Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Stereoselective defluorinative carboxylation of *gem*-difluoroalkenes with carbon dioxide

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Contents

1. General information

Reactions were monitored by thin layer chromatography using UV light or I_2 to visualize the course of reaction. Purification of reaction products was carried out by flash chromatography on silica gel. Chemical yields refer to pure isolated substances. ^{1}H, ^{13}C and ^{19}F NMR spectra were obtained using a Bruker DPX-400 spectrometer. Chemical shifts were reported in ppm with TMS as the internal standard. The following abbreviations were used to designate chemical shift multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, h = heptet, m = multiplet, br = broad.

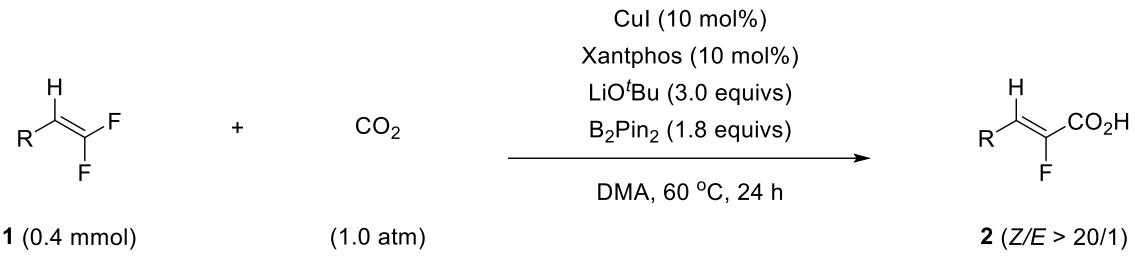
CuI (anhydrous, 99.995% trace metals basis) was purchased from Aldrich. Xantphos, LiO^tBu and B_2Pin_2 was purchased from J&K Scientific. Anhydrous DMA was prepared by first pre-dried with anhydrous Na_2SO_4, then distilled from $CaSO_4$ into 4Å molecular sieves. CO_2 (99.999%) was commercially available and was dried by conc. H_2SO_4. The substrates **1a-t**[1], **3a-d**[1], **5a-d**[2] and **7a-d**[3] were synthesized according to literature methods.

List of abbreviation:

Entry	Chemical name	Abbreviation
1	*N*, *N*-Dimethyl formamide	DMF
2	*N*, *N*-Dimethyl acetamide	DMA
3	Petroleum ether	PE
4	Ethyl acetate	EtOAc
5	Methanol	MeOH

1 H. Sakaguchi, Y. Uetake, M. Ohashi, T. Niwa, S. Ogoshi and T. Hosoya, *J. Am. Chem. Soc.*, 2017, **139**, 12855.
2 J. Wu, J. Xiao, W. Dai and S. Cao, *RSC Adv.*, 2015, **5**, 34498.
3 J. Hu, X. Han, Y. Yuan and Z. Shi, *Angew. Chem. Int. Ed.*, 2017, **56**, 13342.

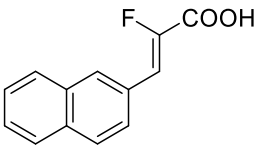
2. General procedure for the defluorinative carboxylation



1 (0.4 mmol) (1.0 atm) **2** (*Z*/*E* > 20/1)

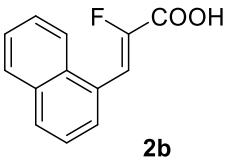
In a glovebox under argon, a flame-dried Schlenk tube (10 mL) fitted with a stirring bar was charged with CuI (7.6 mg, 0.04 mmol), Xantphos (23.1 mg, 0.04 mmol), LiOtBu (96.1 mg, 1.2 mmol), bis(pinacolato)diboron (182.8 mg, 0.72 mmol) and *gem*-difluoroalkenes (0.4 mmol). The Schlenk tube was taken out of the glovebox and then evacuated and back-filled with CO_2 for three times. Subsequently, freshly anhydrous DMA (2.0 mL) was added via syringe under a positive CO_2 atmosphere. Once added, the reaction tube was sealed at atmospheric pressure of CO_2 (1 atm). The mixture was stirred at 60 °C for 24 h. After the tube was cooled to room temperature, the reaction mixture was diluted with EtOAc (3.0 mL), quenched by 2 N HCl (3.0 mL) and then extracted by EtOAc (3×5 mL). Finally, the combined organic phases were dried over Na_2SO_4, filtered and concentrated *in vacuo*. The residue was purified by a silica gel flash column chromatography (petroleum ether/EtOAc 10/1, then EtOAc/MeOH 20/1 with 0.1% AcOH added) to give the desired product.

3. Characterization Data



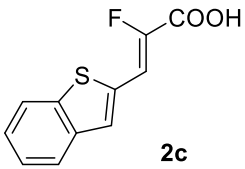
2a

84.0 mg, 97% yield, white solid. ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.26 (s, 1H), 7.99-7.93 (m, 3H), 7.84-7.81 (m, 1H), 7.61-7.55 (m, 2H), 7.20 (d, J = 36.4 Hz, 1H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 162.44 (d, J = 34.4 Hz, 1C), 147.87 (d, J = 263.9 Hz, 1C), 133.52 (d, J = 1.9 Hz, 1C), 133.22, 130.79 (d, J = 7.8 Hz, 1C), 129.01 (d, J = 4.2 Hz, 1C), 128.92 (d, J = 2.5 Hz, 1C), 128.04, 127.86, 127.23, 127.10 (d, J = 8.1 Hz, 1C), 117.13; ^{19}F NMR (376 MHz, DMSO-d_6): δ -123.50; HRMS (ESI): Exact mass calcd for $C_{13}H_8FO_2^-$ [M-H]$^-$: 215.0514, found: 215.0514. The spectroscopic data correspond to those previously reported in the literature.[4]



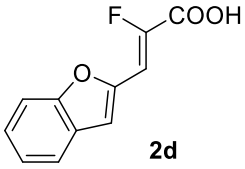
2b

80.0 mg, 93% yield, white solid. ^{1}H NMR (400 MHz, acetone-d_6): δ 8.16 (d, J = 8.4 Hz, 1H), 8.02-7.97 (m, 3H), 7.75 (d, J = 33.6 Hz, 1H), 7.68-7.58 (m, 3H); ^{13}C NMR (125 MHz, acetone-d_6): δ 161.40 (d, J = 35.9 Hz, 1C), 148.20 (d, J = 264.5 Hz, 1C), 133.78, 131.34, 129.96 (d, J = 1.6 Hz, 1C), 128.80, 128.42 (d, J = 10.5 Hz, 1C), 127.23 (d, J = 3.5 Hz, 1C), 127.01, 126.23, 125.46, 123.51, 113.49 (d, J = 5.9 Hz, 1C); ^{19}F NMR (376 MHz, acetone-d_6): δ -125.49; HRMS (ESI): Exact mass calcd for $C_{13}H_8FO_2^-$ [M-H]$^-$: 215.0514, found: 215.0517. The spectroscopic data correspond to those previously reported in the literature.[5]



2c

73.5 mg, 83% yield, yellow solid. ^{1}H NMR (500 MHz, acetone-d_6): δ 8.00-7.98 (m, 1H), 7.93-7.91 (m, 1H), 7.80 (s, 1H), 7.51-7.41 (m, 3H); ^{13}C NMR (125 MHz, acetone-d_6): δ 160.85 (d, J = 33.9 Hz, 1C), 146.83 (d, J = 264.4 Hz, 1C), 141.64 (d, J = 8.5 Hz, 1C), 138.89, 133.62 (d, J = 6.0 Hz, 1C), 128.81 (d, J = 4.3 Hz, 1C), 125.99, 124.91, 124.43 (d, J = 2.1 Hz, 1C), 122.27, 112.09 (d, J = 8.0 Hz, 1C); ^{19}F NMR (376 MHz, acetone-d_6): δ -122.40; HRMS (ESI): Exact mass calcd for $C_{11}H_6FO_2S^-$ [M-H]$^-$: 221.0078, found: 221.0077.

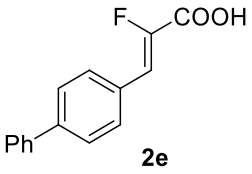


2d

59.0 mg, 72% yield, white solid. ^{1}H NMR (500 MHz, acetone-d_6): δ 7.74 (dd, J = 8.0 Hz, J = 1.5 Hz, 1H), 7.58 (d, J = 8.0 Hz, 1H), 7.45-7.41 (m, 1H), 7.35-7.30 (m, 2H), 7.14 (d, J = 30.0 Hz, 1H); ^{13}C NMR (125 MHz,

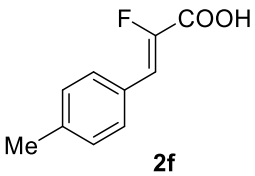
[4] K. Rousée, C. Schneider, S. Couve-Bonnaire, X. Pannecoucke, V. Levacher and C. Hoarau, *Chem. Eur. J.*, 2014, **20**, 15000.
[5] K. Notsu, Y. Zushi, S. Ota, T. Kawasaki-Takasuka and T. Yamazaki, *Chem. Eur. J.*, 2011, **17**, 9200.

acetone-d_6): δ 160.89 (d, J = 33.5 Hz, 1C), 155.17 (d, J = 2.1 Hz, 1C), 148.72 (d, J = 4.9 Hz, 1C), 147.77 (d, J = 269.0 Hz, 1C), 128.46, 126.22, 123.55, 121.90, 111.24, 111.13 (d, J = 10.7 Hz, 1C), 106.50 (d, J = 7.5 Hz, 1C); ^{19}F NMR (376 MHz, acetone-d_6): δ -118.47; HRMS (ESI): Exact mass calcd for $C_{11}H_6FO_3^-$ [M-H]$^-$: 205.0306, found: 205.0311.



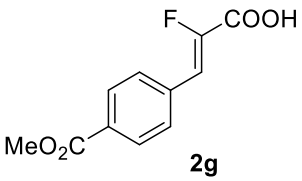
2e

82.0 mg, 85% yield, white solid. ^{1}H NMR (400 MHz, acetone-d_6): δ 7.85-7.83 (m, 2H), 7.79-7.77 (m, 2H), 7.75-7.72 (m, 2H), 7.52-7.48 (m, 2H), 7.43-7.38 (m, 1H), 7.09 (d, J = 35.6 Hz, 1H); ^{13}C NMR (125 MHz, acetone-d_6): δ 161.40 (d, J = 34.9 Hz, 1C), 147.35 (d, J = 263.8 Hz, 1C), 142.04 (d, J = 2.9 Hz, 1C),139.89, 130.77 (d, J = 8.0 Hz, 1C), 130.40 (d, J = 4.4 Hz, 1C), 128.97, 127.88, 127.24, 126.85, 116.71 (d, J = 4.6 Hz, 1C); ^{19}F NMR (376 MHz, acetone-d_6): δ -125.04; HRMS (ESI): Exact mass calcd for $C_{15}H_{10}FO_2^-$ [M-H]$^-$: 241.0670, found: 241.0666.



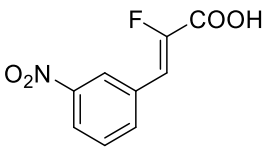
2f

48.0 mg, 67% yield, white solid. ^{1}H NMR (400 MHz, acetone-d_6): δ 7.62 (d, J = 8.0 Hz, 2H), 7.29 (d, J = 8.0 Hz, 2H), 7.00 (d, J = 36.0 Hz, 1H), 2.37 (s, 3H); ^{13}C NMR (125 MHz, acetone-d_6): δ 161.66, 146.81 (d, J = 262.1 Hz, 1C), 139.98 (d, J = 2.8 Hz, 1C), 130.18 (d, J = 7.9 Hz, 1C), 129.53, 128.54 (d, J = 4.3 Hz, 1C), 117.13 (d, J = 4.9 Hz, 1C), 20.51; ^{19}F NMR (376 MHz, acetone-d_6): δ -126.32. The spectroscopic data correspond to those previously reported in the literature.[5]



2g

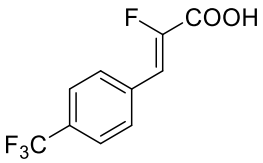
71.5 mg, 80% yield, white solid. ^{1}H NMR (400 MHz, acetone-d_6): δ 8.08 (d, J = 8.4 Hz, 2H), 7.86 (d, J = 8.4 Hz, 2H), 7.11 (d, J = 34.8 Hz, 1H), 3.91 (s, 3H); ^{13}C NMR (125 MHz, acetone-d_6): δ 165.77, 161.05 (d, J = 35.1 Hz, 1C), 148.40 (d, J = 267.5 Hz, 1C), 135.68 (d, J = 4.3 Hz, 1C), 130.76 (d, J = 2.8 Hz, 1C), 130.15 (d, J = 8.1 Hz, 1C), 129.68, 115.83 (d, J = 4.3 Hz, 1C), 51.65; ^{19}F NMR (376 MHz, acetone-d_6): δ -122.08; HRMS (ESI): Exact mass calcd for $C_{11}H_8FO_4^-$ [M-H]$^-$: 223.0412, found: 223.0419.



2h

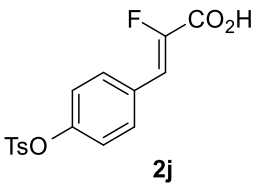
63.0 mg, 75% yield, white solid. ^{1}H NMR (400 MHz, acetone-d_6): δ 8.59 (s, 1H), 8.31-8.28 (m, 1H), 8.17-8.14 (m, 1H), 7.79 (t, J = 8.0 Hz, 1H), 7.23 (d, J = 34.4 Hz, 1H); ^{13}C NMR (125 MHz, acetone-d_6): δ 148.61, 135.88 (d, J =

7.6 Hz, 1C), 133.04 (d, *J* = 3.5 Hz, 1C), 130.27, 124.28 (d, *J* = 8.6 Hz, 1C), 123.88 (d, *J* = 1.8 Hz, 1C), 114.74; ^{19}F NMR (376 MHz, acetone-d_6): δ -121.67; HRMS (ESI): Exact mass calcd for $C_9H_5FO_4^-$ [M-H]$^-$: 210.0208, found: 210.0200.



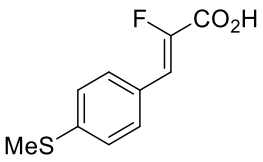
2i

73.0 mg, 78% yield, white solid. ^{1}H NMR (400 MHz, acetone-d_6): δ 7.95 (d, *J* = 8.0 Hz, 2H), 7.81 (d, *J* = 8.4 Hz, 2H), 7.13 (d, *J* = 34.8 Hz, 1H); ^{13}C NMR (125 MHz, acetone-d_6): δ 161.13 (d, *J* = 35.0 Hz, 1C), 148.77 (d, *J* = 267.4 Hz, 1C), 135.29, 130.60 (d, *J* = 8.1 Hz, 1C), 130.42-129.92 (m, 1C), 125.65 (d, *J* = 4.1 Hz, 1C), 124.17 (q, *J* = 269.8 Hz, 1C), 115.26; ^{19}F NMR (376 MHz, acetone-d_6): δ -63.42, -121.77; HRMS (ESI): Exact mass calcd for $C_{10}H_5F_4O_2^-$ [M-H]$^-$: 233.0231, found: 233.0230. The spectroscopic data correspond to those previously reported in the literature.[4]



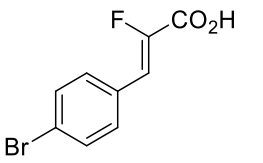
2j

122.0 mg, 91% yield, white solid. ^{1}H NMR (400 MHz, acetone-d_6): δ 7.78-7.72 (m, 4H), 7.49 (d, *J* = 7.6 Hz, 2H), 7.14 (d, *J* = 8.8 Hz, 2H), 7.02 (d, *J* = 35.2 Hz, 1H), 2.47 (s, 3H); ^{13}C NMR (125 MHz, acetone-d_6): δ 161.21 (d, *J* = 35.4 Hz, 1C), 150.15 (d, *J* = 3.8 Hz, 1C), 147.66 (d, *J* = 265.0 Hz, 1C), 146.03, 132.28, 131.65 (d, *J* = 8.0 Hz, 1C), 130.42 (d, *J* = 4.1 Hz, 1C), 130.09, 128.41, 122.72, 115.57 (d, *J* = 4.5 Hz, 1C), 20.71; ^{19}F NMR (376 MHz, acetone-d_6): δ -124.53; HRMS (ESI): Exact mass calcd for $C_{16}H_{12}FO_5S^-$ [M-H]$^-$: 335.0395, found: 335.0386.



2k

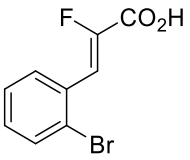
72.5 mg, 86% yield, white solid. ^{1}H NMR (400 MHz, acetone-d_6): δ 7.66 (d, *J* = 8.4 Hz, 2H), 7.34 (d, *J* = 8.8 Hz, 2H), 6.99 (d, *J* = 36.0 Hz, 1H), 2.54 (s, 3H); ^{13}C NMR (125 MHz, acetone-d_6): δ 161.40 (d, *J* = 35.0 Hz, 1C), 146.86 (d, *J* = 262.4 Hz, 1C), 141.58 (d, *J* = 3.1 Hz, 1C), 130.54 (d, *J* = 8.1 Hz, 1C), 127.59 (d, *J* = 4.5 Hz, 1C), 125.70, 116.75 (d, *J* = 4.6 Hz, 1C), 13.87; ^{19}F NMR (376 MHz, acetone-d_6): δ -125.79; HRMS (ESI): Exact mass calcd for $C_{10}H_8FO_2S^-$ [M-H]$^-$: 211.0235, found: 211.0235.



2l

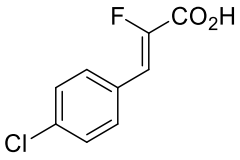
86.0 mg, 88% yield, white solid. ^{1}H NMR (400 MHz, acetone-d_6): δ 7.70-7.65 (m, 4H), 7.03 (d, *J* = 35.2 Hz, 1H); ^{13}C NMR (125 MHz, acetone-d_6): δ 161.19 (d, *J* = 35.4 Hz, 1C), 147.72 (d, *J* = 265.0 Hz, 1C), 132.02, 131.89 (d,

J = 8.4 Hz, 1C), 130.53 (d, *J* = 4.1 Hz, 1C), 123.30 (d, *J* = 3.8 Hz, 1C), 115.86 (d, *J* = 4.5 Hz, 1C); ^{19}F NMR (376 MHz, acetone-*d*$_6$): δ -123.89; HRMS (ESI): Exact mass calcd for $C_9H_5Br^{79}FO_2^-$ [M-H]$^-$: 242.9462, found: 242.9461.



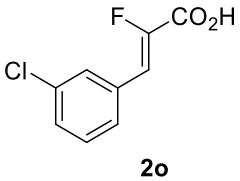
2m

70.0 mg, 72% yield, white solid. ^{1}H NMR (400 MHz, acetone-*d*$_6$): δ 7.92 (dd, *J* = 7.8 Hz, *J* = 2.0 Hz, 1H), 7.75 (dd, *J* = 8.4 Hz, *J* = 1.2 Hz, 1H), 7.52-7.48 (m, 1H), 7.38-7.30 (m, 2H); ^{13}C NMR (125 MHz, acetone-*d*$_6$): δ 148.38 (d, *J* = 268.0 Hz, 1C), 133.17, 131.23 (d, *J* = 12.4 Hz, 1C), 131.11 (d, *J* = 1.6 Hz, 1C), 130.91 (d, *J* = 4.3 Hz, 1C), 128.06, 124.08 (d, *J* = 1.6 Hz, 1C), 114.84; ^{19}F NMR (376 MHz, acetone-*d*$_6$): δ -124.41; HRMS (ESI): Exact mass calcd for $C_9H_5Br^{79}FO_2^-$ [M-H]$^-$: 242.9462, found: 242.9460.



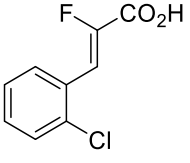
2n

69.5 mg, 87% yield, white solid. ^{1}H NMR (400 MHz, acetone-*d*$_6$): δ 7.75 (d, *J* = 8.8 Hz, 2H), 7.51 (d, *J* = 8.4 Hz, 2H), 7.04 (d, *J* = 35.2 Hz, 1H); ^{13}C NMR (125 MHz, acetone-*d*$_6$): δ 161.26, 147.65 (d, *J* = 264.8 Hz, 1C), 134.92 (d, *J* = 3.6 Hz, 1C), 131.70 (d, *J* = 8.3 Hz, 1C), 130.15 (d, *J* = 4.1 Hz, 1C), 129.00, 115.79 (d, *J* = 4.5 Hz, 1C); ^{19}F NMR (376 MHz, acetone-*d*$_6$): δ -124.27; HRMS (ESI): Exact mass calcd for $C_9H_5Cl^{35}FO_2^-$ [M-H]$^-$: 198.9968, found: 198.9967.



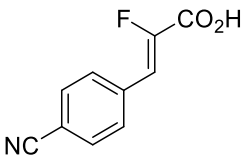
2o

64.0 mg, 80% yield, white solid. ^{1}H NMR (500 MHz, acetone-*d*$_6$): δ 7.75 (s, 1H), 7.68 (d, *J* = 7.5 Hz, 1H), 7.51-7.45 (m, 2H), 7.04 (d, *J* = 35.0 Hz, 1H); ^{13}C NMR (125 MHz, acetone-*d*$_6$): δ 161.25 (d, *J* = 35.1 Hz, 1C), 148.15 (d, *J* = 266.0 Hz, 1C), 134.19, 133.33 (d, *J* = 4.1 Hz, 1C), 130.53, 129.59 (d, *J* = 8.5 Hz, 1C), 129.42 (d, *J* = 2.5 Hz, 1C), 128.53 (d, *J* = 8.1 Hz, 1C), 115.51 (d, *J* = 4.2 Hz, 1C); ^{19}F NMR (376 MHz, acetone-*d*$_6$): δ -122.96; HRMS (ESI): Exact mass calcd for $C_9H_5Cl^{35}FO_2^-$ [M-H]$^-$: 198.9968, found: 198.9965.



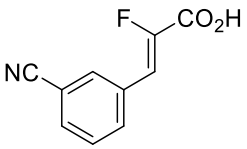
2p

65.5 mg, 82% yield, white solid. ^{1}H NMR (400 MHz, acetone-*d*$_6$): δ 7.96-7.94 (m, 1H), 7.57-7.55 (m, 1H), 7.47-7.44 (m, 2H), 7.37 (d, *J* = 34.0 Hz, 1H); ^{13}C NMR (125 MHz, acetone-*d*$_6$): δ 161.14, 148.44 (d, *J* = 268.0 Hz, 1C), 133.65 (d, *J* = 1.9 Hz, 1C), 131.12 (d, *J* = 12.88 Hz, 1C), 130.98 (d, *J* = 1.8 Hz, 1C), 129.82, 129.10 (d, *J* = 4.5 Hz, 1C), 127.51, 112.16 (d, *J* = 3.8 Hz, 1C); ^{19}F NMR (376 MHz, acetone-*d*$_6$): δ -124.01; HRMS (ESI): Exact mass calcd for $C_9H_5Cl^{35}FO_2^-$ [M-H]$^-$: 198.9968, found: 198.9965.



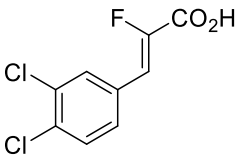
2q

65.5 mg, 86% yield, white solid. ^{1}H NMR (400 MHz, acetone-d_6): δ 7.93 (d, J = 8.8 Hz, 2H), 7.88 (d, J = 8.8 Hz, 2H), 7.13 (d, J = 34.8 Hz, 1H); ^{13}C NMR (125 MHz, acetone-d_6): δ 160.94 (d, J = 34.9 Hz, 1C), 149.00 (d, J = 268.0 Hz, 1C), 135.81 (d, J = 4.0 Hz, 1C), 132.55, 130.70 (d, J = 8.2 Hz, 1C), 118.13, 115.18 (d, J = 4.0 Hz, 1C), 112.57 (d, J = 3.1 Hz, 1C); ^{19}F NMR (376 MHz, acetone-d_6): δ -120.87; HRMS (ESI): Exact mass calcd for $C_{10}H_5FNO_2^-$ [M-H]$^-$: 190.0310, found: 190.0313. The spectroscopic data correspond to those previously reported in the literature. [4]



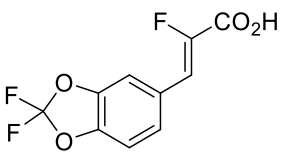
2r

72.5 mg, 95% yield, white solid. ^{1}H NMR (500 MHz, acetone-d_6): δ 8.09 (s, 1H), 8.05 (d, J = 8.0 Hz, 1H), 7.83 (d, J = 7.5 Hz, 1H), 7.71 (t, J = 8.0 Hz, 1H), 7.11 (d, J = 33.5 Hz, 1H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 162.00 (d, J = 34.6 Hz, 1C), 148.83 (d, J = 267.0 Hz, 1C), 134.74 (d, J = 8.7 Hz, 1C), 133.76 (d, J = 7.2 Hz, 1C), 133.34 (d, J = 2.1 Hz, 1C), 132.71 (d, J = 3.6 Hz, 1C), 130.66, 118.79, 114.99 (d, J = 3.7 Hz, 1C), 112.59; ^{19}F NMR (376 MHz, DMSO-d_6): δ -120.91; HRMS (ESI): Exact mass calcd for $C_{10}H_5FNO_2^-$ [M-H]$^-$: 190.0310, found: 190.0312.



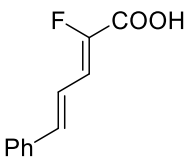
2s

78.0 mg, 83% yield, white solid. ^{1}H NMR (400 MHz, acetone-d_6): δ 7.92 (d, J = 2.0 Hz, 1H), 7.73-7.67 (m, 2H), 7.05 (d, J = 34.4 Hz, 1H); ^{13}C NMR (125 MHz, acetone-d_6): δ 161.13, 148.43 (d, J = 266.7 Hz, 1C), 132.74 (d, J = 3.4 Hz, 1C), 132.28, 131.88 (d, J = 3.9 Hz, 1C), 131.61 (d, J = 8.2 Hz, 1C), 130.99, 129.80 (d, J = 8.5 Hz, 1C), 114.56 (d, J = 4.1 Hz, 1C); ^{19}F NMR (376 MHz, acetone-d_6): δ -122.34; HRMS (ESI): Exact mass calcd for $C_9H_4Cl^{35}{}_2FO_2^-$ [M-H]$^-$: 232.9578, found: 232.9573.



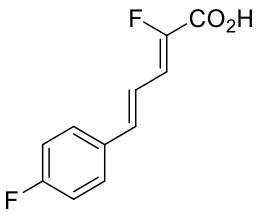
2t

80.5 mg, 82% yield, white solid. ^{1}H NMR (400 MHz, acetone-d_6): δ 7.68 (s, 1H), 7.59 (d, J = 8.4 Hz, 1H), 7.40 (d, J = 8.4 Hz, 1H), 7.08 (d, J = 34.8 Hz, 1H); ^{13}C NMR (125 MHz, acetone-d_6): δ 161.26, 147.42 (d, J = 264.8 Hz, 1C), 143.92 (d, J = 3.1 Hz, 1C), 143.79, 131.63 (t, J = 252.1 Hz, 1C), 128.03 (d, J = 3.8 Hz, 1C), 127.20 (d, J = 7.3 Hz, 1C), 115.81 (d, J = 4.0 Hz, 1C), 110.79 (d, J = 7.9 Hz, 1C), 110.15; ^{19}F NMR (376 MHz, acetone-d_6): δ -51.27, -124.86; HRMS (ESI): Exact mass calcd for $C_{10}H_4F_3O_4^-$ [M-H]$^-$: 245.0067, found: 245.0062.



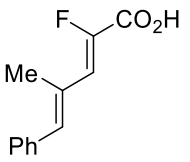
4a

56.0 mg, 73% yield, white solid. ^{1}H NMR (400 MHz, acetone-d_6): δ 7.64-7.61 (m, 2H), 7.43-7.33 (m, 3H), 7.21-7.07 (m, 2H), 6.90 (dd, J = 31.4 Hz, J = 10.4 Hz, 1H); ^{13}C NMR (125 MHz, acetone-d_6): δ 161.04 (d, J = 34.1 Hz, 1C), 146.87 (d, J = 260.6 Hz, 1C), 139.10 (d, J = 4.4 Hz, 1C), 136.30 (d, J = 1.9 Hz, 1C), 129.03, 128.84, 127.24, 118.65 (d, J = 8.9 Hz, 1C), 118.53 (d, J = 2.4 Hz, 1C); ^{19}F NMR (376 MHz, acetone-d_6): δ -128.60; HRMS (ESI): Exact mass calcd for $C_{11}H_8FO_2^-$ [M-H]$^-$: 191.0514, found: 191.0514.



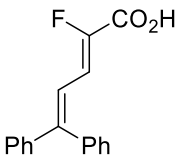
4b

56.0 mg, 67% yield, white solid. ^{1}H NMR (500 MHz, acetone-d_6): δ 7.70-7.66 (m, 2H), 7.19-7.14 (m, 2H), 7.12-7.04 (m, 2H), 6.87 (dd, J = 31.5 Hz, J = 10.0 Hz, 1H); ^{13}C NMR (125 MHz, acetone-d_6): δ 163.04 (d, J = 246.1 Hz, 1C), 161.17 (d, J = 34.2 Hz, 1C), 146.92 (d, J = 260.5 Hz, 1C), 137.71 (d, J = 4.5 Hz, 1C), 132.83 (dd, J = 3.6 Hz, J = 1.7 Hz, 1C), 129.29 (d, J = 8.2 Hz, 1C), 118.53 (d, J = 14.6 Hz, 1C), 118.50 (d, J = 3.0 Hz, 1C), 115.70 (d, J = 21.7 Hz, 1C); ^{19}F NMR (376 MHz, acetone-d_6): δ -113.57 (d, J = 2.6 Hz, 1F), -128.47 (d, J = 2.3 Hz, 1F); HRMS (ESI): Exact mass calcd for $C_{11}H_7F_2O_2^-$ [M-H]$^-$: 209.0420, found: 209.0415.



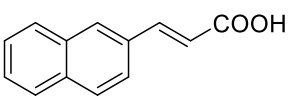
4c

52.0 mg, 63% yield, white solid. ^{1}H NMR (400 MHz, acetone-d_6): δ 7.42-7.41 (m, 4H), 7.35-7.30 (m, 1H), 6.97 (s, 1H), 6.76 (d, J = 35.6 Hz, 1H), 2.22 (dd, J = 2.8 Hz, J = 1.2 Hz, 3H); ^{13}C NMR (125 MHz, acetone-d_6): δ 161.77 (d, J = 34.9 Hz, 1C), 146.07 (d, J = 262.7 Hz, 1C), 137.18 (d, J = 6.1 Hz, 1C), 136.60, 131.09 (d, J = 5.7 Hz, 1C), 129.37, 128.32, 127.64, 121.91 (d, J = 4.0 Hz, 1C), 15.82 (d, J = 7.1 Hz, 1C); ^{19}F NMR (376 MHz, acetone-d_6): δ -127.49; HRMS (ESI): Exact mass calcd for $C_{12}H_{10}FO_2^-$ [M-H]$^-$: 205.0665, found: 205.0656.



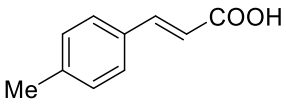
4d

75.0 mg, 70% yield, white solid. ^{1}H NMR (500 MHz, acetone-d_6): δ 7.54-7.48 (m, 3H), 7.38 (s, 5H), 7.28-7.26 (m, 2H), 7.07 (d, J = 11.5 Hz, 1H), 6.69 (dd, J = 31.0 Hz, J = 12.0 Hz, 1H); ^{13}C NMR (125 MHz, acetone-d_6): δ 161.09 (d, J = 34.4 Hz, 1C), 149.76 (d, J = 5.2 Hz, 1C), 147.81 (d, J = 263.0 Hz, 1C), 141.04, 138.52, 130.13, 128.82, 128.58, 128.51, 128.45, 127.94, 117.04 (d, J = 1.7 Hz, 1C), 115.78 (d, J = 7.4 Hz, 1C); ^{19}F NMR (376 MHz, acetone-d_6): δ -127.59; HRMS (ESI): Exact mass calcd for $C_{17}H_{12}FO_2^-$ [M-H]$^-$: 267.0827, found: 267.0823.



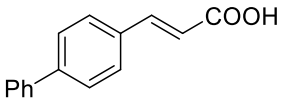
6a

34.2 mg, 87% yield, white solid. ^{1}H NMR (400 MHz, acetone-d_6): δ 8.16 (s, 1H), 7.99-7.93 (m, 3H), 7.88-7.84 (m, 2H), 7.58-7.56 (m, 2H), 6.67 (d, J = 16.0 Hz, 1H); ^{13}C NMR (125 MHz, acetone-d_6): δ 166.85, 144.55, 134.31, 133.49, 132.19, 129.79, 128.65, 128.54, 127.73, 127.22, 126.71, 123.71, 118.66; HRMS (ESI): Exact mass calcd for $C_{13}H_9O_2^-$ [M-H]$^-$: 197.0603, found: 197.0606.



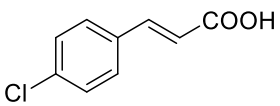
6b

18.4 mg, 57% yield, white solid. ^{1}H NMR (500 MHz, DMSO-d_6): δ 12.4 (br, 1H), 7.58 (d, J = 16.0 Hz, 1H), 7.53 (dd, J = 6.5 Hz, J = 2.0 Hz, 2H), 7.18 (d, J = 8.0 Hz, 2H), 6.47 (d, J = 16.0 Hz, 1H), 2.29 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 168.22, 144.37, 140.54, 131.93, 129.93, 128.58, 118.52, 21.41. The spectroscopic data correspond to those previously reported in the literature.[6]



6c

36.2 mg, 81% yield, white solid. ^{1}H NMR (500 MHz, DMSO-d_6): δ 12.44 (br, 1H), 7.79-7.75 (m, 2H), 7.73-7.63 (m, 5H), 7.50-7.43 (m, 2H), 7.41-7.36 (m, 1H), 6.58 (dd, J = 16.0 Hz, J = 9.5 Hz, 1H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 168.08, 143.88, 142.17, 139.70, 133.84, 129.45, 129.29, 128.36, 127.52, 127.12, 119.66. The spectroscopic data correspond to those previously reported in the literature.[7]

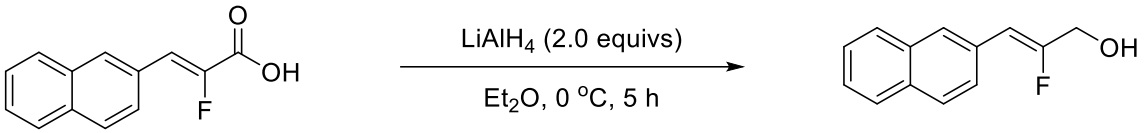


6d

25.2 mg, 70% yield, white solid. ^{1}H NMR (500 MHz, DMSO-d_6): δ 12.49 (br, 1H), 7.64 (d, J = 8.5 Hz, 2H), 7.57 (d, J = 16.5 Hz, 1H), 7.38 (d, J = 8.5 Hz, 2H), 6.51 (dd, J = 16.0 Hz, J = 5.5 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 167.91, 142.91, 135.18, 133.55, 130.19, 129.26, 120.42. The spectroscopic data correspond to those previously reported in the literature.[7]

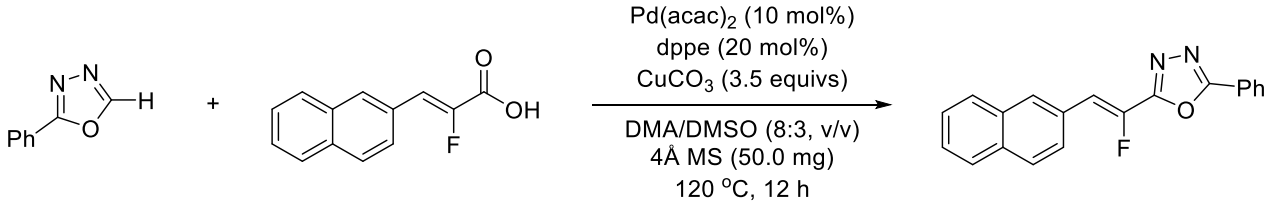
6 D. Basavaiah and A. J. Rao, *Synth. Commun.* 2002, **32**, 195.
7 S. T. Ahmed, F. Parmeggiani, N. J. Weise, S. L. Flitsch and N. J. Turner, *ACS Catal*. 2015, **5**, 5410.

4. Product elaboration



2a **8**, 87% yield

In a flame-dried Schlenk tube (10 mL) containing a stirring bar, a solution of **2a** (43.0 mg, 0.2 mmol, 1.0 equiv) and $LiAlH_4$ (15.0 mg, 0.4 mmol, 2.0 equivs) in Et_2O (2.0 mL) was stirred for 5 h at 0 °C and then quenched by water (0.5 mL). The solution was extracted with EtOAc (3×5 mL). The organic extracts were washed with water and brine, dried over anhydrous Na_2SO_4, filtered, and concentrated *in vacuo*. The residue was purified by chromatography on silica gel (petroleum ether/ethyl acetate 7/1 to 3/1) to give the product **8** (35.0 mg, 87% yield) as a white solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.93 (s, 1H), 7.82–7.79 (m, 3H), 7.69–7.67 (m, 1H), 7.47–7.45 (m, 2H), 5.94 (d, *J* = 38.5 Hz, 1H), 4.33 (d, *J* = 14.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 158.43 (d, *J* = 265.5 Hz, 1C), 133.37, 132.60 (d, *J* = 1.9 Hz, 1C), 130.24 (d, *J* = 3.0 Hz, 1C), 128.11, 128.09, 127.92 (d, *J* = 7.0 Hz, 1C), 127.59, 126.52 (d, *J* = 7.5 Hz, 1C), 126.24, 126.16, 107.66 (d, *J* = 6.5 Hz, 1C), 62.02 (d, *J* = 32.2 Hz, 1C); ^{19}F NMR (376 MHz, $CDCl_3$): δ -112.99. The spectroscopic data correspond to those previously reported in the literature.[8]



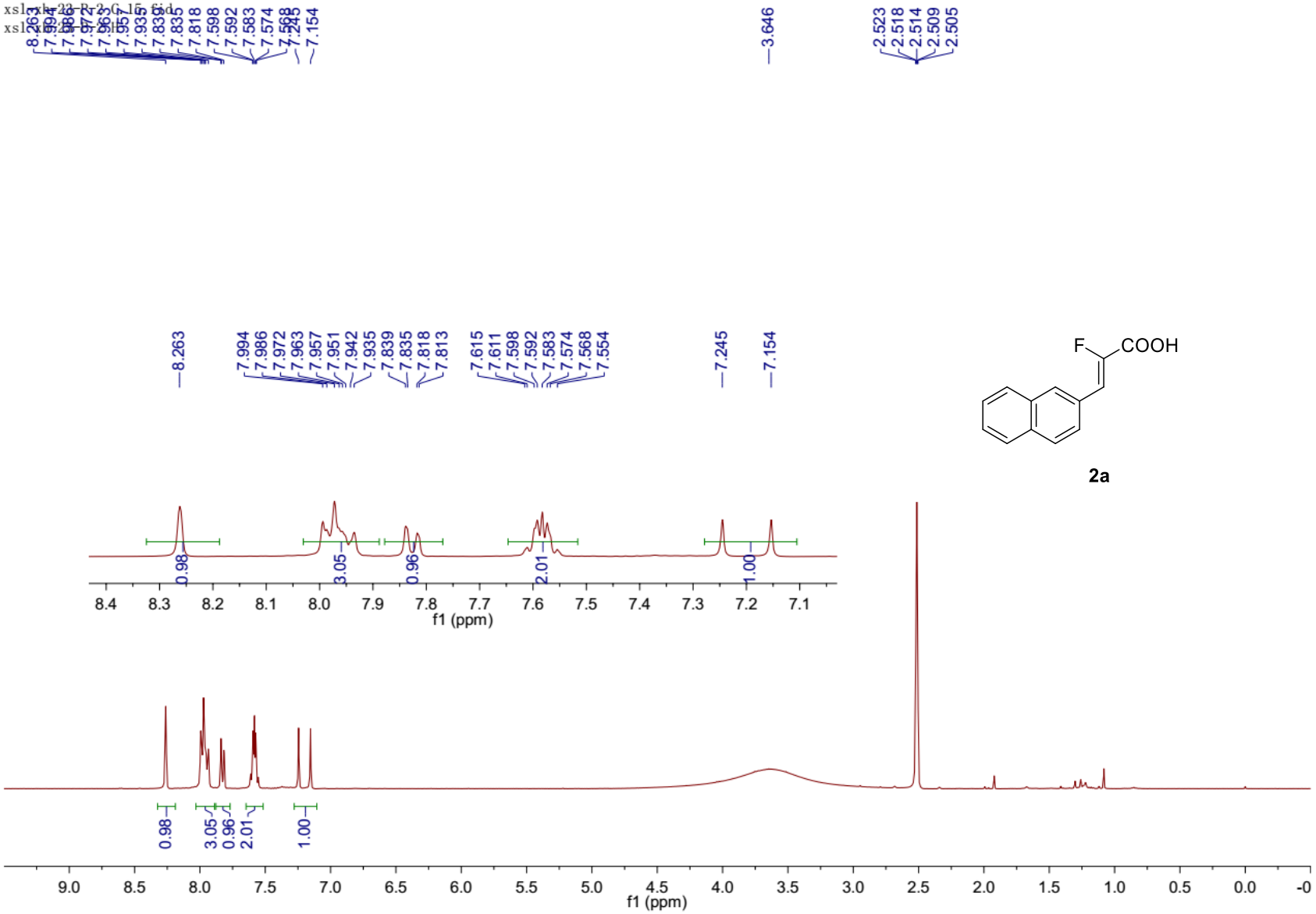
(0.1 mmol) **2a** (2.0 equivs) **9**, 75% yield

A flame-dried tube filled with nitrogen was charged with **2a** (43.0 mg, 0.2 mmol, 2.0 equivs), 1,3,4-oxadiazole (0.1 mmol, 14.5 mg, 1.0 equiv), dppe (8.0 mg, 0.02 mmol), $Pd(acac)_2$ (3.0 mg, 0.01 mmol), $CuCO_3$ (77.5 mg, 0.35 mmol, 3.5 equivs), 4 Å MS (50 mg). After which, the tube was purged and back-filled with nitrogen (this operation was repeated three times), then anhydrous DMA (0.7 mL) and DMSO (0.3 mL) were added. The tube was sealed and heated at 140 °C for 12 hours then cooled to room temperature. The reaction mixture was poured into NH_4Cl (10 mL)/EtOAc (10 mL), then was filtered through a plug of celite (washed with EtOAc) and extracted with EtOAc (3x10 mL). The combined organic layer was dried over Na_2SO_4, filtrated and the solvent was removed under reduced pressure. The crude product was then purified by flash silica gel column chromatography (petroleum ether/EtOAc 15/1 to 10/1) affording the desired compound **9** in 75% yield (23.5 mg) as a white solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.17-8.15 (m, 3H),

[8] J. Zhou, X. Jiang, C. Jin, Z. Guo, B. Su and W. Su, *Eur. J. Org. Chem.*, 2017, 3631.

