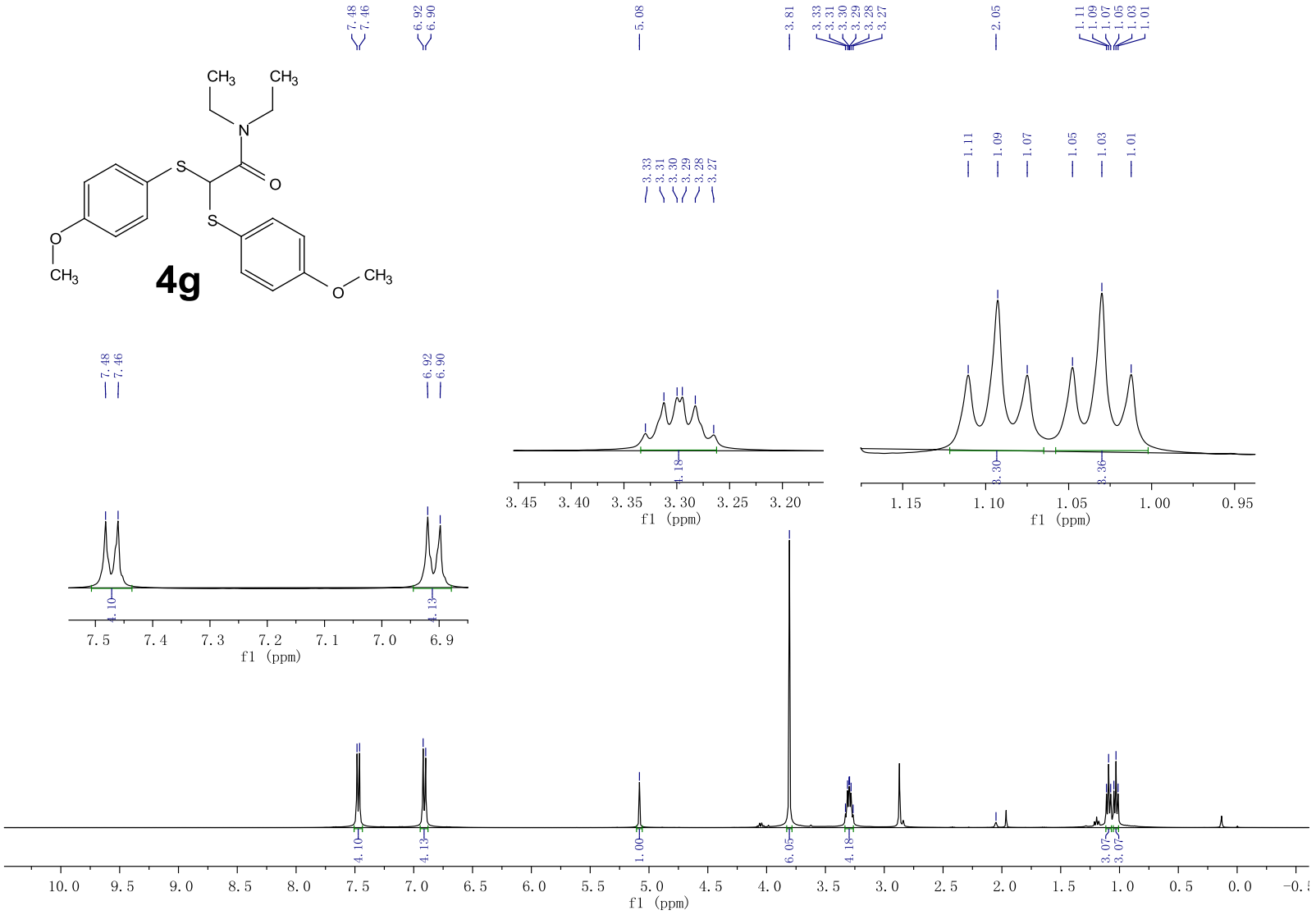

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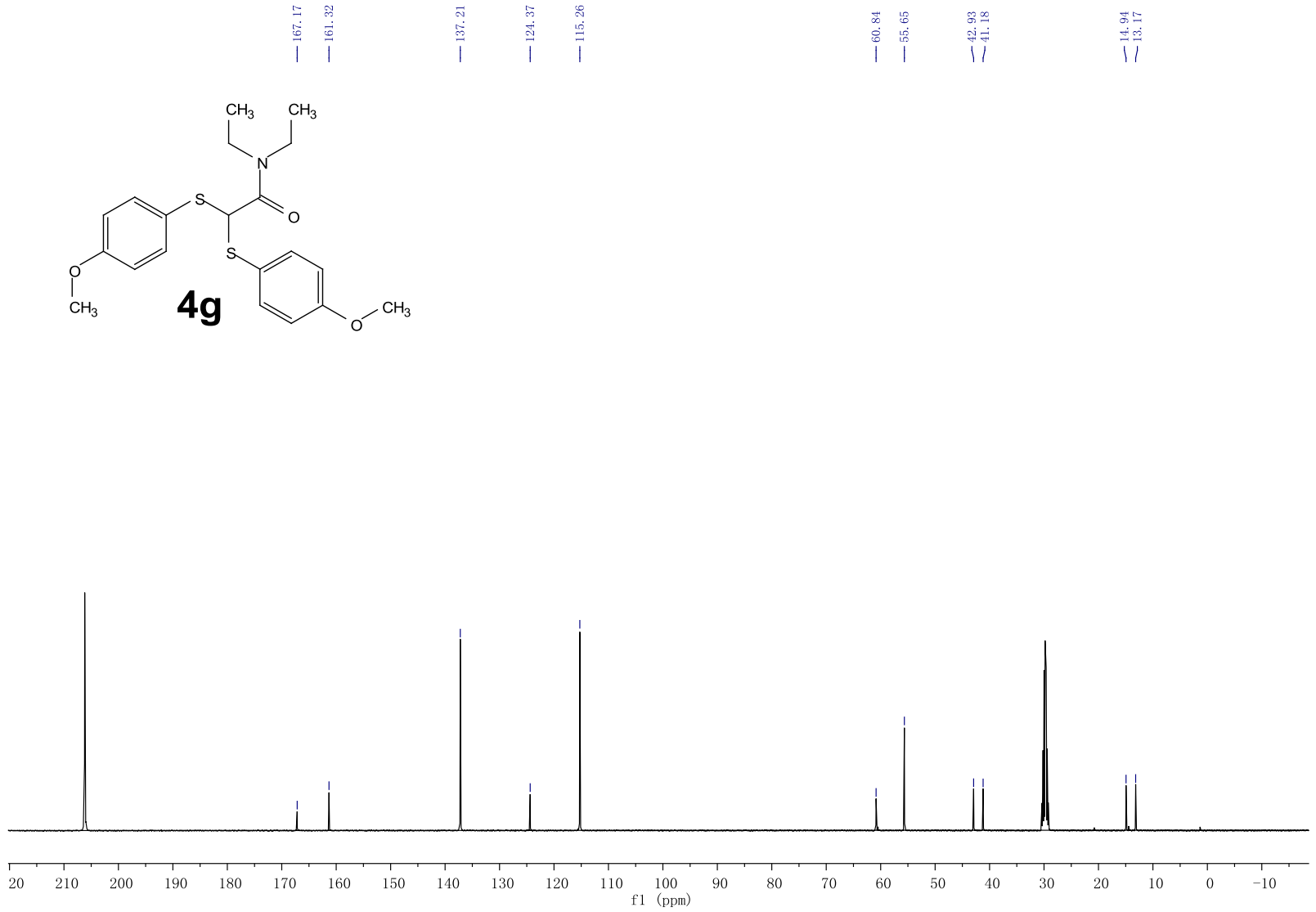

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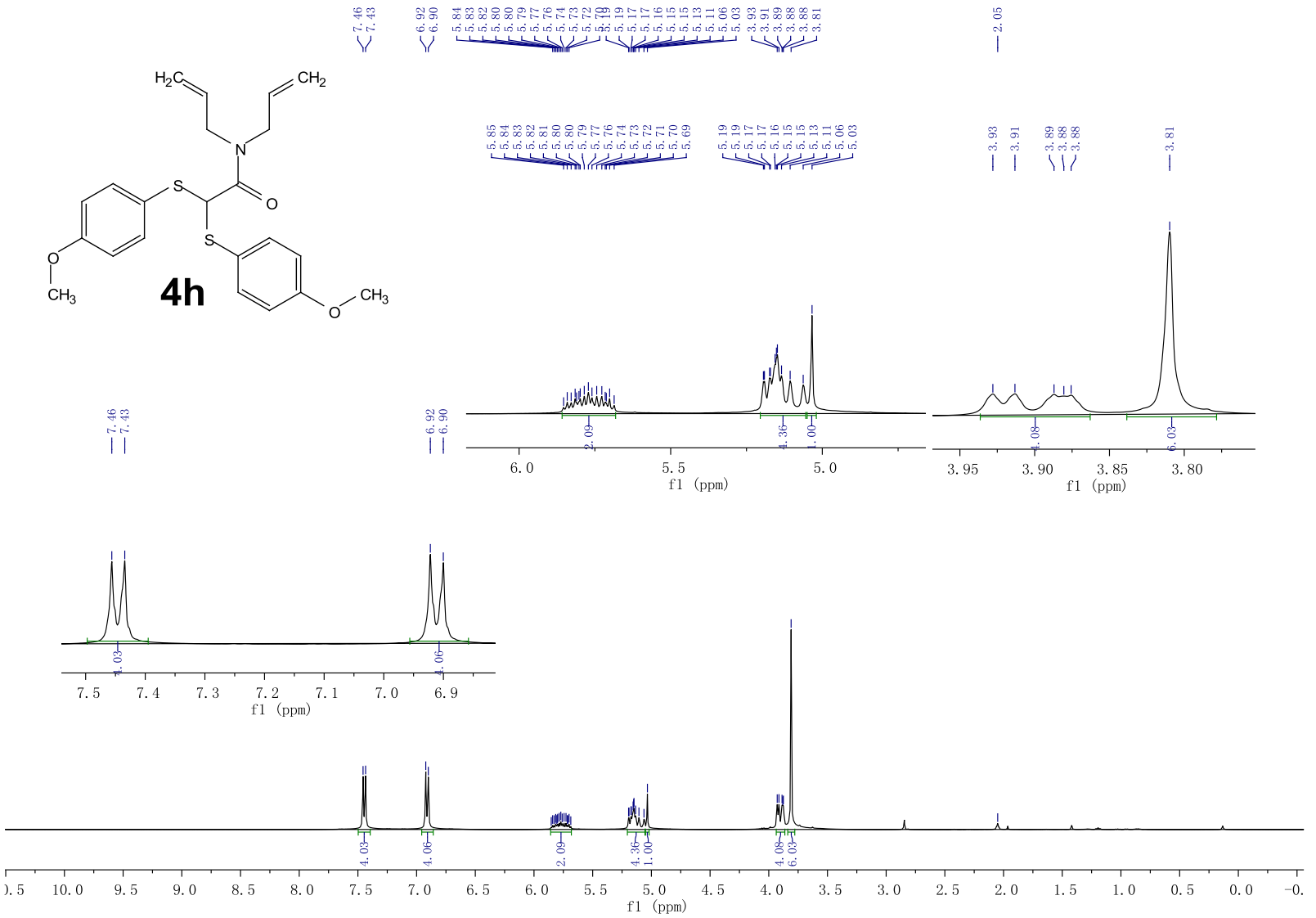

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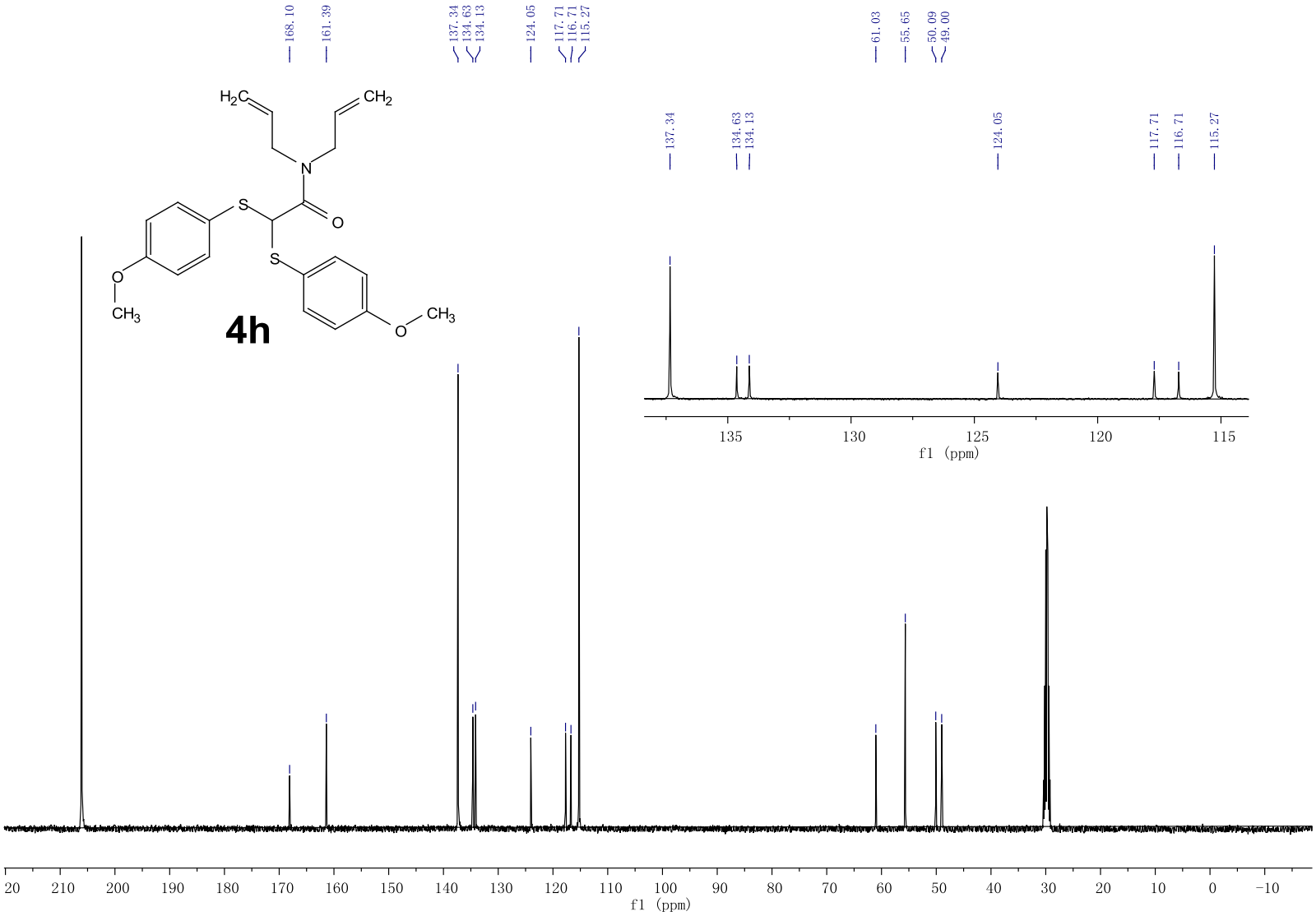

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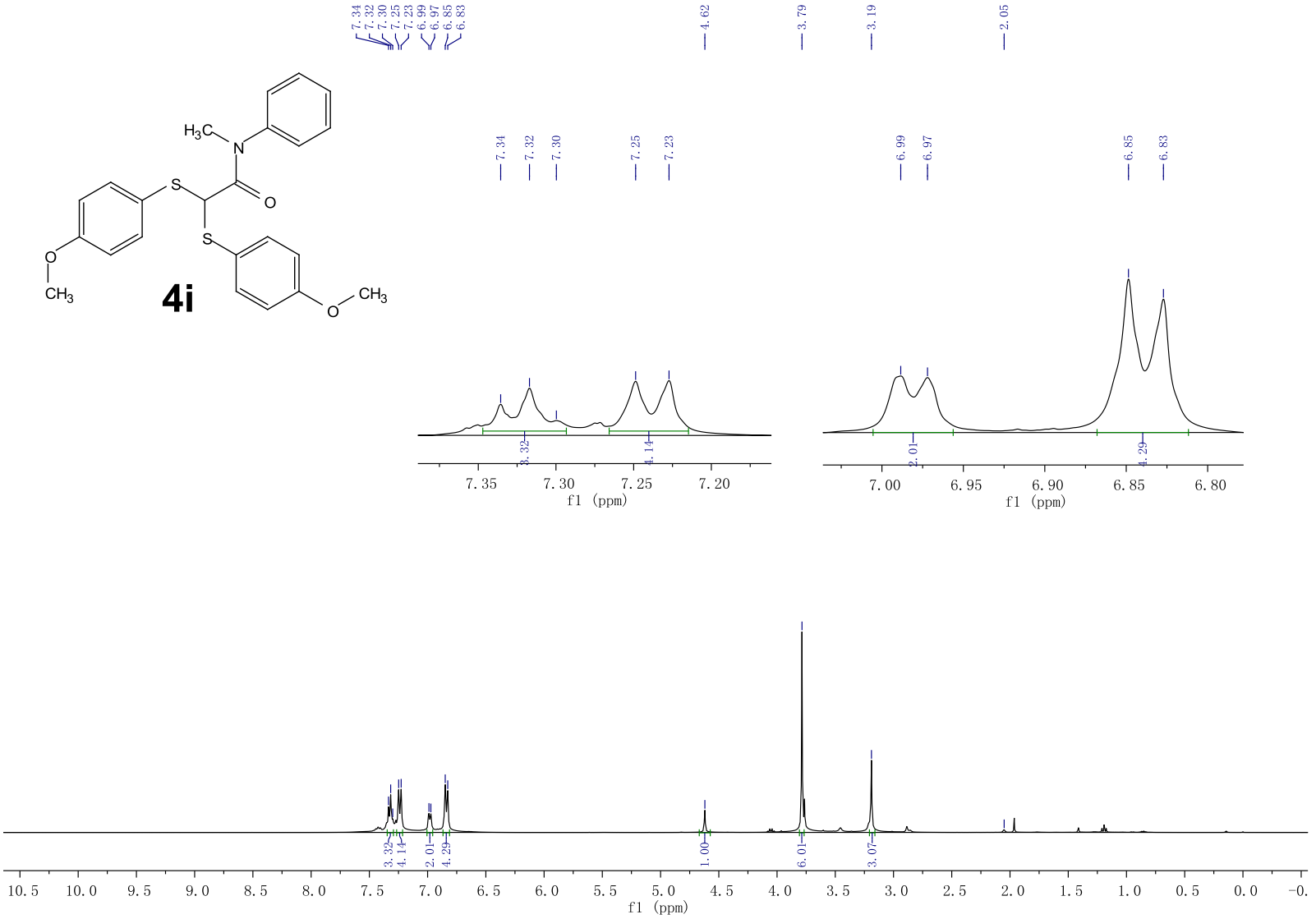

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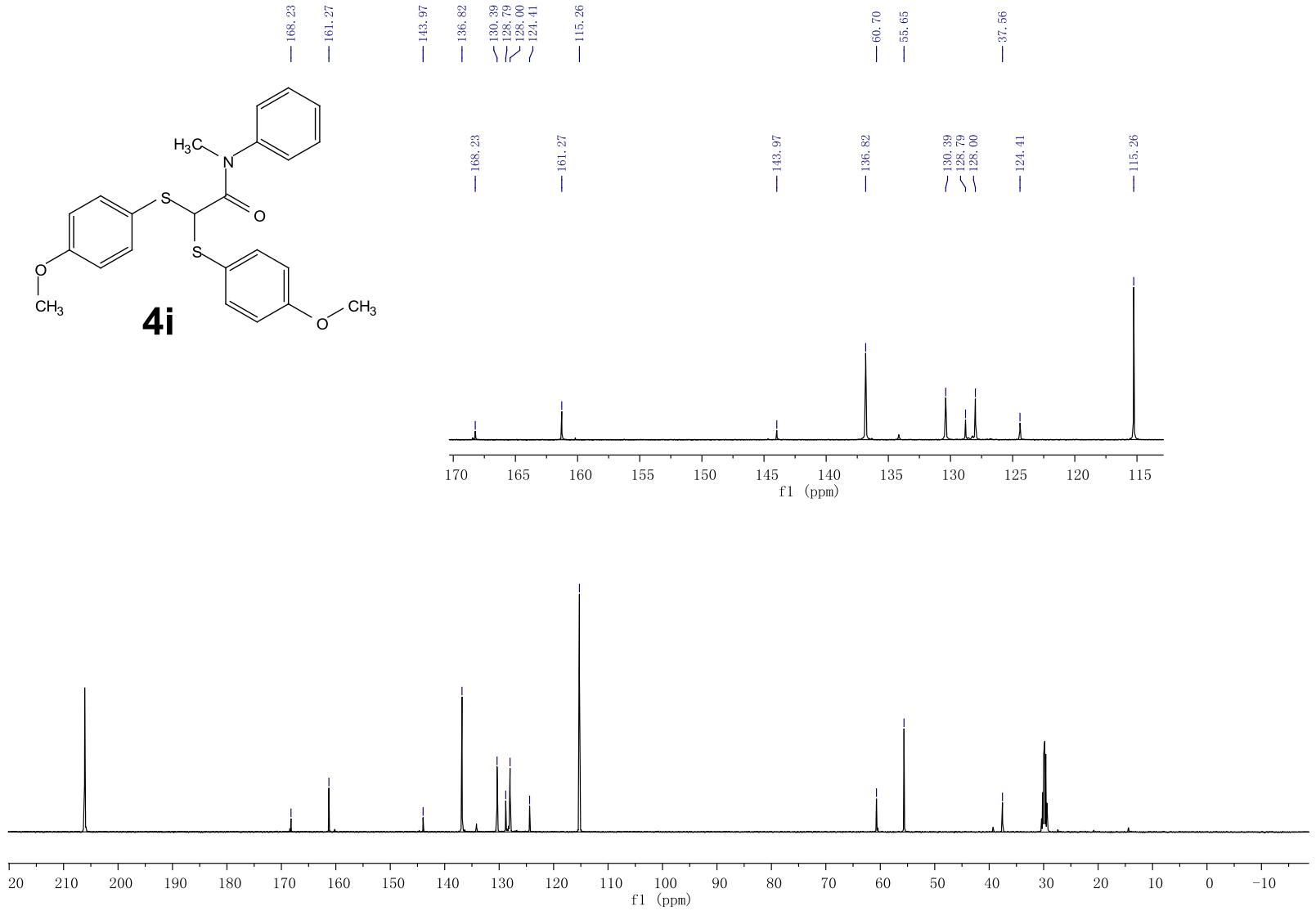

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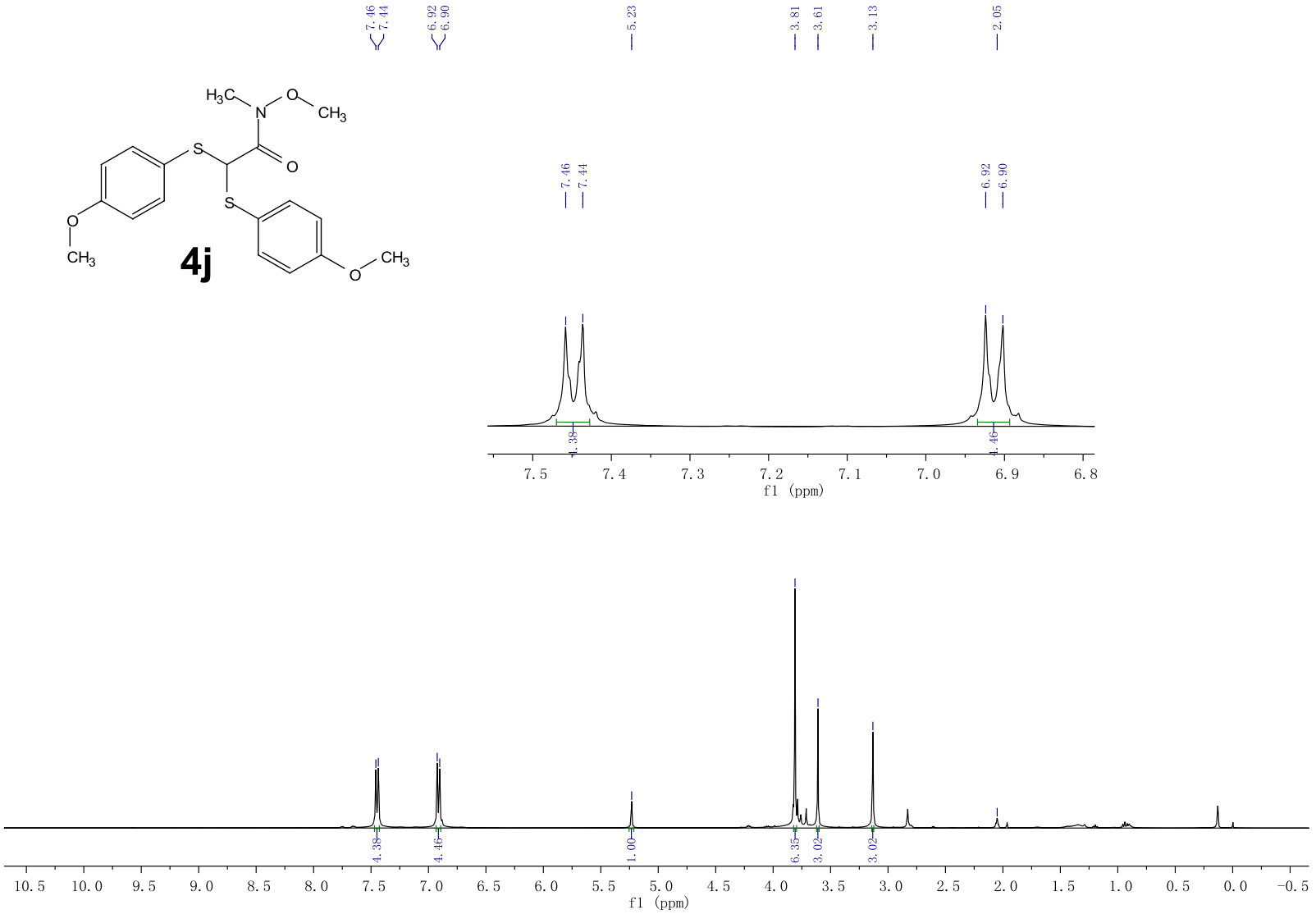

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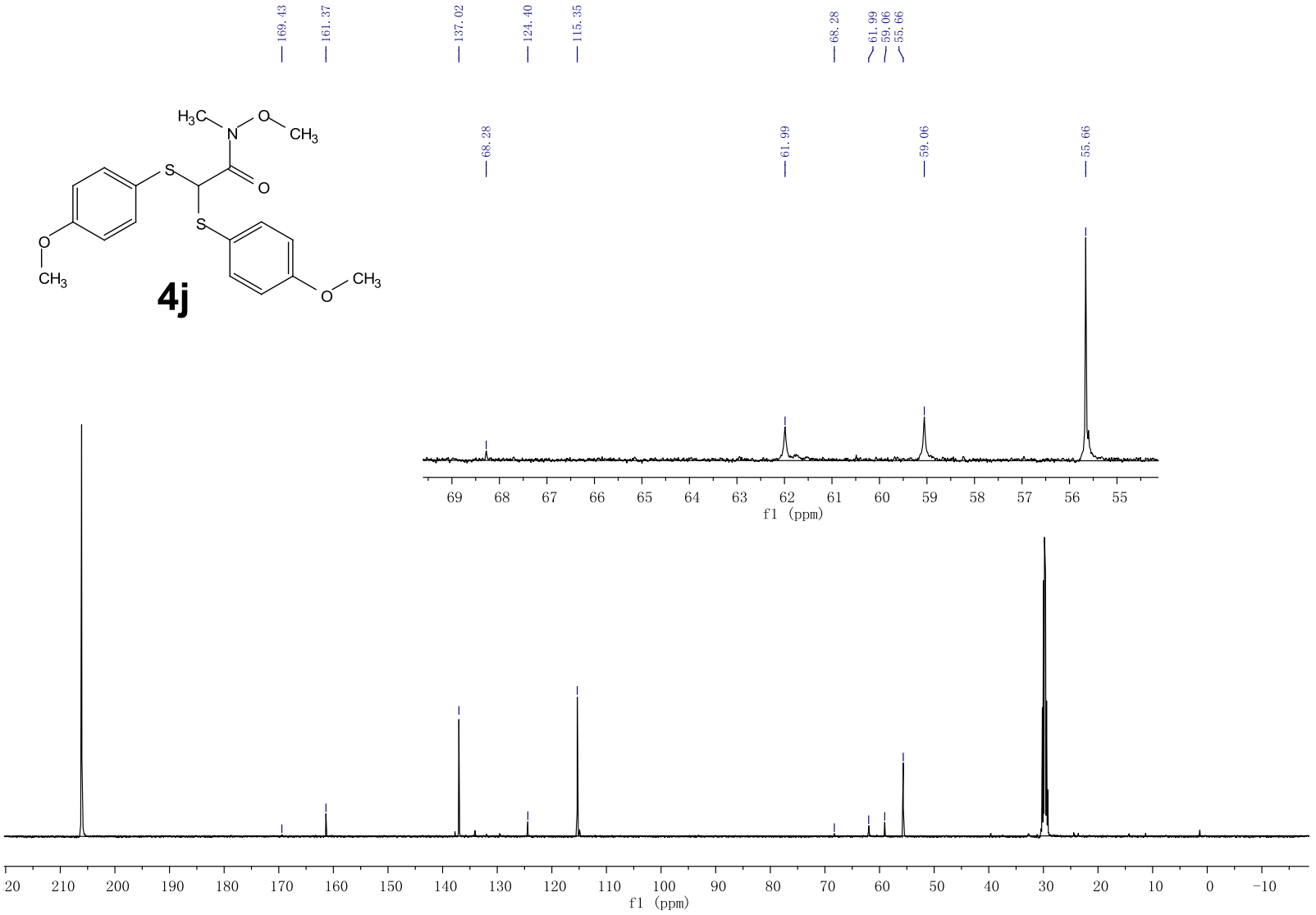

