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Gold-Catalyzed Intramolecular Diazo Coupling: An Efficient Macrocyclization towards Cyclic Olefins

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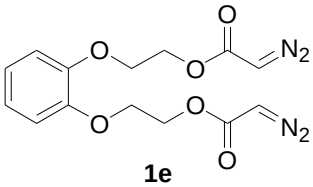
General Information

All of the reactions were carried out in a flame-dried tube. Solvents were dried prior to use. For column chromatography, 200-300 mesh silica gel was used. ^{1}H NMR were recorded on Bruker 400MHz spectrometer and ^{13}C NMR were recorded on Bruker 100MHz, 125MHz spectrometer in $CDCl_3$. HRMS were performed on Agilent 6230 TOF mass spectrometer, Waters Micromass GCT Premier and Thermo Fisher Scientific LTQ FT Ultra mass spectrometer. GC analysis was recorded on Agilent 7890/5975C-GC/MSD. Melting points were determined on a SGW X-4B melting point apparatus.

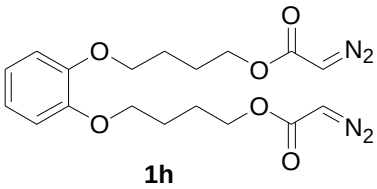
Preparation of Substrate

Preparation of diazo compounds: **1a-1m**[1], **1n-1v**[2] were prepared according to the literature procedures.



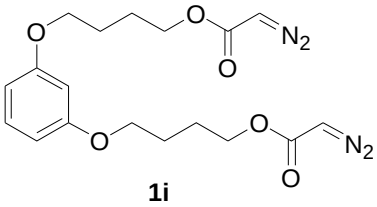
1e

1e: ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.94 (m, 4H), 4.82 (s, 2H), 4.57-4.47 (m, 4H), 4.25-4.20 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.94, 129.72, 122.41, 116.03, 67.88, 63.36, 62.21. HRMS (APCI) calcd. for $C_{14}H_{15}O_6$ $[M+H-2N_2]^+$: 279.0863, found: 279.0869.



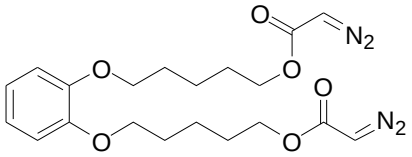
1h

1h: ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.92-6.87 (m, 4H), 4.74 (s, 1H), 4.36-4.24 (m, 4H), 4.05-4.00 (m, 4H), 2.47 (s, 1H), 1.92-1.74 (m, 8H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.96, 121.29, 114.08, 68.52, 64.61, 46.16, 25.86, 25.67. HRMS (APCI) calcd. for $C_{18}H_{23}O_6$ $[M+H-2N_2]^+$: 335.1489, found: 335.1475.



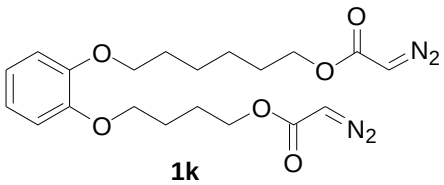
1i

1i: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.16 (t, J = 8.2 Hz, 1H), 6.49 (d, J = 2.3 Hz, 1H), 6.47 (d, J = 2.3 Hz, 1H), 6.44 (t, J = 2.3 Hz, 1H), 4.74 (s, 2H), 4.24 (t, J = 6.1 Hz, 4H), 3.96 (t, J = 5.7 Hz, 4H), 1.84 (dt, J = 6.0, 3.0 Hz, 8H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.16, 129.88, 106.77, 101.47, 67.23, 64.53, 46.19, 25.79, 25.63. HRMS (APCI) calcd. for $C_{18}H_{23}O_6$ $[M+H-2N_2]^+$: 335.1489, found: 335.1479.



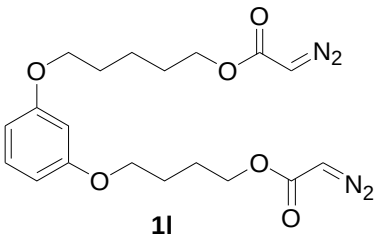
1j

1j: ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.89 (s, 4H), 4.74 (s, 2H), 4.19 (t, *J* = 12.0 Hz, 4H), 4.01 (t, *J* = 12.0 Hz, 4H), 1.84 (m, 4H), 1.73 (m, 4H), 1.60-1.50 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.09, 121.21, 114.18, 68.93, 64.82, 46.14, 28.95, 28.60, 22.54. HRMS (APCI) calcd. for $C_{20}H_{27}O_6$ $[M+H-2N_2]^+$: 363.1802, found: 363.1802.



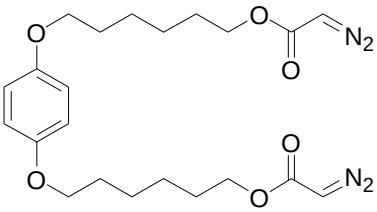
1k

1k: ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.89 (s, 4H), 4.73 (s, 1H), 4.35 (s, 1H), 4.31-4.11 (m, 4H), 4.00 (m, 4H), 1.91 (m, 1H), 1.87 (m, 3H), 1.84-1.78 (m, 2H), 1.68 (m, 2H), 1.55-1.48 (m, 2H), 1.48-1.40 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.33, 165.04, 149.20, 148.92, 121.35, 121.16, 113.99, 68.94, 68.88, 68.61, 65.40, 65.23, 64.63, 46.17, 29.19, 28.78, 28.25, 25.87, 25.62. HRMS (APCI) calcd. for $C_{20}H_{27}O_6$ $[M+H-2N_2]^+$: 363.1802, found: 363.1801.



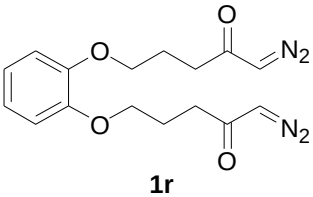
1l

1l: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.15 (t, *J* = 8.2 Hz, 1H), 6.48 (d, *J* = 2.3 Hz, 1H), 6.46 (d, *J* = 2.3 Hz, 1H), 6.44 (t, *J* = 2.2 Hz, 1H), 4.74 (s, 2H), 4.18 (t, *J* = 6.6 Hz, 4H), 3.94 (t, *J* = 6.4 Hz, 4H), 1.84-1.76 (m, 4H), 1.76-1.67 (m, 4H), 1.58-1.48 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.27, 129.84, 106.71, 101.47, 67.59, 64.76, 46.17, 28.89, 28.58, 22.54. HRMS (APCI) calcd. for $C_{20}H_{27}O_6$ $[M+H-2N_2]^+$: 363.1802, found: 363.1805.



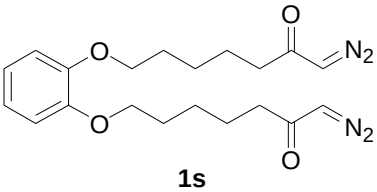
1m

1m: ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.81 (s, 4H), 4.73 (s, 2H), 4.17 (t, *J* = 6.6 Hz, 4H), 3.90 (t, *J* = 6.4 Hz, 4H), 1.76 (dd, *J* = 14.4, 6.6 Hz, 4H), 1.68 (dd, *J* = 14.3, 7.0 Hz, 4H), 1.54-1.36 (m, 8H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.18, 115.41, 68.41, 64.88, 46.16, 29.28, 28.77, 25.77, 25.67. HRMS (APCI) calcd. for $C_{22}H_{31}O_6$ $[M+H-2N_2]^+$: 391.2115, found: 391.2109.



1r

1r: ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.88 (s, 4H), 5.36 (s, 2H), 4.02 (t, *J* = 6.1 Hz, 4H), 2.57 (s, 4H), 2.24-2.04 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.42, 148.84, 121.49, 114.29, 68.05, 54.57, 24.80. HRMS (DART) calcd. for $C_{16}H_{19}N_4O_4$ $[M+H]^+$: 331.1401, found: 331.1399.

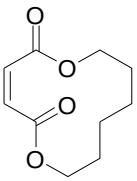


1s

1s: ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.87 (s, 4H), 5.29 (s, 2H), 3.98 (t, *J* = 12.0 Hz, 4H), 2.34 (s, 4H), 1.88-1.77 (m, 4H), 1.70 (m, 4H), 1.56-1.46 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.04, 149.07, 121.19, 114.15, 68.90, 29.71, 29.08, 25.74, 24.95. HRMS (DART) calcd. for $C_{20}H_{27}N_4O_4$ $[M+H]^+$: 387.2027, found:387.2026.

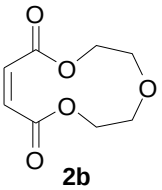
General procedure for table 2

To a solution of **1** (1 mmol) in 10 mL of 1,2-dichloroethane (DCE) was added 2.5 mol% of *t*BuXphosAuCl/ $NaBAr_F$ at room temperature. Then the mixture was heated 80°C and stirred for 12 h under argon atmosphere. The mixture was cooled to room temperature and solvent was removed under vacuum. The crude residue was purified by column chromatography to afford product **2**.



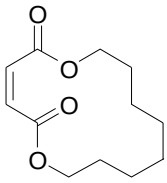
2a

2a:[1] This compound was prepared via general procedure (Yield: 65%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.13 (s, 2H), 4.26-4.12 (m, 4H), 1.77-1.65 (m, 4H), 1.51-1.46 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.21, 128.92, 66.37, 24.57, 24.19.



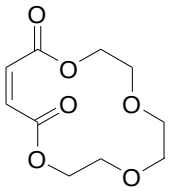
2b

2b:[1] This compound was prepared via general procedure (Yield: 63%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.32 (s, 2H), 4.42-4.30 (m, 4H), 3.93-3.82 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.13, 129.81, 71.57, 66.06.



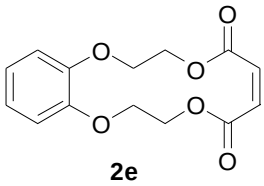
2c

2c:[1] This compound was prepared via general procedure (Yield: 62%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.24 (s, 2H), 4.32-4.24 (m, 4H), 1.73-1.64 (m, 4H), 1.41 (s, 8H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.28, 129.58, 64.02, 26.81, 24.84, 21.65.



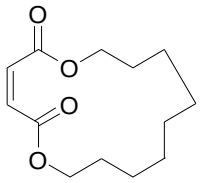
2d

2d:[1] This compound was prepared via general procedure (Yield: 62%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.27 (s, 2H), 4.44-4.32 (m, 4H), 3.84-3.71 (m, 4H), 3.65 (s, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.14, 129.61, 69.45, 68.67, 63.79.



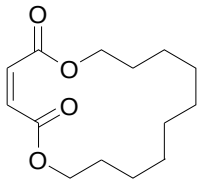
2e

2e: This compound was prepared via general procedure (Yield: 68%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.95 (tdd, J = 9.6, 5.9, 3.8 Hz, 4H), 6.30 (s, 2H), 4.52 (dd, J = 5.1, 3.3 Hz, 4H), 4.27 (dd, J = 5.1, 3.3 Hz, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.10, 149.04, 129.79, 122.40, 115.58, 67.73, 63.40. HRMS (APCI) calcd. for $C_{14}H_{15}O_6$ $[M+H]^+$: 279.0863, found: 279.0855.



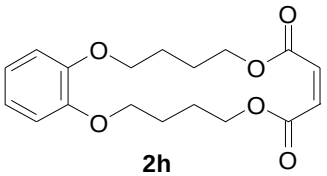
2f

2f:[1] This compound was prepared via general procedure (Yield: 58%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.23 (s, 2H), 4.34-4.23 (m, 4H), 1.74-1.62 (m, 4H), 1.39 (m, 10H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.46, 129.73, 64.55, 28.30, 26.70, 25.73, 23.54.



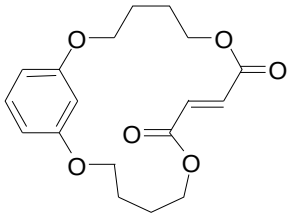
2g

2g:[1] This compound was prepared via general procedure (Yield: 54%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.25 (s, 2H), 4.36-4.22 (m, 4H), 1.79-1.64 (m, 4H), 1.35 (m, 12H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.49, 129.95, 63.90, 28.10, 26.55, 25.33, 24.25.



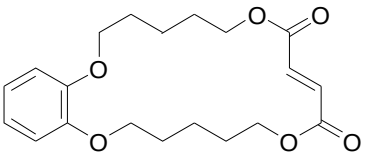
2h

2h: This compound was prepared via general procedure as a solid (Yield: 66%), mp: 80-82°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.95-6.81 (m, 4H), 6.25 (s, 2H), 4.34 (t, *J* = 7.1 Hz, 4H), 4.03 (t, *J* = 5.3 Hz, 4H), 2.00-1.92 (m, 4H), 1.89 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.35, 148.84, 129.69, 121.03, 113.02, 68.29, 65.61, 26.13, 25.87. HRMS (APCI) calcd. for $C_{18}H_{23}O_6$ $[M+H]^+$: 335.1489, found: 335.1519.



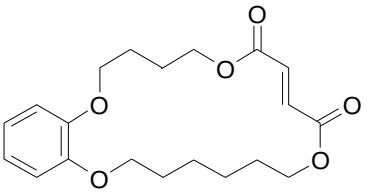
2i

2i: This compound was prepared via general procedure as a solid (Yield: 42%), mp: 113-115°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.13 (t, *J* = 8.2 Hz, 1H), 6.76 (s, 2H), 6.51 (d, *J* = 2.3 Hz, 1H), 6.49 (d, *J* = 2.3 Hz, 1H), 6.44 (t, *J* = 2.3 Hz, 1H), 4.36-4.28 (t, *J* = 12.0 Hz, 4H), 4.07 (t, *J* = 16.0 Hz, 4H), 2.01 (dd, *J* = 13.1, 6.3 Hz, 4H), 1.98-1.91 (m, 4H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.46, 160.23, 133.35, 129.98, 108.69, 98.27, 67.62, 66.61, 28.13, 24.64. HRMS (APCI) calcd. for $C_{18}H_{23}O_6$ $[M+H]^+$: 335.1489, found: 335.1509.



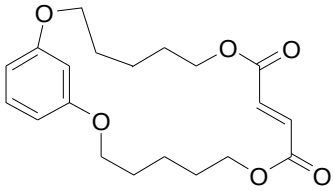
2j

2j: This compound was prepared via general procedure (Yield: 48%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.86 (s, 6H), 4.33-4.24 (t, *J* = 12.0 Hz, 4H), 3.97 (t, *J* = 12.0 Hz, 4H), 2.01-1.93 (m, 4H), 1.84-1.77 (m, 4H), 1.68 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.74, 148.34, 133.69, 120.40, 111.55, 68.05, 65.88, 29.52, 28.16, 24.10. HRMS (APCI) calcd. for $C_{20}H_{27}O_6$ $[M+H]^+$: 363.1802, found: 363.1797.



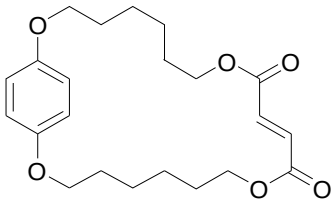
2k

2k: This compound was prepared via general procedure as a solid (Yield: 58%), mp: 97-99°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.90 (d, *J* = 1.4 Hz, 4H), 6.88 (s, 2H), 4.30-4.26 (m, 2H), 4.26-4.22 (m, 2H), 4.03 (t, *J* = 3.9 Hz, 2H), 4.00 (t, *J* = 4.6 Hz, 2H), 2.04-1.96 (m, 2H), 1.93-1.84 (m, 4H), 1.76 (dd, *J* = 11.0, 5.6 Hz, 2H), 1.57-1.49 (m, 4H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.86, 149.40, 133.80, 133.75, 121.35, 121.26, 115.00, 114.11, 69.96, 68.52, 66.08, 64.60, 29.84, 27.26, 27.05, 26.84, 26.03, 25.41. HRMS (APCI) calcd. for $C_{20}H_{27}O_6$ $[M+H]^+$: 363.1802, found: 363.1796.



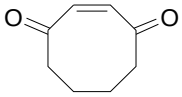
2l

2l: This compound was prepared via general procedure (Yield: 52%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18 (t, *J* = 8.1 Hz, 1H), 6.82 (s, 2H), 6.50 (d, *J* = 2.3 Hz, 1H), 6.48 (d, *J* = 2.1 Hz, 1H), 6.48-6.45 (m, 1H), 4.32-4.21 (t, *J* = 12.0 Hz, 4H), 4.03 (t, *J* = 12.0 Hz, 4H), 1.84-1.70 (m, 8H), 1.64 (dt, *J* = 9.0, 4.6 Hz, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.96, 160.19, 133.52, 129.94, 106.33, 103.74, 67.29, 64.89, 28.20, 27.85, 22.54. HRMS (APCI) calcd. for $C_{20}H_{27}O_6$ $[M+H]^+$: 363.1802, found: 363.1799.



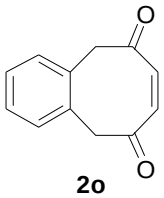
2m

2m: This compound was prepared via general procedure (Yield: 45%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.80 (s, 4H), 6.70 (s, 2H), 4.27-4.18 (t, *J* = 12.0 Hz, 4H), 4.00 (t, *J* = 12.0 Hz, 4H), 1.73 (m, 4H), 1.65 (m, 4H), 1.51 (m, 4H), 1.47-1.37 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.05, 152.69, 133.37, 116.17, 67.74, 64.92, 28.34, 27.72, 24.82, 24.75. HRMS (APCI) calcd. for $C_{22}H_{31}O_6$ $[M+H]^+$: 391.2115, found: 391.2107.



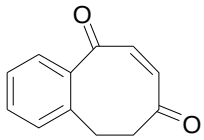
2n

2n:[2] This compound was prepared via general procedure (Yield: 62%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.40 (s, 2H), 2.57 (s, 4H), 1.90 (m, 4H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 204.69, 136.82, 40.34, 23.37.



2o

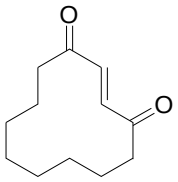
2o:[2] This compound was prepared via general procedure (Yield: 55%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35-7.23 (m, 4H), 6.18 (s, 2H), 3.95 (s, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.06, 134.39, 133.00, 130.84, 128.36, 48.63.



2p

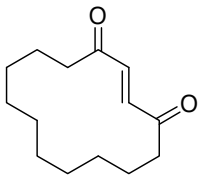
2p:[2] This compound was prepared via general procedure (Yield: 65%). ^{1}H NMR (400 MHz,

$CDCl_3$) δ 7.51 (d, *J* = 7.7 Hz, 1H), 7.47-7.44 (m, 1H), 7.32 (d, *J* = 7.6 Hz, 1H), 7.17 (d, *J* = 7.6 Hz, 1H), 6.59 (q, *J* = 13.7 Hz, 2H), 3.25 (dd, *J* = 7.4, 5.5 Hz, 2H), 2.86 (dd, *J* = 7.4, 5.5 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 204.00, 194.74, 137.43, 135.04, 132.58, 130.48, 129.14, 127.73, 48.41, 28.45.



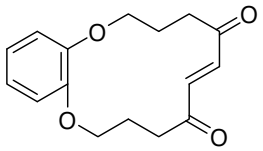
2q

2q:[3] This compound was prepared via general procedure (Yield: 45%). ^1H NMR (400 MHz, $CDCl_3$) δ 7.03 (s, 2H), 2.71-2.59 (m, 4H), 1.89-1.71 (m, 4H), 1.46 (m, 4H), 1.28 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.18, 137.22, 39.12, 24.75, 24.51, 24.18.



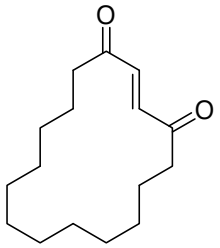
2r

2r:[3] This compound was prepared via general procedure as a solid (Yield: 61%). ^1H NMR (400 MHz, $CDCl_3$) δ 6.99 (s, 2H), 2.71-2.56 (m, 4H), 1.88-1.67 (m, 4H), 1.40-1.26 (m, 12H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 202.52, 136.13, 41.11, 26.32, 26.19, 25.66, 24.59.



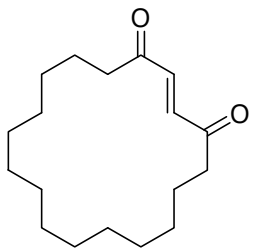
2s

2s: This compound was prepared via general procedure as a solid (Yield: 72%), mp: 108-110°C. ^1H NMR (400 MHz, $CDCl_3$) δ 7.34 (s, 2H), 6.90 (m, 2H), 6.86-6.80 (m, 2H), 4.05-4.03 (t, *J* = 8.0 Hz, 4H), 2.84 (t, *J* = 12.0 Hz, 4H), 2.27-2.15 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.00, 148.09, 137.12, 121.12, 112.01, 67.34, 38.51, 25.70. HRMS (EI) calcd. for $C_{16}H_{18}O_4$ [M]: 274.1205, found: 274.1199.



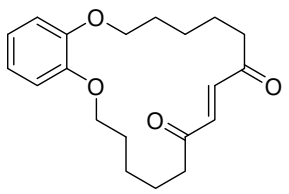
2t

2t:[3] This compound was prepared via general procedure as a solid (Yield: 56%). ^1H NMR (400 MHz, $CDCl_3$) δ 7.00 (s, 2H), 2.65-2.59 (m, 4H), 1.78-1.69 (m, 4H), 1.26 (m, 16H). ^{13}C NMR (125MHz, $CDCl_3$) δ 201.88, 136.07, 41.64, 27.23, 27.10, 26.92, 26.62, 24.63.



2u

2u: This compound was prepared via general procedure as white solid (Yield: 61%), mp: 121-123°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.94 (s, 2H), 2.63 (t, *J* = 12.0 Hz, 4H), 1.70 (dd, *J* = 13.7, 6.9 Hz, 4H), 1.29 (m, 20H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 201.62, 136.31, 41.14, 29.71, 27.59, 27.50, 27.44, 27.42, 24.39. HRMS (APCI) calcd. for $C_{18}H_{31}O_2$ $[M+H]^+$: 279.2319, found: 279.2316.



2v

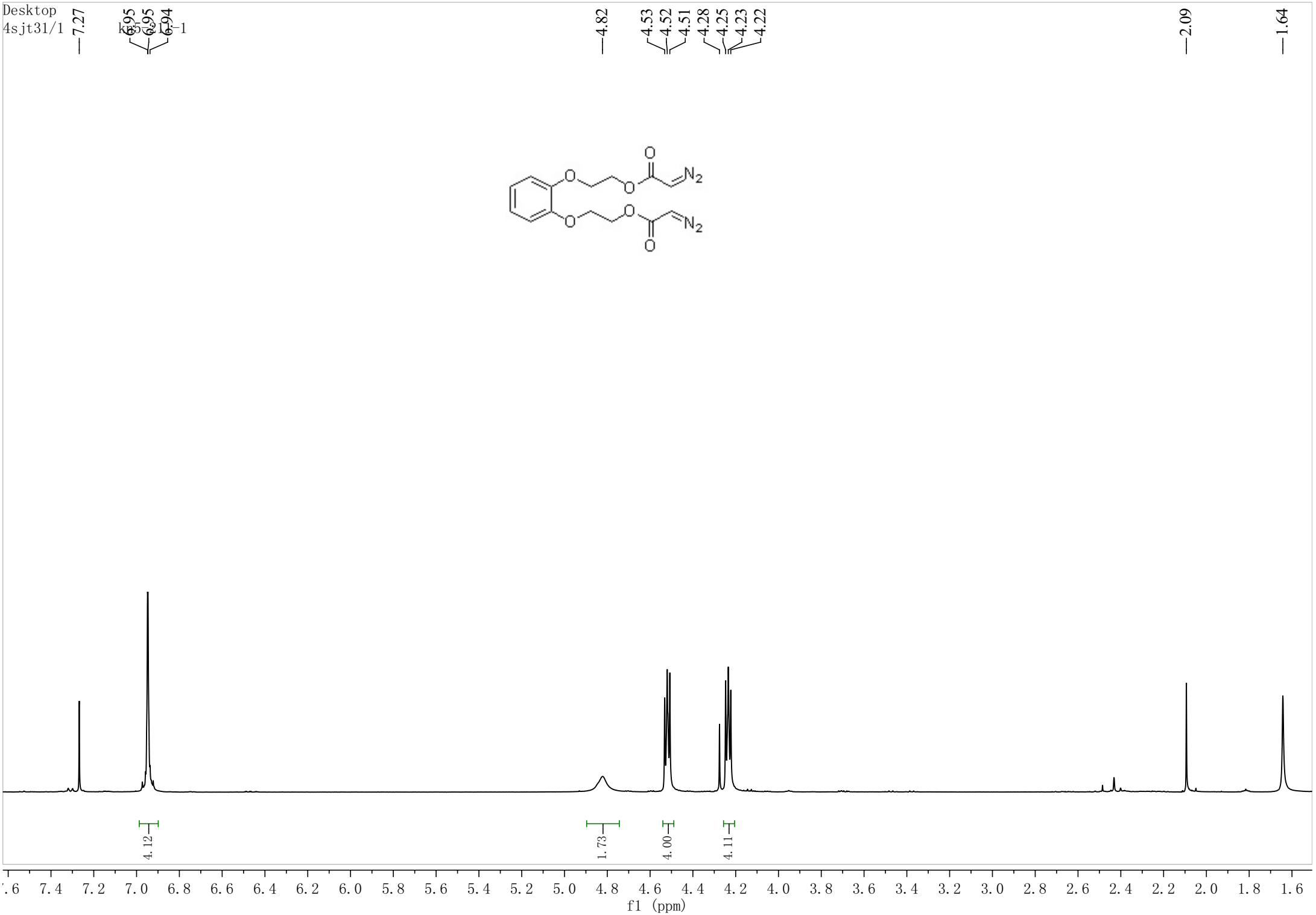
2v: This compound was prepared via general procedure as a solid (Yield: 69%), mp: 83-85°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.99 (s, 2H), 6.93-6.75 (m, 4H), 3.96 (t, *J* = 5.5 Hz, 4H), 2.67-2.64 (t, *J* = 12.0 Hz, 4H), 1.88-1.70 (m, 8H), 1.51 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.21, 148.54, 136.57, 120.66, 112.36, 67.94, 41.48, 29.57, 26.33, 26.13. HRMS (EI) calcd. for $C_{20}H_{26}O_4$ [M]: 330.1831, found: 330.1829.

References:

[1] Li, G.; Che, C. *Org. Lett.,* **2004**, *6*, 1621.

[2] Chandler, C. L.; List, B. *J. Am. Chem. Soc.,* **2008**, *130,* 6737.