7.92-7.85 (m, 4H), 7.63-7.52 (m, 5H), 7.07 (d, *J* = 37.0 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 164.86, 159.40 (d, *J* = 37.2 Hz, 1C), 143.42 (d, *J* = 254.7 Hz, 1C), 133.49 (d, *J* = 2.0 Hz, 1C), 132.23, 130.34 (d, *J* = 7.6 Hz, 1C), 129.21, 128.76, 128.72, 128.69, 128.56, 127.72, 127.33, 127.17, 126.73, 126.56 (d, *J* = 8.1 Hz, 1C), 123.25, 114.71 (d, *J* = 4.1 Hz, 1C); ^{19}F NMR (376 MHz, $CDCl_3$): δ -126.68. The spectroscopic data correspond to those previously reported in the literature.[9]

[9] C. Schneider, D. Masi, S. Couve-Bonnaire, X. Pannecoucke and C. Hoarau, *Angew. Chem. Int. Ed.*, 2013, **52**, 3246.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7.154
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COOH
2a
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0.96
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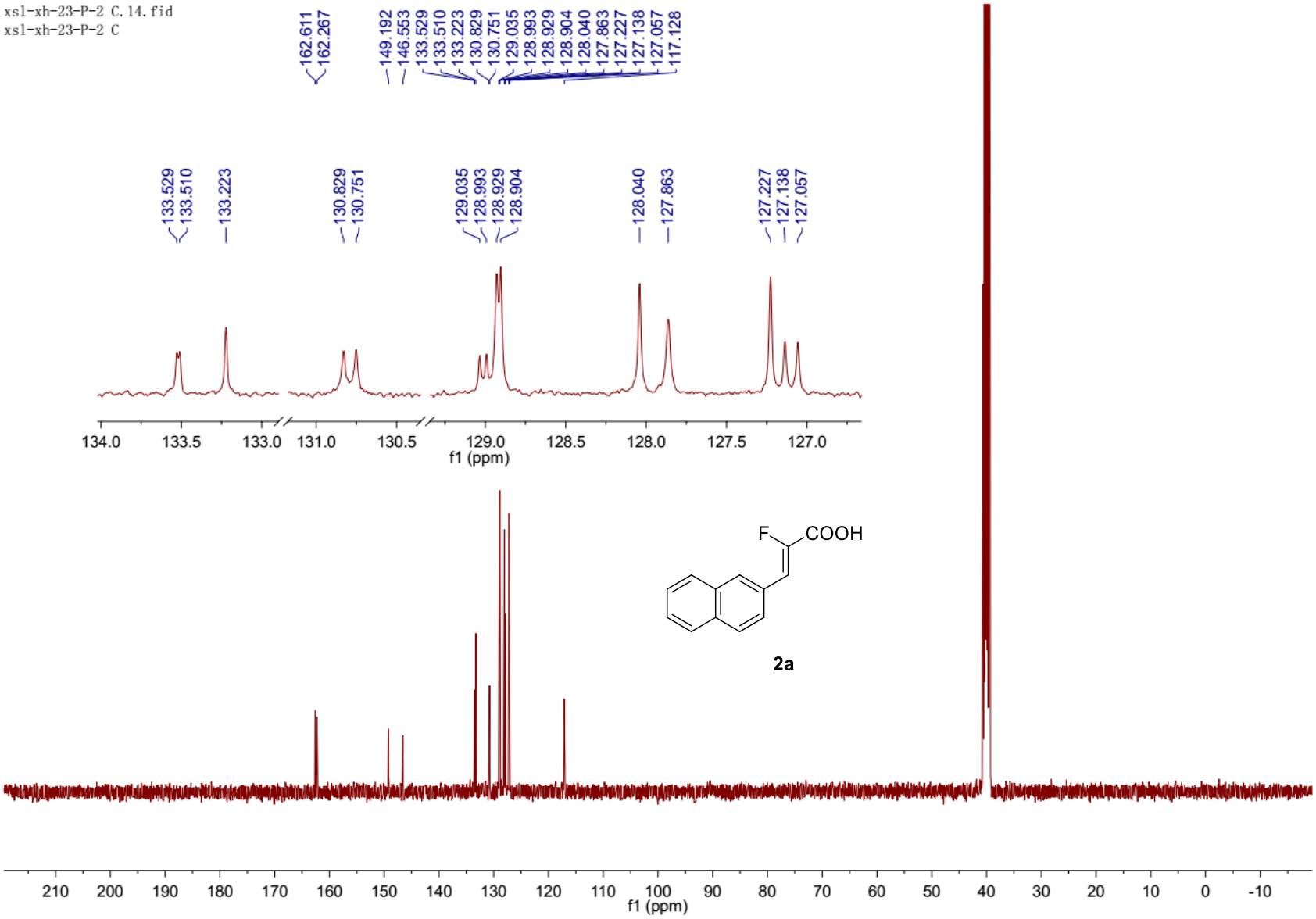
f1 (ppm)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COOH
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180
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160
150
140
130
120
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80
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20
10
0
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f1 (ppm)

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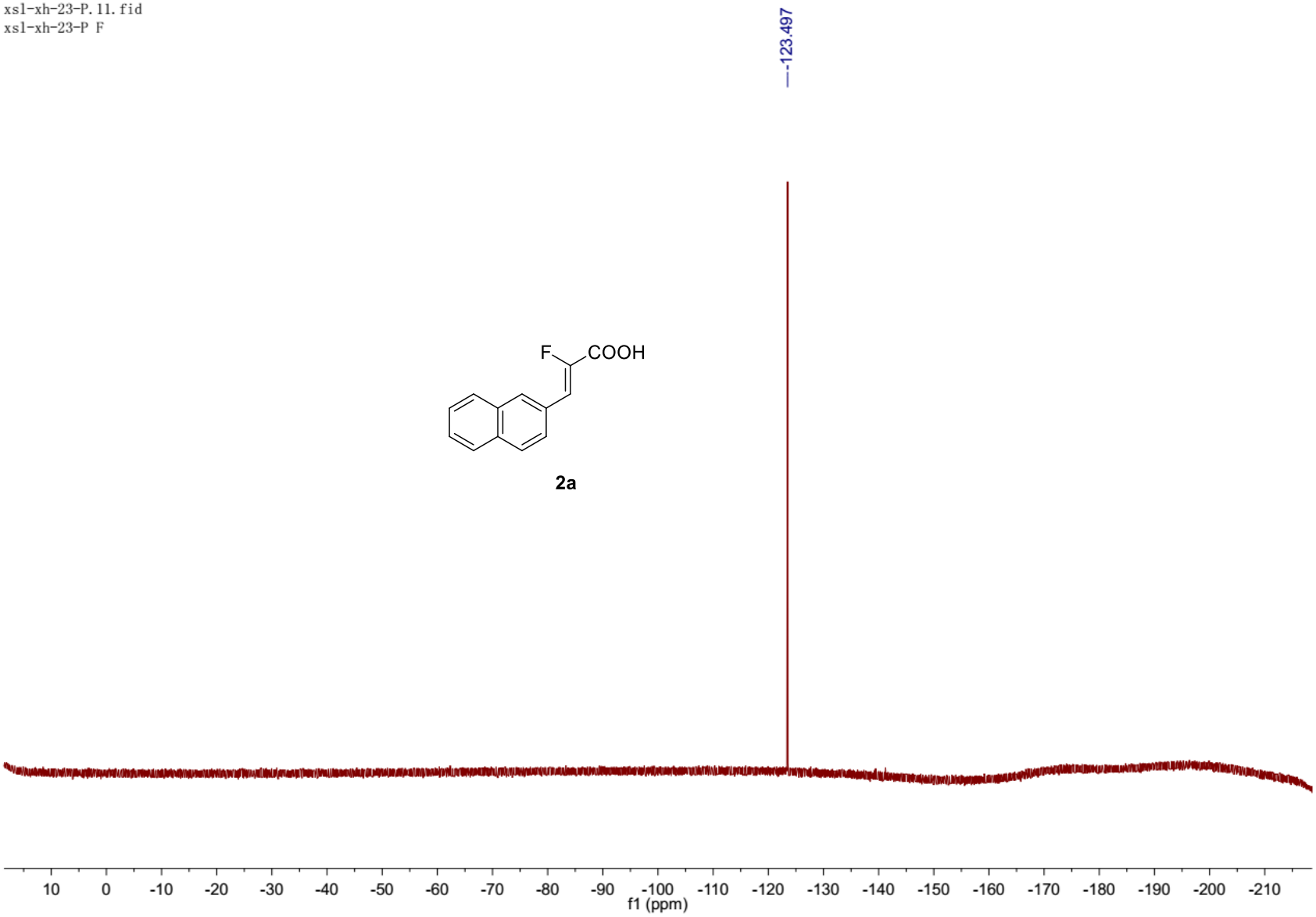
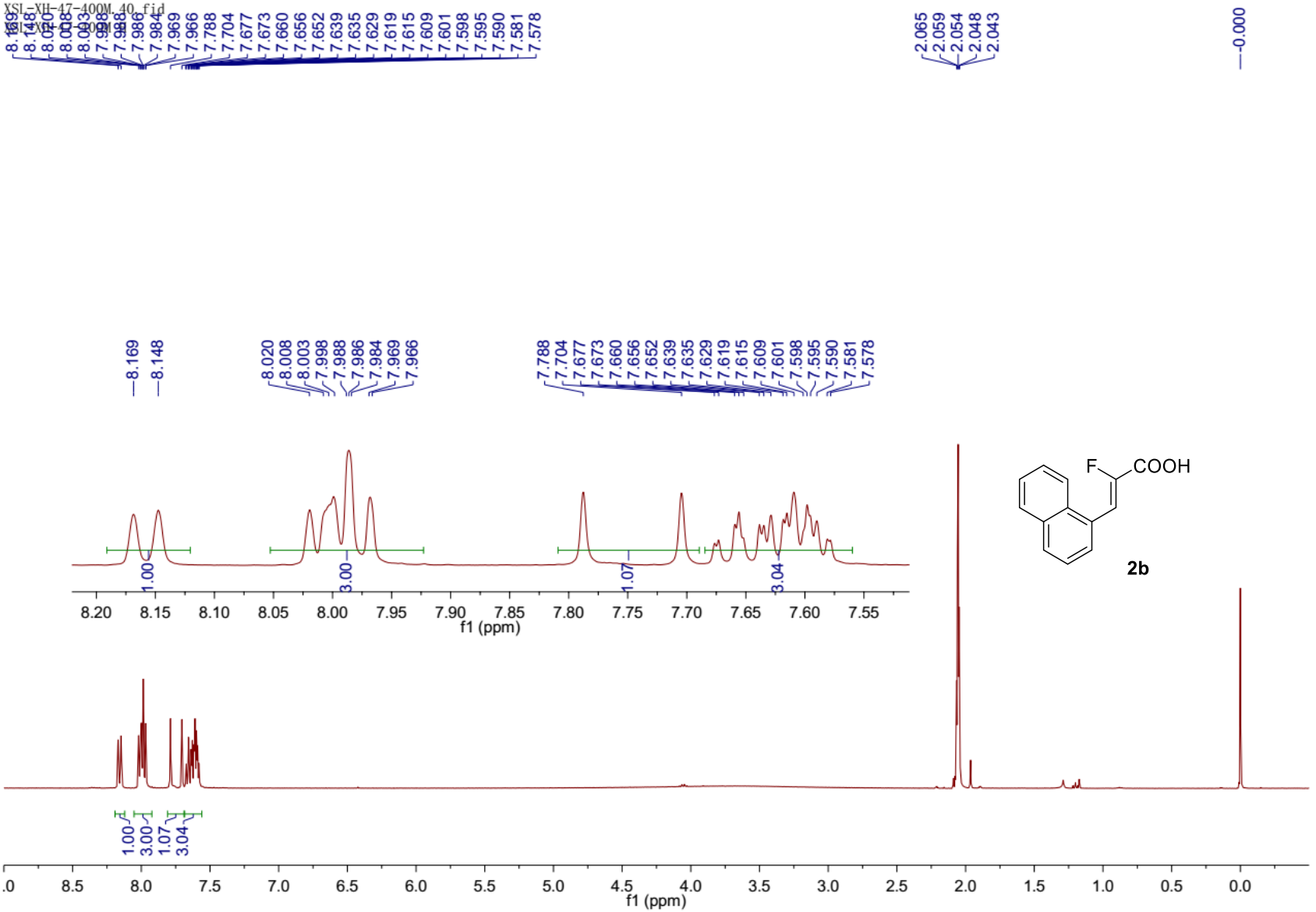


2a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7.609
7.601
7.598
7.595
7.590
7.581
7.578
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2.043
0.000
1.00
3.00
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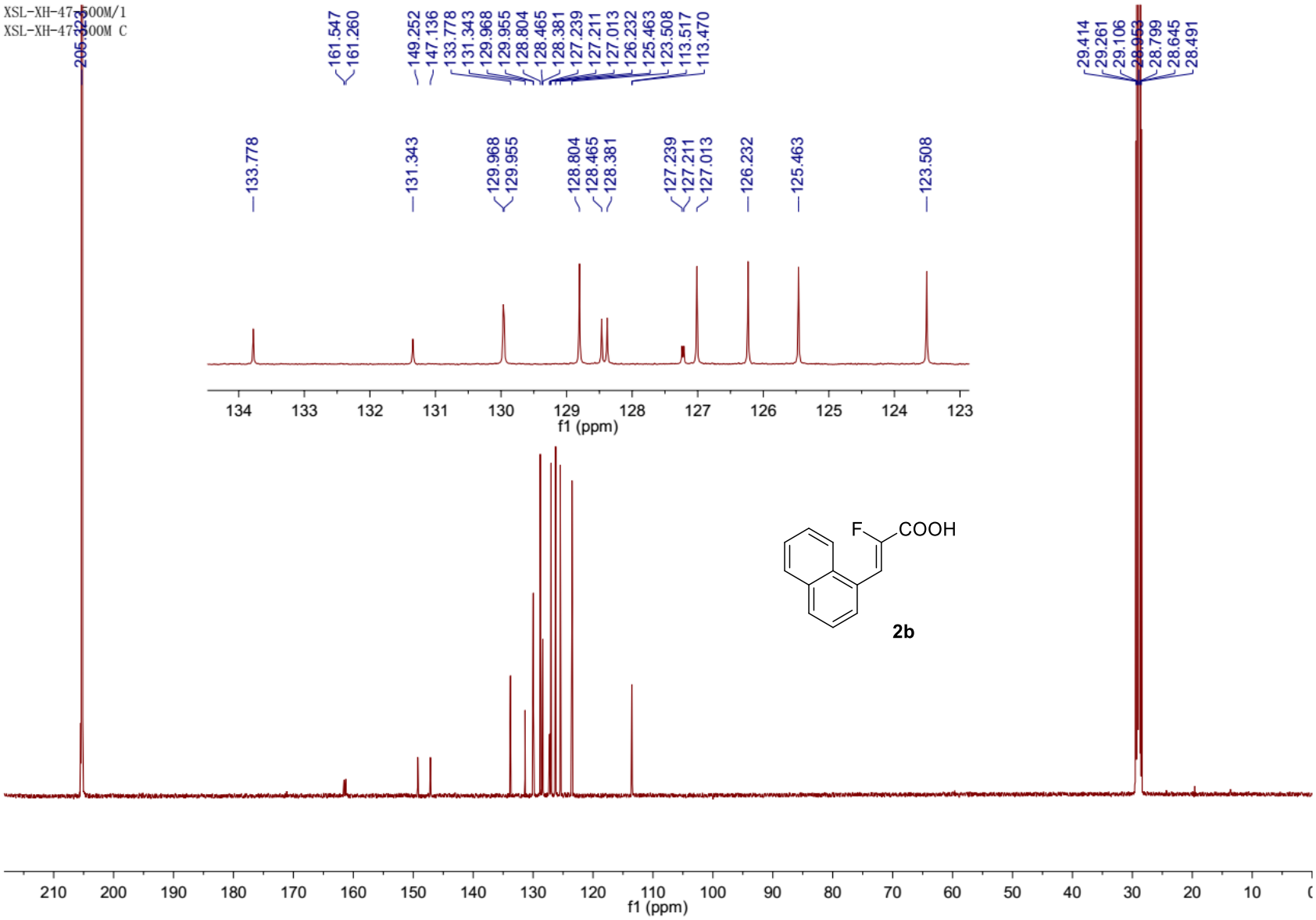
3.04
f1 (ppm)
F
COOH
2b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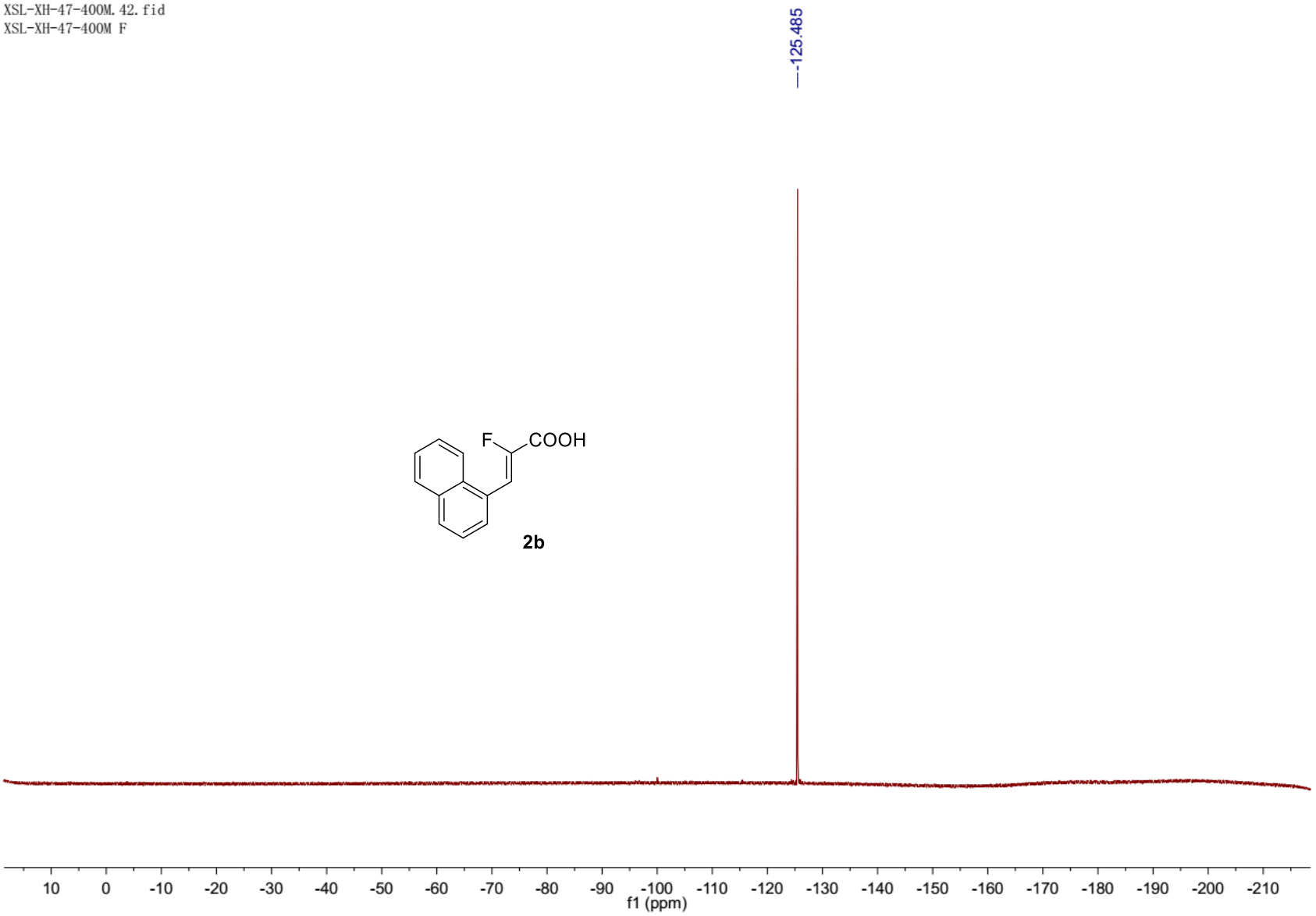
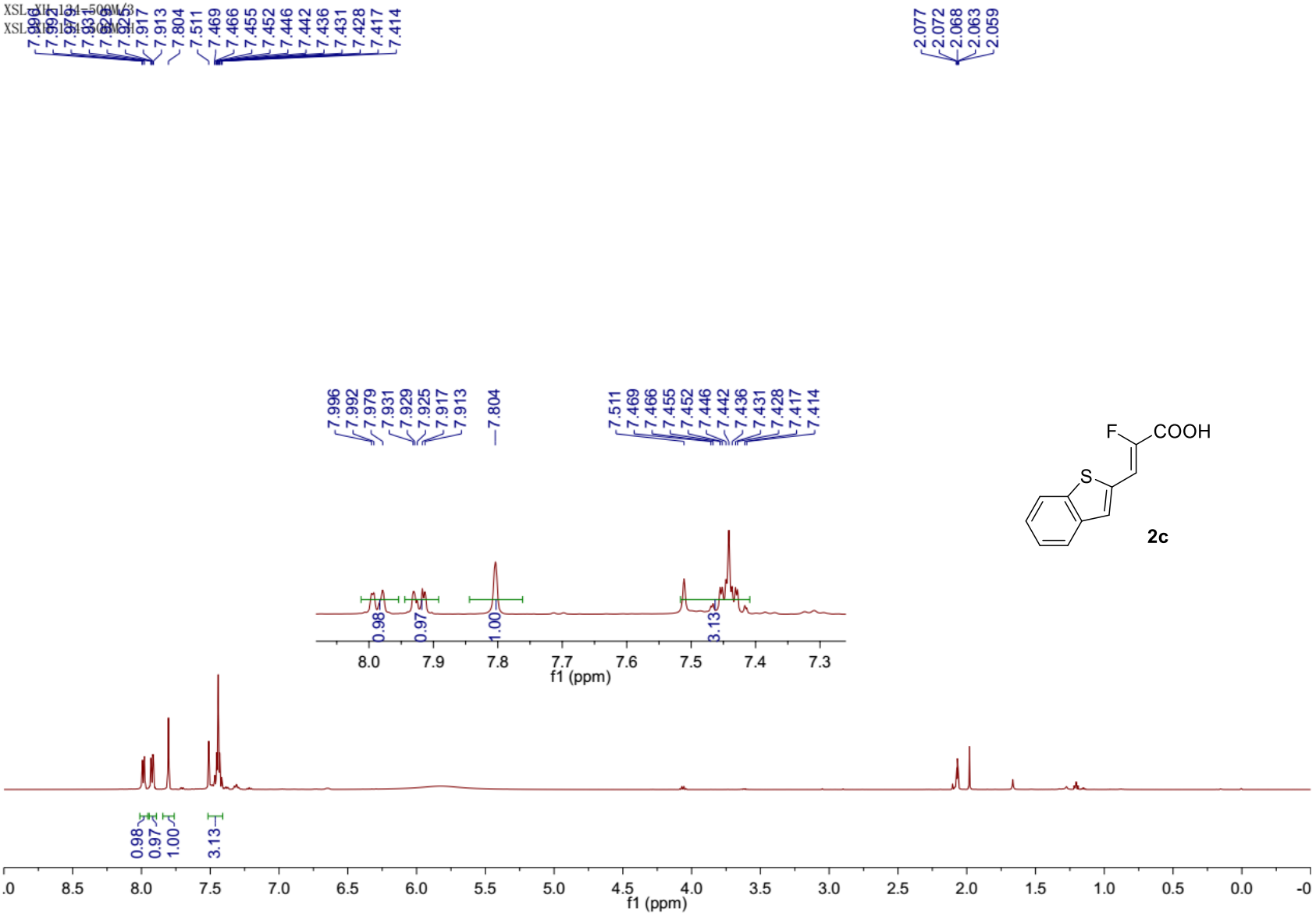
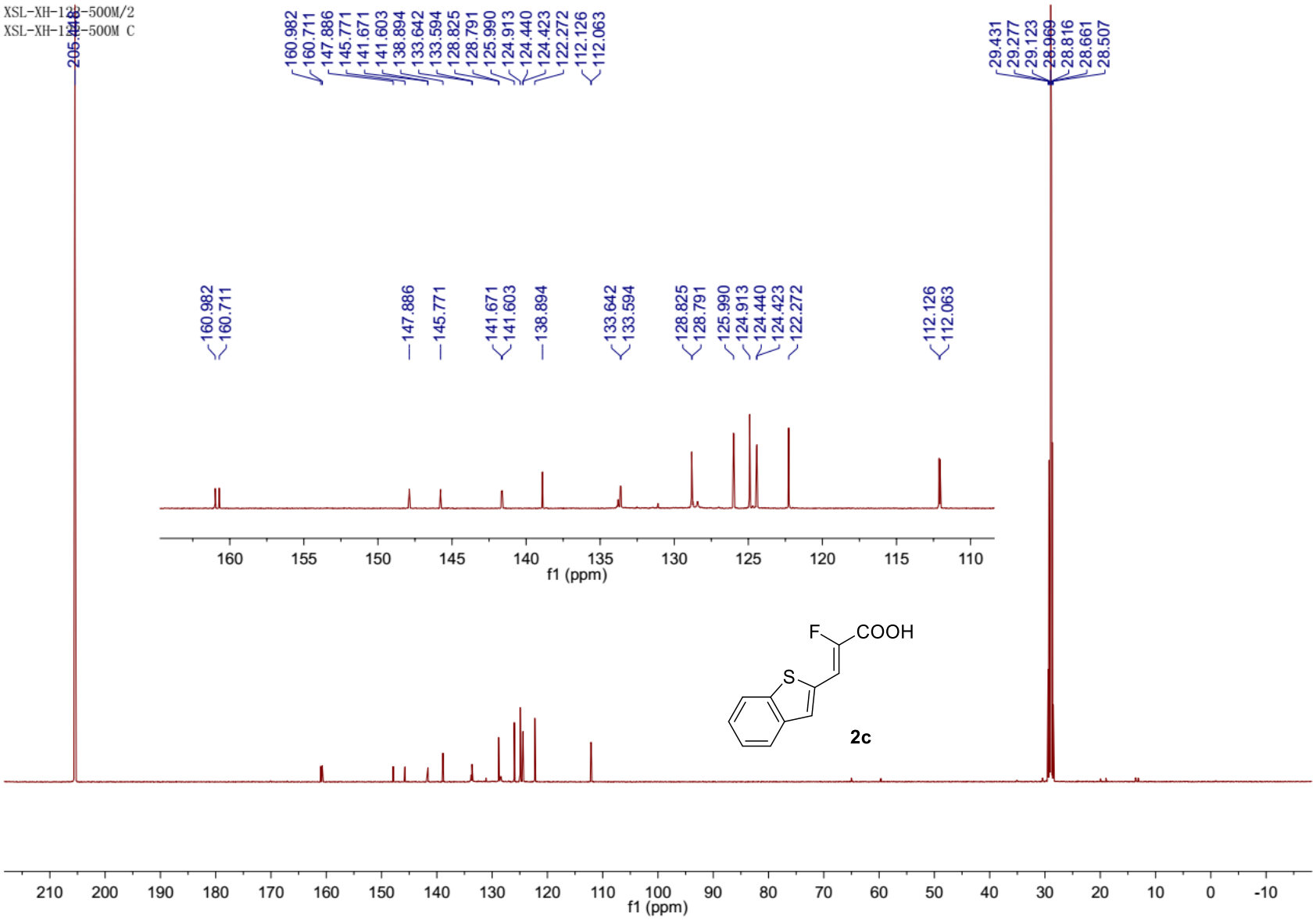
128.804
128.465
128.381
127.239
127.211
127.013
126.232
125.463
123.508
113.517
113.470
29.414
29.261
29.106
28.953
28.799
28.645
28.491
f1 (ppm)
F
COOH
2b

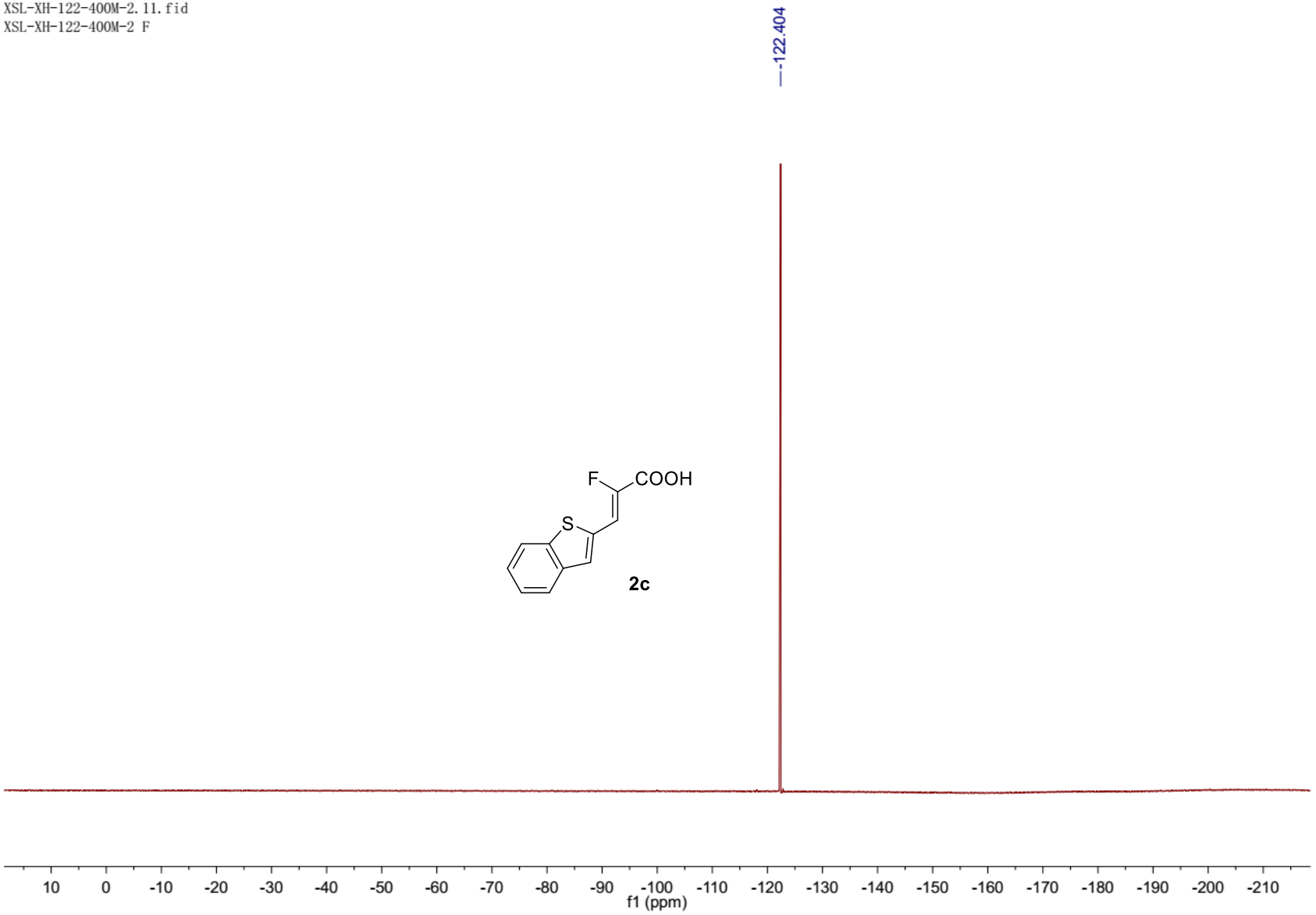
XSL-XH-47-400M.42.fid
XSL-XH-47-400M F

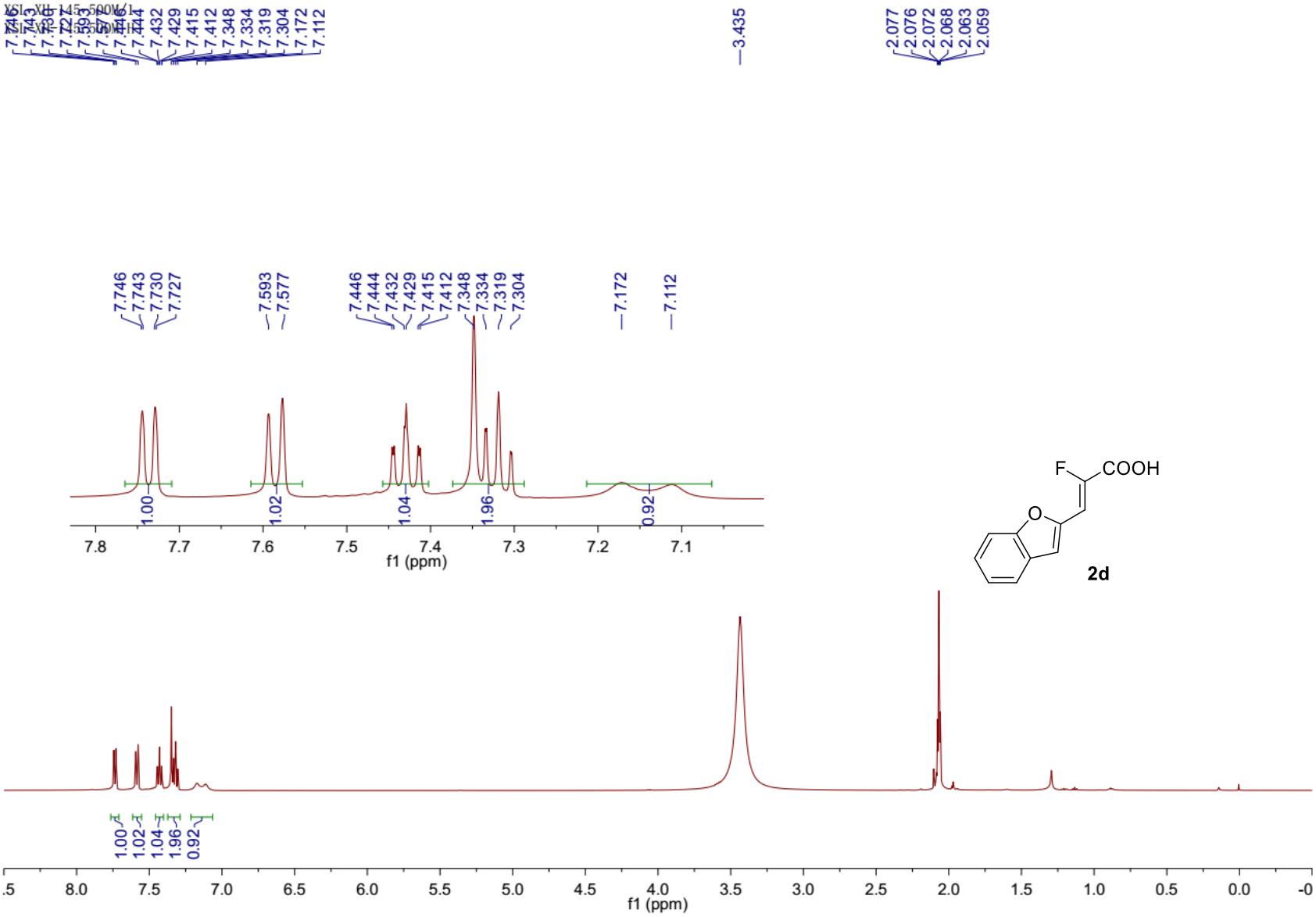


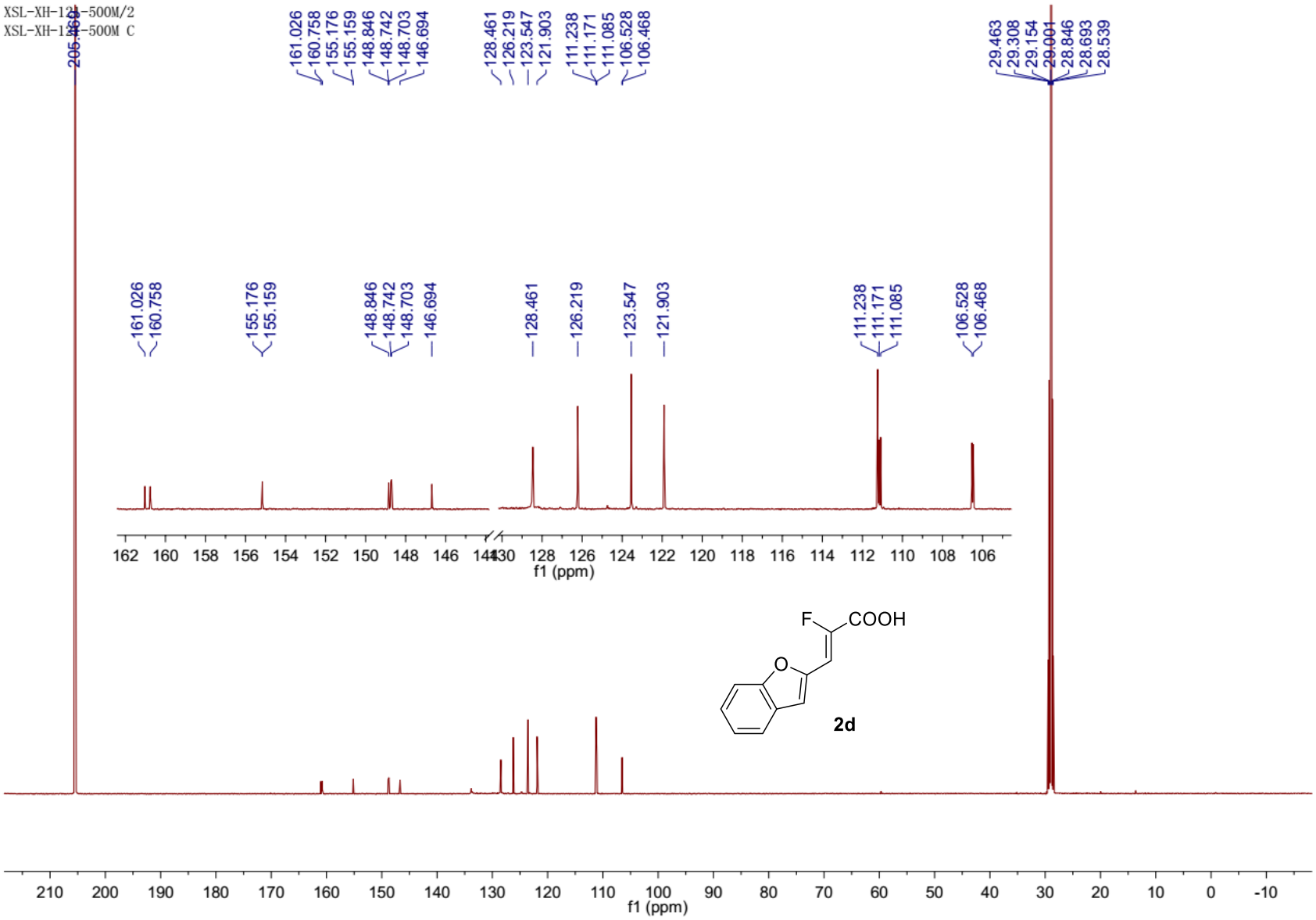

7.996
7.992
7.979
7.931
7.929
7.925
7.917
7.913
7.804
7.511
7.469
7.466
7.455
7.452
7.446
7.442
7.436
7.431
7.428
7.417
7.414
2.077
2.072
2.068
2.063
2.059
0.98
0.97
1.00
3.13
f1 (ppm)
F
COOH
S
2c


XSL-XH-123-500M/2
XSL-XH-123-500M C
205.418
160.982
160.711
147.886
145.771
141.671
141.603
138.894
133.642
133.594
128.825
128.791
125.990
124.913
124.440
124.423
122.272
112.126
112.063
29.431
29.277
29.123
28.969
28.816
28.661
28.507
f1 (ppm)
F
COOH
S
2c

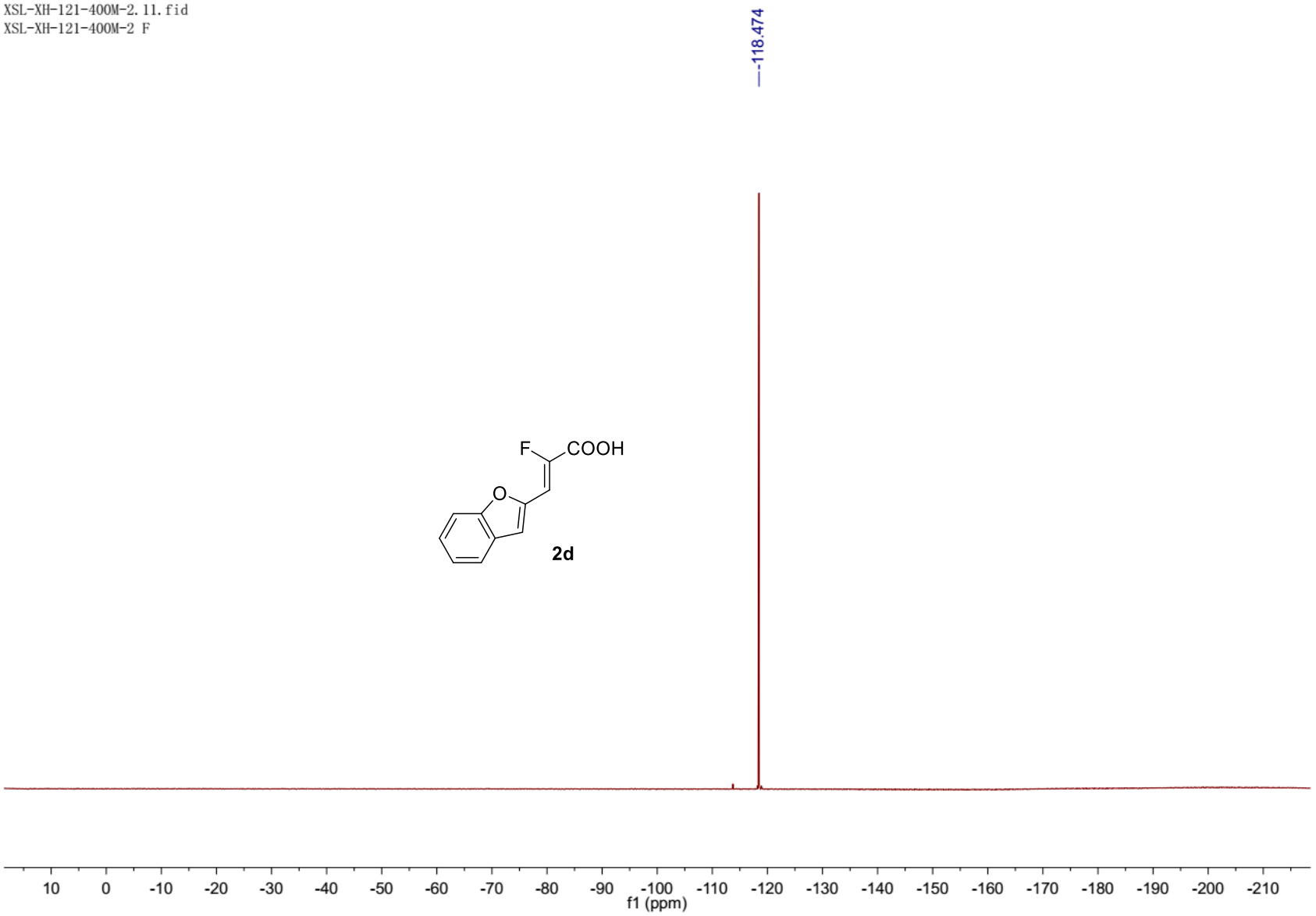
XSL-XH-122-400M-2.11.fid
XSL-XH-122-400M-2 F

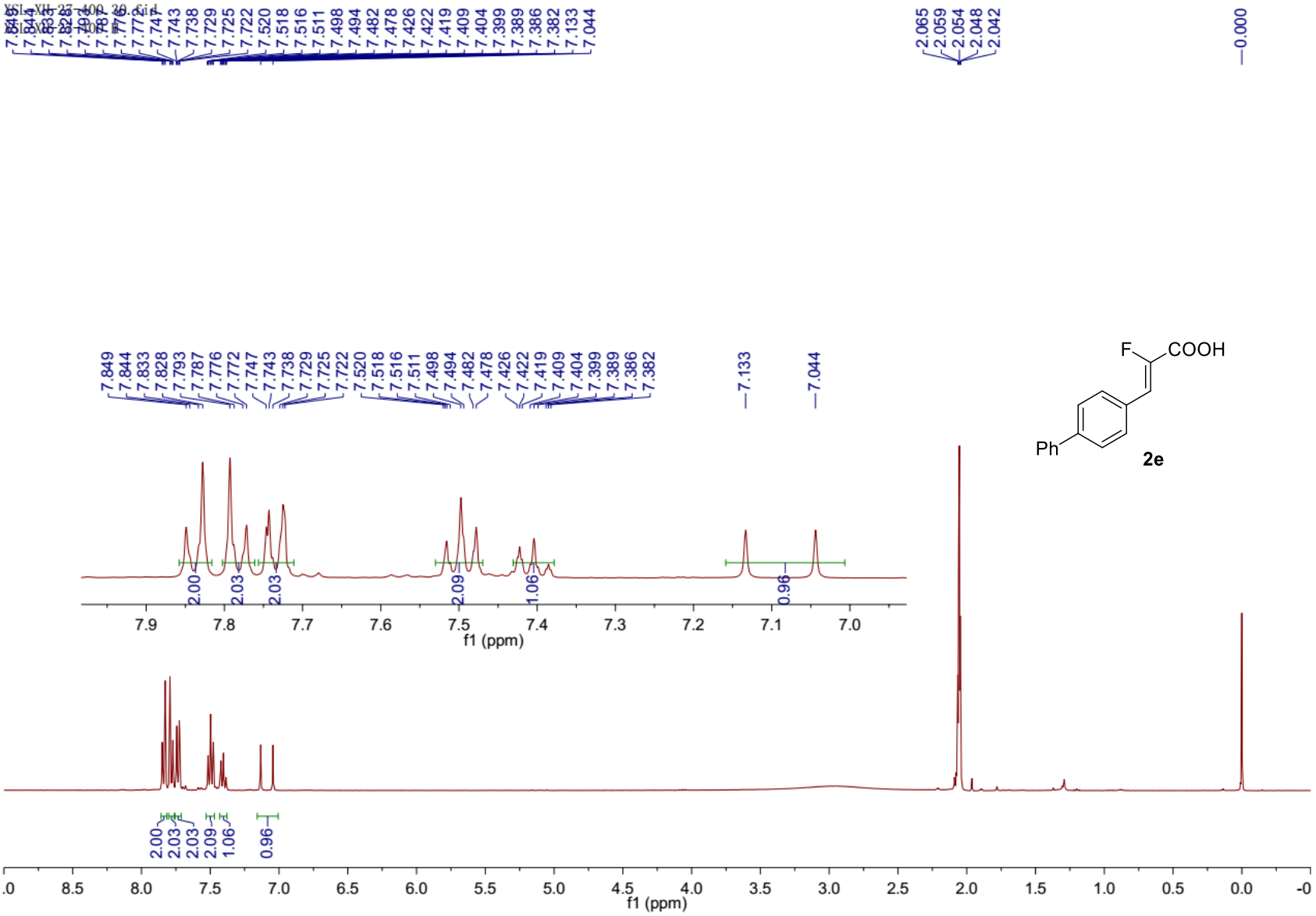


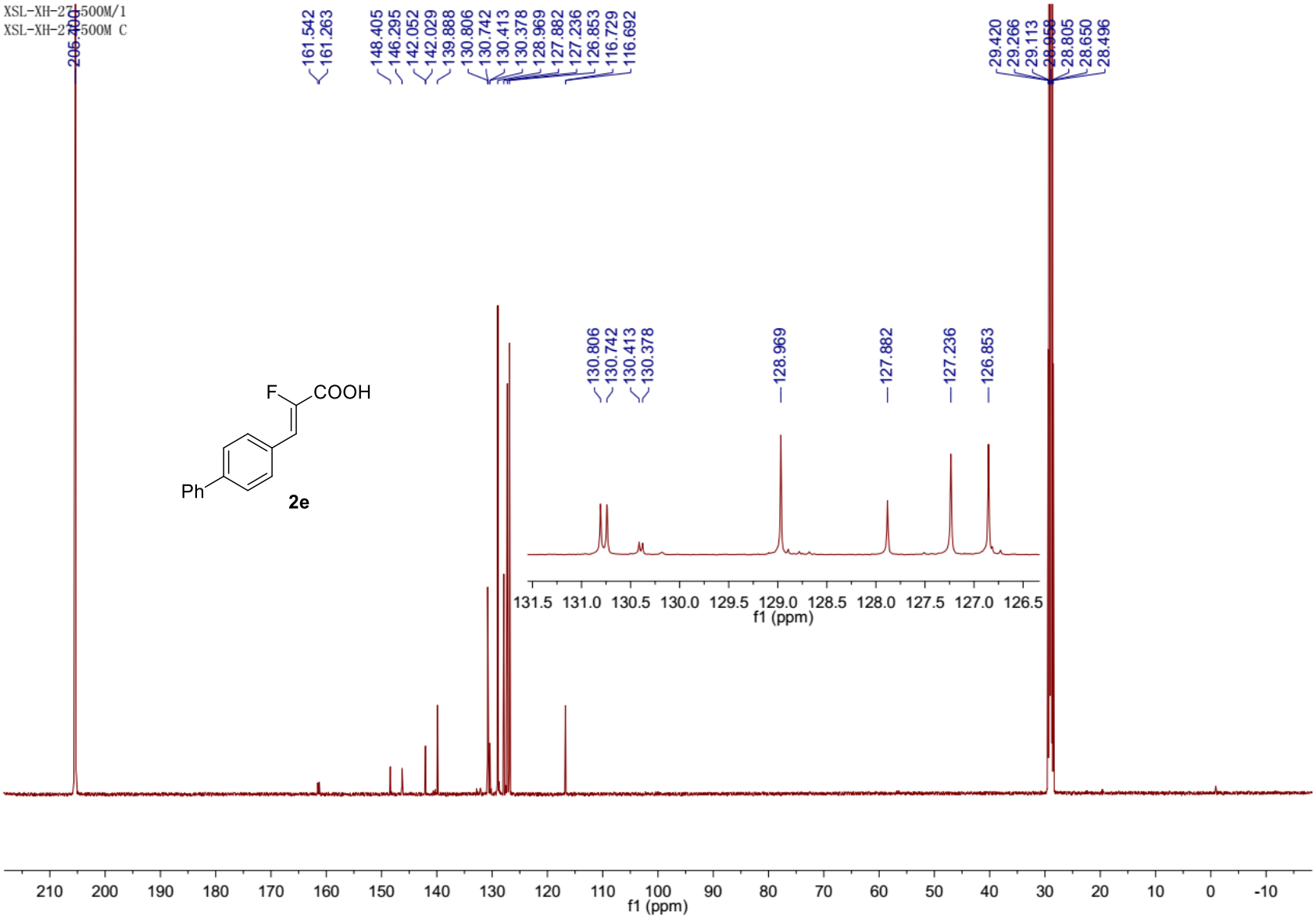

7.746
7.743
7.730
7.727
7.593
7.577
7.446
7.444
7.432
7.429
7.415
7.412
7.348
7.334
7.319
7.304
7.172
7.112
3.435
2.077
2.076
2.072
2.068
2.063
2.059
1.00
1.02
1.04
1.96
0.92
f1 (ppm)
F
COOH
O
2d


XSL-XH-12d-500M/2
XSL-XH-12d-500M C
205.468
161.026
160.758
155.176
155.159
148.846
148.742
148.703
146.694
128.461
126.219
123.547
121.903
111.238
111.171
111.085
106.528
106.468
29.463
29.308
29.154
29.001
28.846
28.693
28.539
f1 (ppm)
F
COOH
O
2d