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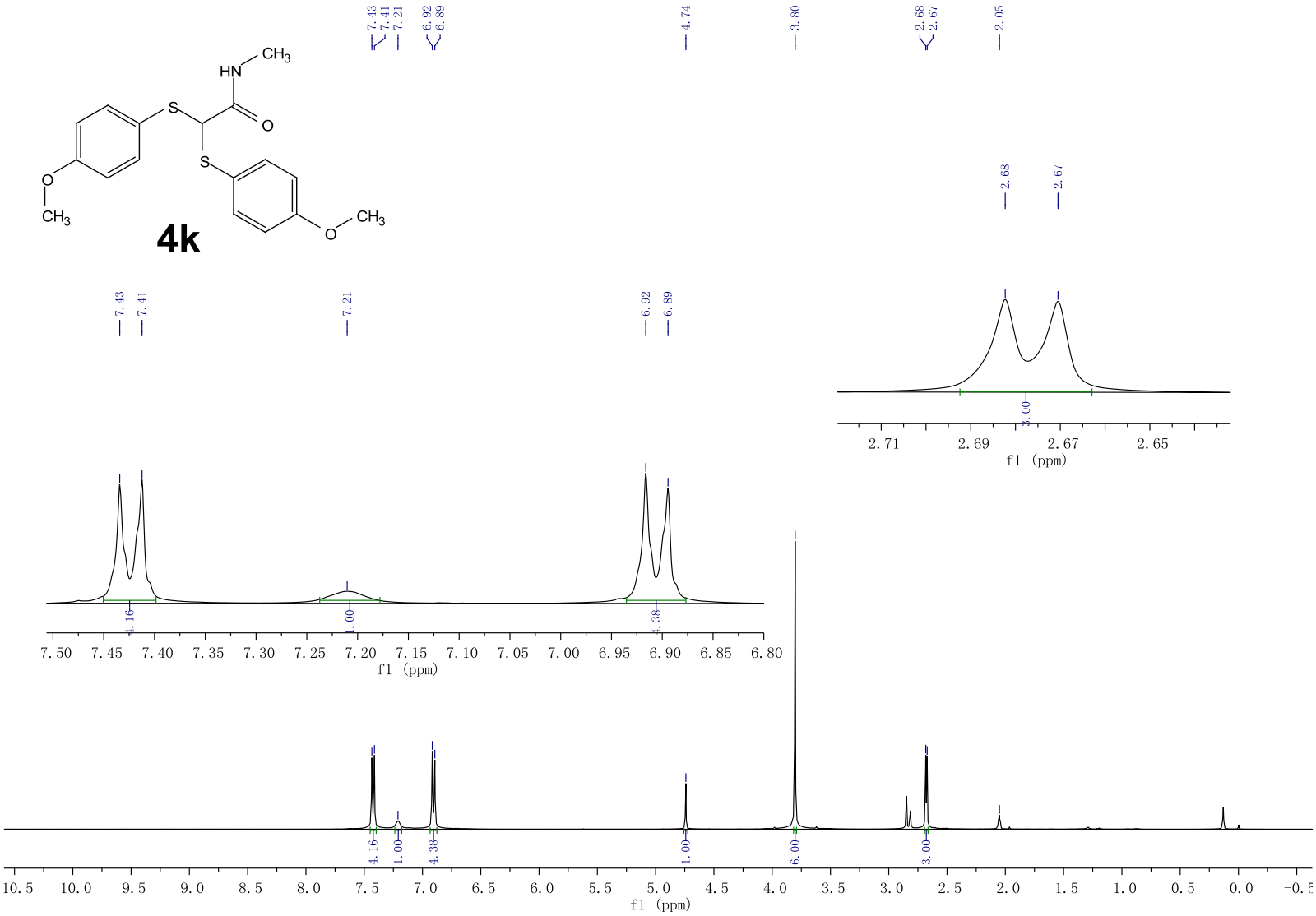

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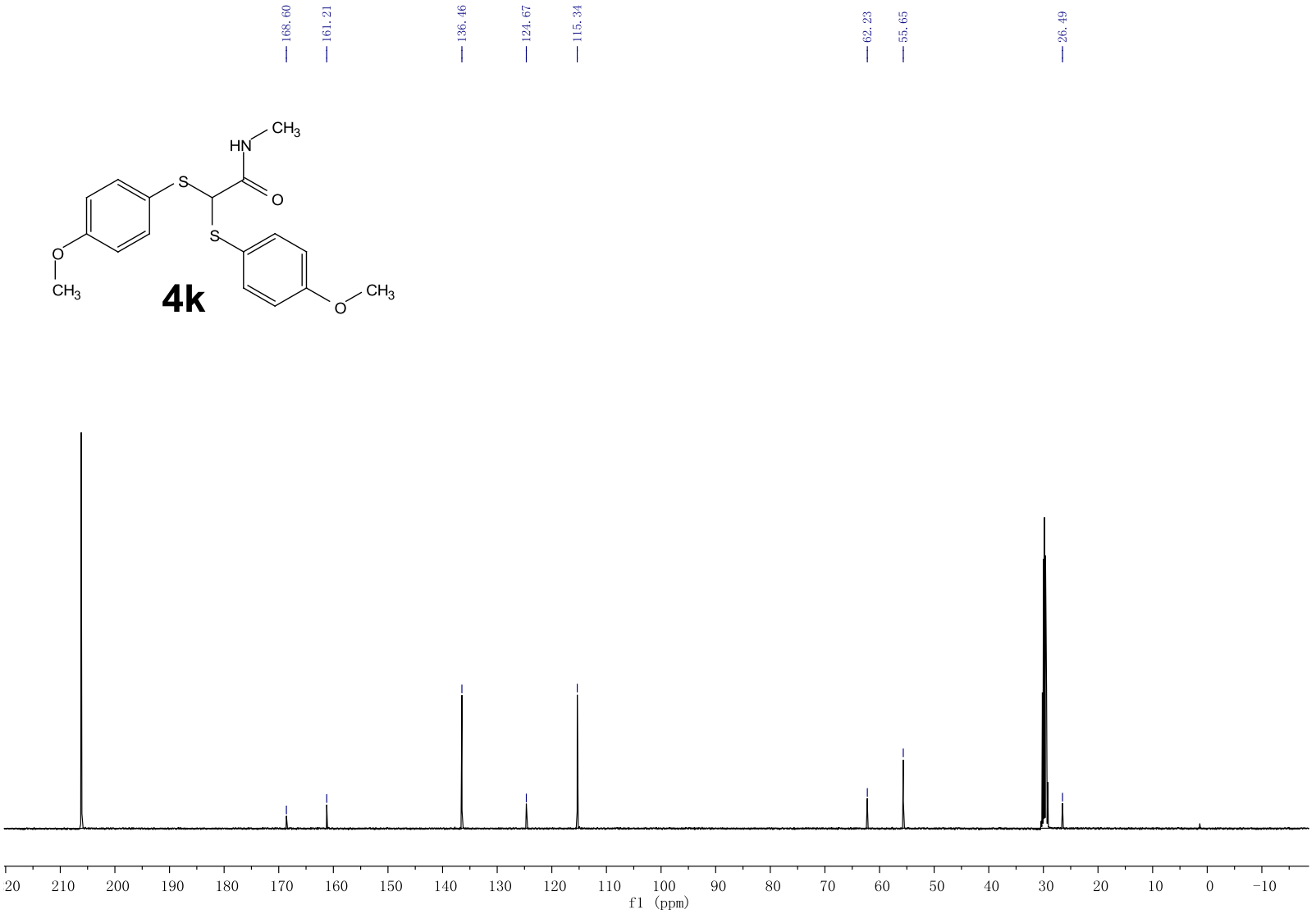

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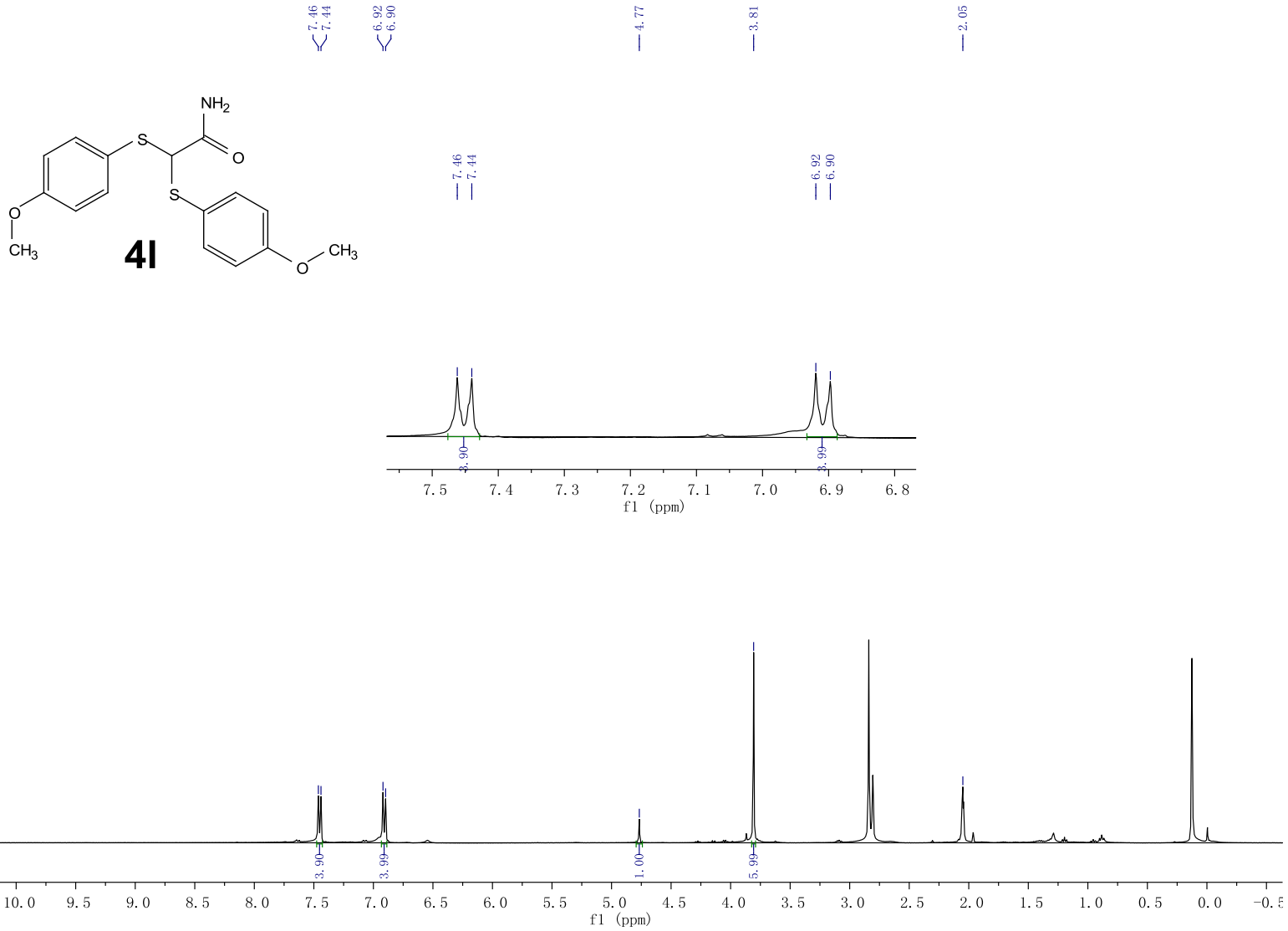

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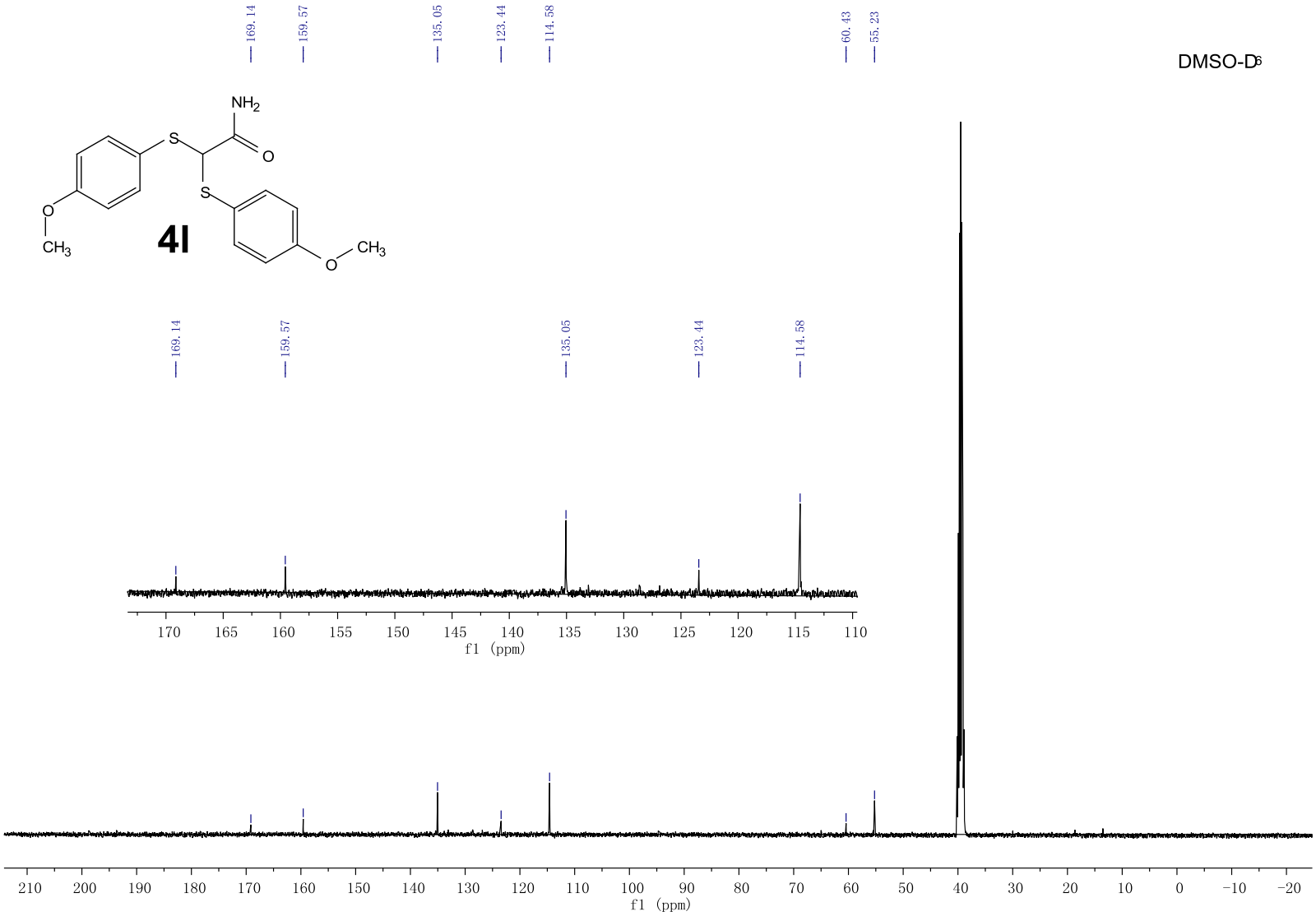

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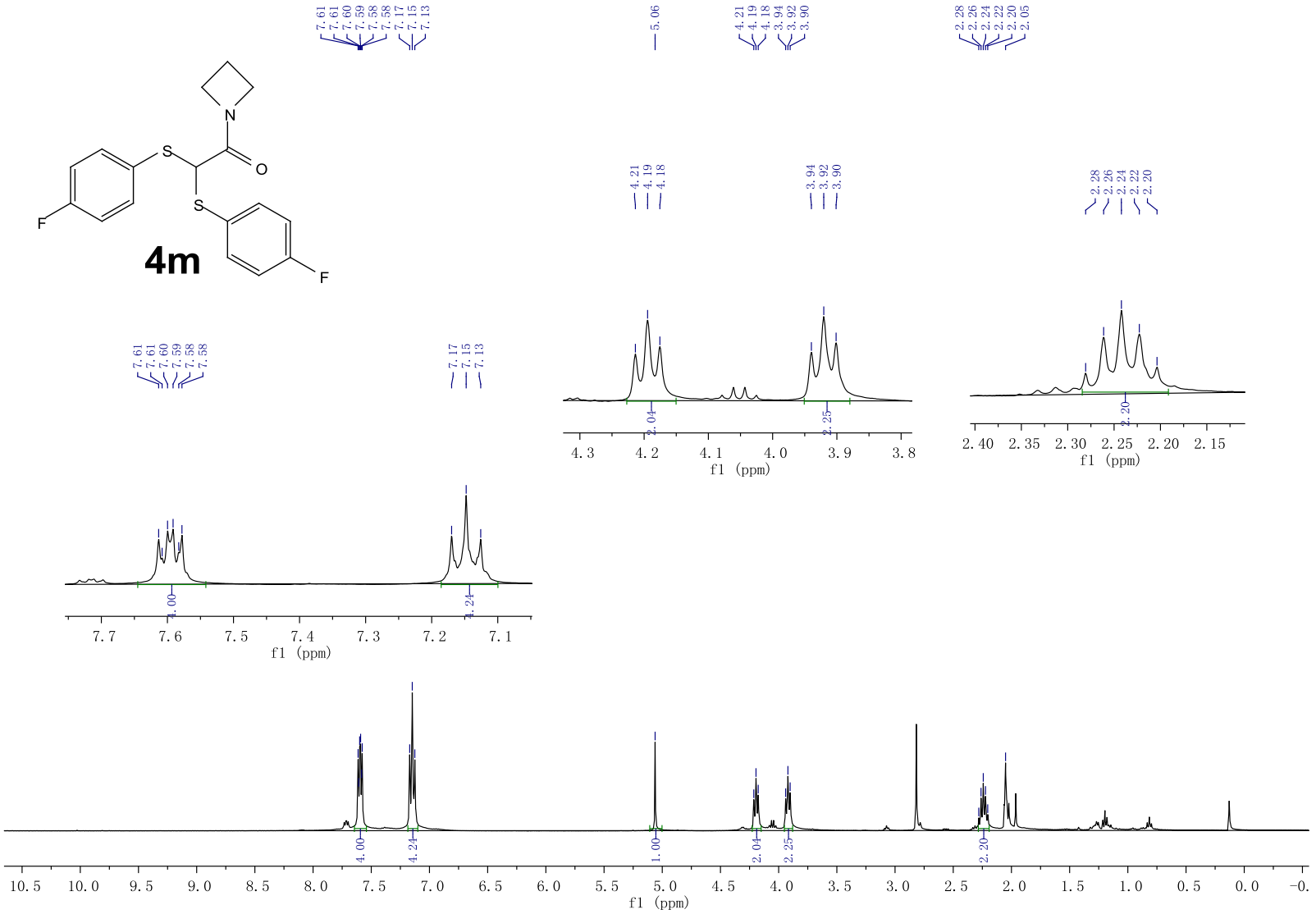

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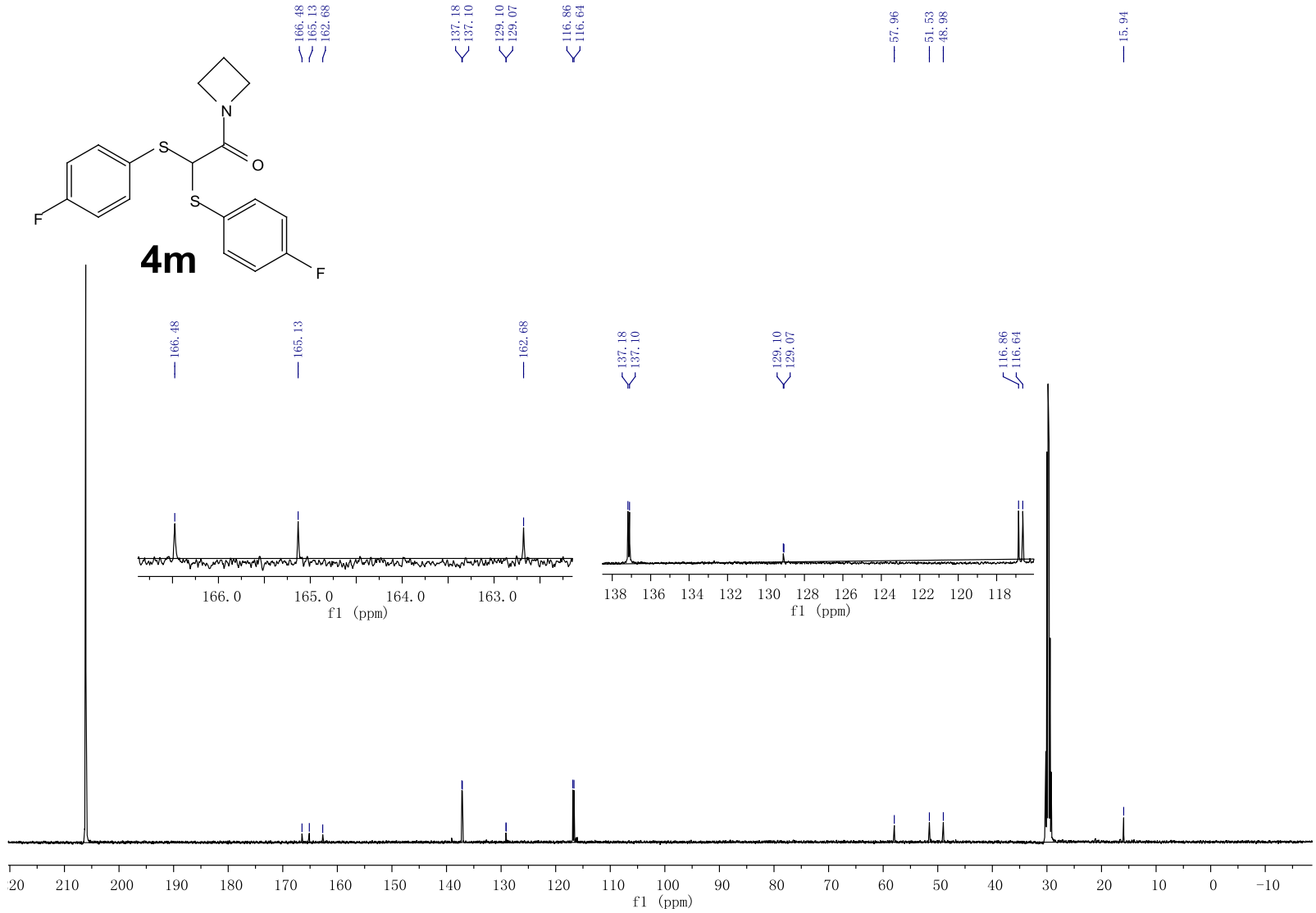

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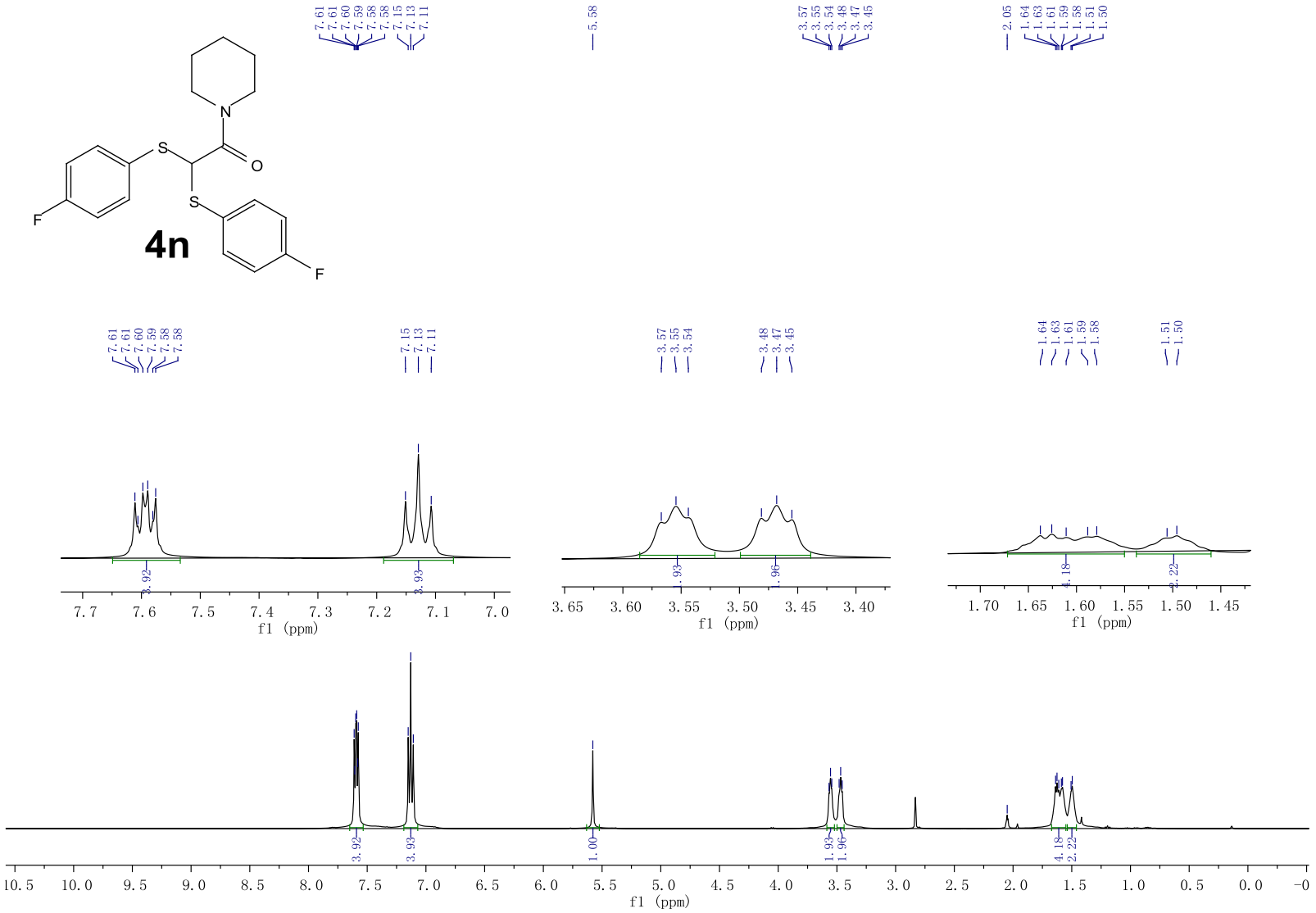

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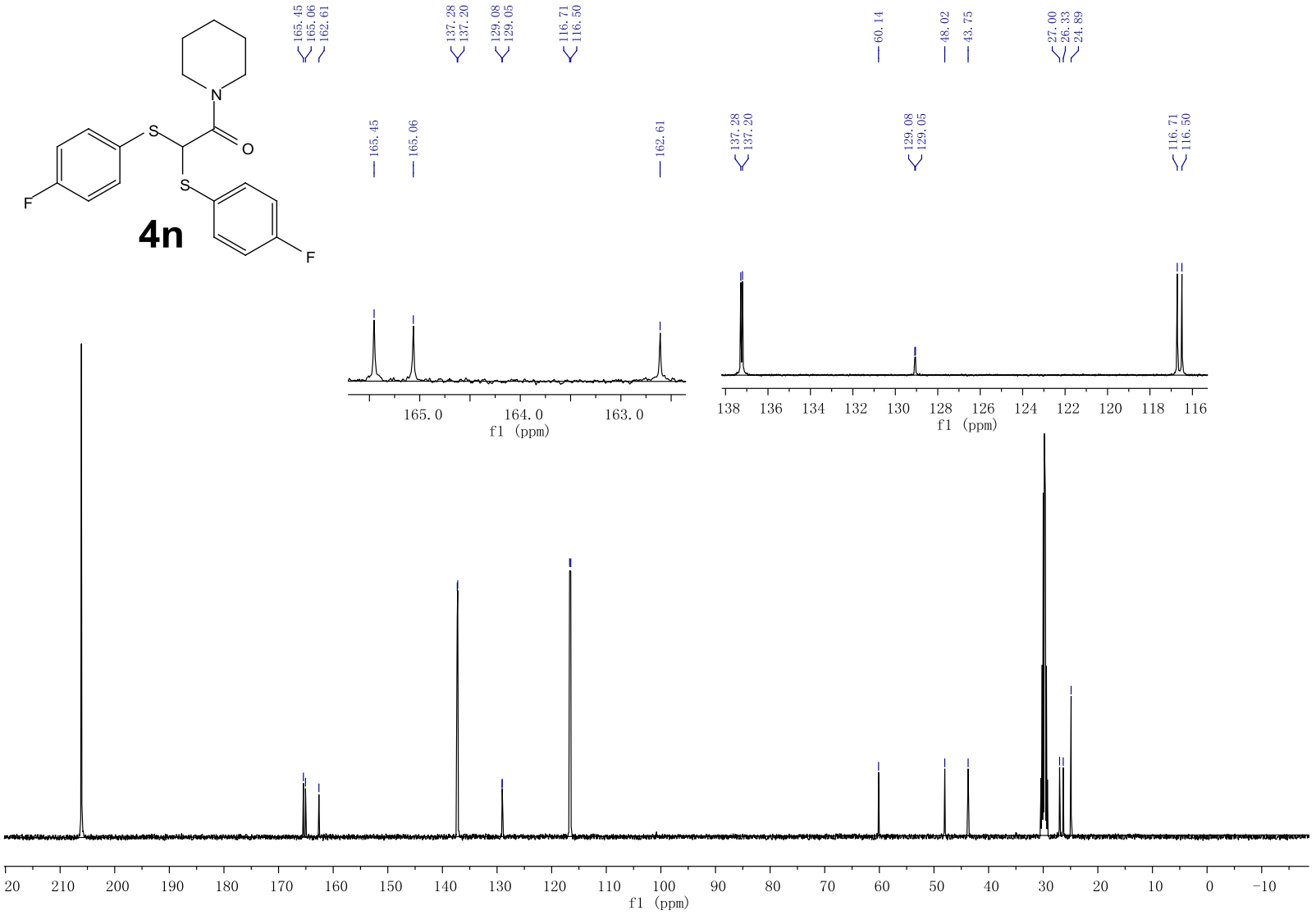