[3] Sunanta, K.; Anthony, M. *Chem. Comm.,* **1978**, *23*, 1069.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1.64
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O
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O
N2
N2
O
O
4.12
1.73
4.00
4.11
7.6 7.4 7.2 7.0 6.8 6.6 6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4 3.2 3.0 2.8 2.6 2.4 2.2 2.0 1.8 1.6
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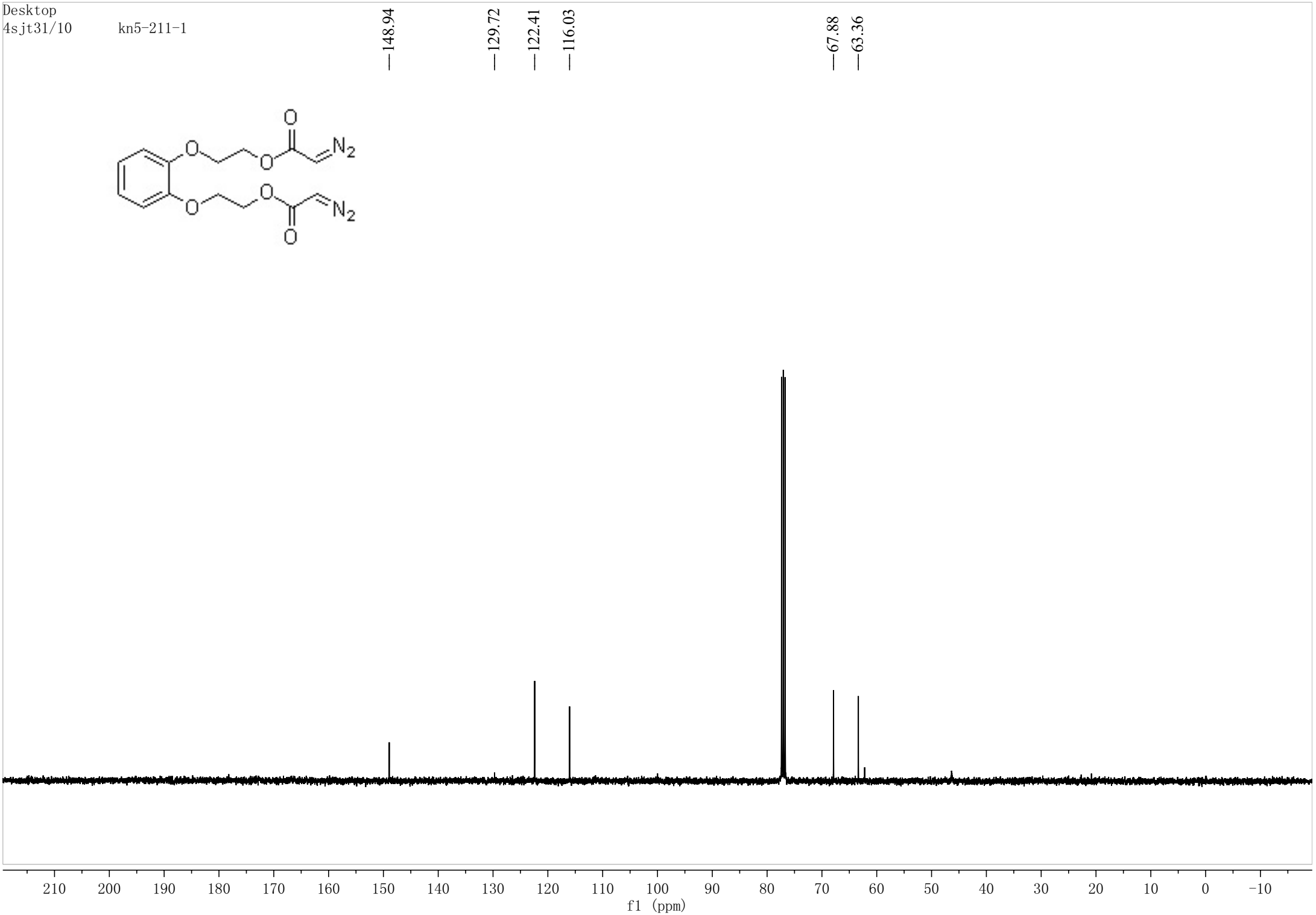

2014-7-3
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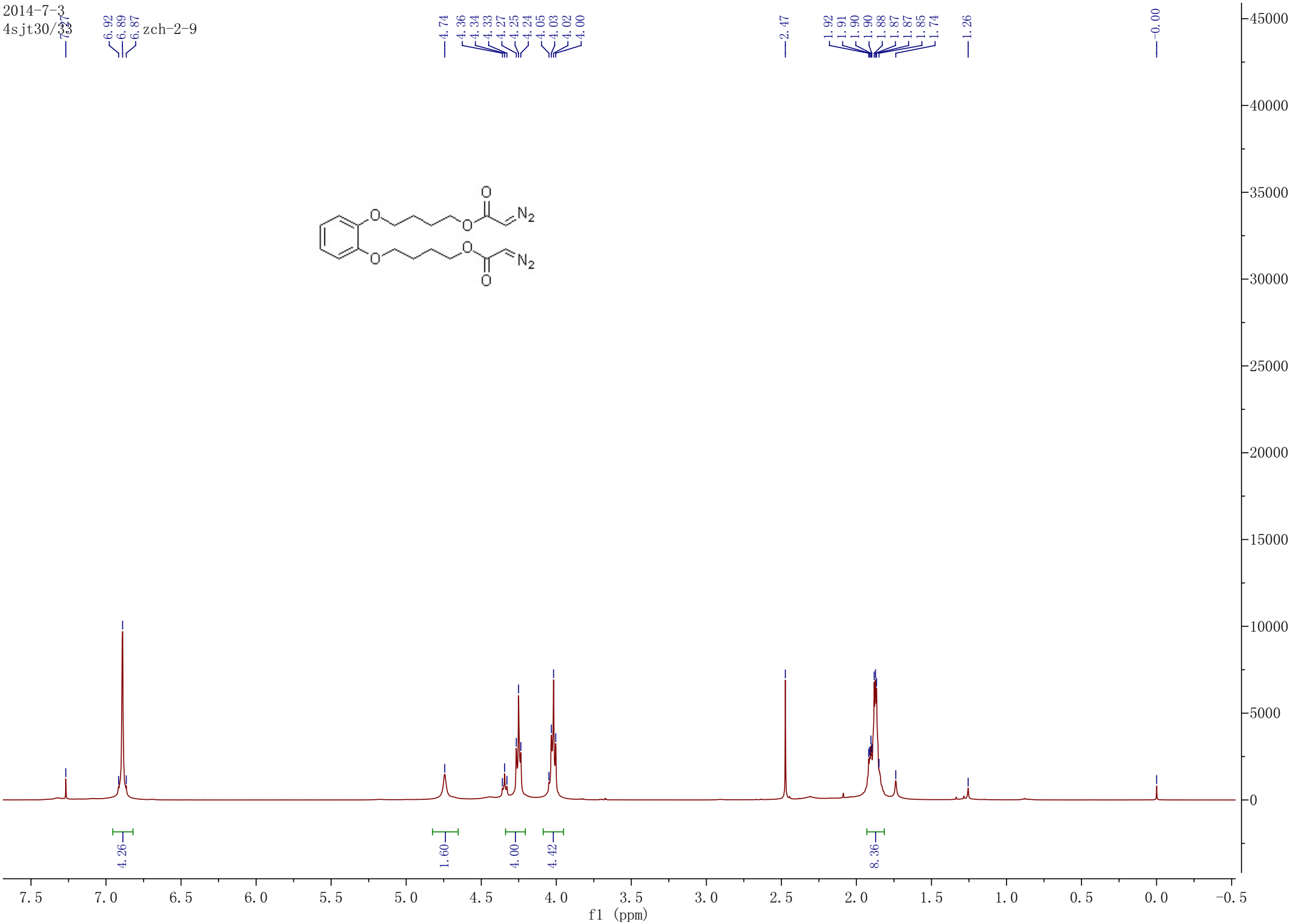

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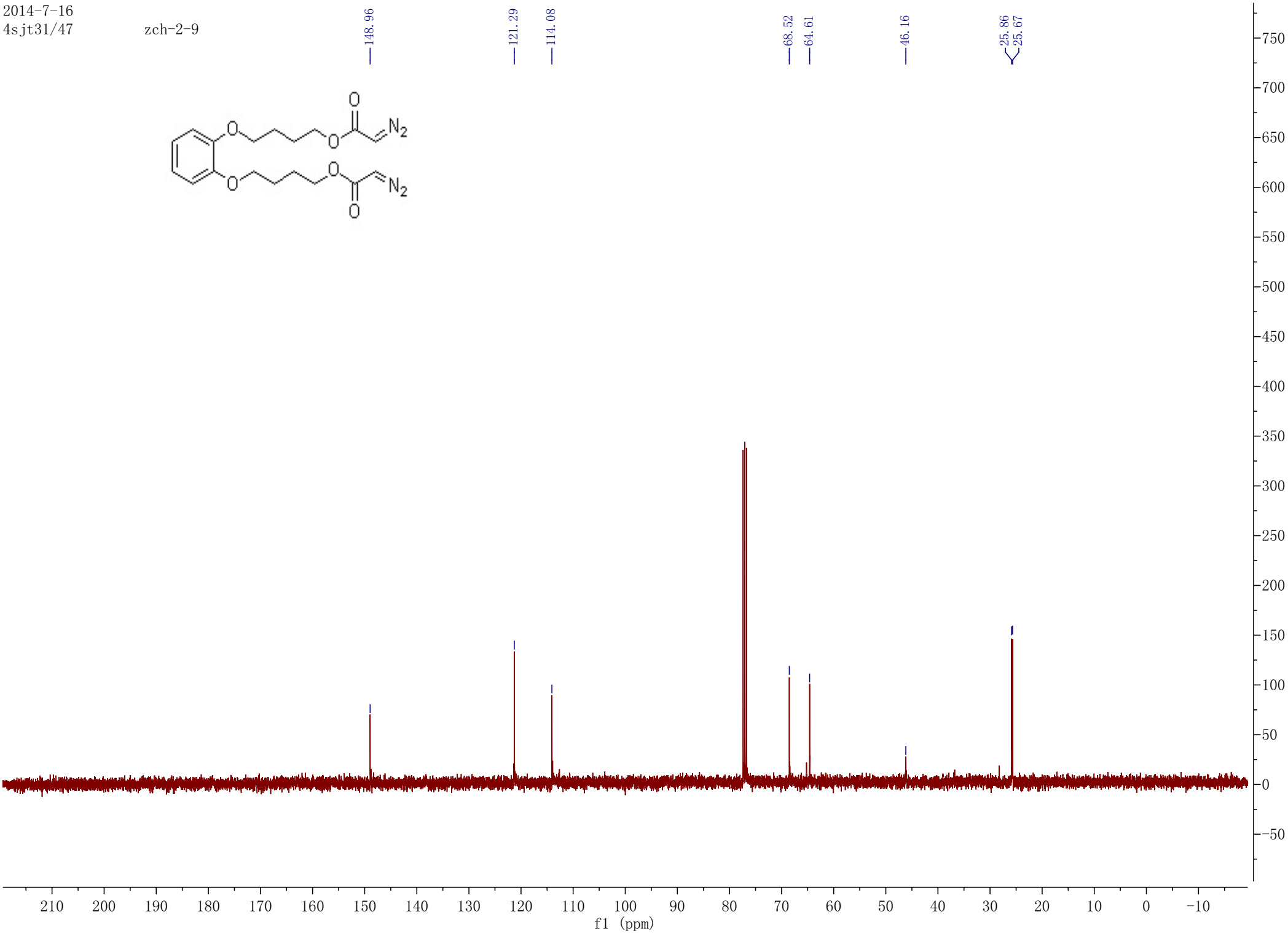
4sjt31/47

zch-2-9



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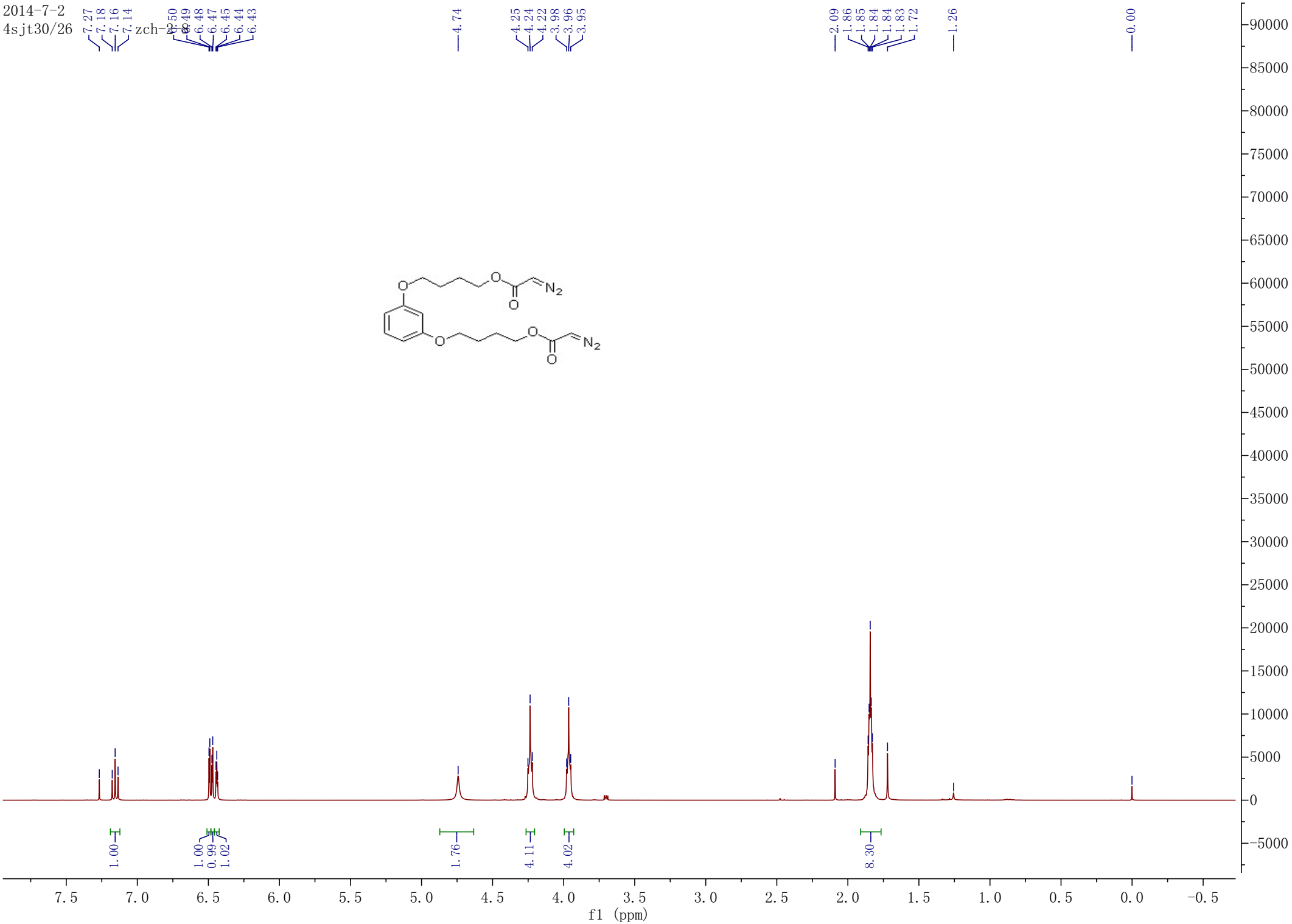

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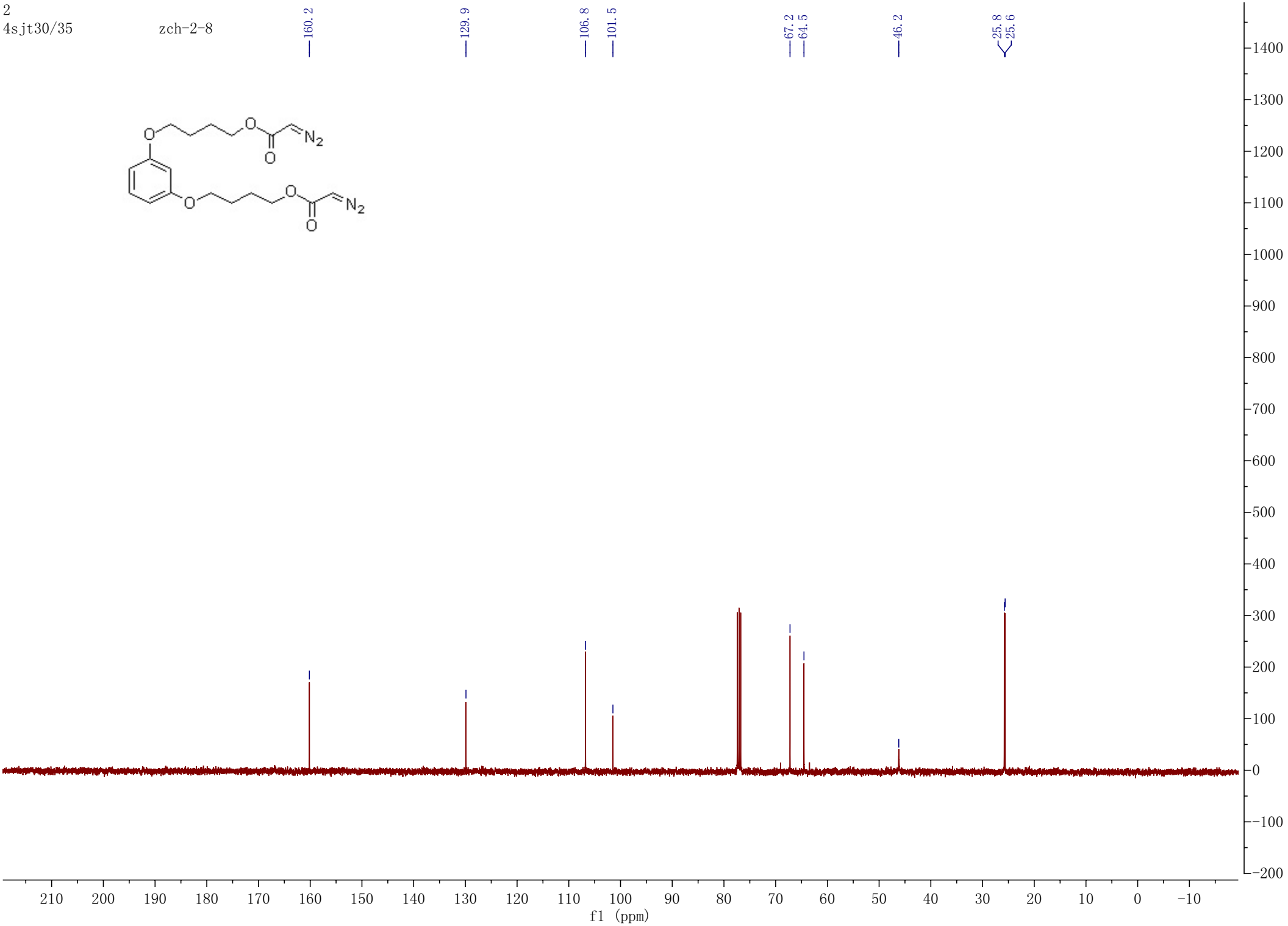

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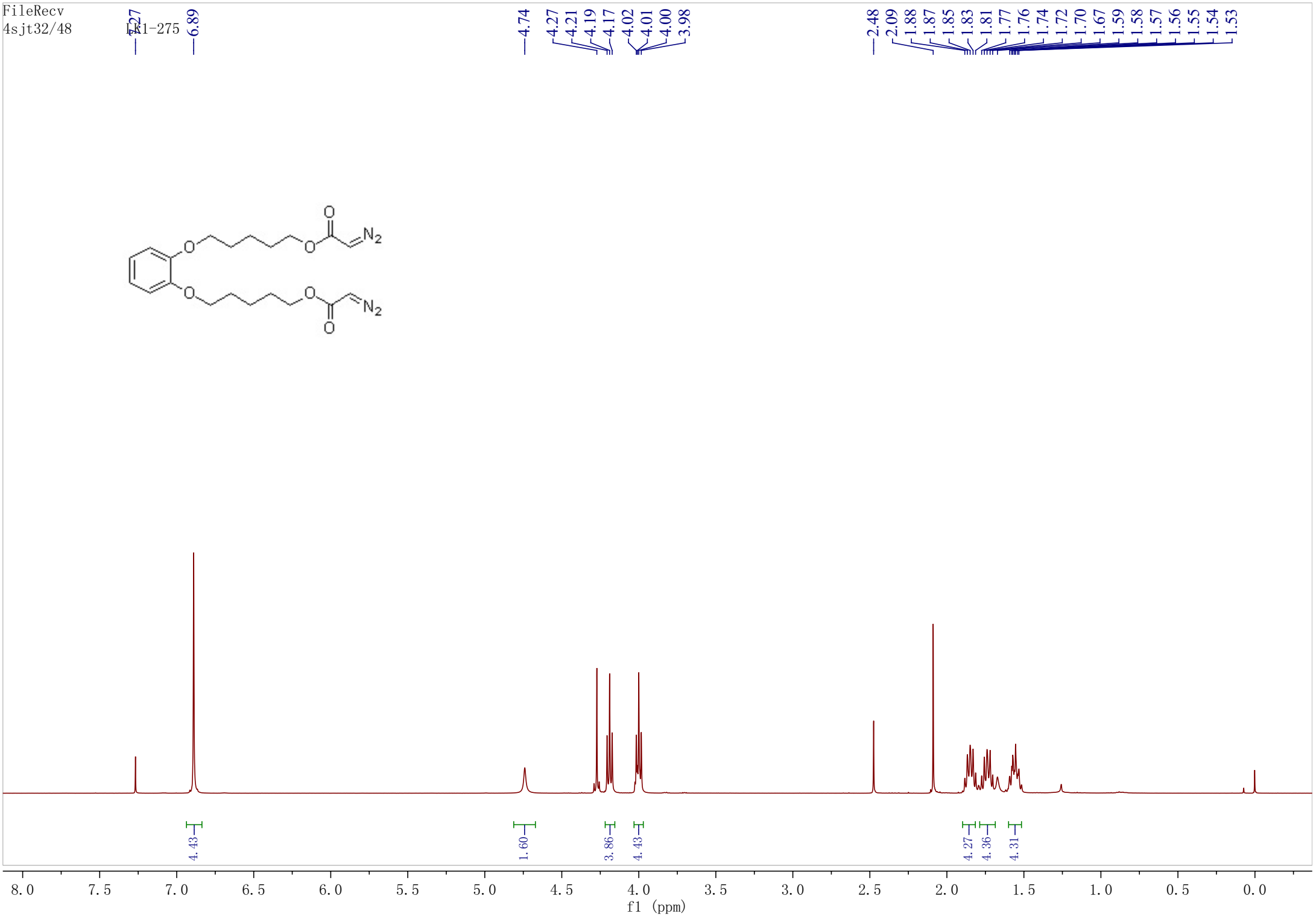

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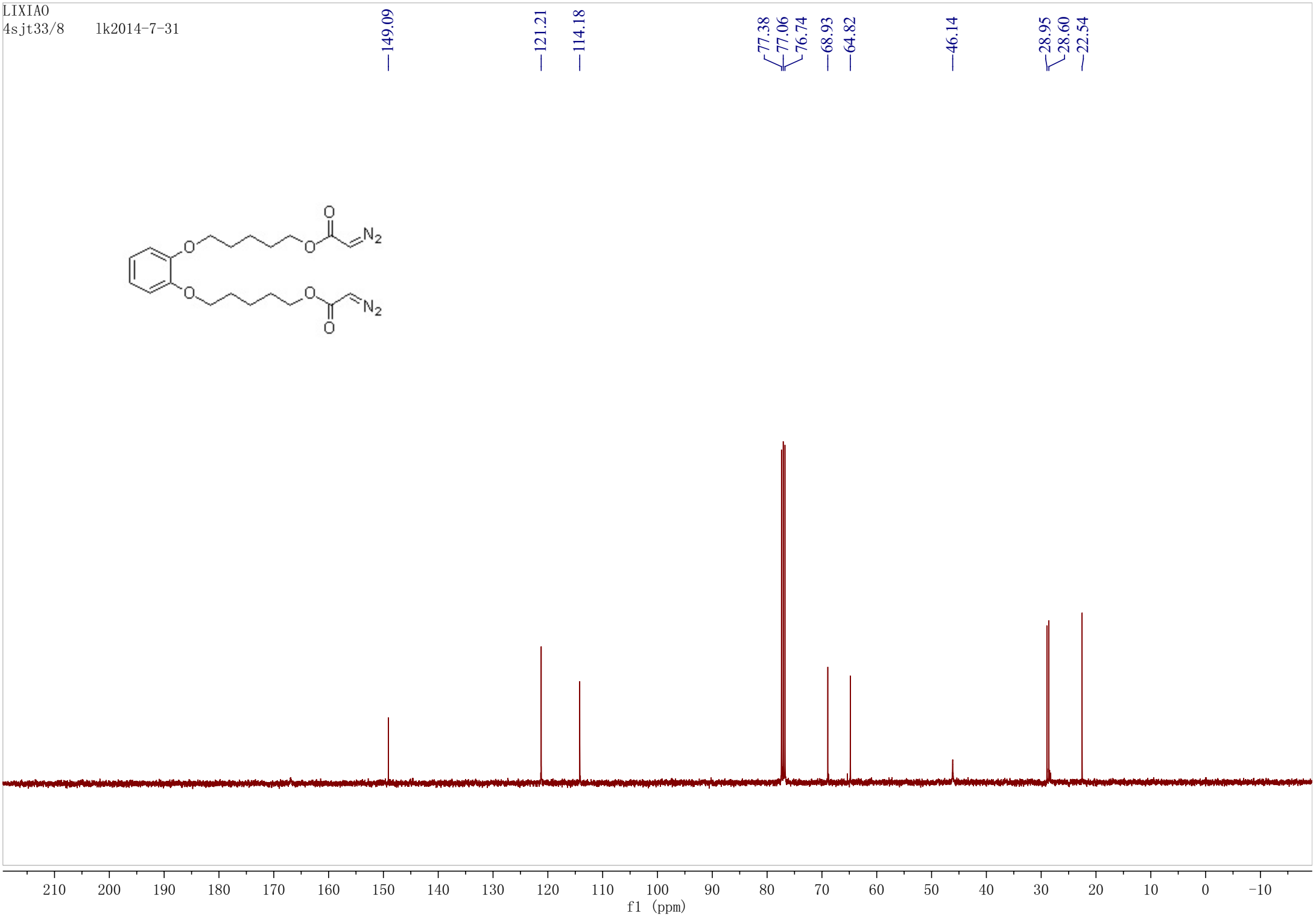

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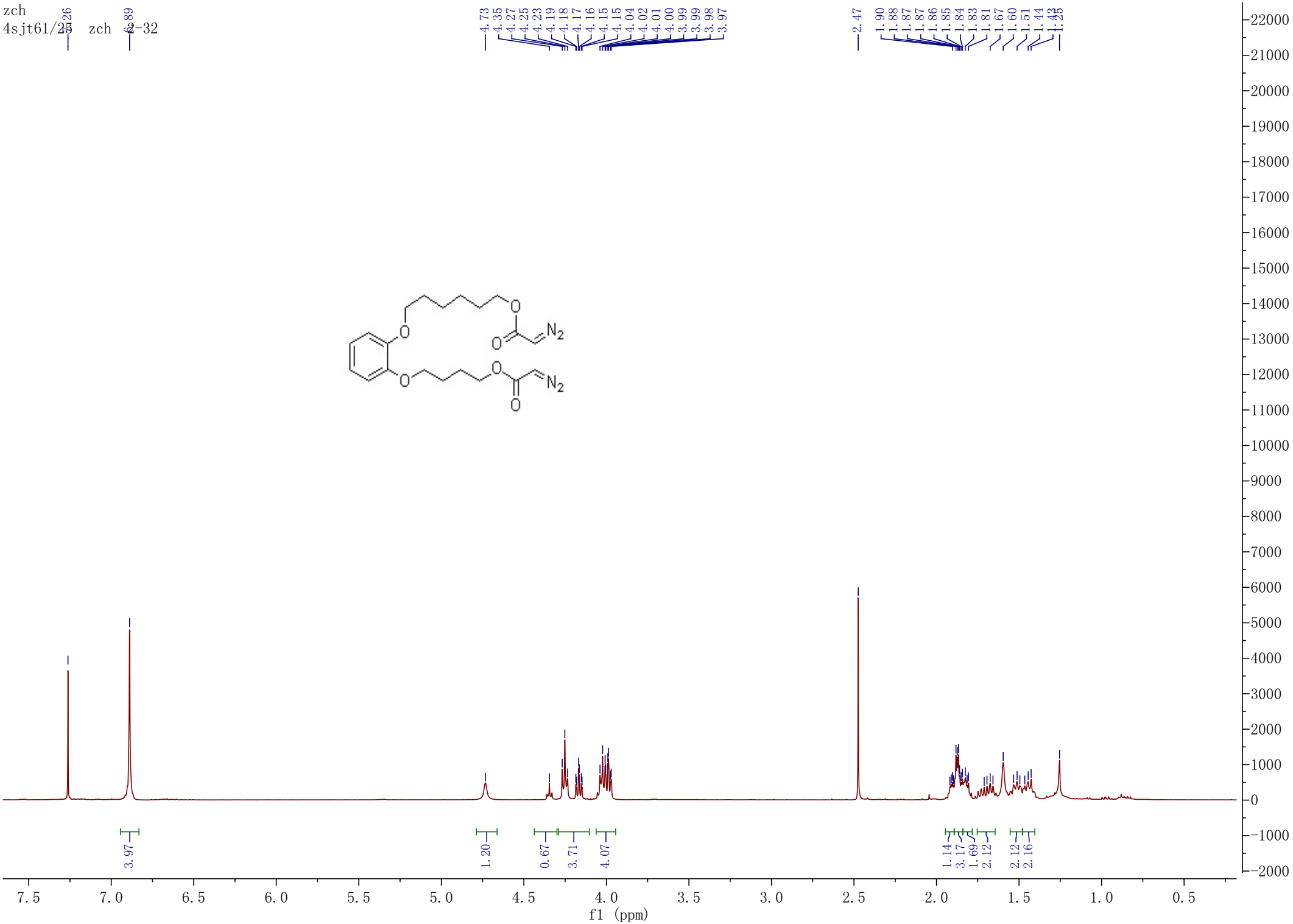

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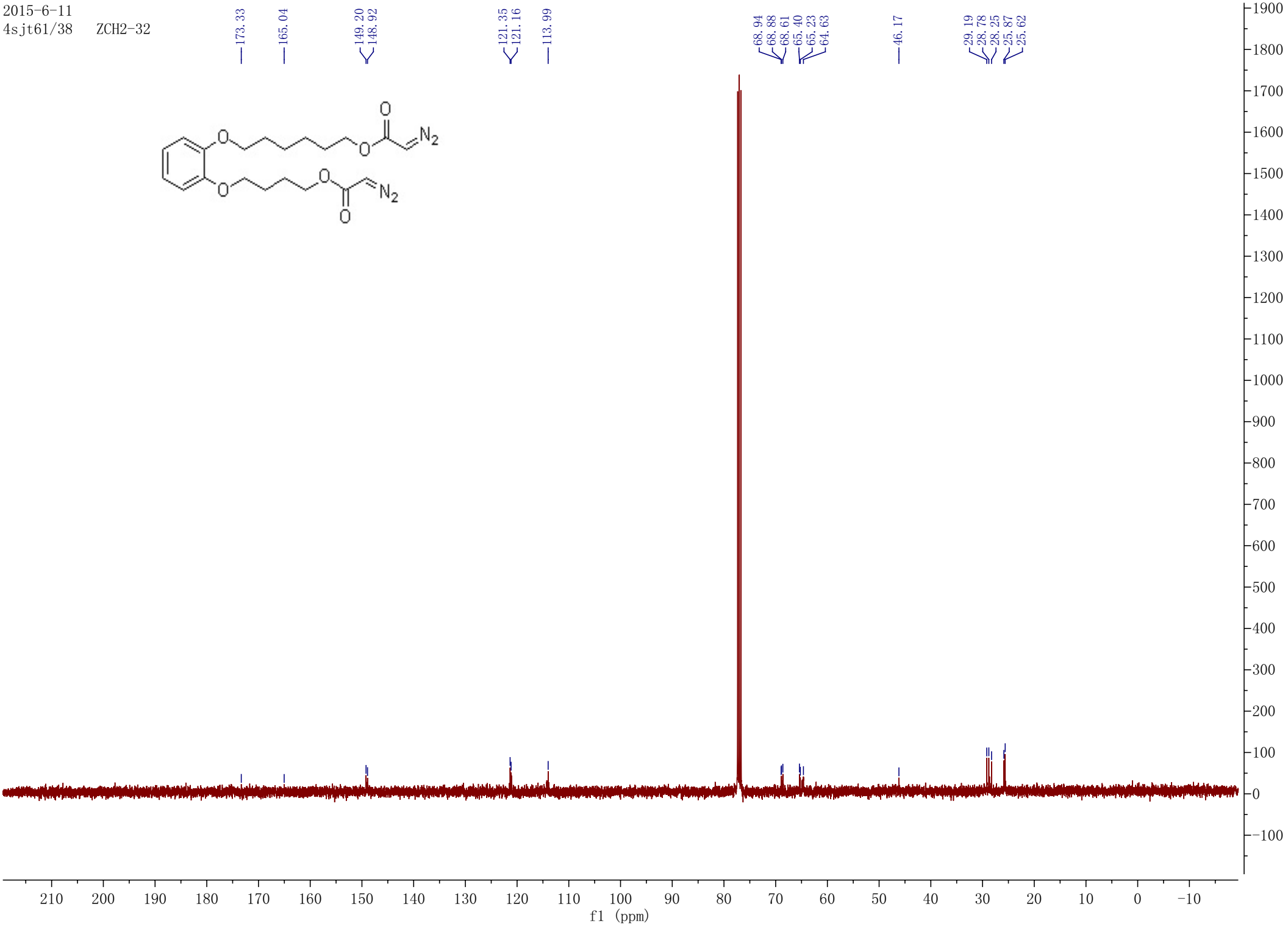