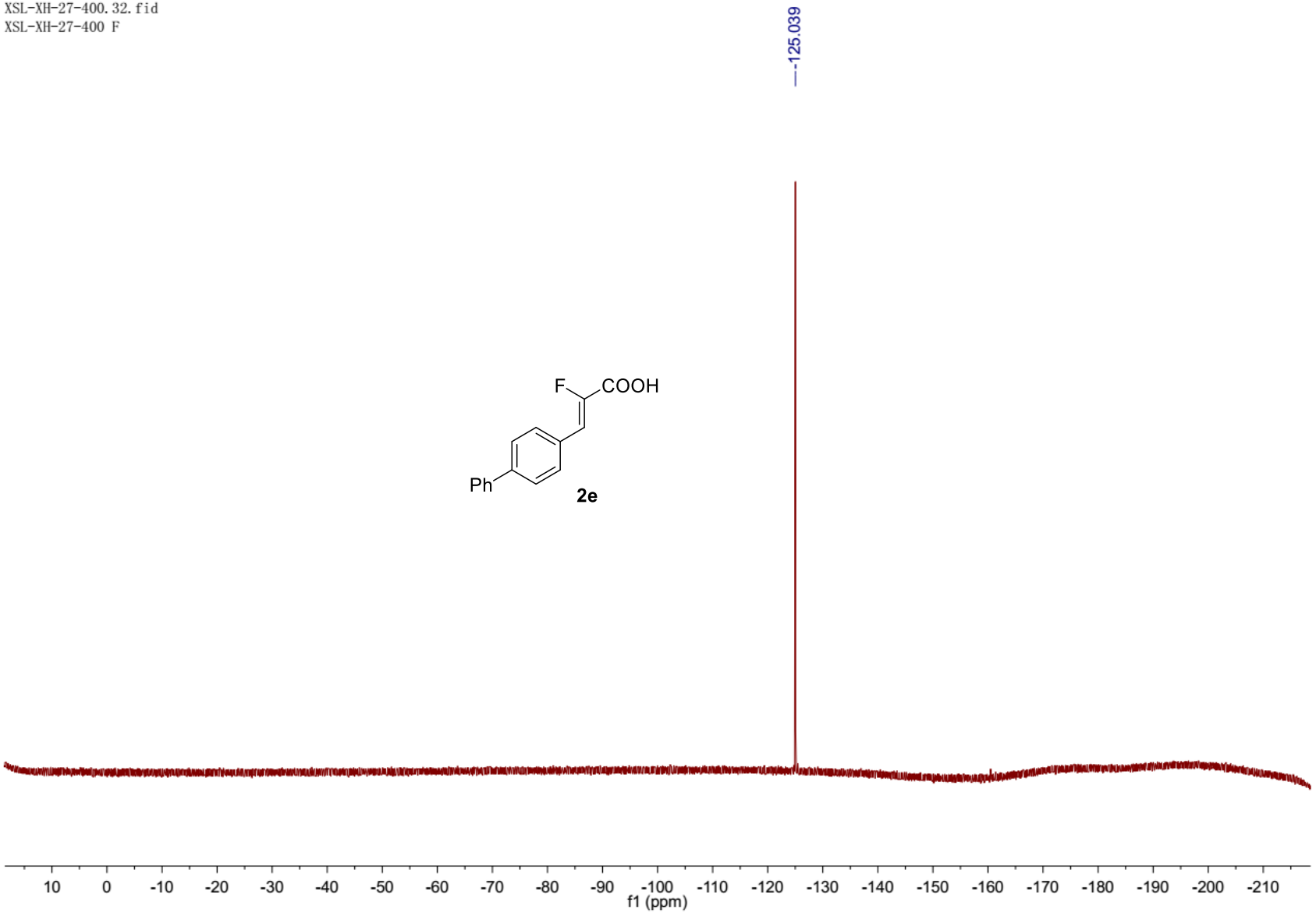
XSL-XH-121-400M-2.11.fid
XSL-XH-121-400M-2 F

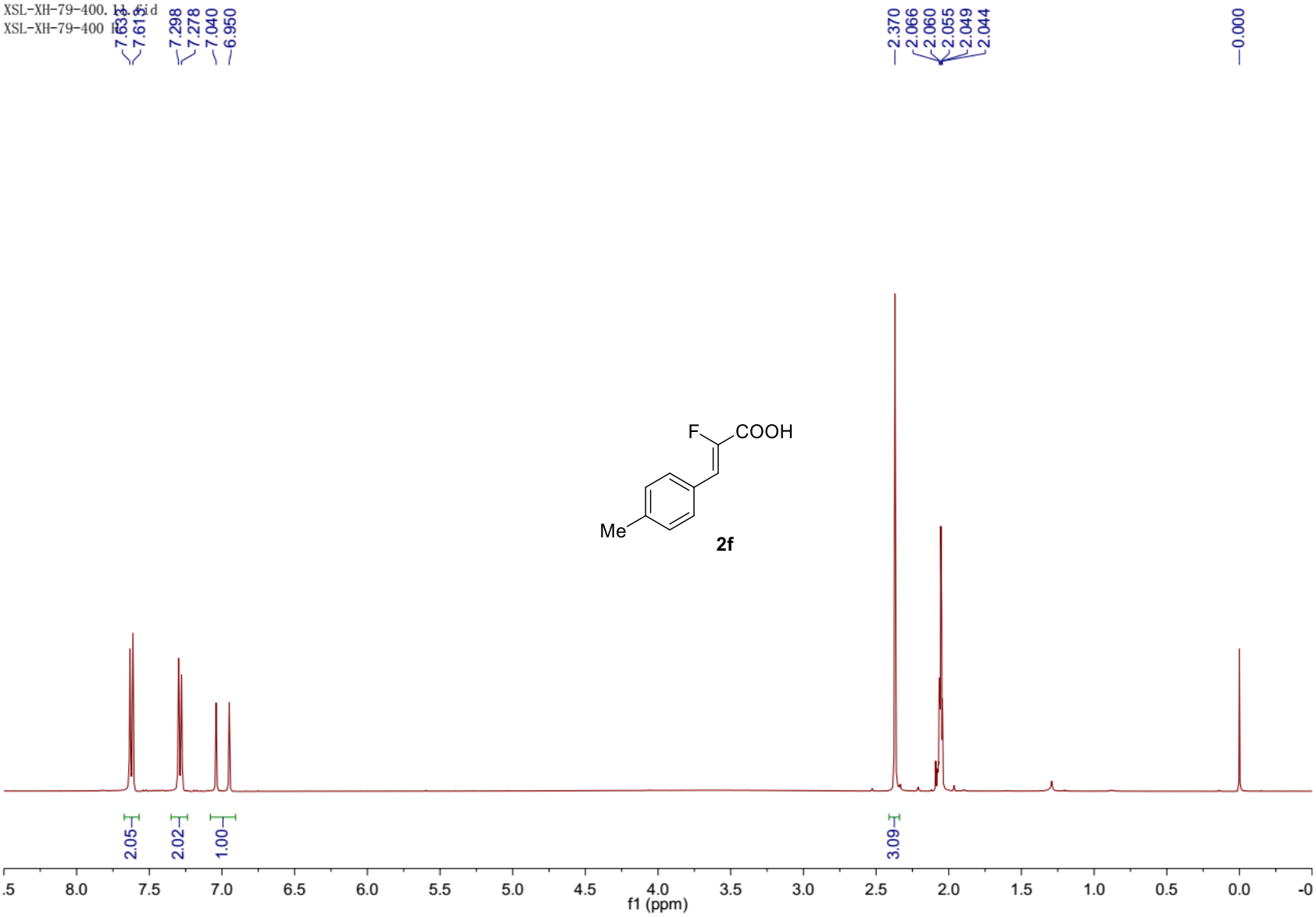


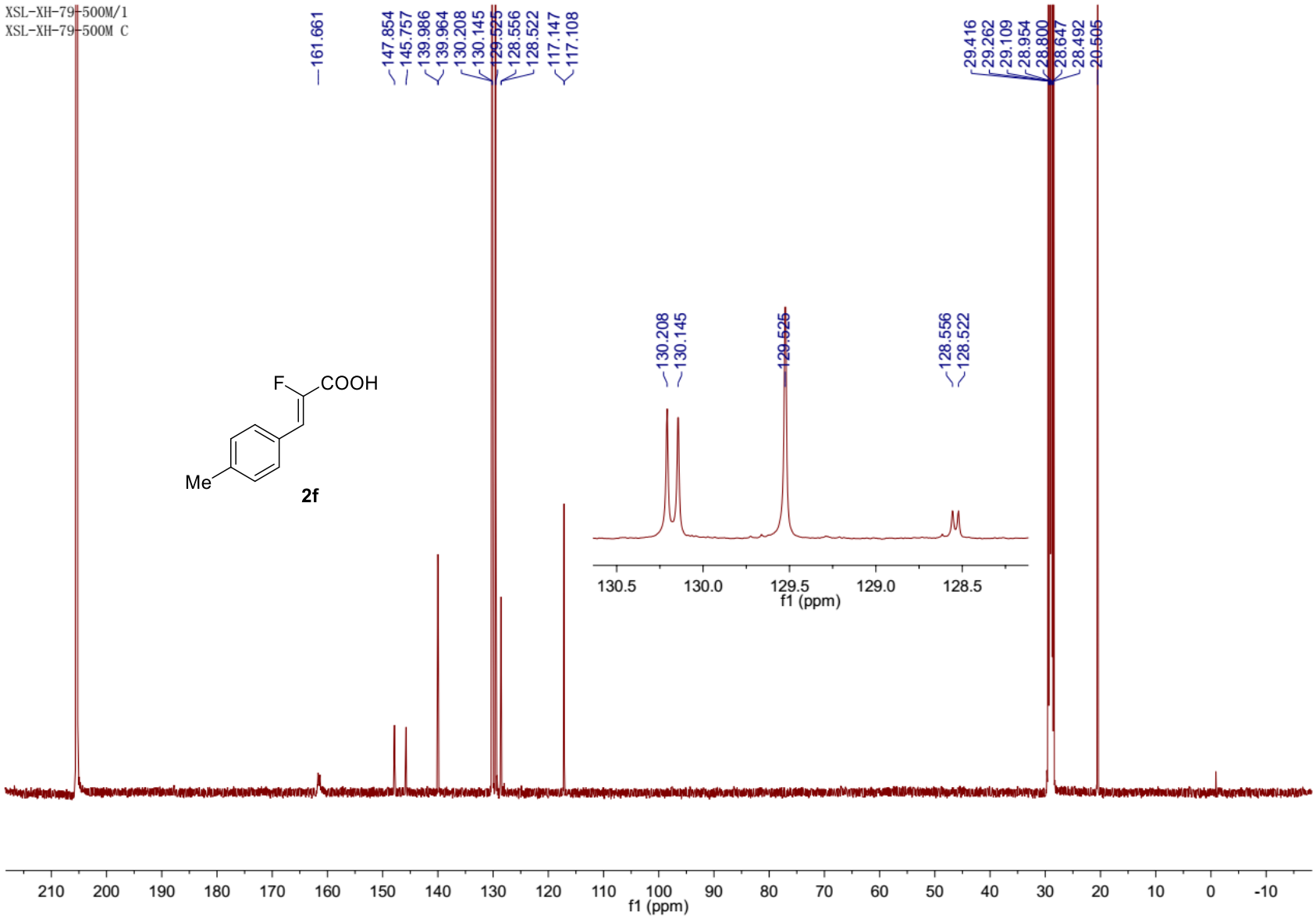

7.849
7.844
7.833
7.828
7.793
7.787
7.776
7.772
7.747
7.743
7.738
7.729
7.725
7.722
7.520
7.518
7.516
7.511
7.498
7.494
7.482
7.478
7.426
7.422
7.419
7.409
7.404
7.399
7.389
7.386
7.382
7.133
7.044
2.00
2.03
2.03
2.09
1.06
0.96
f1 (ppm)
F
COOH
Ph
2e


XSL-XH-27-500M/1
XSL-XH-27-500M C
161.542
161.263
148.405
146.295
142.052
142.029
139.888
130.806
130.742
130.413
130.378
128.969
127.882
127.236
126.853
116.729
116.692
29.420
29.266
29.113
28.805
28.650
28.496
F
COOH
Ph
2e
130.806
130.742
130.413
130.378
128.969
127.882
127.236
126.853
131.5 131.0 130.5 130.0 129.5 129.0 128.5 128.0 127.5 127.0 126.5
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

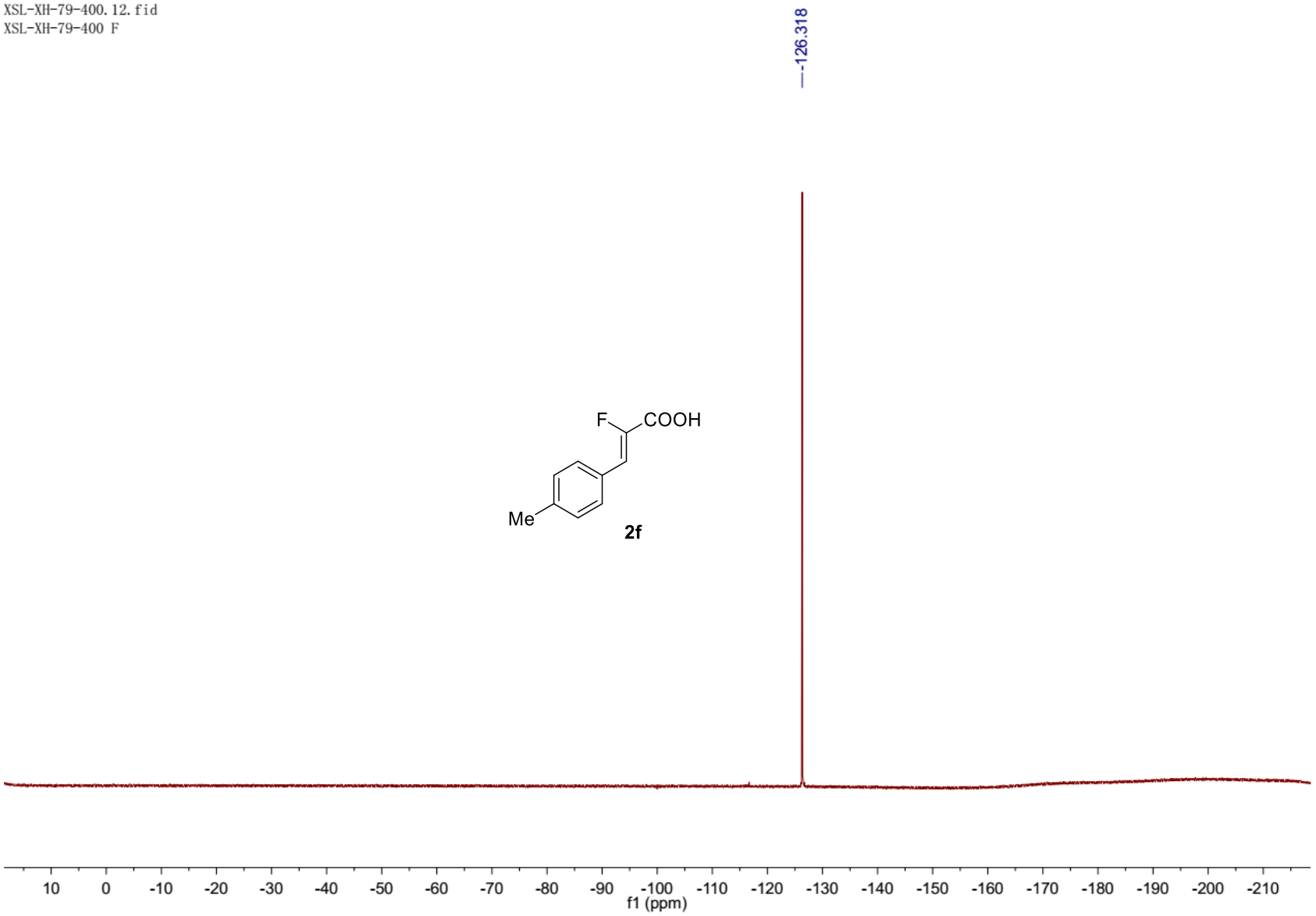
XSL-XH-27-400.32.fid
XSL-XH-27-400 F

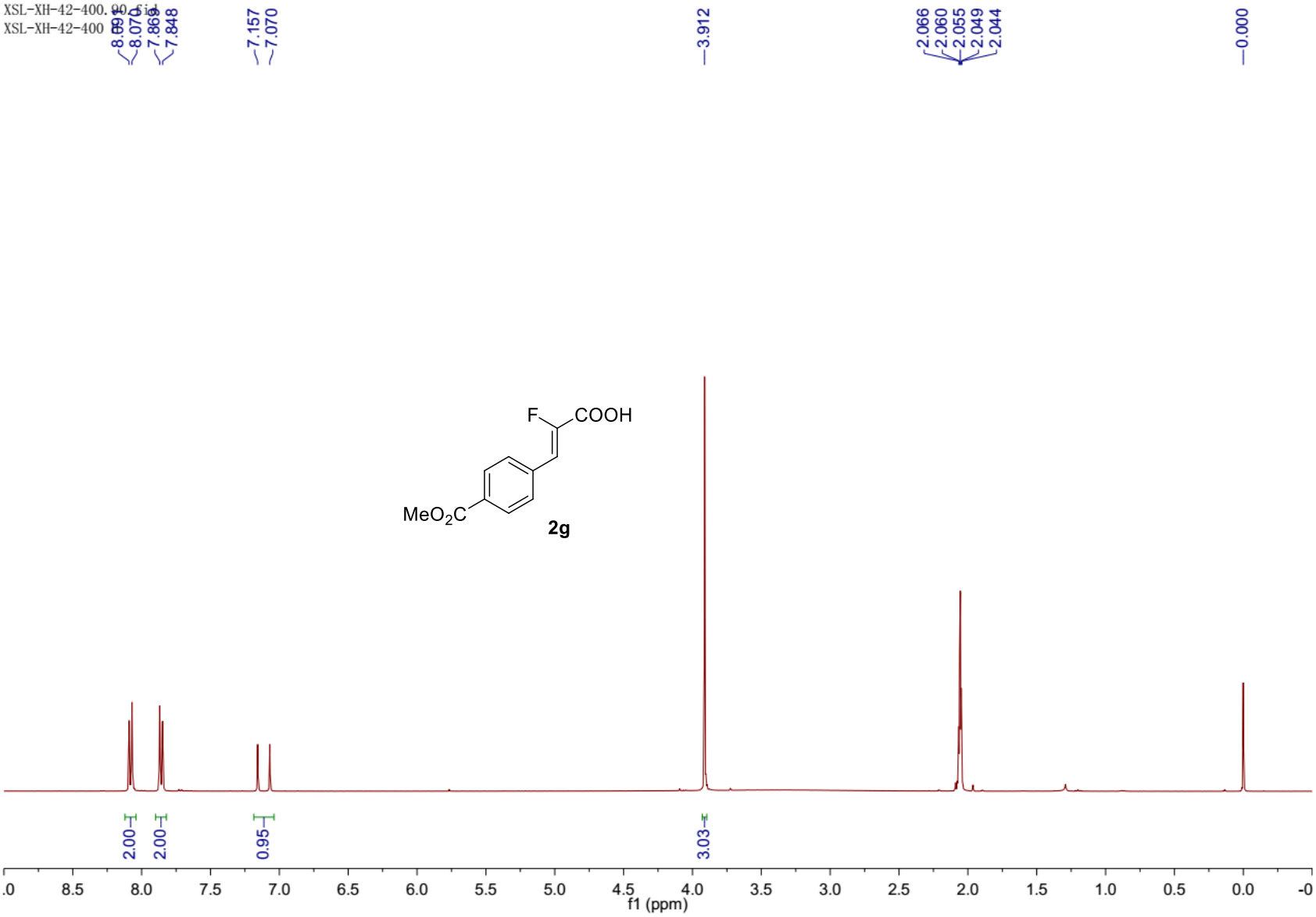


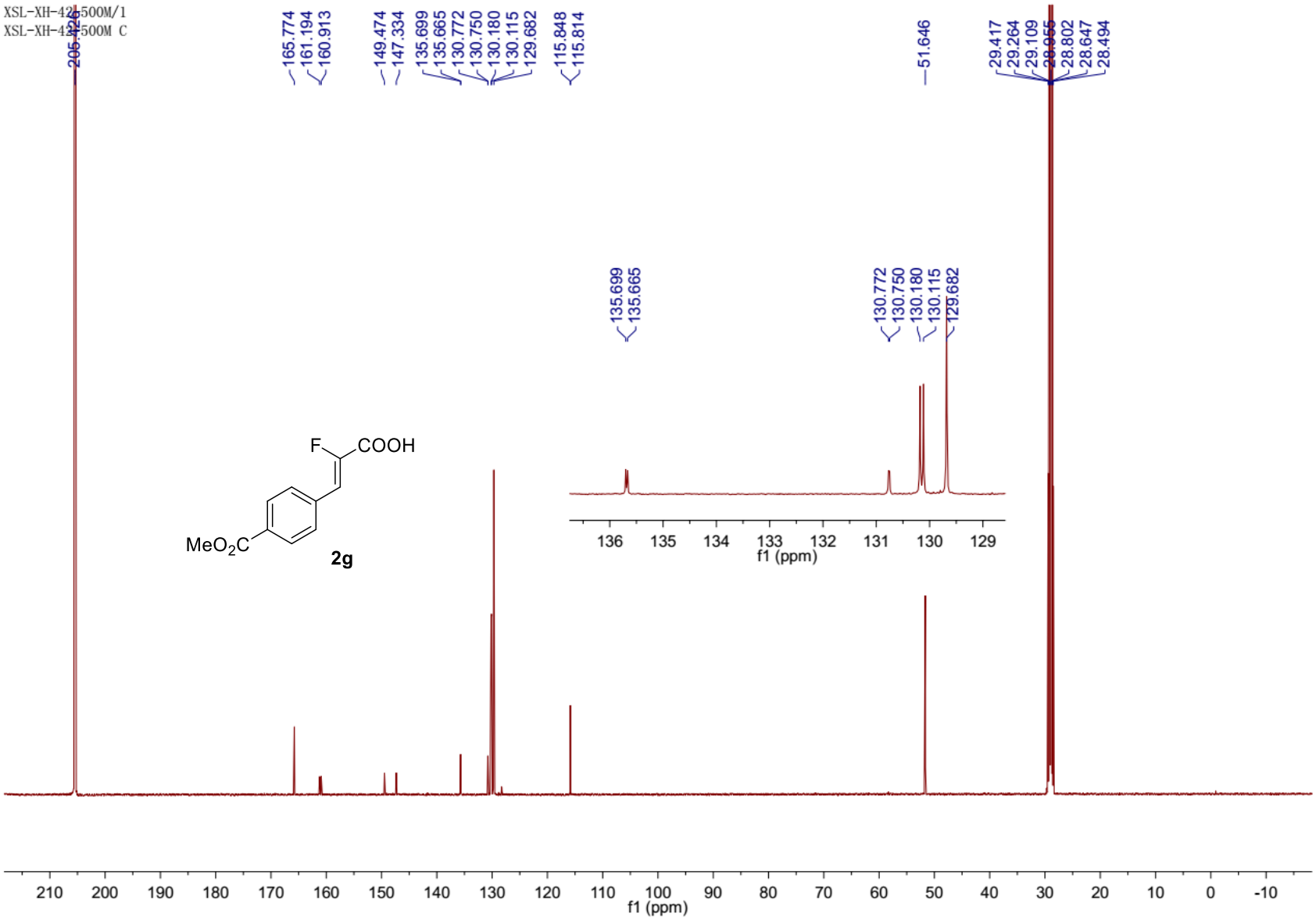

XSL-XH-79-400.11.fid
XSL-XH-79-400 H1
7.639
7.613
7.298
7.278
7.040
6.950
2.370
2.066
2.060
2.055
2.049
2.044
0.000
F
COOH
Me
2f
2.05
2.02
1.00
3.09
f1 (ppm)


XSL-XH-79-500M/1
XSL-XH-79-500M C
161.661
147.854
145.757
139.986
139.964
130.208
130.145
129.525
128.556
128.522
117.147
117.108
29.416
29.262
29.109
28.954
28.800
28.647
28.492
20.606
F
COOH
Me
2f
130.208
130.145
129.525
128.556
128.522
130.5
130.0
129.5
129.0
128.5
f1 (ppm)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

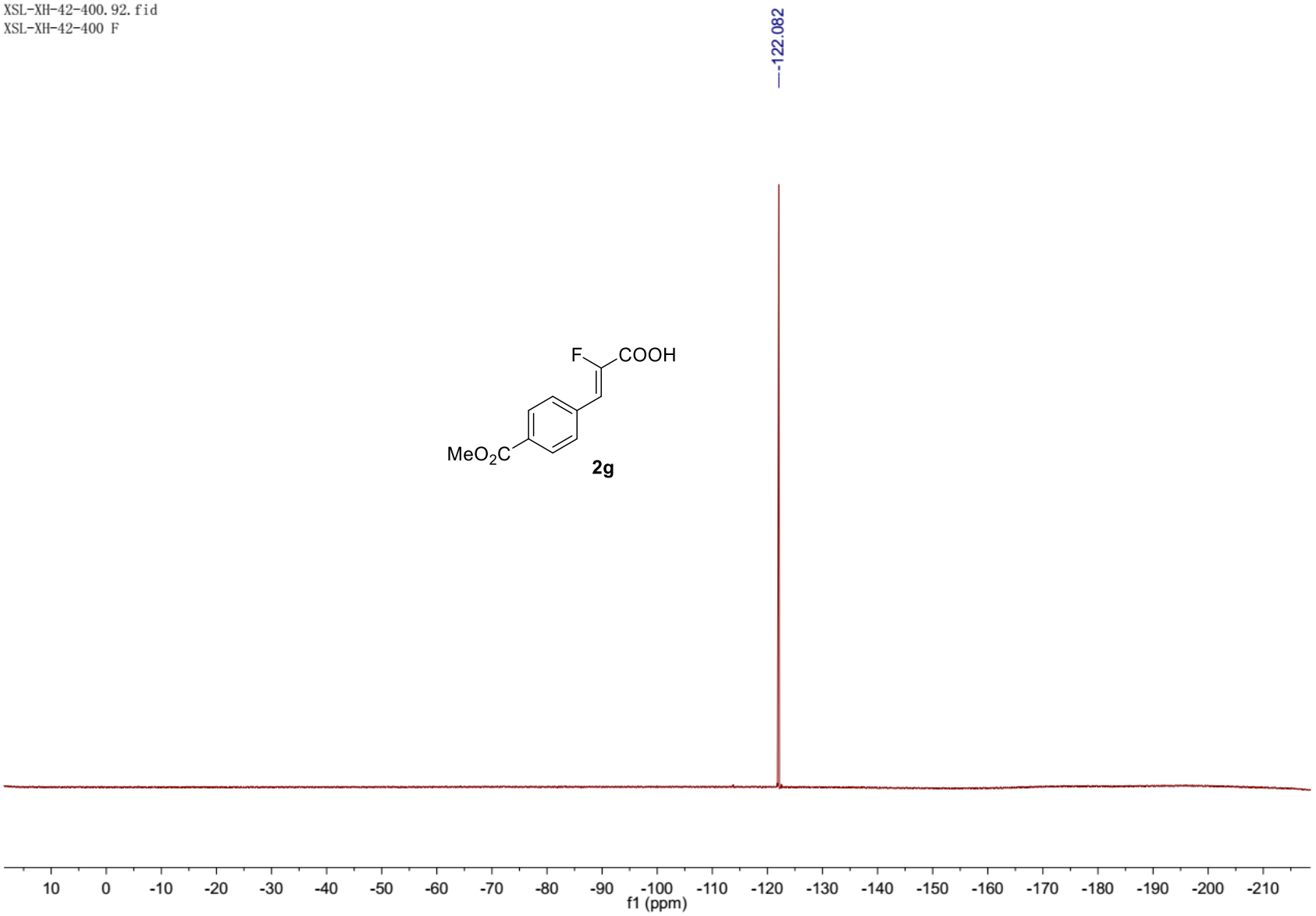
XSL-XH-79-400.12.fid
XSL-XH-79-400 F

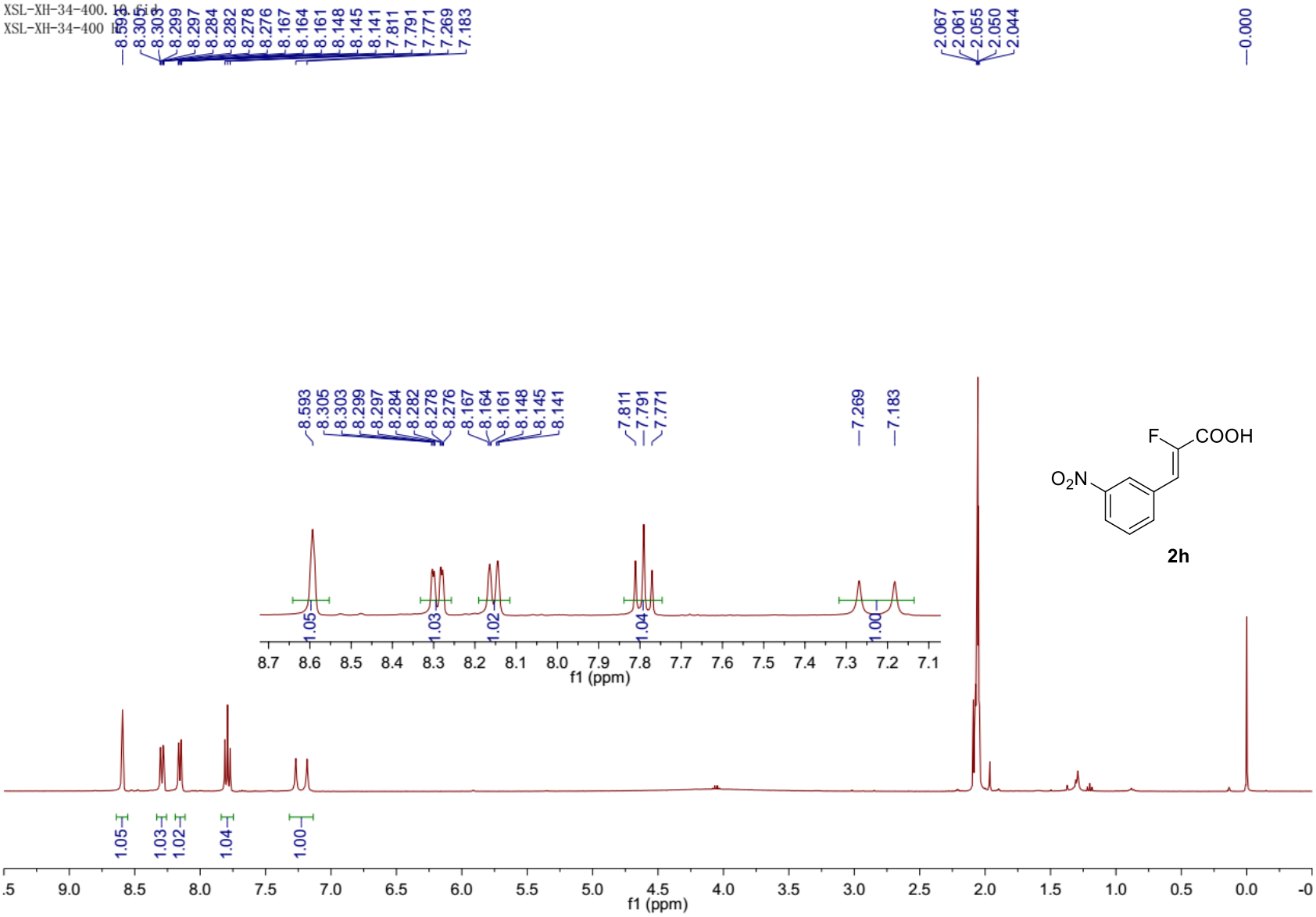


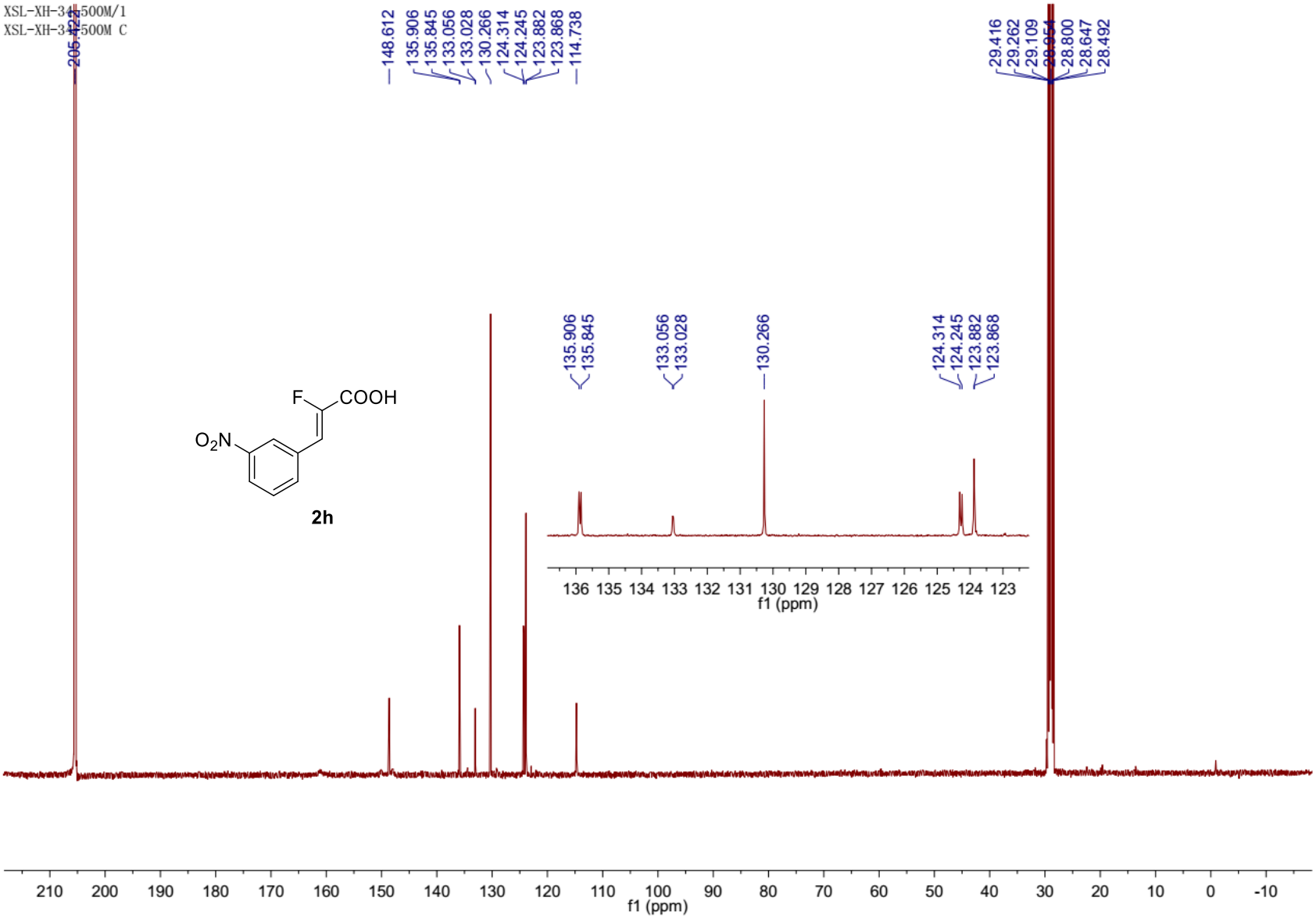

XSL-XH-42-400.90.fid
XSL-XH-42-400
8.091
8.070
7.869
7.848
7.157
7.070
3.912
2.066
2.060
2.055
2.049
2.044
0.000
F
COOH
MeO2C
2g
2.00
2.00
0.95
3.03
f1 (ppm)


XSL-XH-426-500M/1
XSL-XH-426-500M C
205.426
165.774
161.194
160.913
149.474
147.334
135.699
135.665
130.772
130.750
130.180
130.115
129.682
115.848
115.814
51.646
29.417
29.264
29.109
28.955
28.802
28.647
28.494
F
COOH
MeO2C
2g
f1 (ppm)
f1 (ppm)

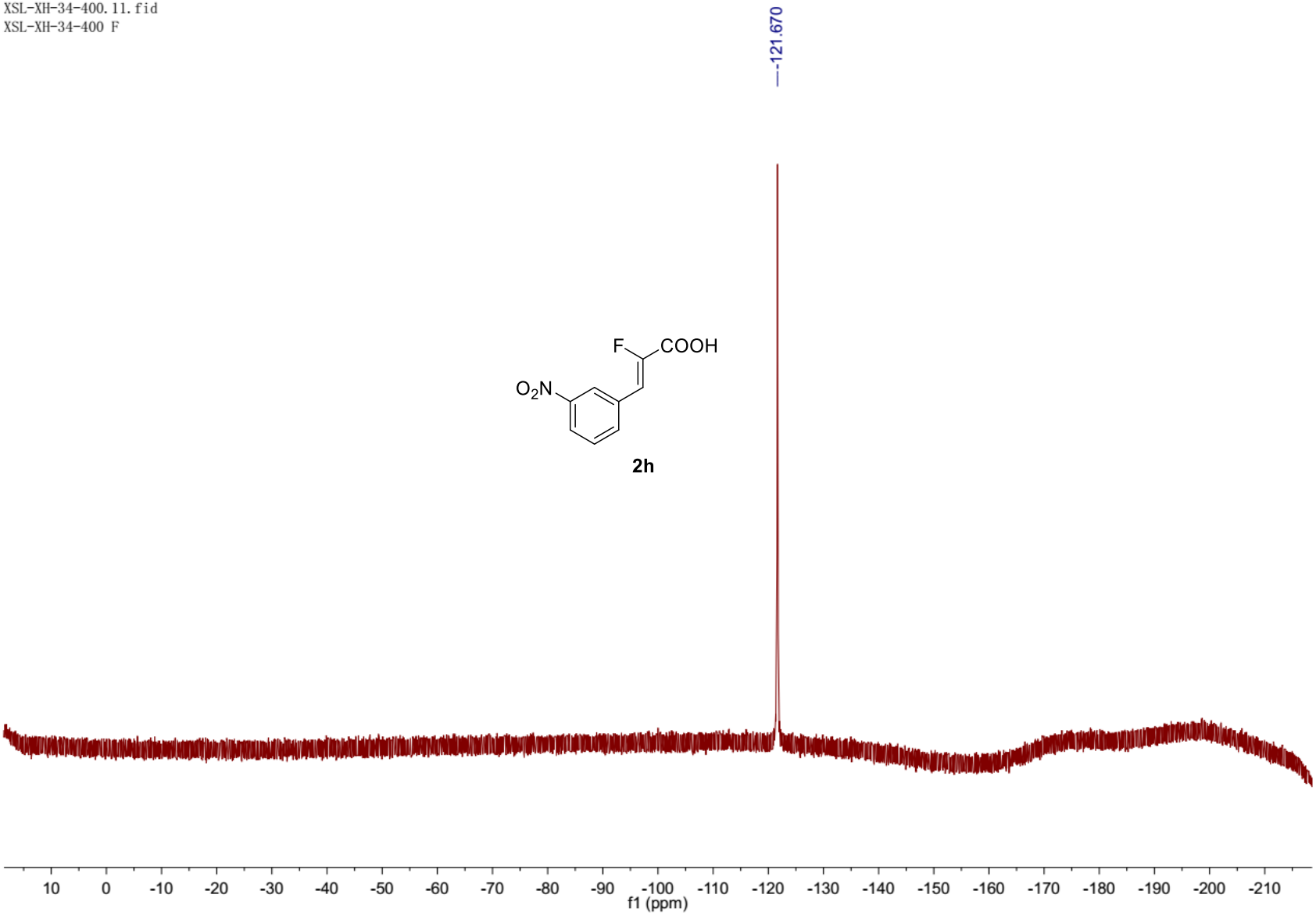
XSL-XH-42-400.92.fid
XSL-XH-42-400 F



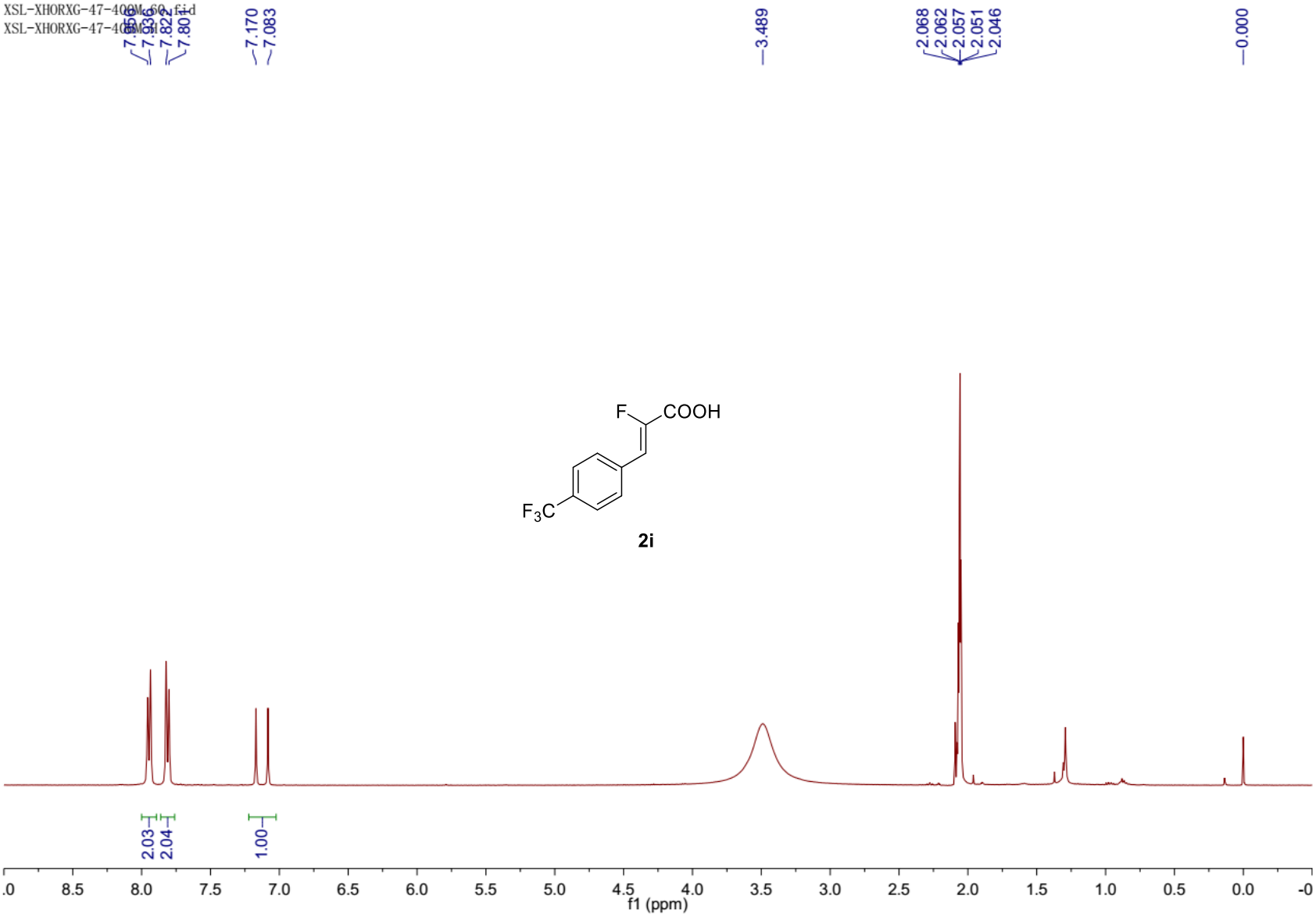

XSL-XH-34-400.10.fid
XSL-XH-34-400 F
8.593
8.305
8.303
8.299
8.297
8.284
8.282
8.278
8.276
8.167
8.164
8.161
8.148
8.145
8.141
7.811
7.791
7.771
7.269
7.183
2.067
2.061
2.055
2.050
2.044
0.000
F
COOH
O2N
2h
1.05
1.03
1.02
1.04
1.00
f1 (ppm)

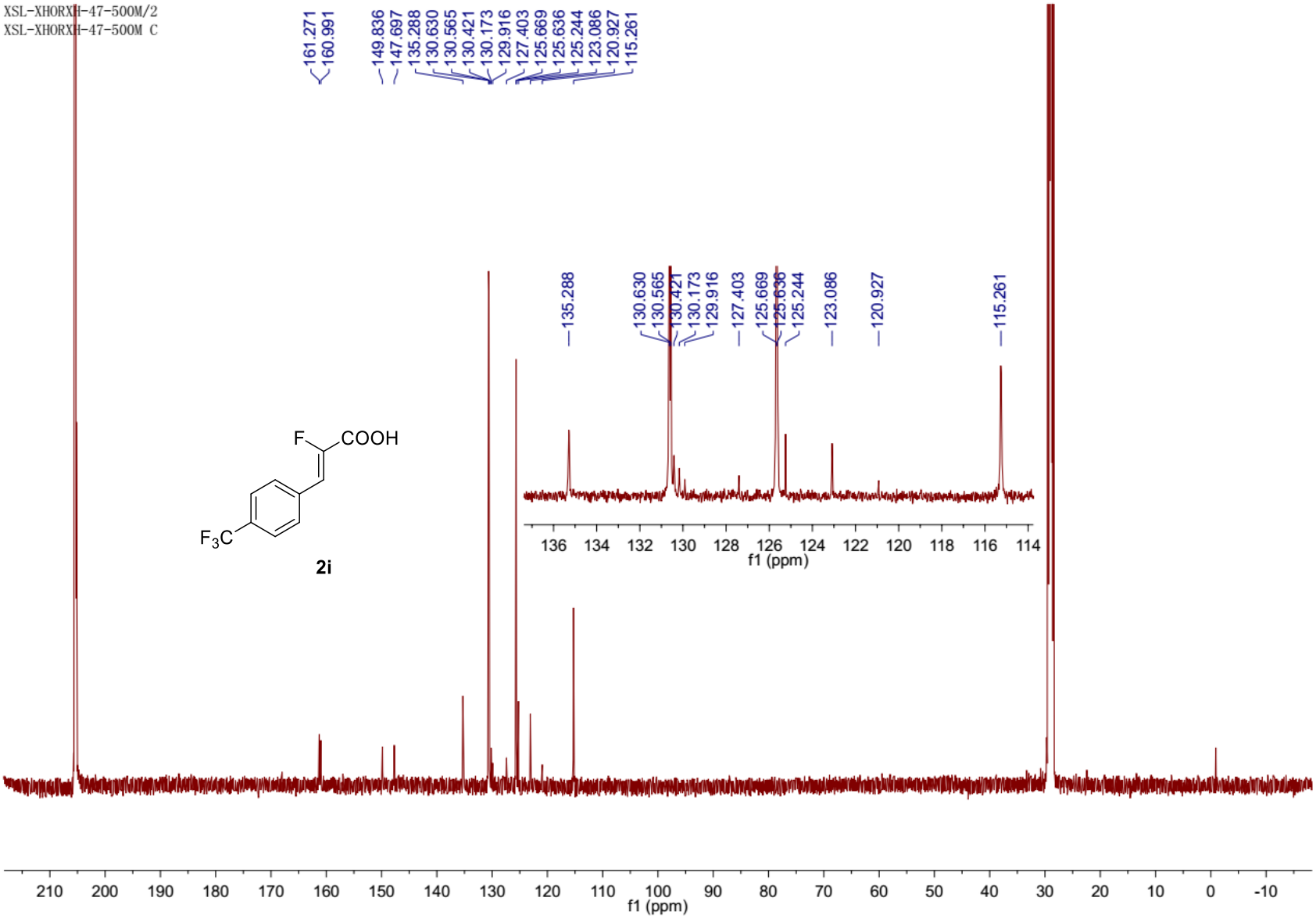

XSL-XH-34-500M/1
XSL-XH-34-500M C
148.612
135.906
135.845
133.056
133.028
130.266
124.314
124.245
123.882
123.868
114.738
29.416
29.262
29.109
28.800
28.647
28.492
F
COOH
O2N
2h
f1 (ppm)
f1 (ppm)