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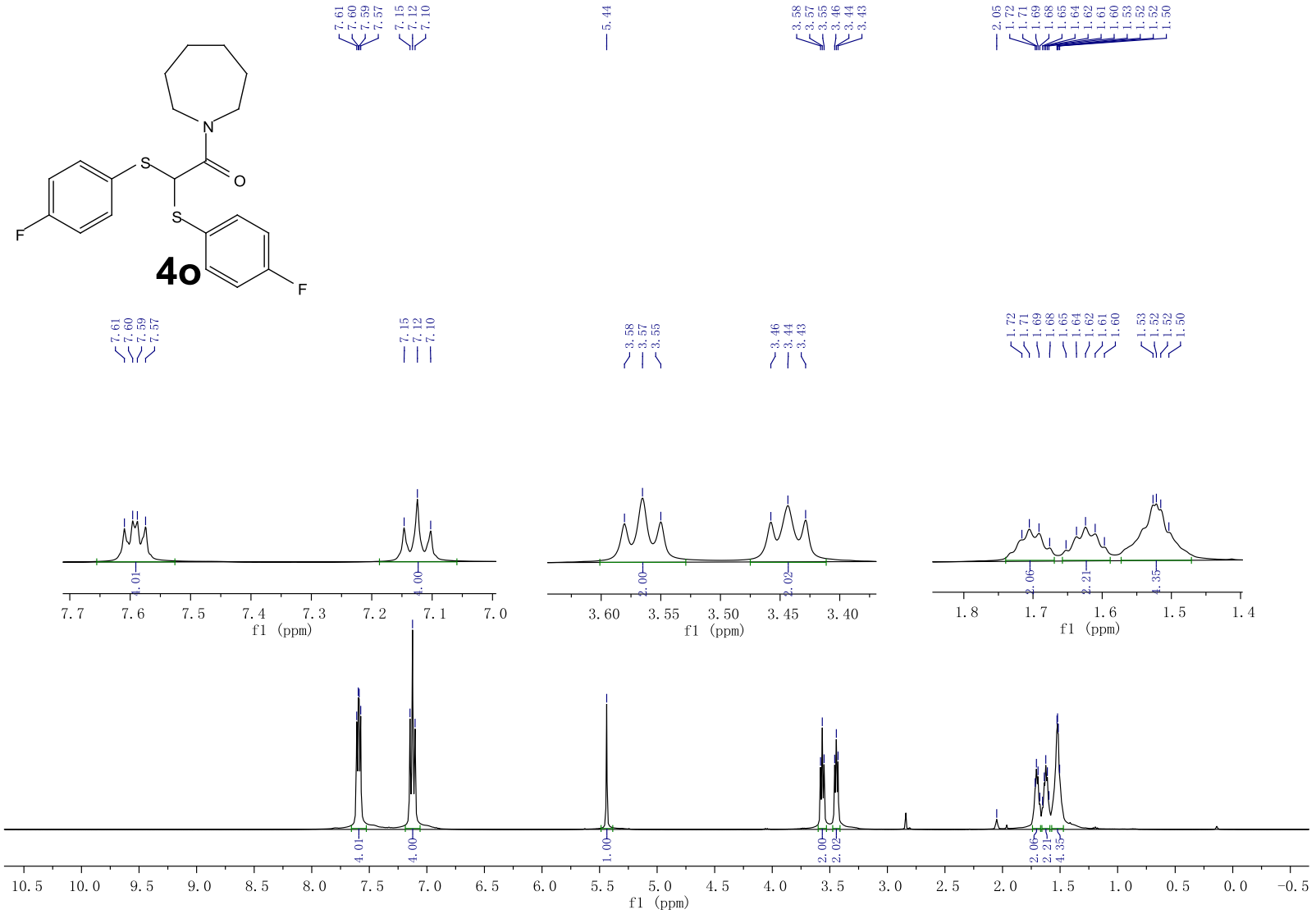

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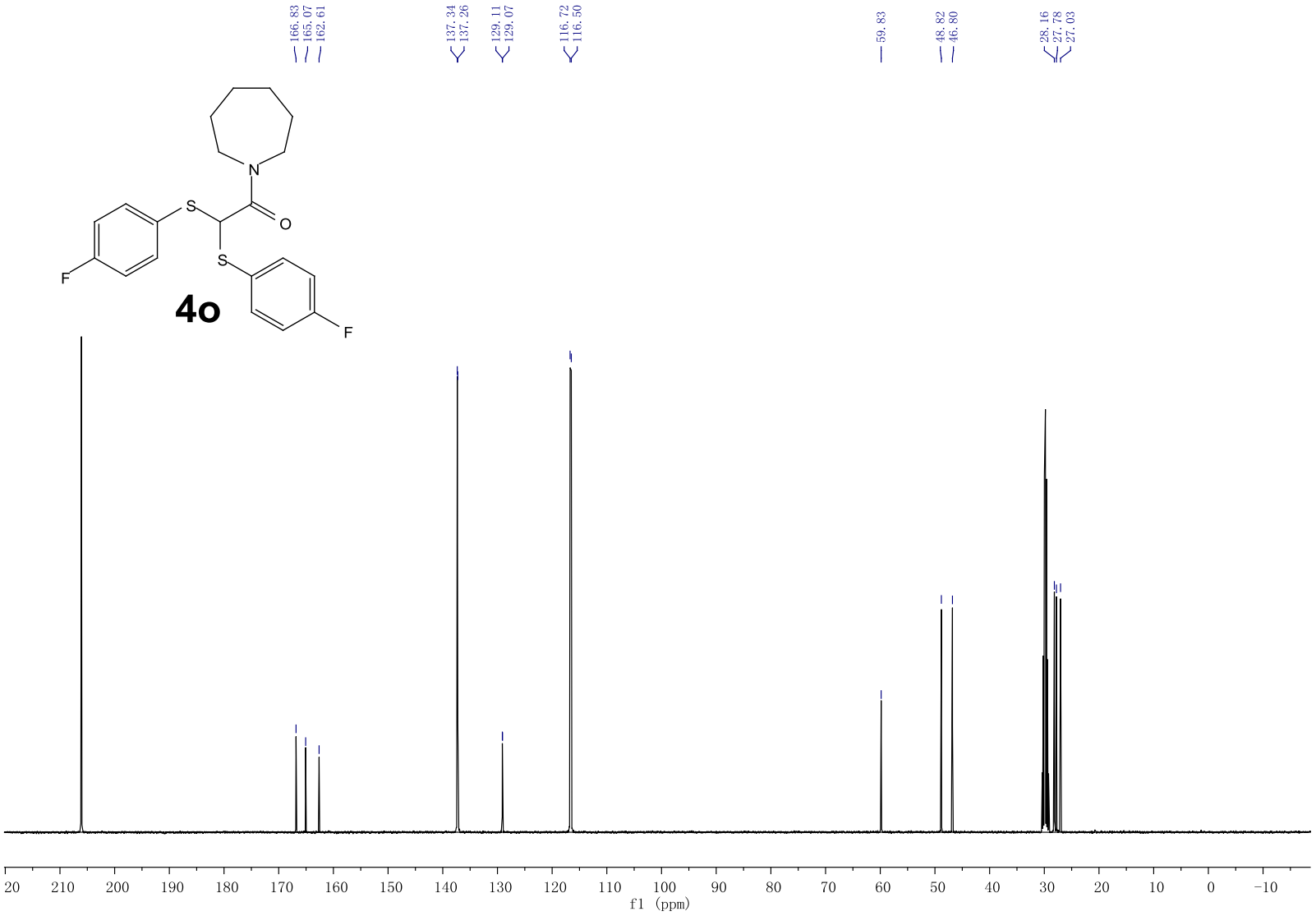

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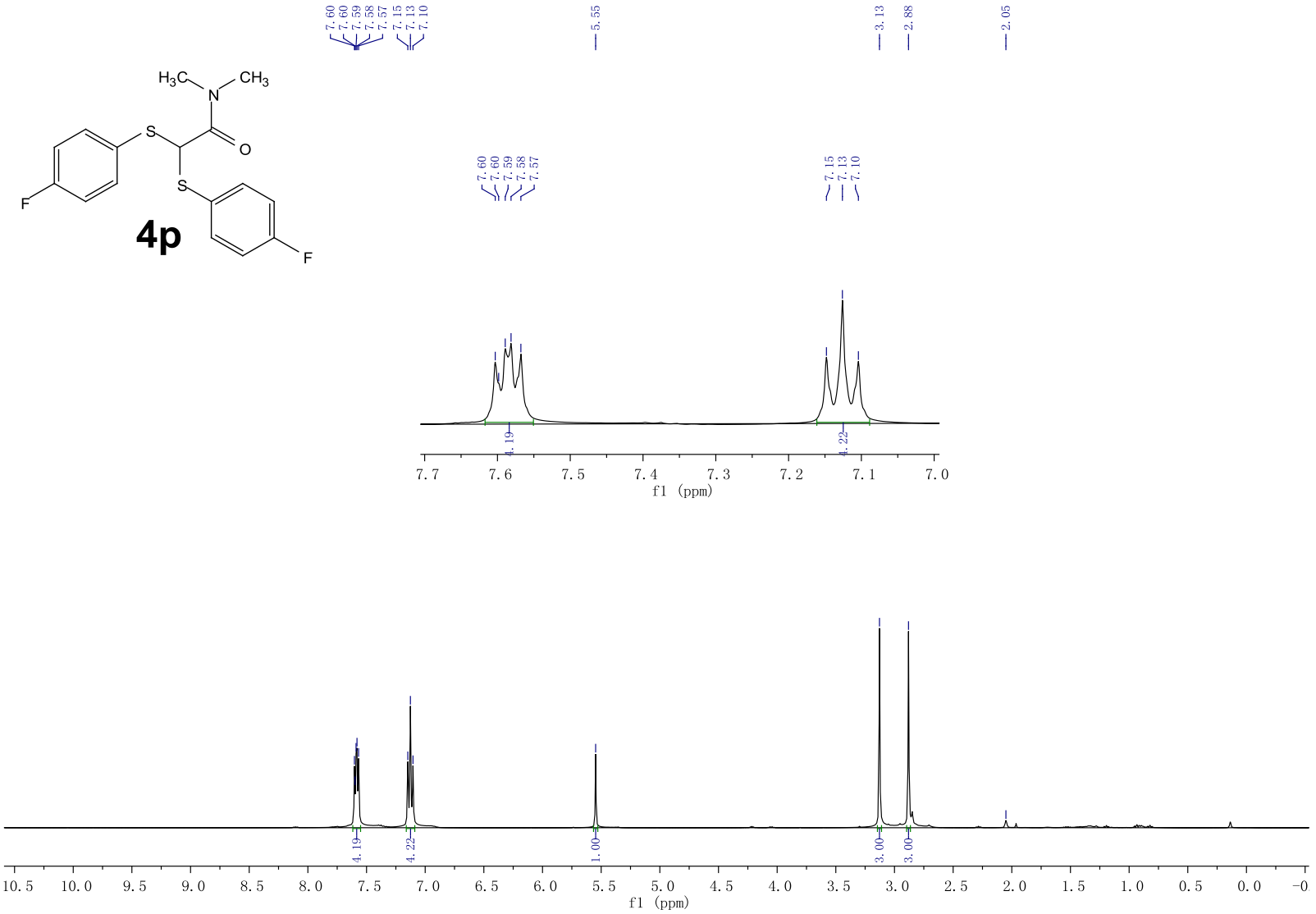

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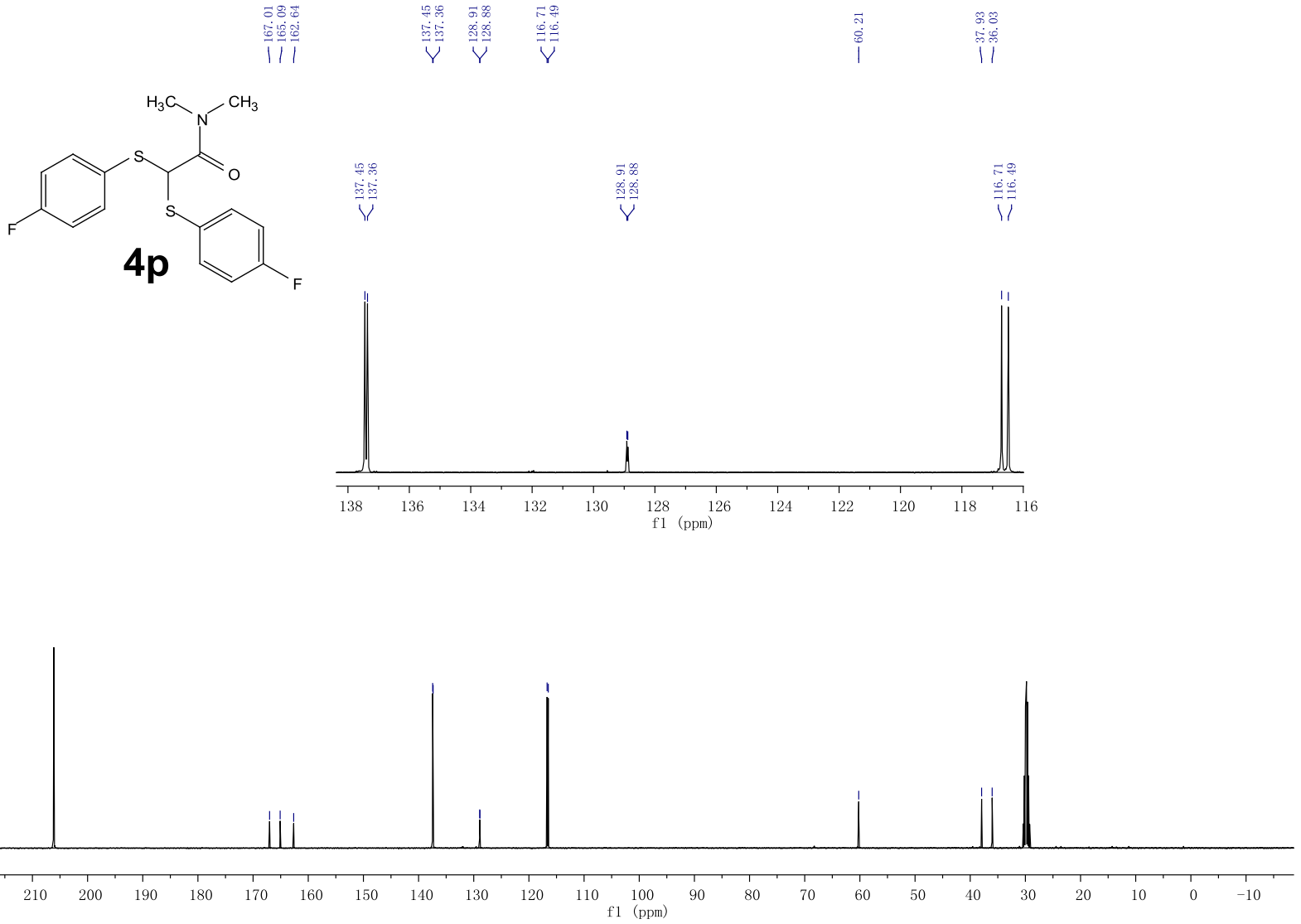

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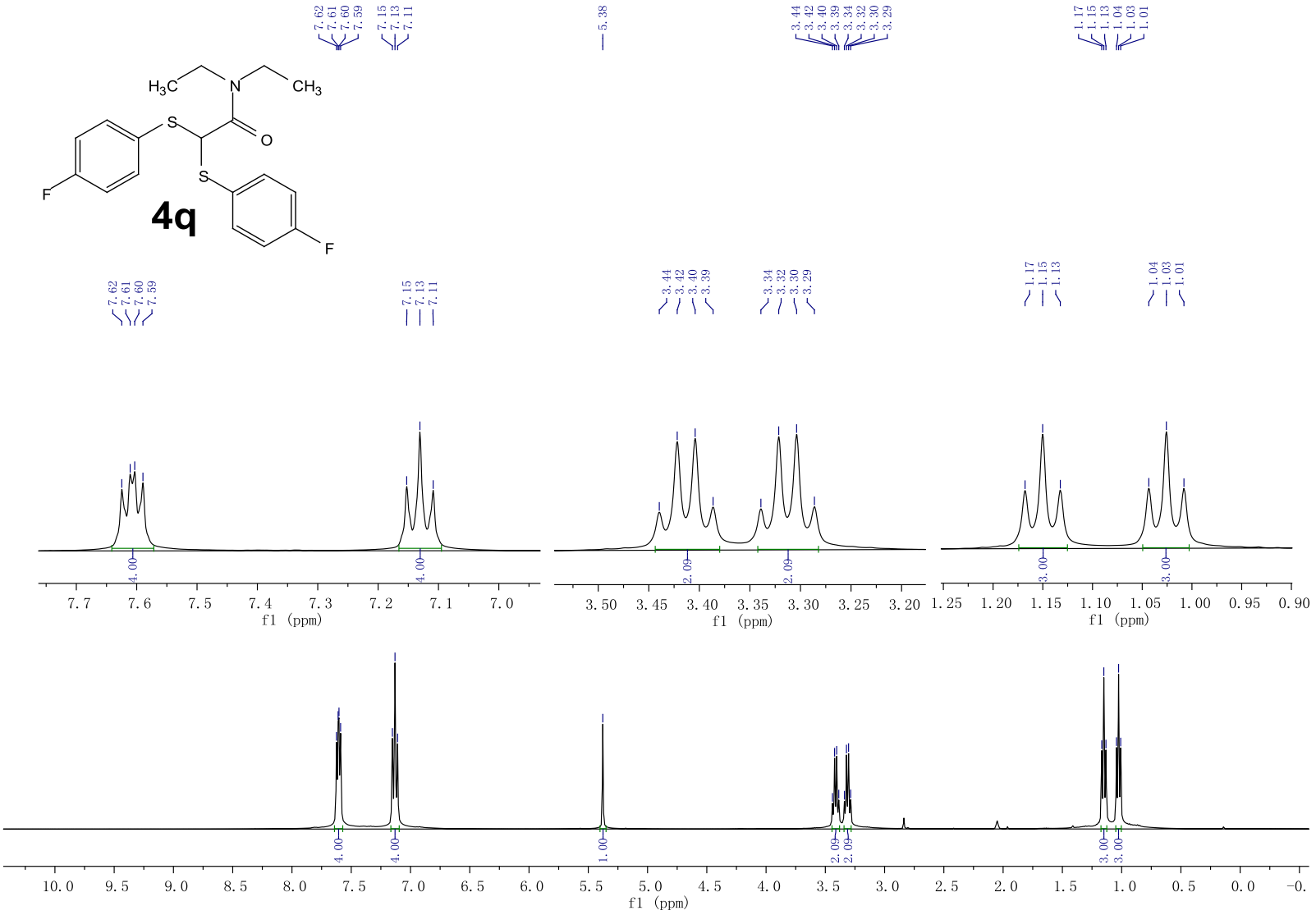

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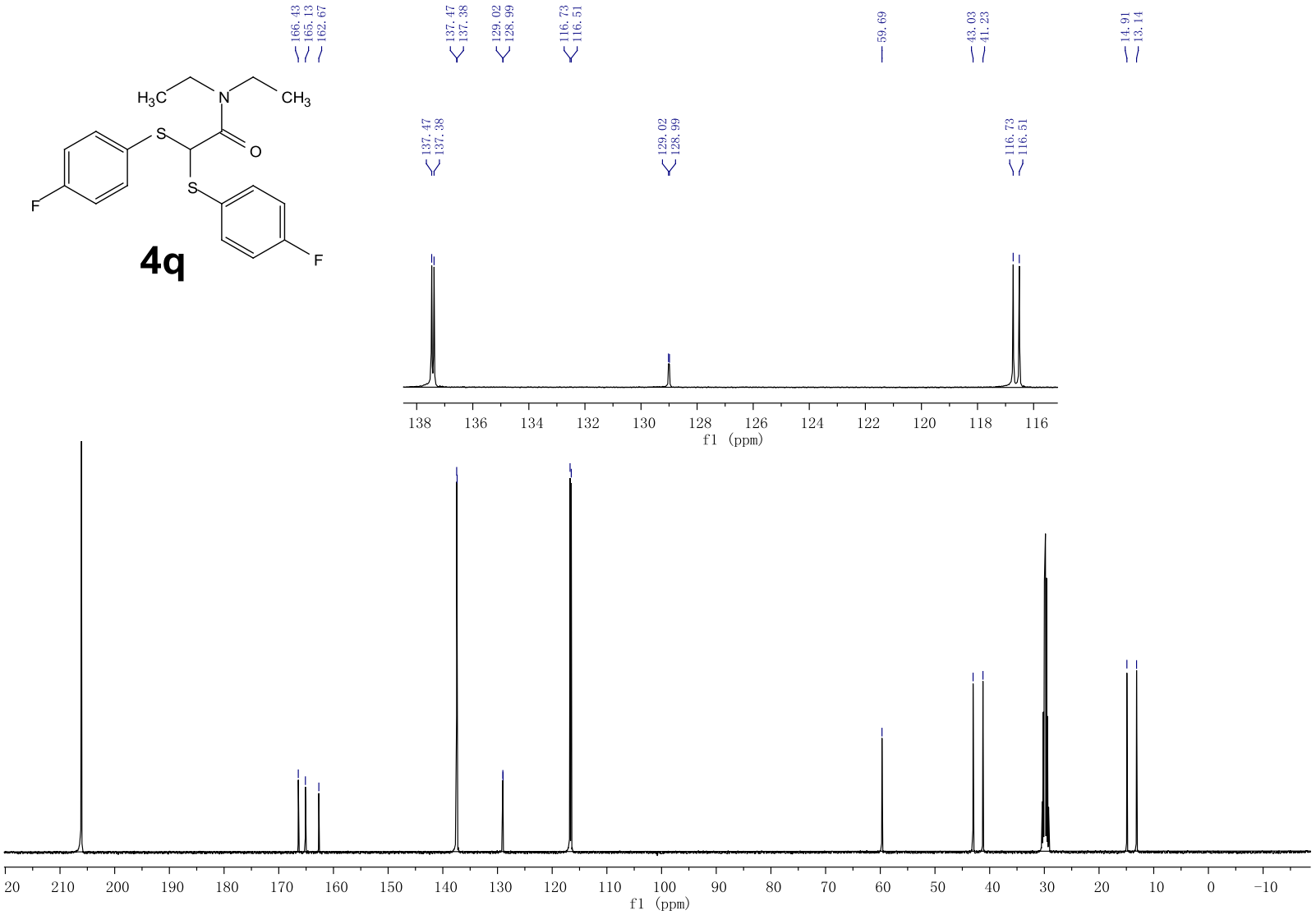

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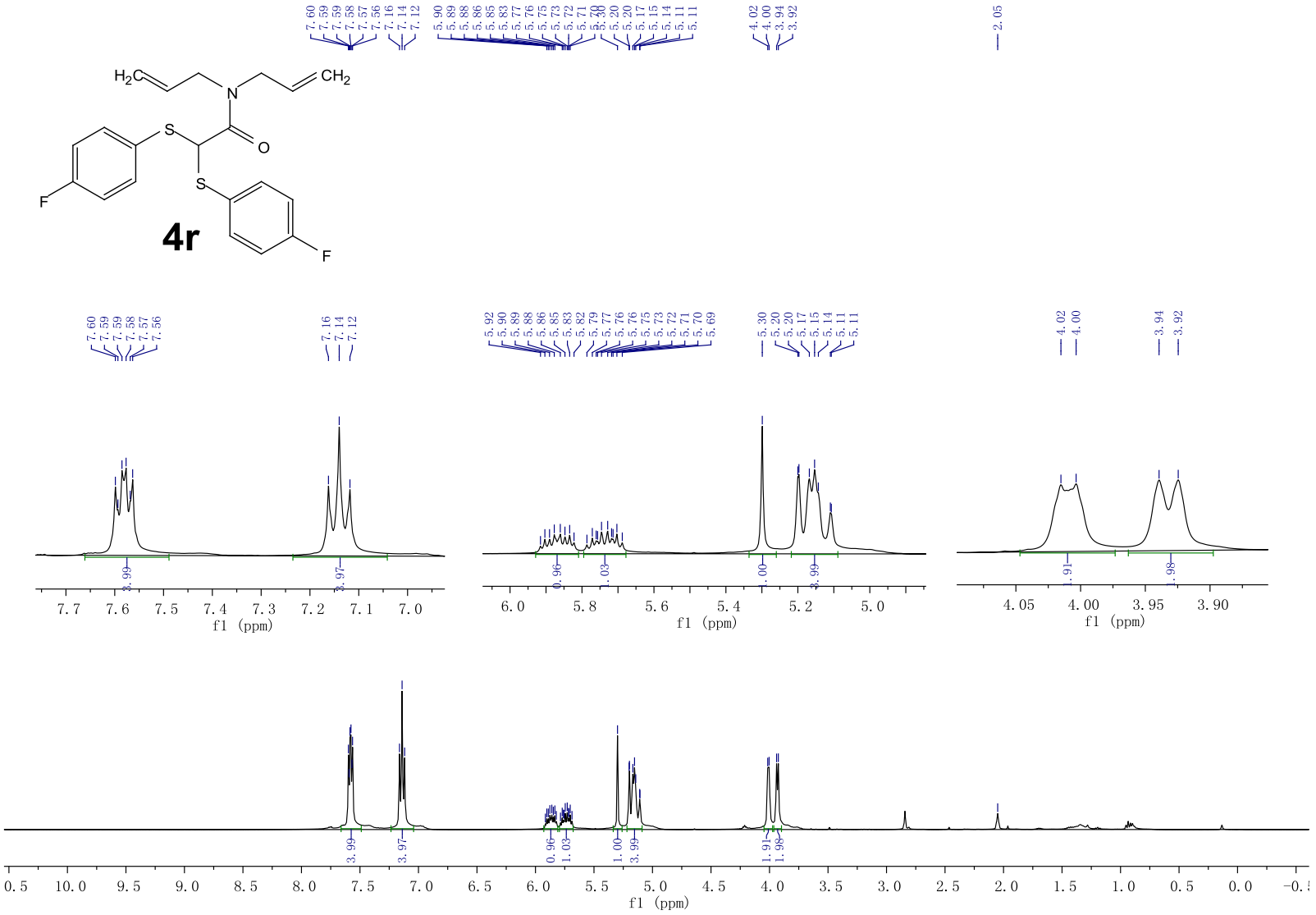

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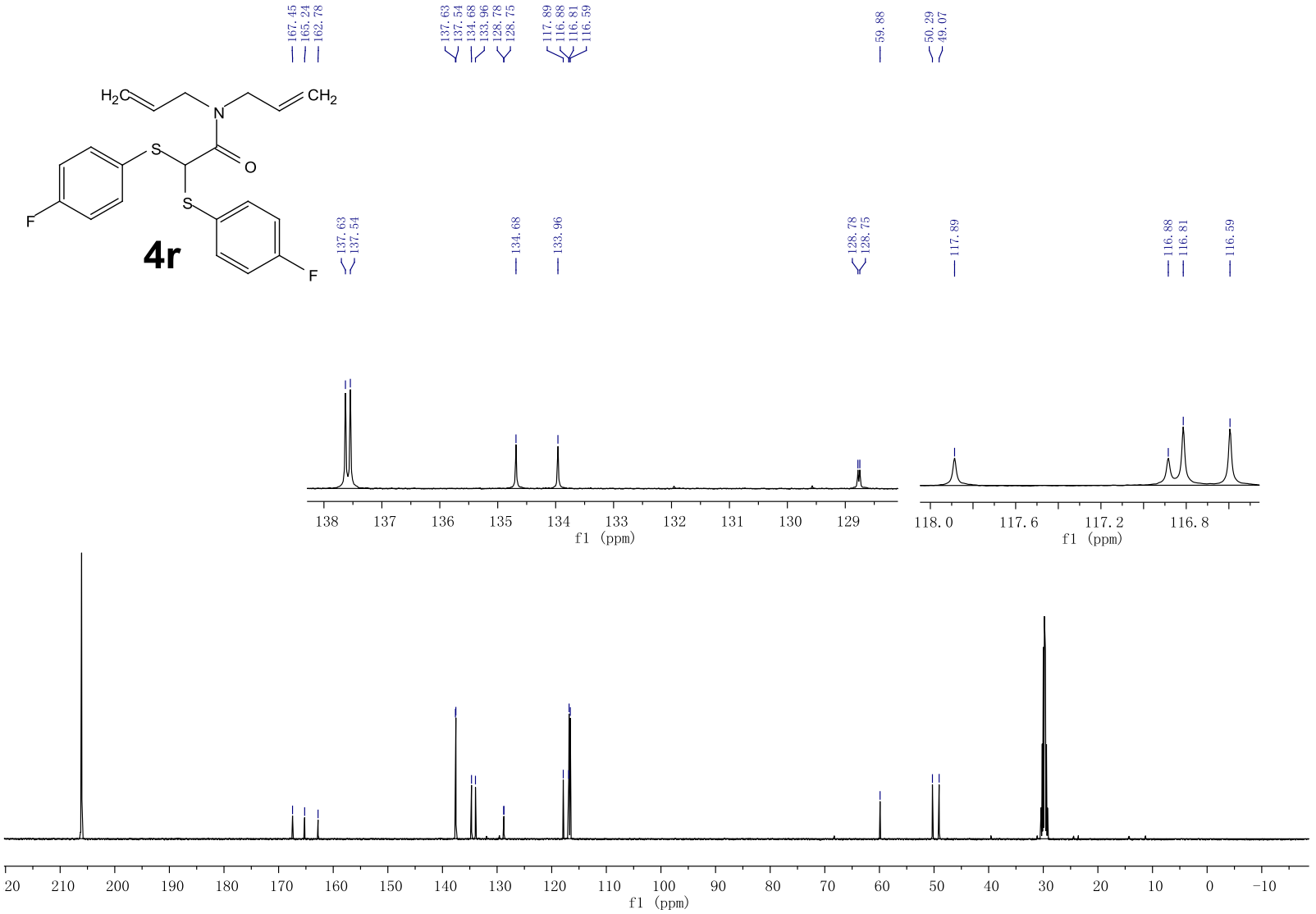