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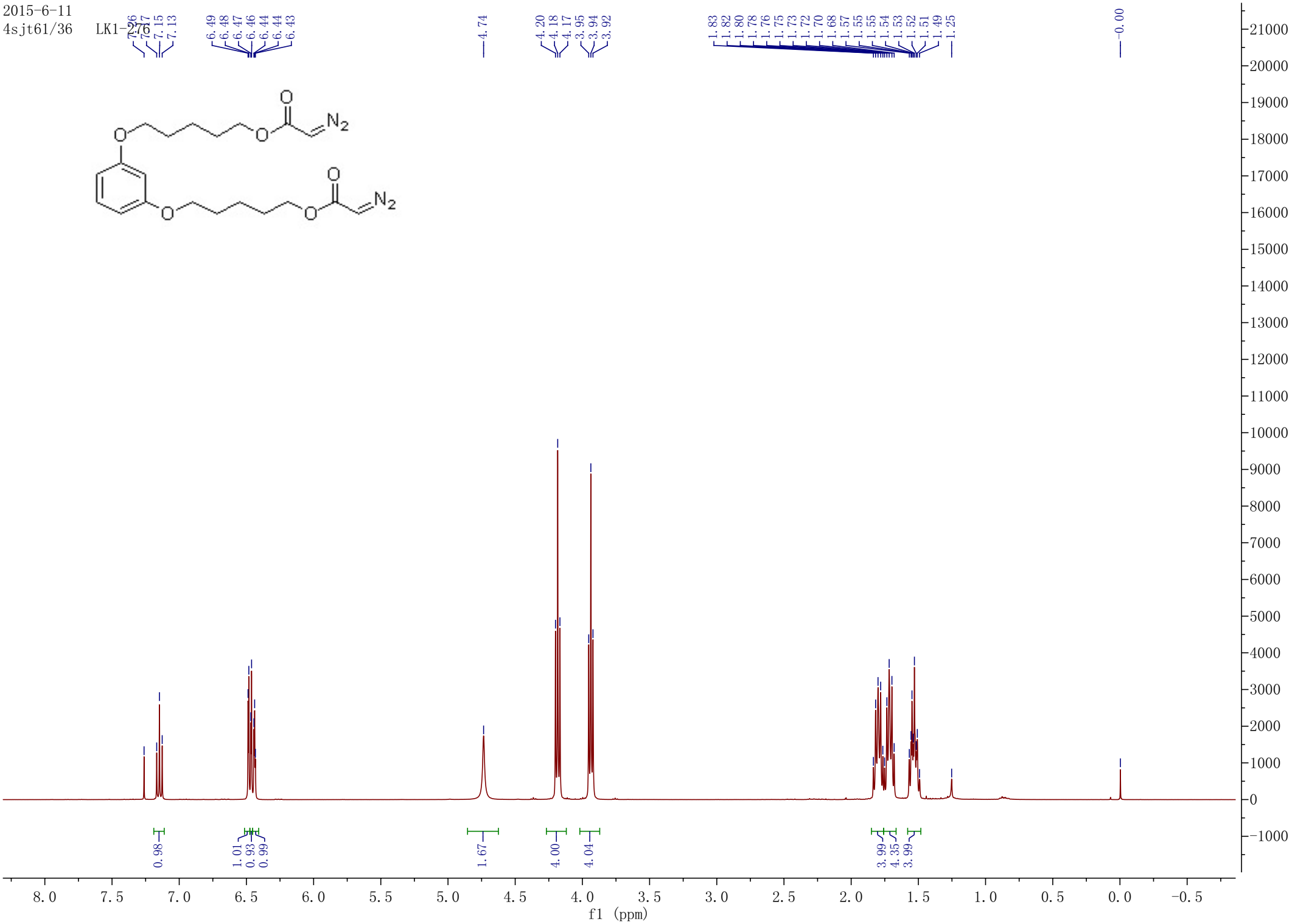

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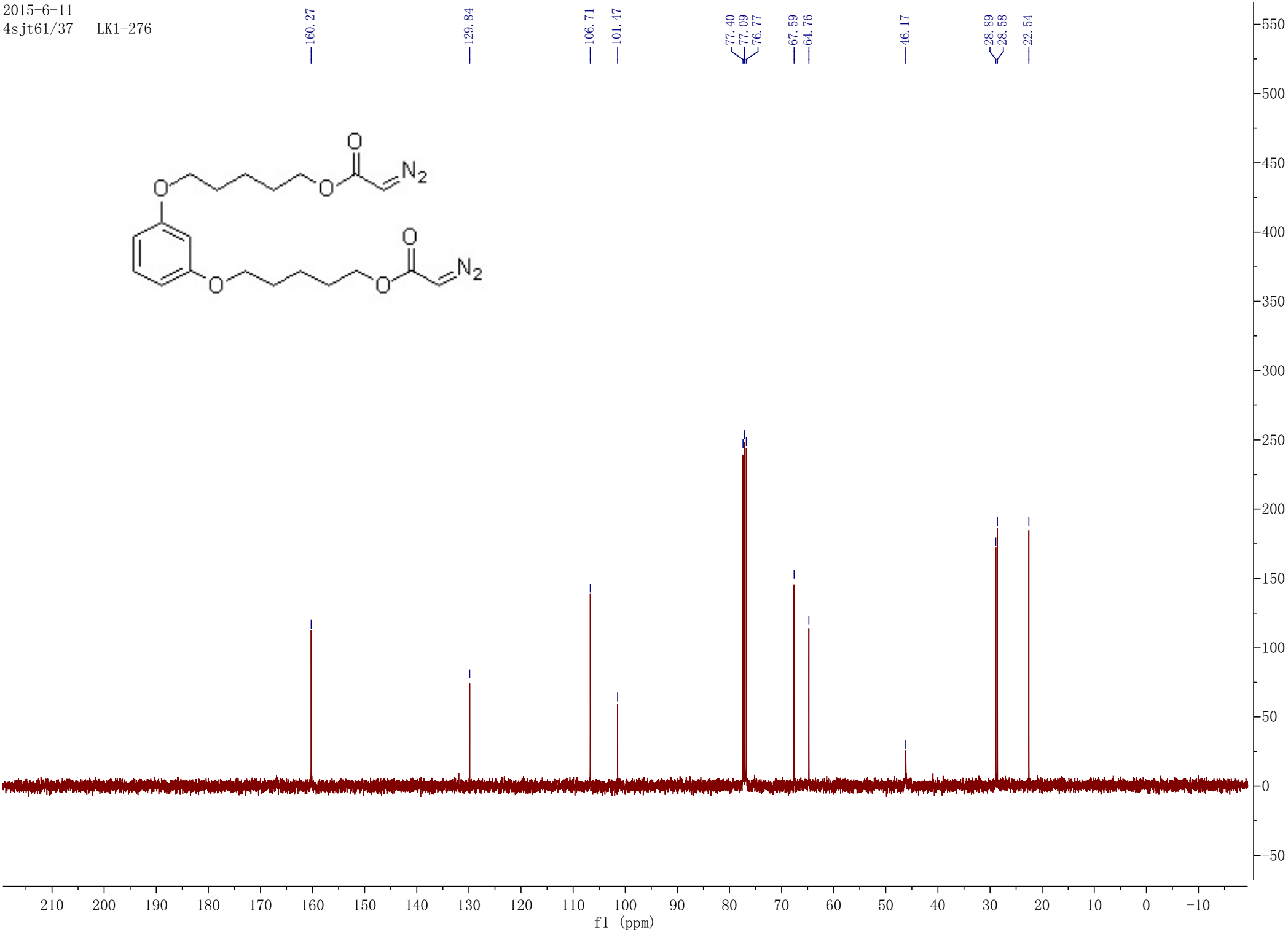

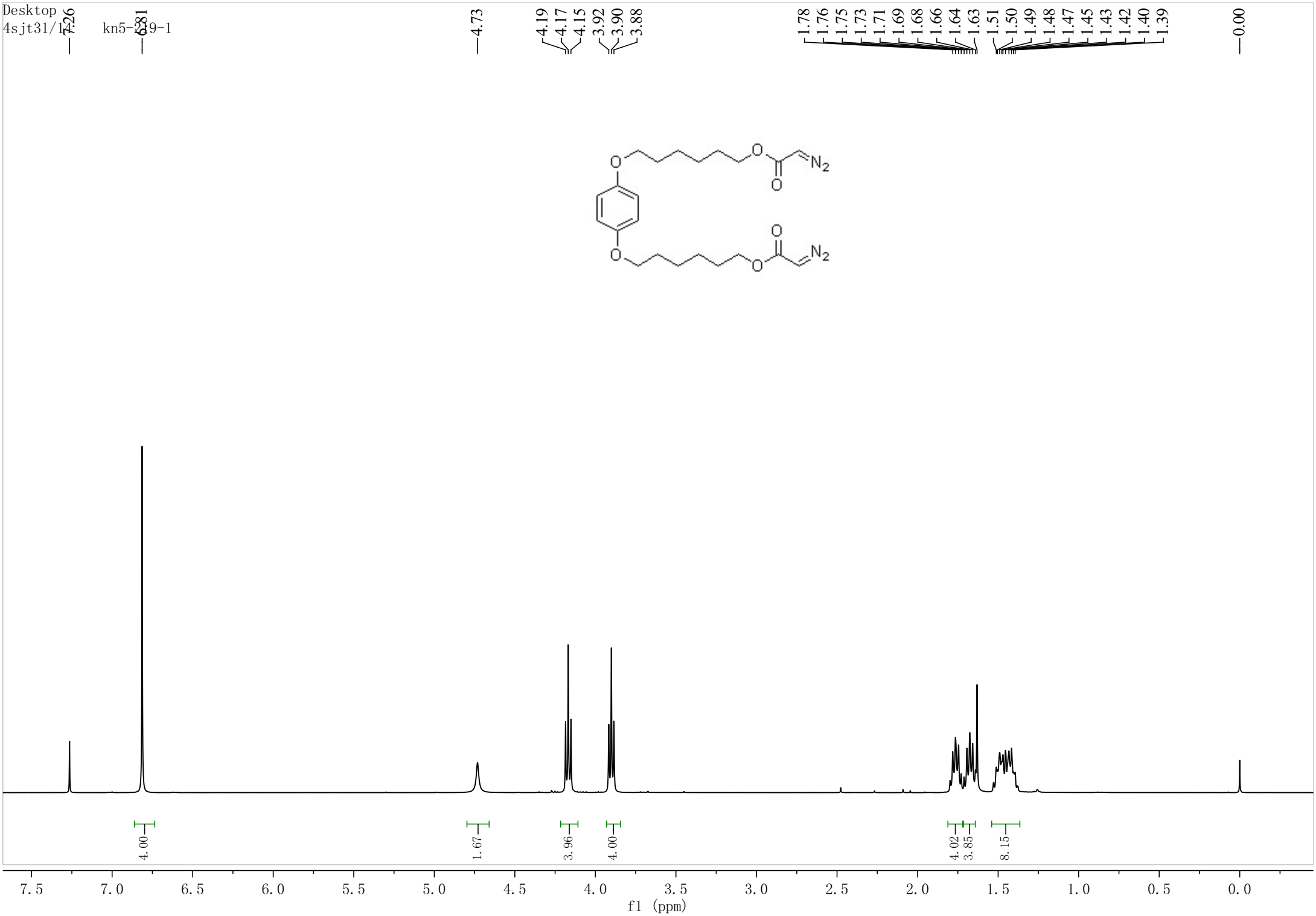
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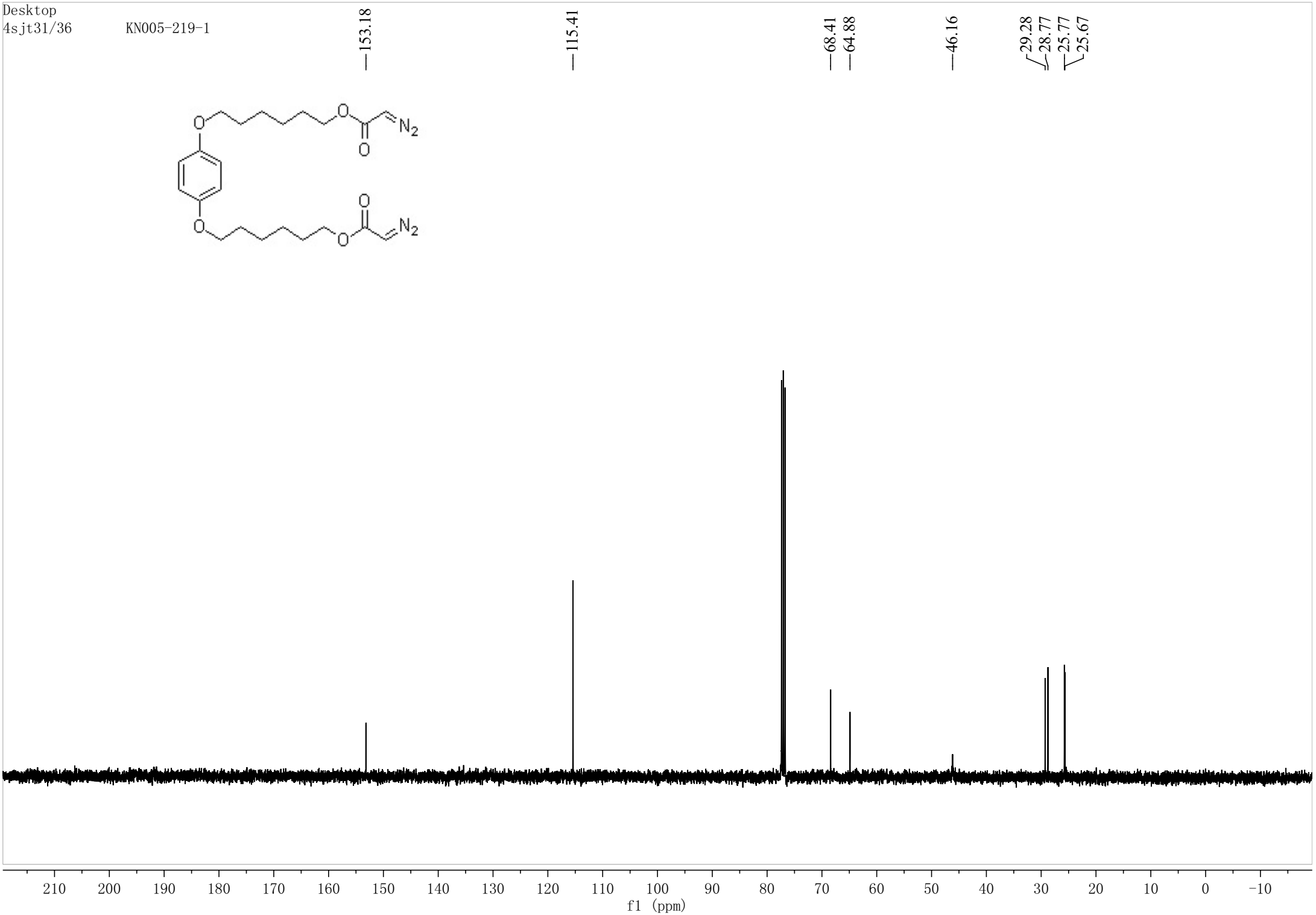

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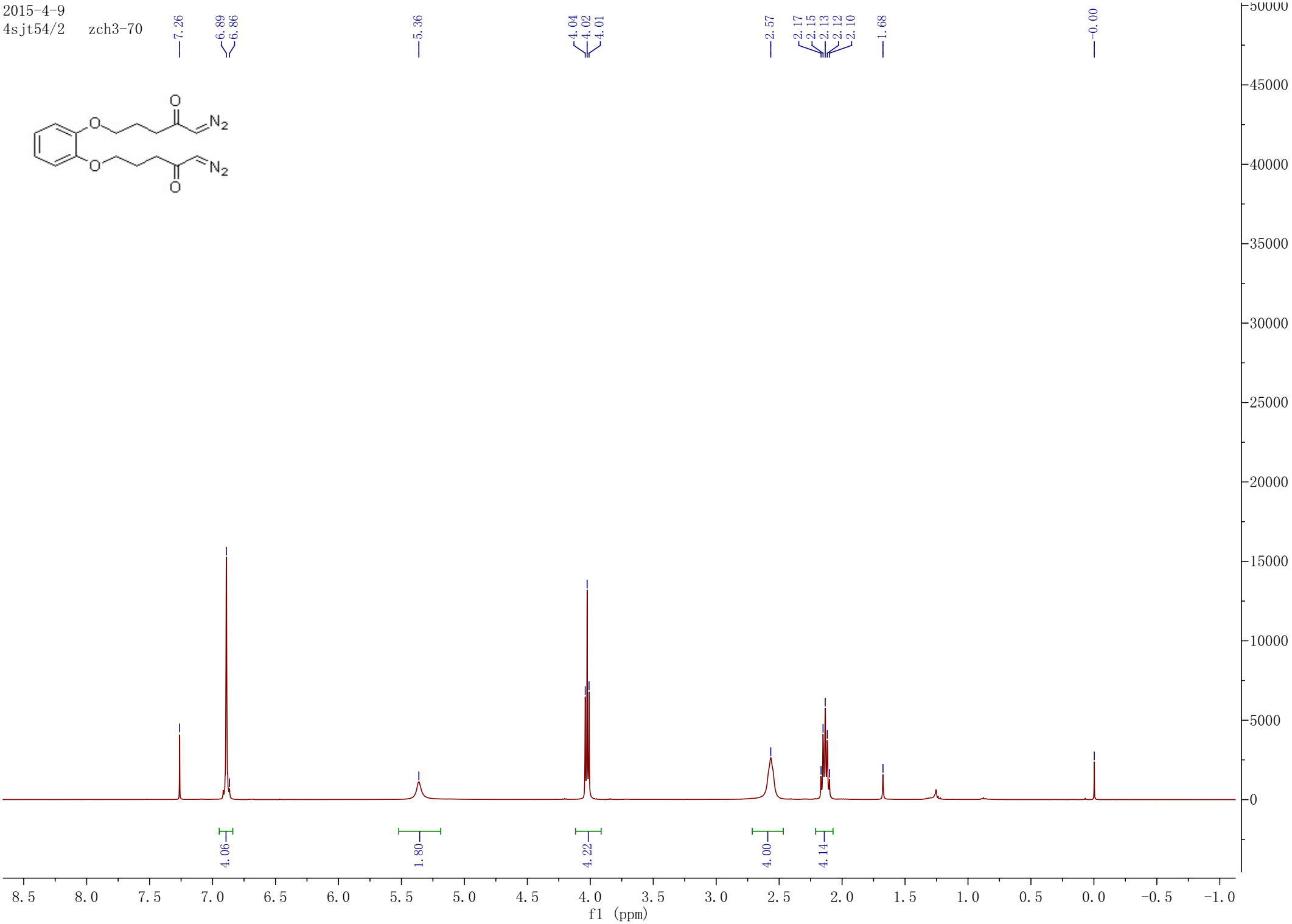

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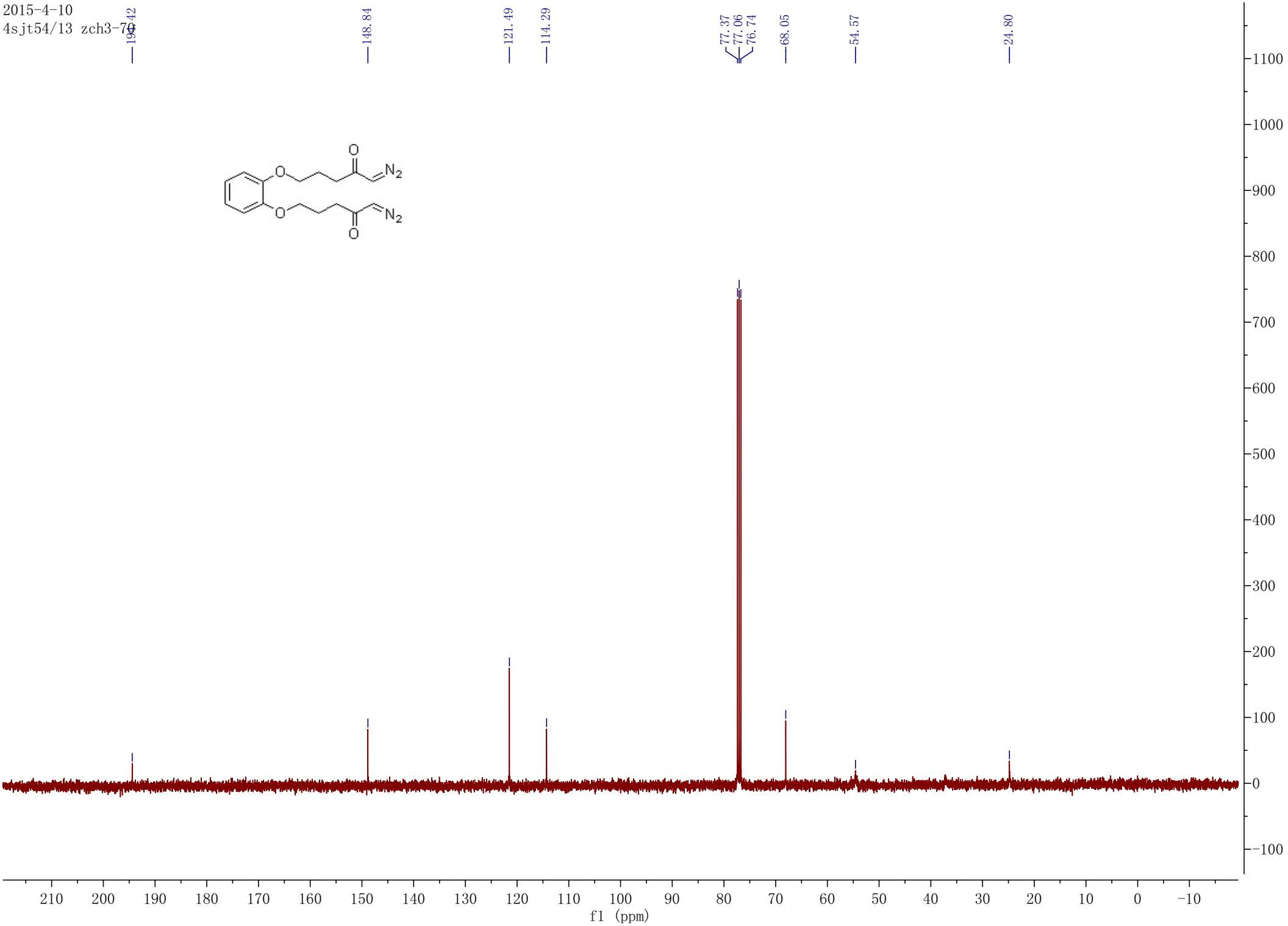

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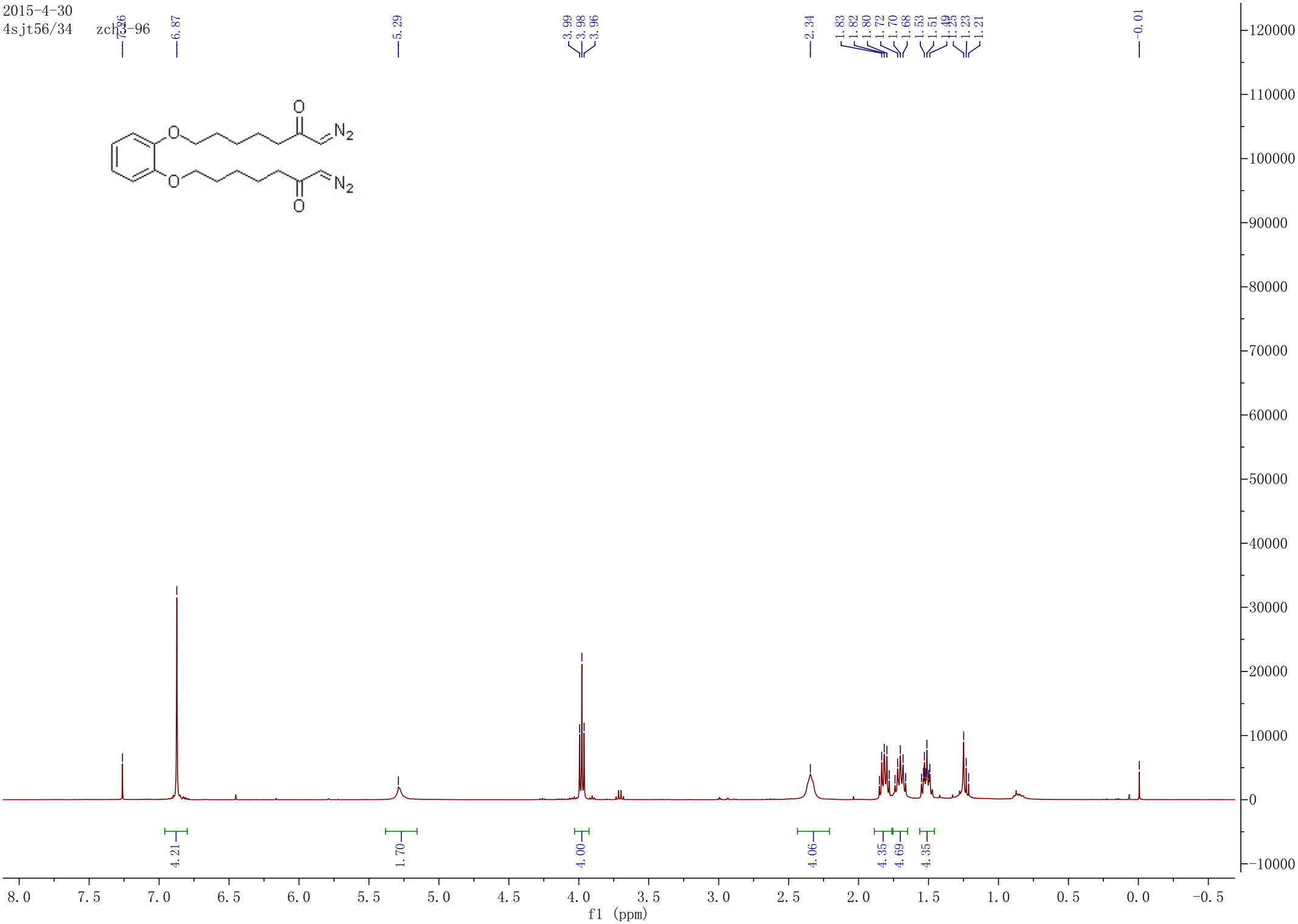

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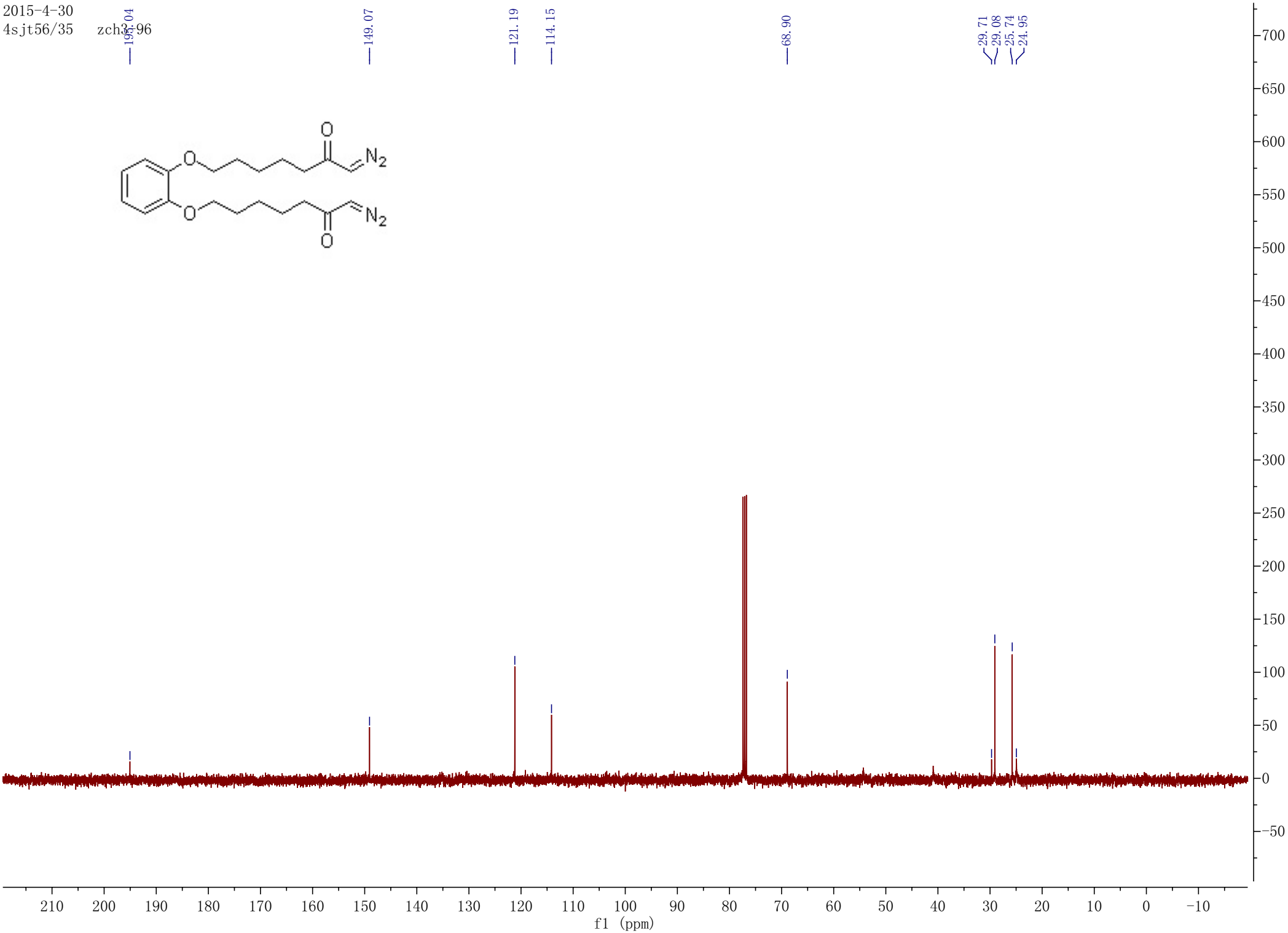