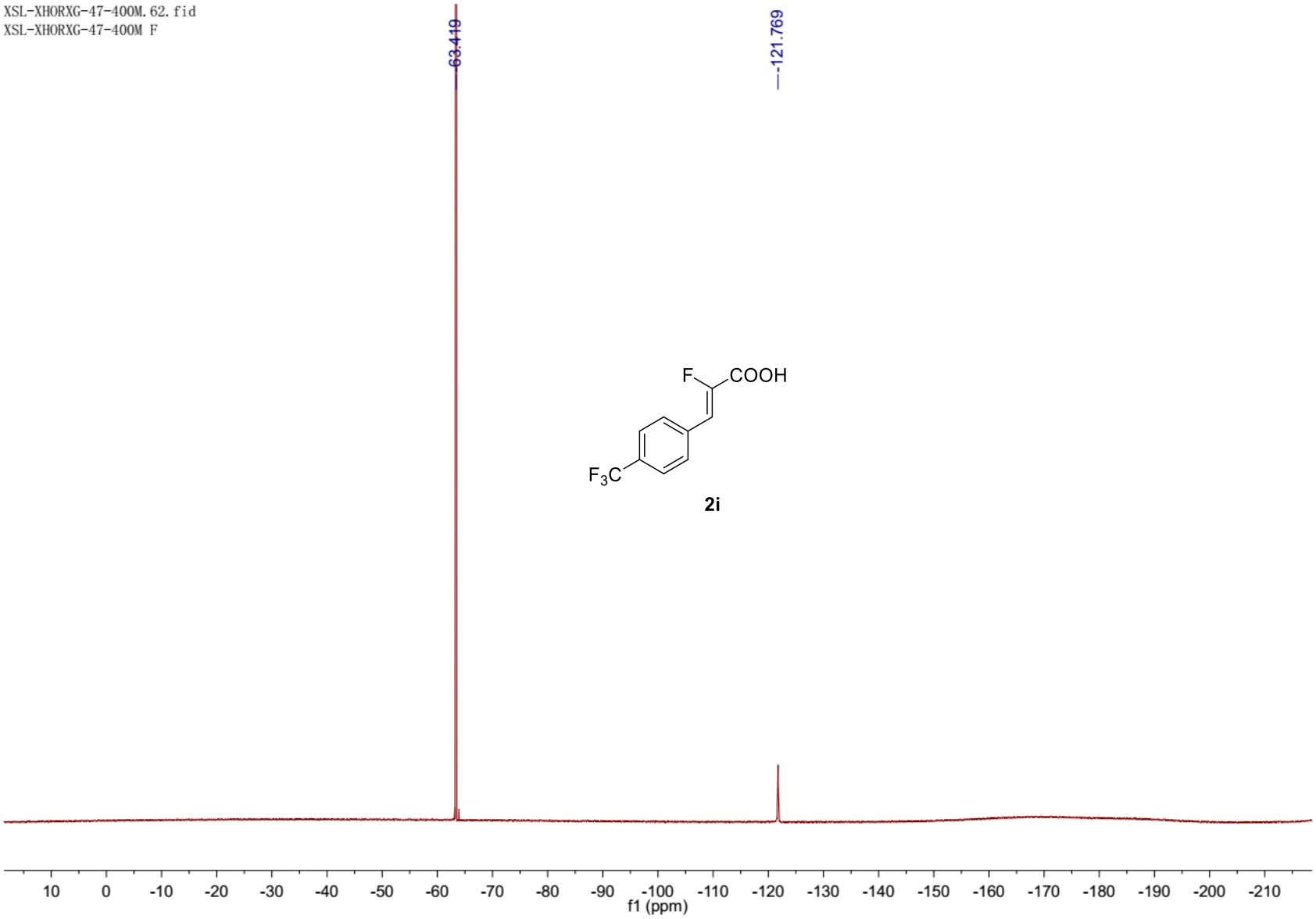
XSL-XH-34-400.11.fid
XSL-XH-34-400 F

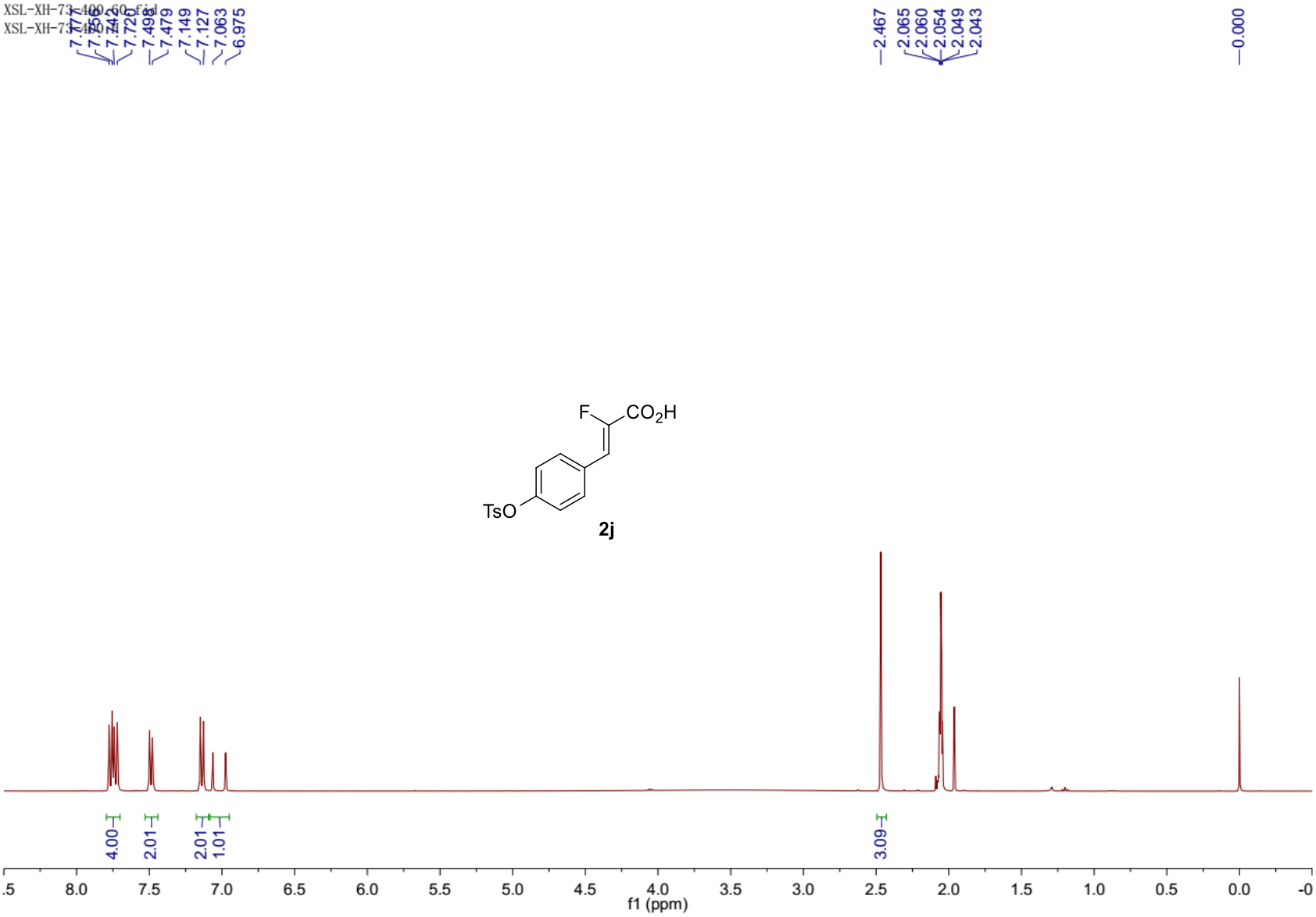


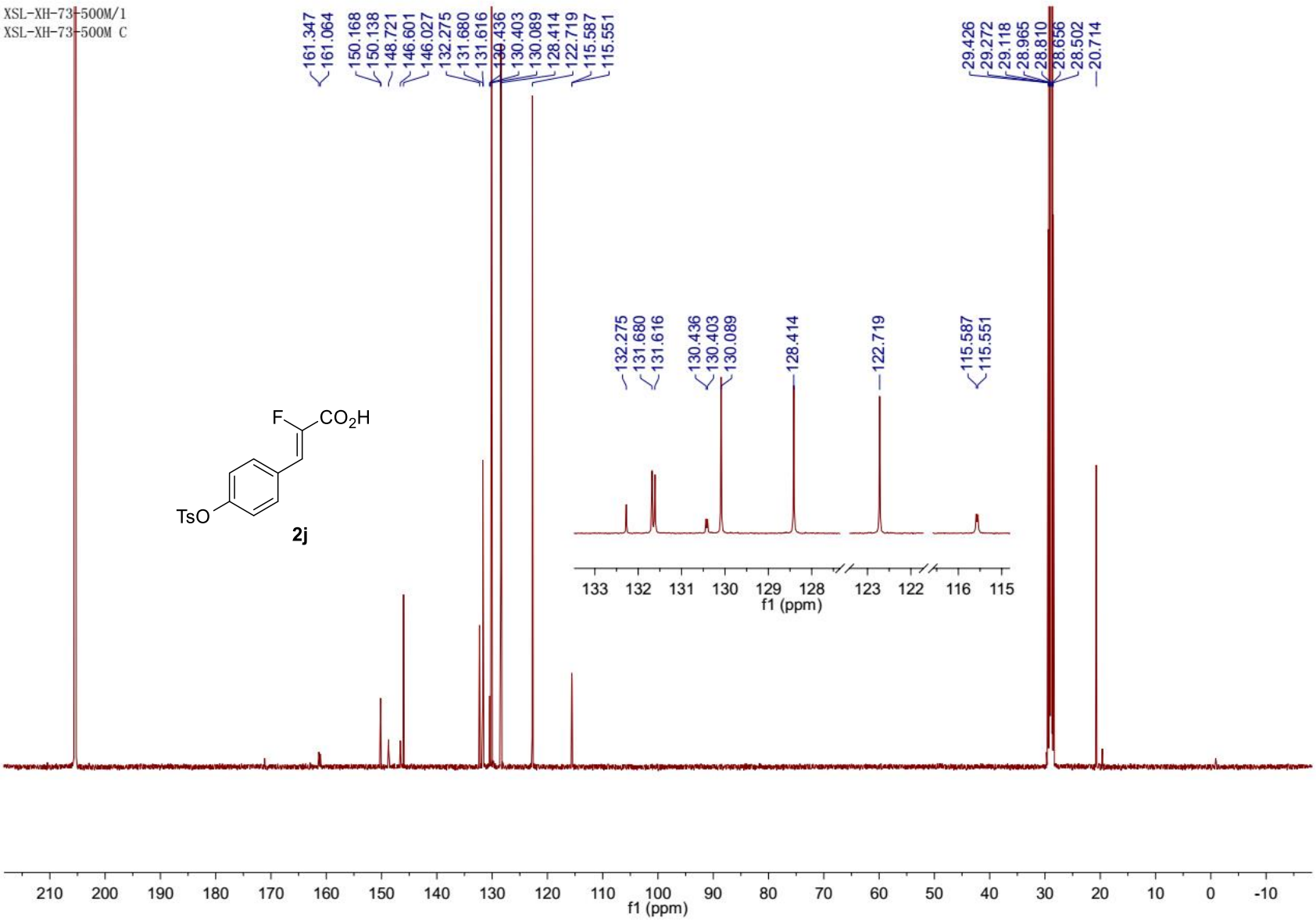
2h


XSL-XHORXG-47-400M-60.fid
7.936
7.822
7.801
7.170
7.083
3.489
2.068
2.062
2.057
2.051
2.046
0.000
F
COOH
F3C
2i
2.03
2.04
1.00
f1 (ppm)

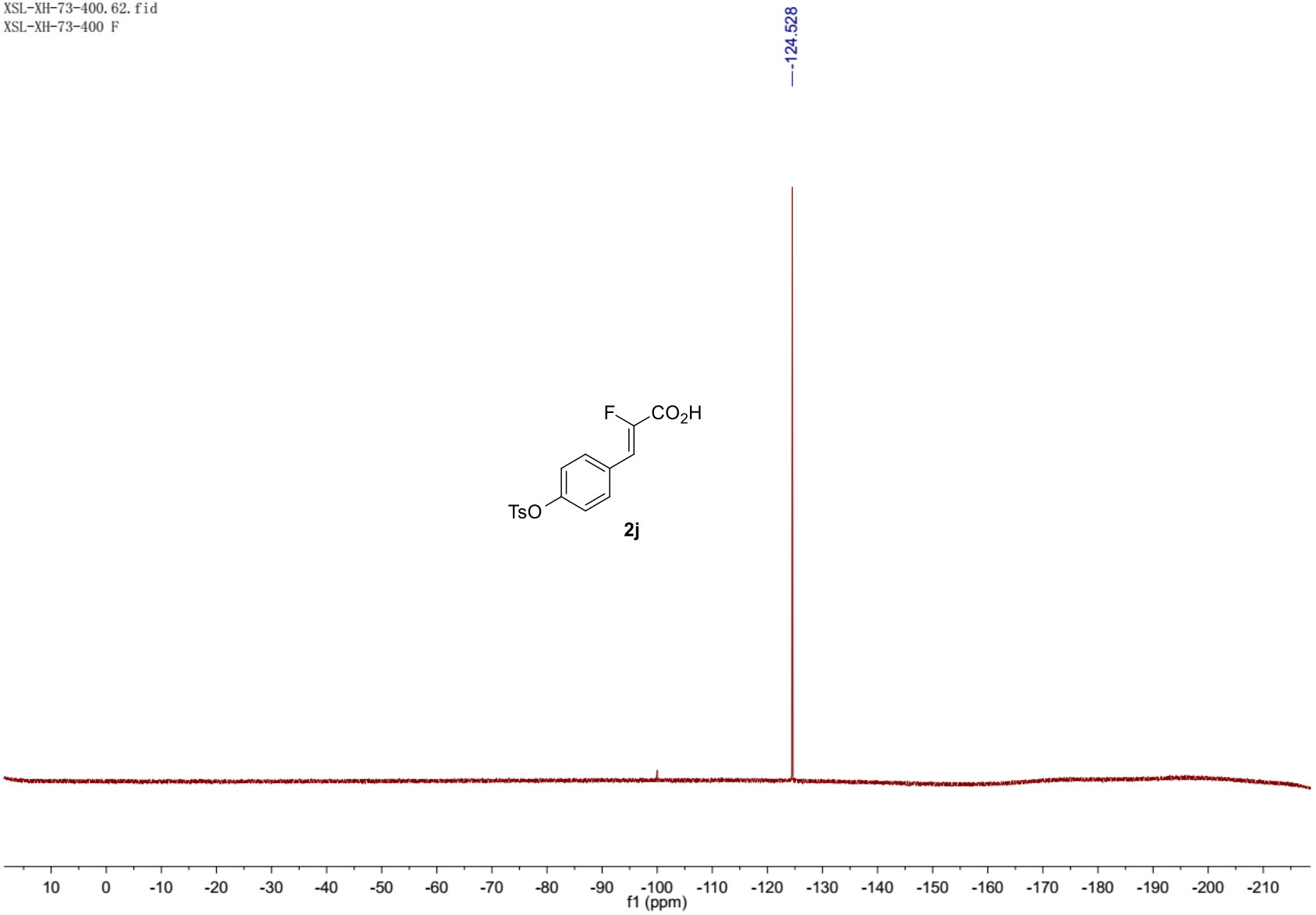

XSL-XHORXH-47-500M/2
XSL-XHORXH-47-500M C
161.271
160.991
149.836
147.697
135.288
130.630
130.565
130.421
130.173
129.916
127.403
125.669
125.636
125.244
123.086
120.927
115.261
F
COOH
F3C
2i
f1 (ppm)
f1 (ppm)

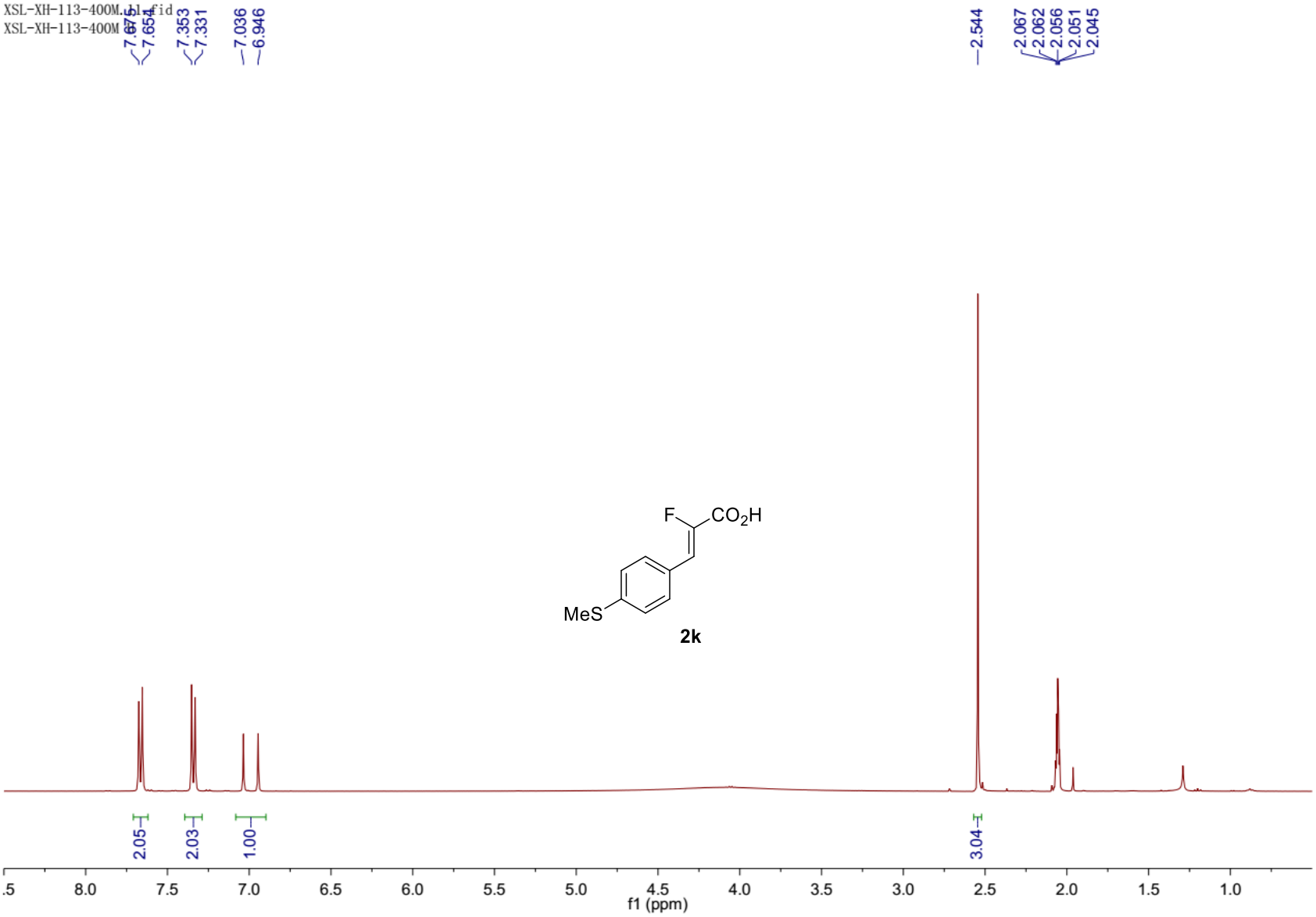

XSL-XHORXG-47-400M.62.fid
XSL-XHORXG-47-400M F
-63.419
-121.769
F
COOH
F3C
2i
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

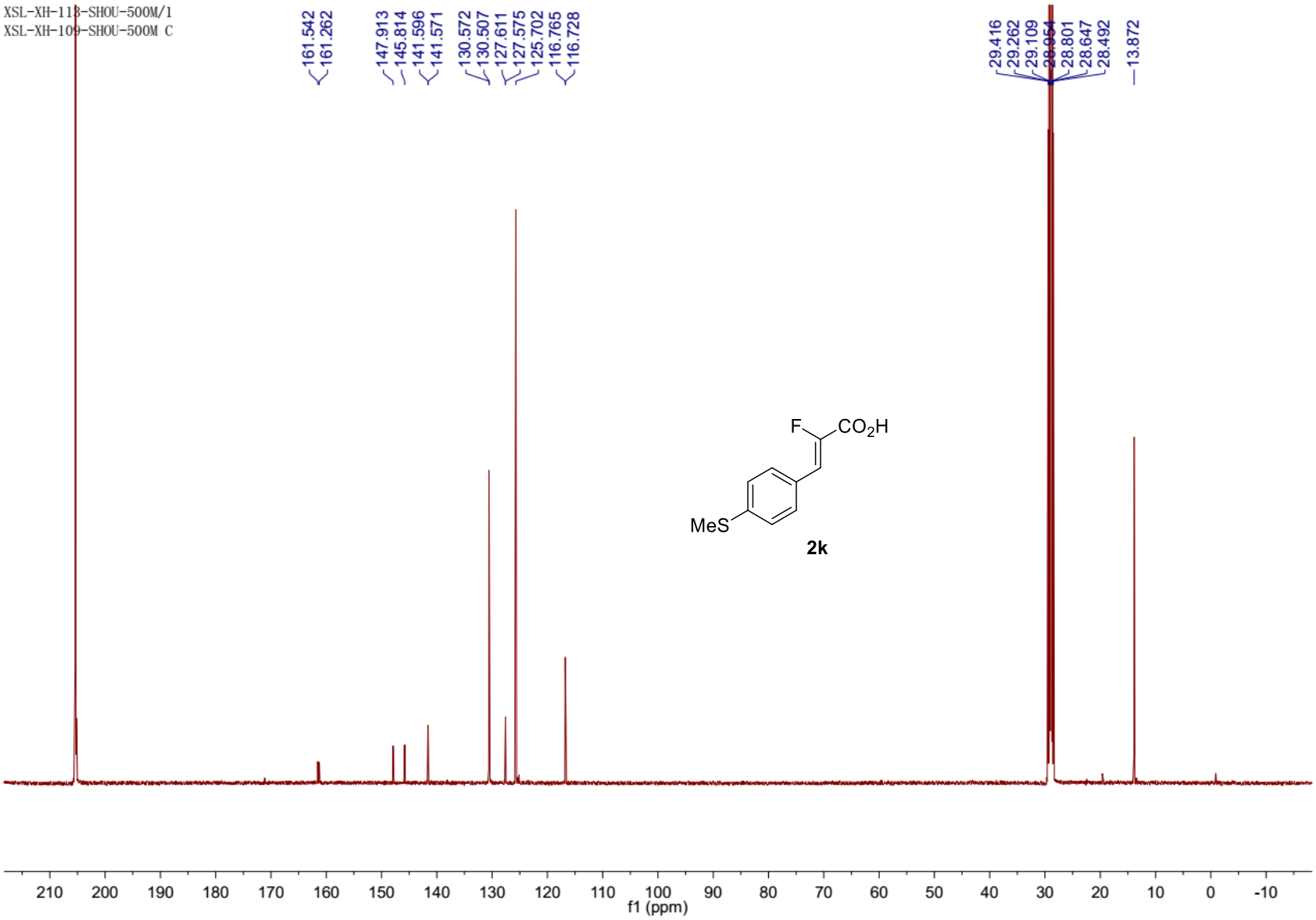

XSL-XH-73-400-60_fid
7.756
7.742
7.721
7.498
7.479
7.149
7.127
7.063
6.975
2.467
2.065
2.060
2.054
2.049
2.043
0.000
F
CO2H
TsO
2j
4.00
2.01
2.01
1.01
3.09
f1 (ppm)


XSL-XH-73-500M/1
XSL-XH-73-500M C
161.347
161.064
150.168
150.138
148.721
146.601
146.027
132.275
131.680
131.616
130.436
130.403
130.089
128.414
122.719
115.587
115.551
29.426
29.272
29.118
28.965
28.810
28.656
28.502
20.714
F
CO2H
TsO
2j
f1 (ppm)
f1 (ppm)

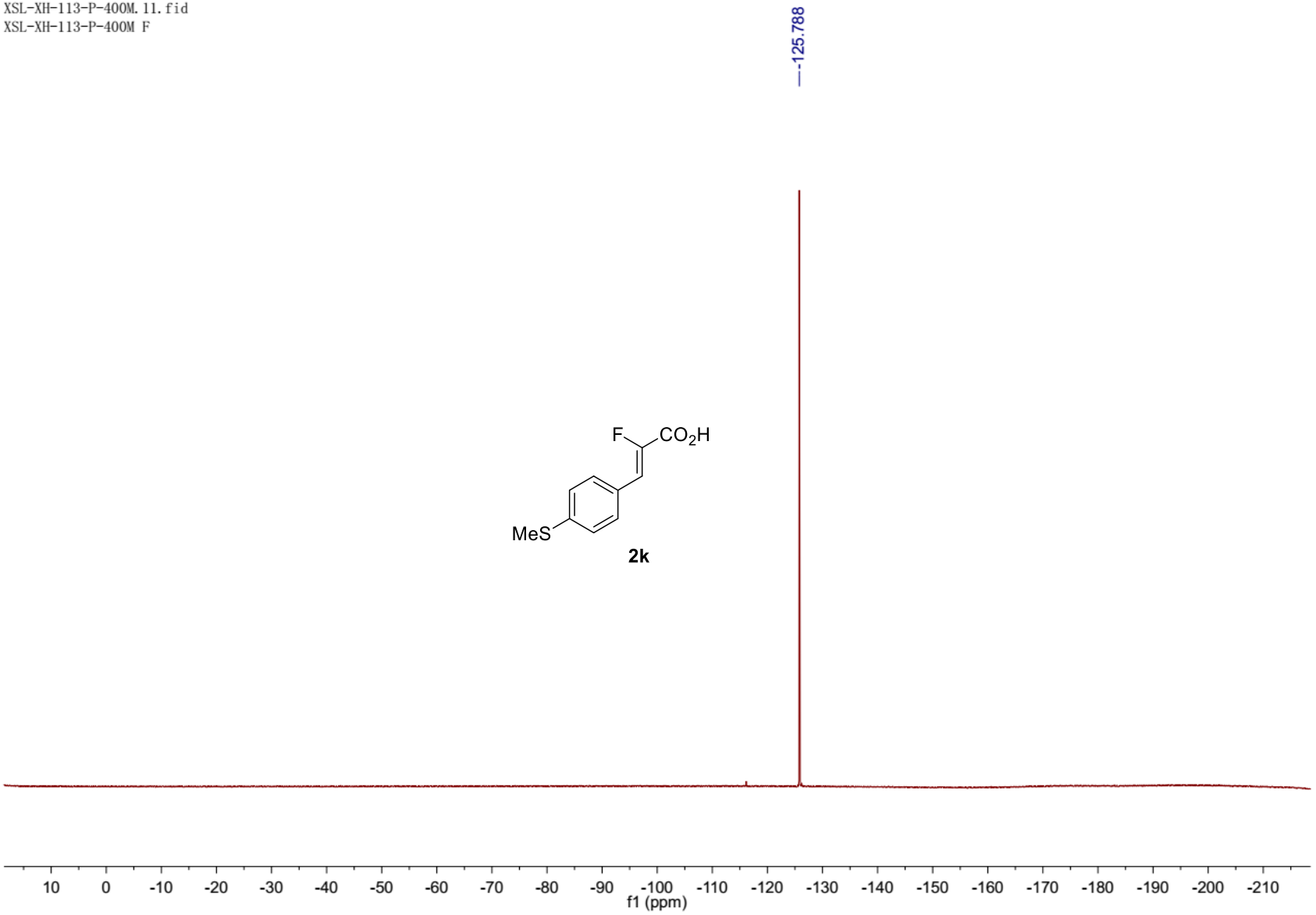
XSL-XH-73-400.62.fid
XSL-XH-73-400 F

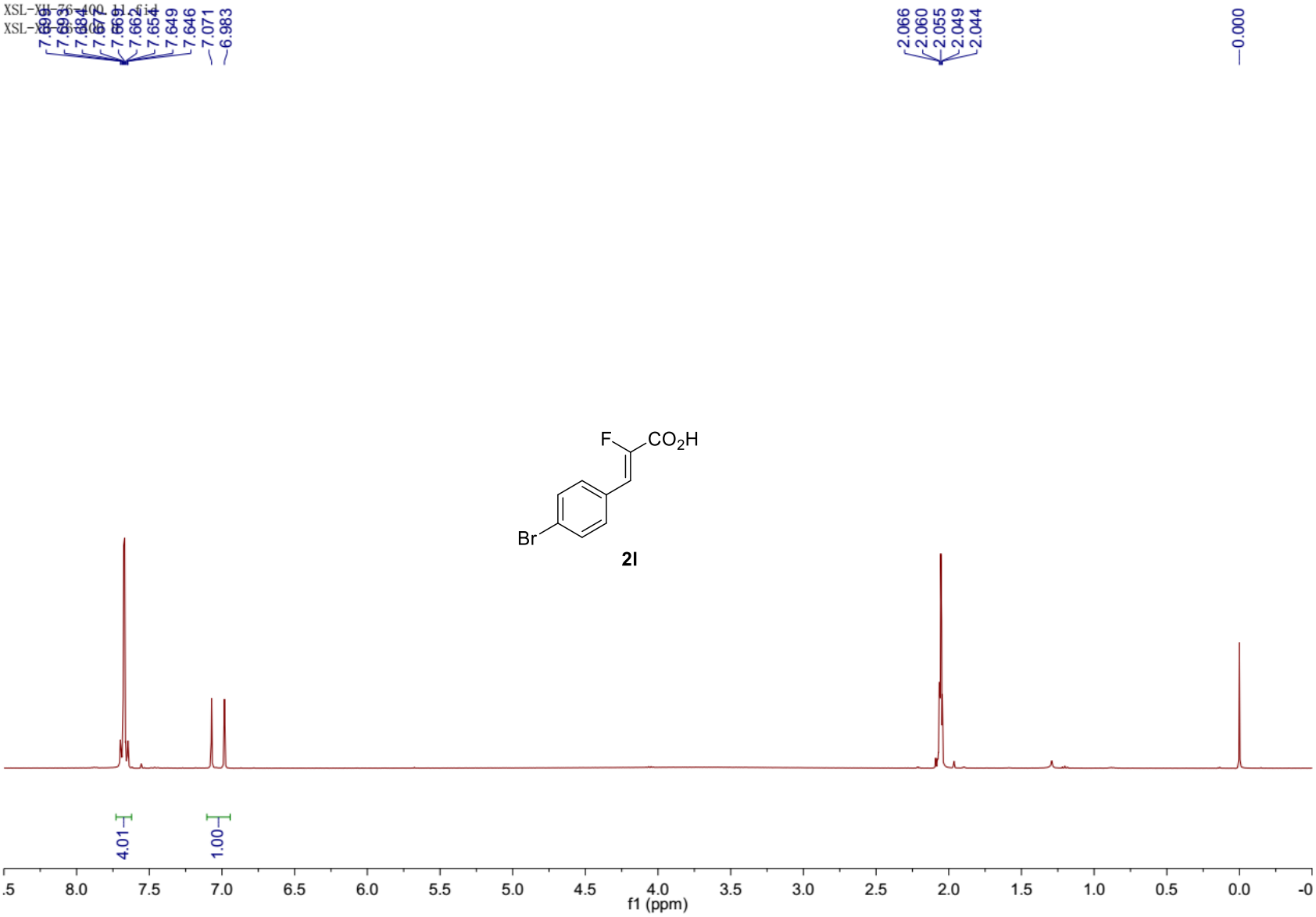


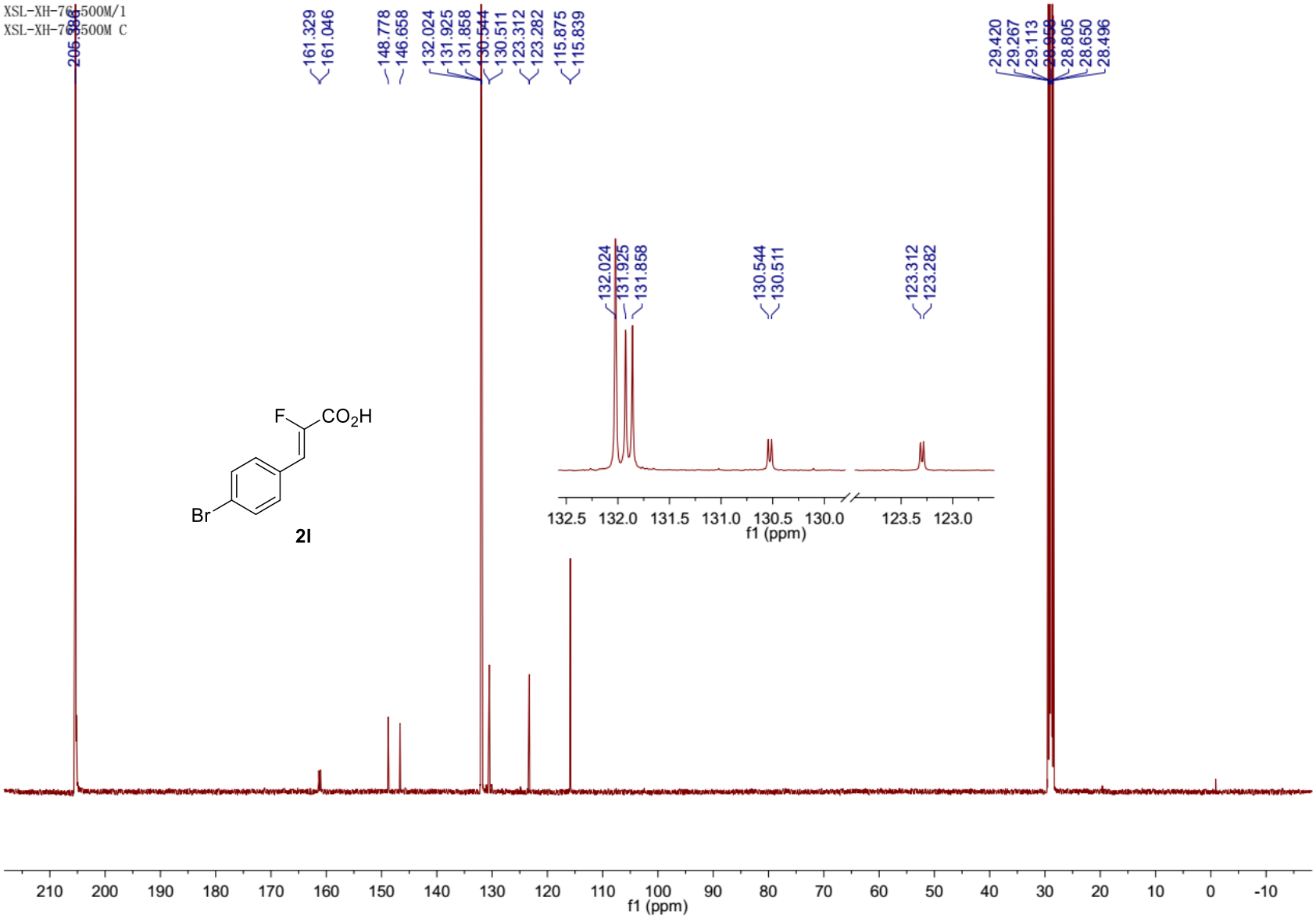

XSL-XH-113-400M.jl.fid
XSL-XH-113-400M.jl.fid
7.675
7.654
7.353
7.331
7.036
6.946
2.544
2.067
2.062
2.056
2.051
2.045
F
CO2H
MeS
2k
2.05
2.03
1.00
3.04
.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
f1 (ppm)


XSL-XH-113-SHOU-500M/1
XSL-XH-109-SHOU-500M C
161.542
161.262
147.913
145.814
141.596
141.571
130.572
130.507
127.611
127.575
125.702
116.765
116.728
29.416
29.262
29.109
28.954
28.801
28.647
28.492
13.872
F
CO2H
MeS
2k
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

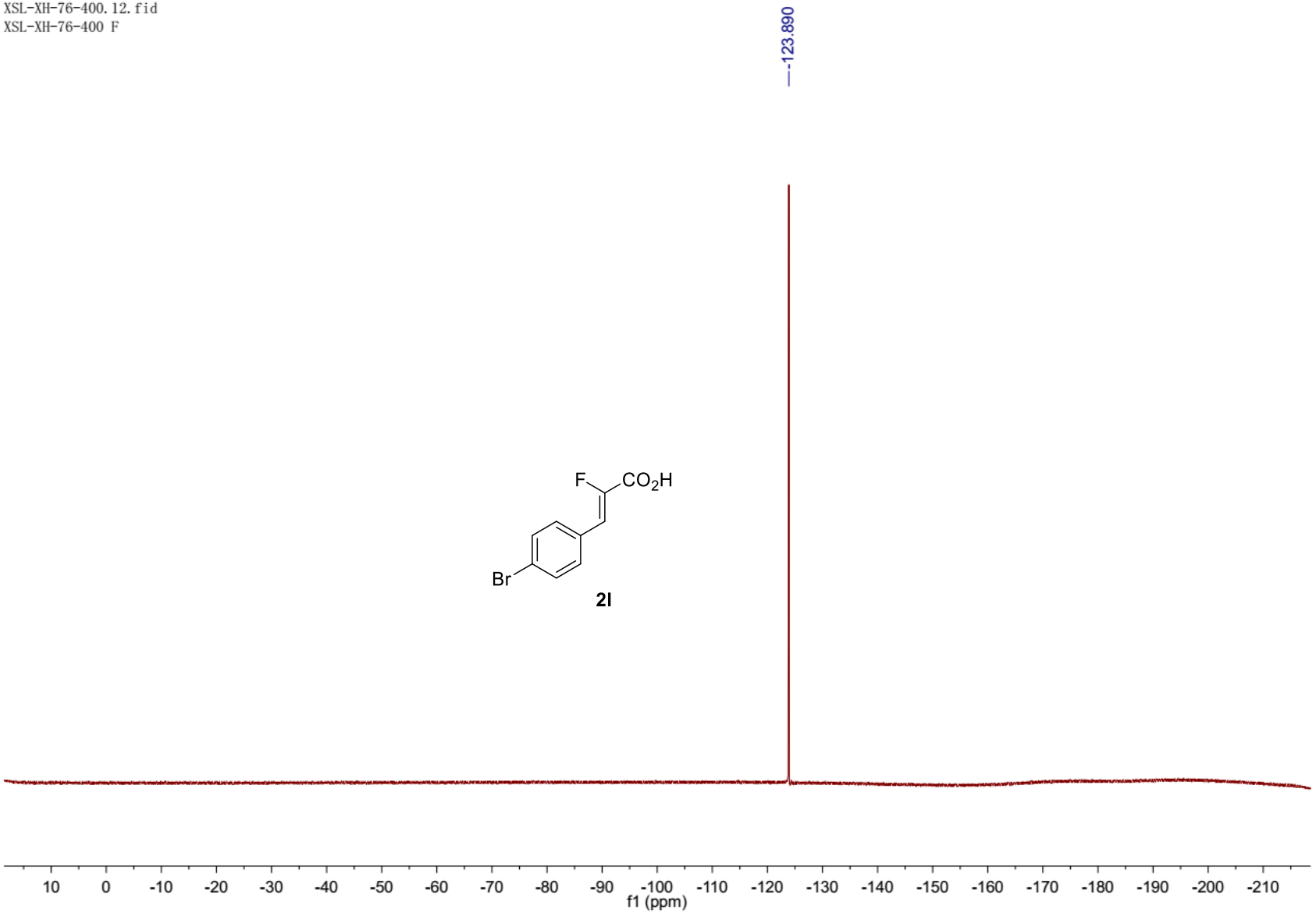
XSL-XH-113-P-400M.11.fid
XSL-XH-113-P-400M F



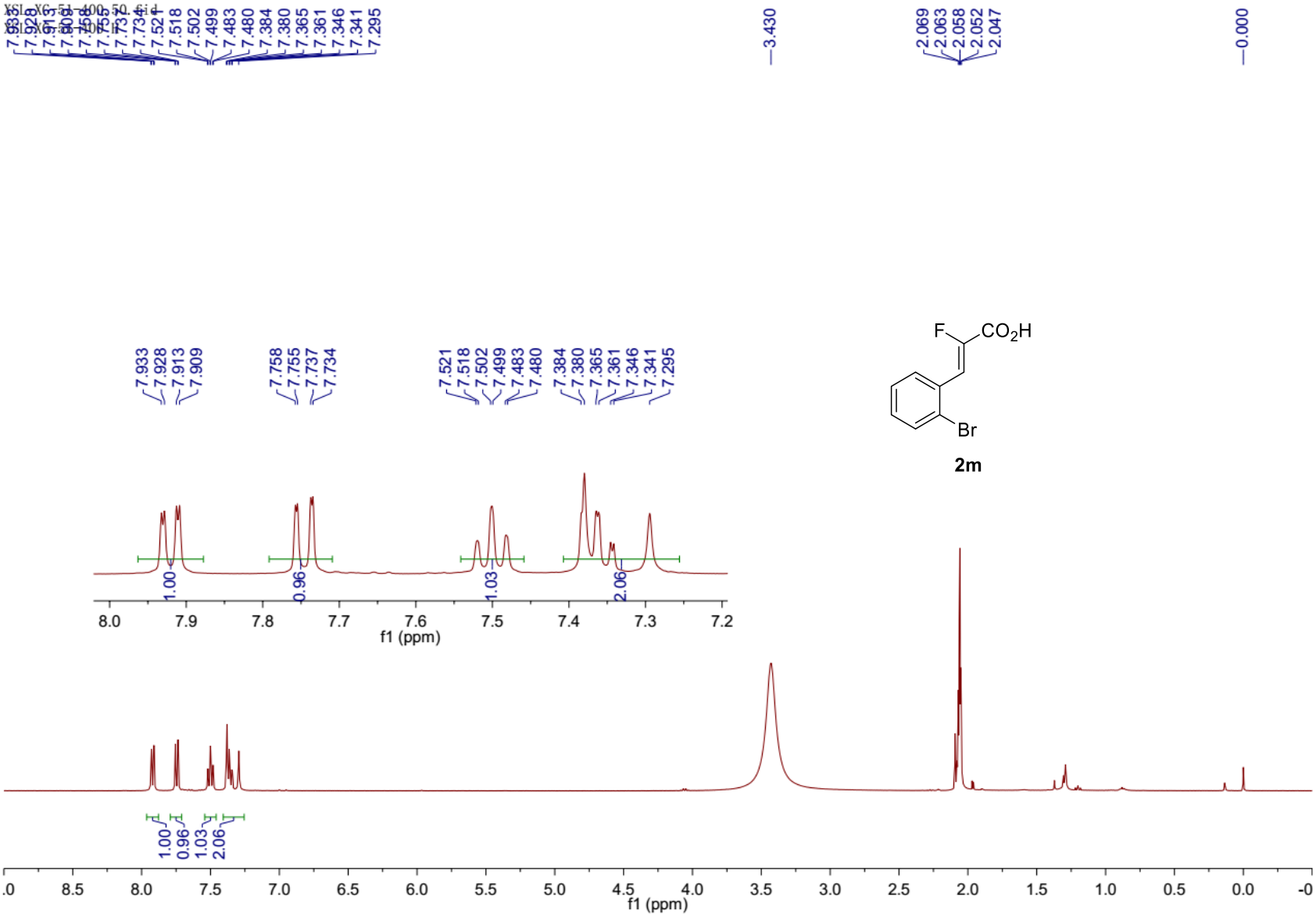

XSL-XH-76-400 1l.fid
7.695
7.693
7.684
7.677
7.669
7.662
7.654
7.649
7.646
7.071
6.983
2.066
2.060
2.055
2.049
2.044
0.000
F
CO2H
Br
2l
4.01
1.00
f1 (ppm)

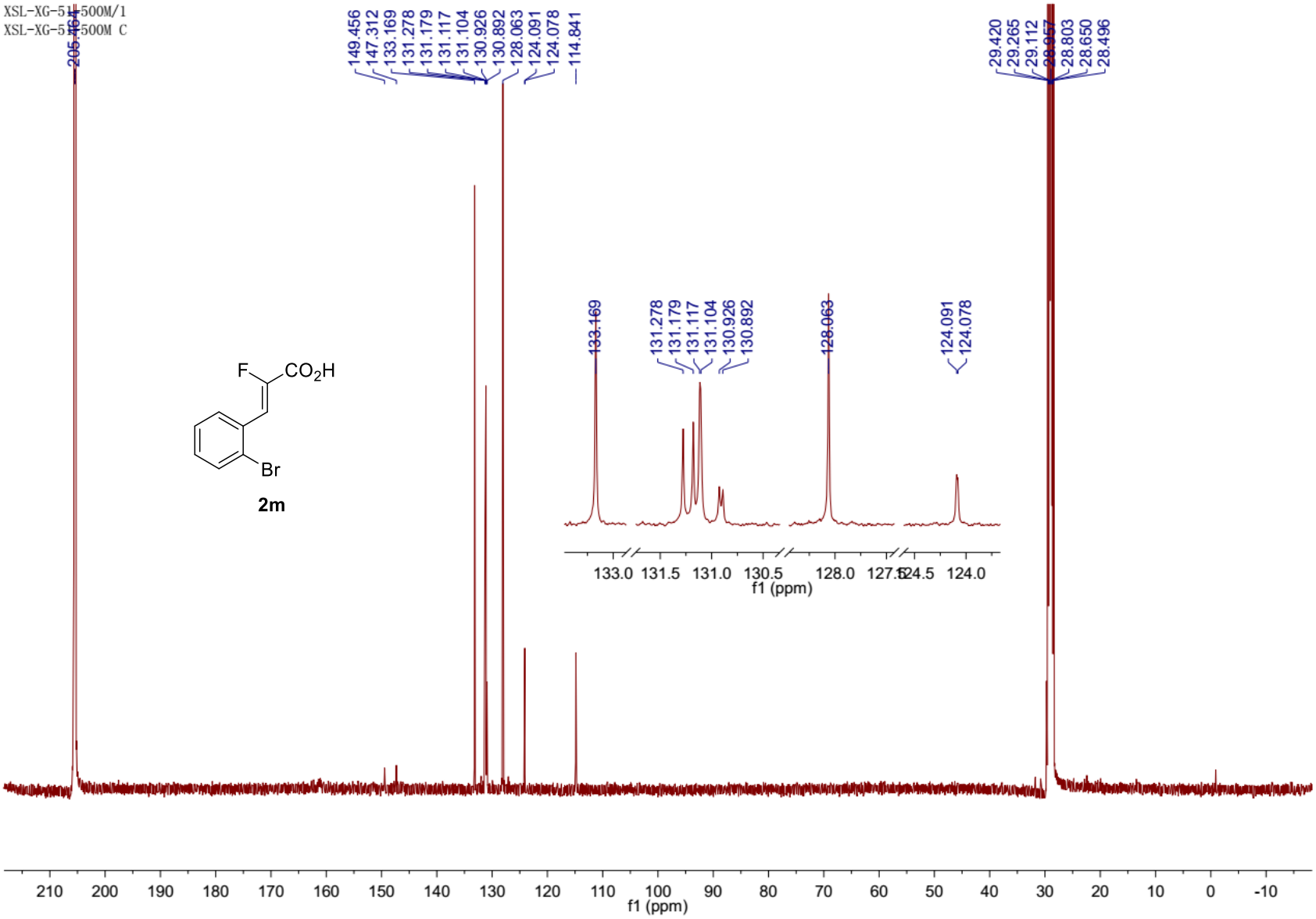

XSL-XH-76-500M/1
XSL-XH-76-500M C
205.386
161.329
161.046
148.778
146.658
132.024
131.925
131.858
130.544
130.511
123.312
123.282
115.875
115.839
29.420
29.267
29.113
28.805
28.650
28.496
F
CO2H
Br
2l
f1 (ppm)
f1 (ppm)