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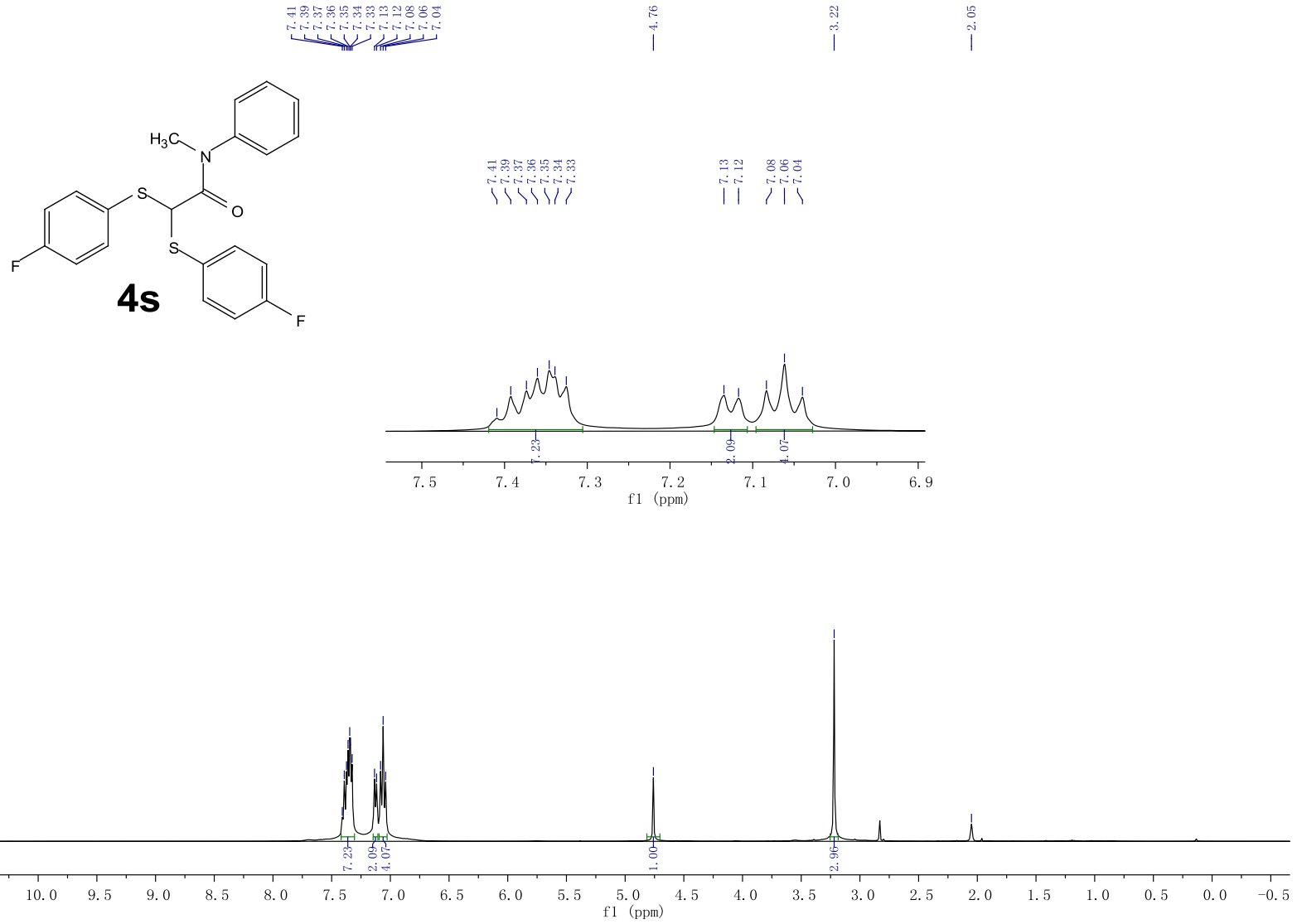

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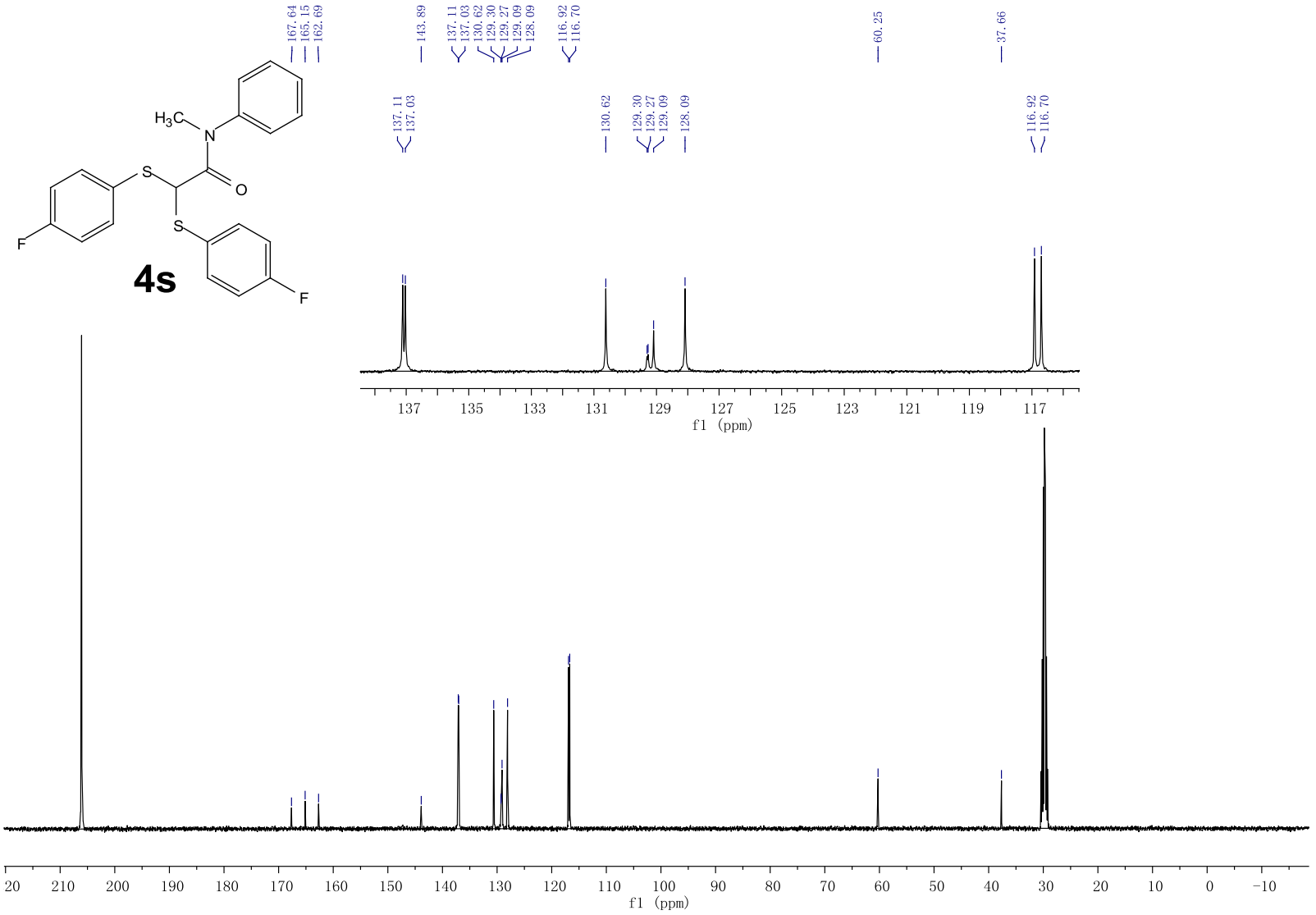

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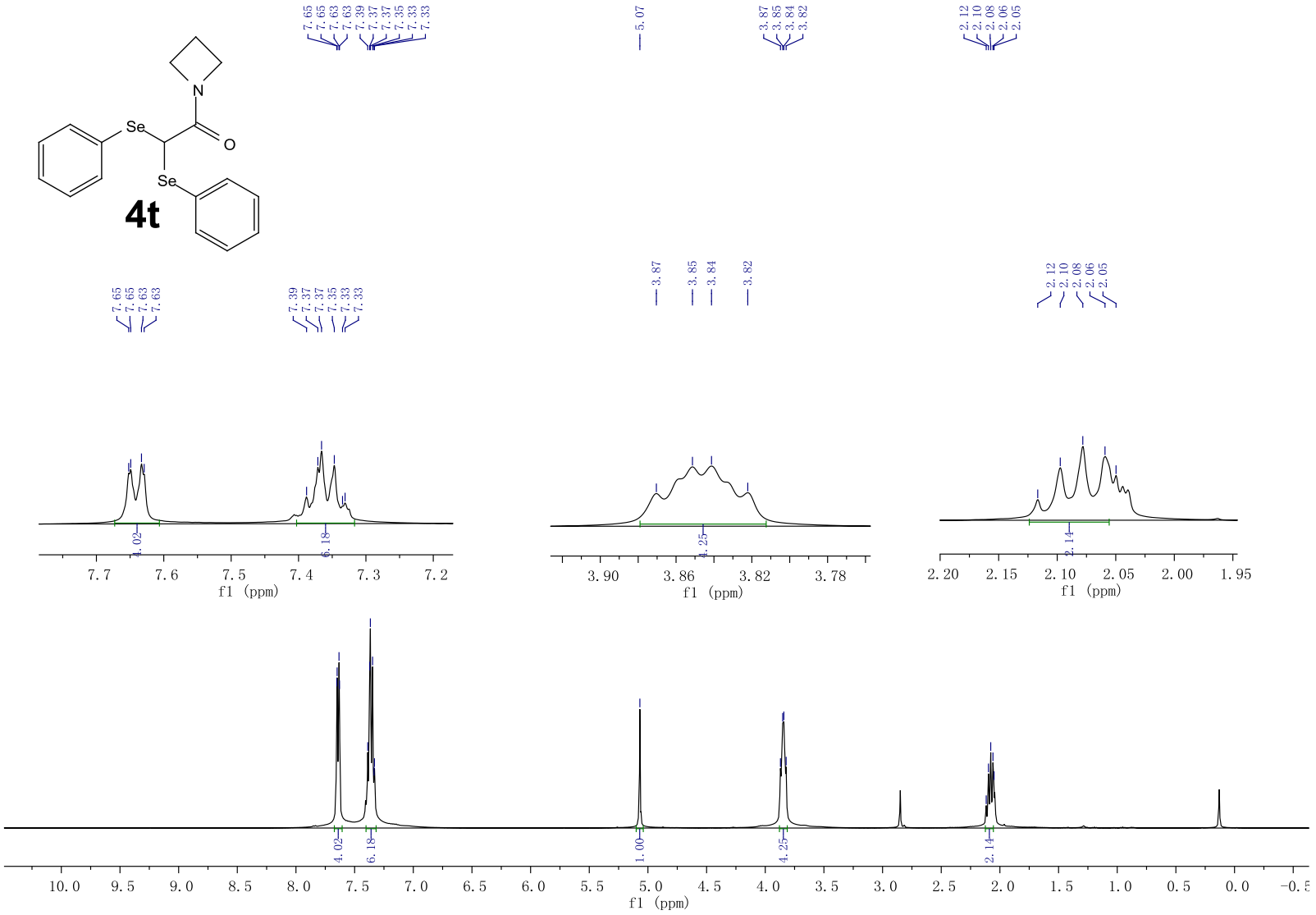

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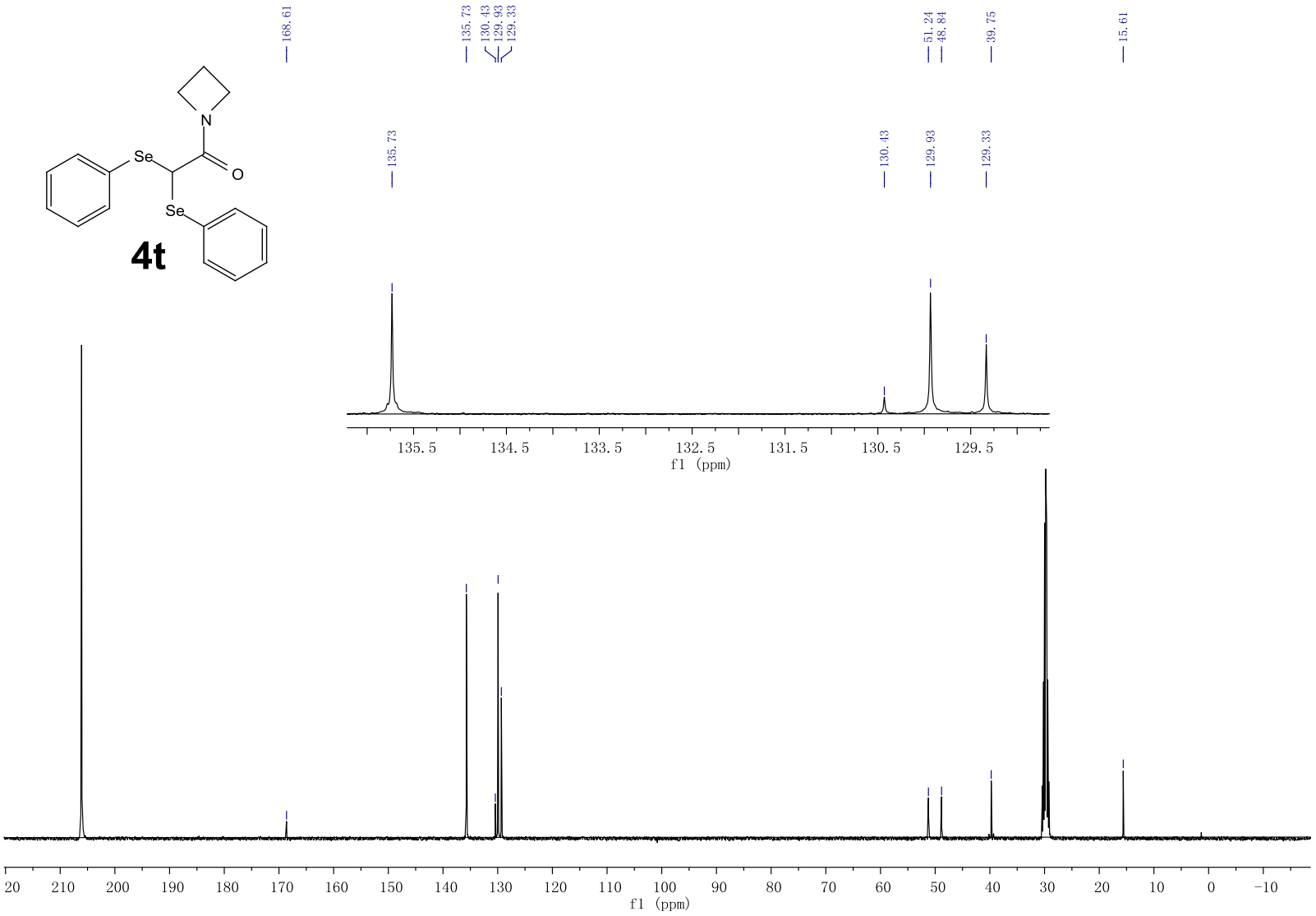

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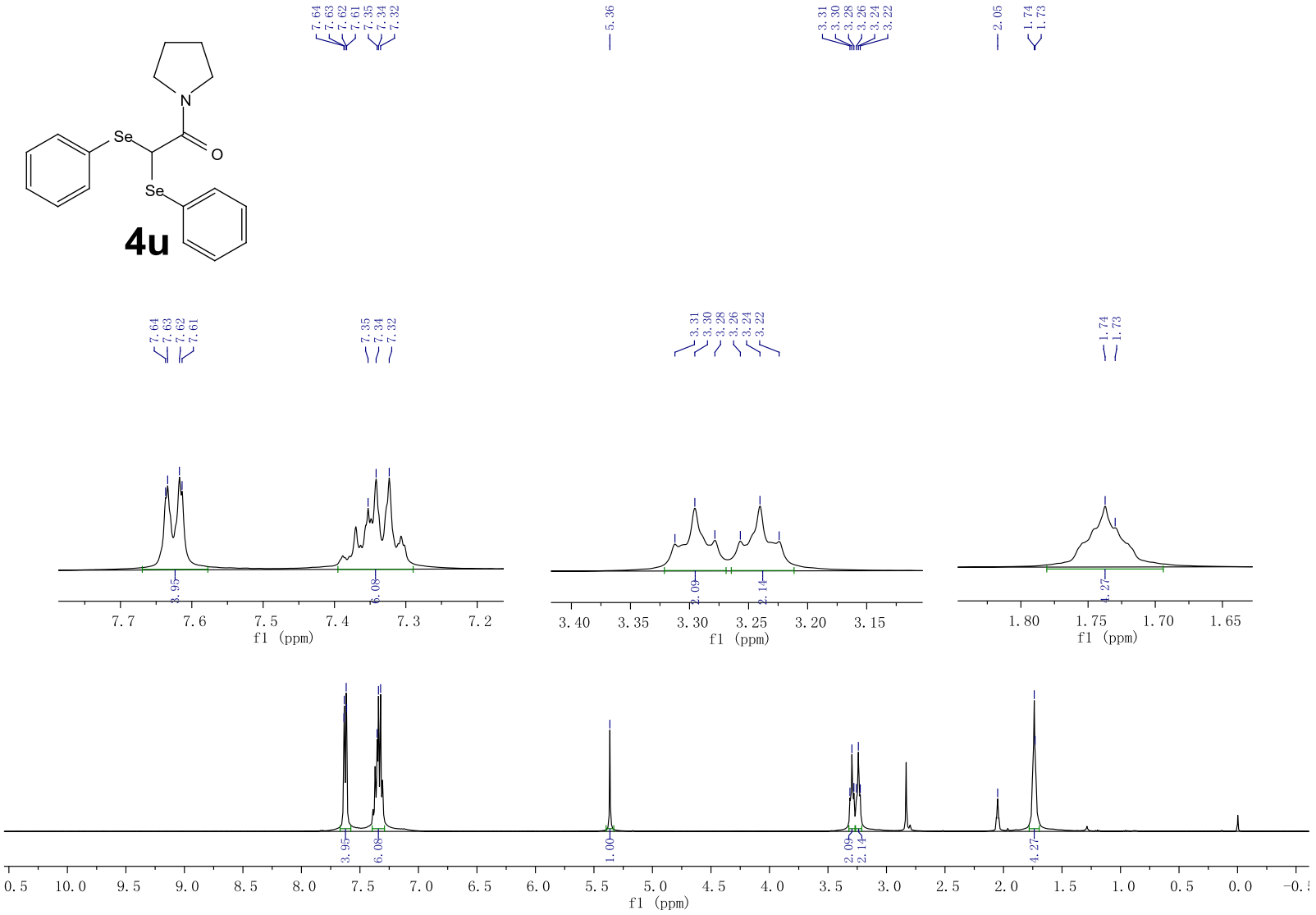

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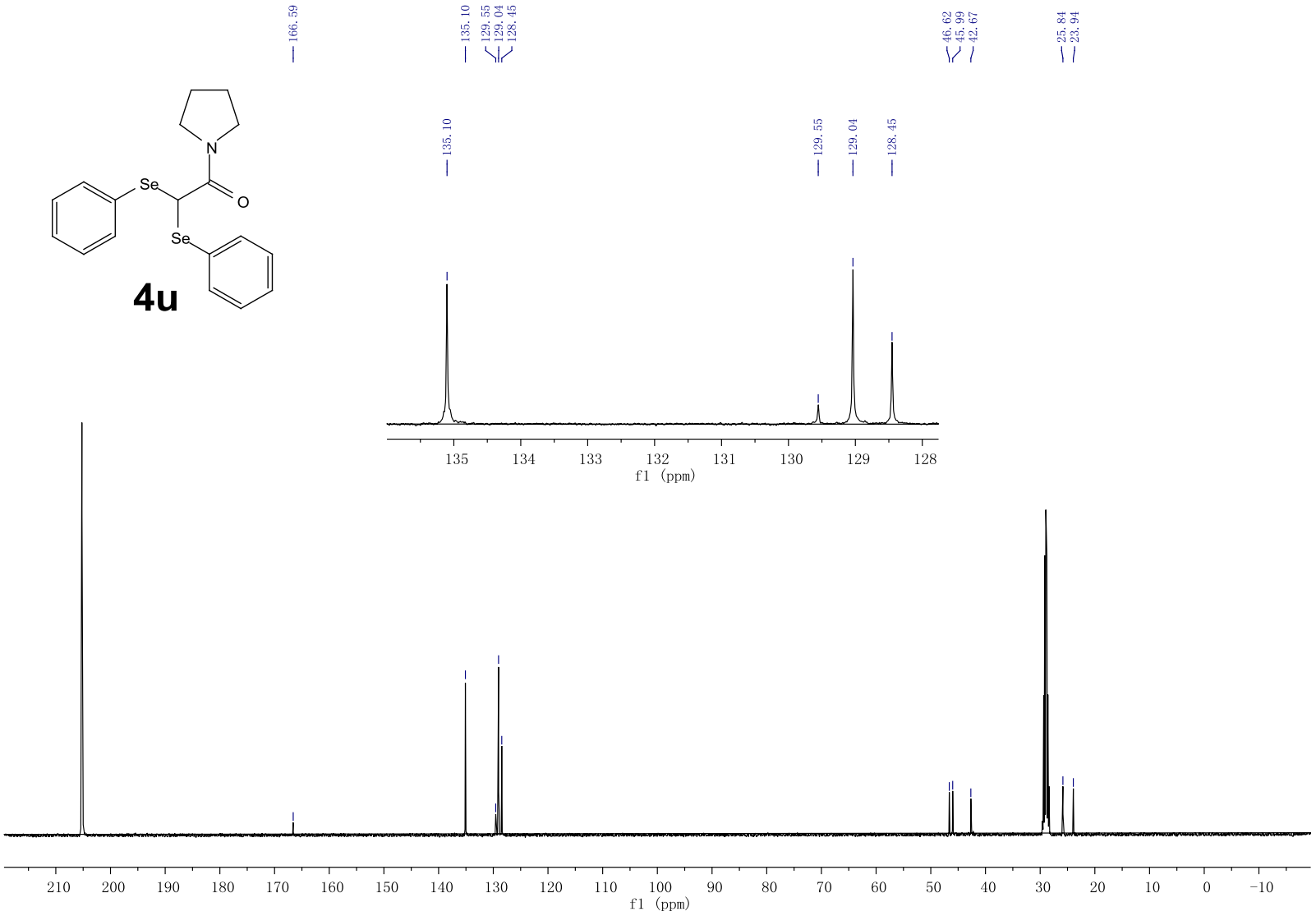

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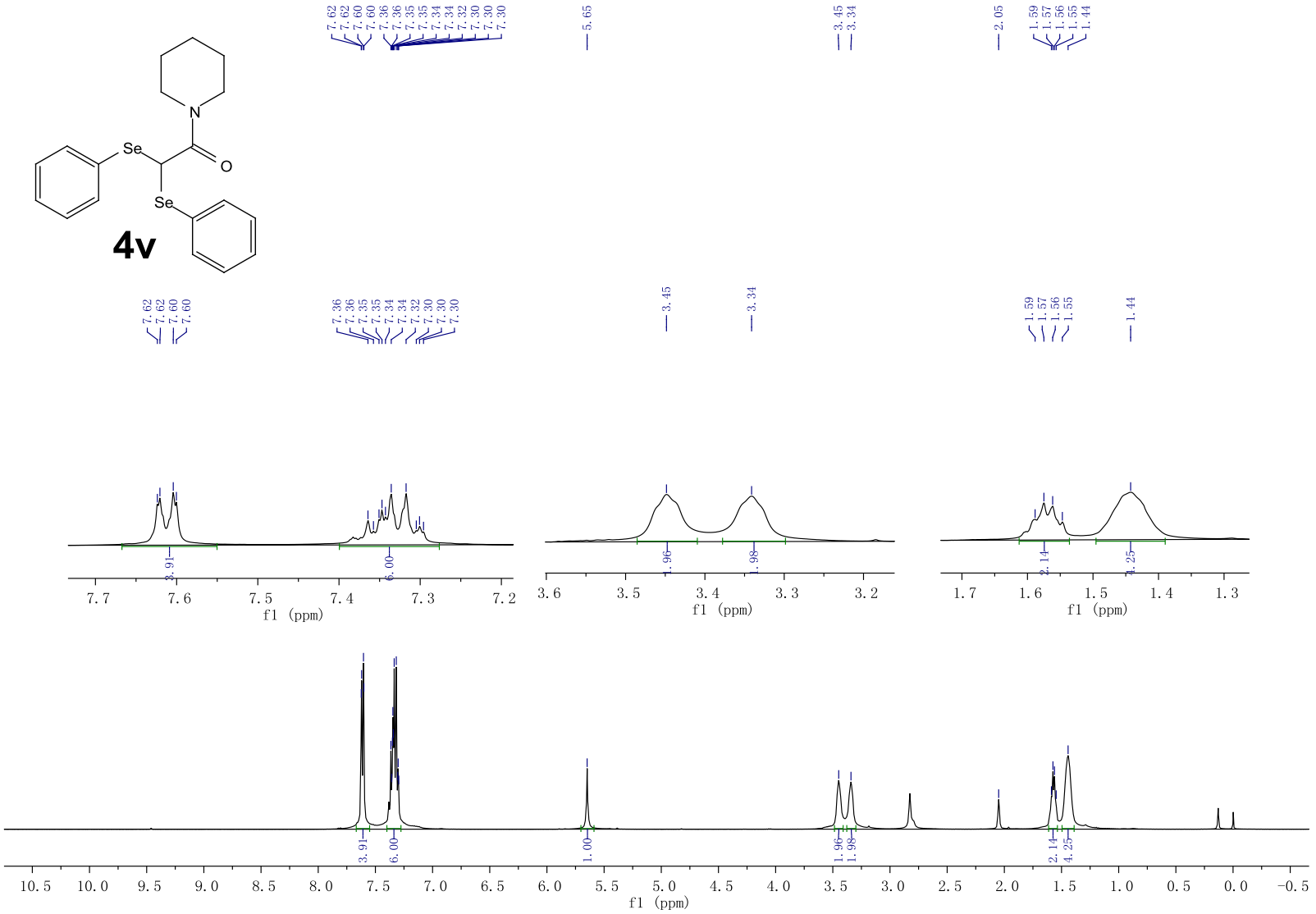

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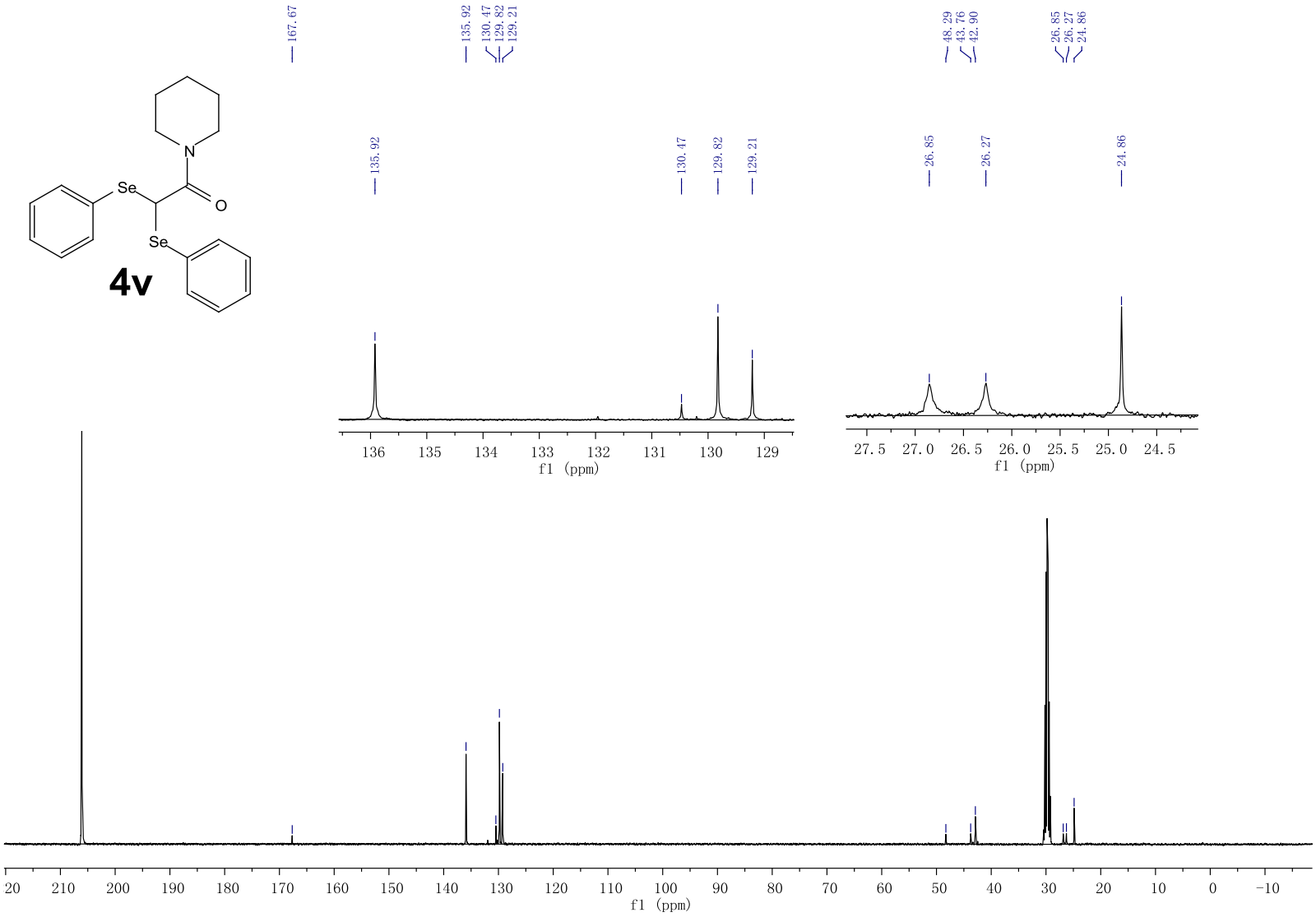