146-H
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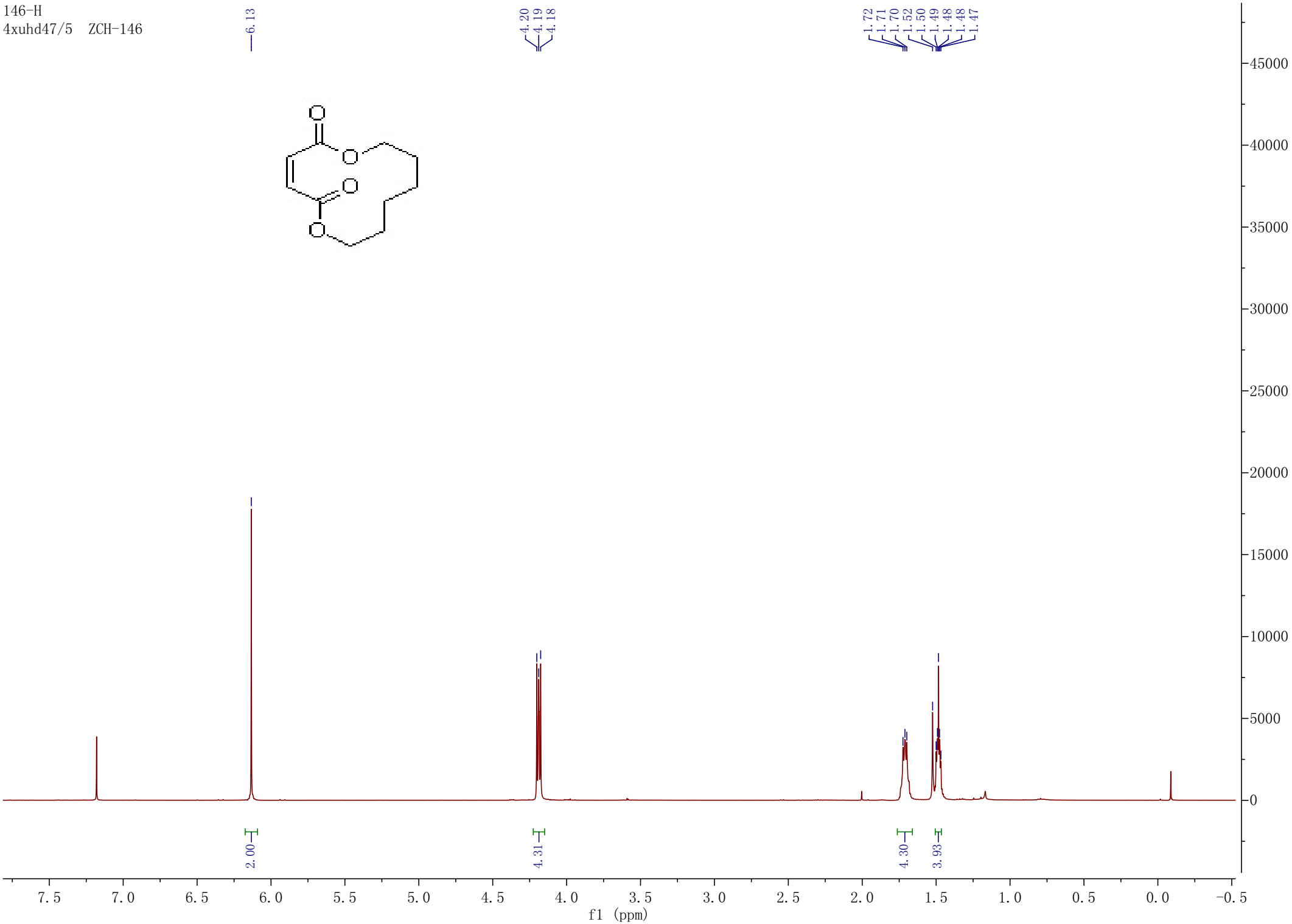

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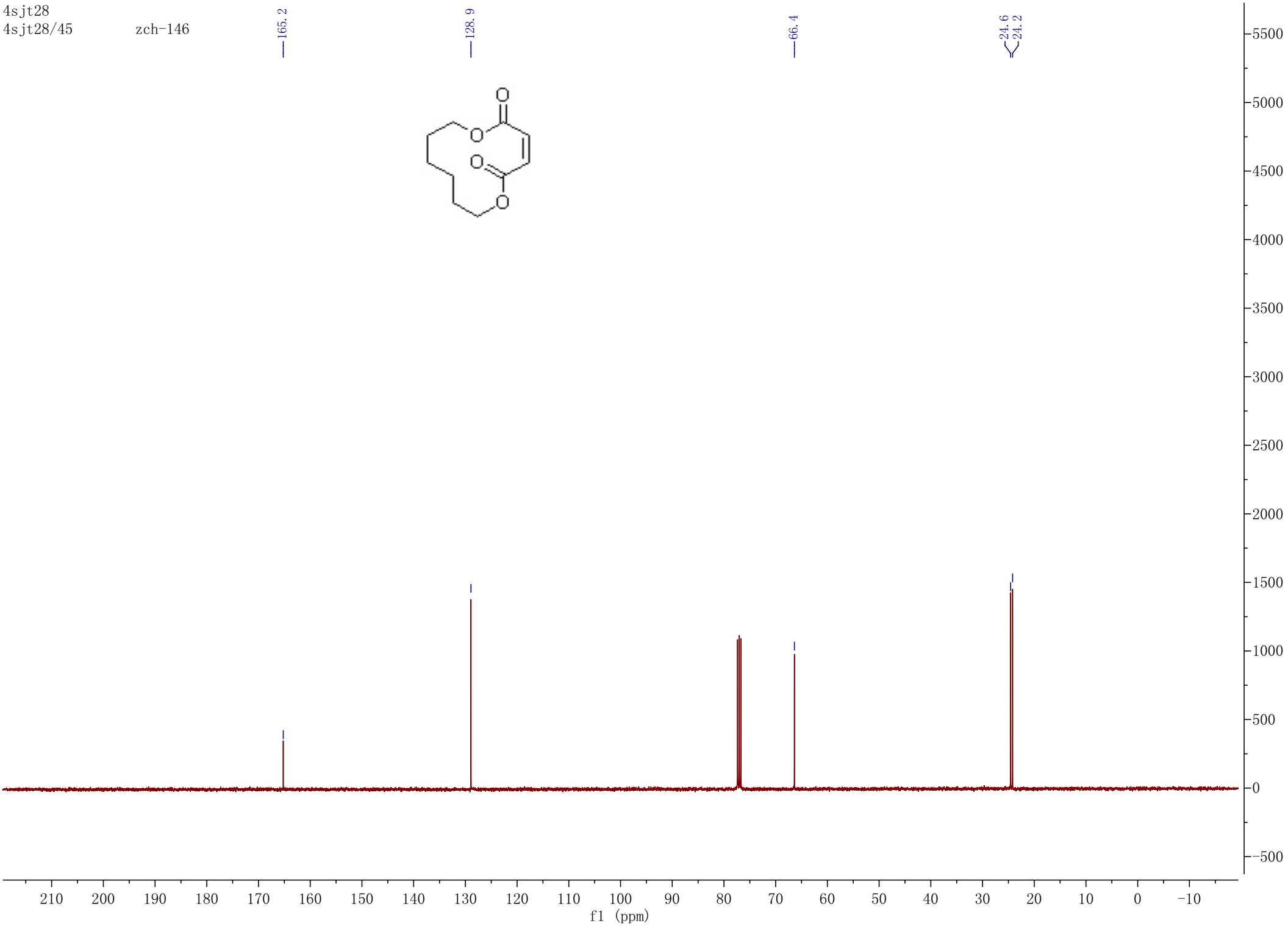

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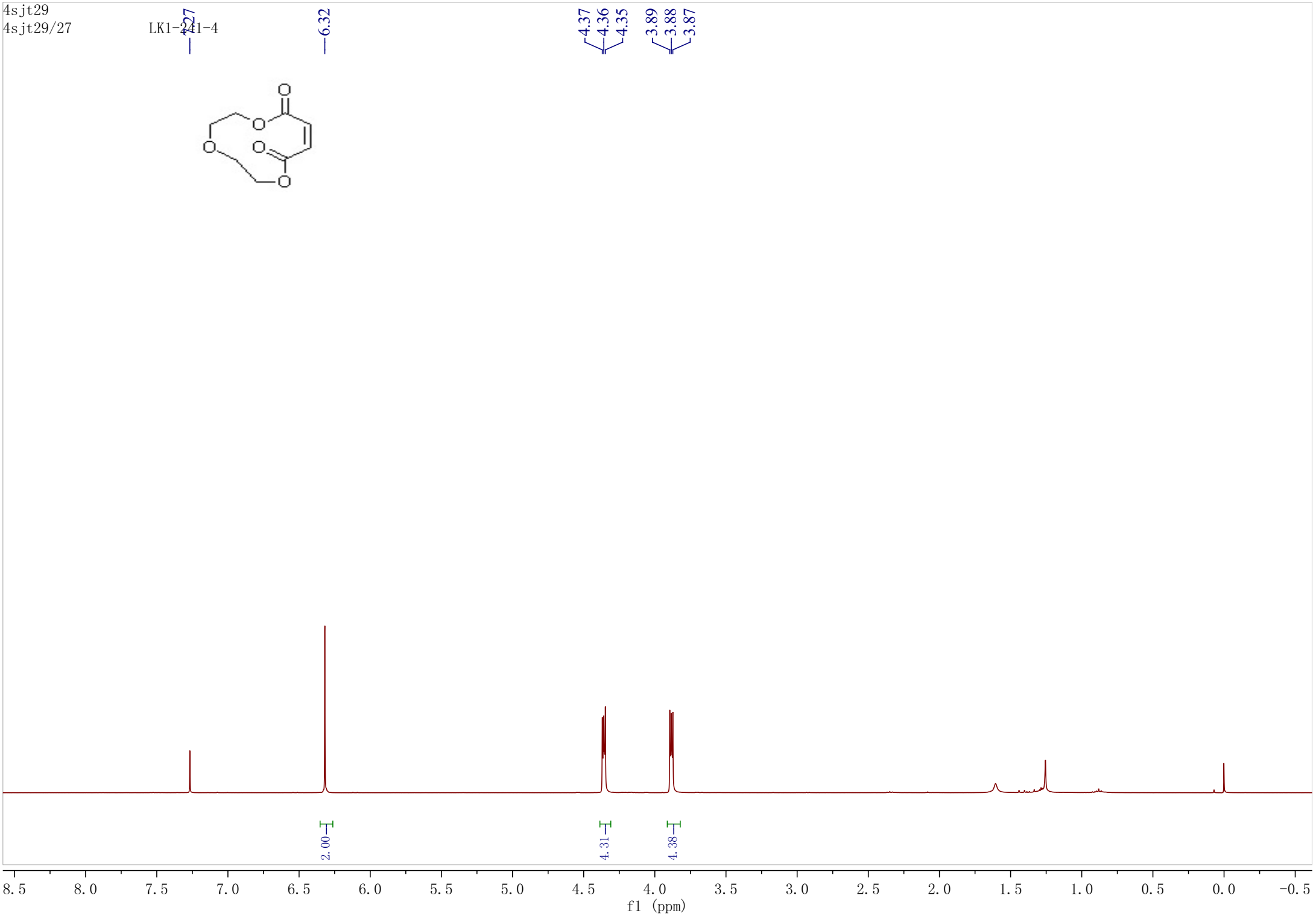

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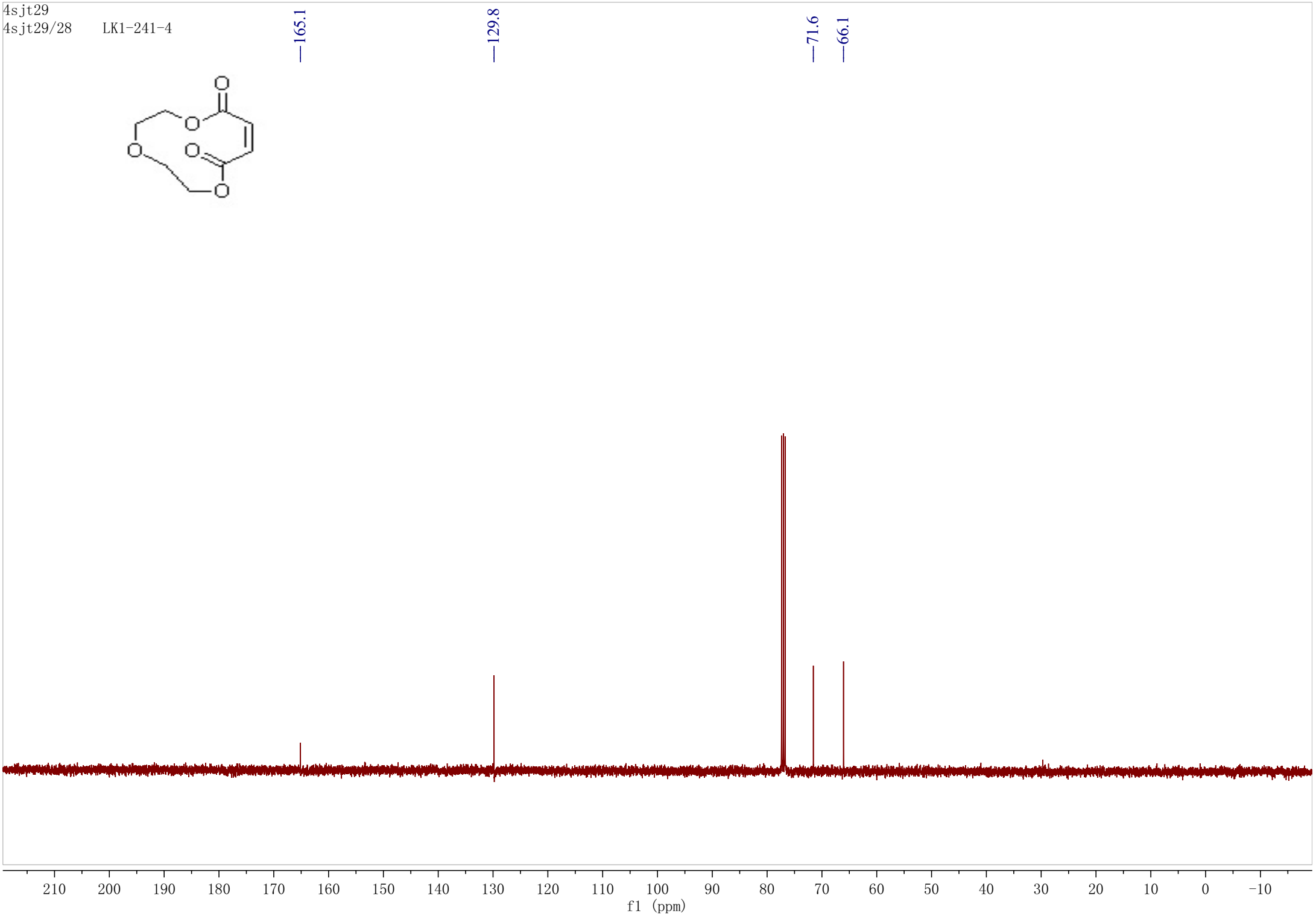

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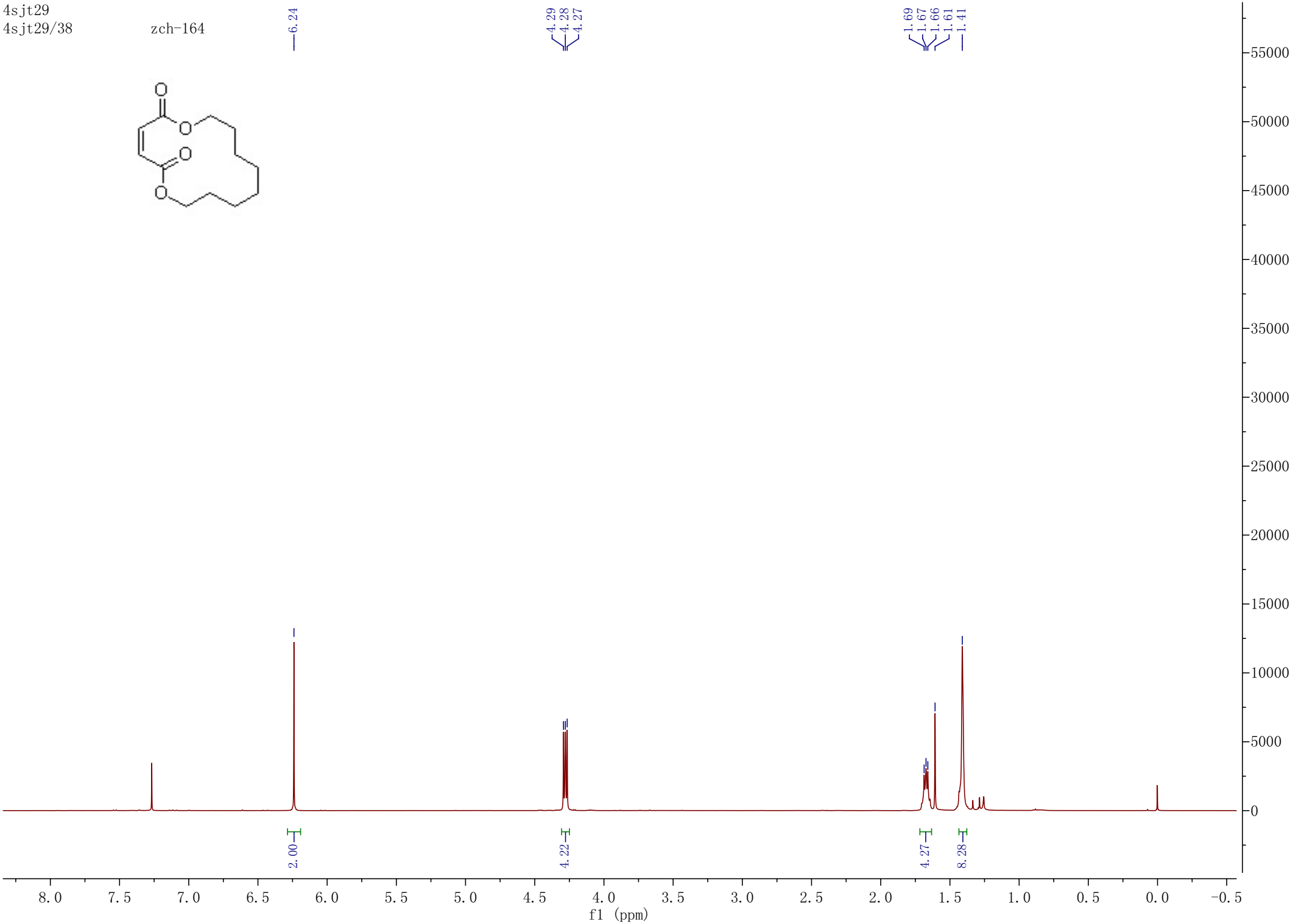

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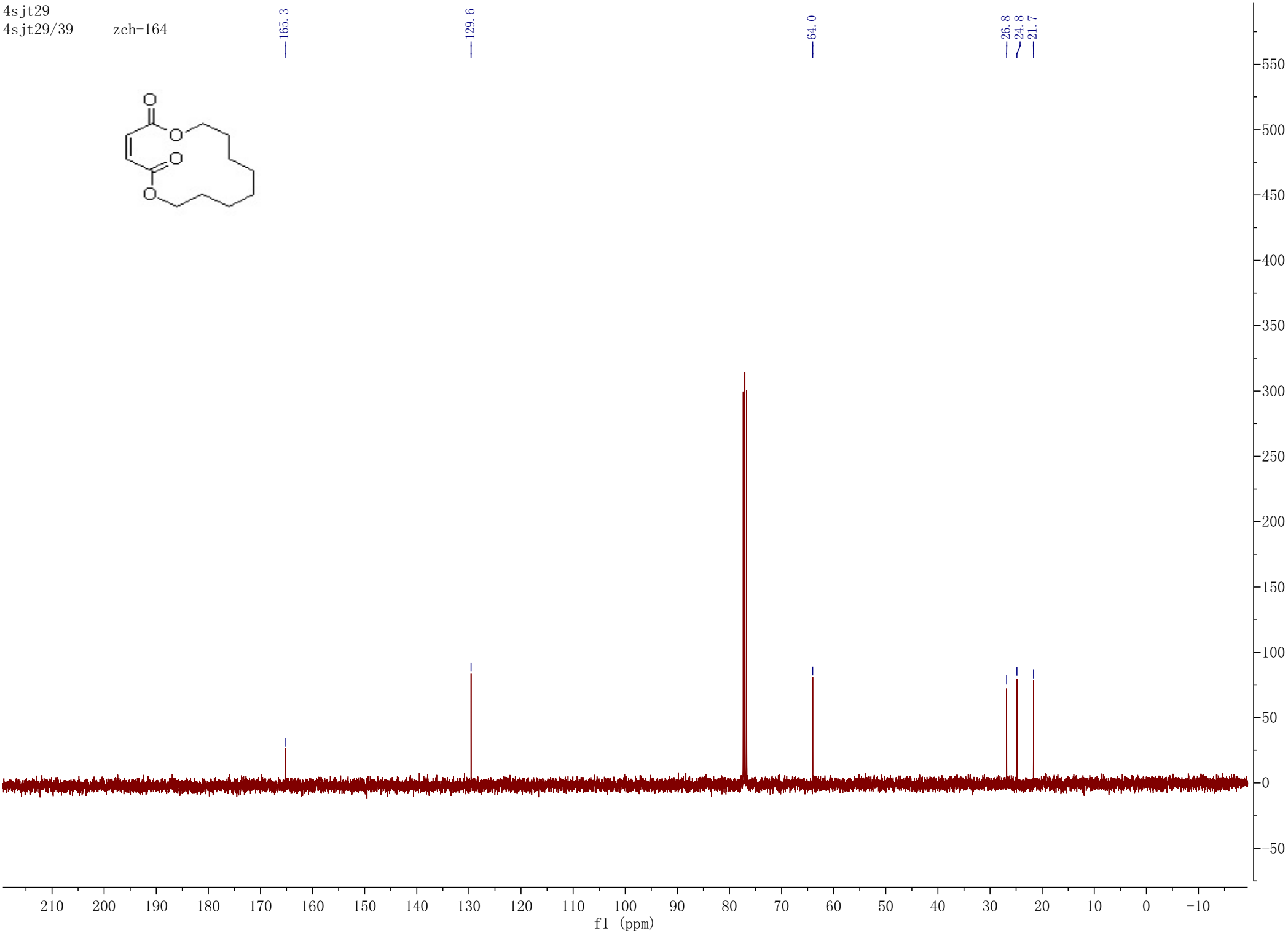

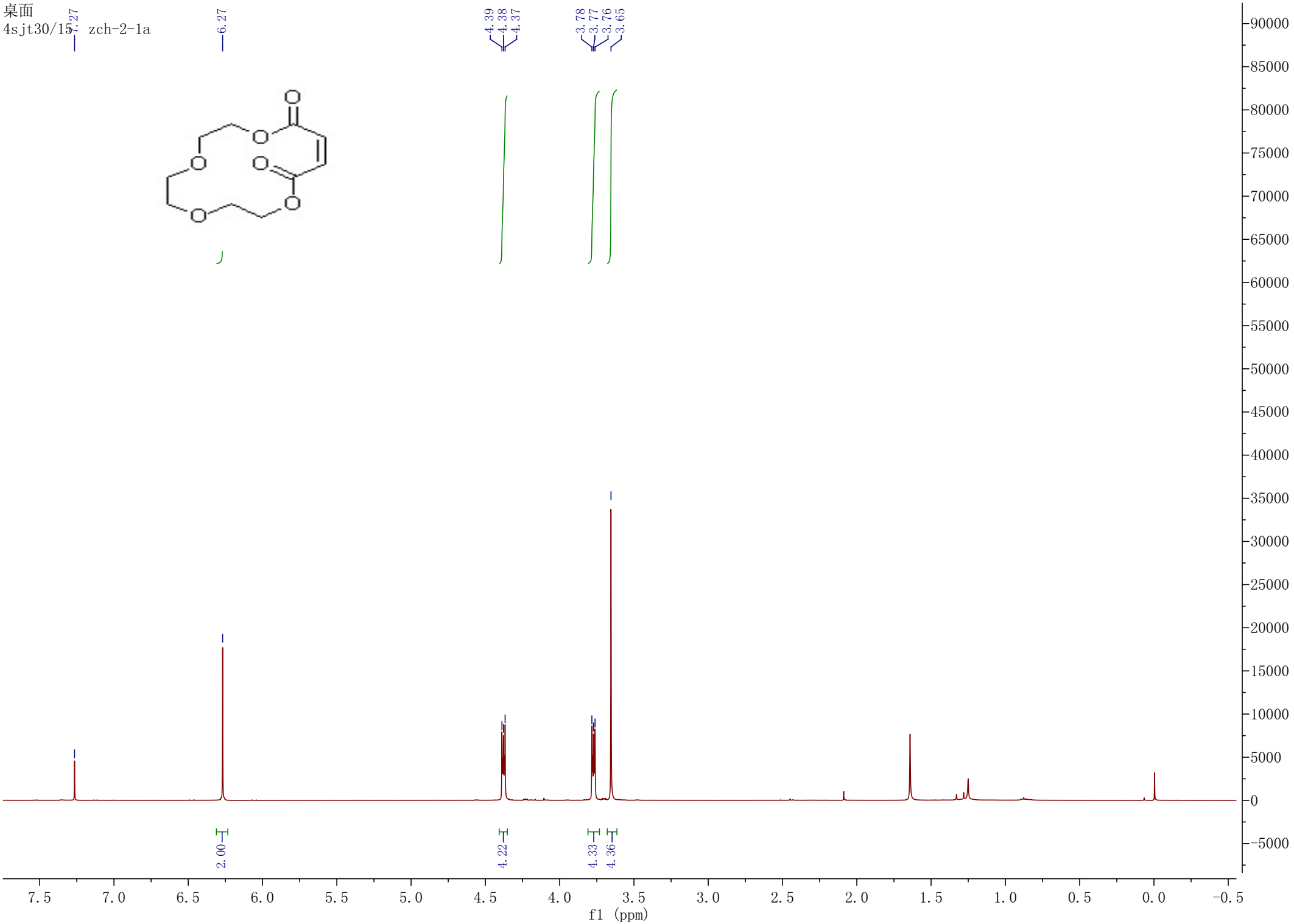
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f1 (ppm)

2014-6-30
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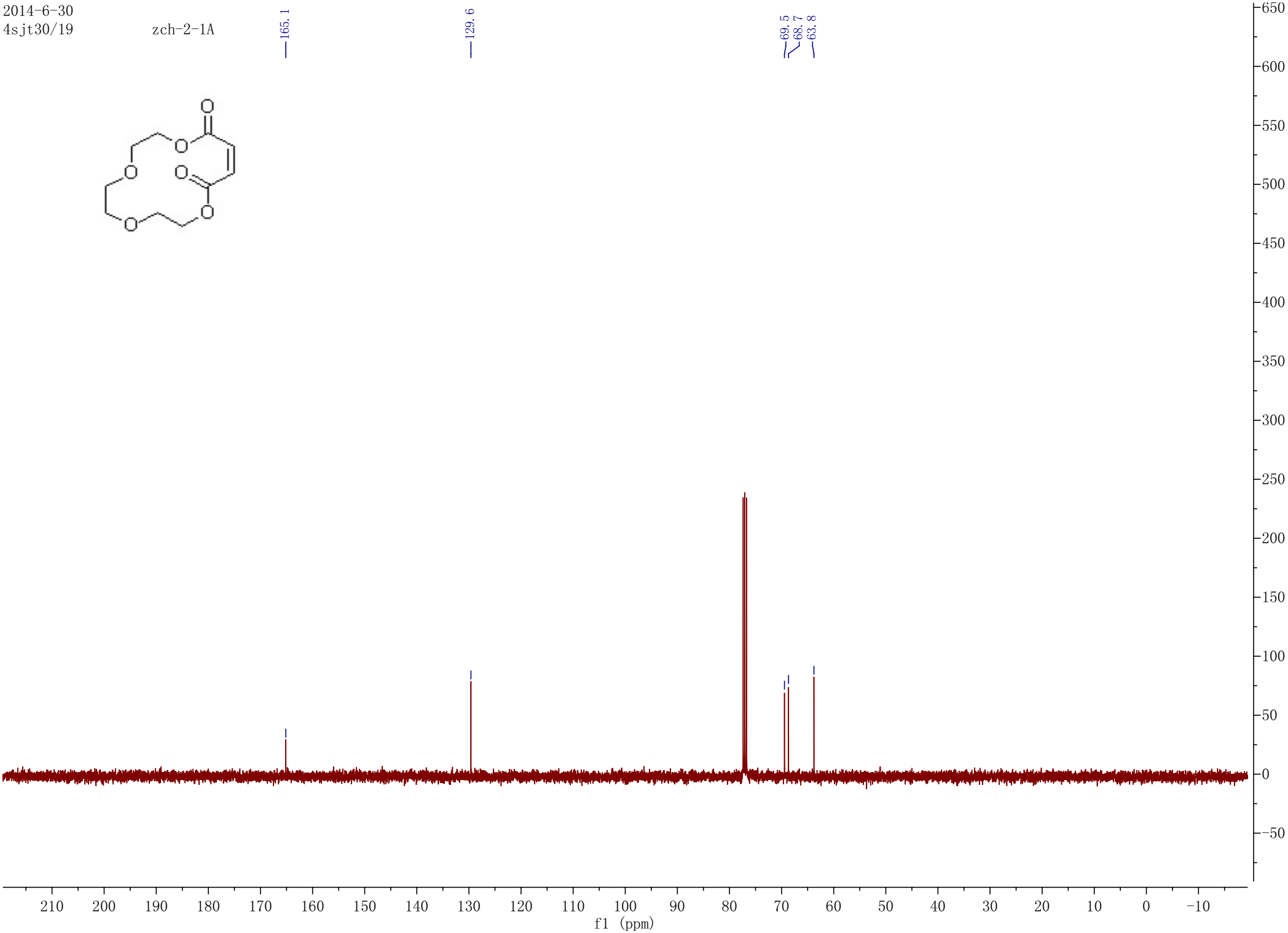