XSL-XH-76-400.12.fid
XSL-XH-76-400 F

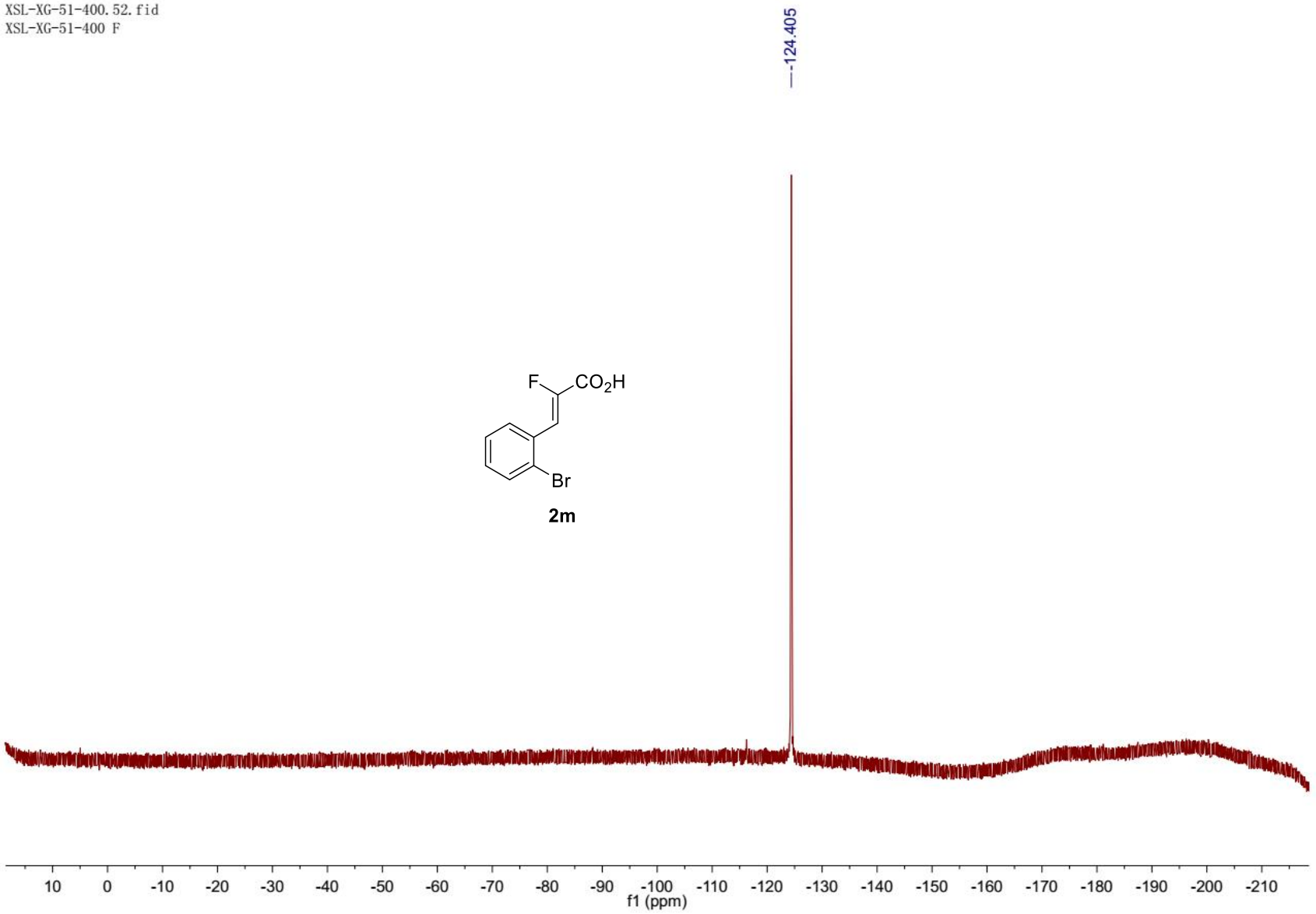


2l

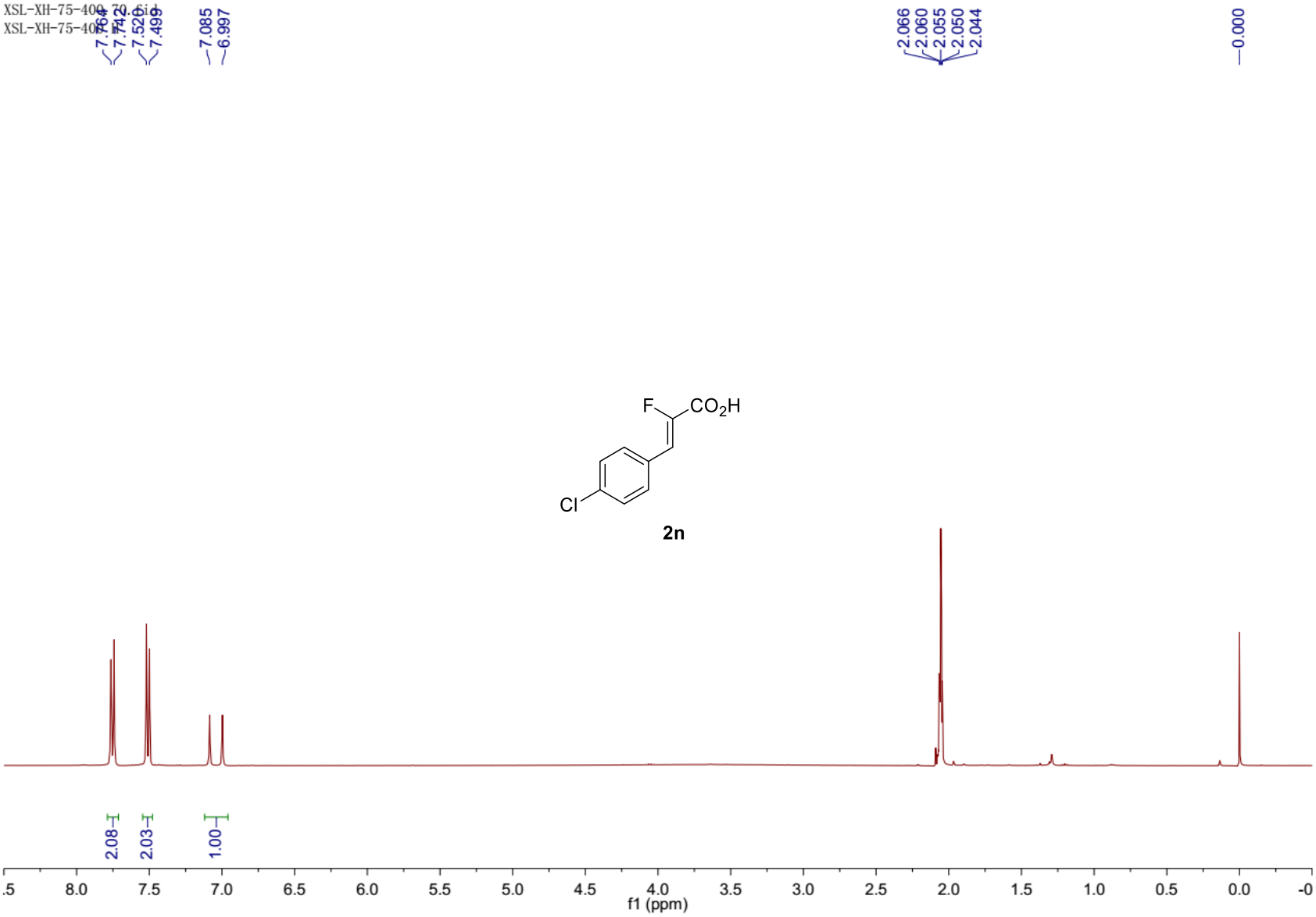

7.933
7.928
7.913
7.909
7.758
7.755
7.737
7.734
7.521
7.518
7.502
7.499
7.483
7.480
7.384
7.380
7.365
7.361
7.346
7.341
7.295
3.430
2.069
2.063
2.058
2.052
2.047
0.000
F
CO2H
Br
2m
1.00
0.96
1.03
2.06
f1 (ppm)

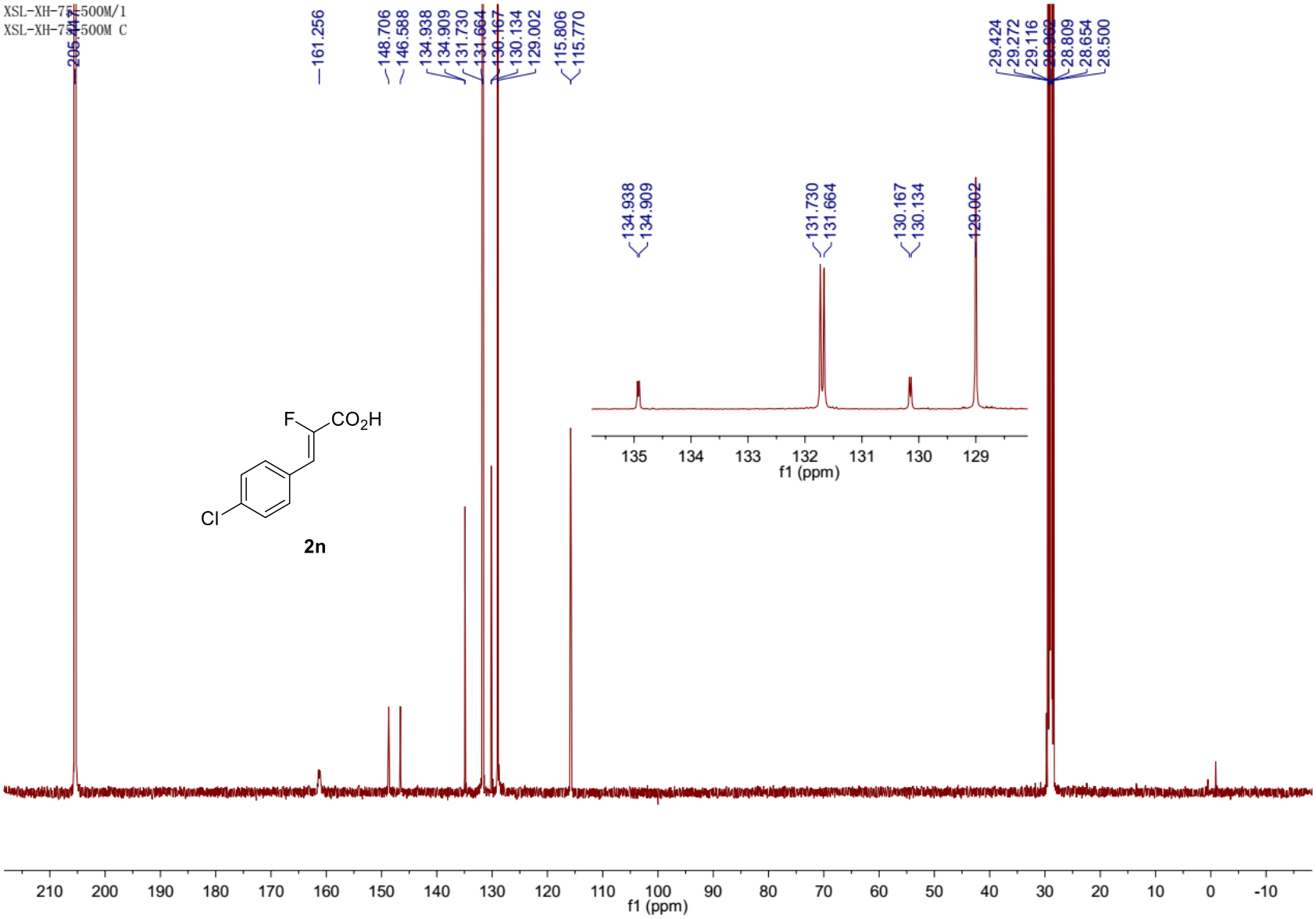

XSL-XG-51-500M/1
XSL-XG-51-500M C
149.456
147.312
133.169
131.278
131.179
131.117
131.104
130.926
130.892
128.063
124.091
124.078
114.841
29.420
29.265
29.112
28.957
28.803
28.650
28.496
F
CO2H
Br
2m
133.169
131.278
131.179
131.117
131.104
130.926
130.892
128.063
124.091
124.078
133.0 131.5 131.0 130.5 128.0 127.5 124.5 124.0
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

XSL-XG-51-400.52.fid
XSL-XG-51-400 F

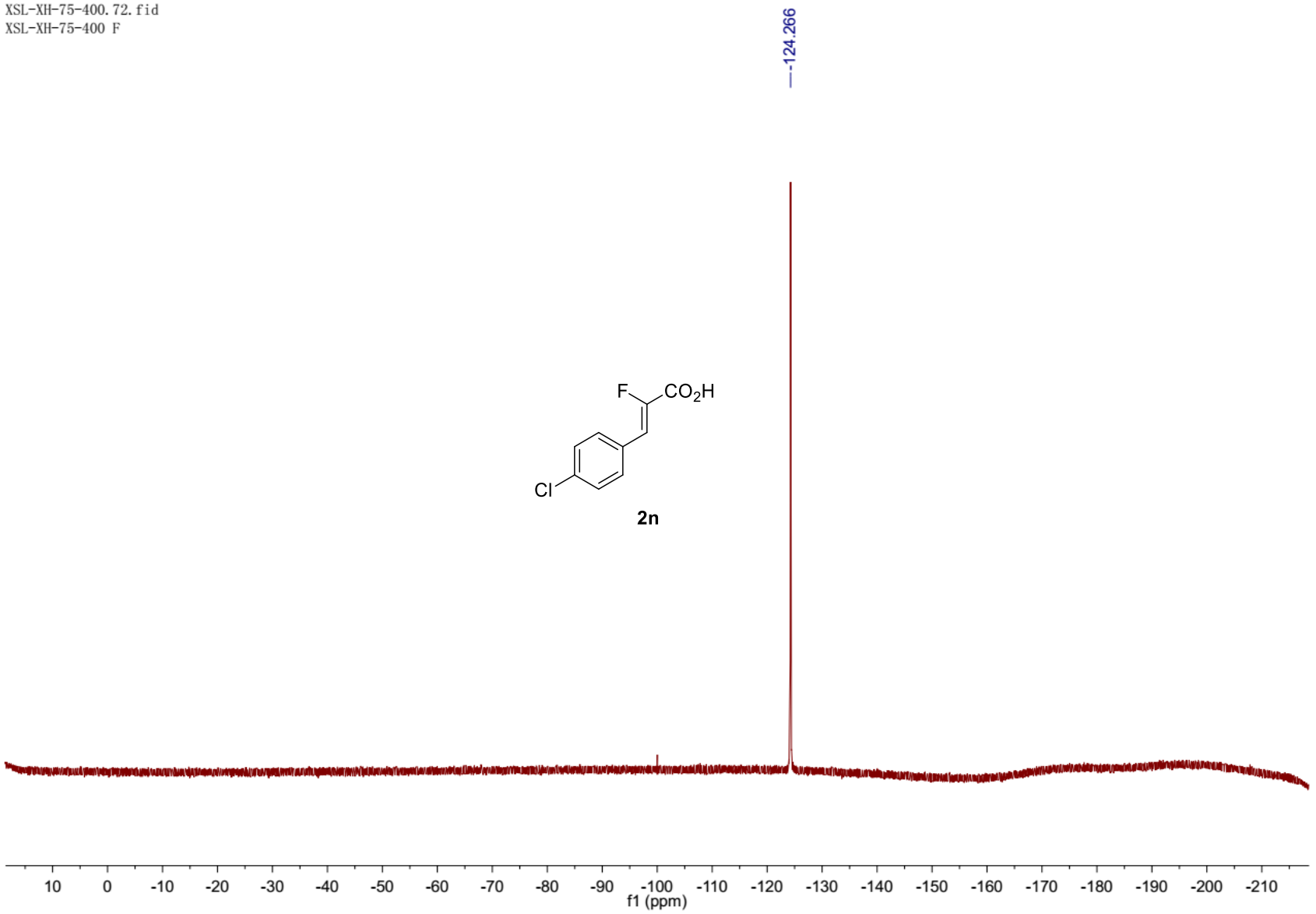


2m

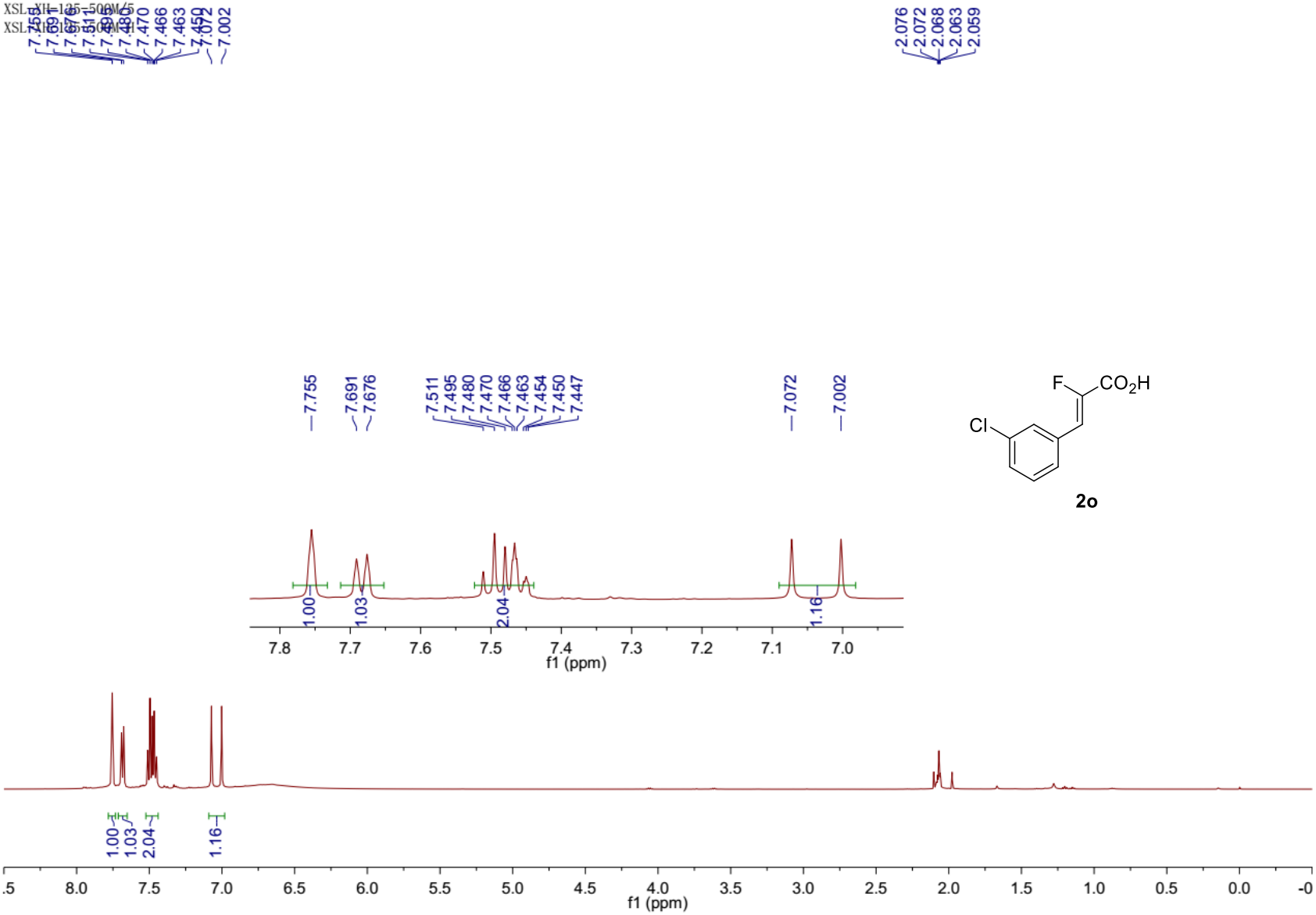

XSL-XH-75-400-70.6.1
XSL-XH-75-40
7.764
7.742
7.526
7.499
7.085
6.997
2.066
2.060
2.055
2.050
2.044
0.000
F
CO2H
Cl
2n
2.08
2.03
1.00
f1 (ppm)

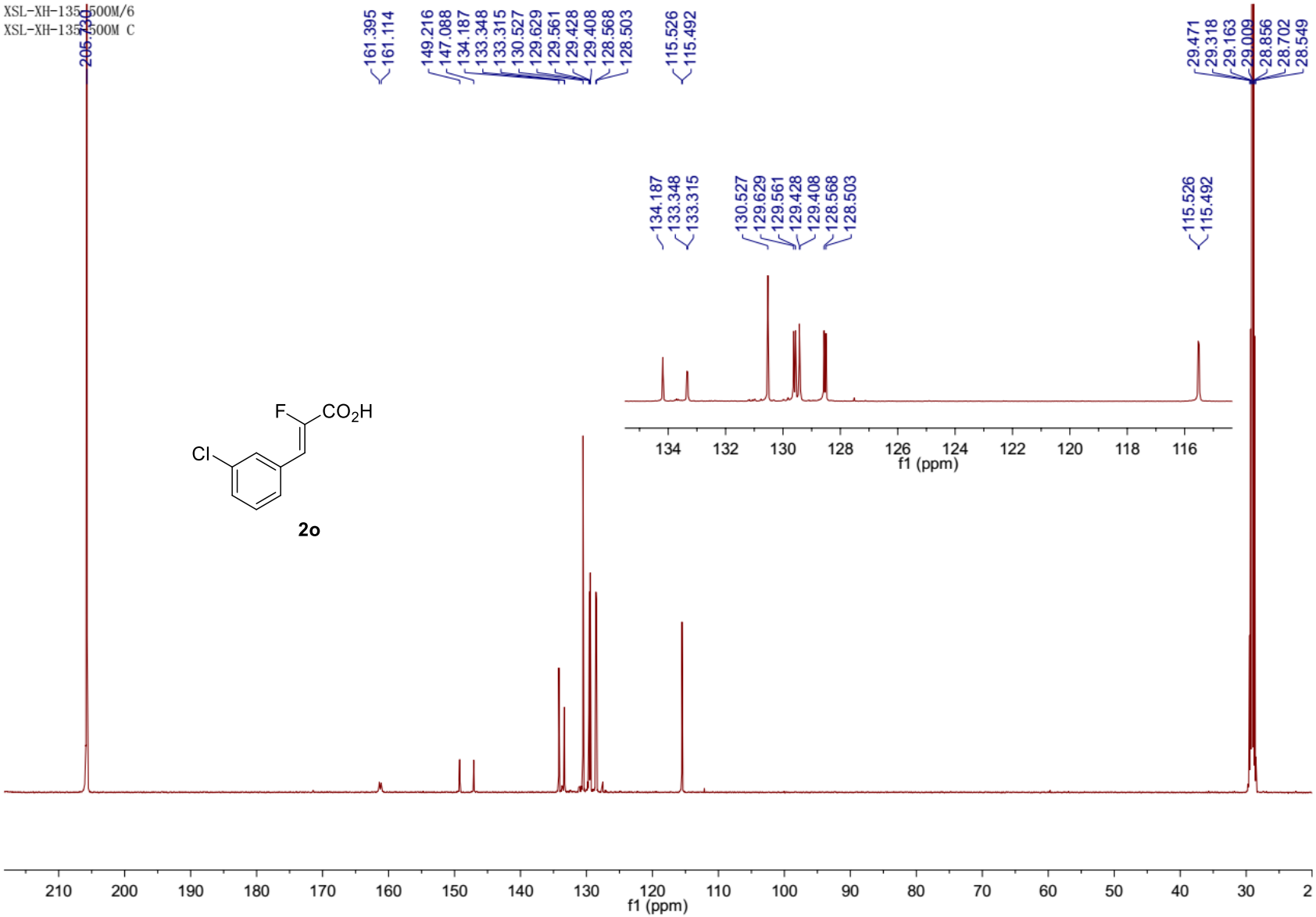

XSL-XH-75-500M/1
XSL-XH-7 500M C
161.256
148.706
146.588
134.938
134.909
131.730
131.664
130.167
130.134
129.002
115.806
115.770
29.424
29.272
29.116
28.809
28.654
28.500
F
CO2H
Cl
2n
f1 (ppm)
f1 (ppm)

XSL-XH-75-400.72.fid
XSL-XH-75-400 F

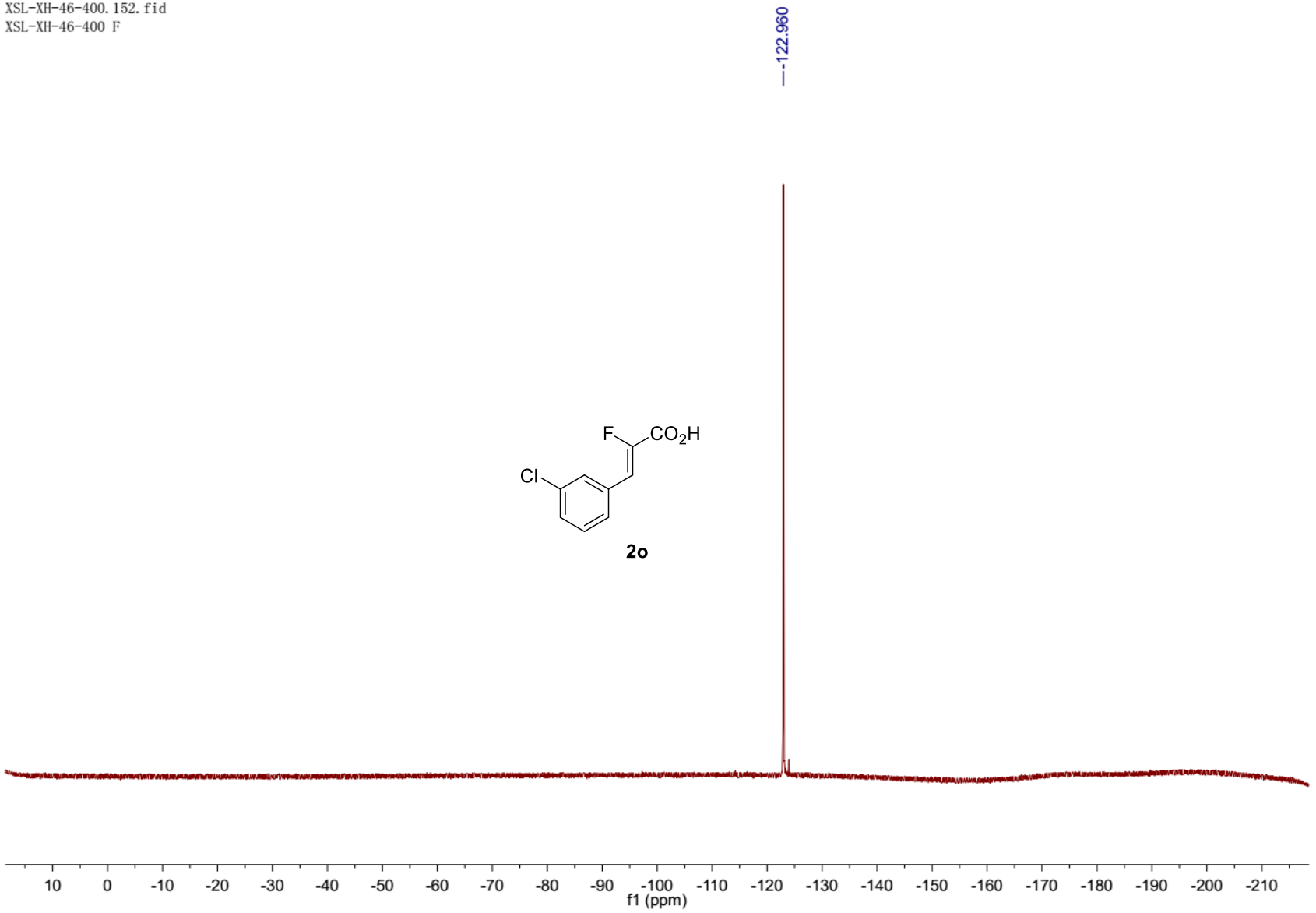


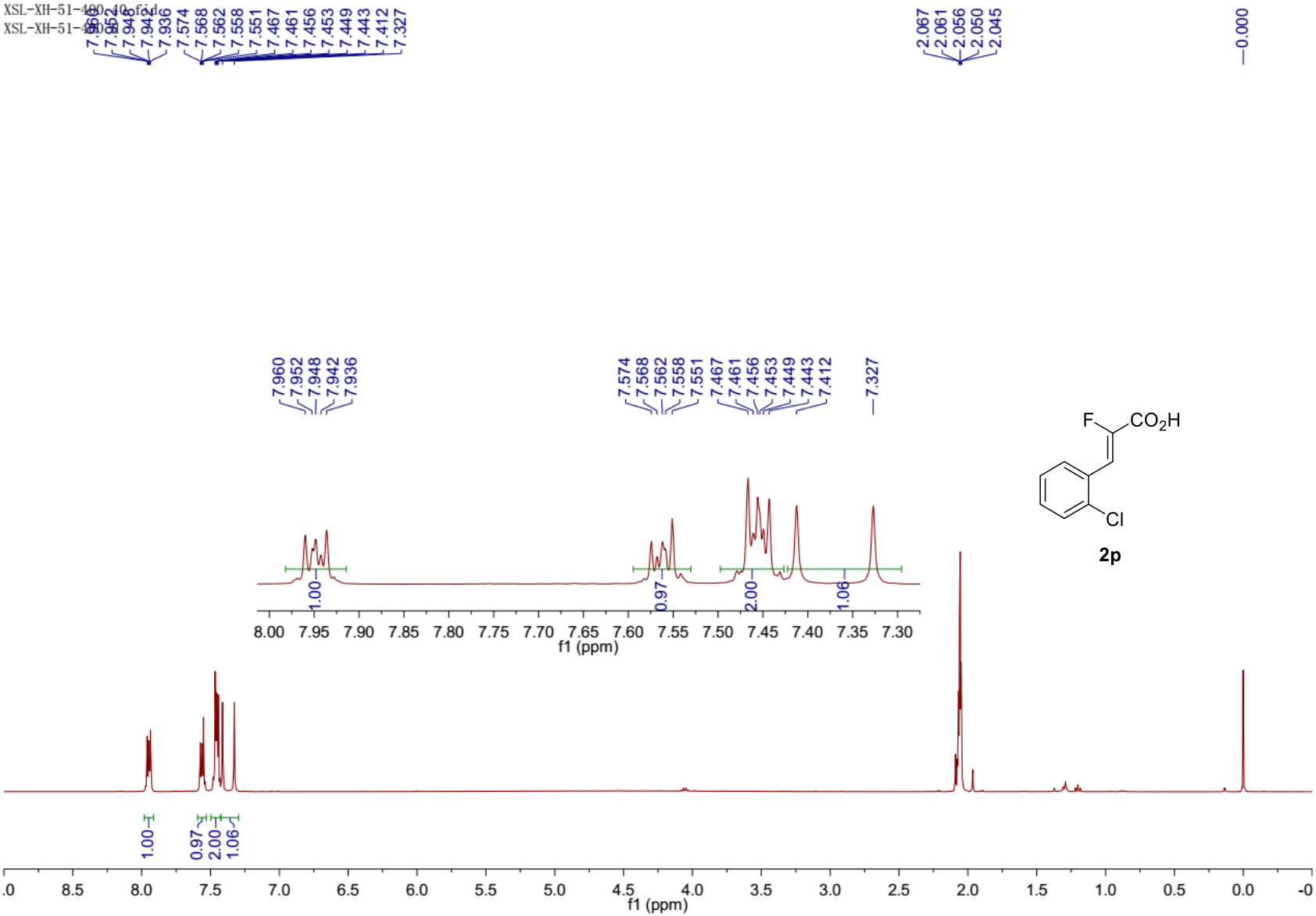
2n

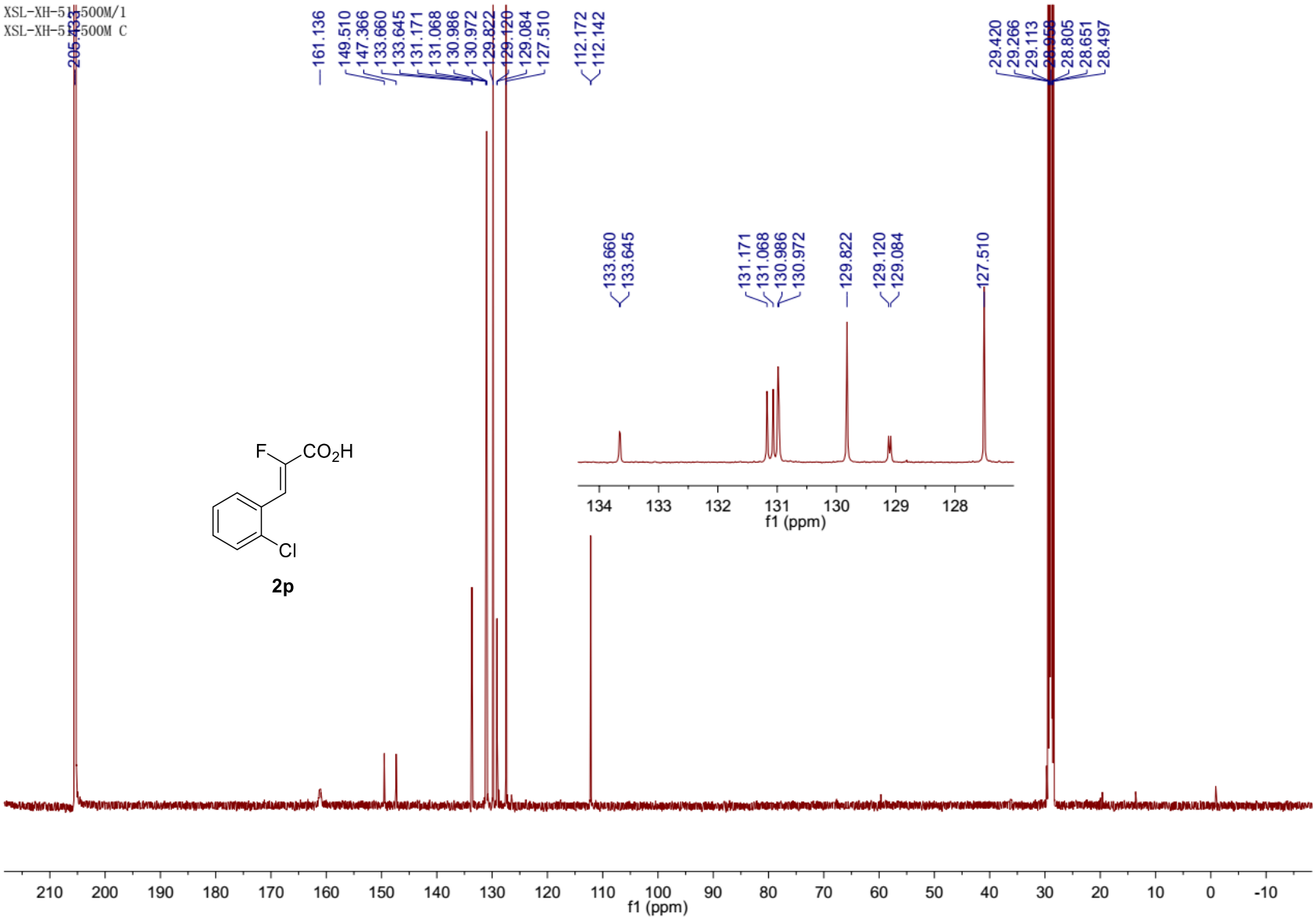

7.755
7.691
7.676
7.511
7.495
7.480
7.470
7.466
7.463
7.454
7.450
7.447
7.072
7.002
2.076
2.072
2.068
2.063
2.059
F
CO2H
Cl
2o
1.00
1.03
2.04
1.16
f1 (ppm)


XSL-XH-135-500M/6
XSL-XH-135-500M C
206.799
161.395
161.114
149.216
147.088
134.187
133.348
133.315
130.527
129.629
129.561
129.428
129.408
128.568
128.503
115.526
115.492
29.471
29.318
29.163
29.009
28.856
28.702
28.549
F
CO2H
Cl
2o
f1 (ppm)
f1 (ppm)

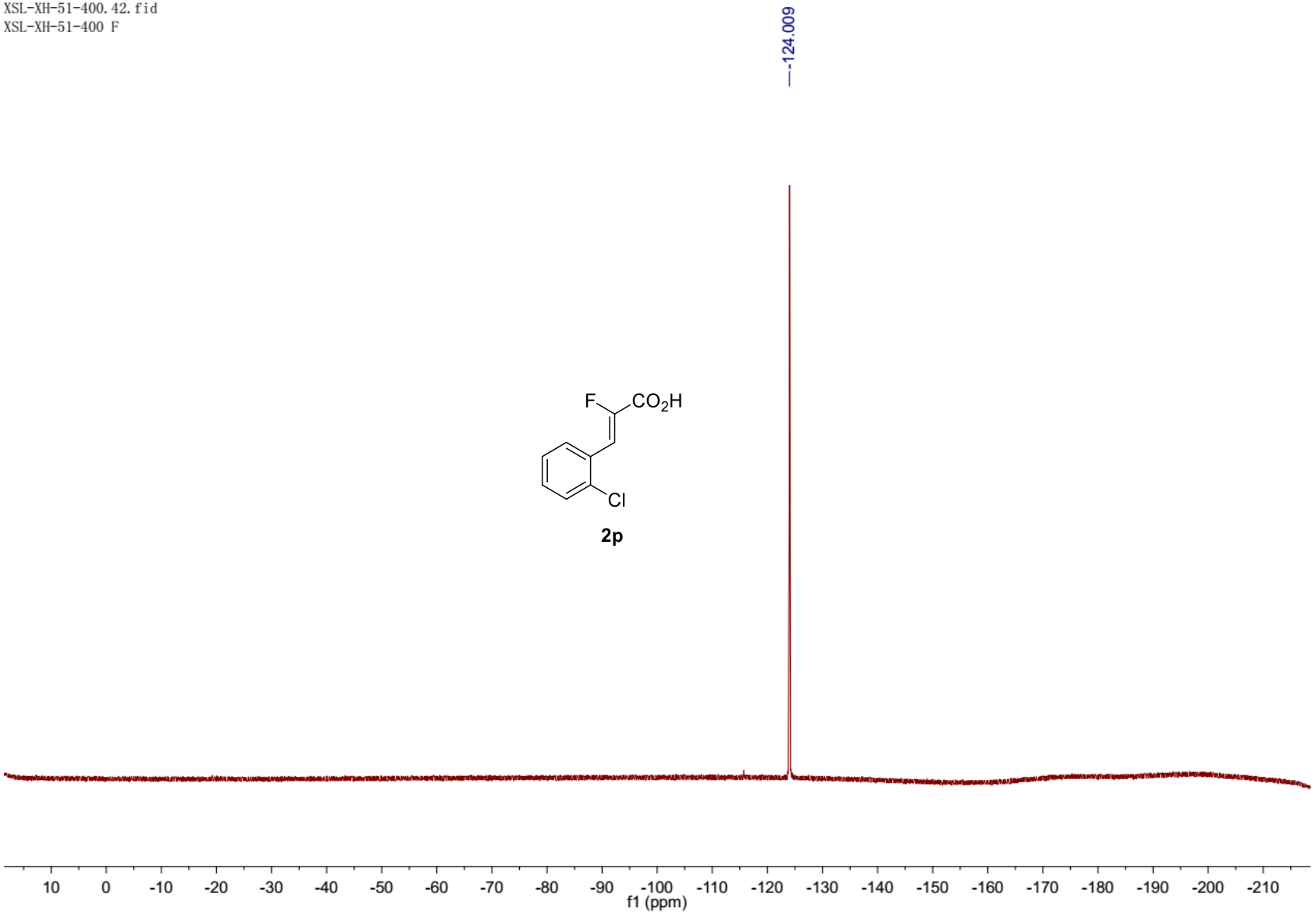
XSL-XH-46-400.152.fid
XSL-XH-46-400 F

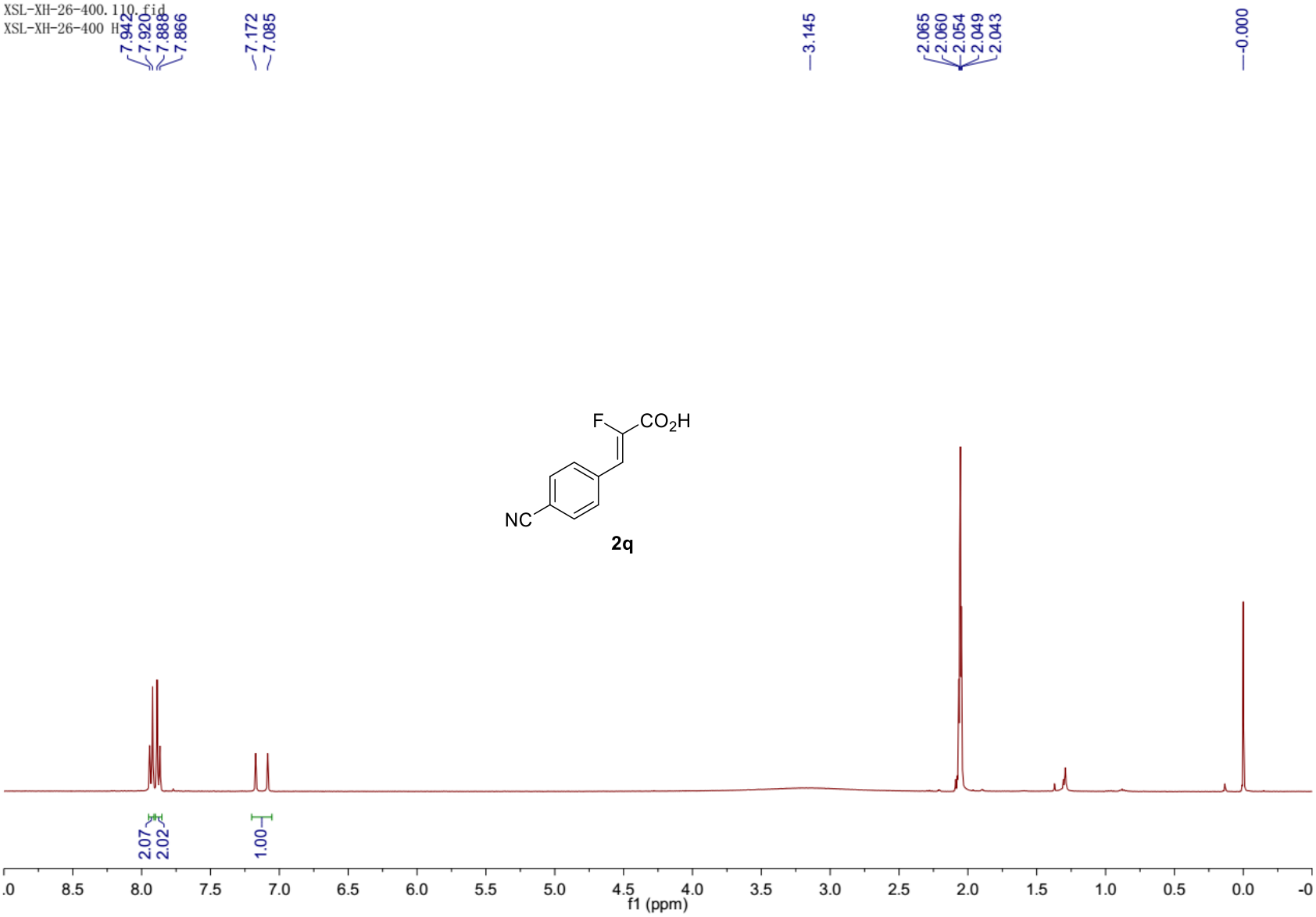


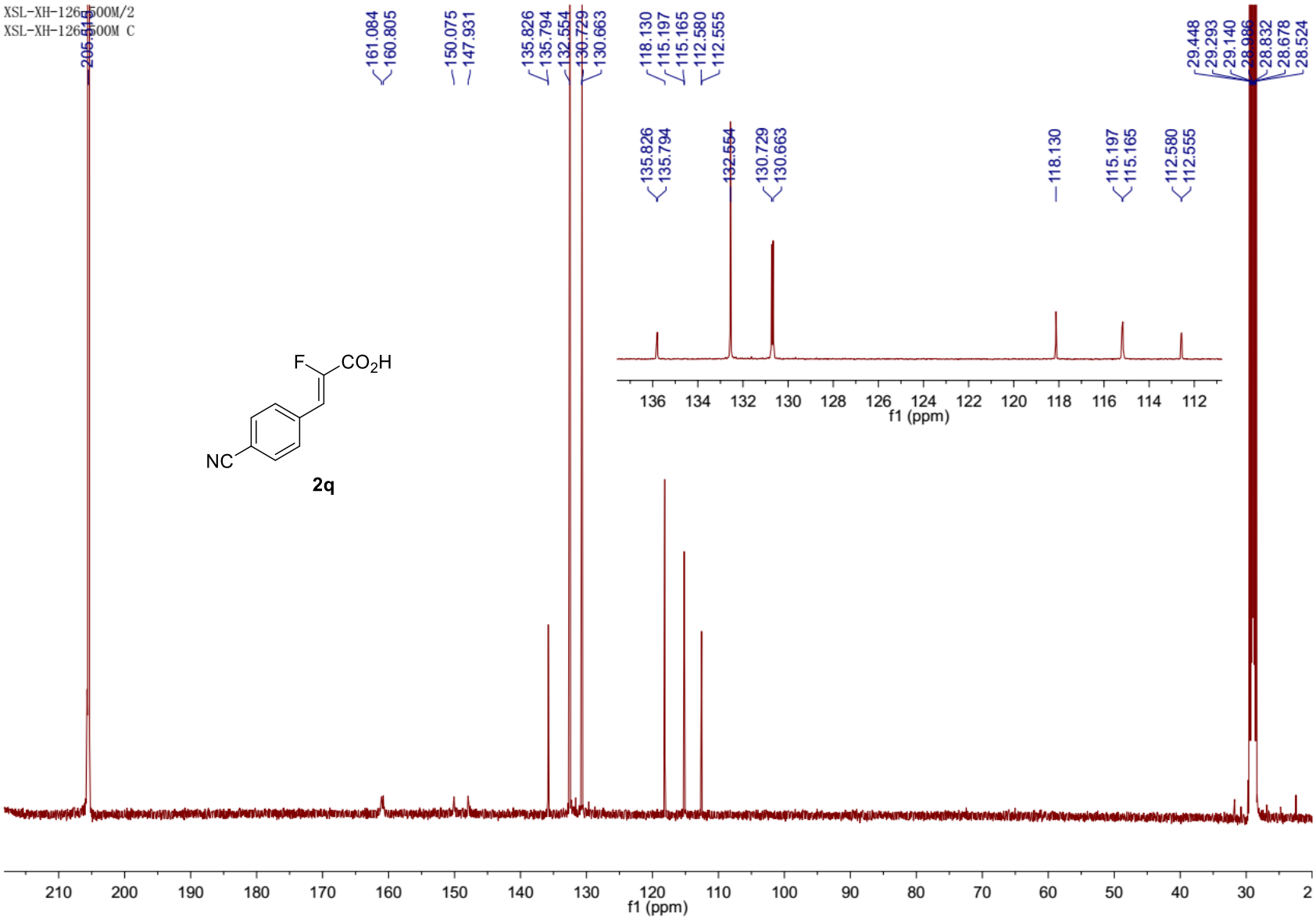

XSL-XH-51-400-10.fid
7.960
7.952
7.948
7.942
7.936
7.574
7.568
7.562
7.558
7.551
7.467
7.461
7.456
7.453
7.449
7.443
7.412
7.327
2.067
2.061
2.056
2.050
2.045
0.000
F
CO2H
Cl
2p
1.00
0.97
2.00
1.06
f1 (ppm)


XSL-XH-51-500M/1
XSL-XH-51-500M C
161.136
149.510
147.366
133.660
133.645
131.171
131.068
130.986
130.972
129.822
129.120
129.084
127.510
112.172
112.142
29.420
29.266
29.113
28.805
28.651
28.497
F
CO2H
Cl
2p
f1 (ppm)