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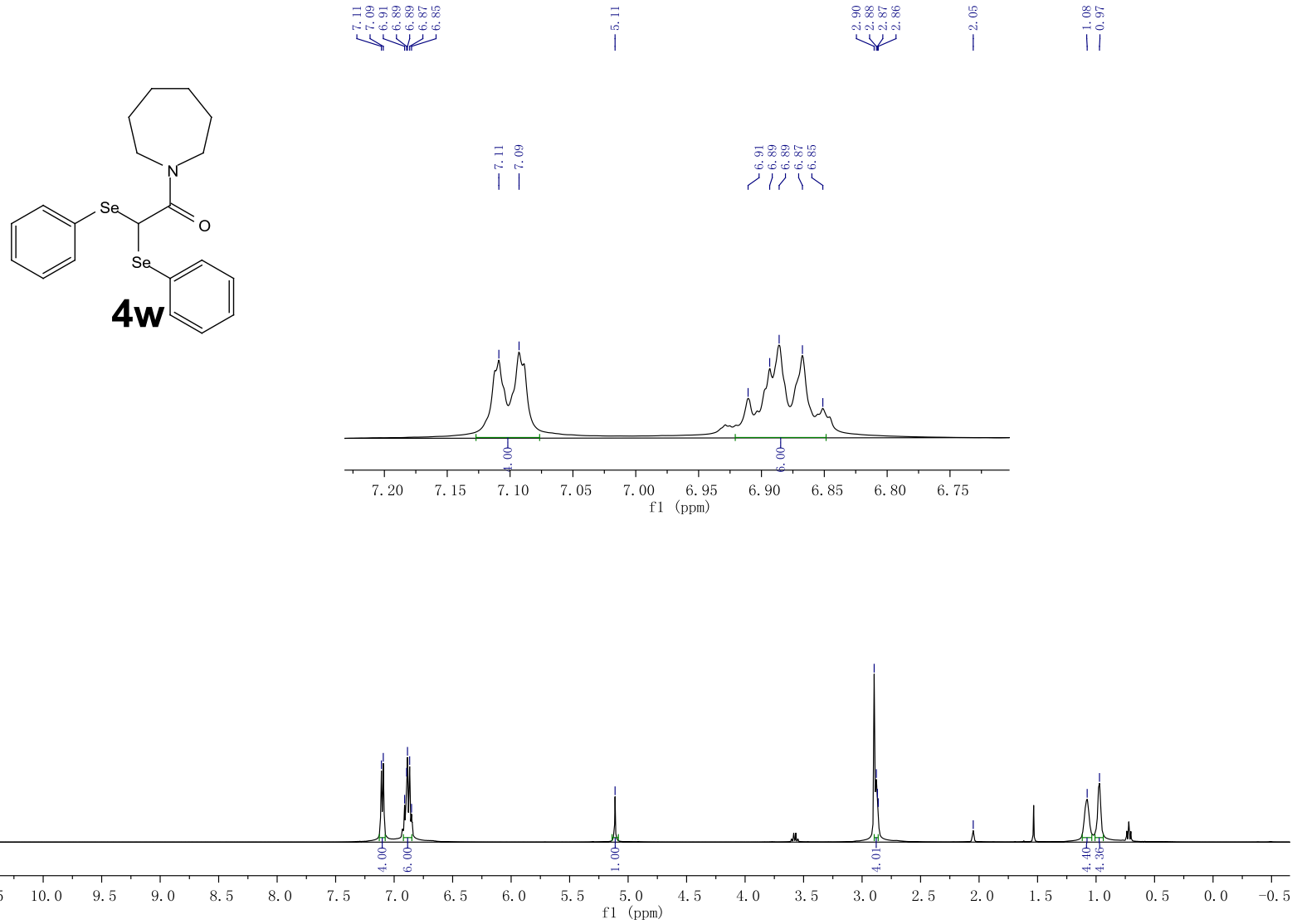

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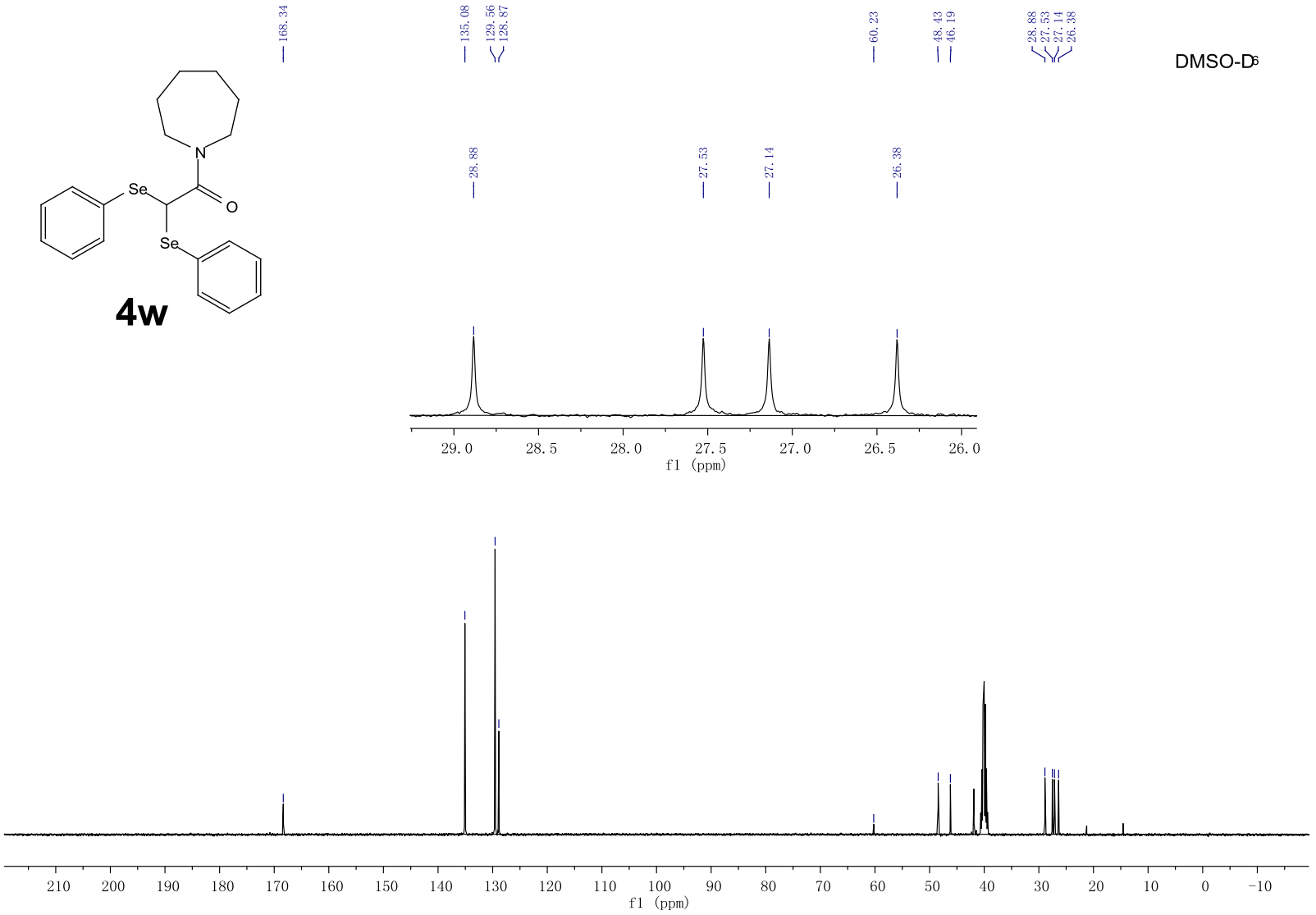

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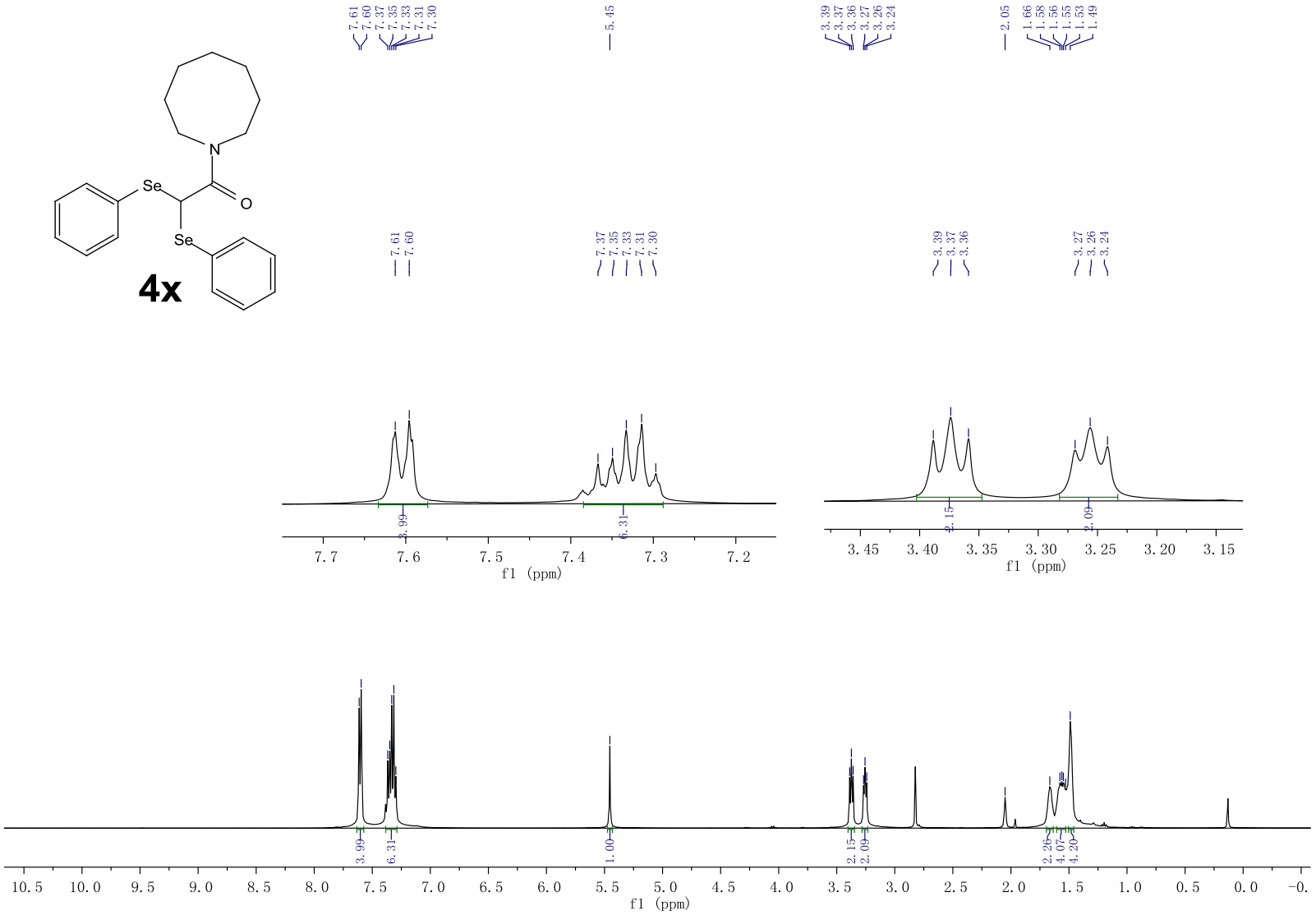

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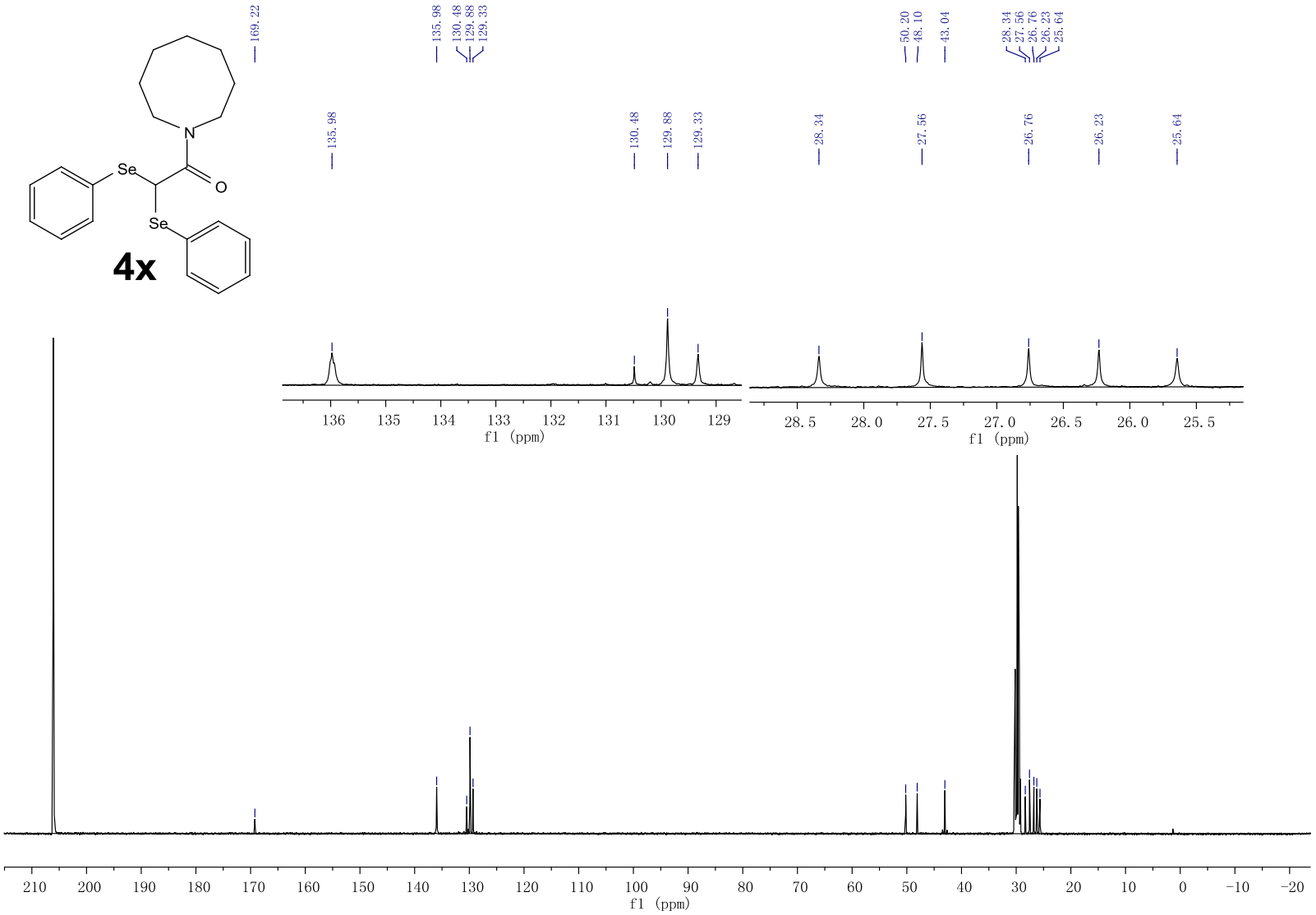

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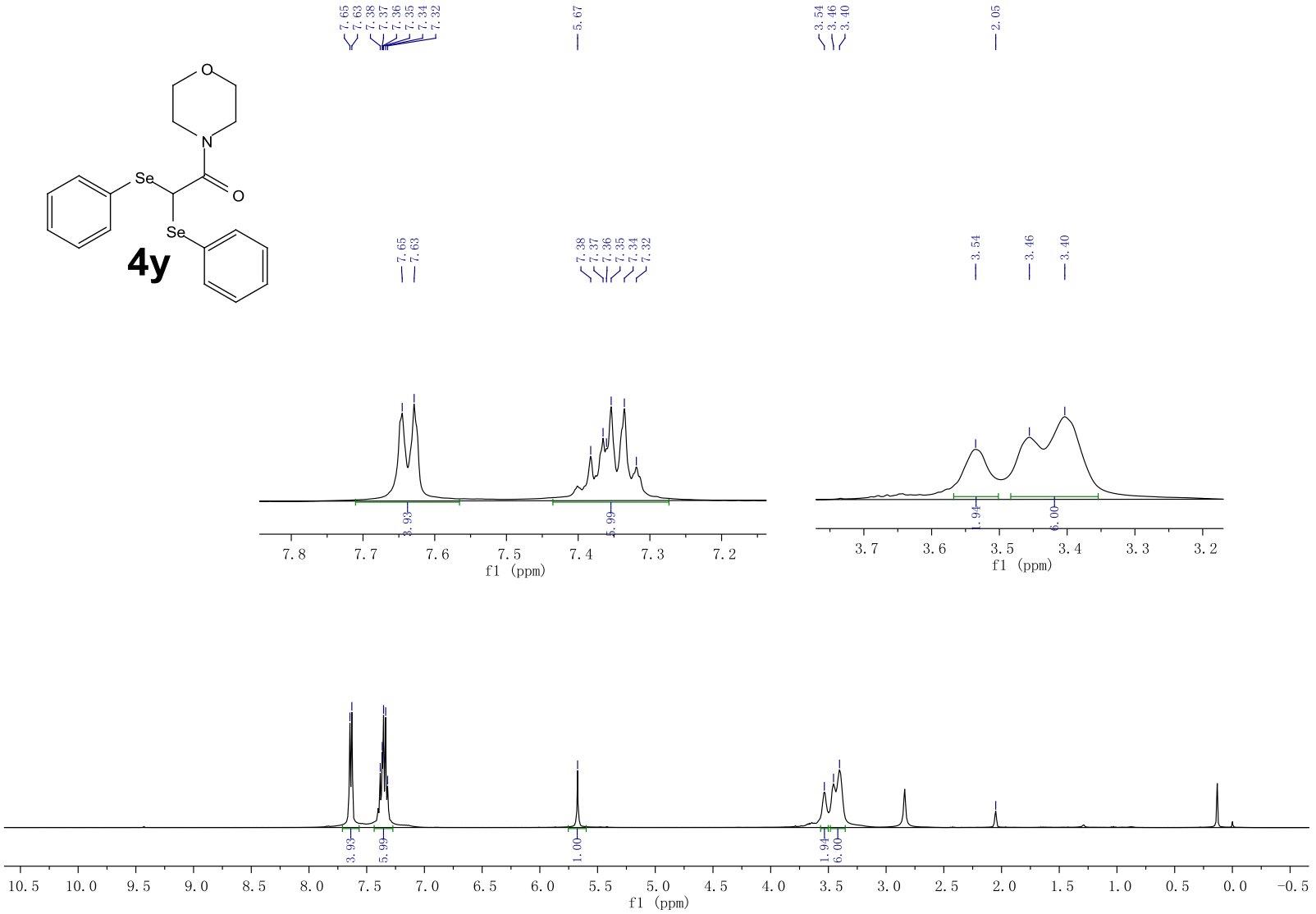

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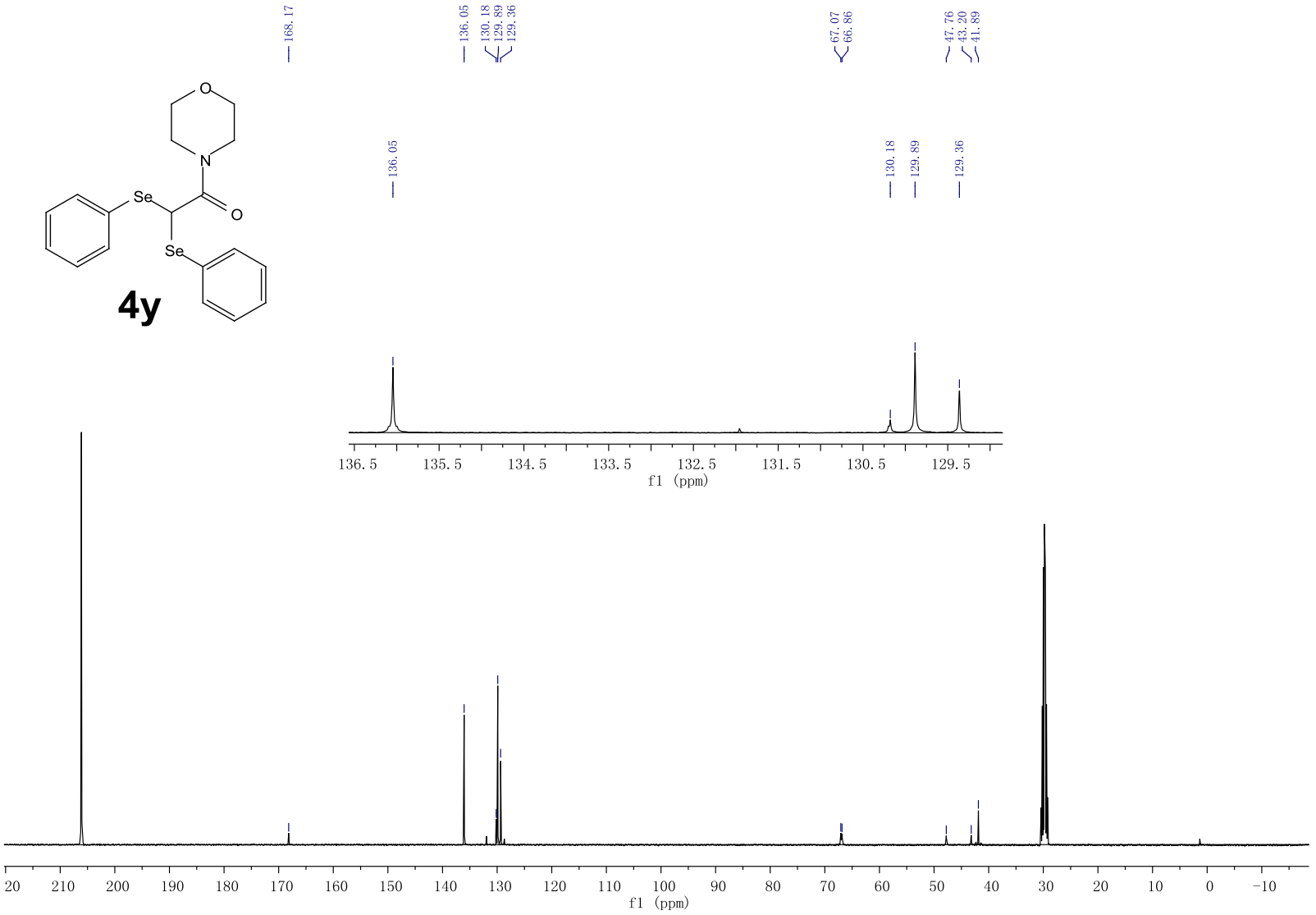

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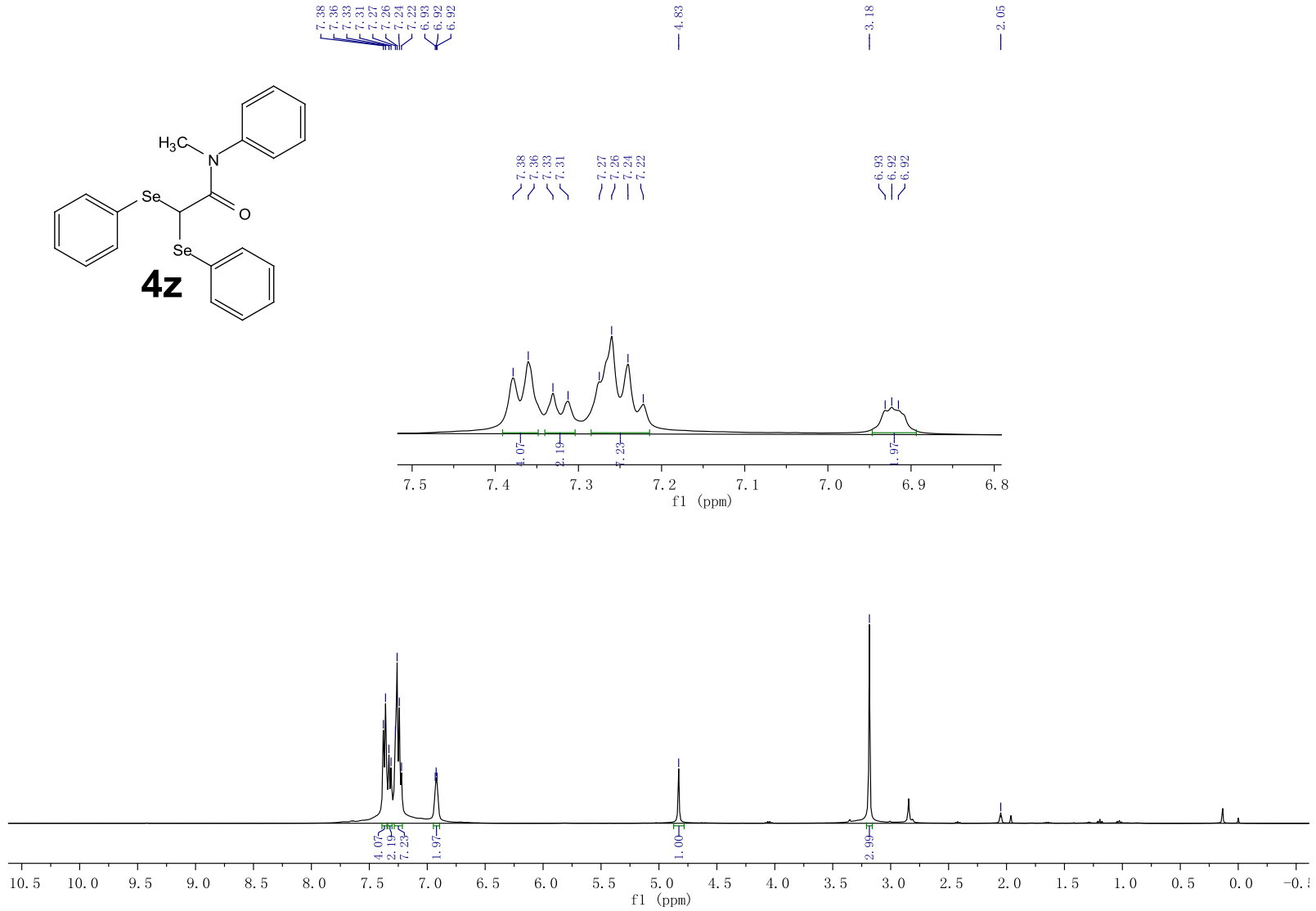

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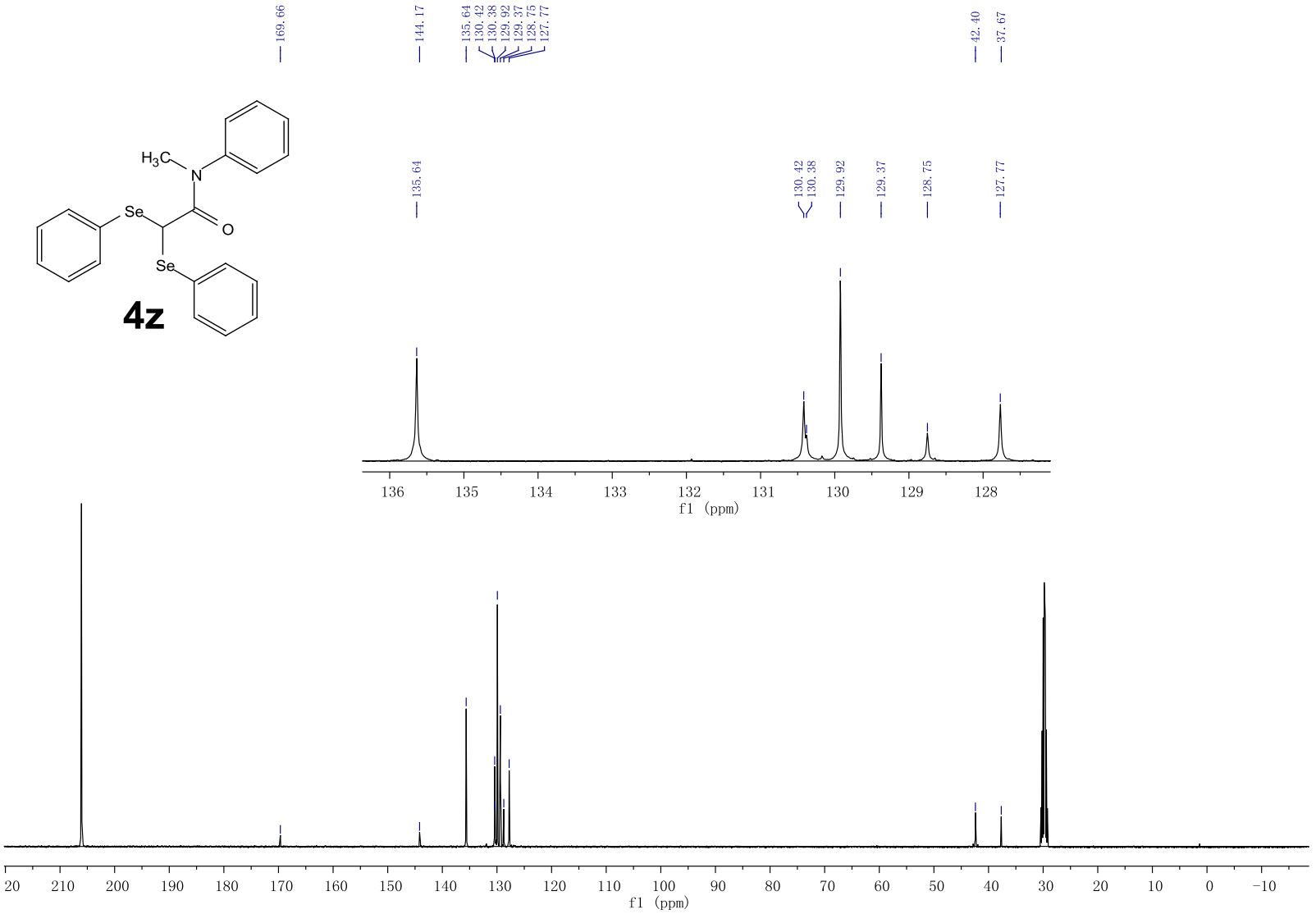