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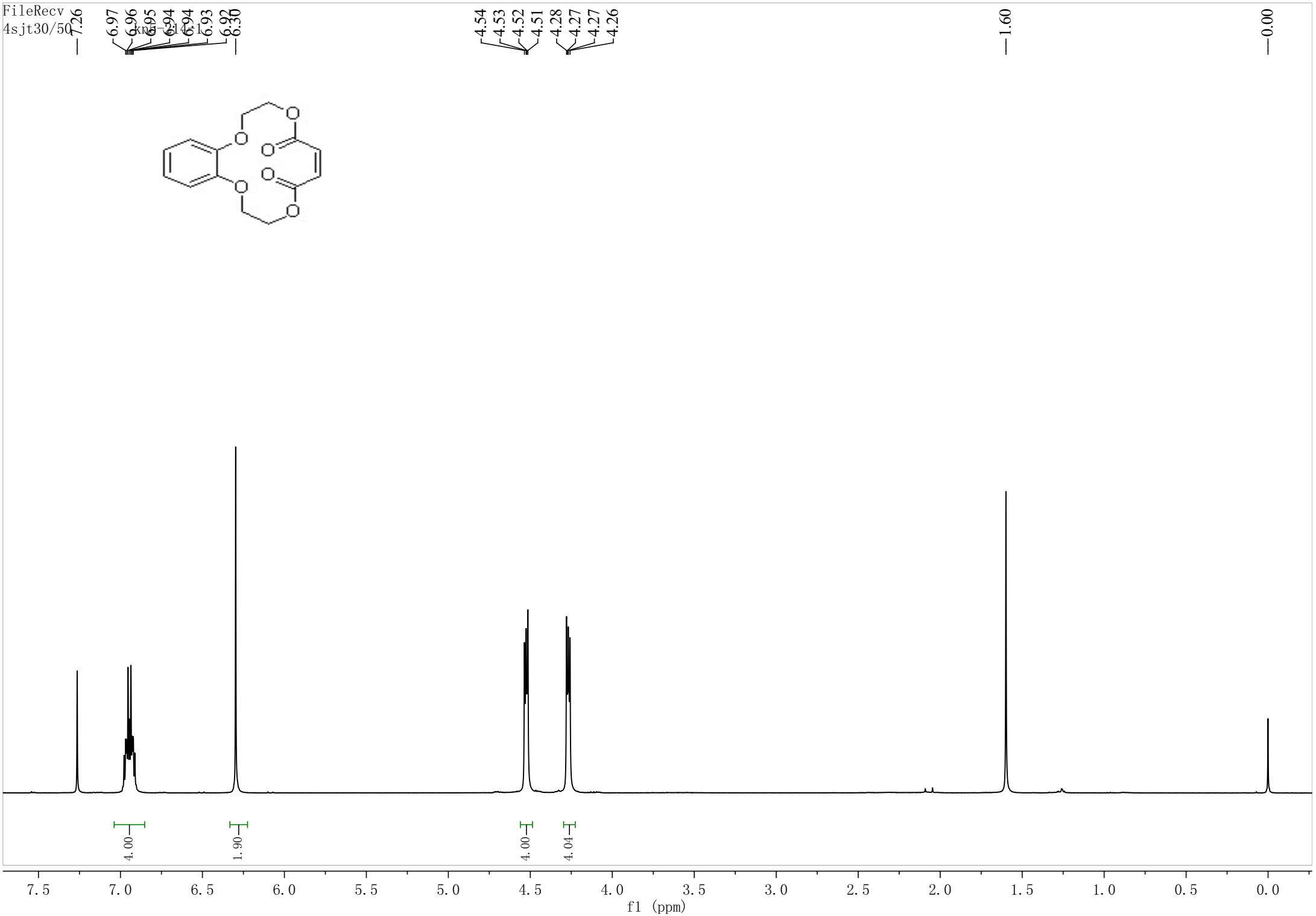

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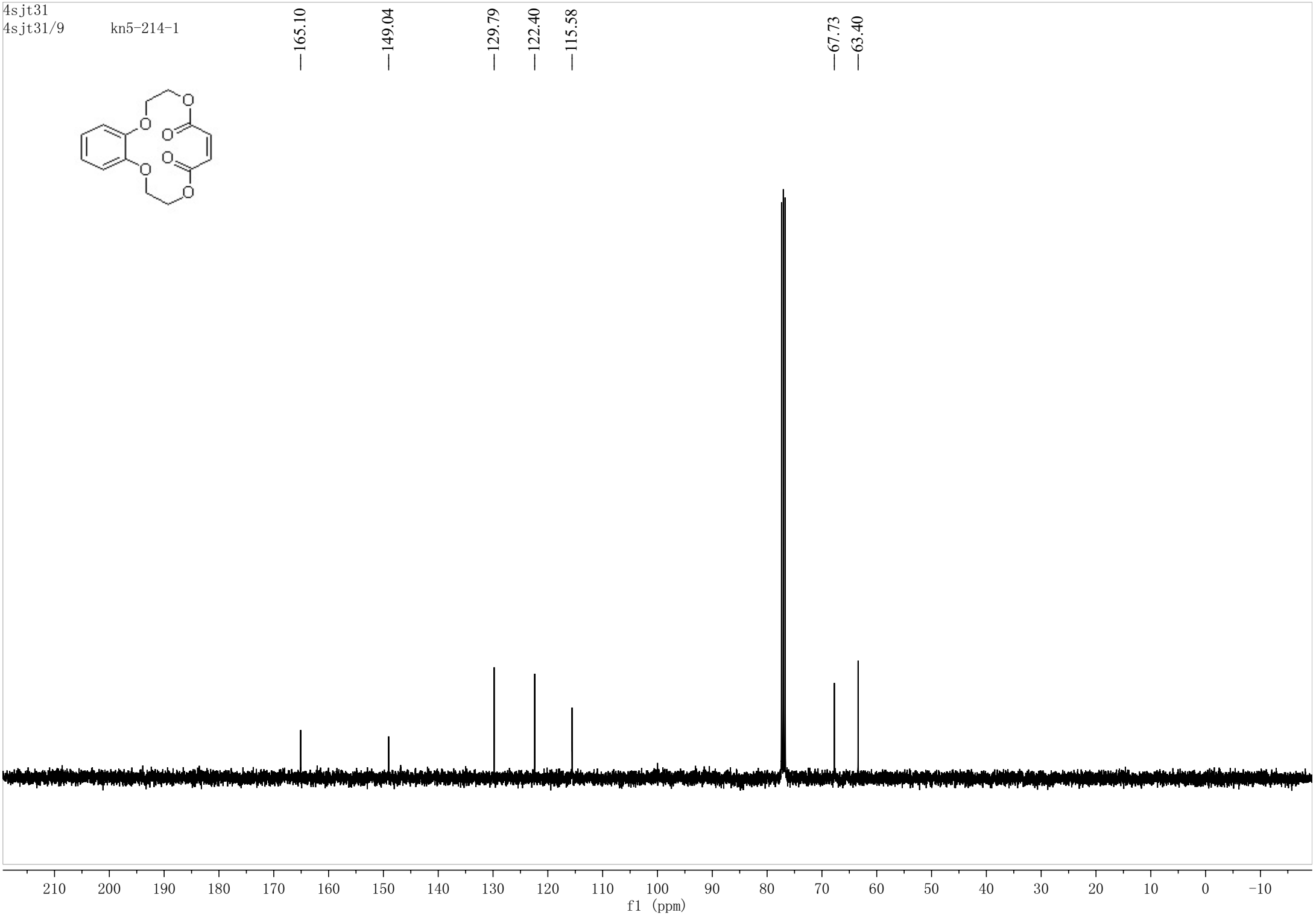

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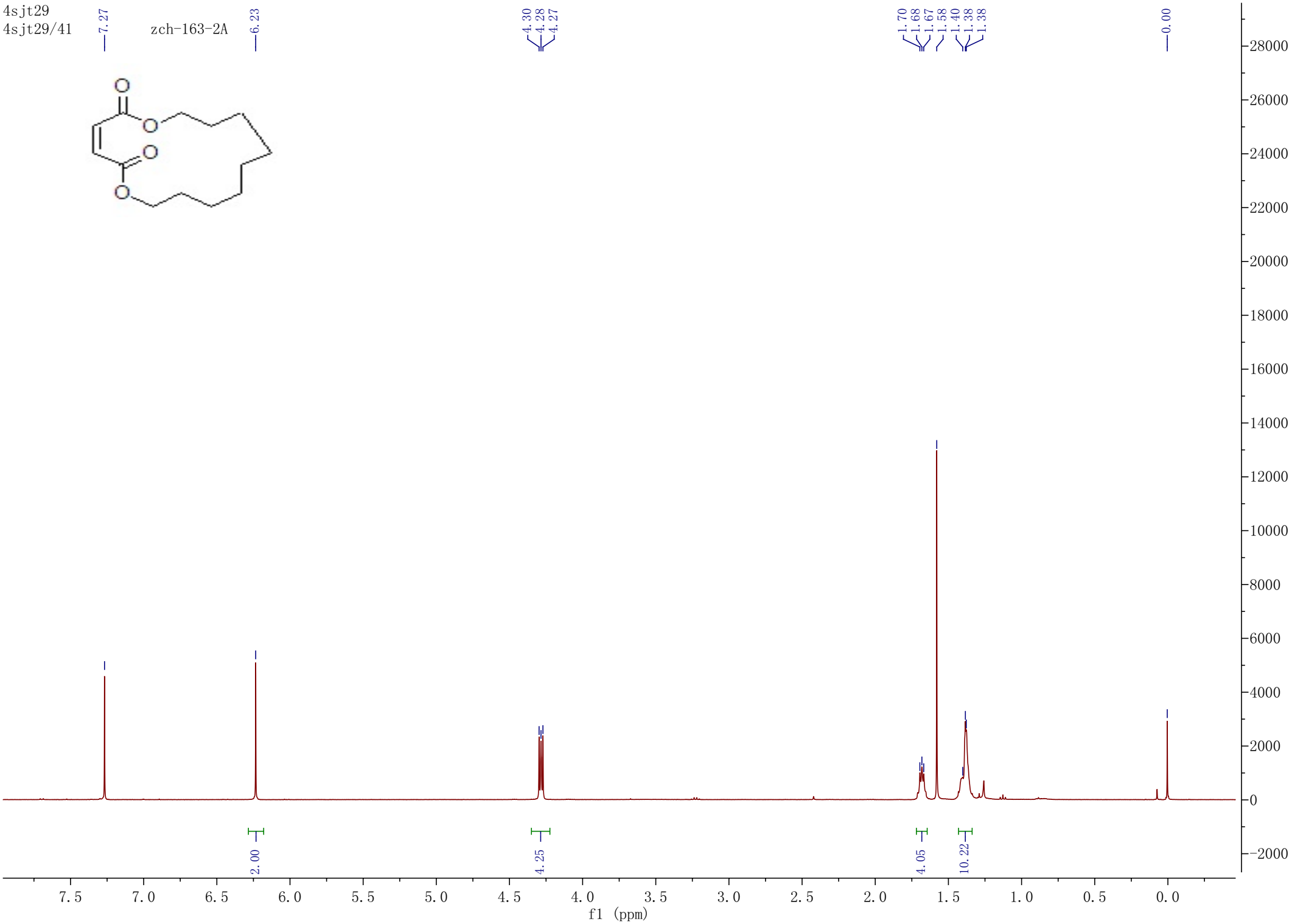

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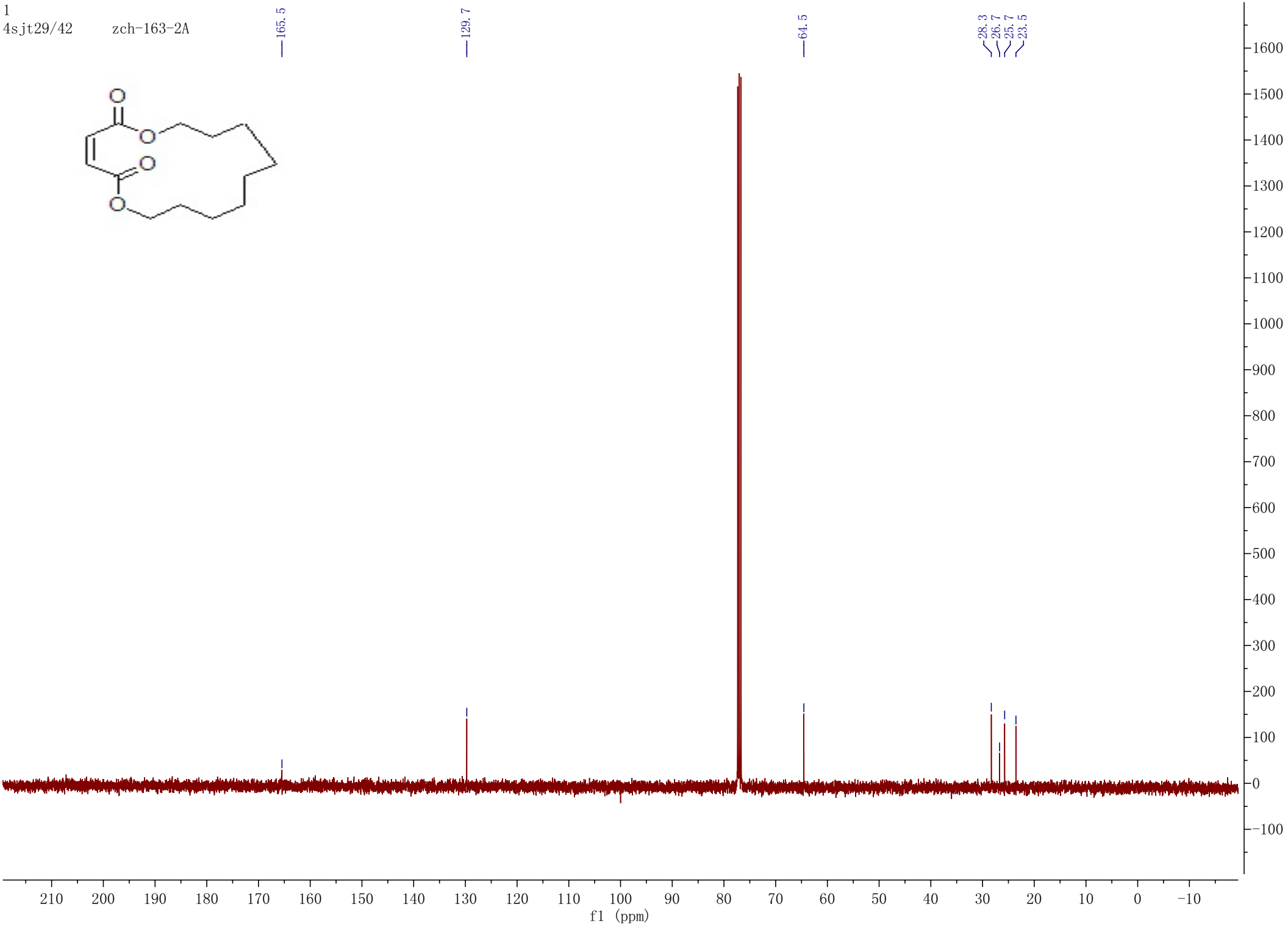
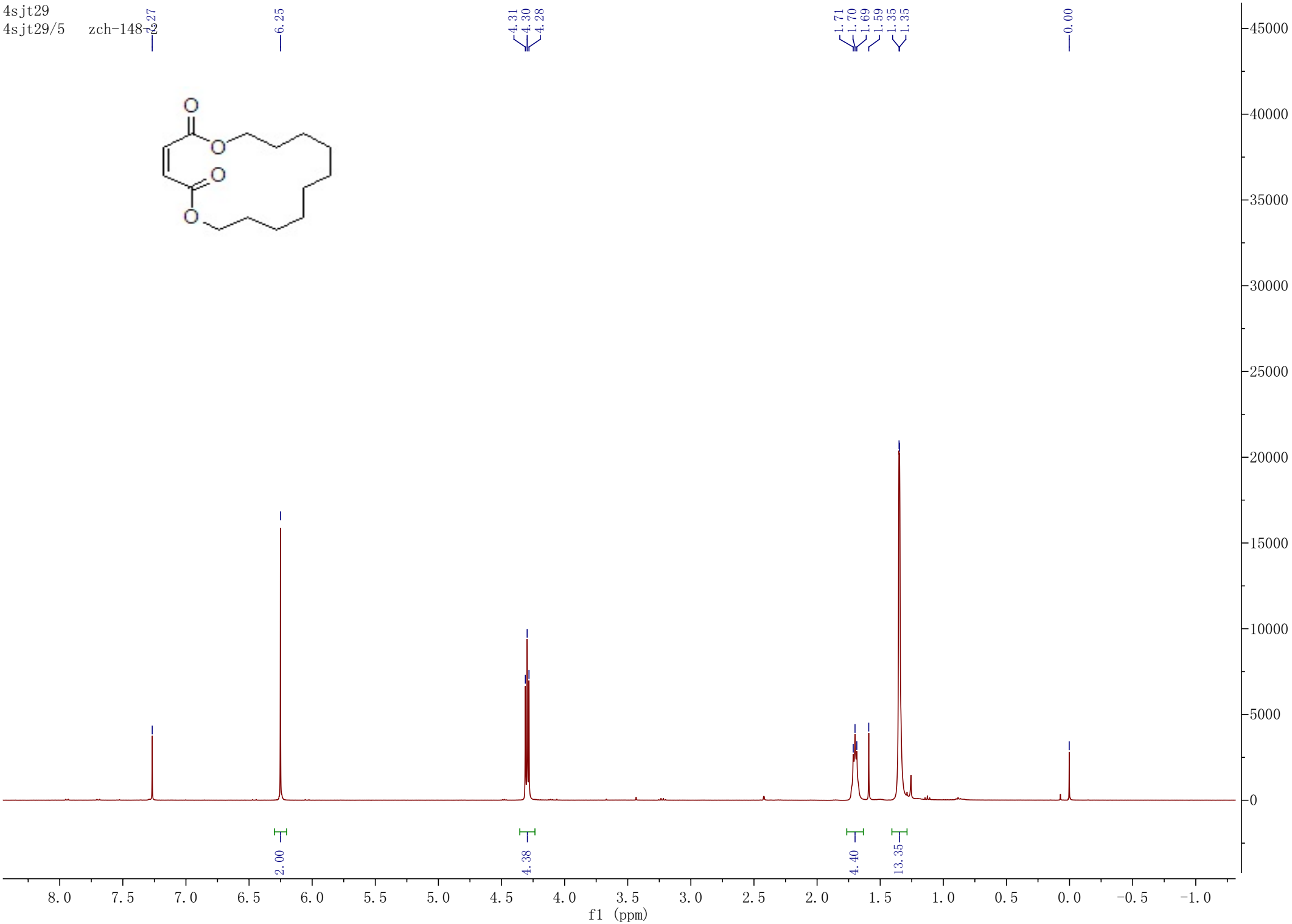
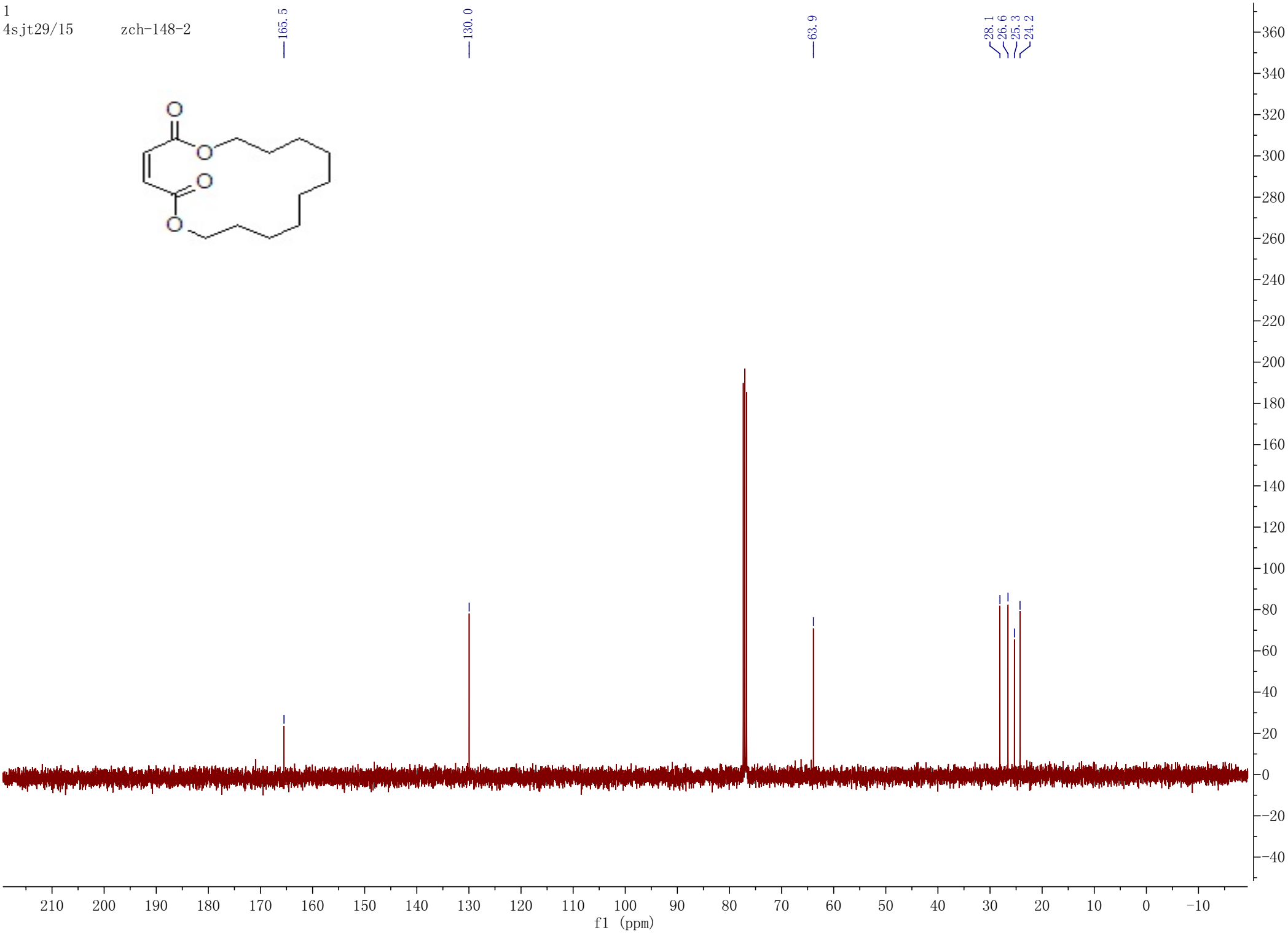
4sjt29/42 zch-163-2A