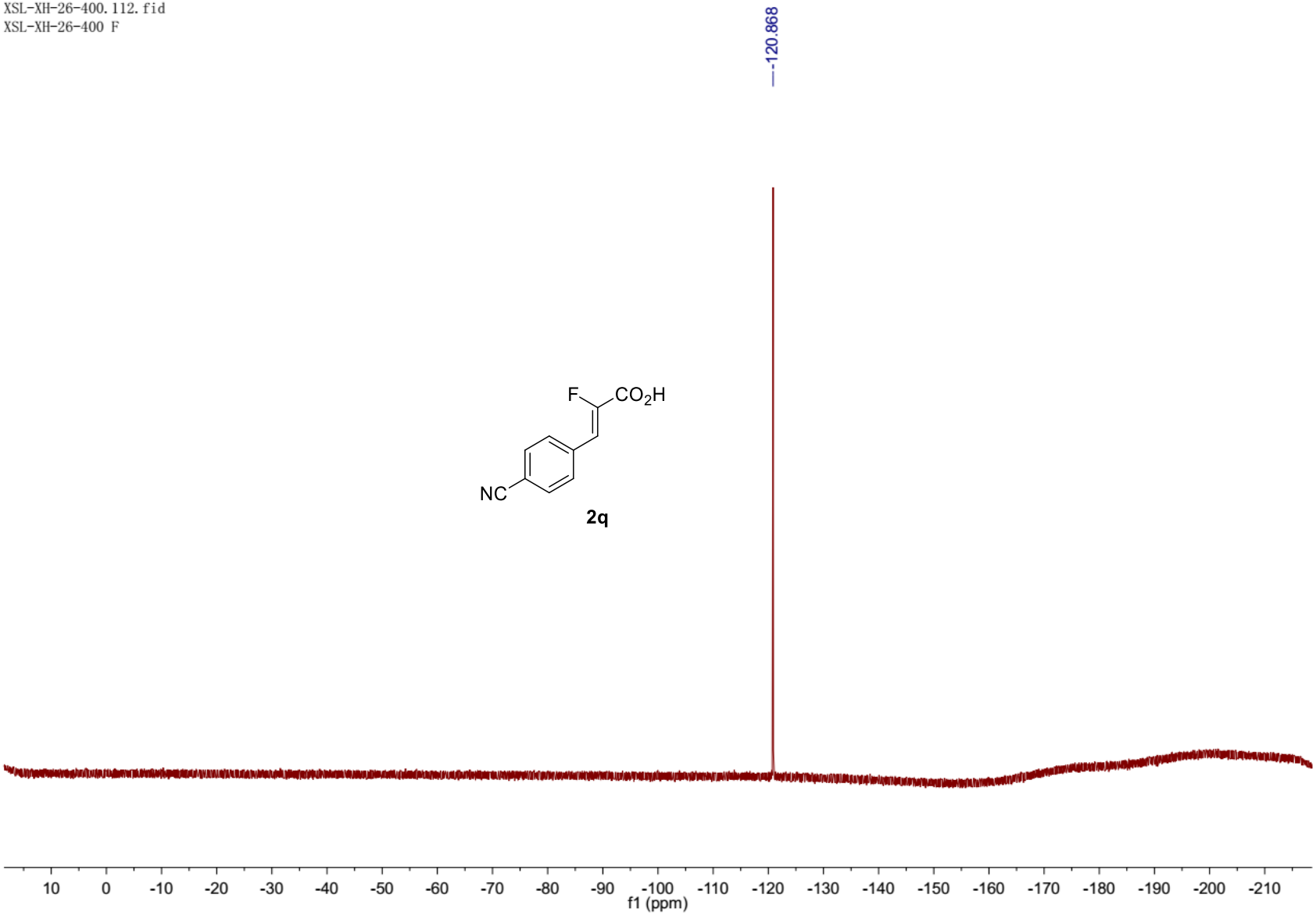
XSL-XH-51-400.42.fid
XSL-XH-51-400 F



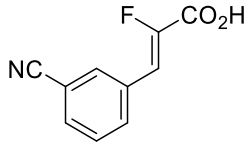

XSL-XH-26-400.110.fid
XSL-XH-26-400 H
7.942
7.920
7.888
7.866
7.172
7.085
3.145
2.065
2.060
2.054
2.049
2.043
0.000
F
CO2H
NC
2q
2.07
2.02
1.00
f1 (ppm)


XSL-XH-126-600M/2
XSL-XH-126-600M C
205.815
161.084
160.805
150.075
147.931
135.826
135.794
132.554
130.729
130.663
118.130
115.197
115.165
112.580
112.555
29.448
29.293
29.140
28.986
28.832
28.678
28.524
F
CO2H
NC
2q
f1 (ppm)
f1 (ppm)

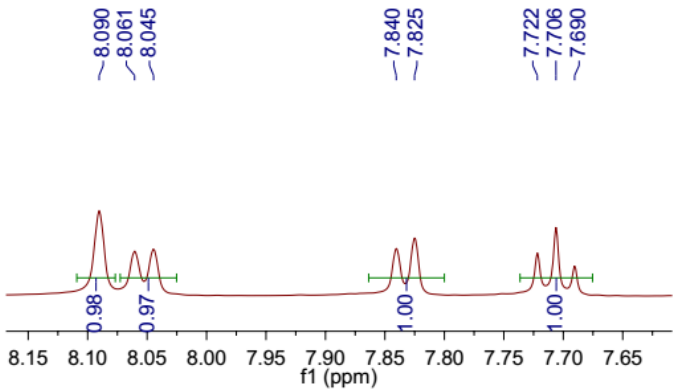
XSL-XH-26-400.112.fid
XSL-XH-26-400 F

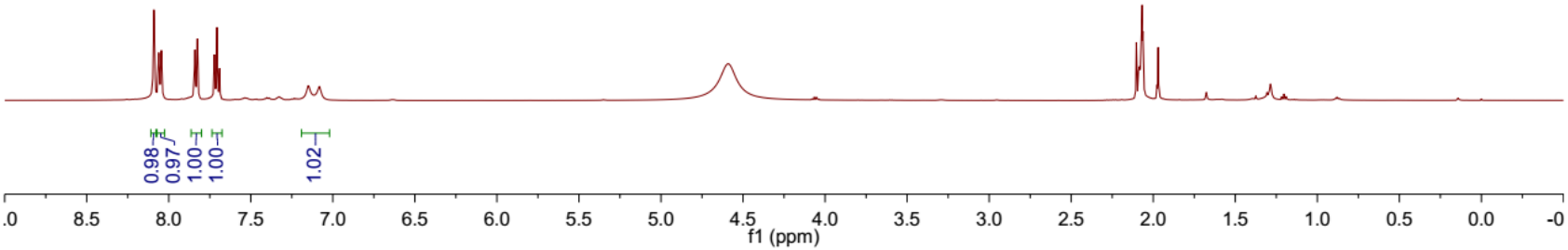


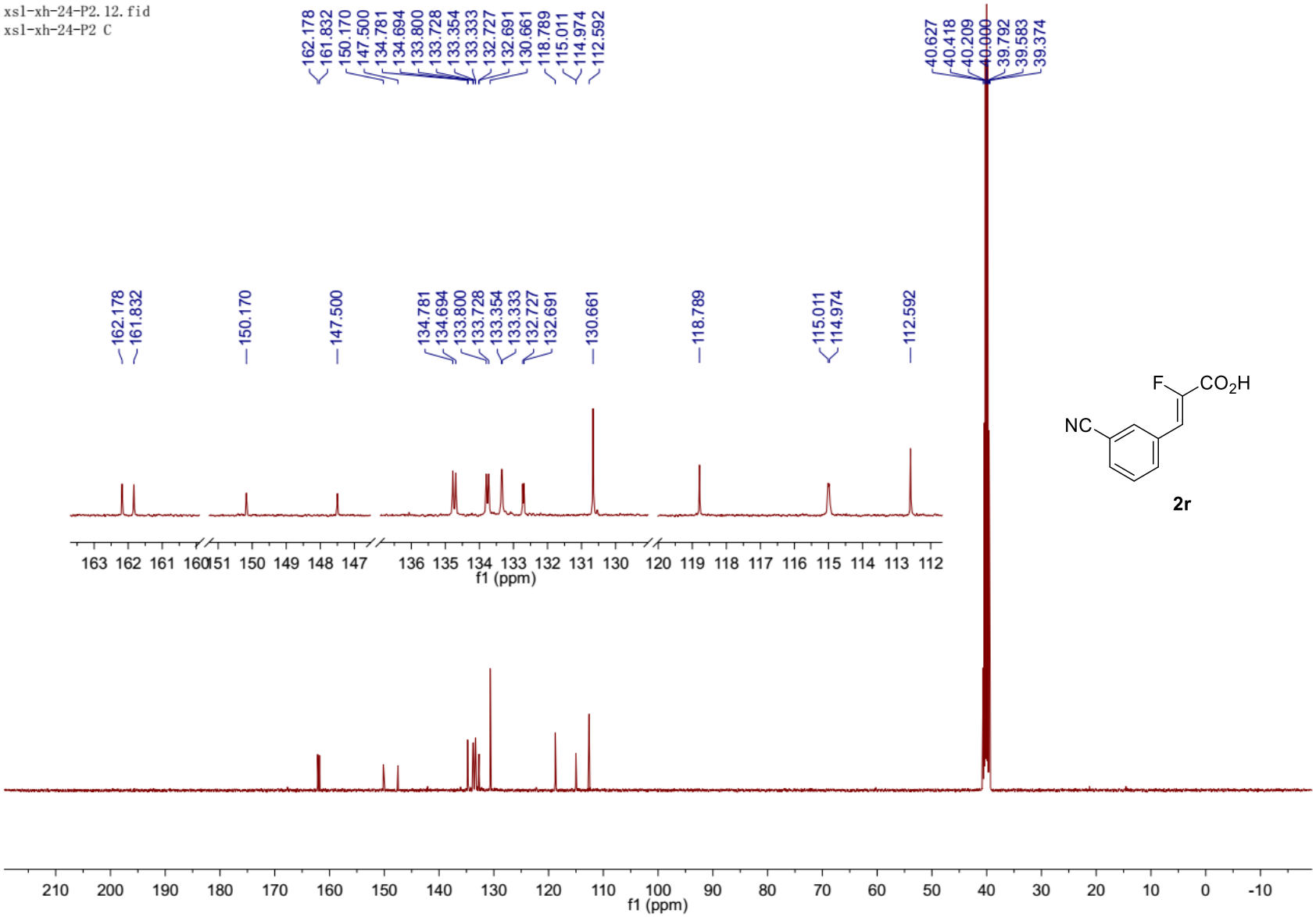

XSL-XH-139-3-CN-500M-1
XSL-XH-139-3-CN-500M-1
8.090
8.061
8.045
7.840
7.825
7.722
7.706
7.690
7.149
7.082
4.591
2.077
2.072
2.068
2.063
2.059


F
CO2H
NC

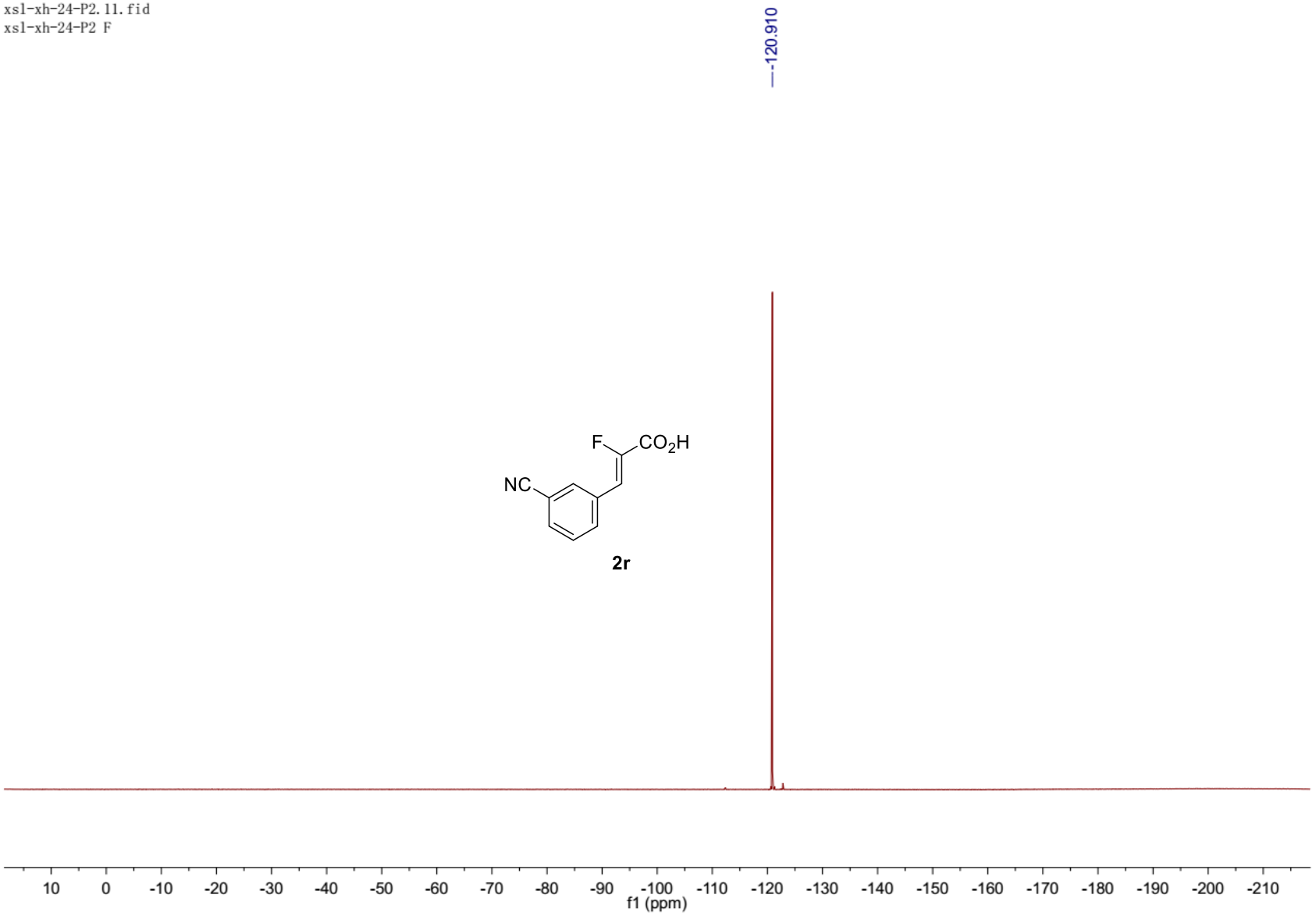
2r

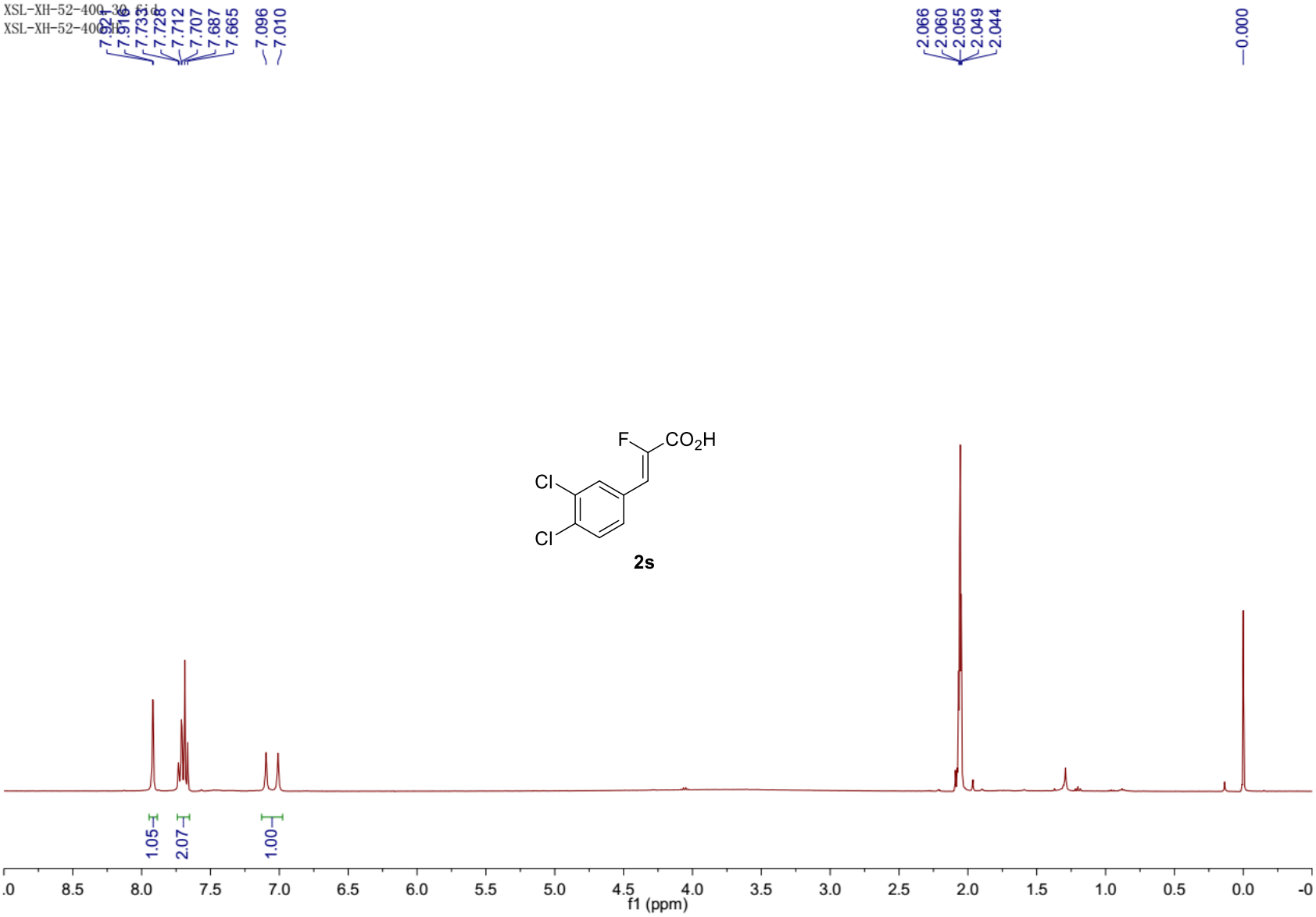

8.090
8.061
8.045
7.840
7.825
7.722
7.706
7.690
0.98
0.97
1.00
1.00
8.15 8.10 8.05 8.00 7.95 7.90 7.85 7.80 7.75 7.70 7.65
f1 (ppm)

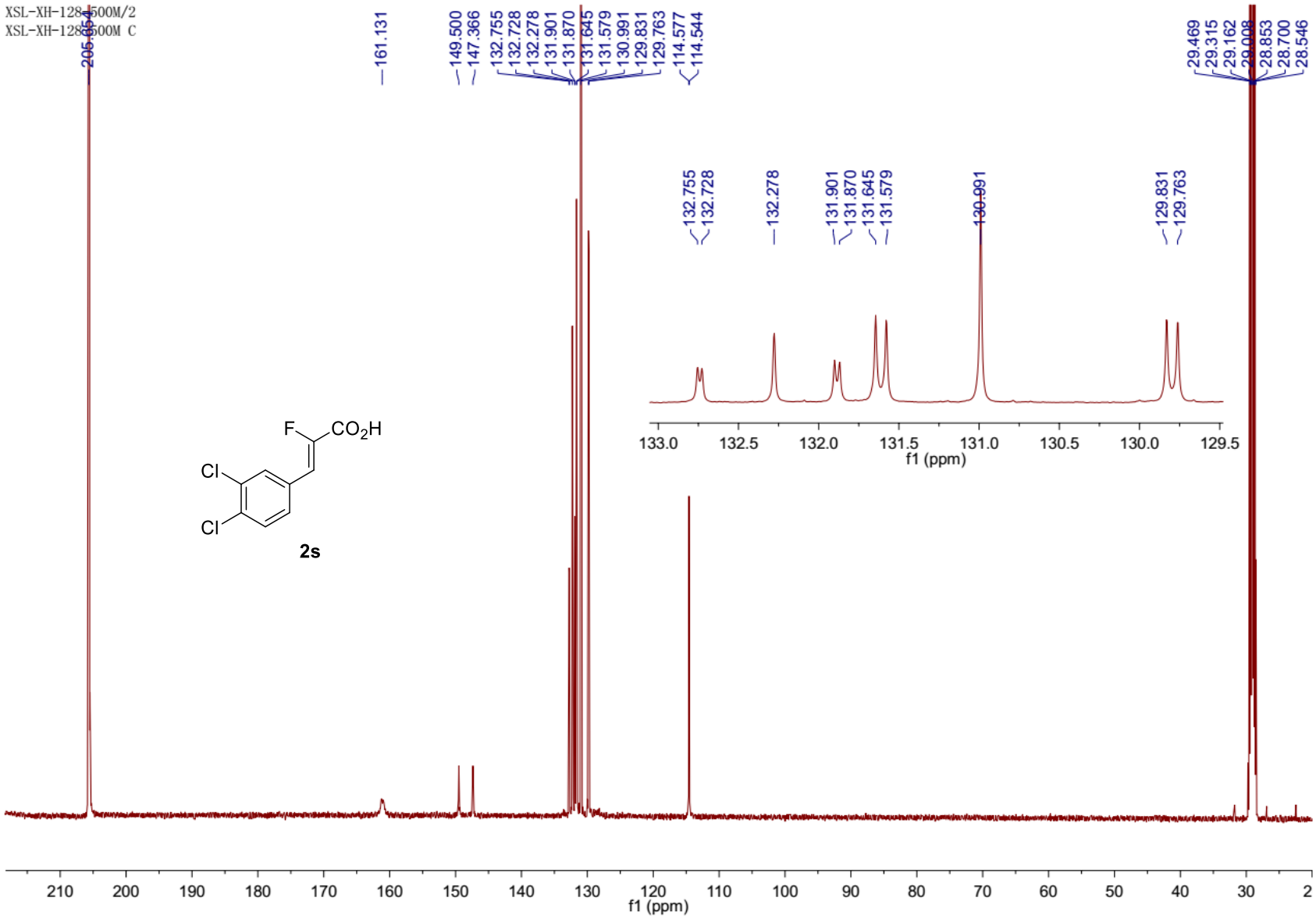

0.98
0.97
1.00
1.00
1.02
.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0
f1 (ppm)


xsl-xh-24-P2.12.fid
xsl-xh-24-P2 C
162.178
161.832
150.170
147.500
134.781
134.694
133.800
133.728
133.354
133.333
132.727
132.691
130.661
118.789
115.011
114.974
112.592
40.627
40.418
40.209
40.000
39.792
39.583
39.374
F
CO2H
NC
2r
f1 (ppm)

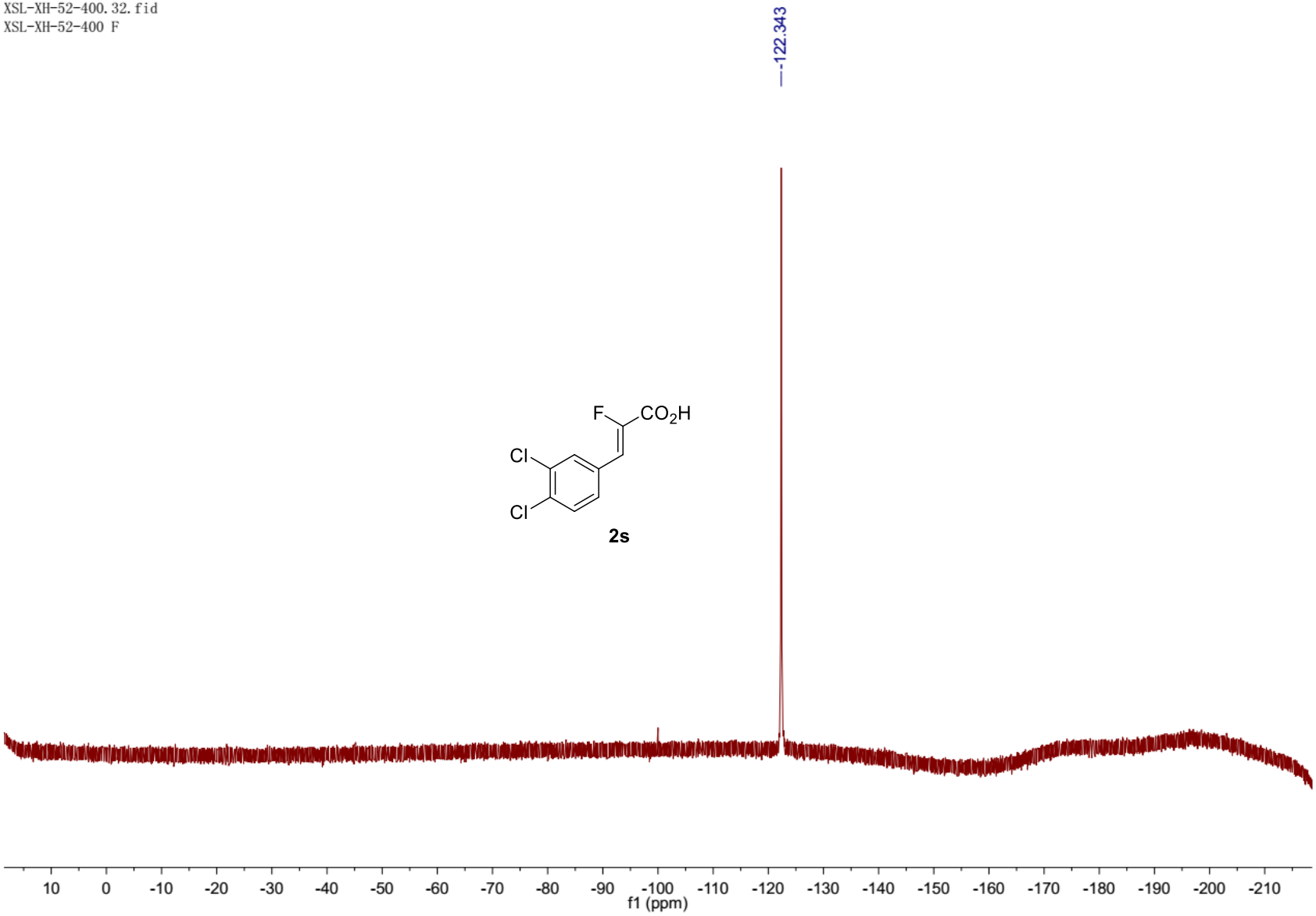
xsl-xh-24-P2.11.fid
xsl-xh-24-P2 F



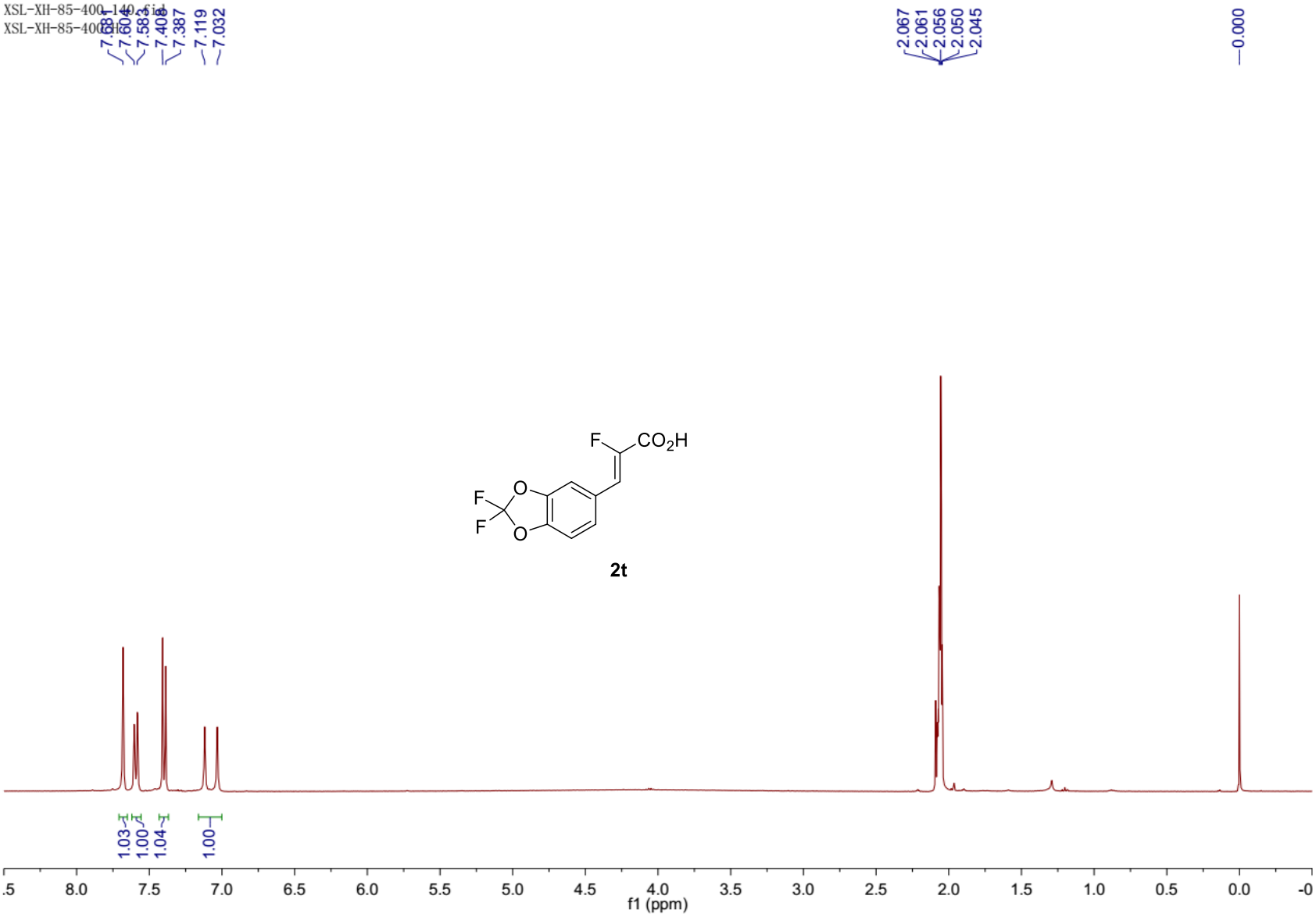

XSL-XH-52-400-30.fid
XSL-XH-52-400-2.fid
7.924
7.916
7.733
7.728
7.712
7.707
7.687
7.665
7.096
7.010
2.066
2.060
2.055
2.049
2.044
0.000
F
CO2H
Cl
Cl
2s
1.05
2.07
1.00
.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)

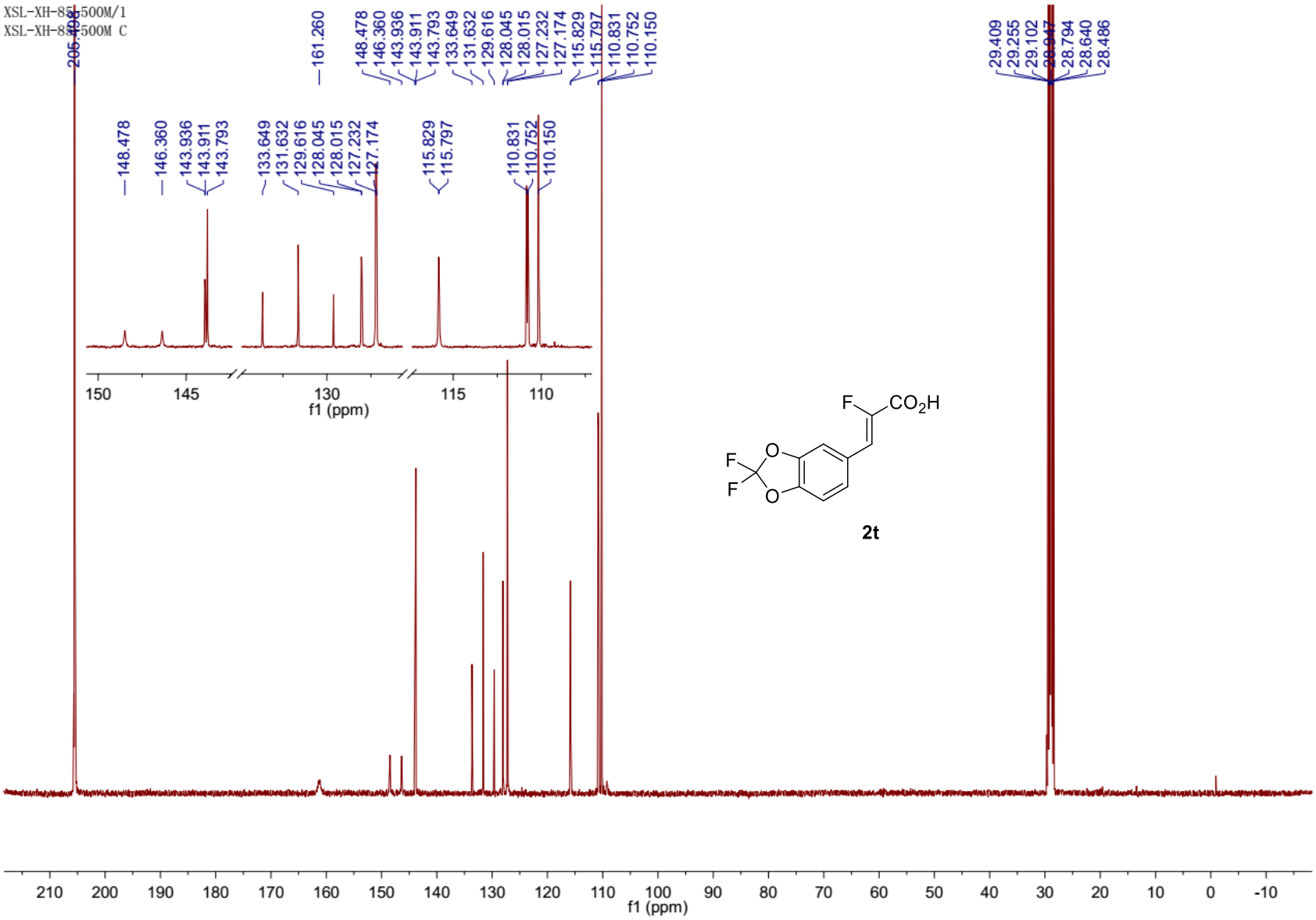

XSL-XH-128-500M/2
XSL-XH-128-500M C
205.603
161.131
149.500
147.366
132.755
132.728
132.278
131.901
131.870
131.645
131.579
130.991
129.831
129.763
114.577
114.544
29.469
29.315
29.162
29.008
28.853
28.700
28.546
F
CO2H
Cl
Cl
2s
f1 (ppm)
f1 (ppm)

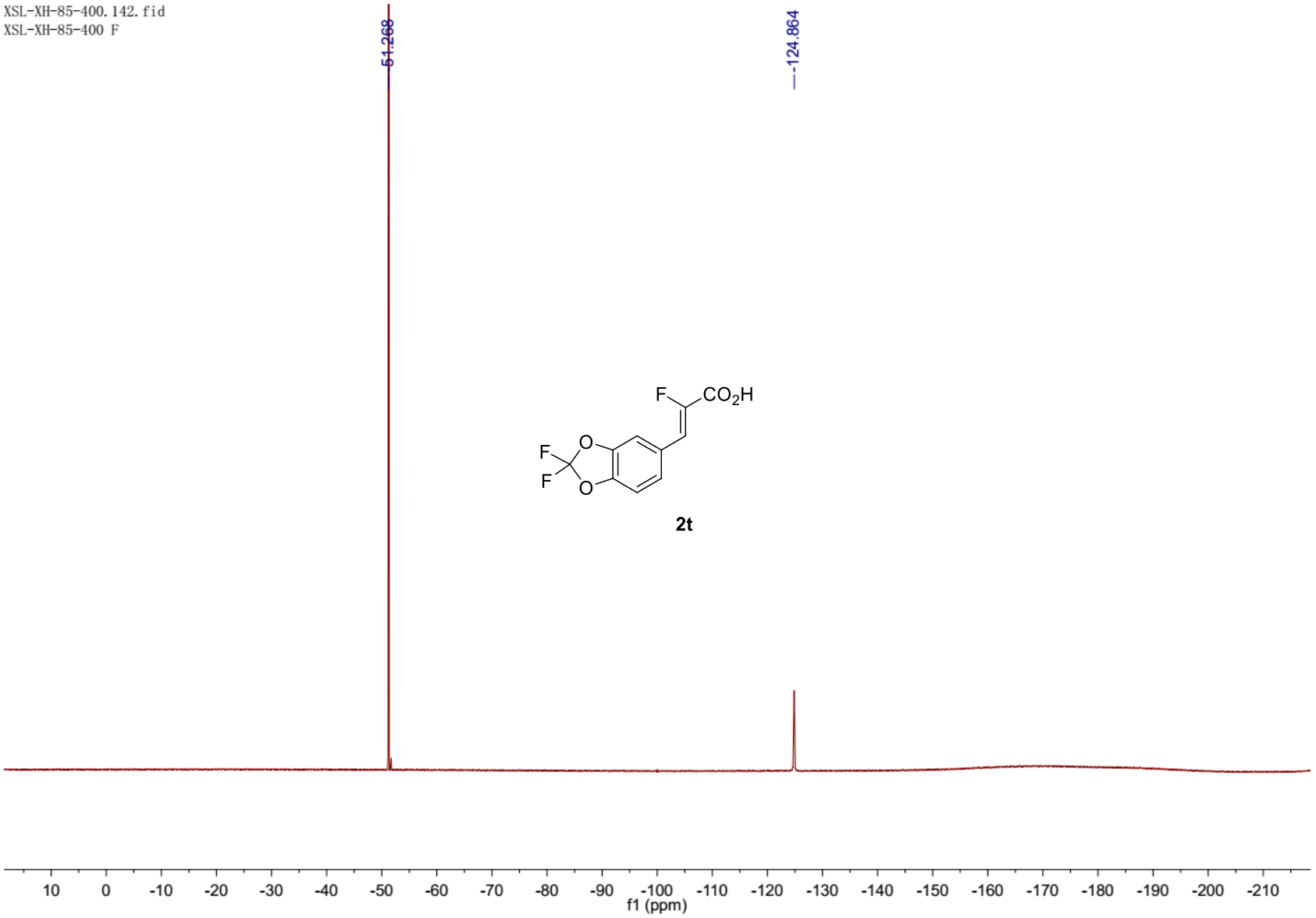
XSL-XH-52-400.32.fid
XSL-XH-52-400 F



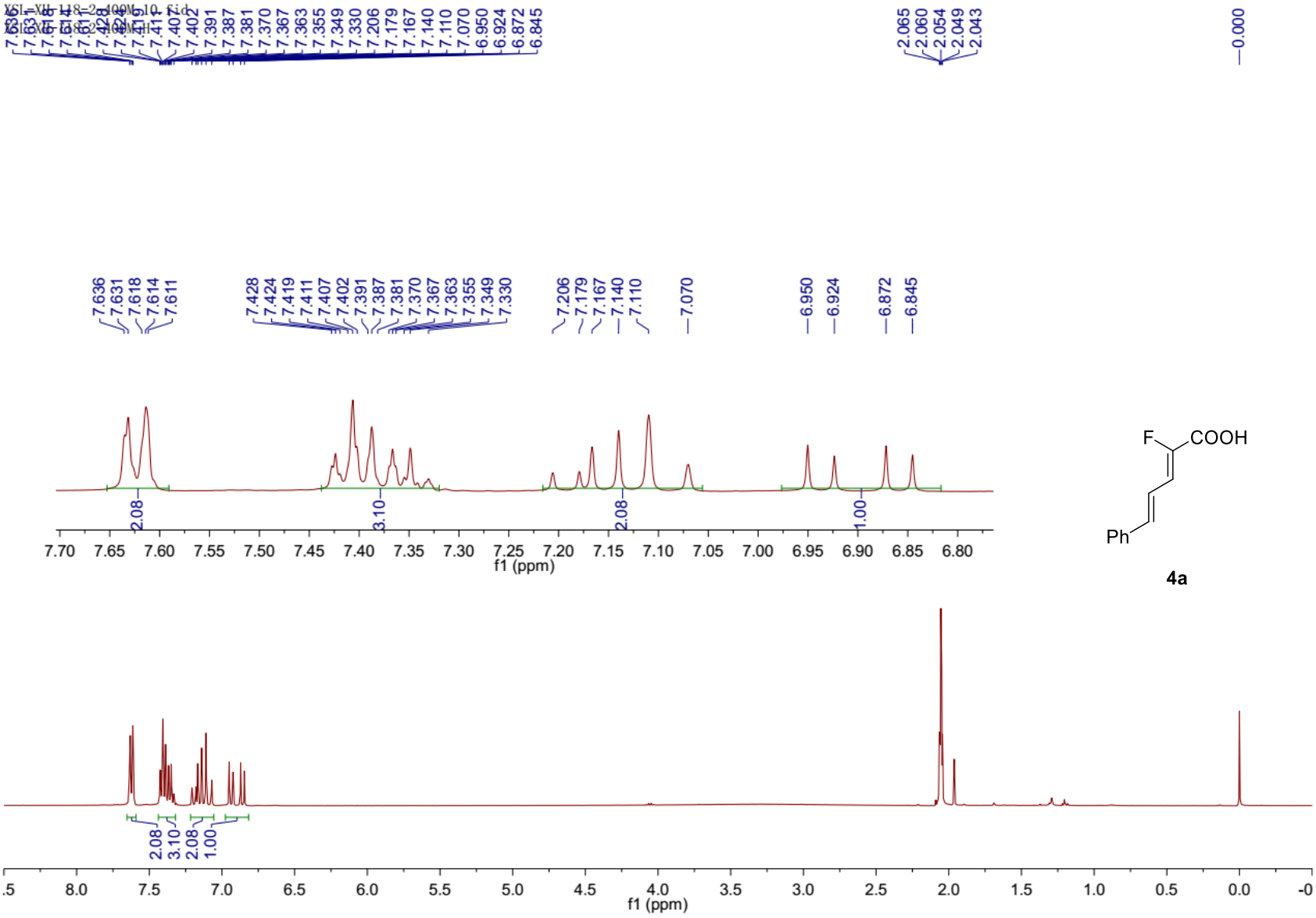
2s

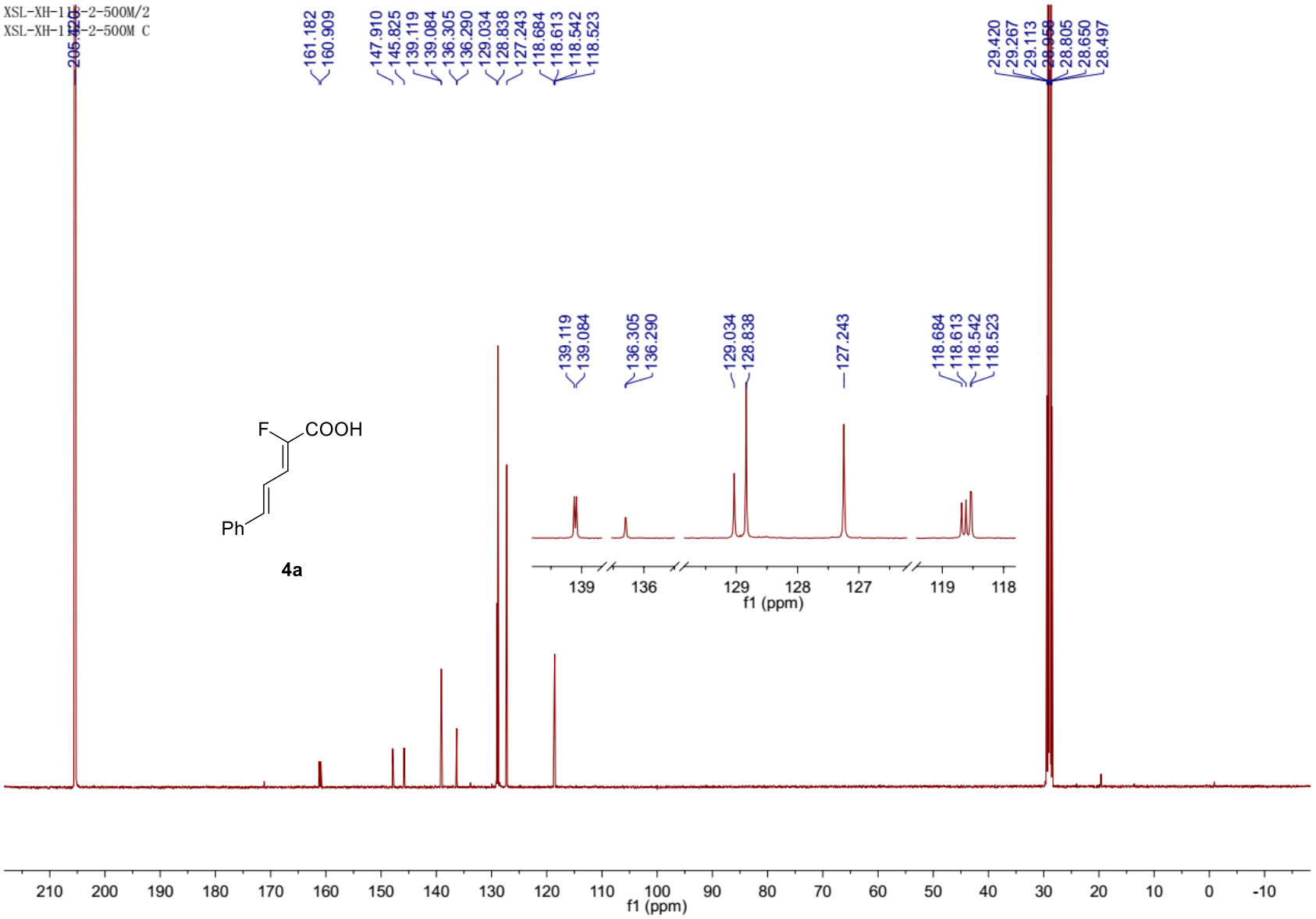

XSL-XH-85-400-140.fid
XSL-XH-85-400
7.623
7.604
7.584
7.408
7.387
7.119
7.032
2.067
2.061
2.056
2.050
2.045
0.000
F
CO2H
O
F
F
O
2t
1.03
1.00
1.04
1.00
f1 (ppm)


XSL-XH-85-500M/1
XSL-XH-85-500M C
205.408
161.260
148.478
146.360
143.936
143.911
143.793
133.649
131.632
129.616
128.045
128.015
127.232
127.174
115.829
115.797
110.831
110.752
110.150
29.409
29.255
29.102
28.947
28.794
28.640
28.486
f1 (ppm)
F
CO2H
O
F
F
O
2t