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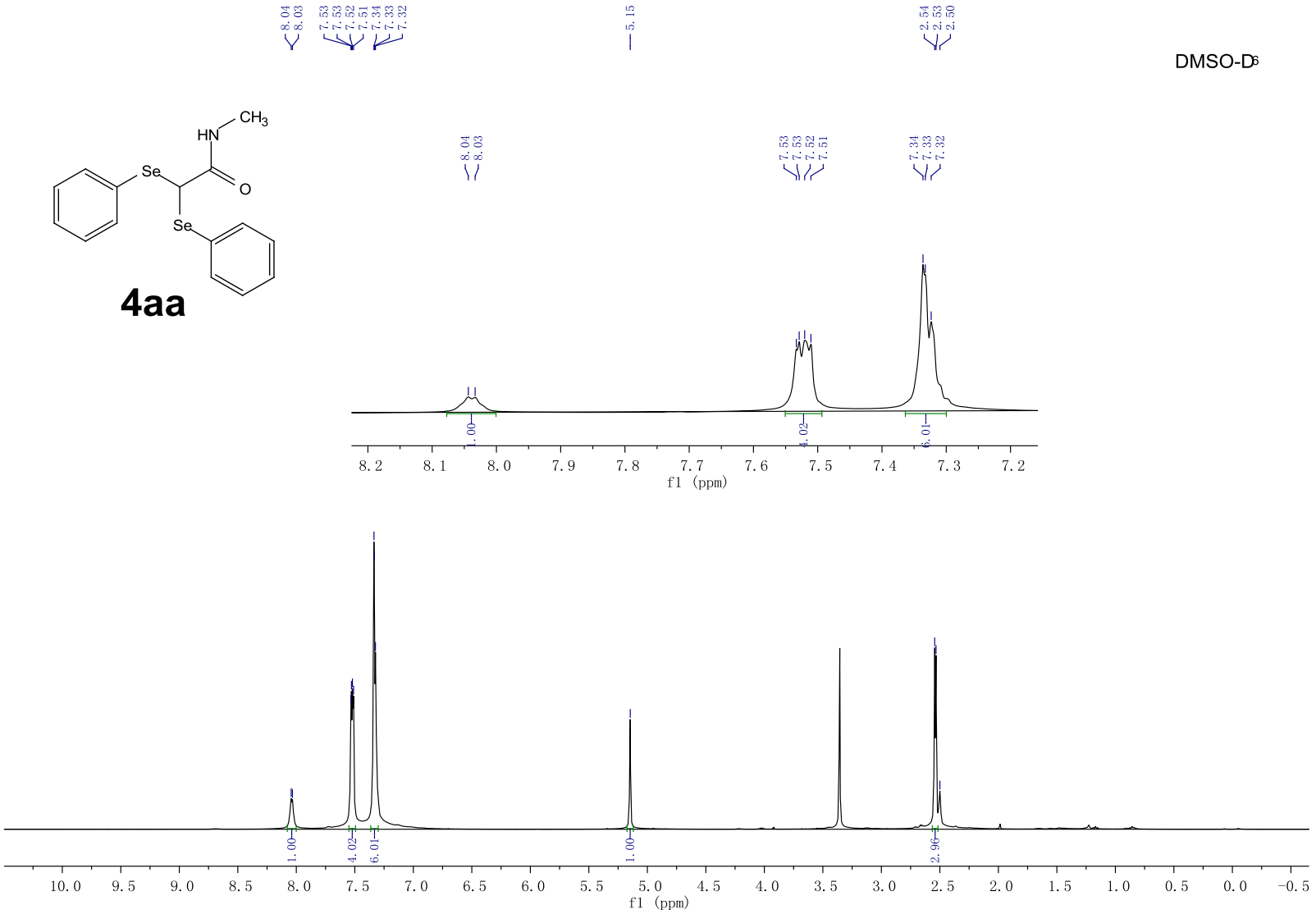

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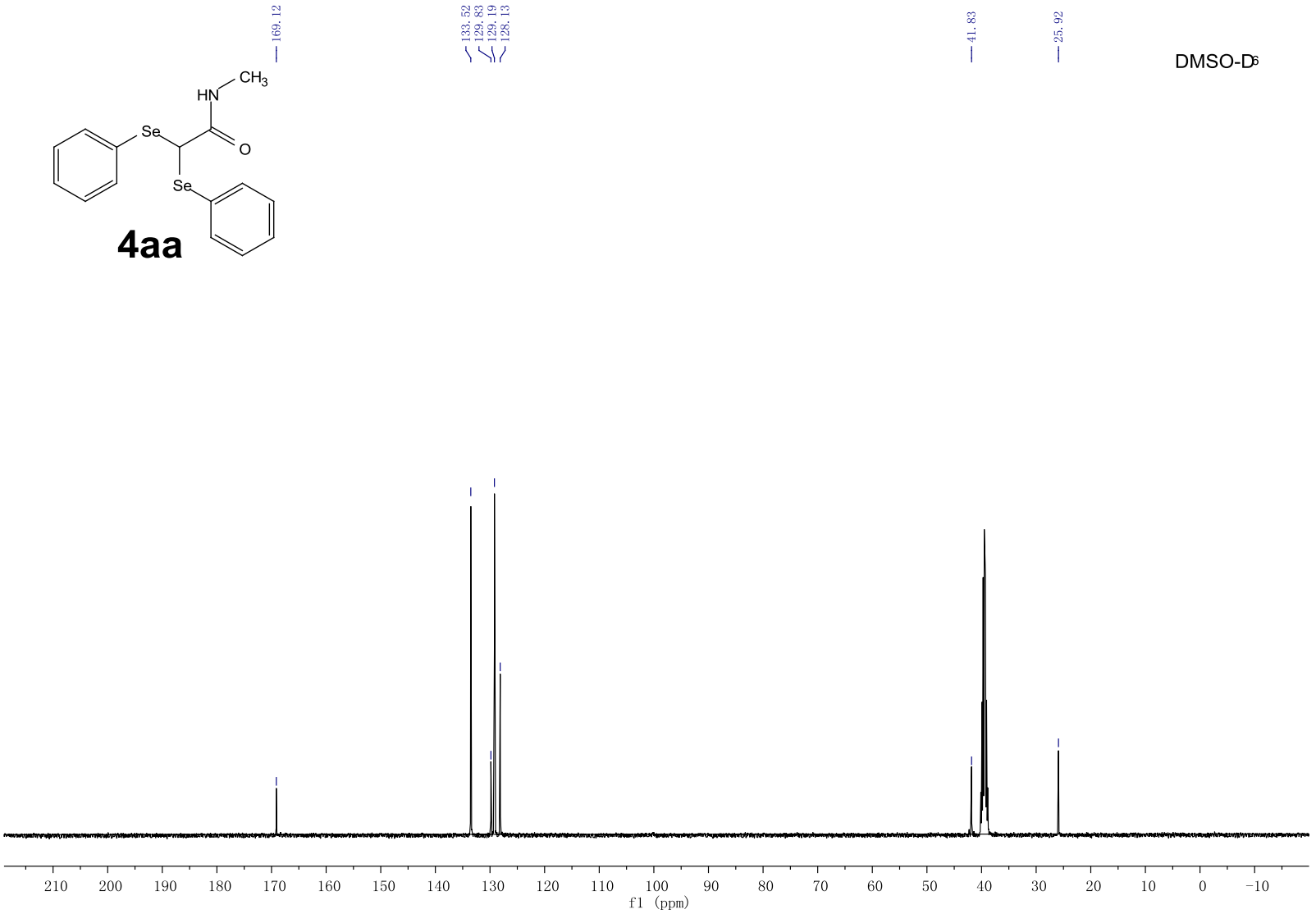
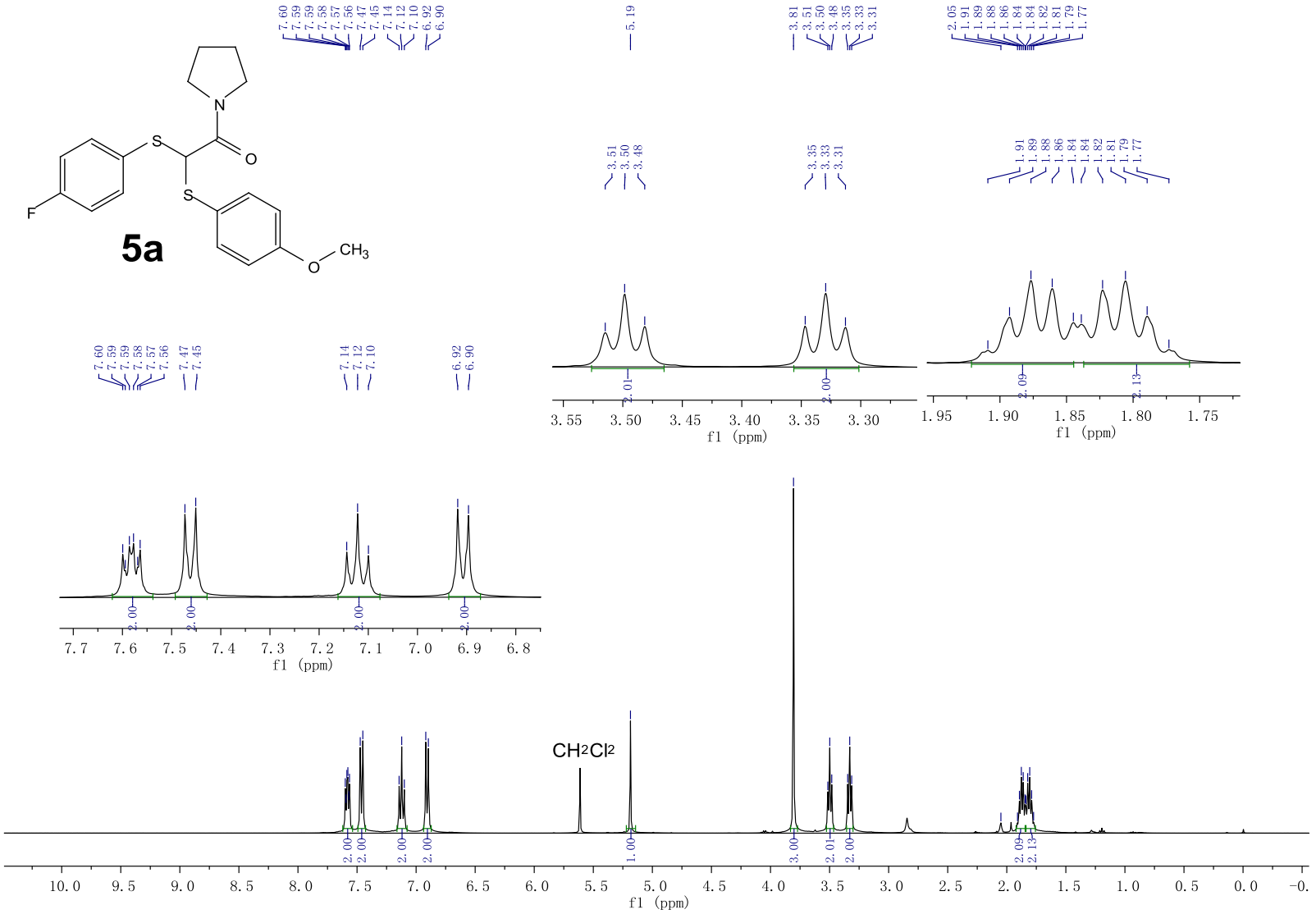
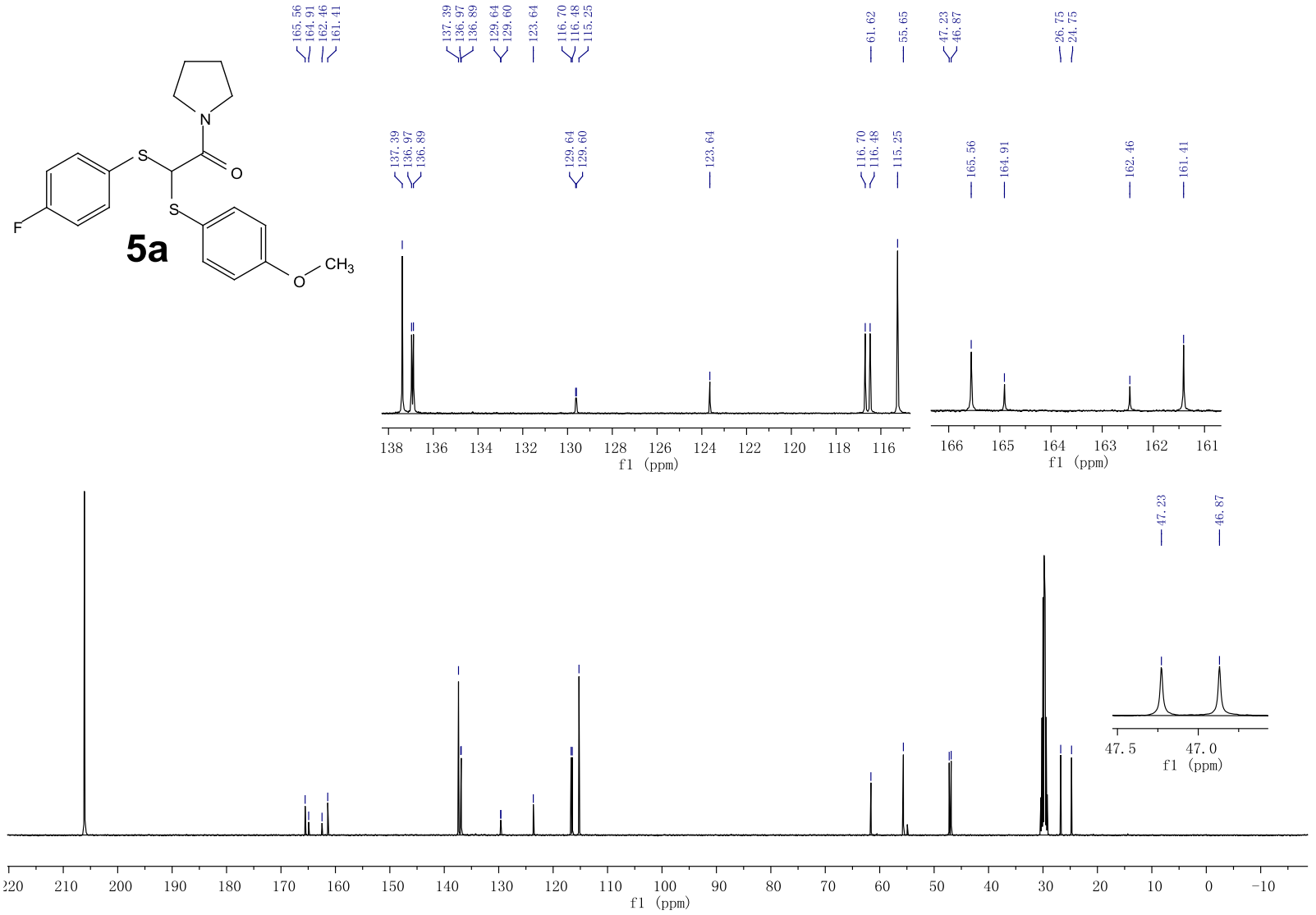


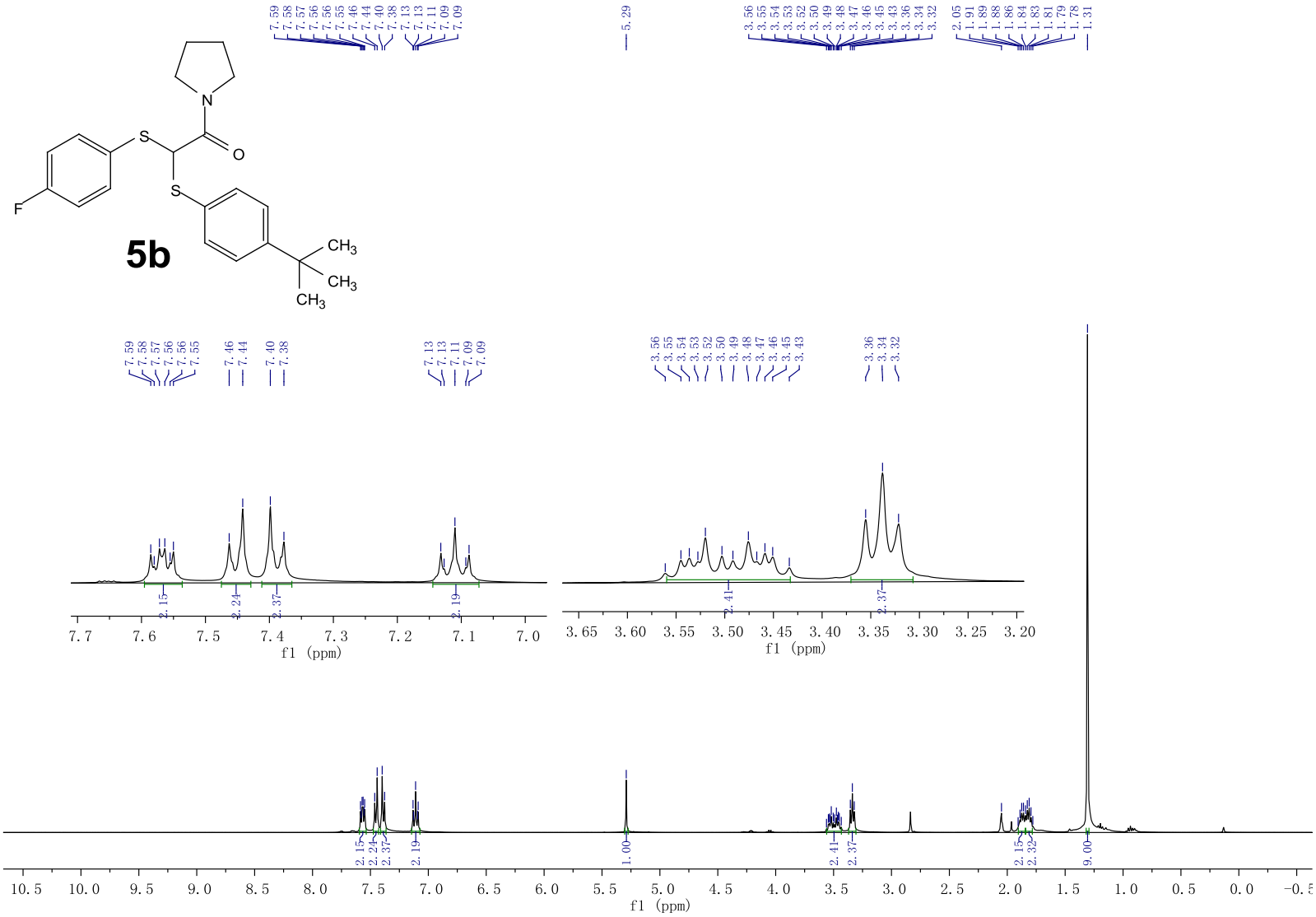
^{13}C NMR (100 MHz, CD_3SOCD_3)



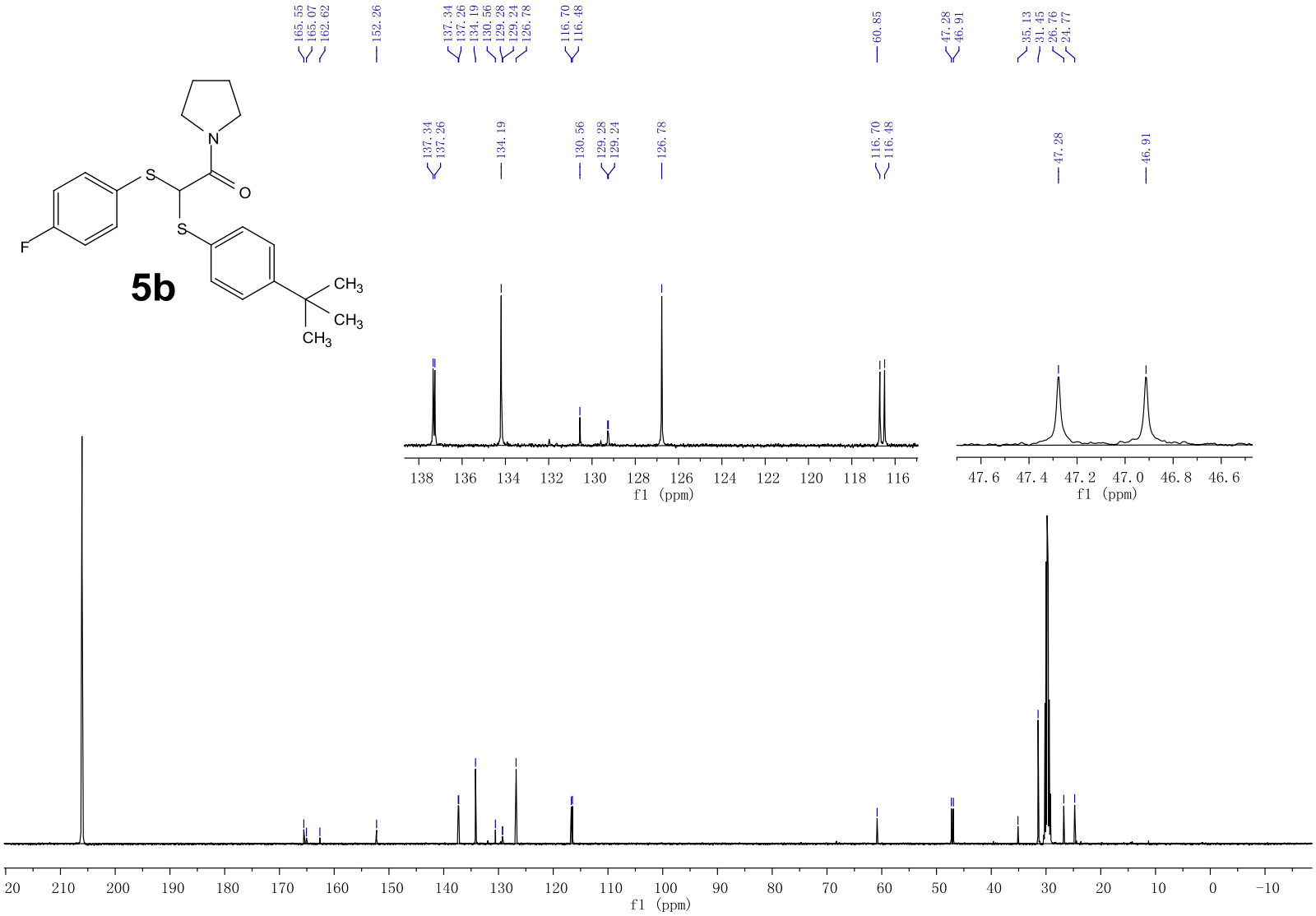
^{1}H NMR (400 MHz, CD_3COCD_3)



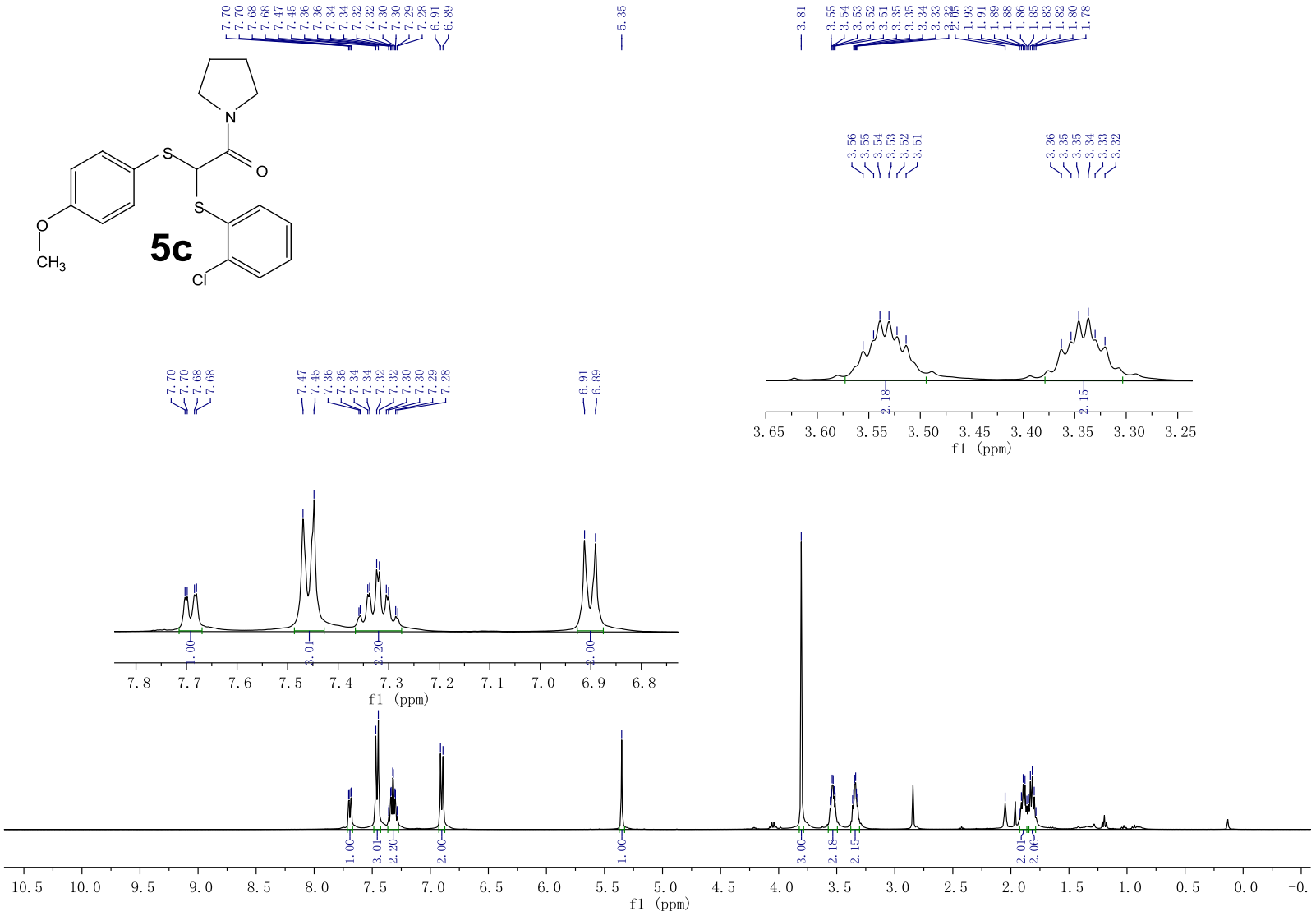
^{13}C NMR (100 MHz, CD_3COCD_3)



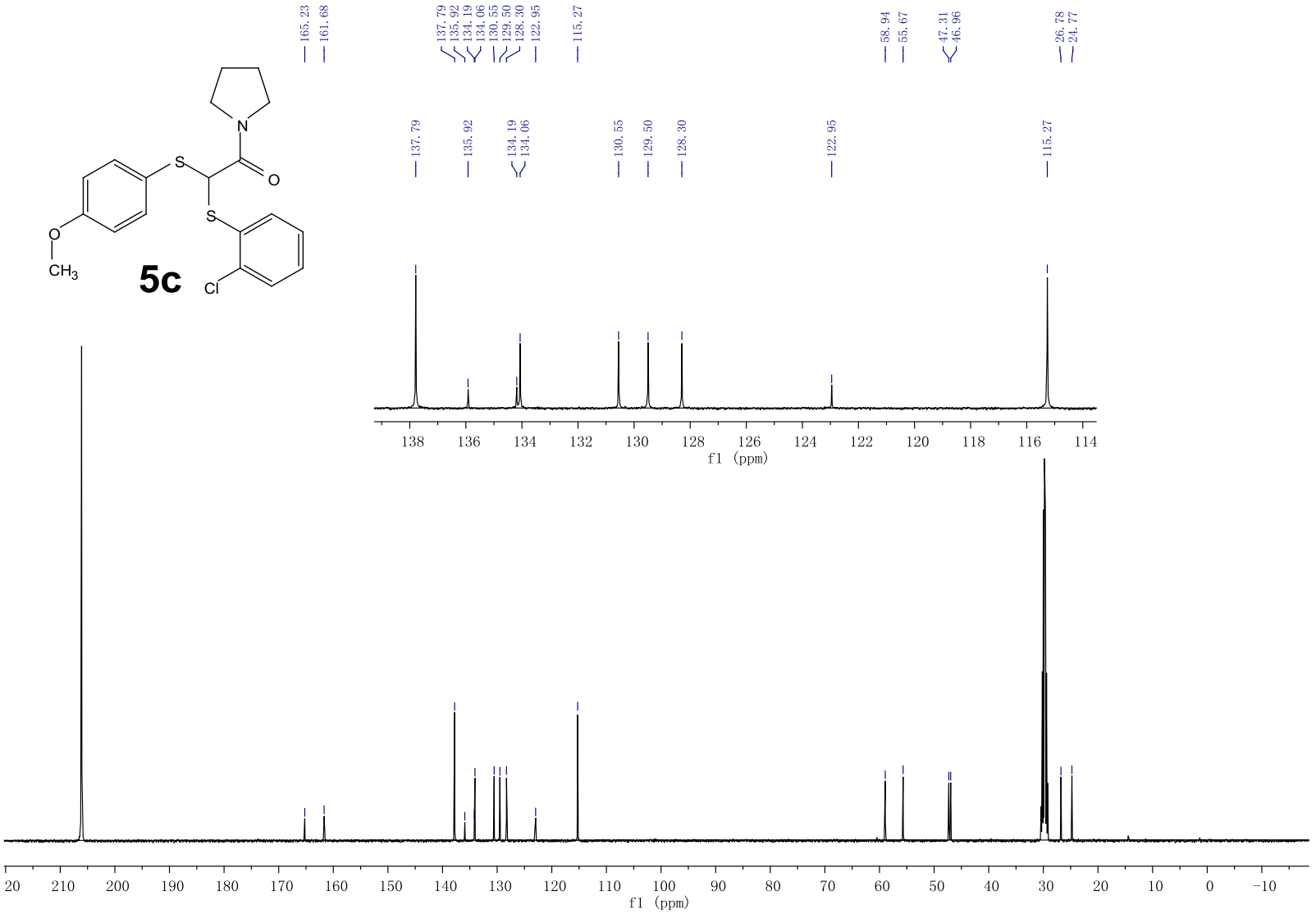
^{1}H NMR (400 MHz, CD_3COCD_3)