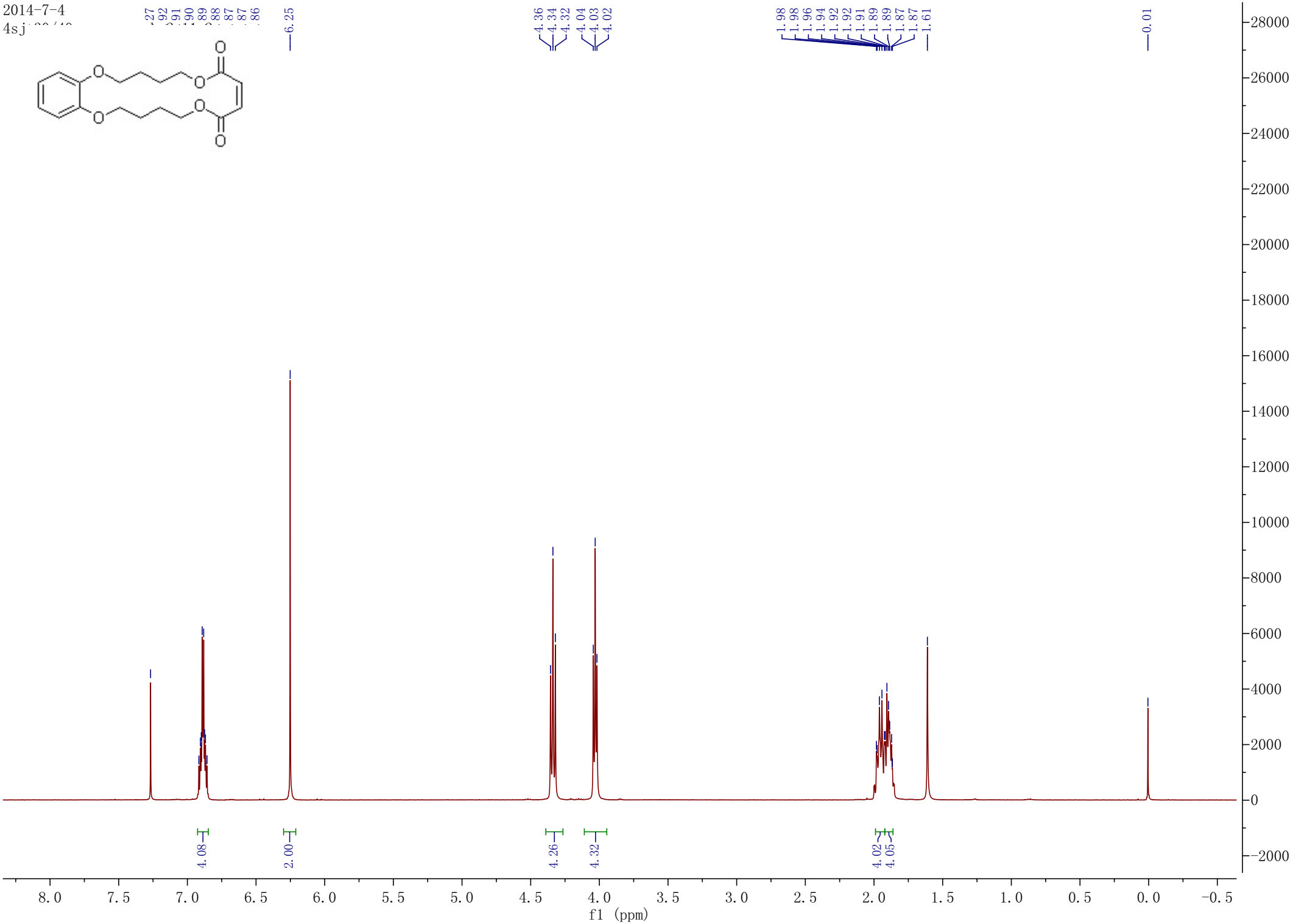
4sjt29
4sjt29/5 zch-148-2



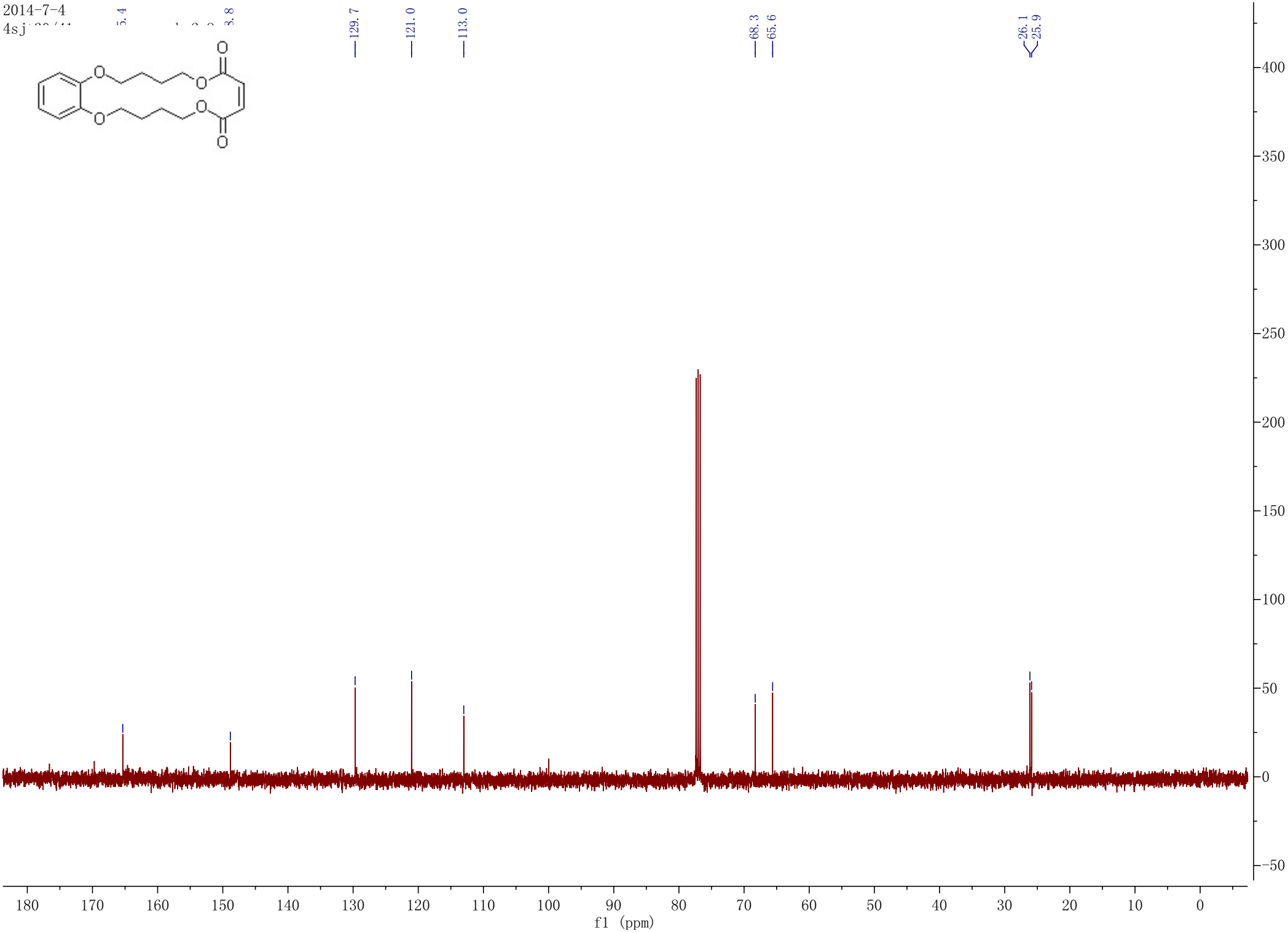
1
4sjt29/15 zch-148-2



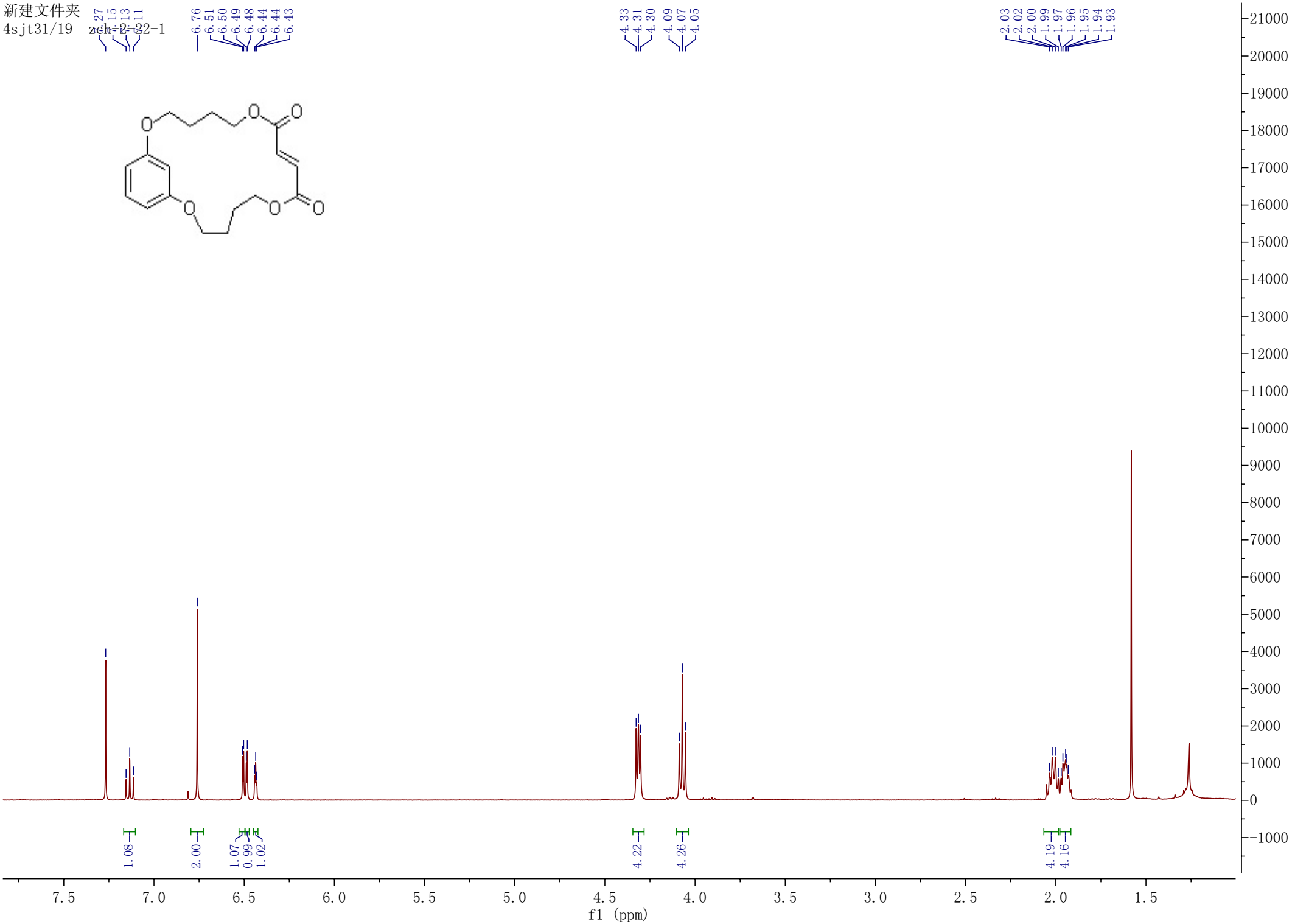
2014-7-4
4sj


6.25
4.36
4.34
4.32
4.04
4.03
4.02
1.98
1.98
1.96
1.94
1.92
1.92
1.91
1.89
1.89
1.87
1.87
1.61
0.01
4.08
2.00
4.26
4.32
4.02
4.05
f1 (ppm)

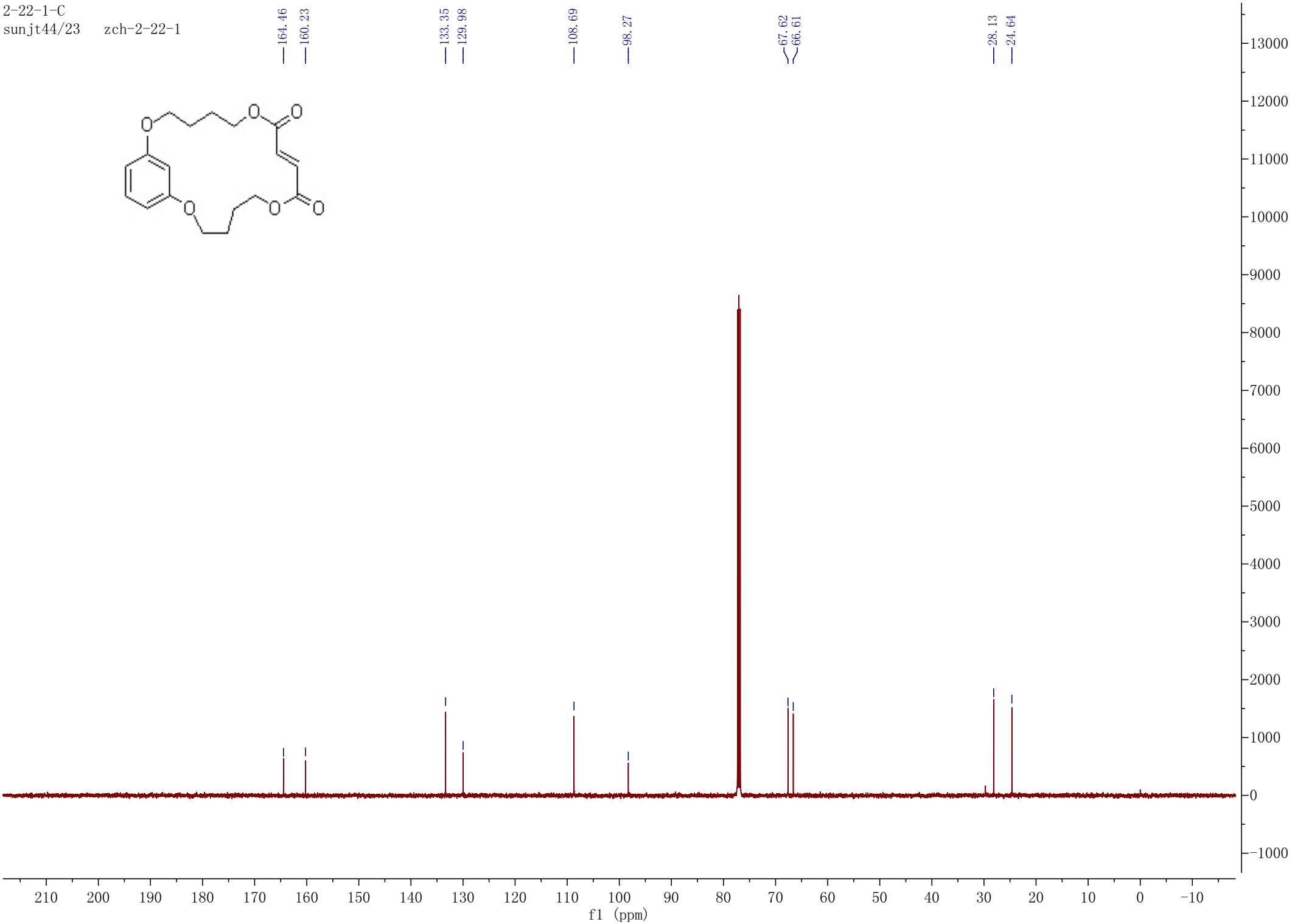
2014-7-4
4sj


5.4
3.8
129.7
121.0
113.0
68.3
65.6
26.1
25.9
400
350
300
250
200
150
100
50
0
−50
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

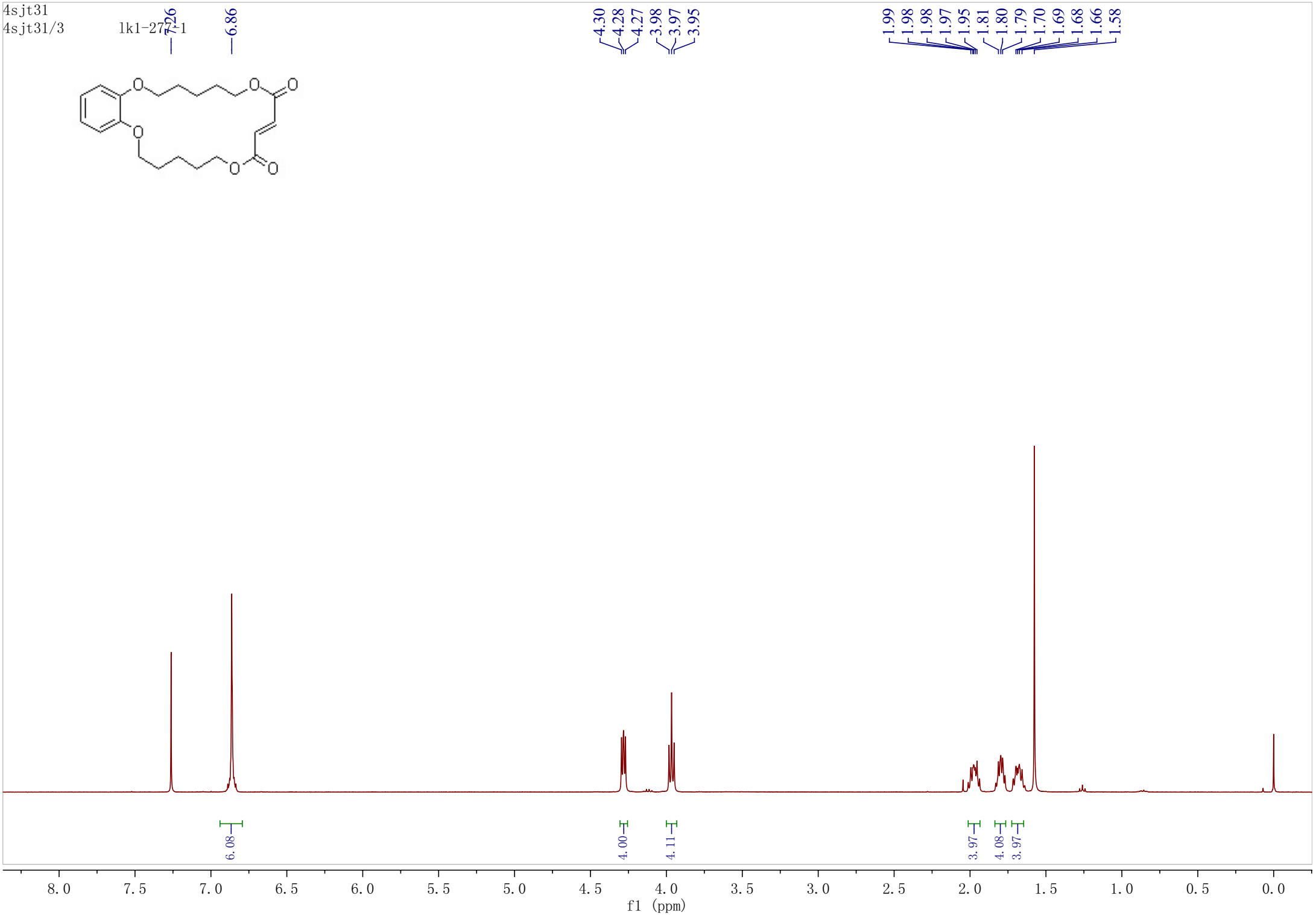
新建文件夹
4sjt31/19 zch-2-22-1



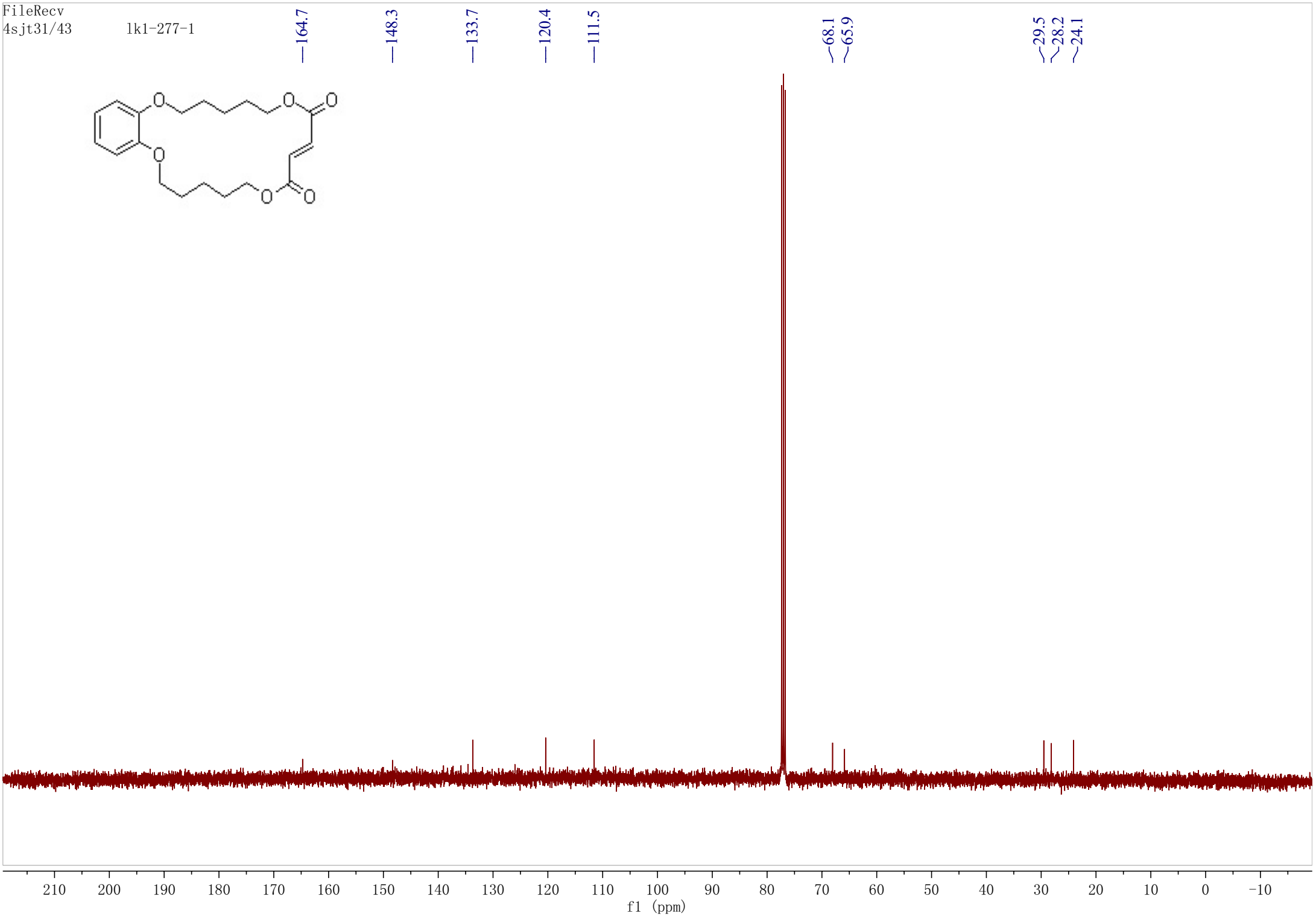
2-22-1-C
sunjt44/23 zch-2-22-1



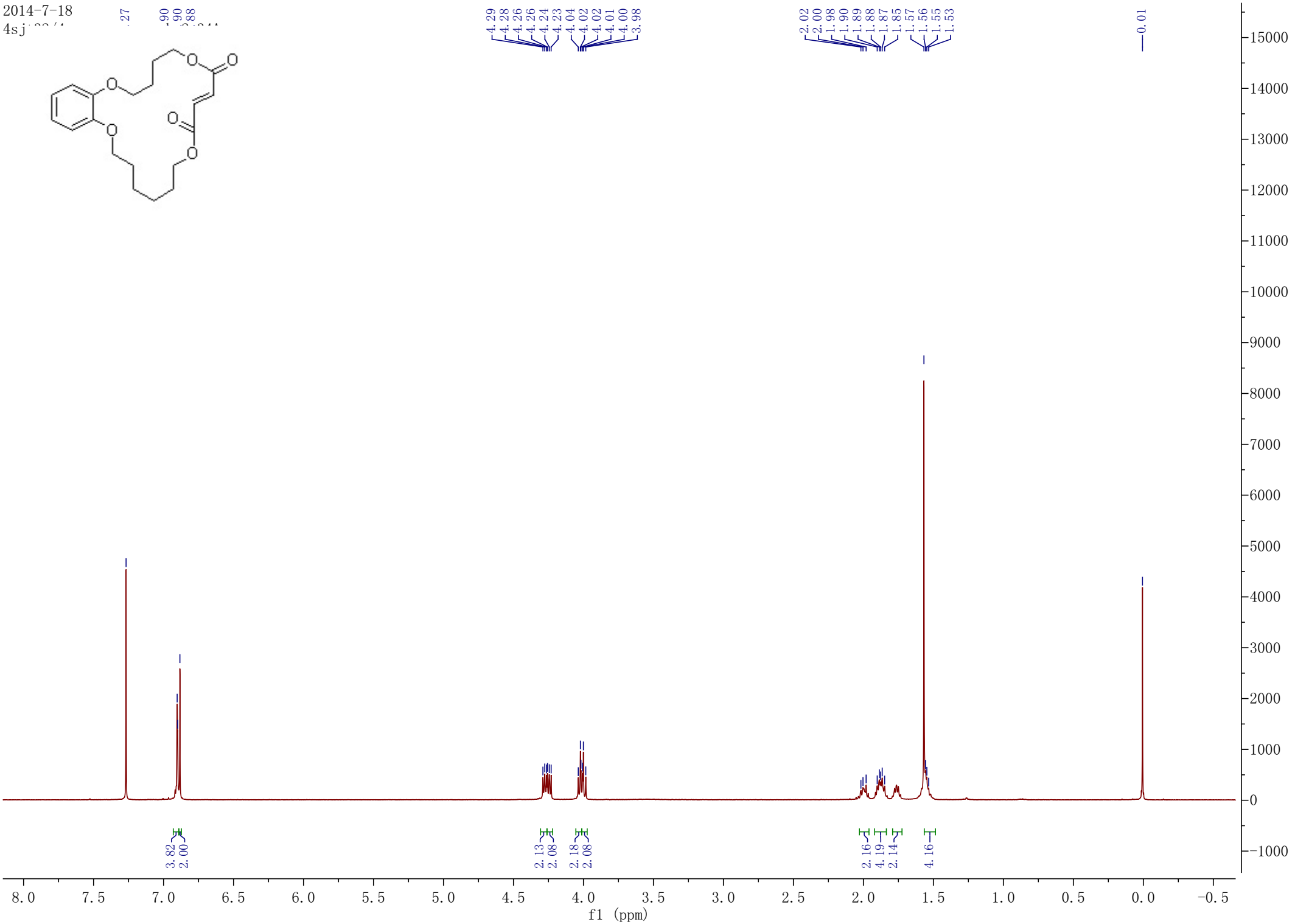
4sjt31
4sjt31/3 lk1-277-1



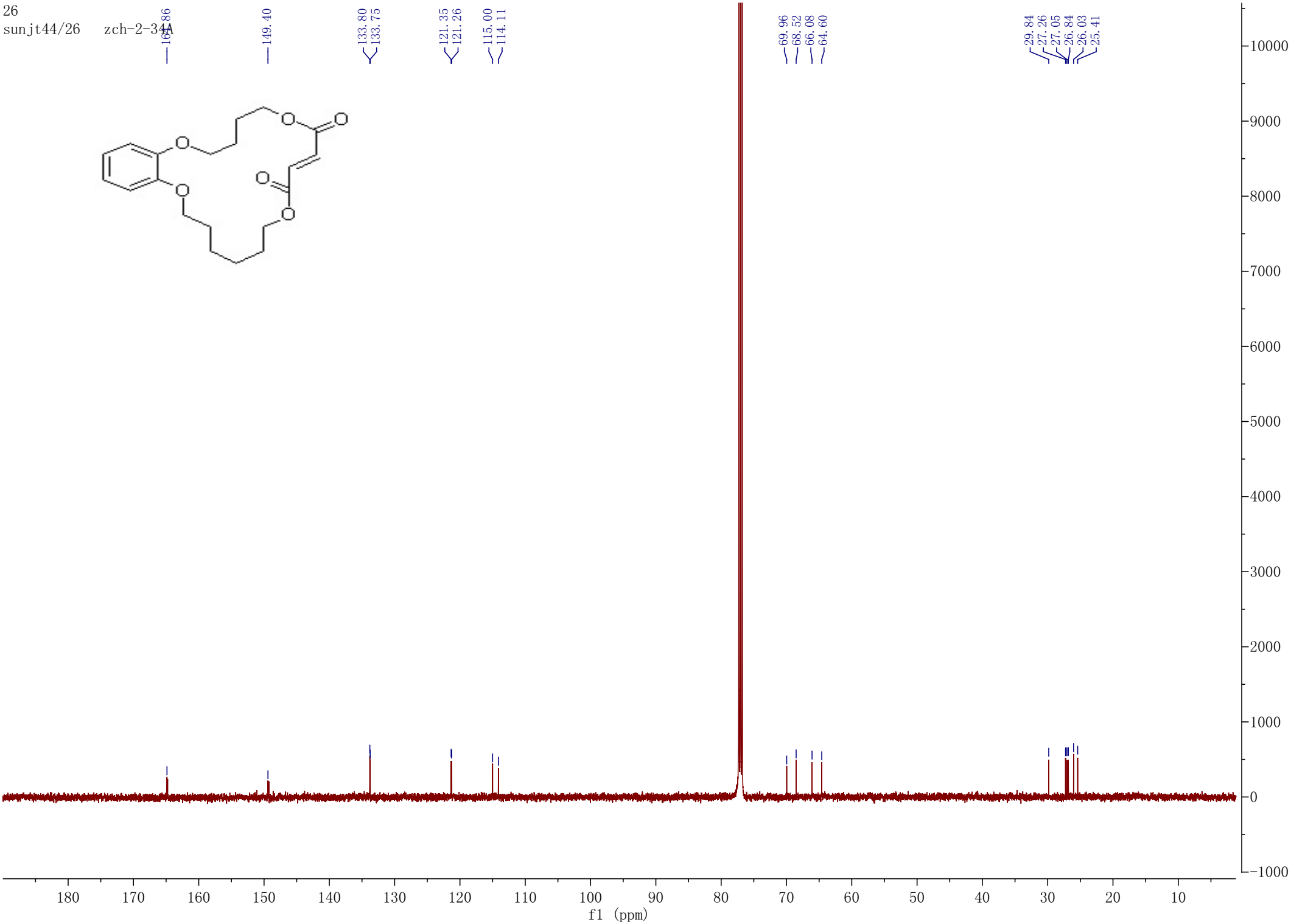
FileRecv
4sjt31/43 lk1-277-1



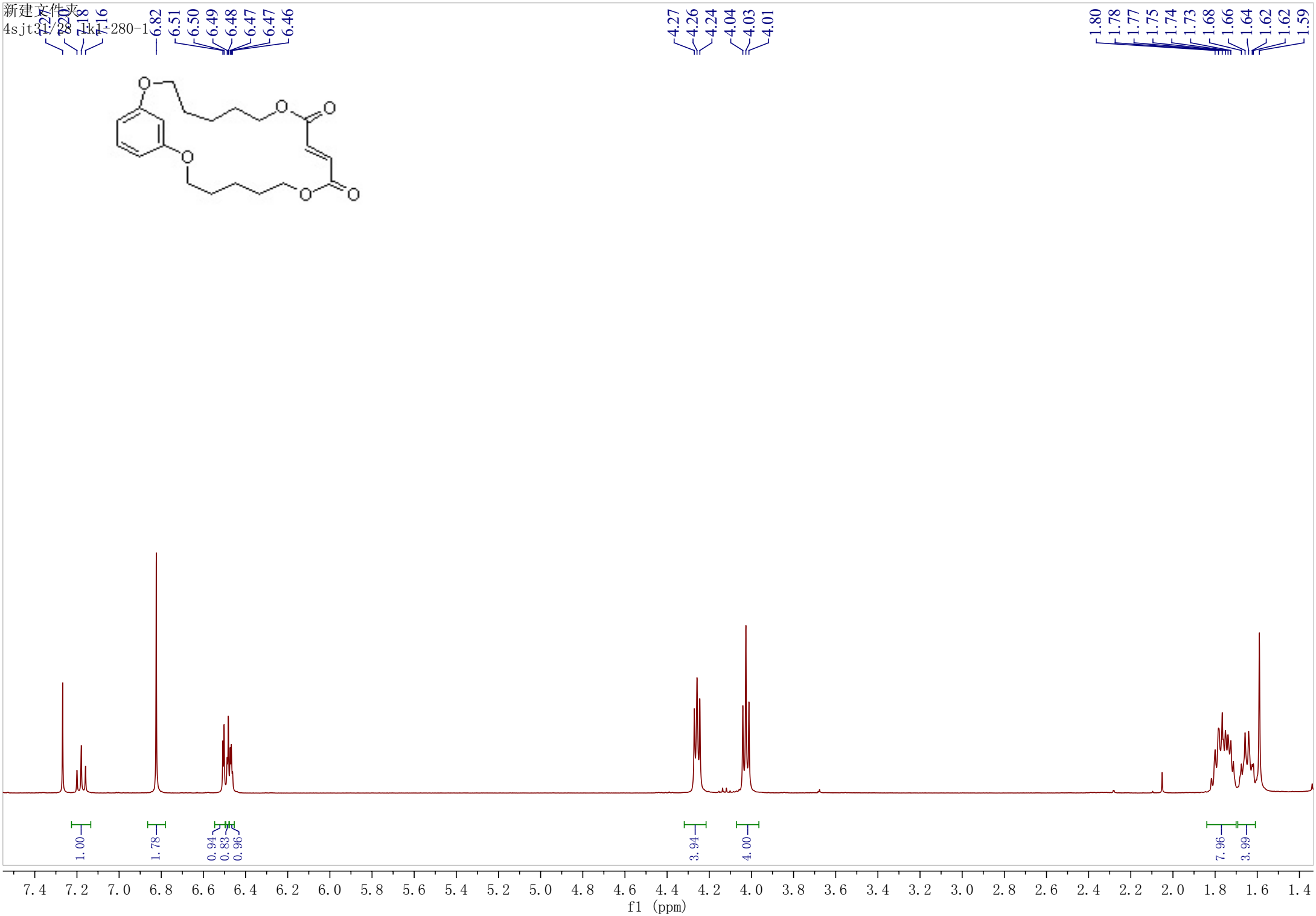
2014-7-18
4sj


4.29
4.28
4.26
4.26
4.24
4.23
4.04
4.02
4.02
4.01
4.00
3.98
2.02
2.00
1.98
1.90
1.89
1.88
1.87
1.85
1.57
1.56
1.55
1.53
0.01
3.82
2.00
2.13
2.08
2.18
2.08
2.16
4.19
2.14
4.16
f1 (ppm)