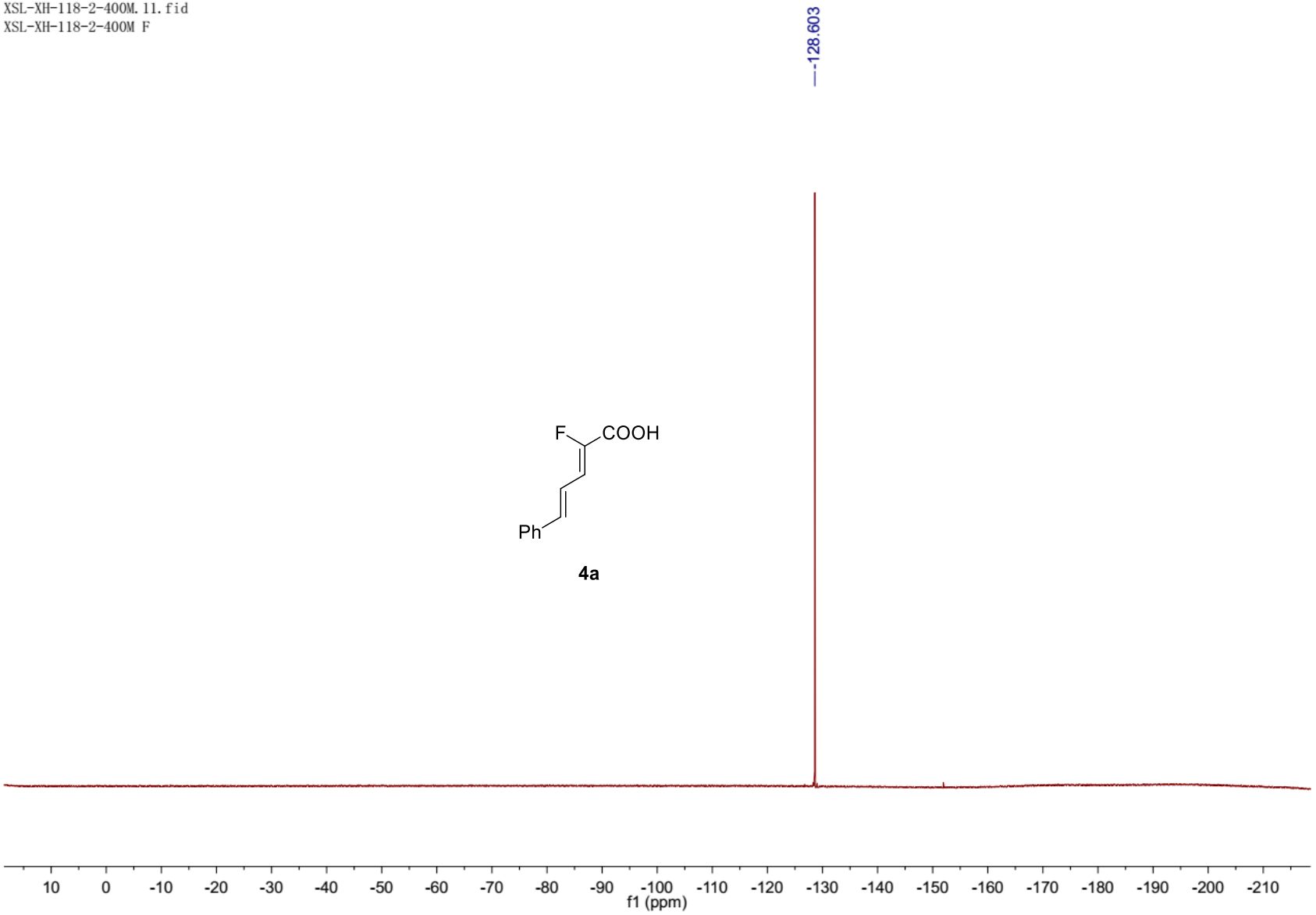

XSL-XH-85-400.142.fid
XSL-XH-85-400 F
-51.268
-124.864
F
CO2H
O
F
F
O
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

2t

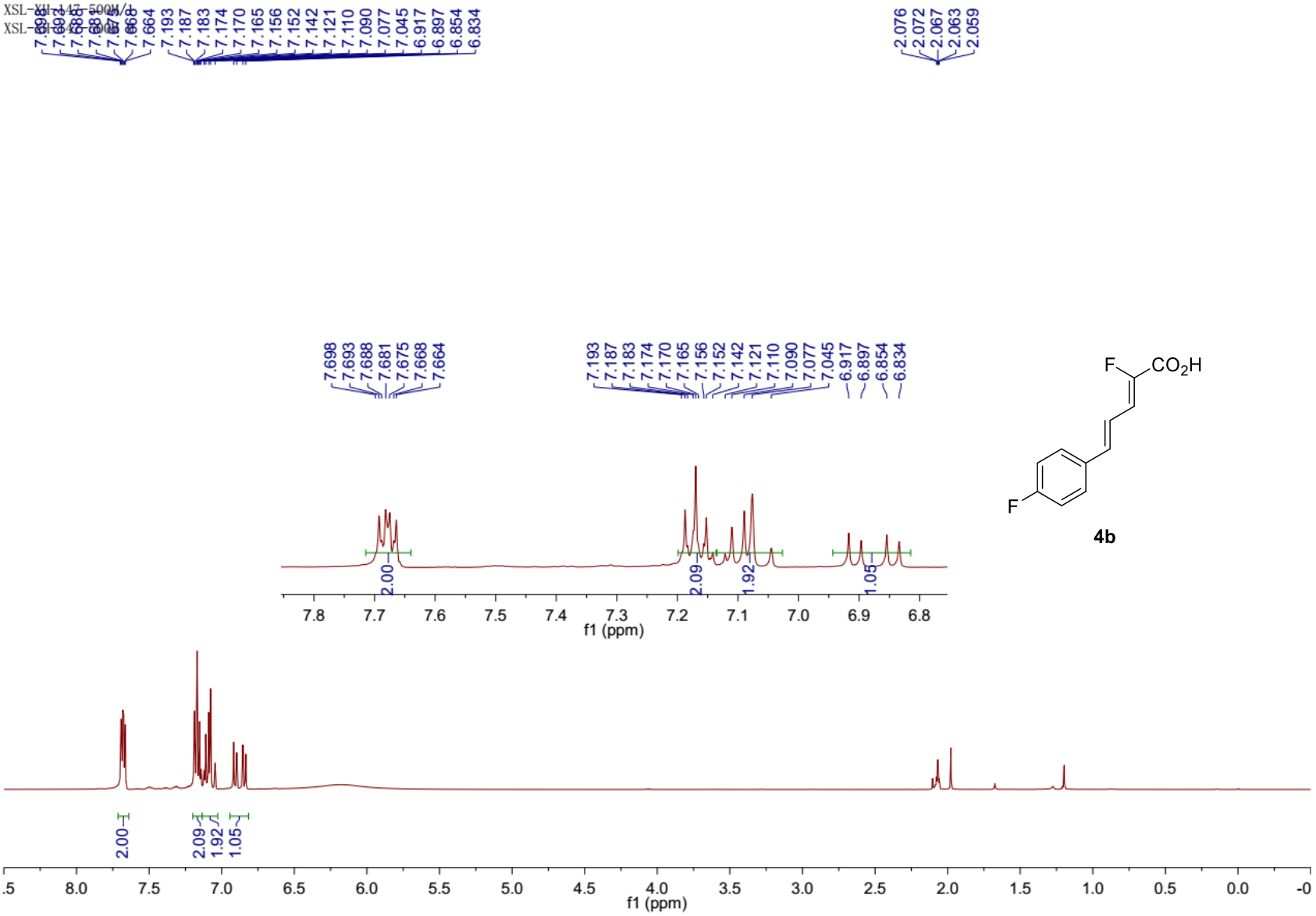

7.636
7.631
7.618
7.614
7.611
7.428
7.424
7.419
7.411
7.407
7.402
7.391
7.387
7.381
7.370
7.367
7.363
7.355
7.349
7.330
7.206
7.179
7.167
7.140
7.110
7.070
6.950
6.924
6.872
6.845
2.065
2.060
2.054
2.049
2.043
0.000
2.08
3.10
2.08
1.00
f1 (ppm)
F
COOH
Ph
4a

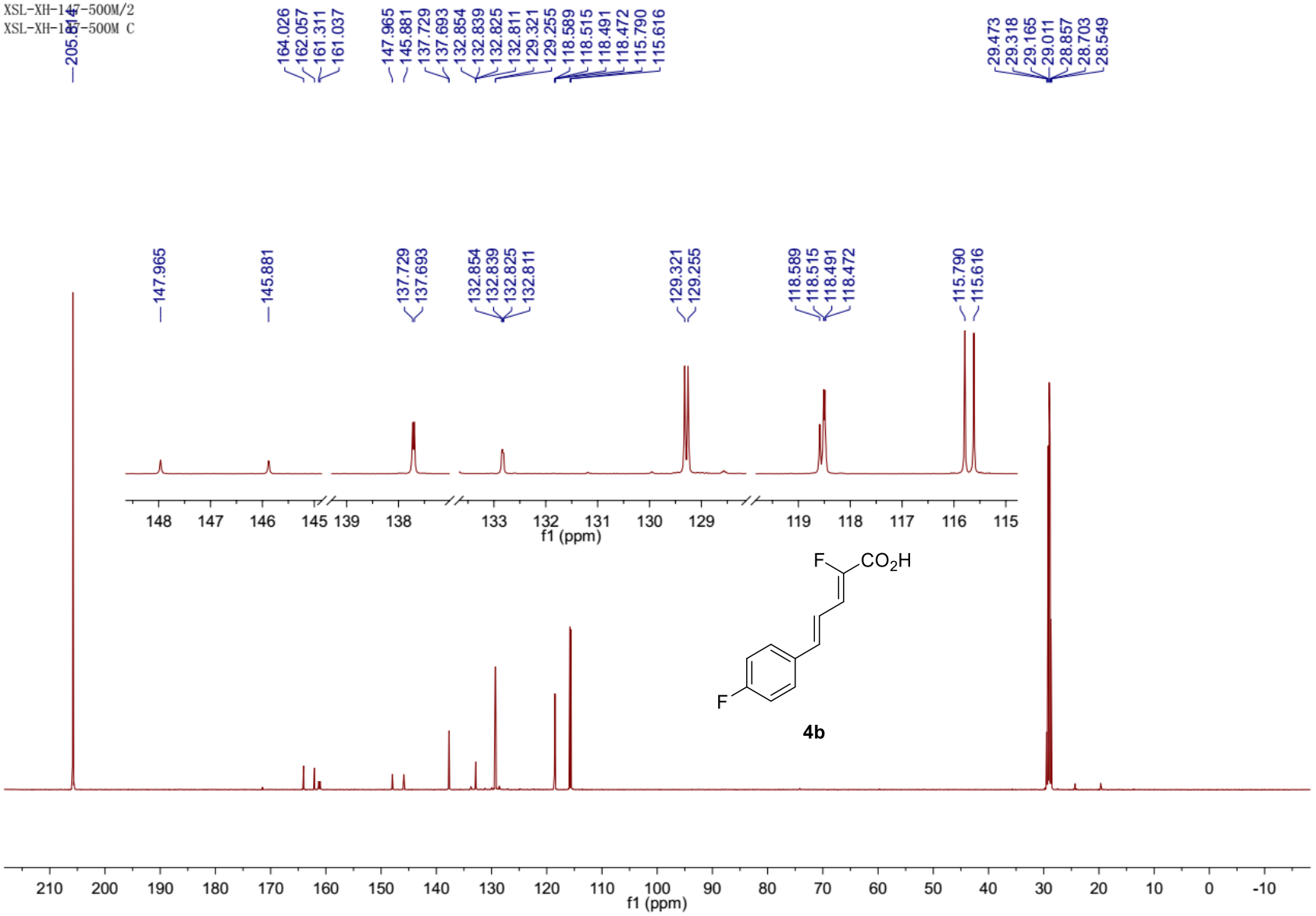

XSL-XH-1
-2-500M/2
XSL-XH-1
-2-500M C
161.182
160.909
147.910
145.825
139.119
139.084
136.305
136.290
129.034
128.838
127.243
118.684
118.613
118.542
118.523
29.420
29.267
29.113
28.805
28.650
28.497
F
COOH
Ph
4a
f1 (ppm)
f1 (ppm)

XSL-XH-118-2-400M.11.fid
XSL-XH-118-2-400M F

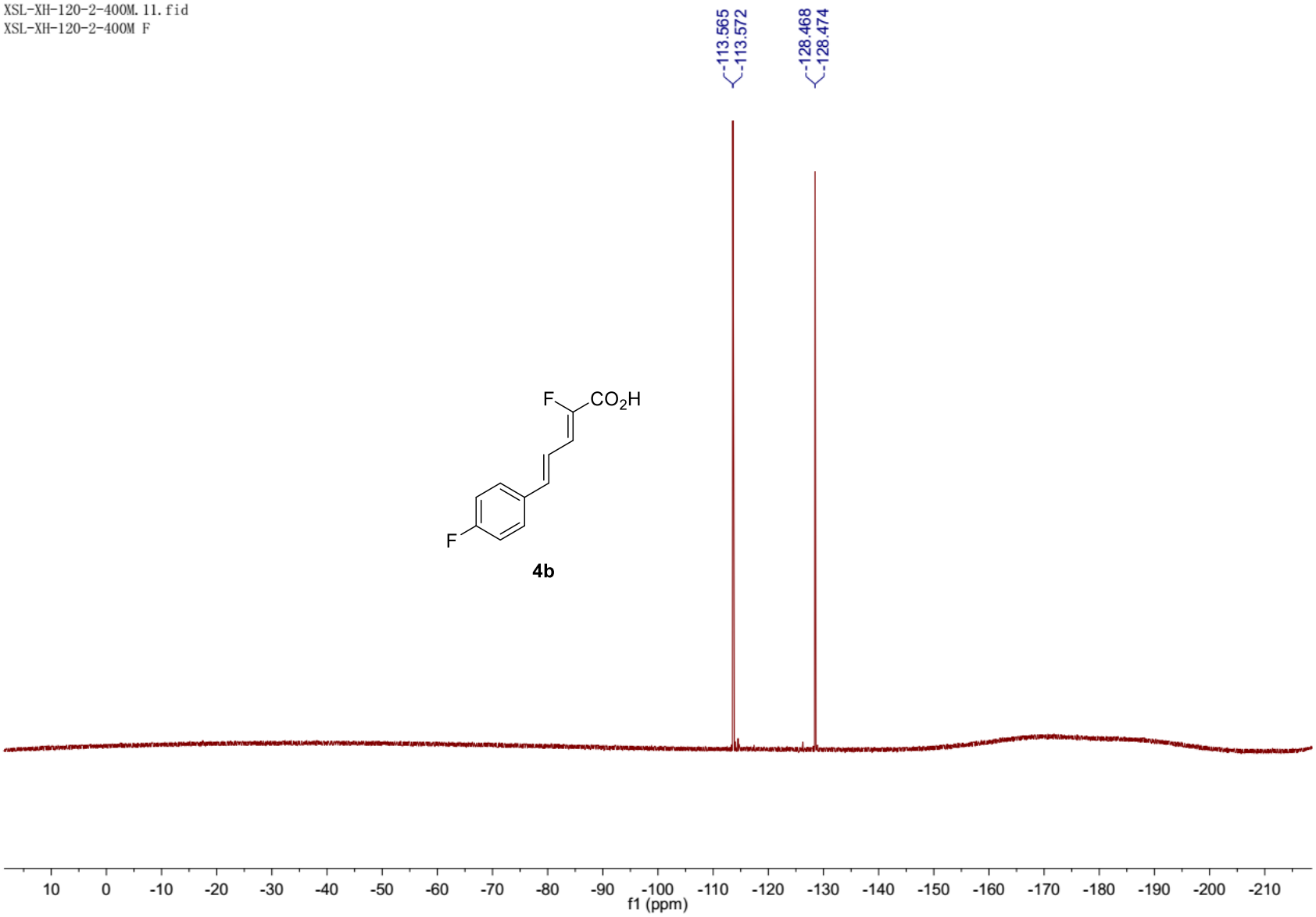


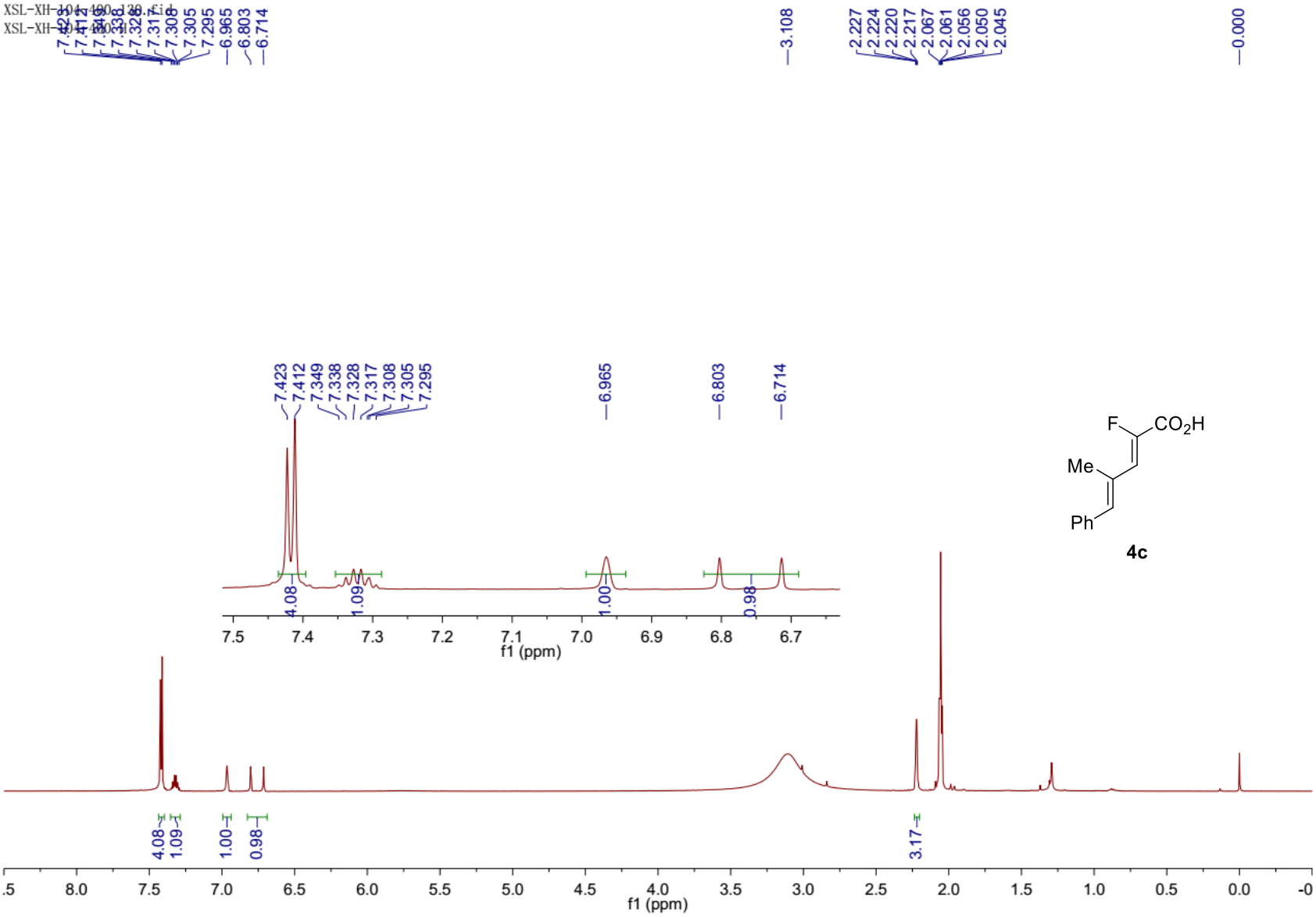
4a

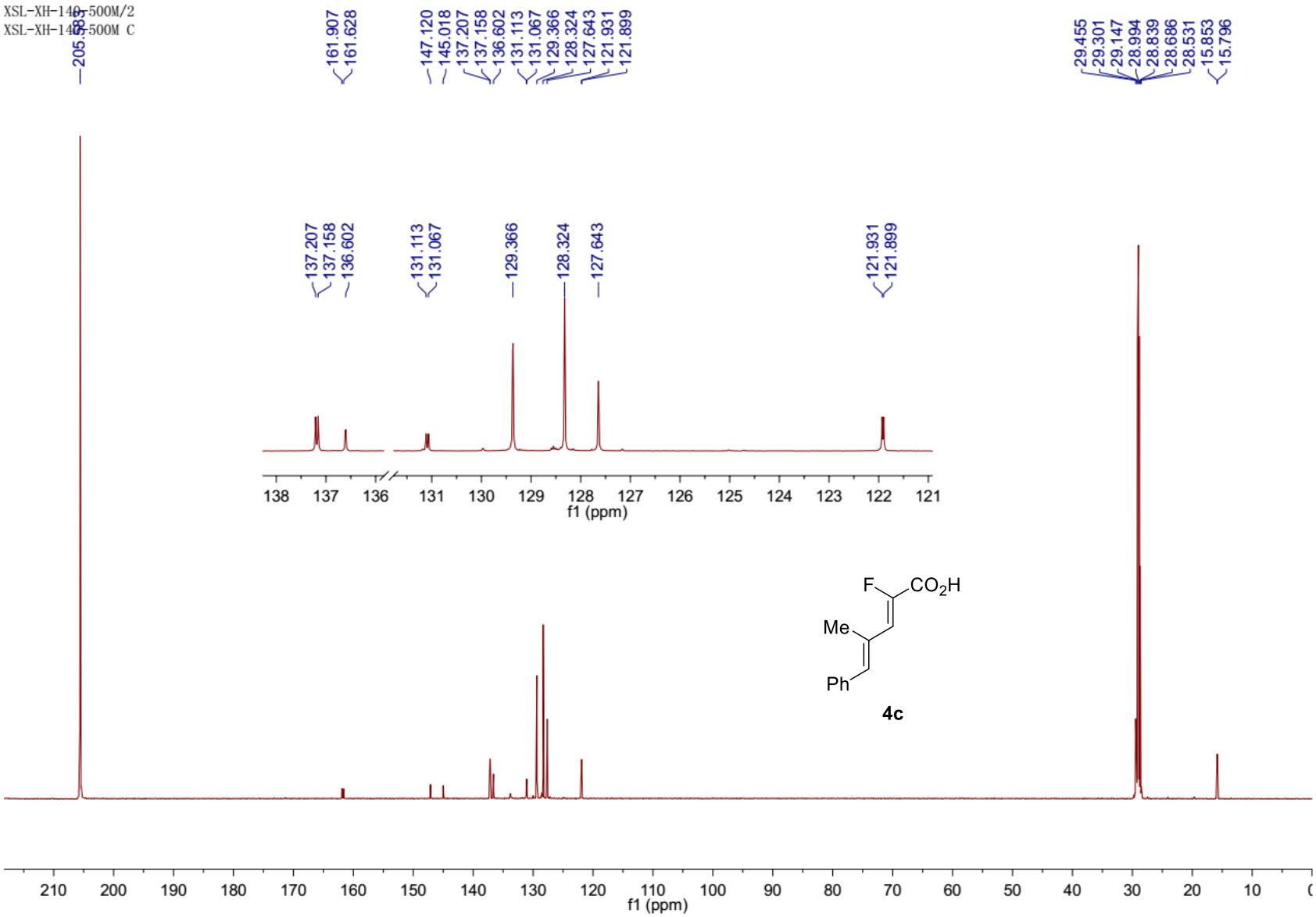

XSL-XU-147-500M-1
7.698
7.693
7.688
7.681
7.675
7.668
7.664
7.193
7.187
7.183
7.174
7.170
7.165
7.156
7.152
7.142
7.121
7.110
7.090
7.077
7.045
6.917
6.897
6.854
6.834
2.076
2.072
2.067
2.063
2.059
F
CO2H
F
4b
2.00
2.09
1.92
1.05
f1 (ppm)


XSL-XH-147-500M/2
XSL-XH-147-500M C
205.814
164.026
162.057
161.311
161.037
147.965
145.881
137.729
137.693
132.854
132.839
132.825
132.811
129.321
129.255
118.589
118.515
118.491
118.472
115.790
115.616
29.473
29.318
29.165
29.011
28.857
28.703
28.549
148 147 146 145 139 138 133 132 131 130 129 119 118 117 116 115
f1 (ppm)
F
CO2H
F
4b
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

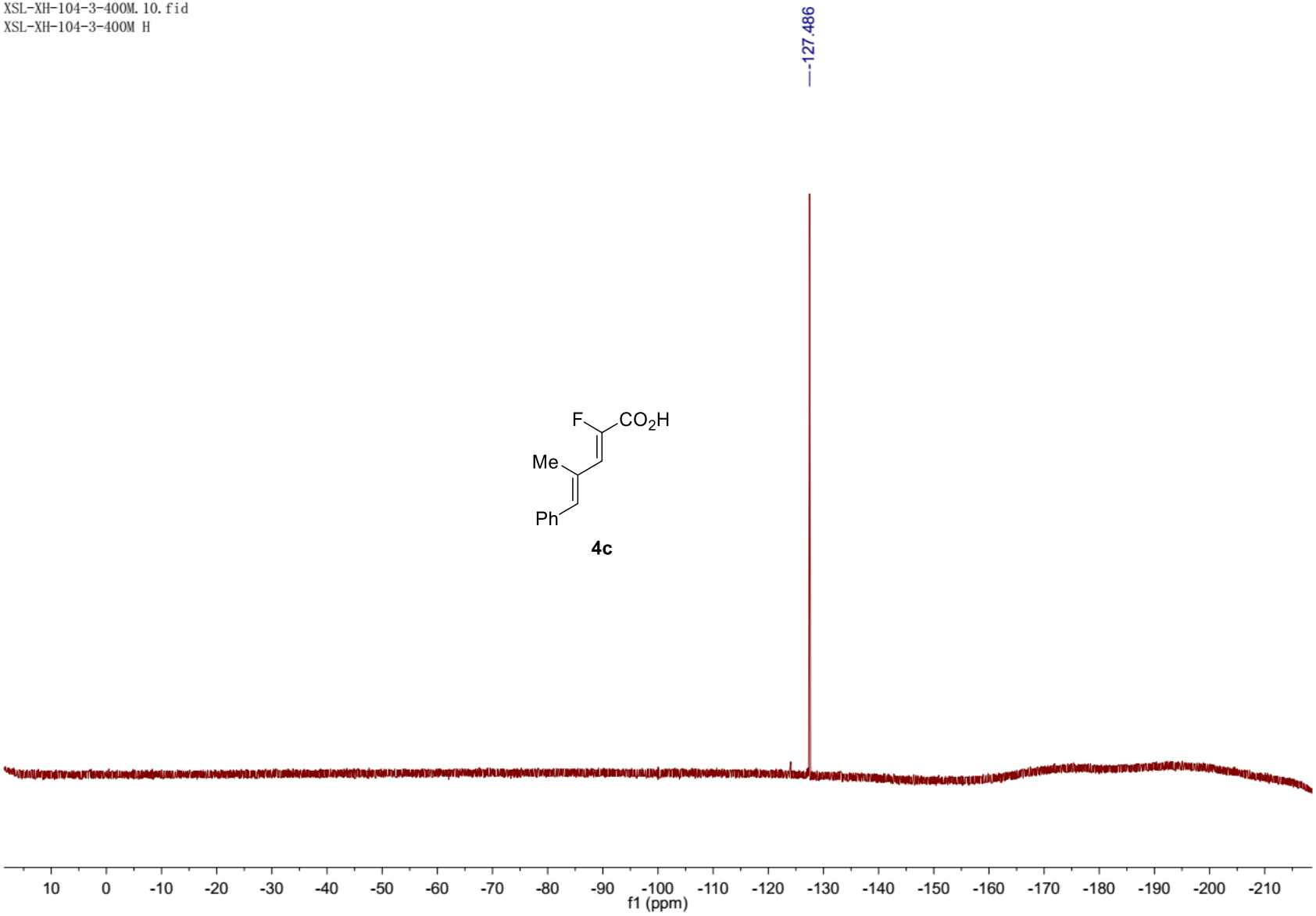
XSL-XH-120-2-400M.11.fid
XSL-XH-120-2-400M F



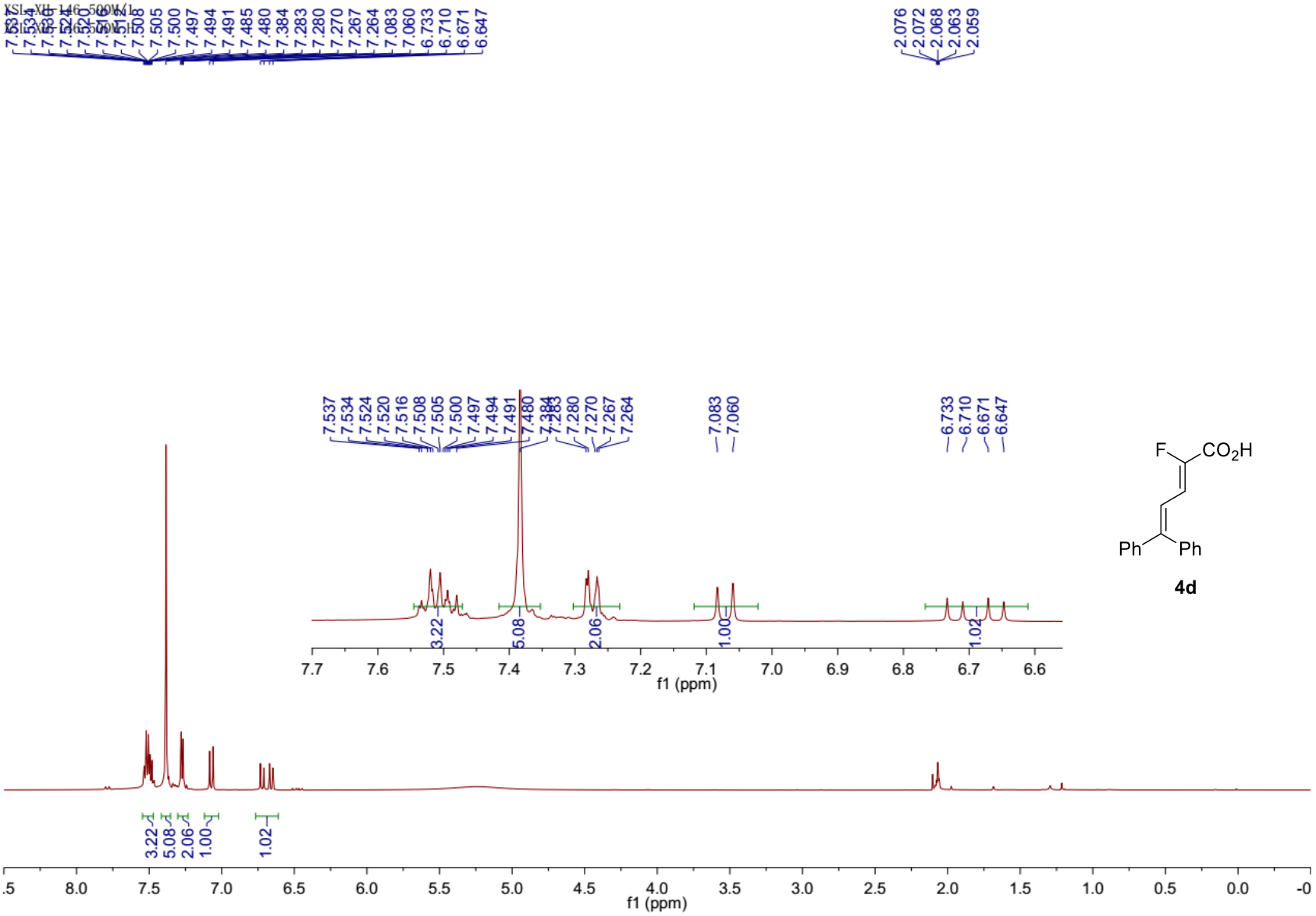

7.423
7.412
7.349
7.338
7.328
7.317
7.308
7.305
7.295
6.965
6.803
6.714
3.108
2.227
2.224
2.220
2.217
2.067
2.061
2.056
2.050
2.045
0.000
4.08
1.09
1.00
0.98
3.17
f1 (ppm)
F
CO2H
Me
Ph
4c

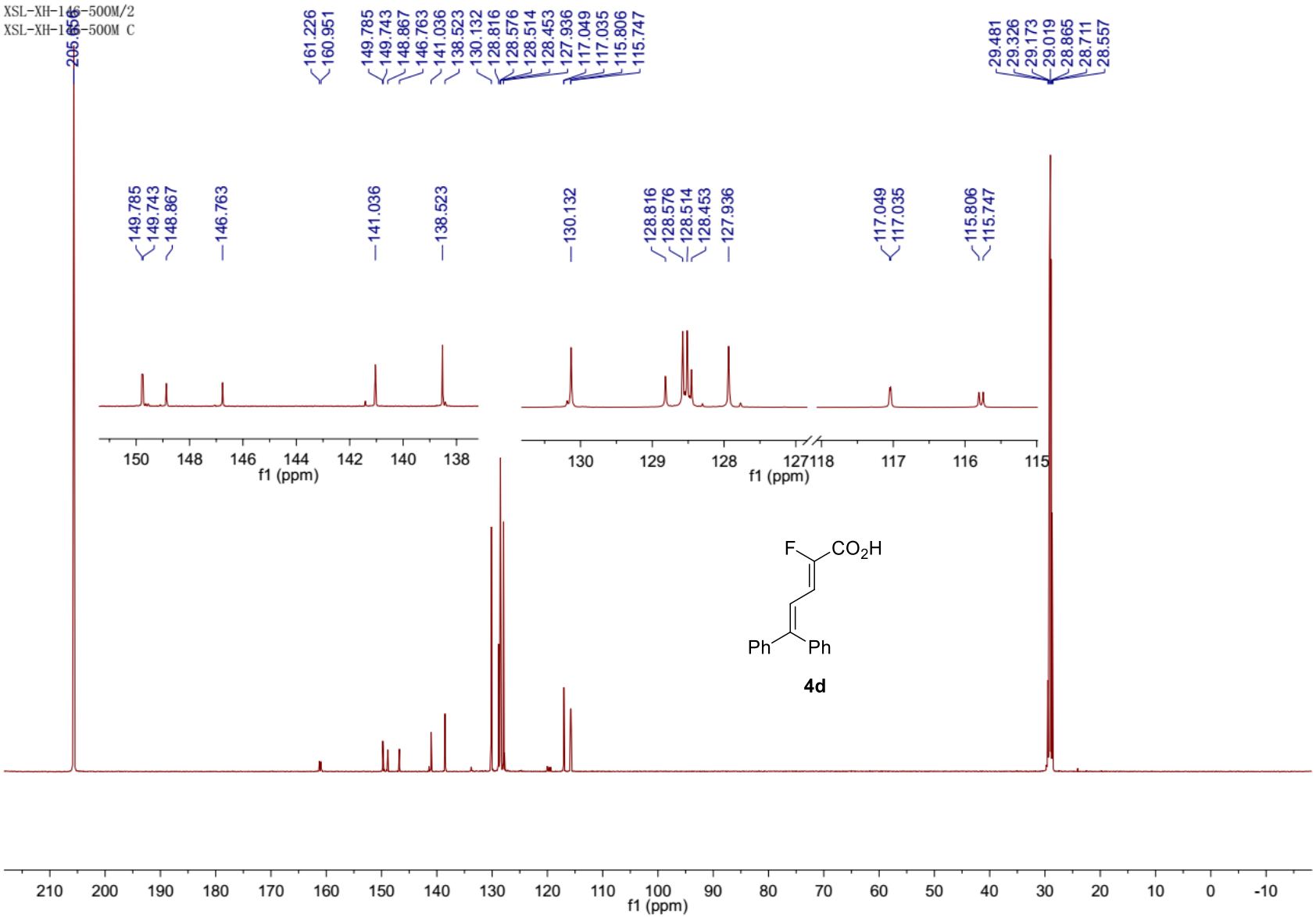

XSL-XH-140-500M/2
XSL-XH-140-500M C
205.583
161.907
161.628
147.120
145.018
137.207
137.158
136.602
131.113
131.067
129.366
128.324
127.643
121.931
121.899
29.455
29.301
29.147
28.994
28.839
28.686
28.531
15.853
15.796
f1 (ppm)
F
CO2H
Me
Ph
4c

XSL-XH-104-3-400M.10.fid
XSL-XH-104-3-400M H

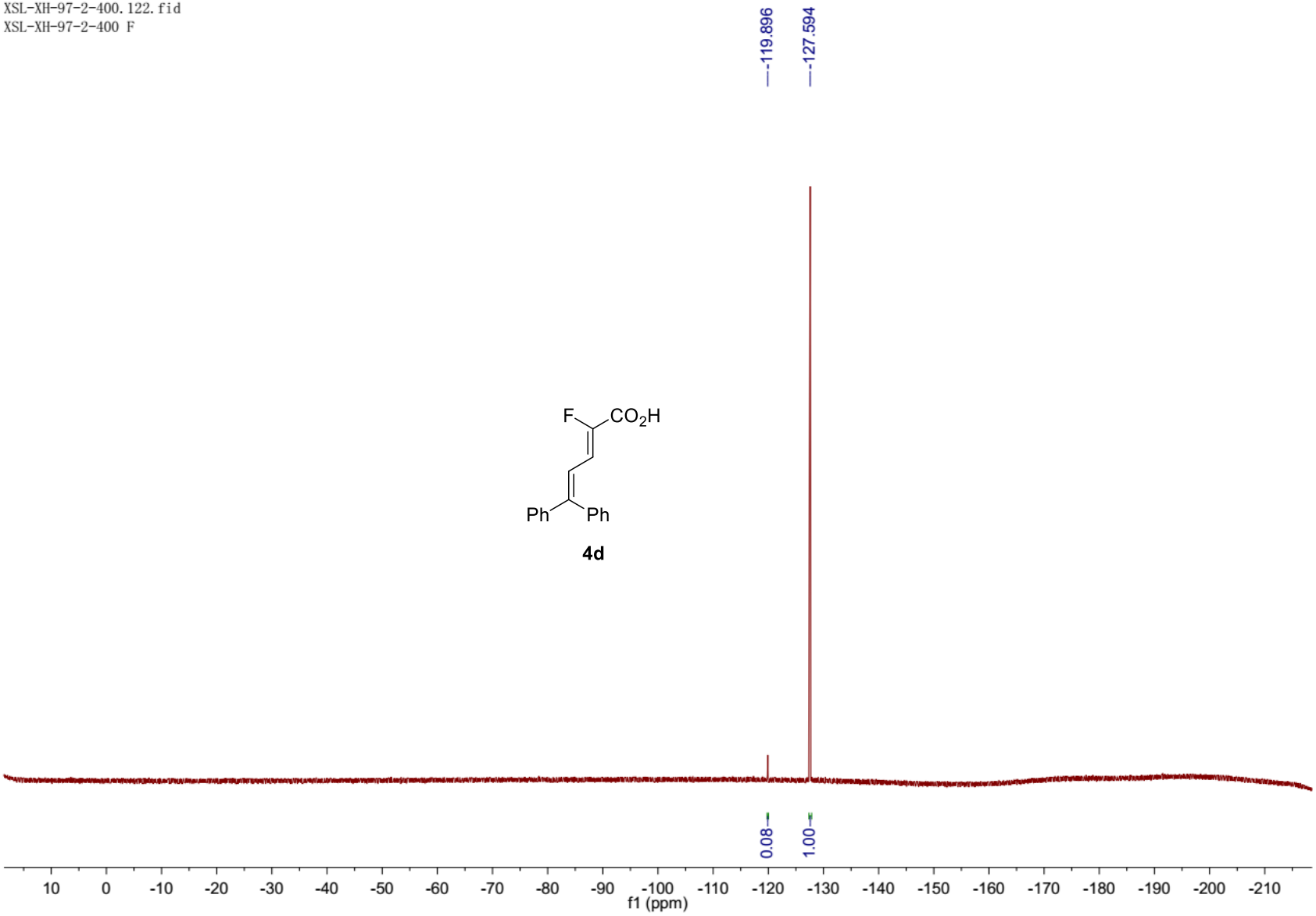


4c

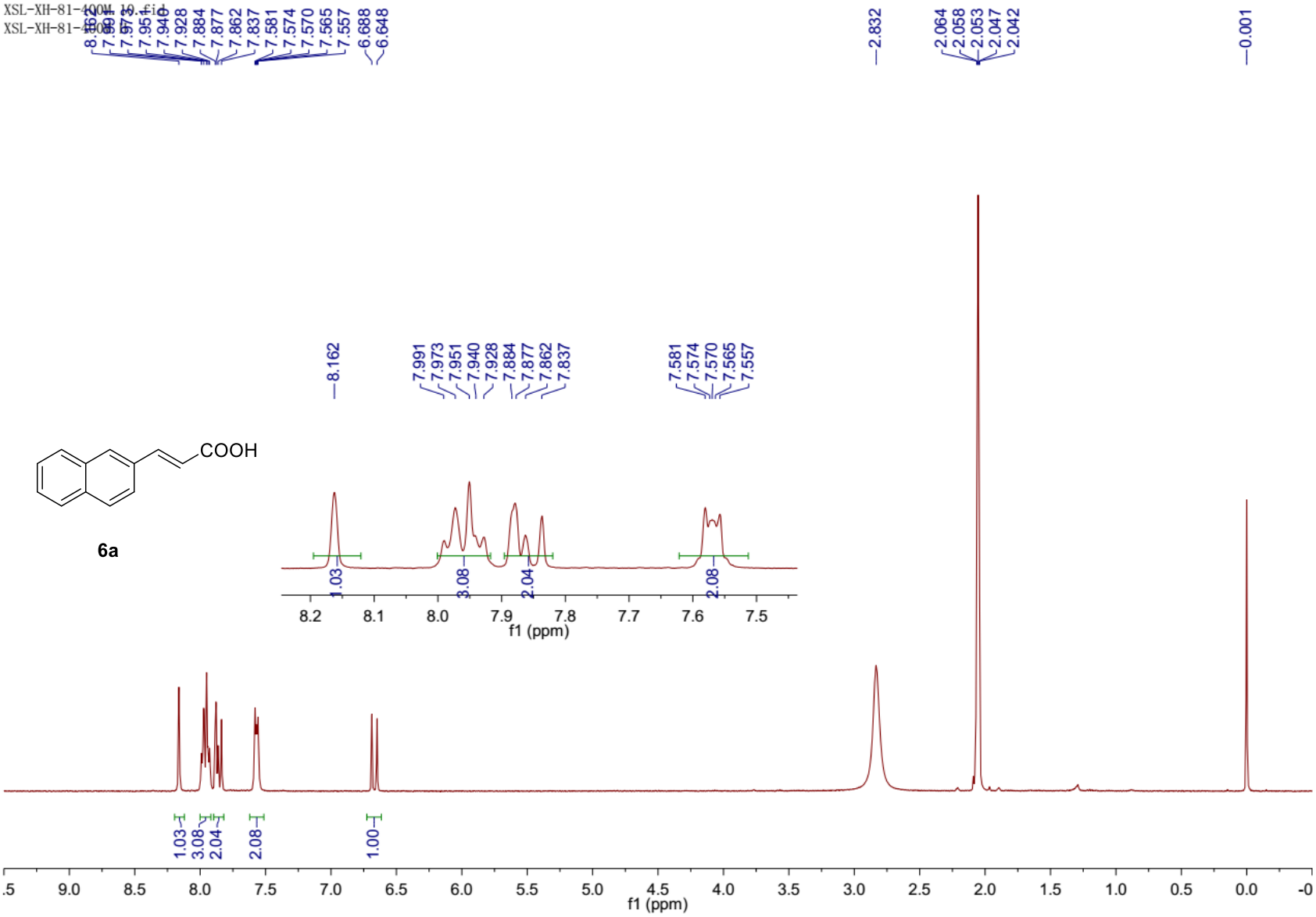

7.537
7.534
7.524
7.520
7.516
7.508
7.505
7.500
7.497
7.494
7.491
7.480
7.384
7.283
7.280
7.270
7.267
7.264
7.083
7.060
6.733
6.710
6.671
6.647
2.076
2.072
2.068
2.063
2.059
3.22
5.08
2.06
1.00
1.02
f1 (ppm)
F
CO2H
Ph
Ph
4d

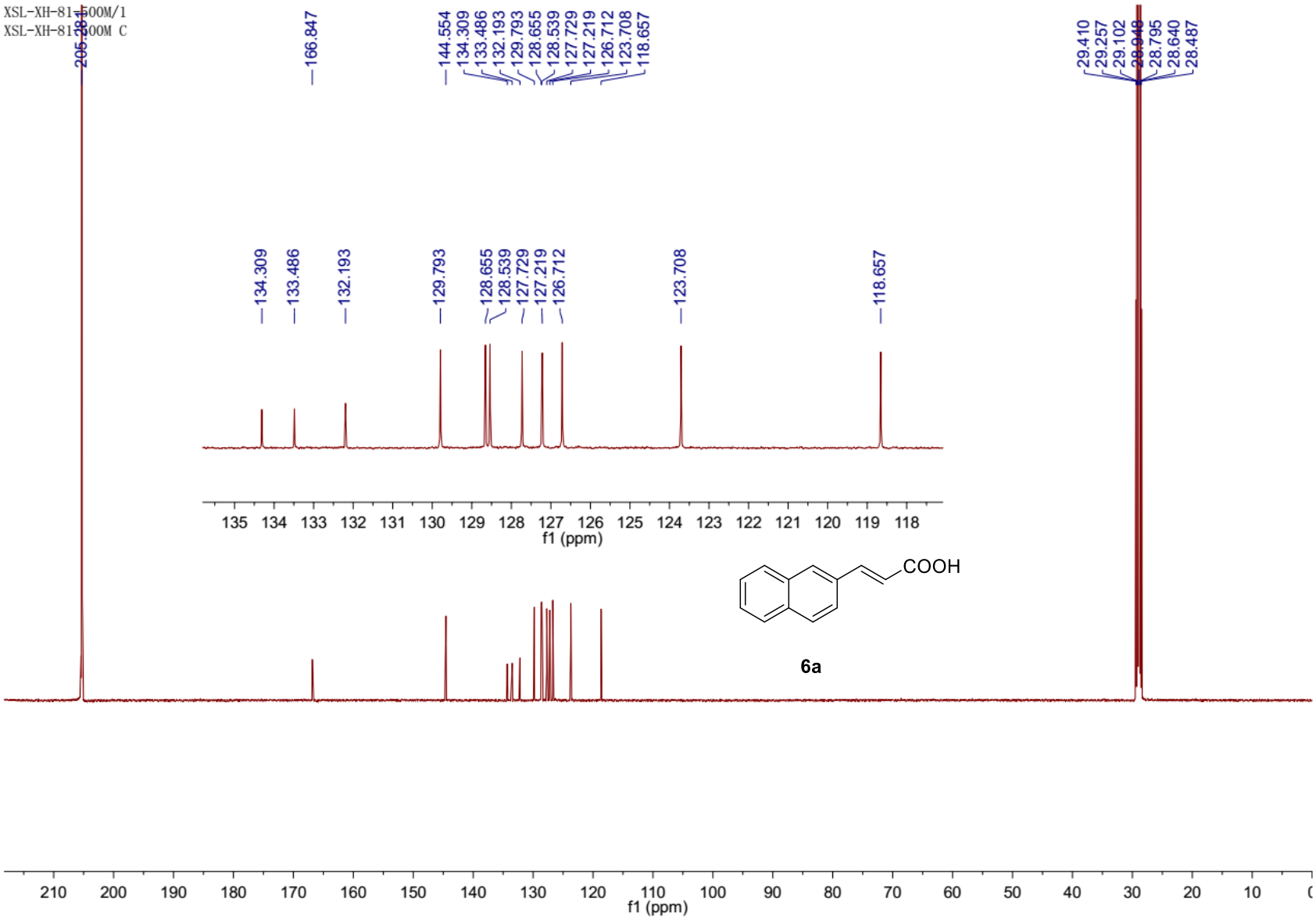

XSL-XH-146-500M/2
XSL-XH-146-500M C
205.650
161.226
160.951
149.785
149.743
148.867
146.763
141.036
138.523
130.132
128.816
128.576
128.514
128.453
127.936
117.049
117.035
115.806
115.747
29.481
29.326
29.173
29.019
28.865
28.711
28.557
f1 (ppm)
F
CO2H
Ph
Ph
4d