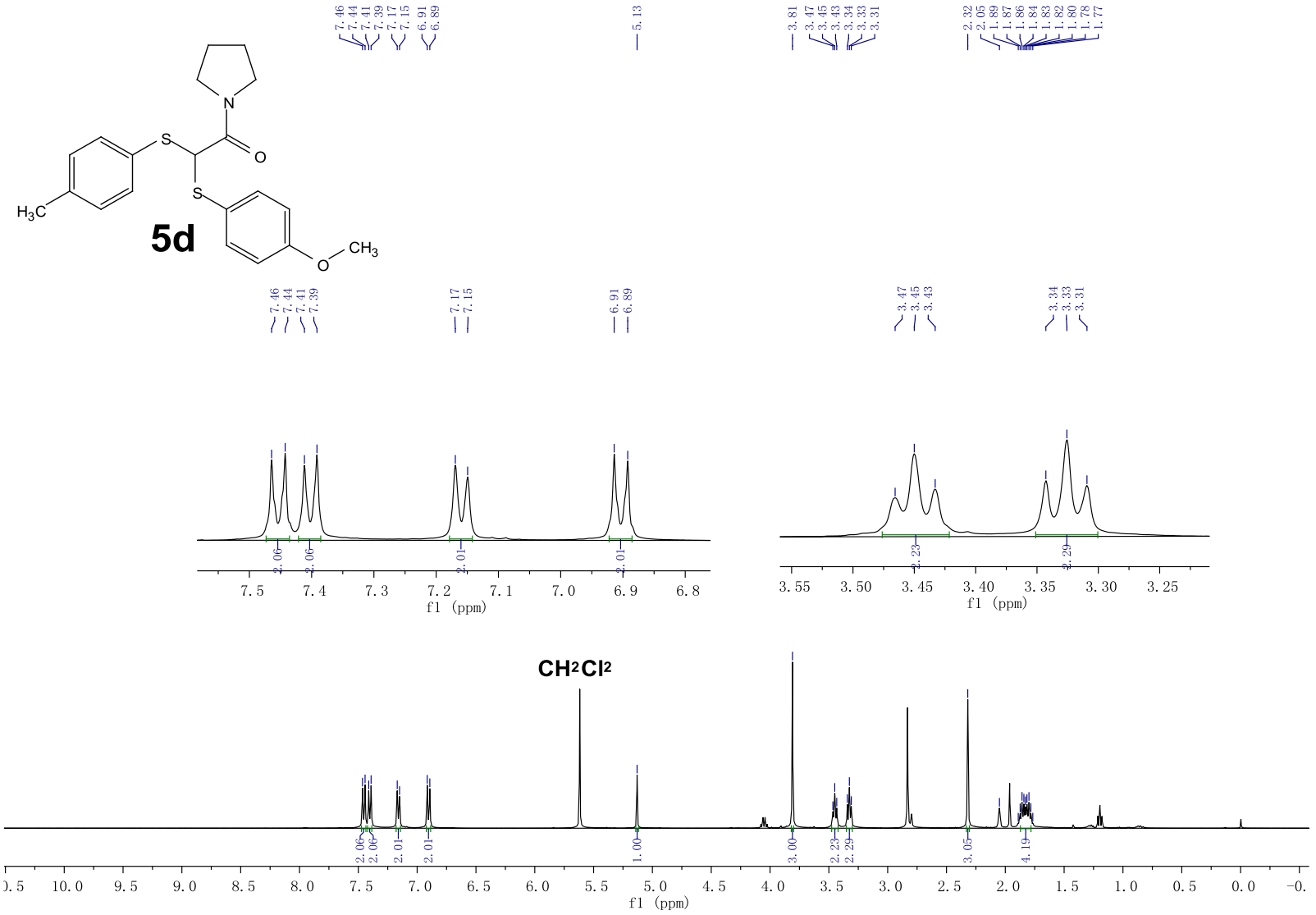
^{13}C NMR (100 MHz, CD_3COCD_3)



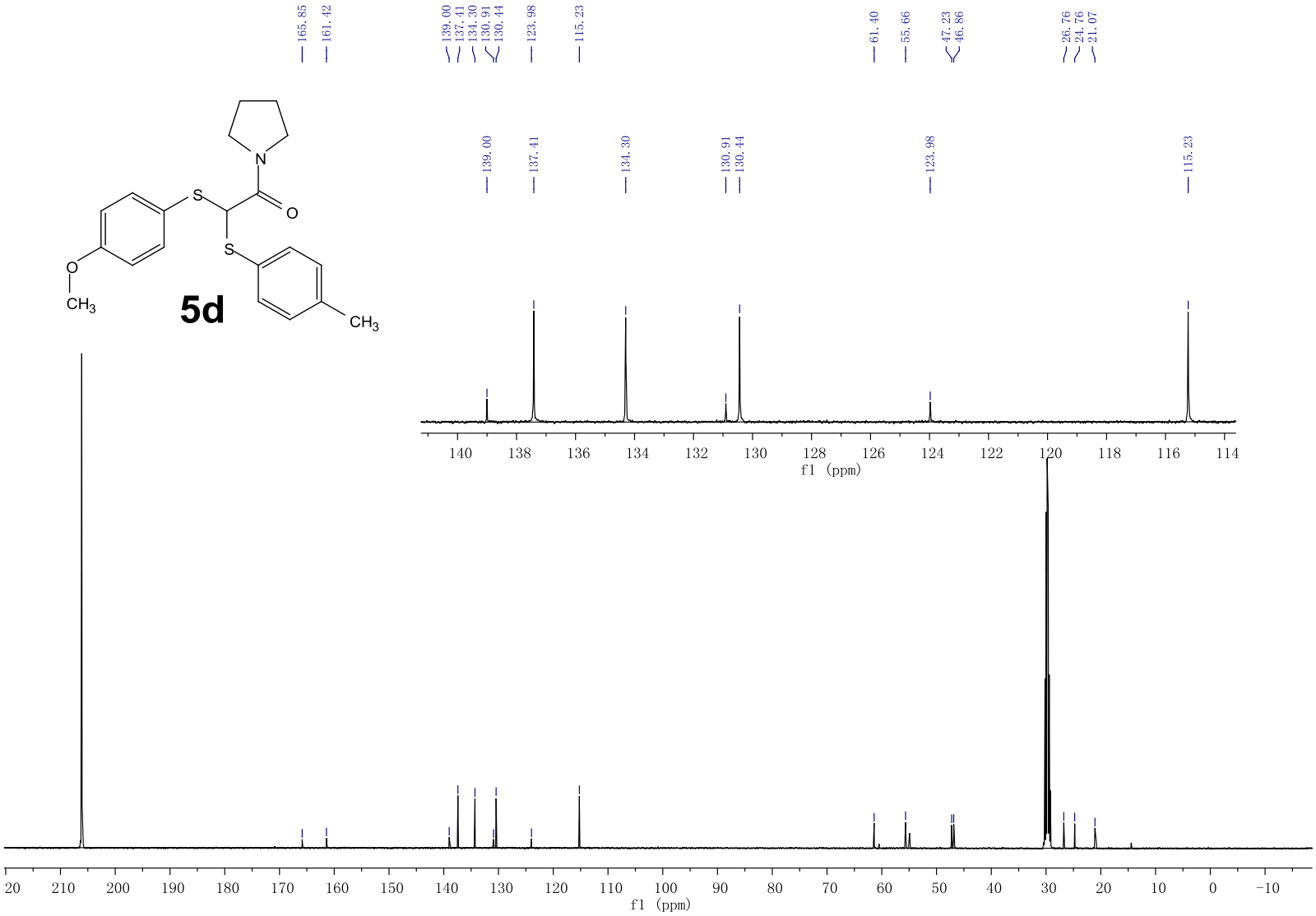
^{1}H NMR (400 MHz, CD_3COCD_3)



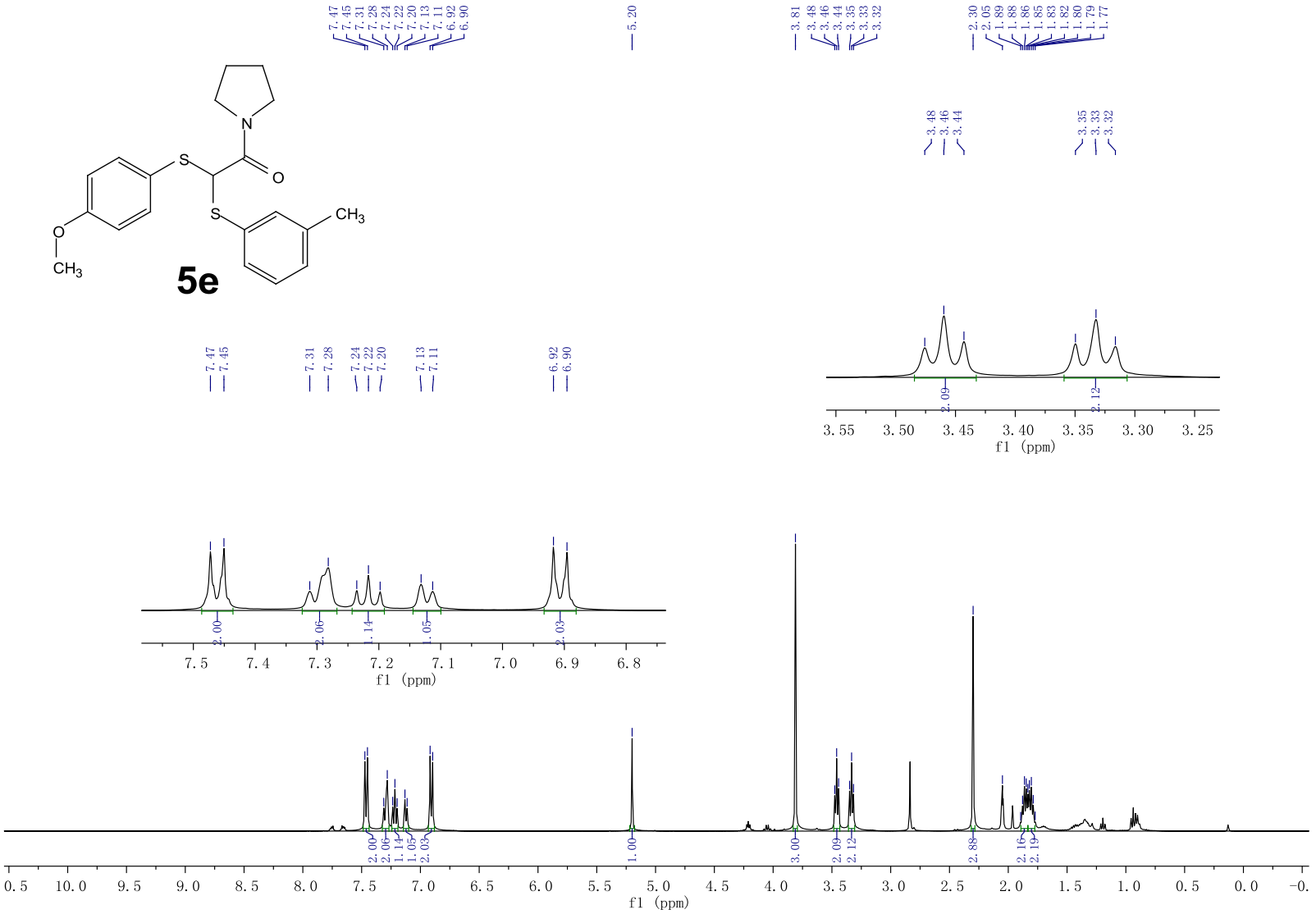
^{13}C NMR (100 MHz, CD_3COCD_3)



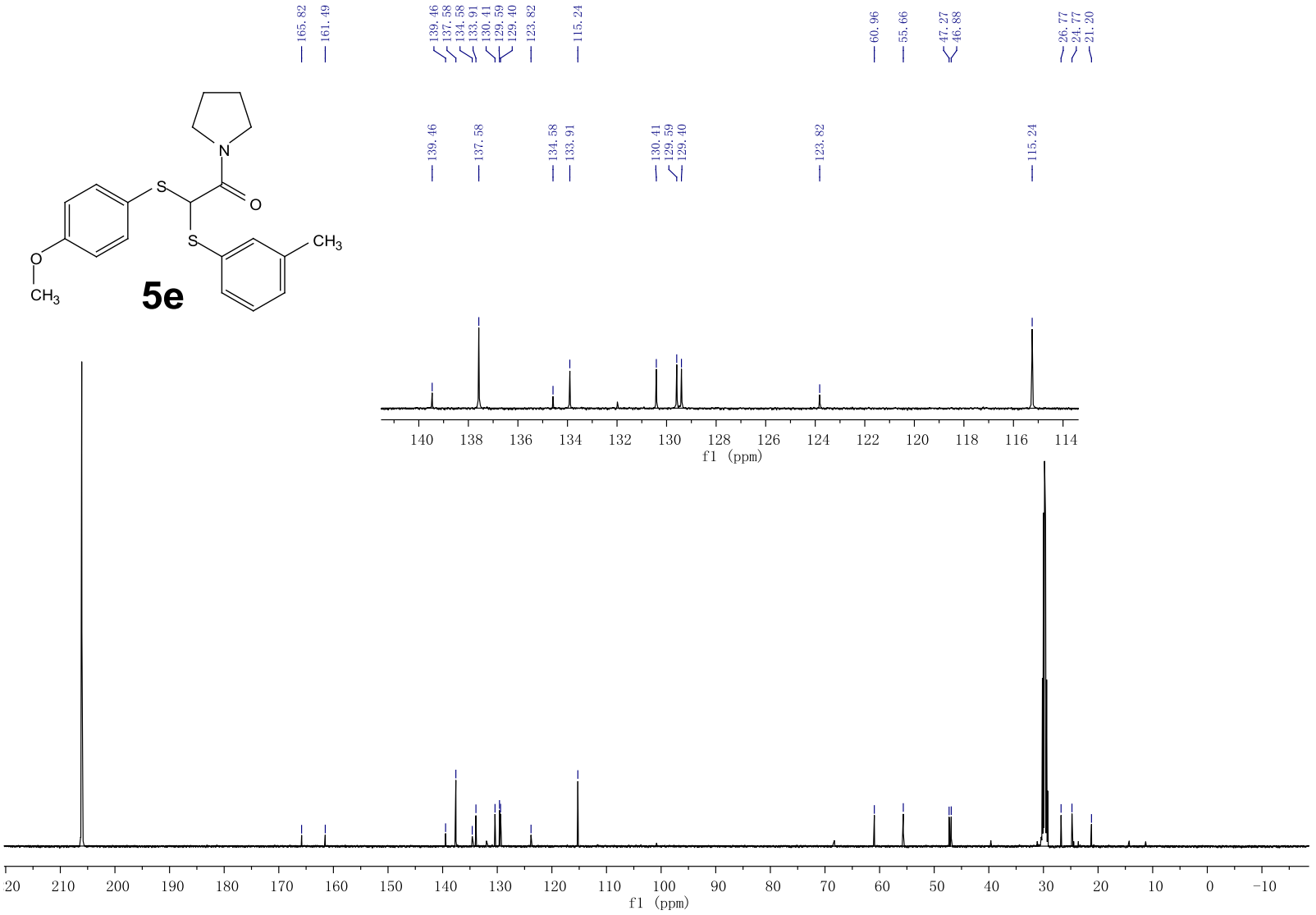
^{1}H NMR (400 MHz, CD_3COCD_3)



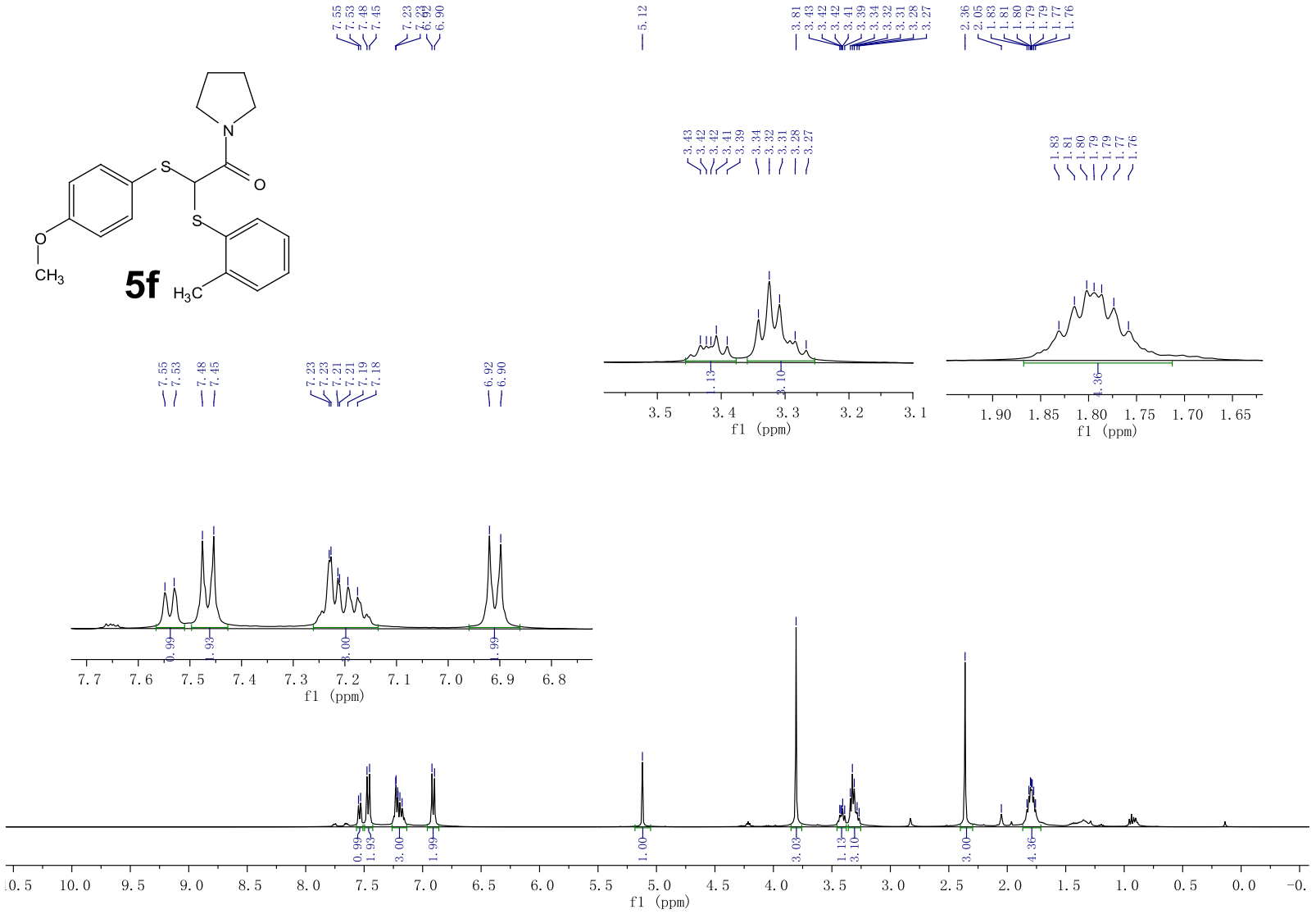
^{13}C NMR (100 MHz, CD_3COCD_3)



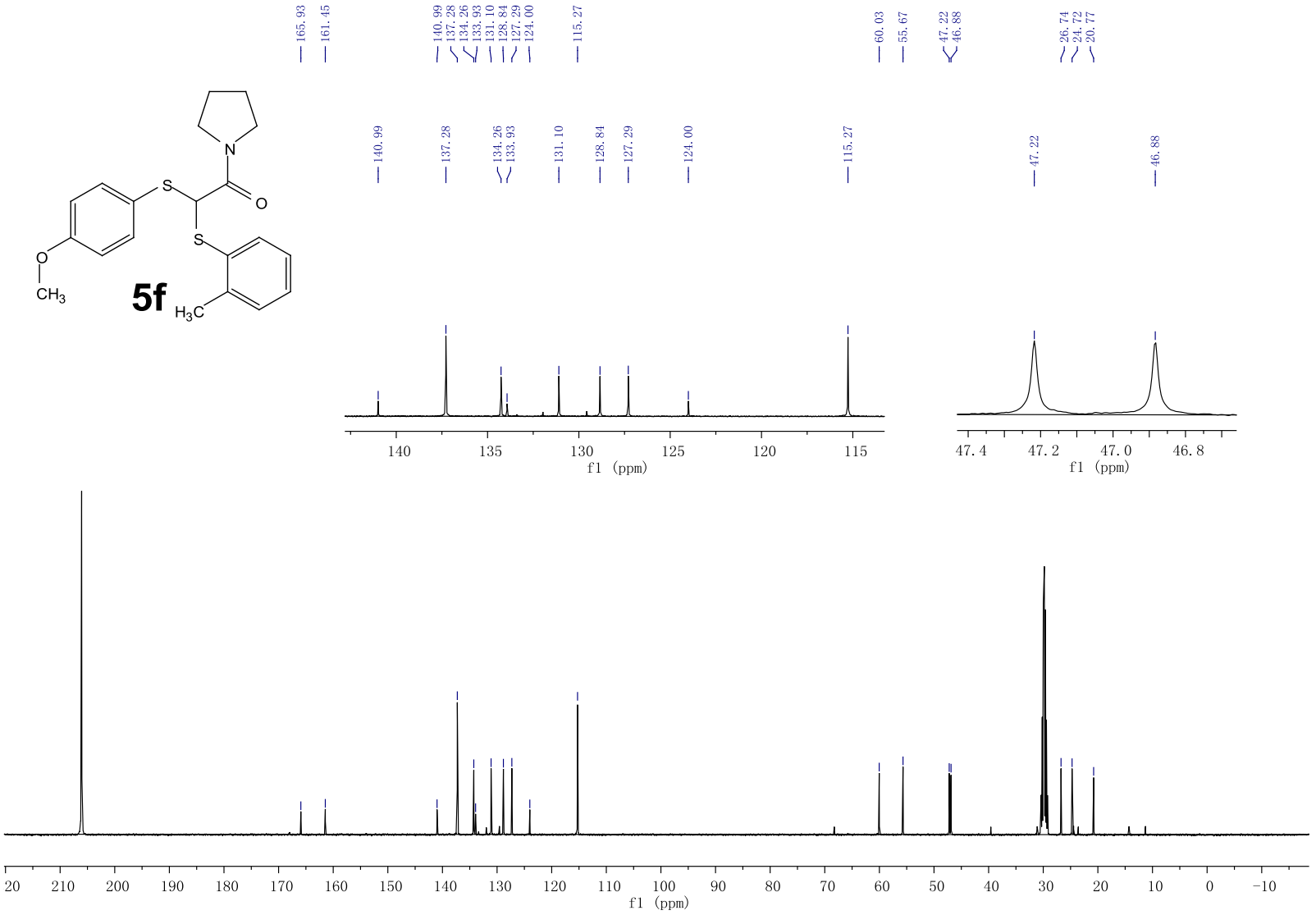
^{1}H NMR (400 MHz, CD_3COCD_3)



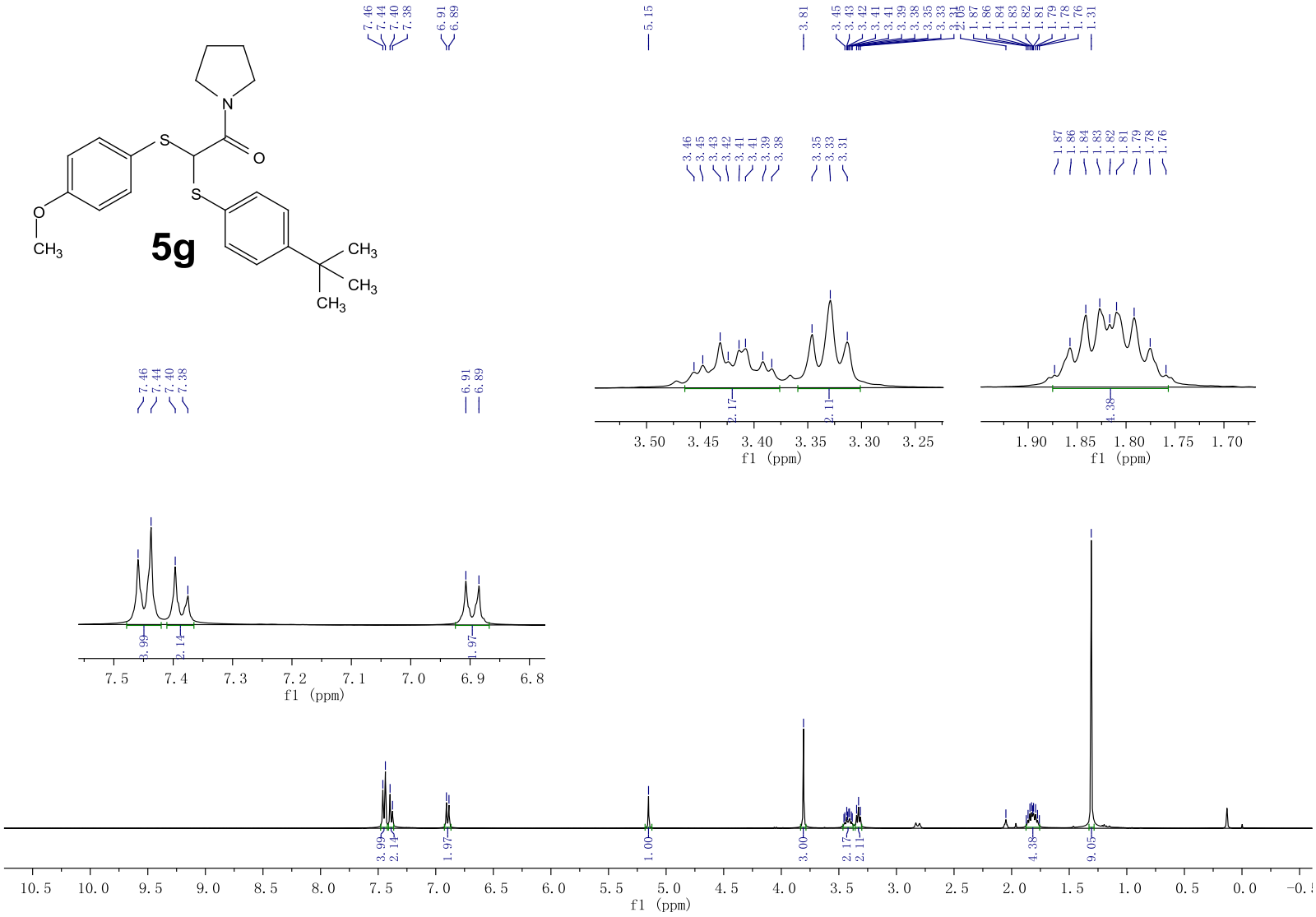
^{13}C NMR (100 MHz, CD_3COCD_3)



^{1}H NMR (400 MHz, CD_3COCD_3)



^{13}C NMR (100 MHz, CD_3COCD_3)