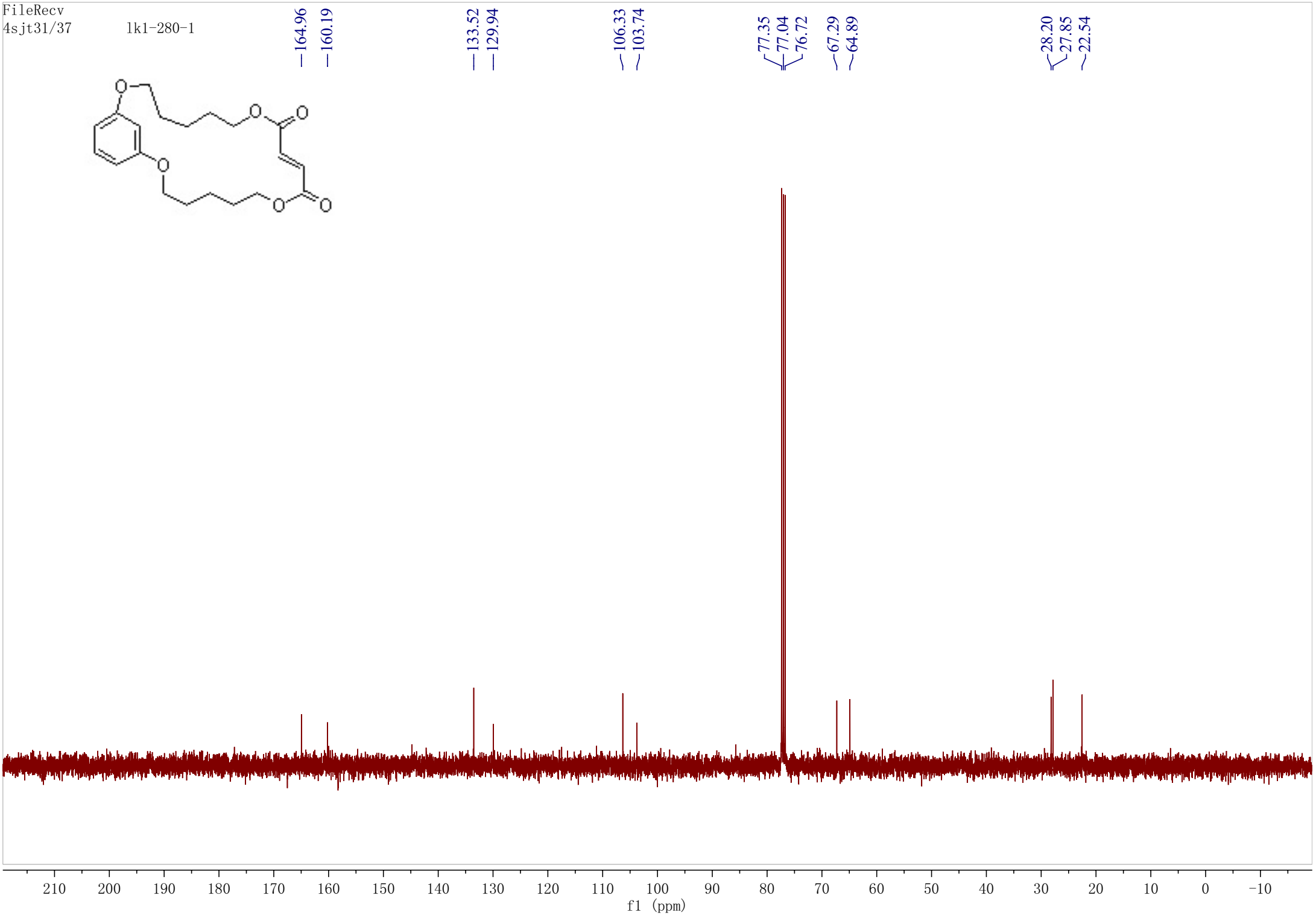
26
sunjt44/26 zch-2-34A

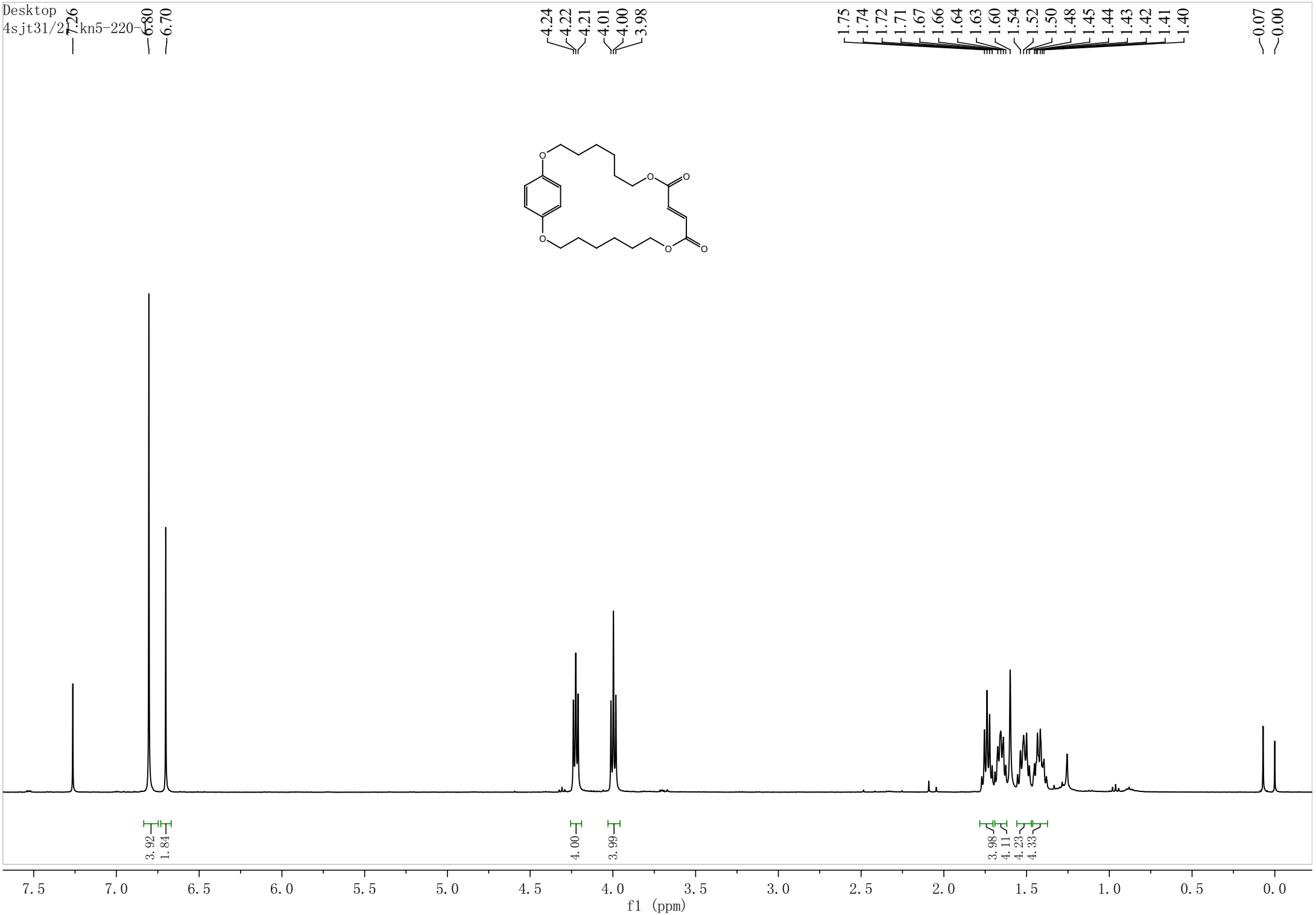


新建文件夹
4sjt31/28 1kl-280-1

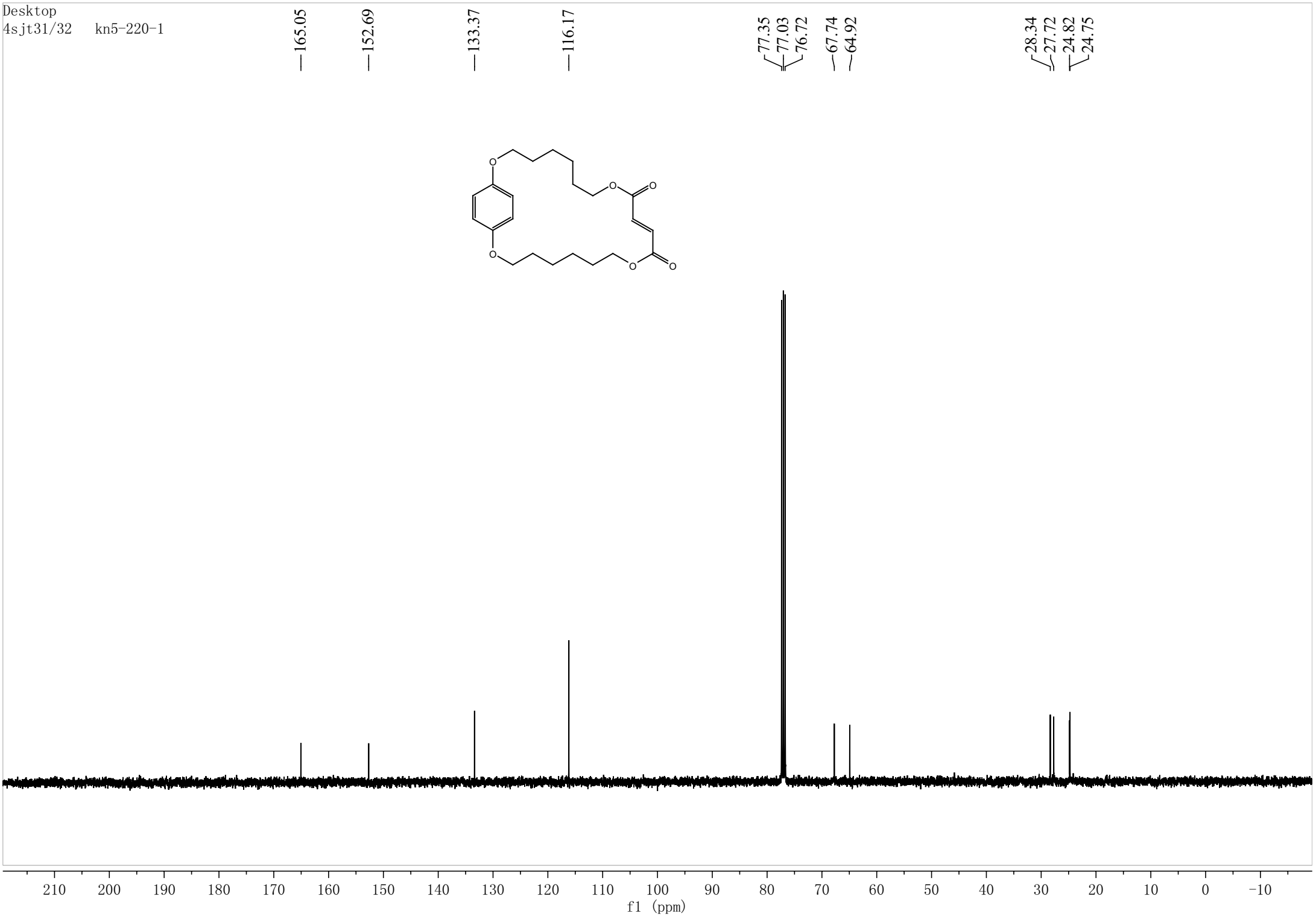


FileRecv
4sjt31/37 lk1-280-1

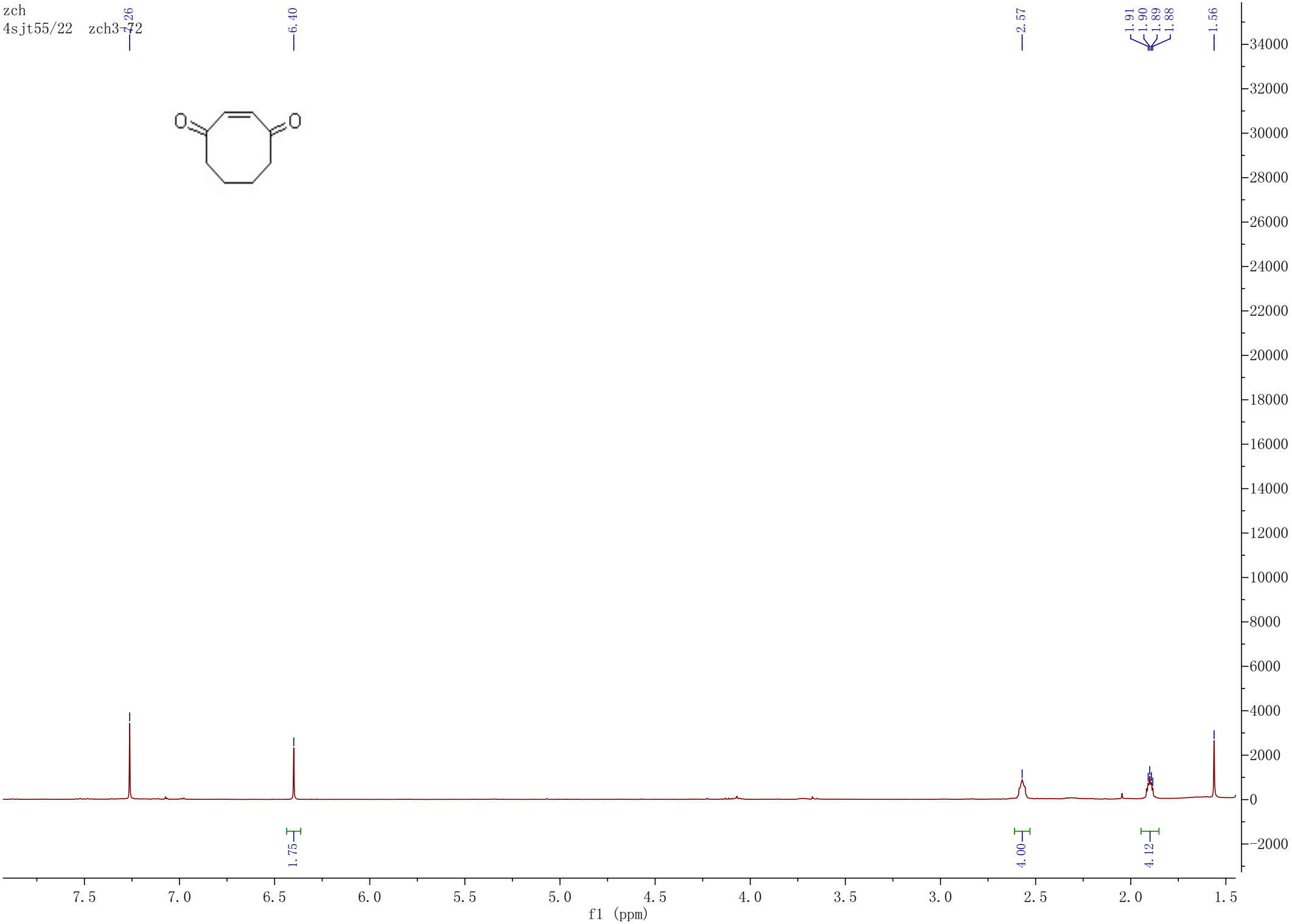



Desktop
4sjt31/21-kn5-220-1
7.26
6.80
6.70
4.24
4.22
4.21
4.01
4.00
3.98
1.75
1.74
1.72
1.71
1.67
1.66
1.64
1.63
1.60
1.54
1.52
1.50
1.48
1.45
1.44
1.43
1.42
1.41
1.40
0.07
0.00
3.92
1.84
4.00
3.99
3.98
4.11
4.23
4.33
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