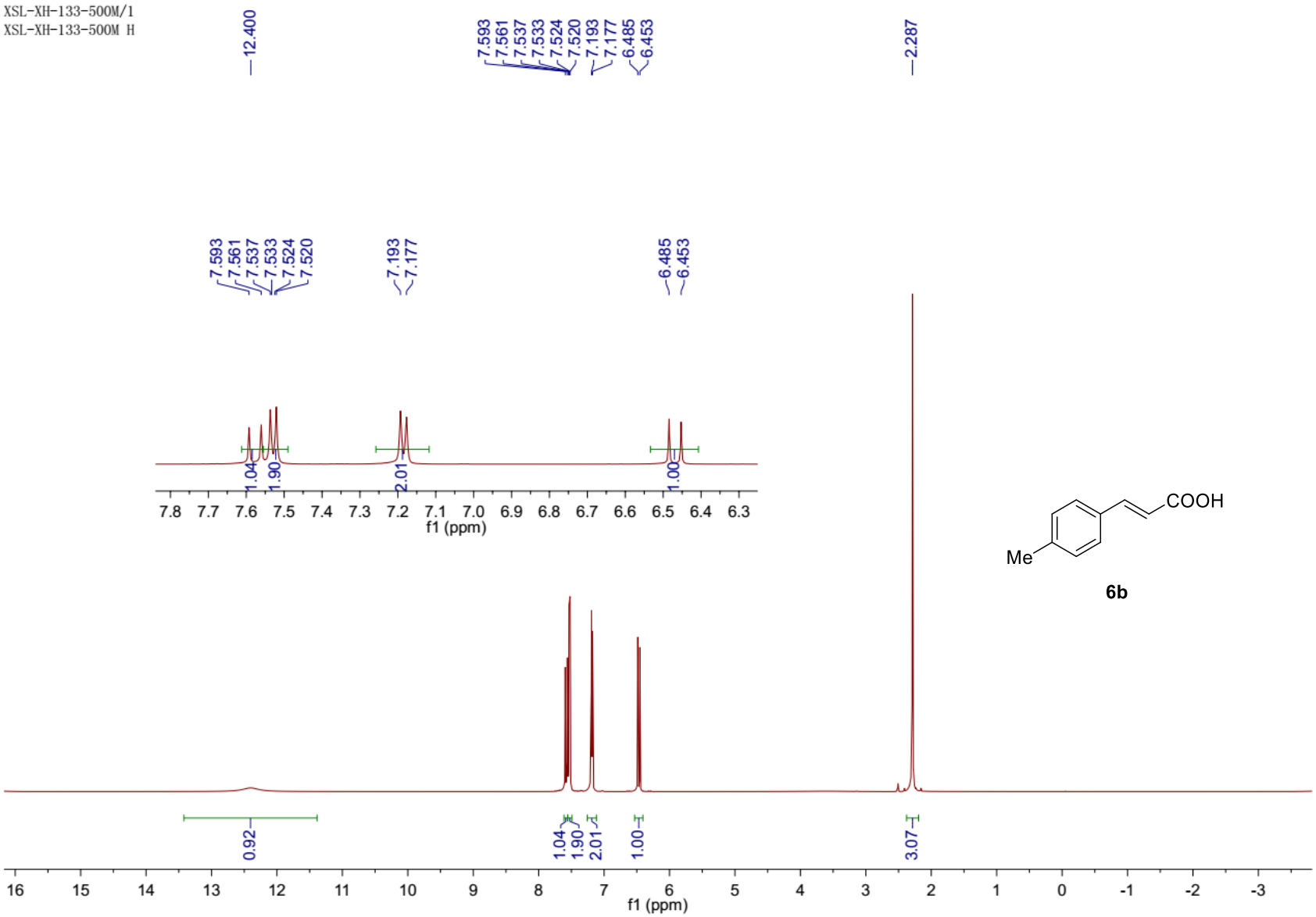
XSL-XH-97-2-400.122.fid
XSL-XH-97-2-400 F



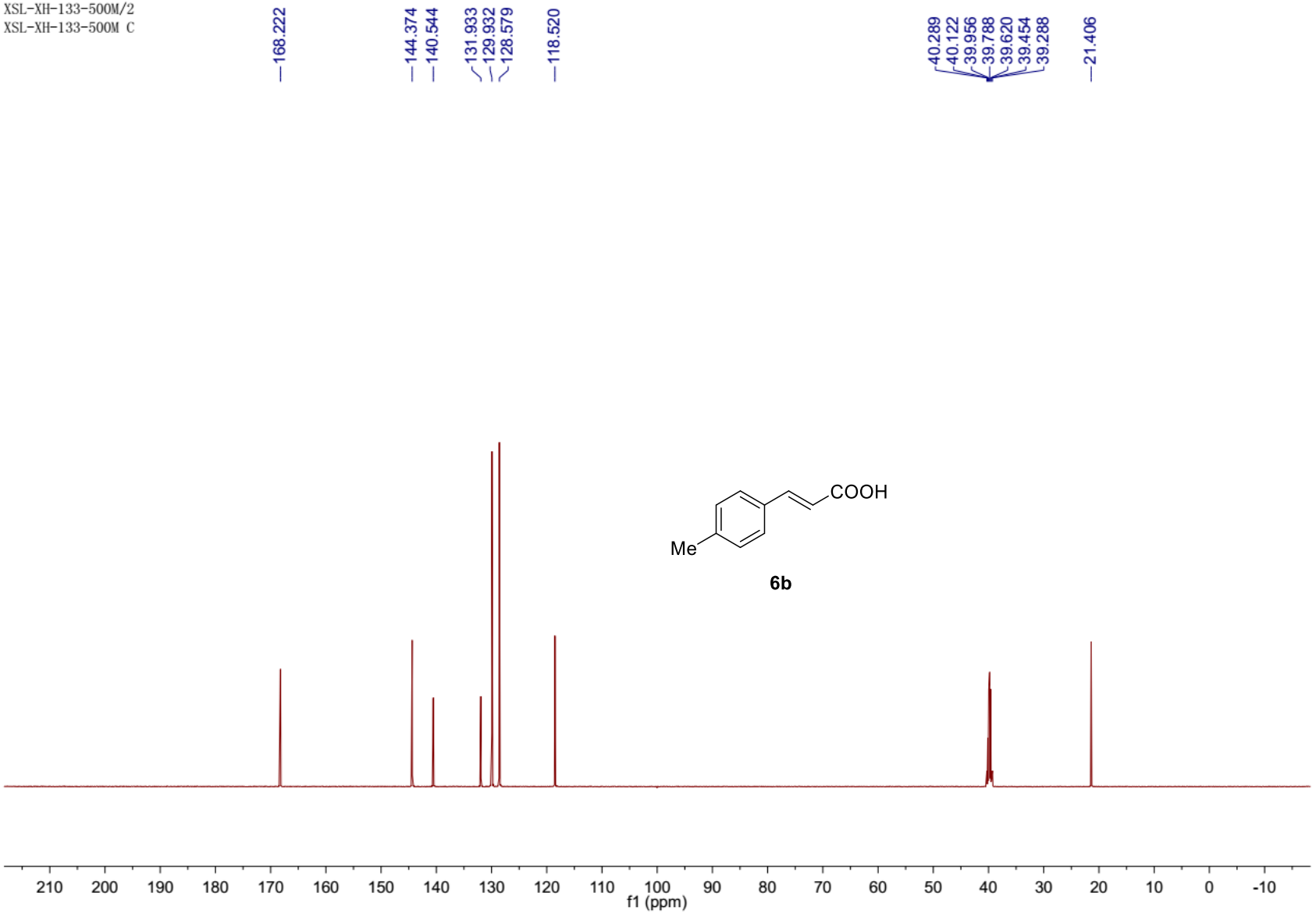
4d

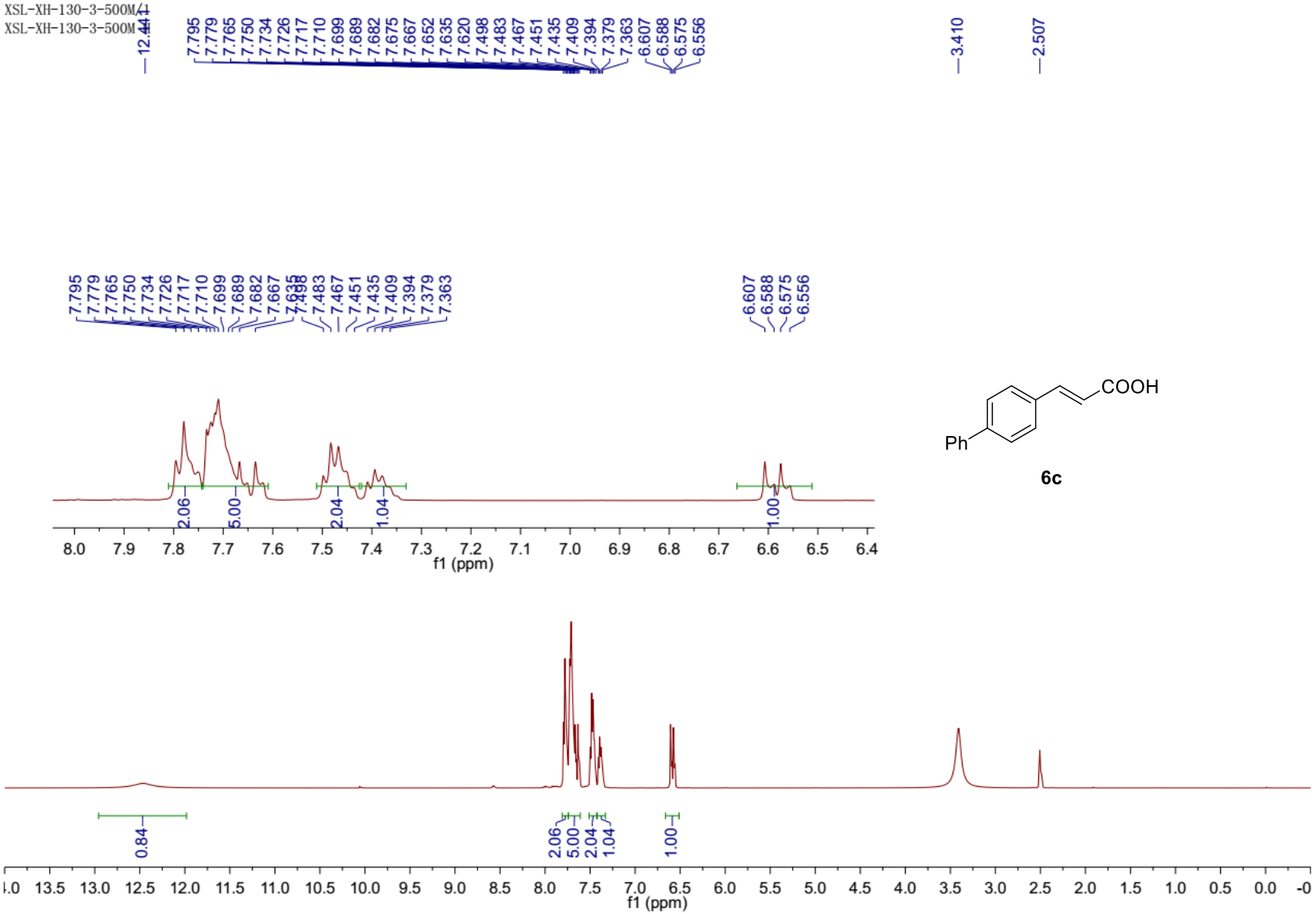

XSL-XH-81-400M-10.fid
8.162
7.991
7.973
7.951
7.940
7.928
7.884
7.877
7.862
7.837
7.581
7.574
7.570
7.565
7.557
6.688
6.648
2.832
2.064
2.058
2.053
2.047
2.042
0.001
COOH
6a
1.03
3.08
2.04
2.08
1.00
f1 (ppm)

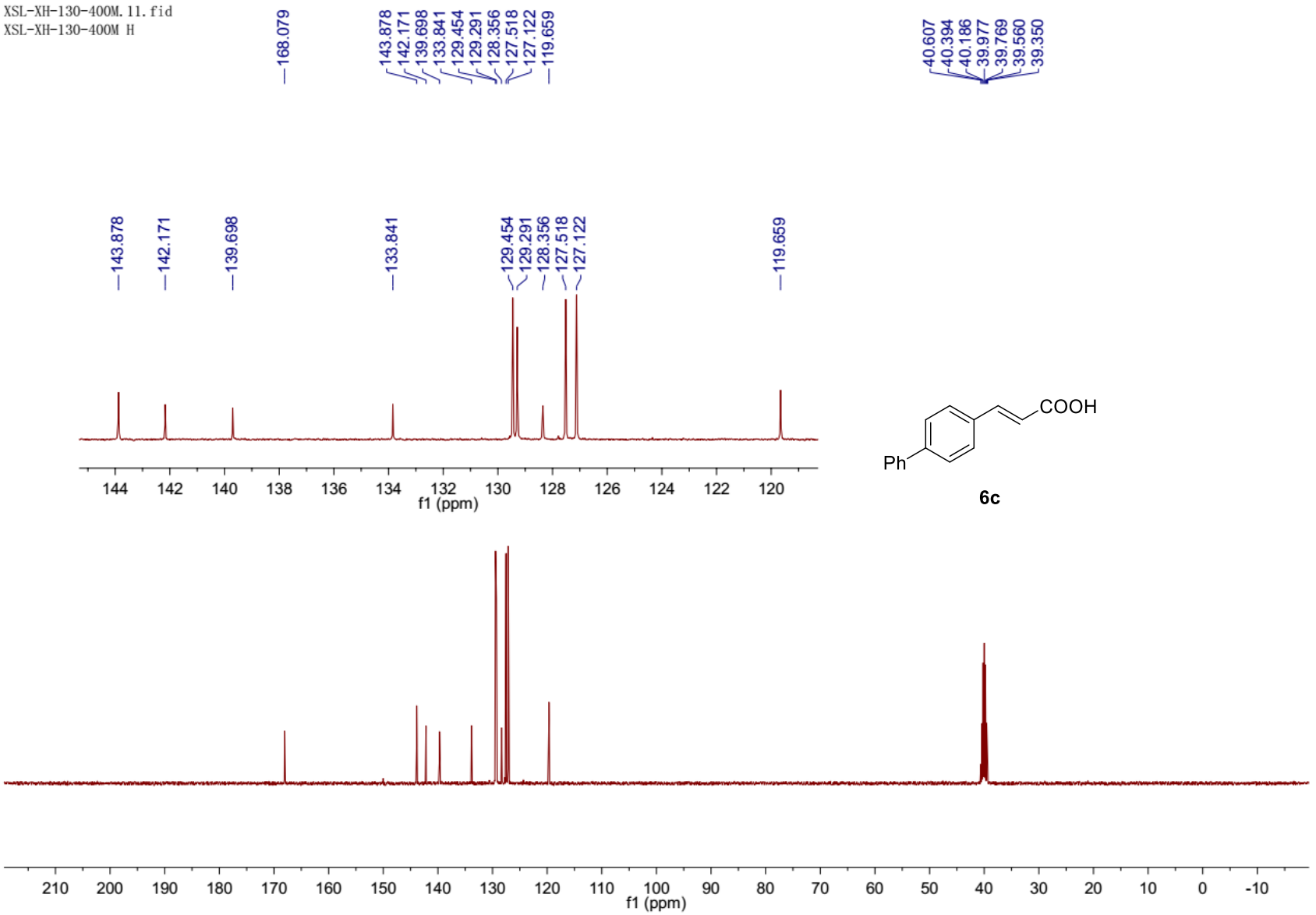

XSL-XH-81-500M/1
XSL-XH-81-500M C
206.281
166.847
144.554
134.309
133.486
132.193
129.793
128.655
128.539
127.729
127.219
126.712
123.708
118.657
29.410
29.257
29.102
28.948
28.795
28.640
28.487
135 134 133 132 131 130 129 128 127 126 125 124 123 122 121 120 119 118
f1 (ppm)
COOH
6a
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

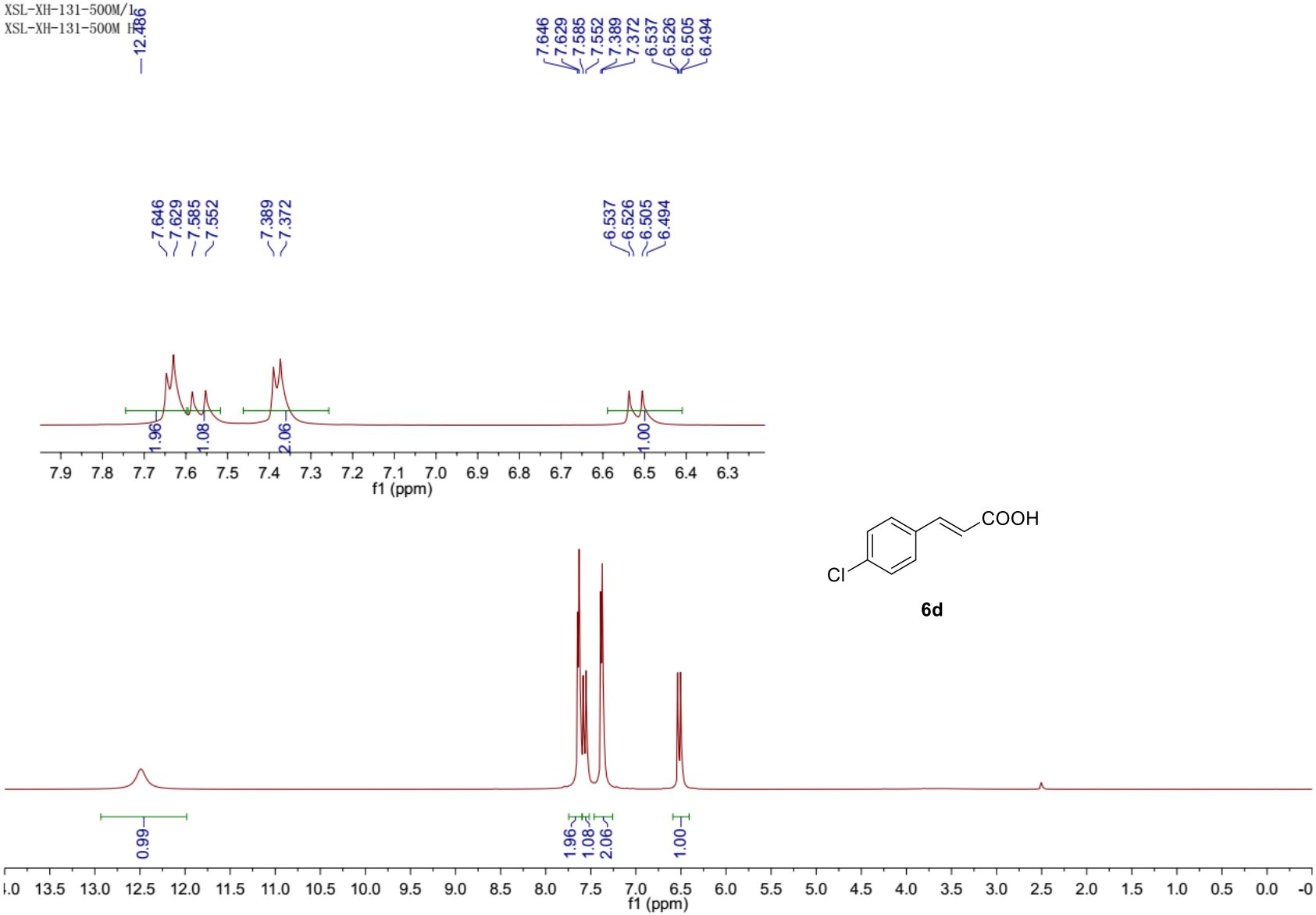

XSL-XH-133-500M/1
XSL-XH-133-500M H
12.400
7.593
7.561
7.537
7.533
7.524
7.520
7.193
7.177
6.485
6.453
2.287
1.04
1.90
2.01
1.00
0.92
3.07
f1 (ppm)
COOH
Me
6b

XSL-XH-133-500M/2
XSL-XH-133-500M C

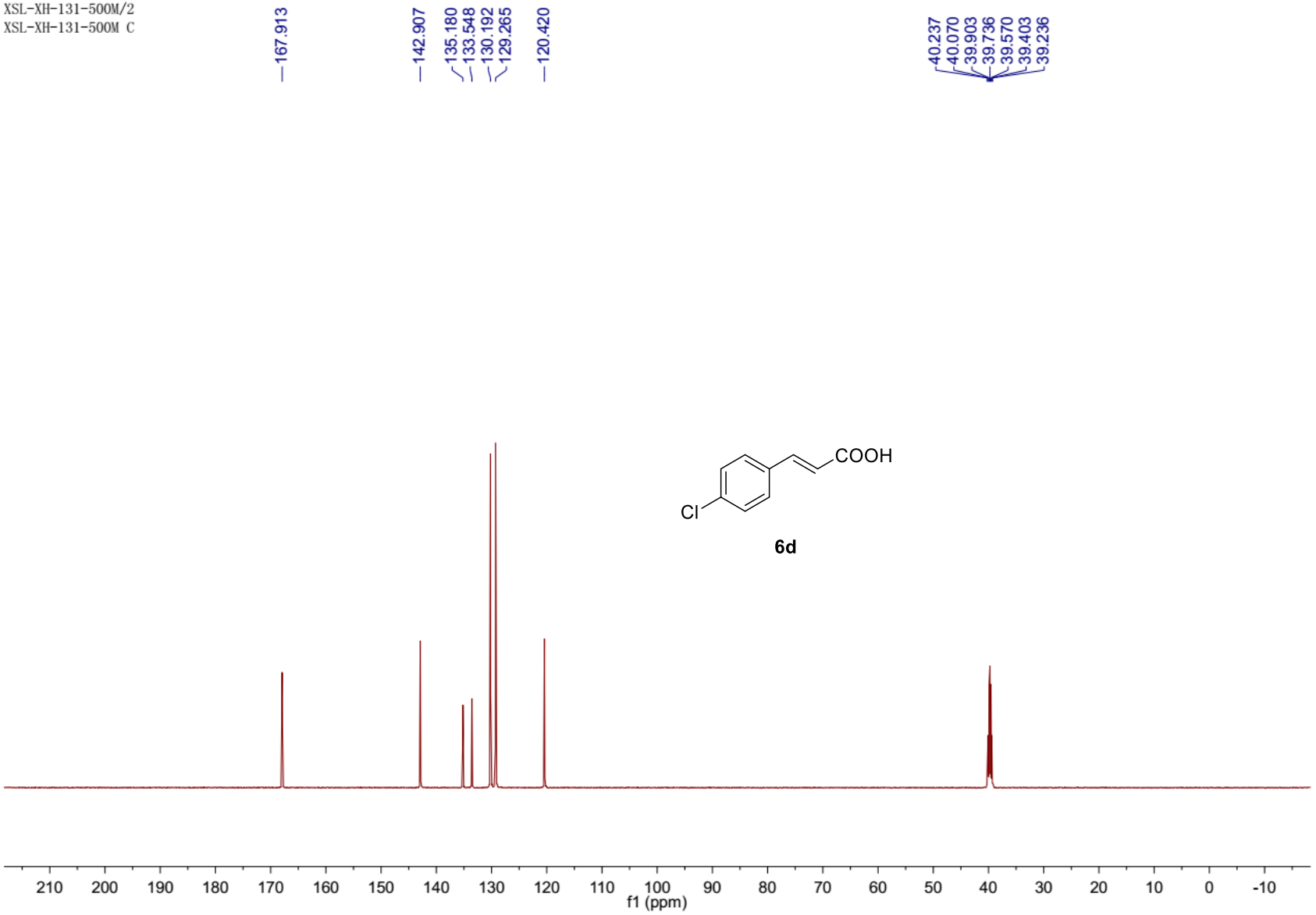



XSL-XH-130-3-500M-1
XSL-XH-130-3-500M
12.441
7.795
7.779
7.765
7.750
7.734
7.726
7.717
7.710
7.699
7.689
7.682
7.675
7.667
7.652
7.635
7.620
7.498
7.483
7.467
7.451
7.435
7.409
7.394
7.379
7.363
6.607
6.588
6.575
6.556
3.410
2.507
COOH
Ph
6c
2.06
5.00
2.04
1.04
1.00
0.84
f1 (ppm)

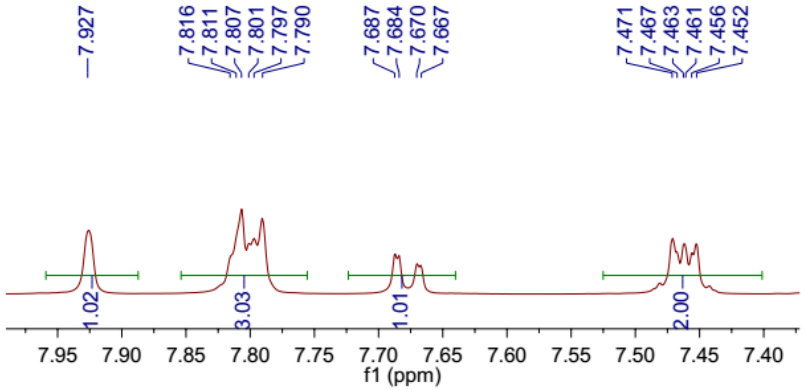

XSL-XH-130-400M.11.fid
XSL-XH-130-400M H
168.079
143.878
142.171
139.698
133.841
129.454
129.291
128.356
127.518
127.122
119.659
40.607
40.394
40.186
39.977
39.769
39.560
39.350
f1 (ppm)
COOH
Ph
6c
f1 (ppm)

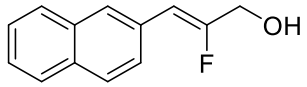

XSL-XH-131-500M/1
XSL-XH-131-500M H
12.486
7.646
7.629
7.585
7.552
7.389
7.372
6.537
6.526
6.505
6.494
1.96
1.08
2.06
1.00
f1 (ppm)
COOH
Cl
6d
0.99
1.96
1.08
2.06
1.00
f1 (ppm)

XSL-XH-131-500M/2
XSL-XH-131-500M C

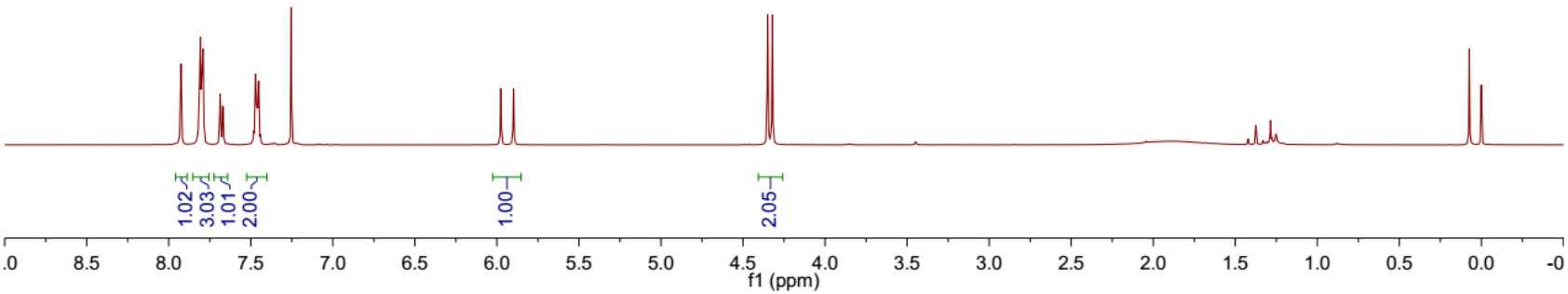


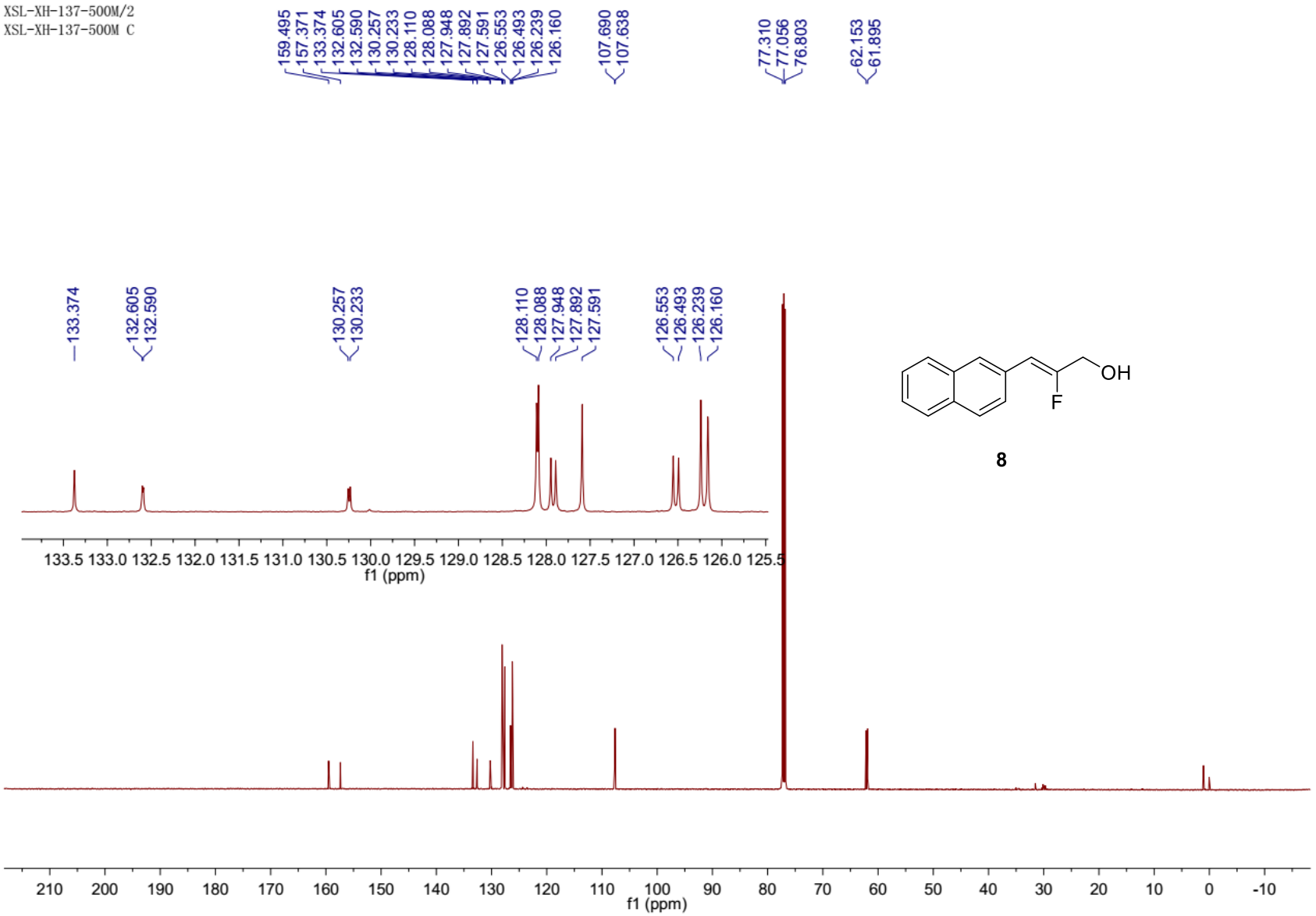

XSL-XH-127-500M-1
7.927
7.816
7.811
7.807
7.801
7.797
7.790
7.687
7.684
7.670
7.667
7.471
7.467
7.463
7.461
7.456
7.452
7.253
5.976
5.899
4.350
4.321
0.000


7.927
7.816
7.811
7.807
7.801
7.797
7.790
7.687
7.684
7.670
7.667
7.471
7.467
7.463
7.461
7.456
7.452
1.02
3.03
1.01
2.00
7.95 7.90 7.85 7.80 7.75 7.70 7.65 7.60 7.55 7.50 7.45 7.40
f1 (ppm)

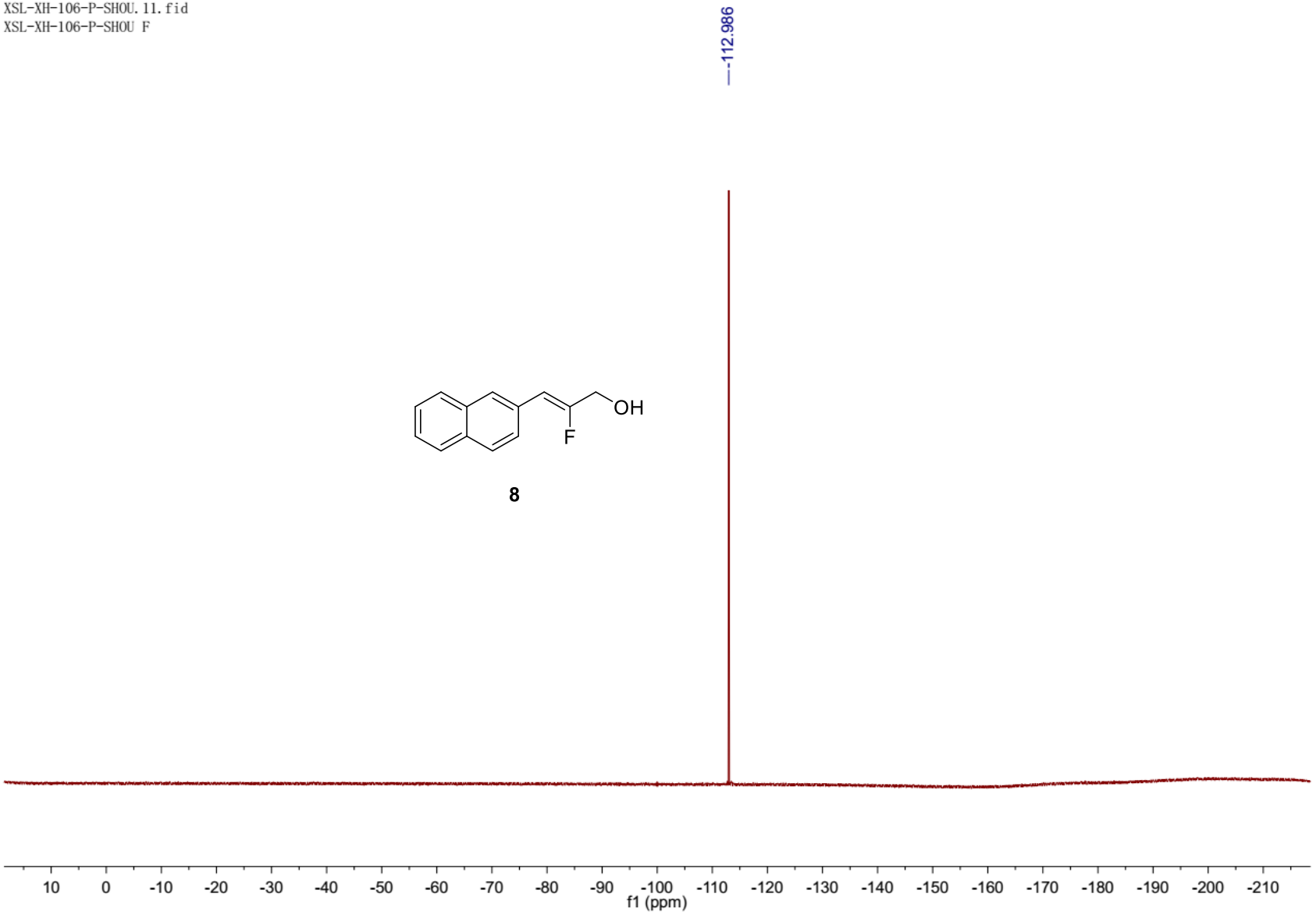

OH
F

8

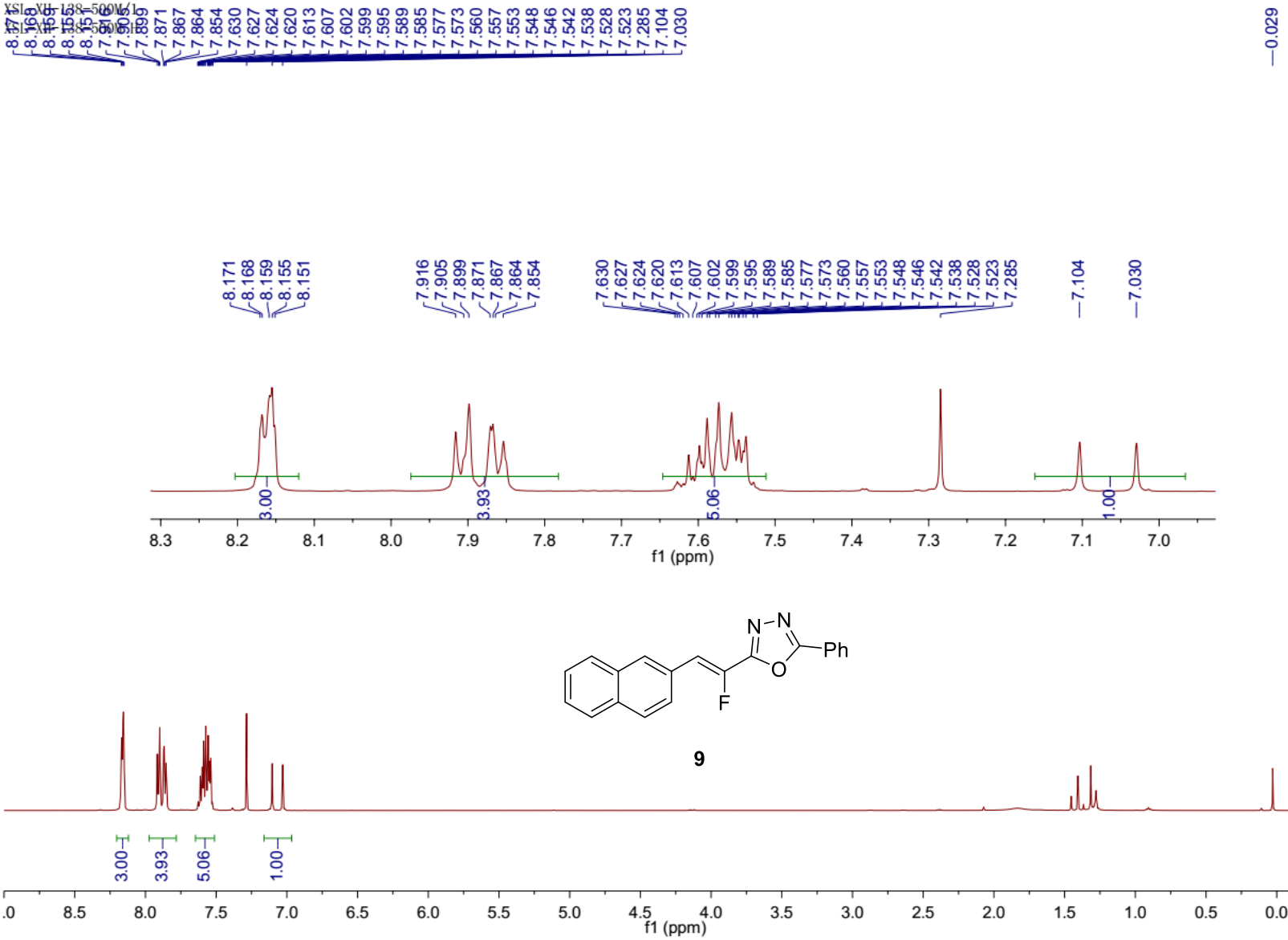

1.02
3.03
1.01
2.00
1.00
2.05
.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0
f1 (ppm)


XSL-XH-137-500M/2
XSL-XH-137-500M C
159.495
157.371
133.374
132.605
132.590
130.257
130.233
128.110
128.088
127.948
127.892
127.591
126.553
126.493
126.239
126.160
107.690
107.638
77.310
77.056
76.803
62.153
61.895
OH
F
8
f1 (ppm)
f1 (ppm)

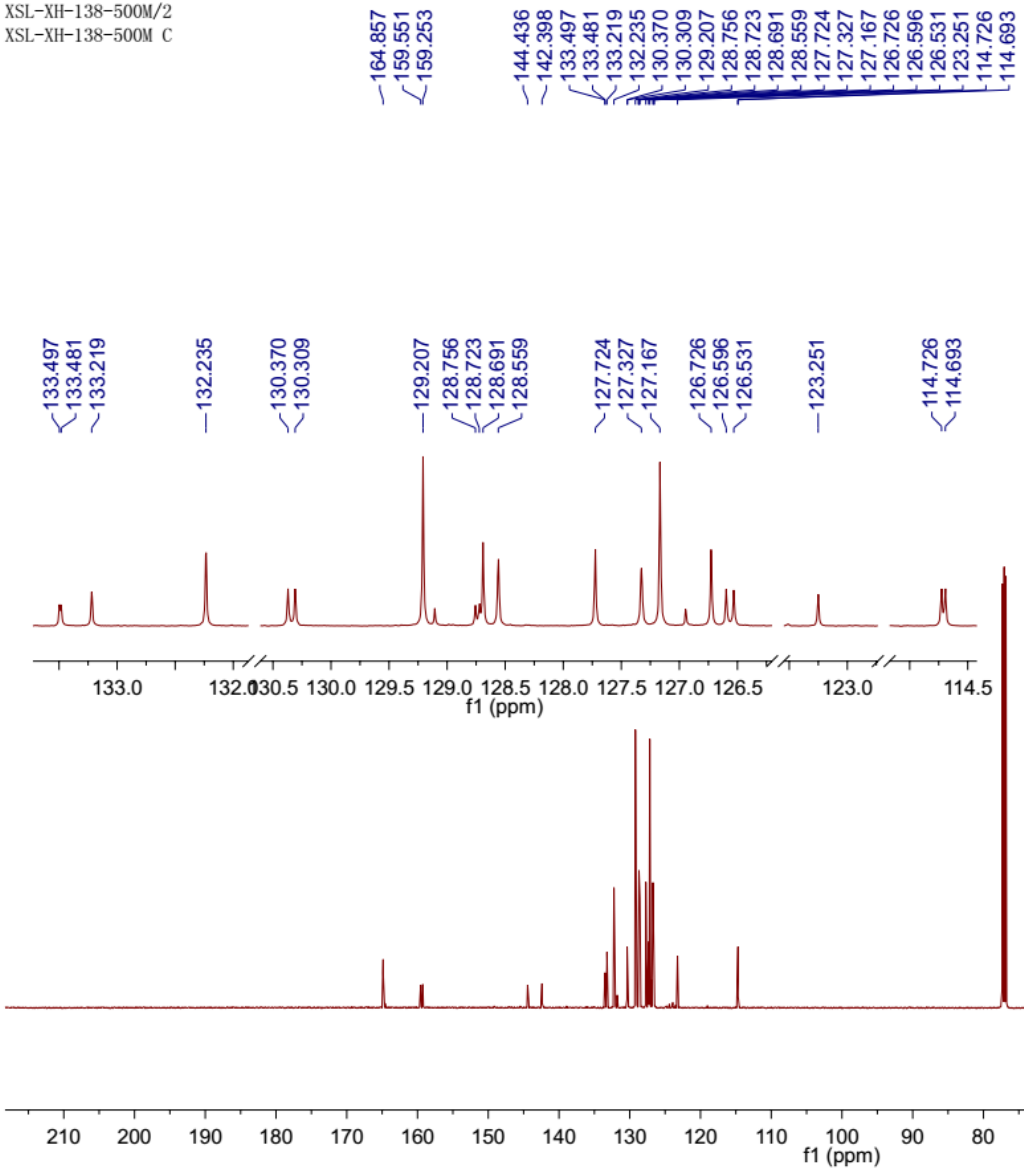
XSL-XH-106-P-SHOU.11.fid
XSL-XH-106-P-SHOU F

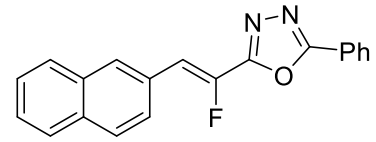


8


8.171
8.168
8.159
8.155
8.151
7.916
7.905
7.899
7.871
7.867
7.864
7.854
7.630
7.627
7.624
7.620
7.613
7.607
7.602
7.599
7.595
7.589
7.585
7.577
7.573
7.560
7.557
7.553
7.548
7.546
7.542
7.538
7.528
7.523
7.285
7.104
7.030
0.029
3.00
3.93
5.06
1.00
f1 (ppm)
N
N
O
Ph
F
9
3.00
3.93
5.06
1.00
f1 (ppm)

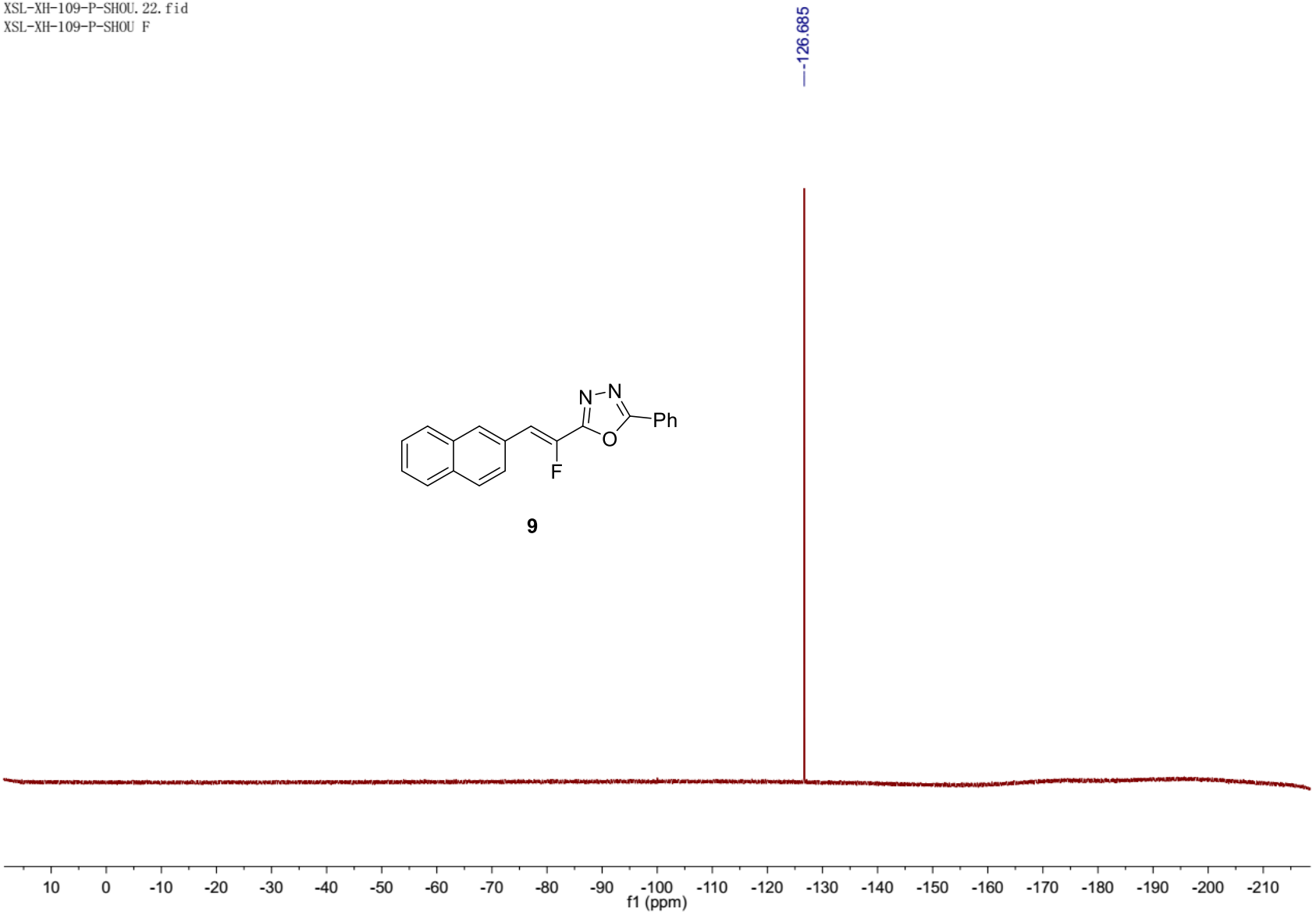
XSL-XH-138-500M/2
XSL-XH-138-500M C





9

XSL-XH-109-P-SHOU.22.fid
XSL-XH-109-P-SHOU F



9