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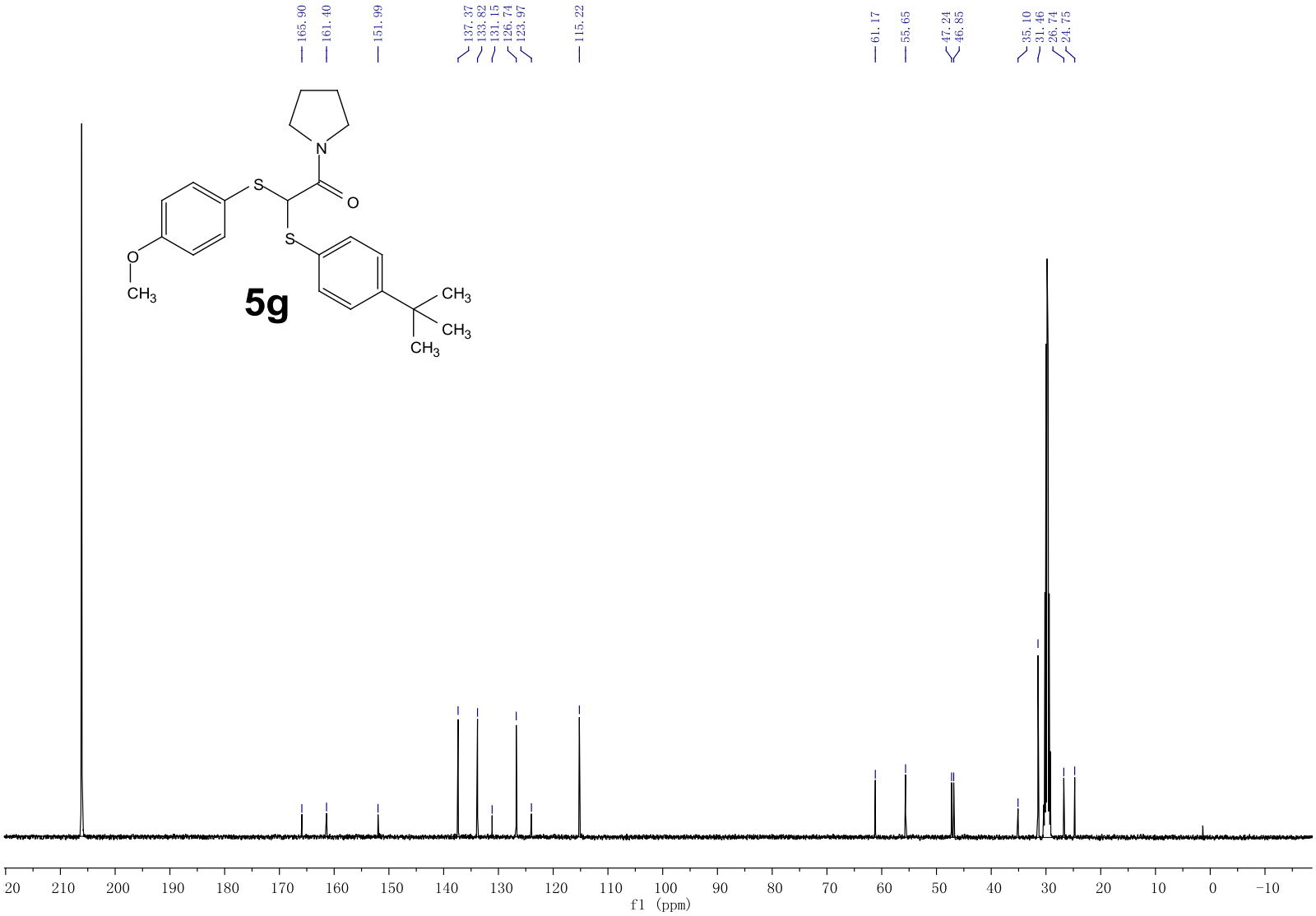
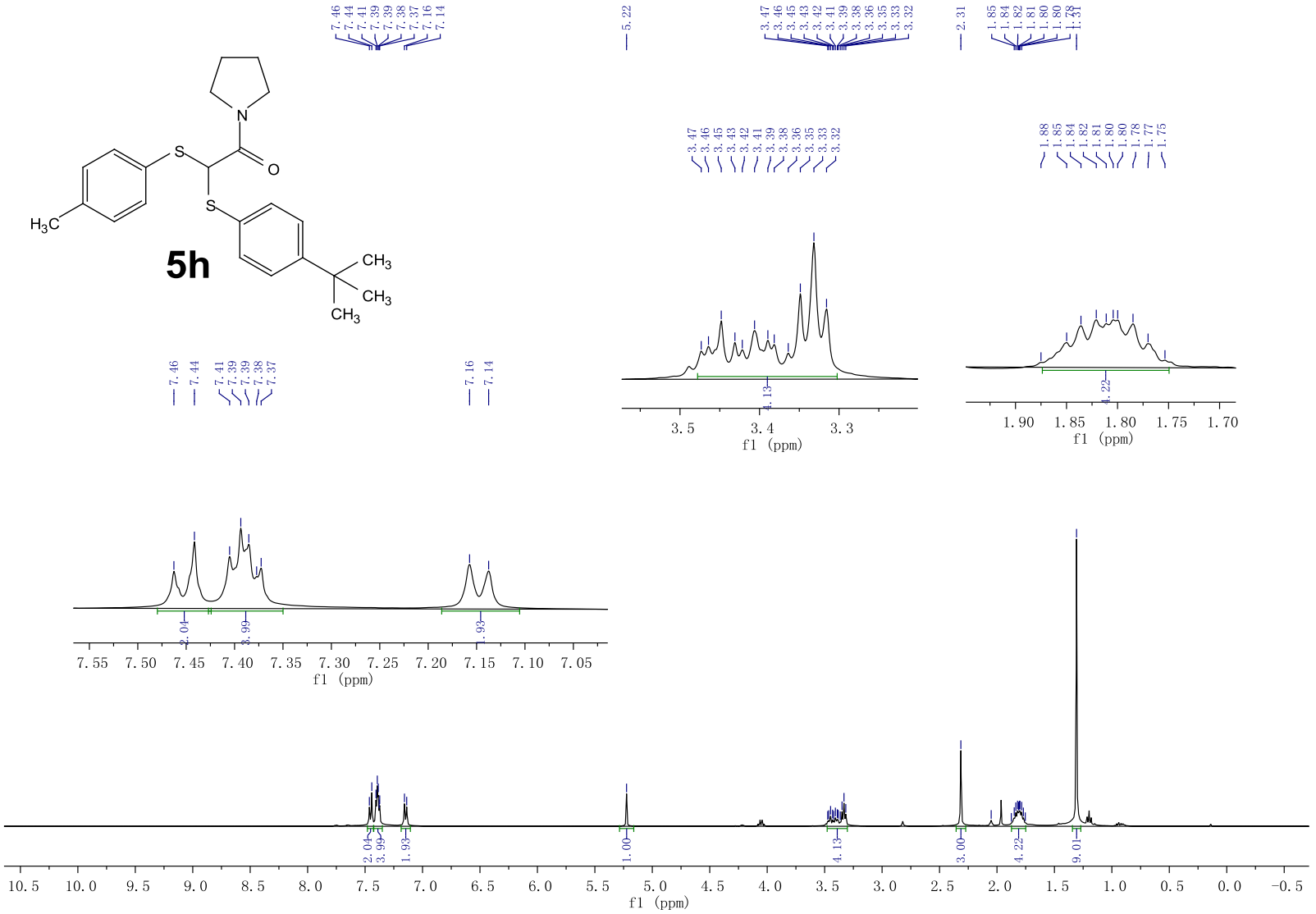
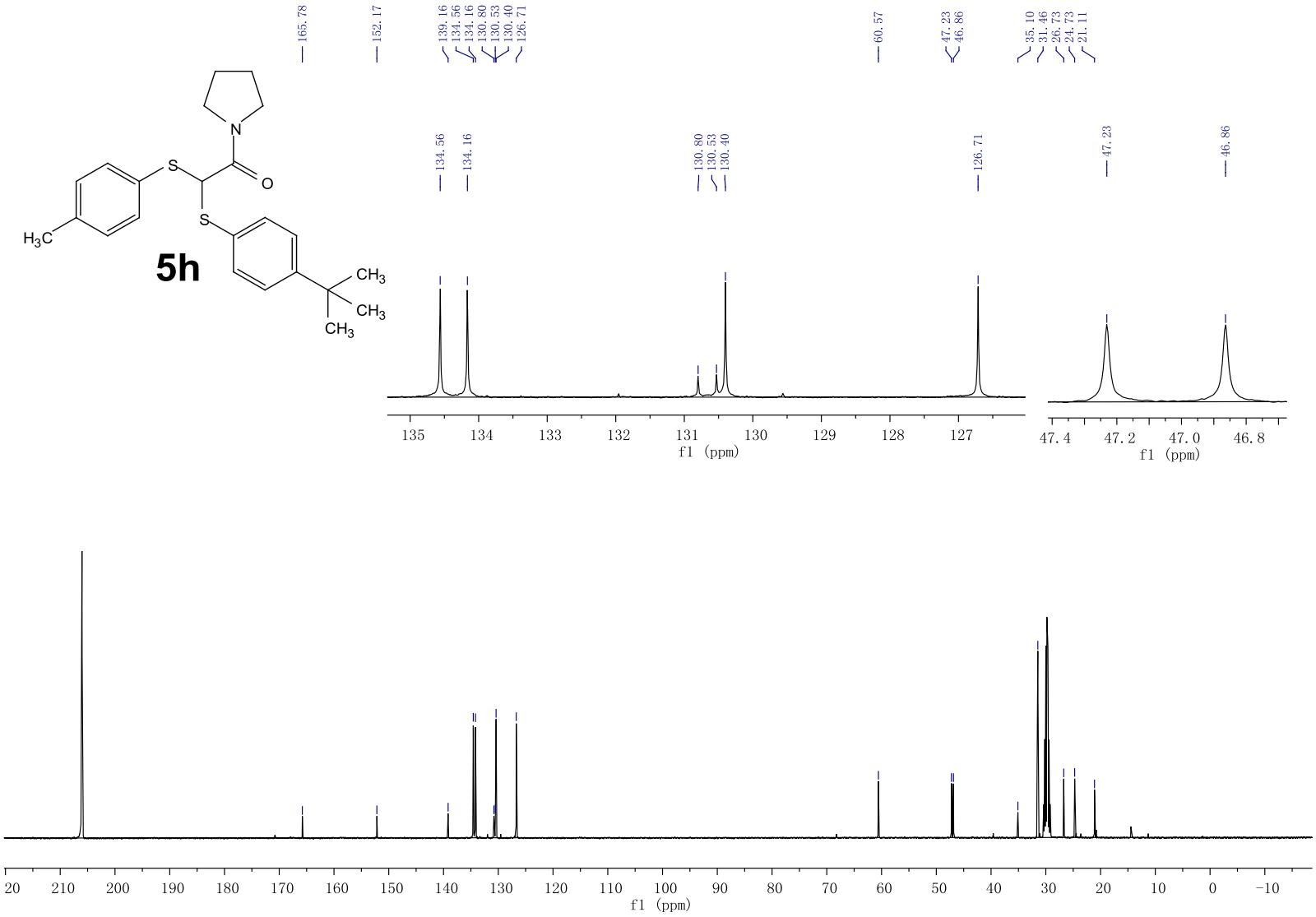


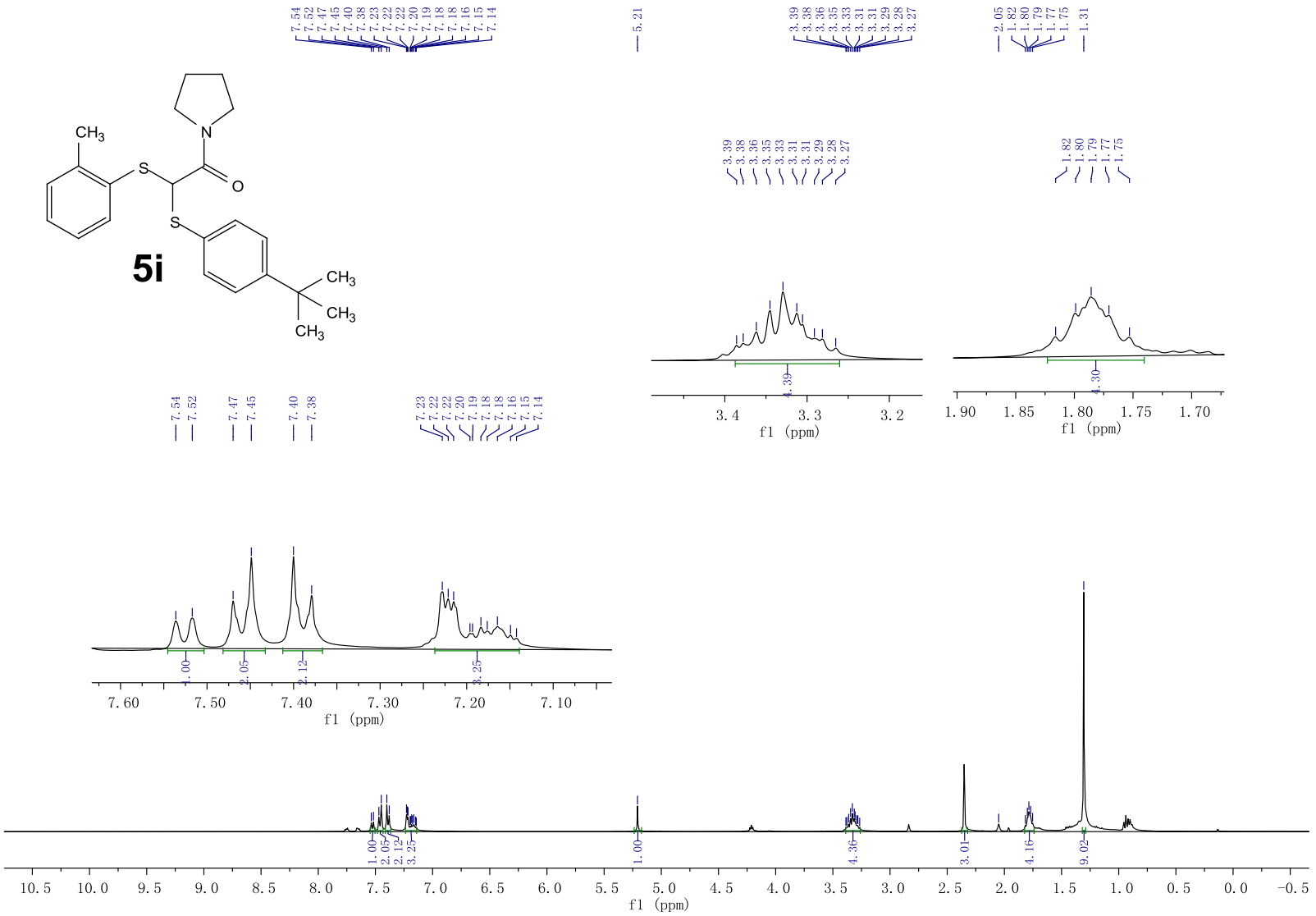
^{13}C NMR (100 MHz, CD_3COCD_3)



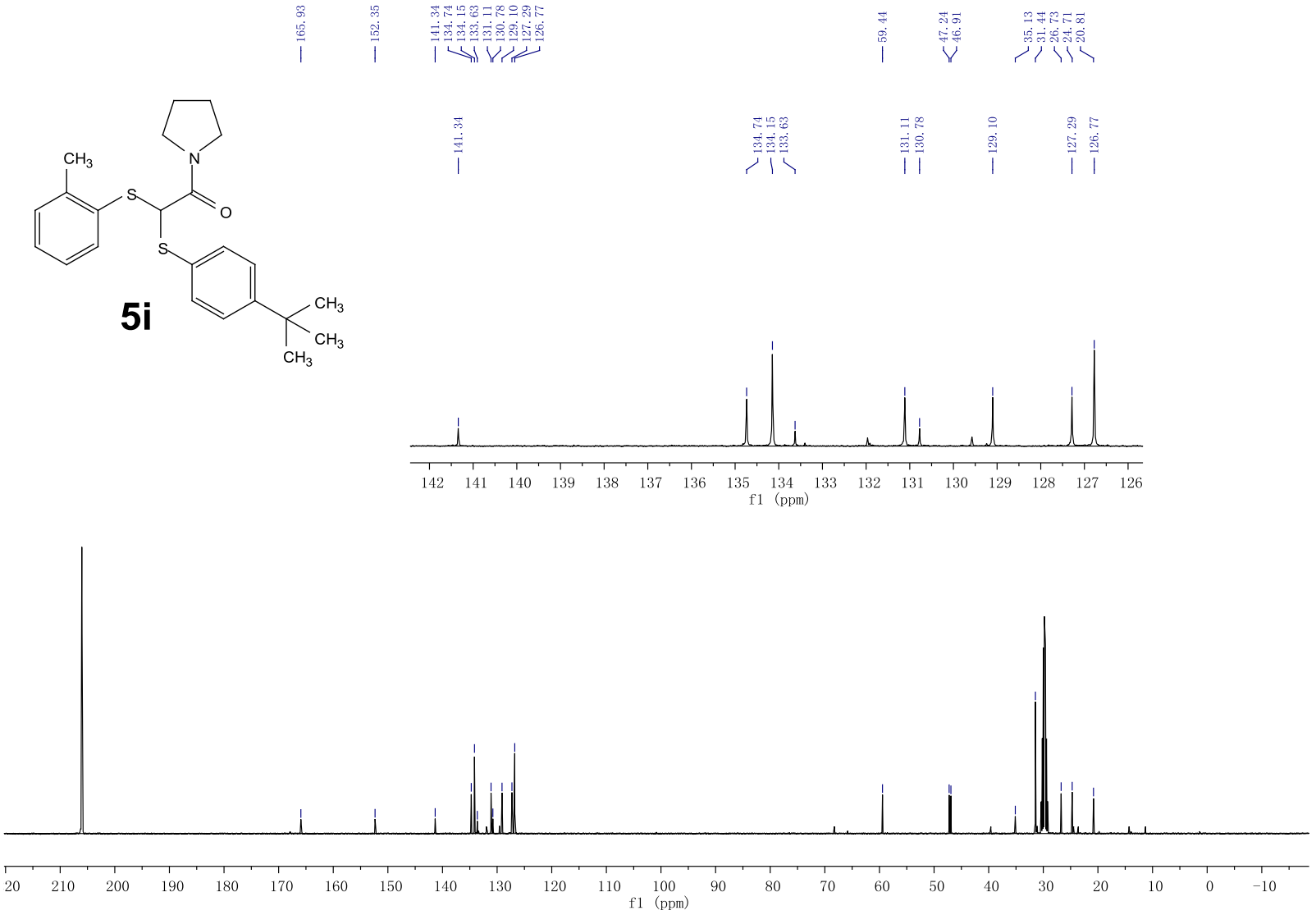
^{1}H NMR (400 MHz, CD_3COCD_3)



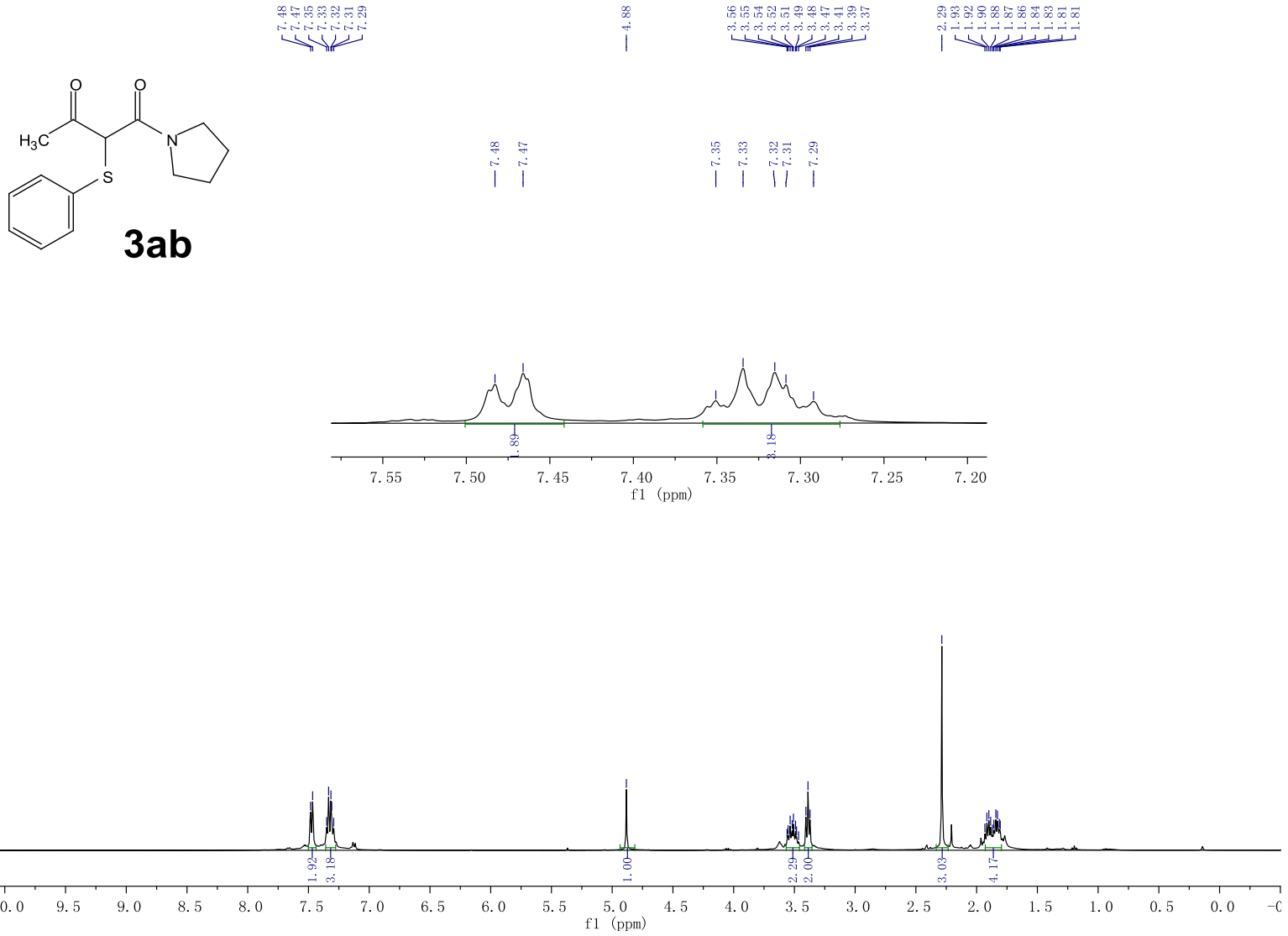
^{13}C NMR (100 MHz, CD_3COCD_3)



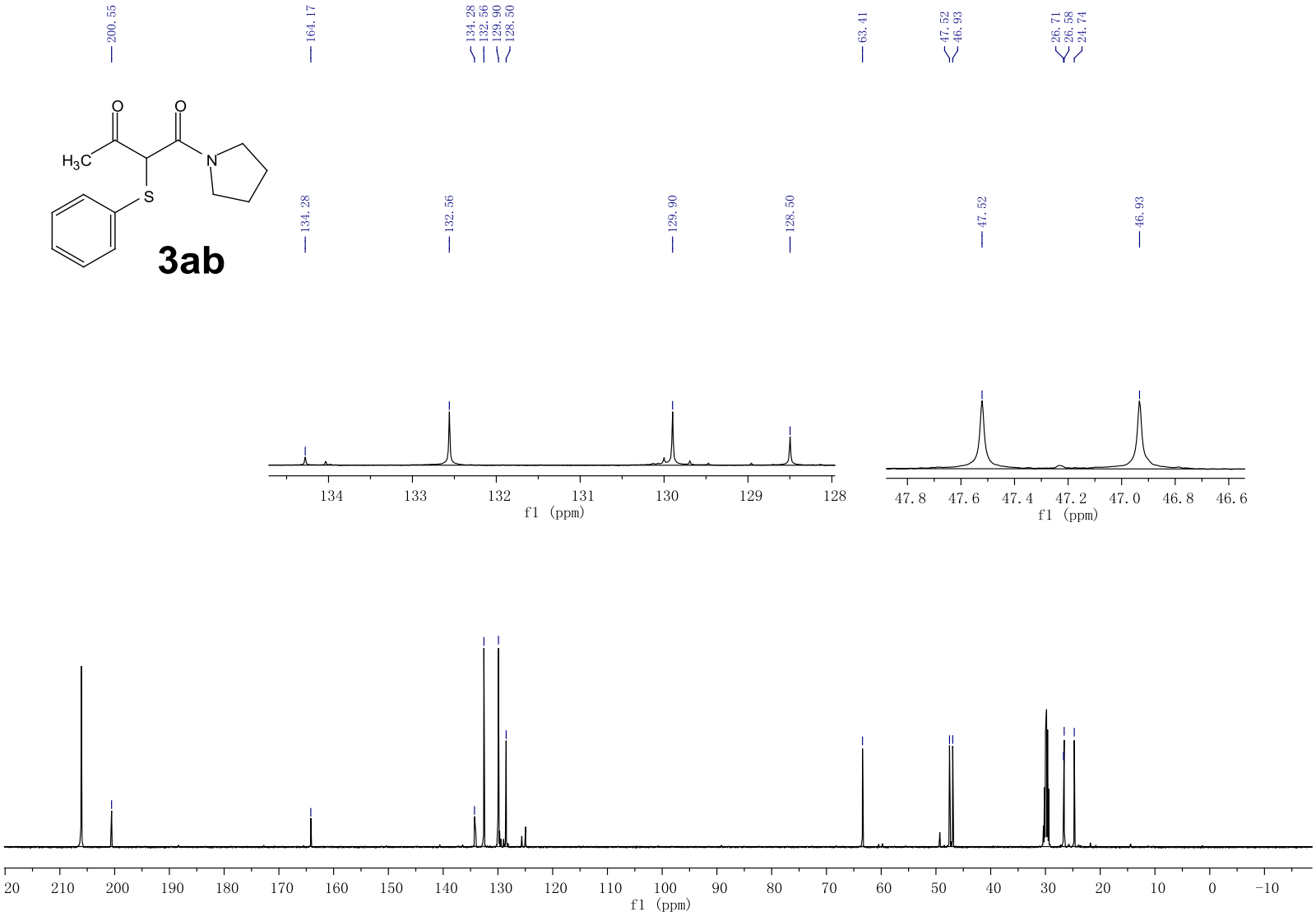
^{1}H NMR (400 MHz, CD_3COCD_3)



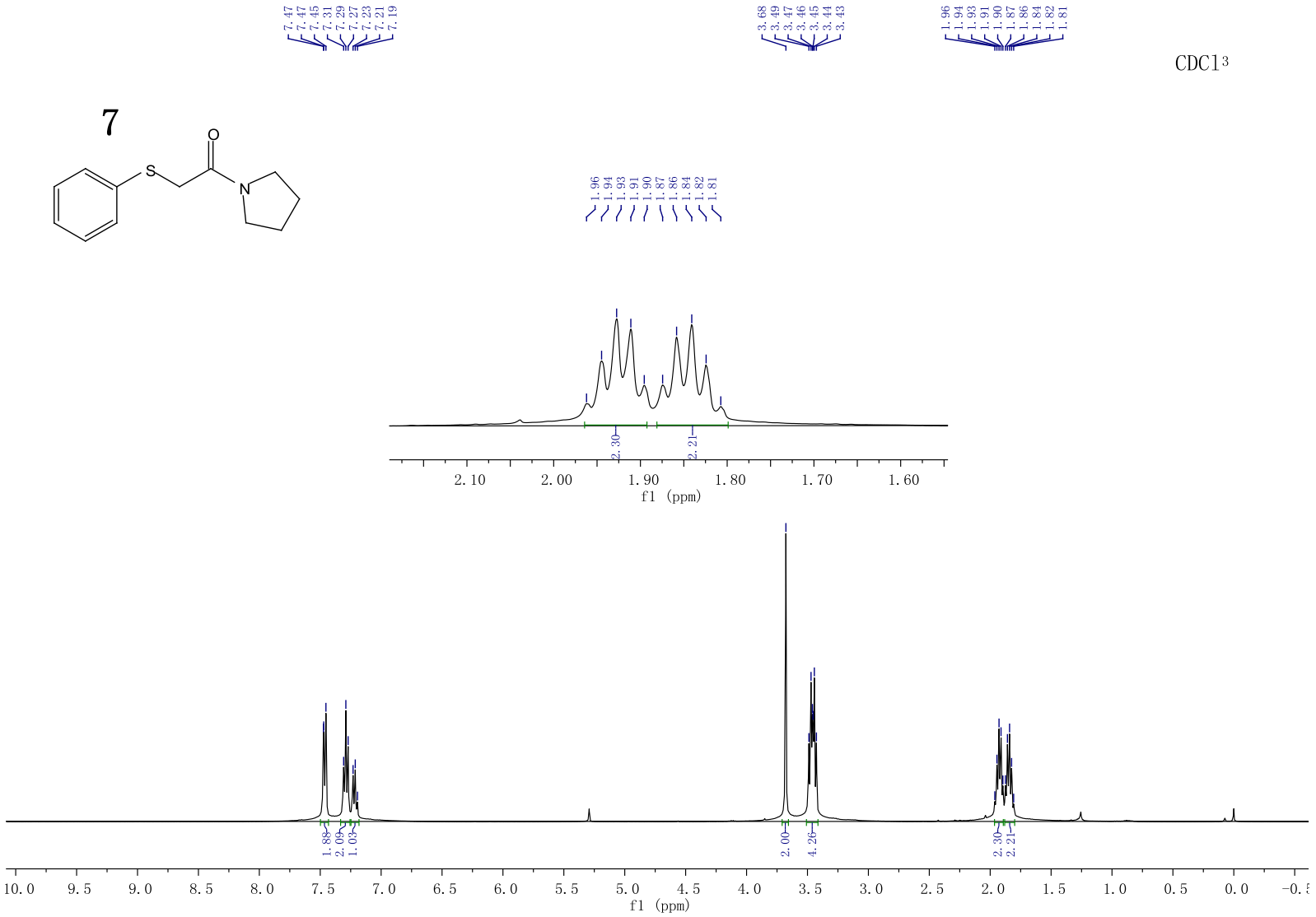
^{13}C NMR (100 MHz, CD_3COCD_3)



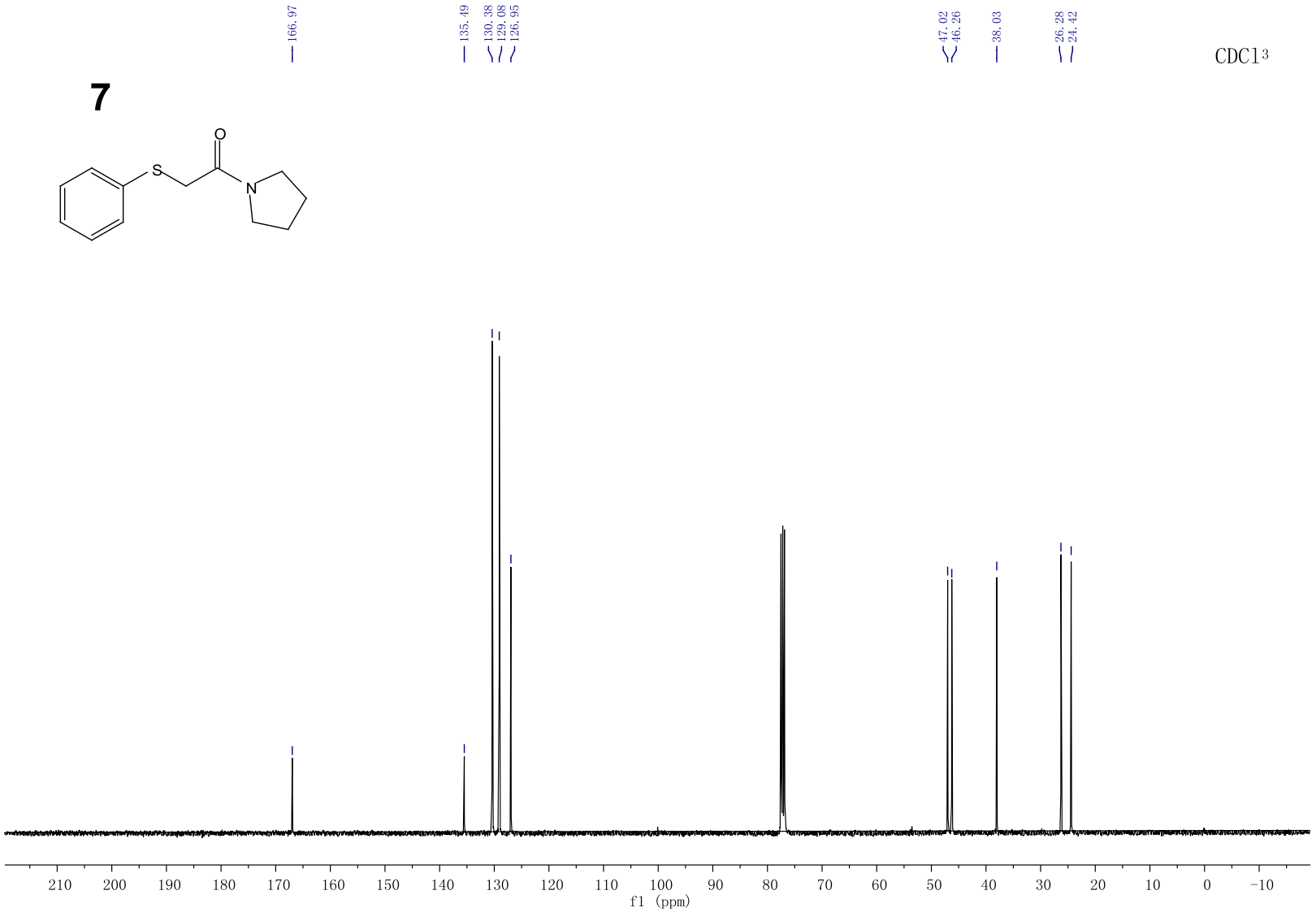
^{1}H NMR (400 MHz, CD_3COCD_3)



^{13}C NMR (100 MHz, CD_3COCD_3)



^{1}H NMR (400 MHz, CD_3Cl)



^{13}C NMR (100 MHz, CD_3Cl)