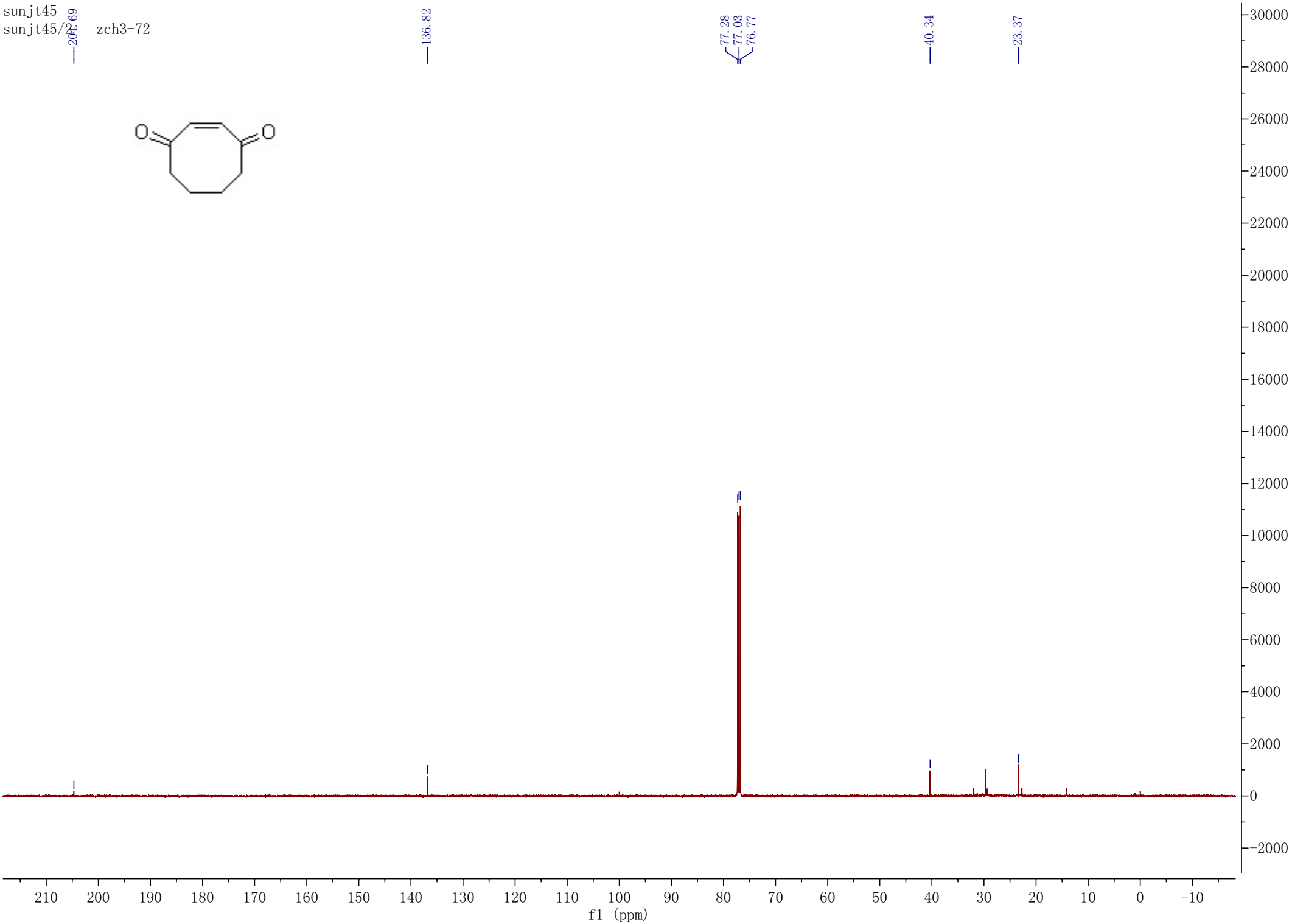
Desktop
4sjt31/32 kn5-220-1



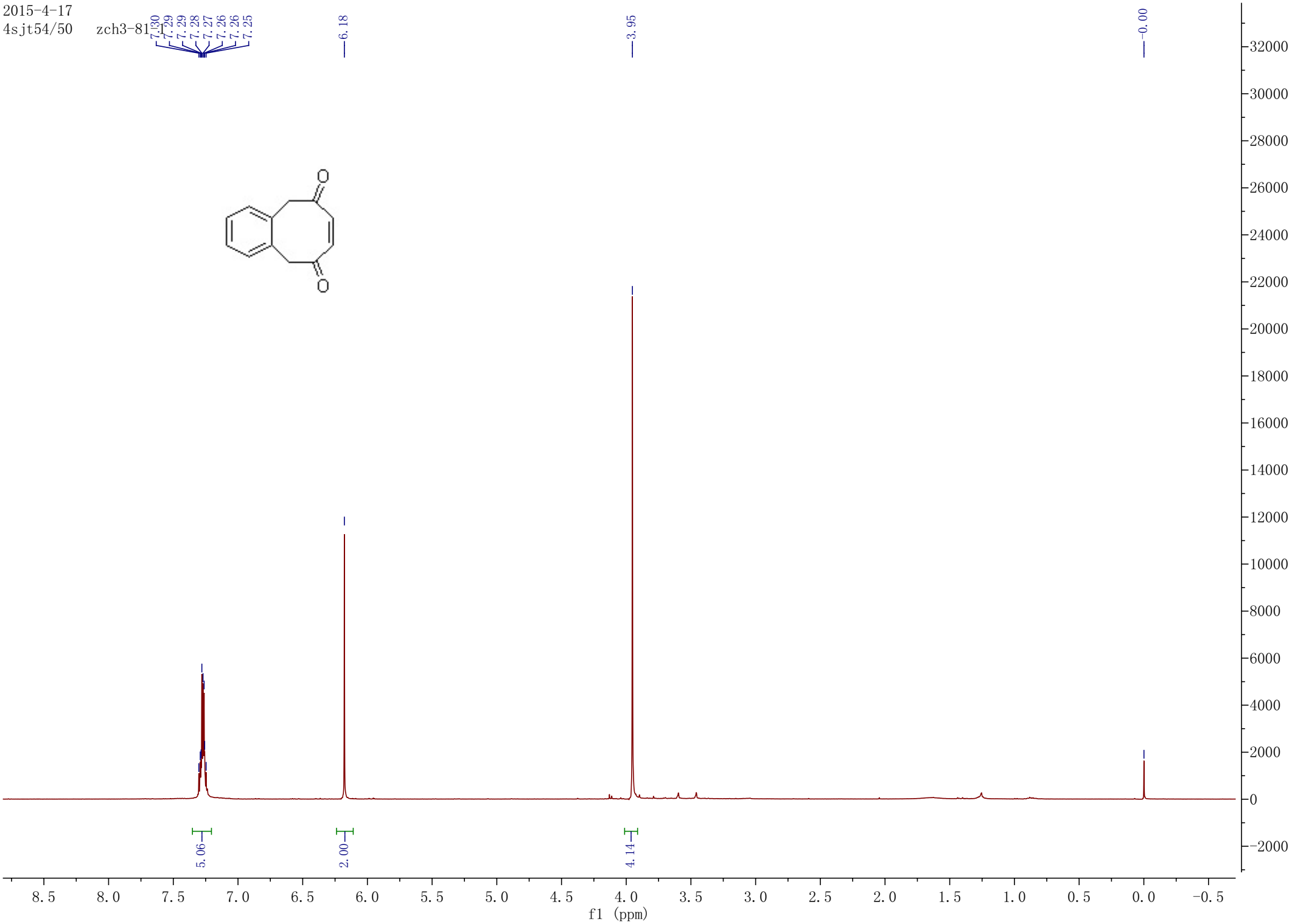
zch
4sjt55/22 zch3-72



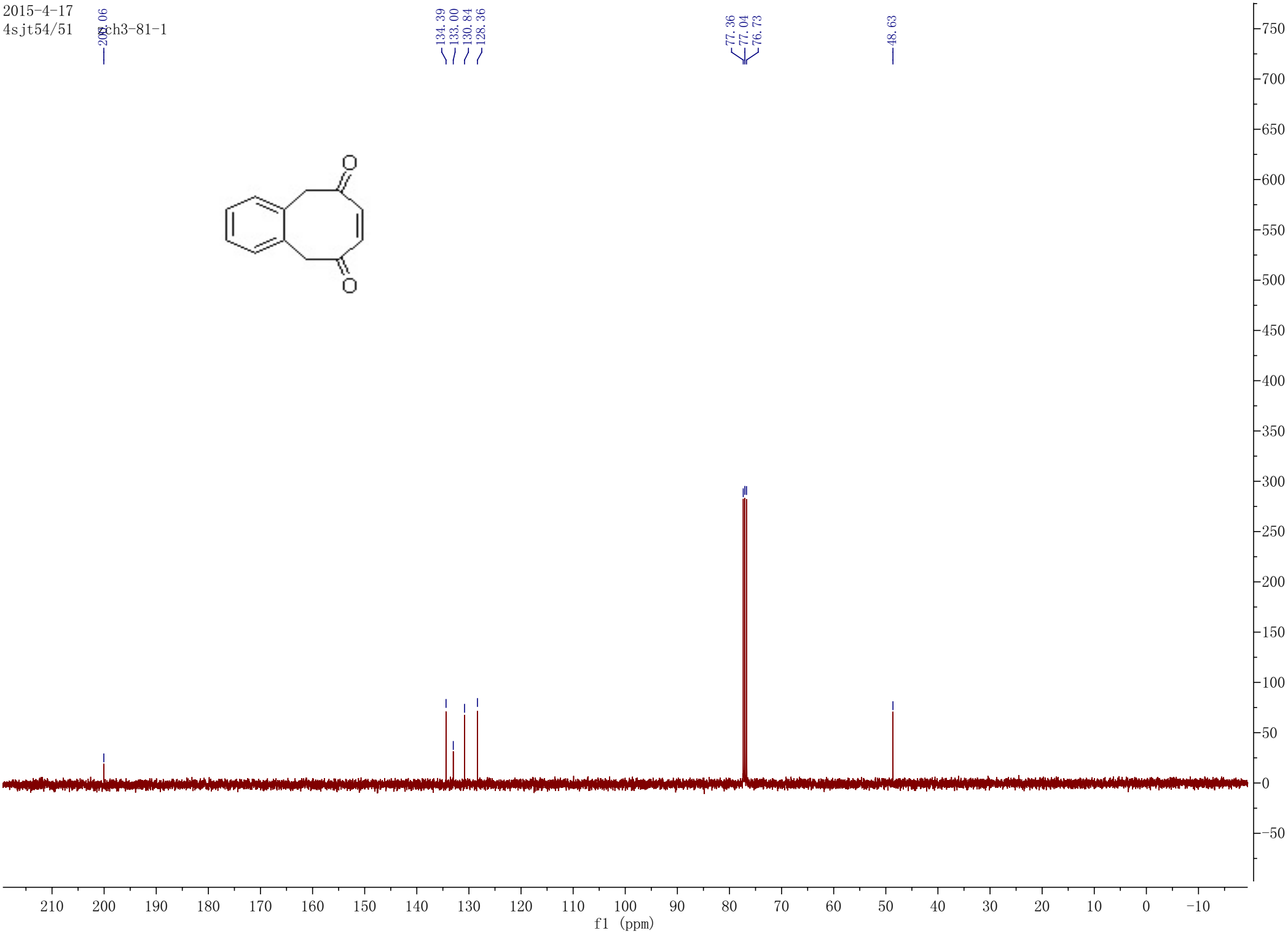
sunjt45
sunjt45/2 zch3-72



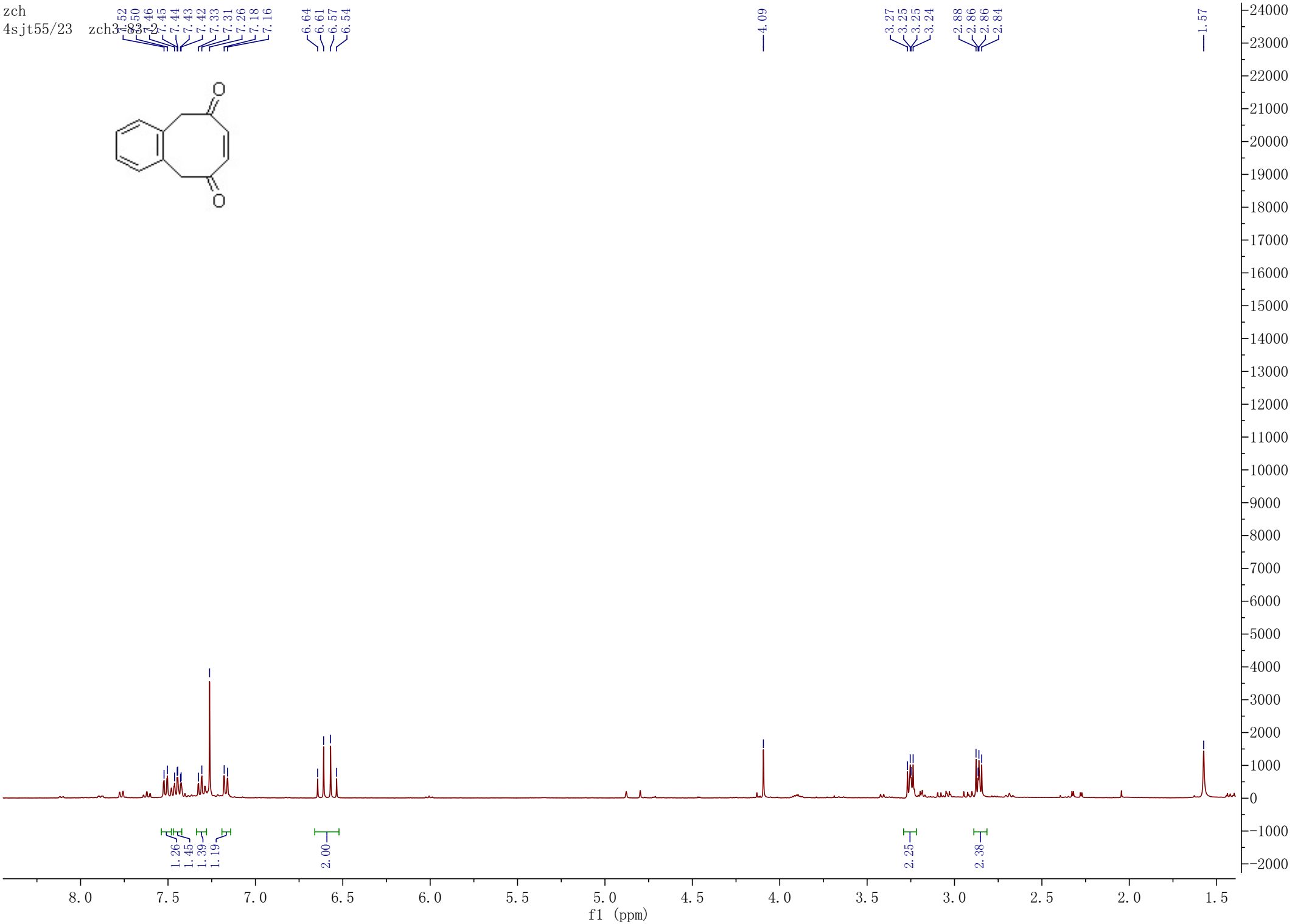
2015-4-17
4sjt54/50 zch3-81-1



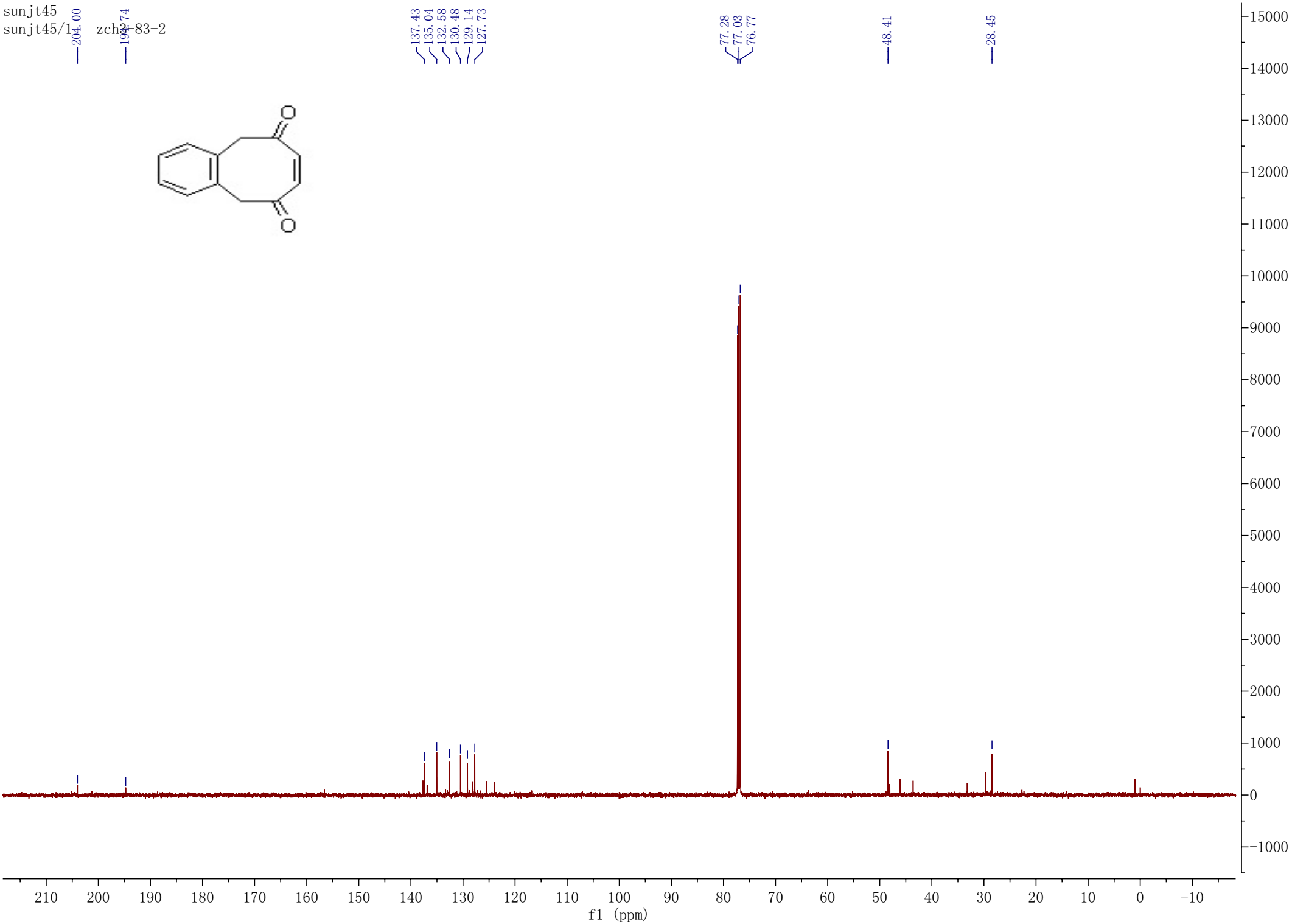
2015-4-17
4sjt54/51 zch3-81-1



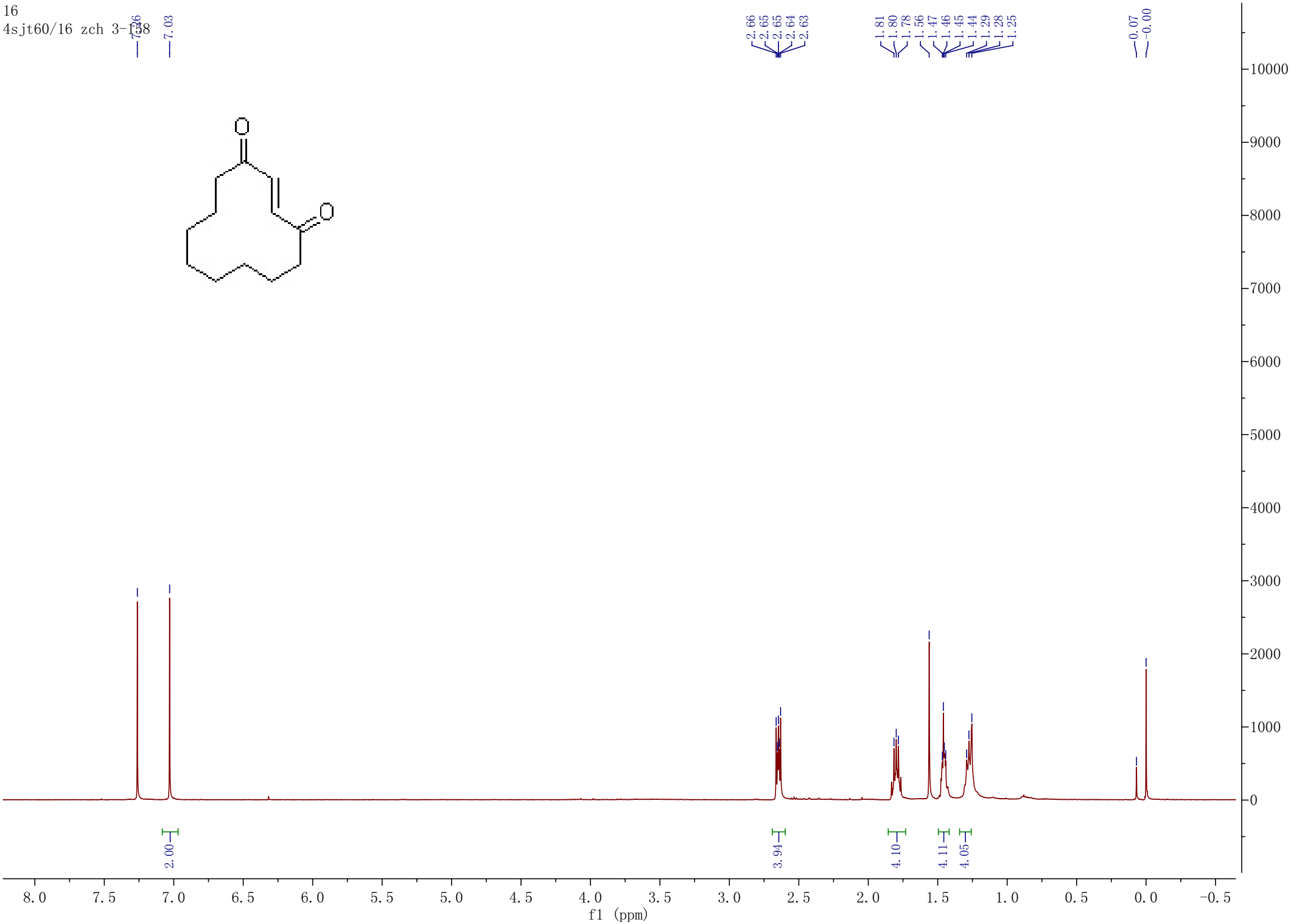
zch
4sjt55/23 zch3-83-2



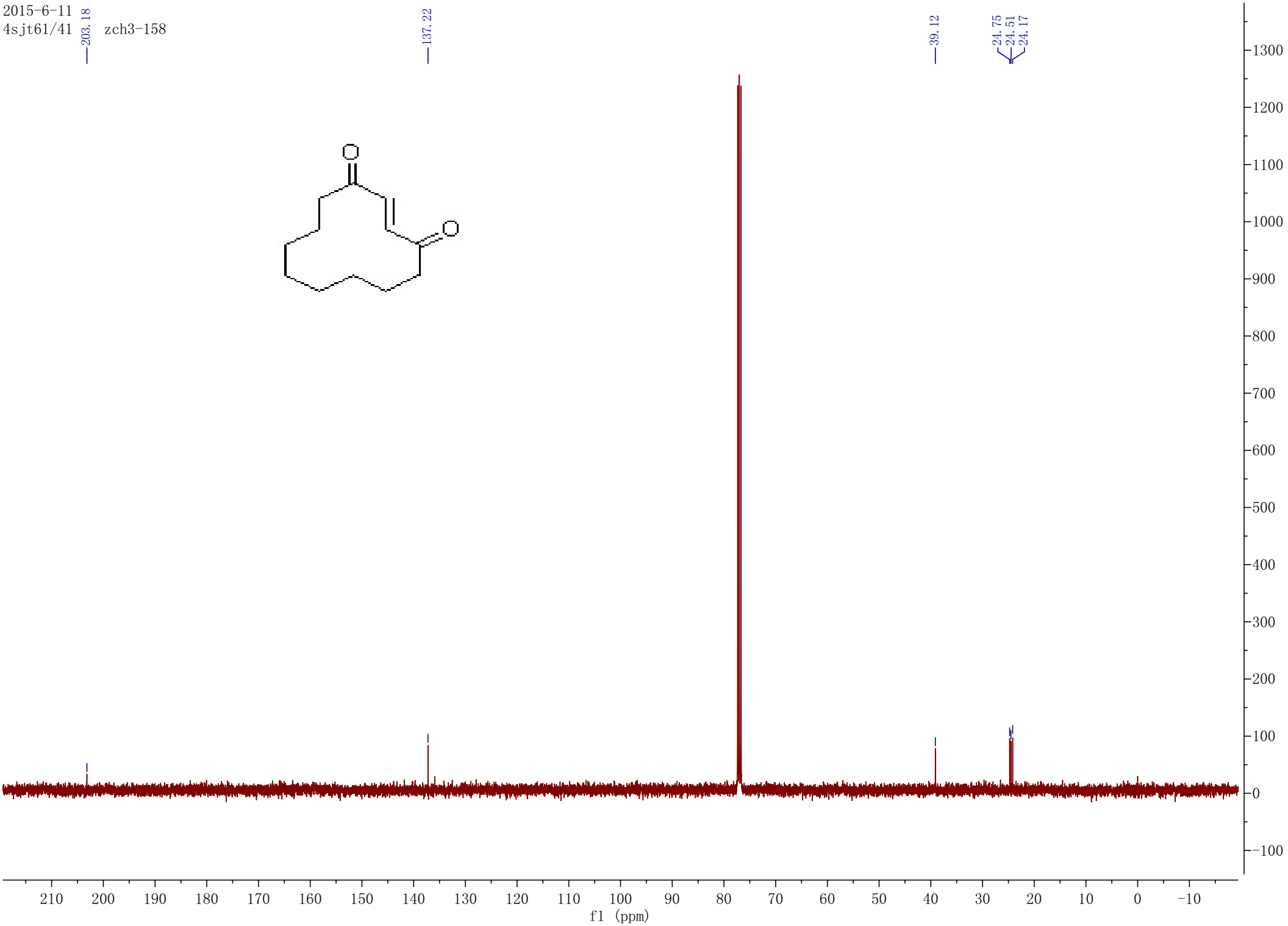
sunjt45
sunjt45/1 zch3-83-2



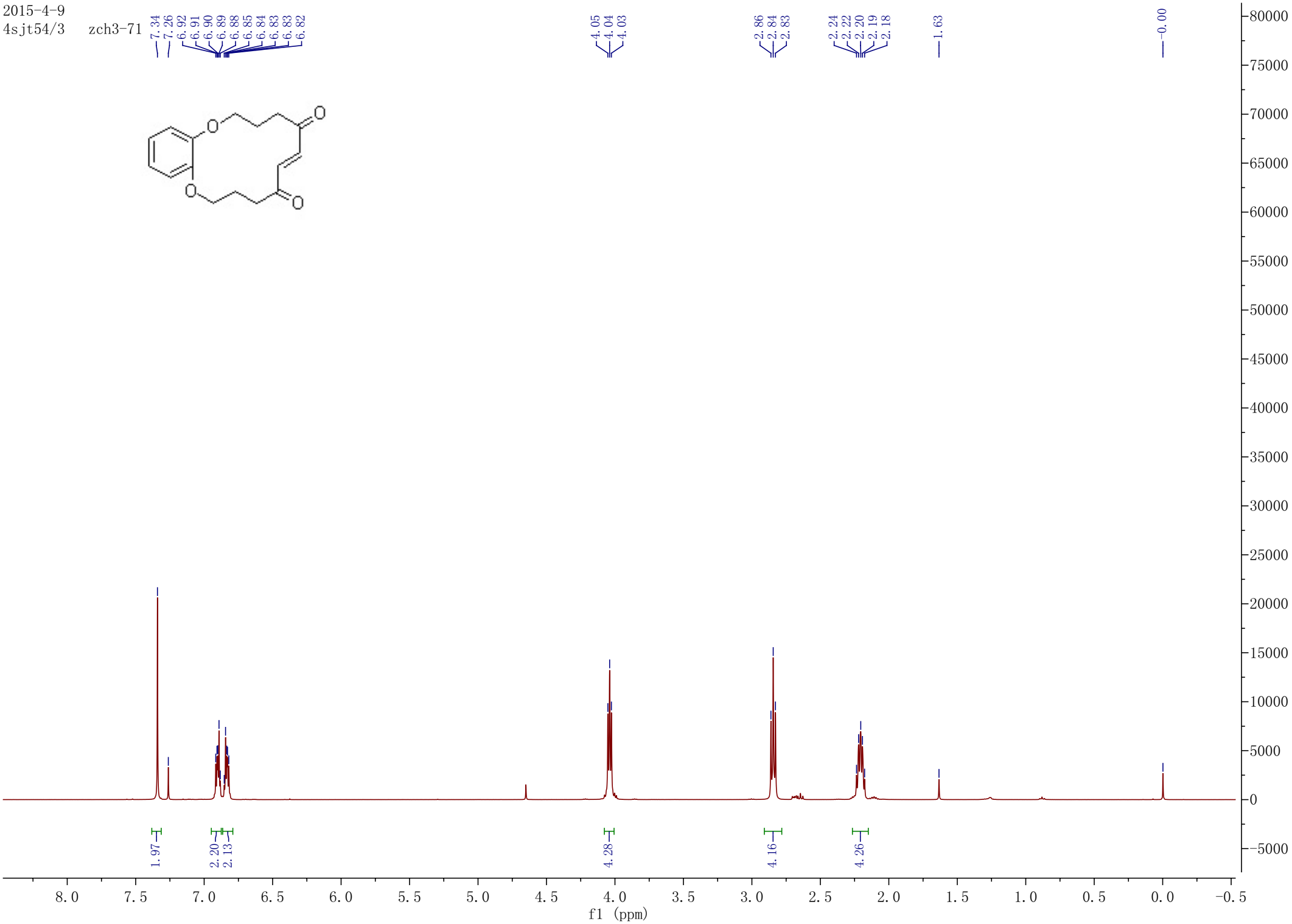
16
4sjt60/16 zch 3-138



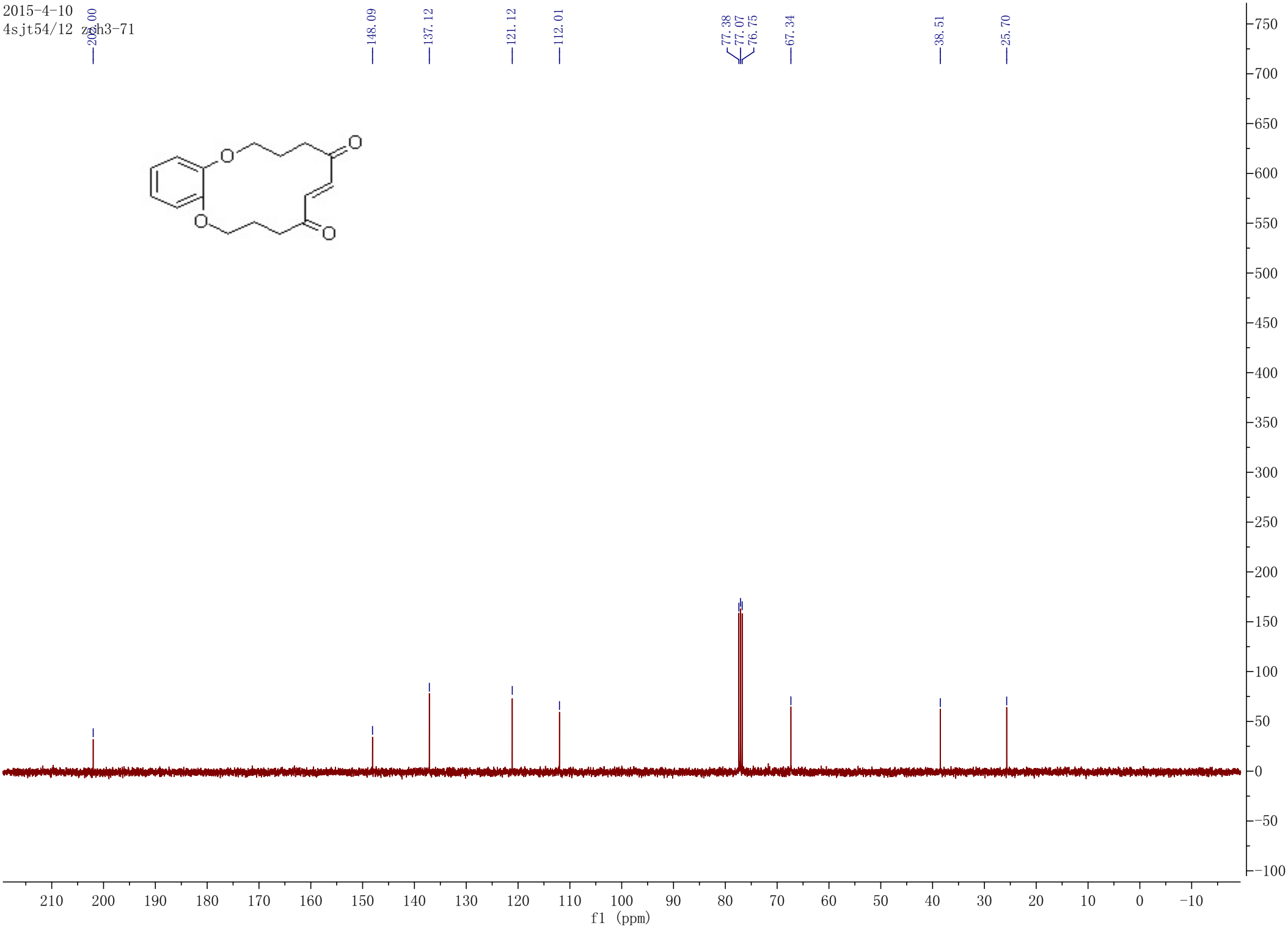
2015-6-11
4sjt61/41 zch3-158



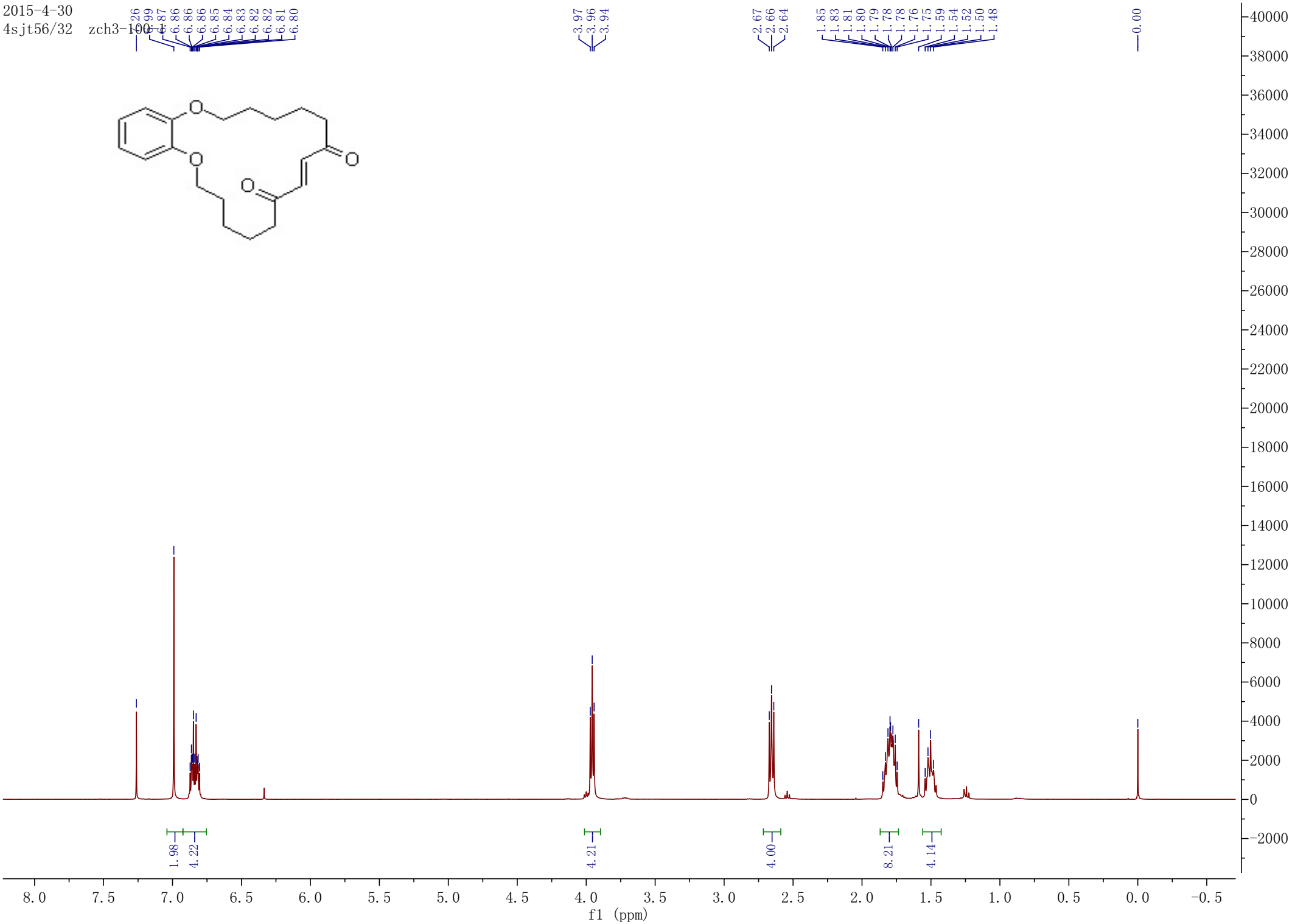
2015-4-9
4sjt54/3 zch3-71



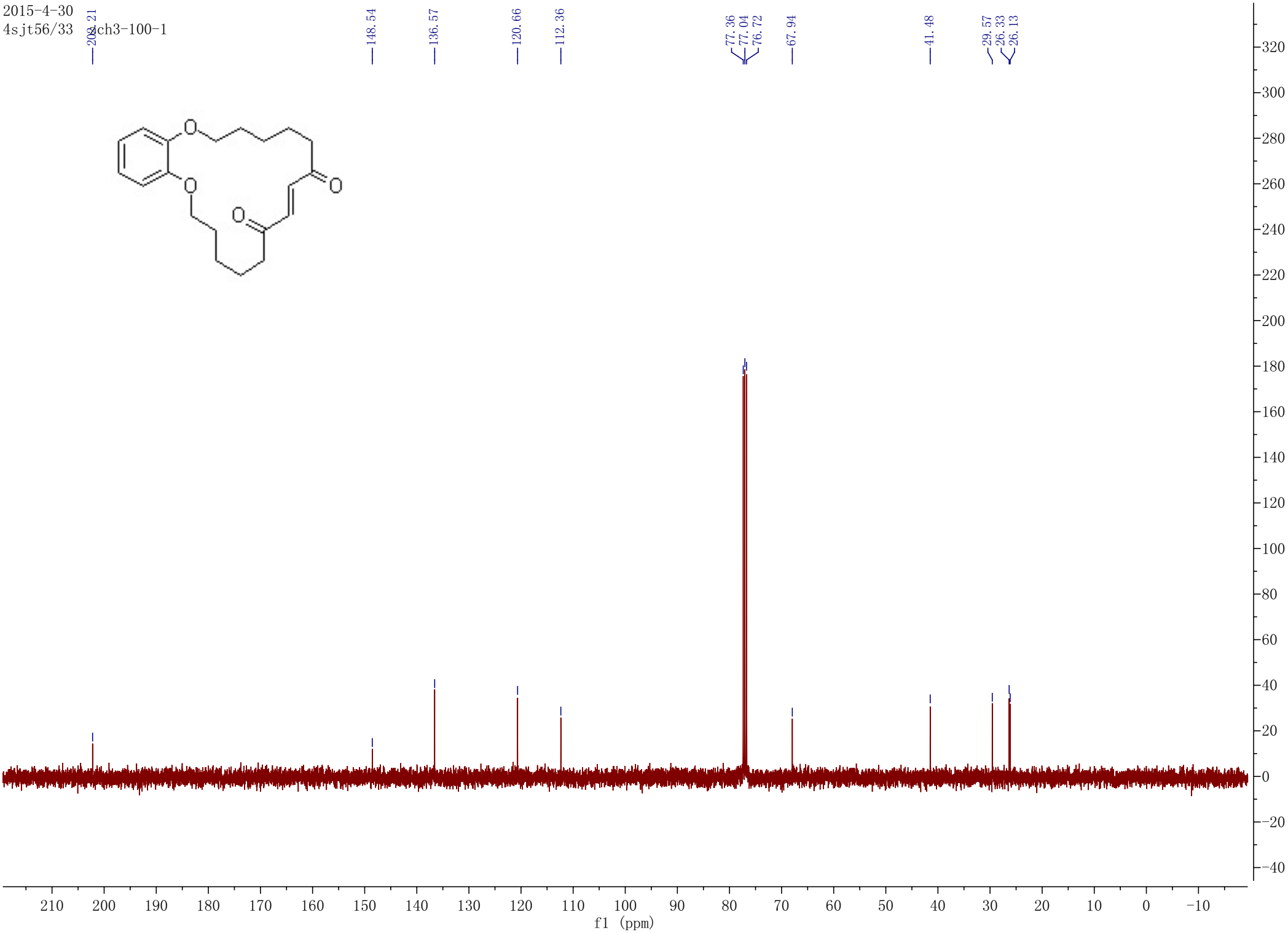
2015-4-10
4sjt54/12 zqh3-71



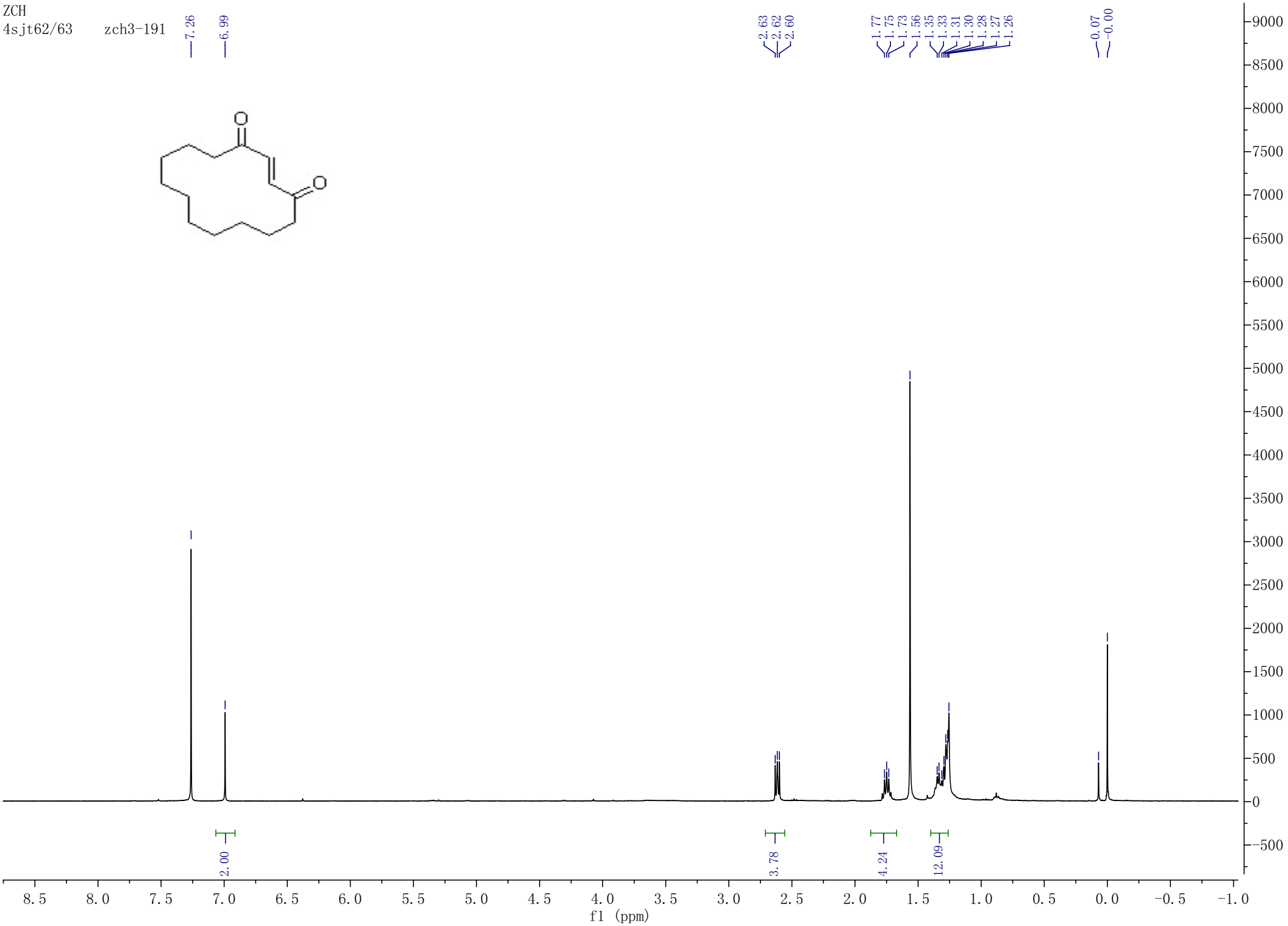
2015-4-30
4sjt56/32 zch3-100-1



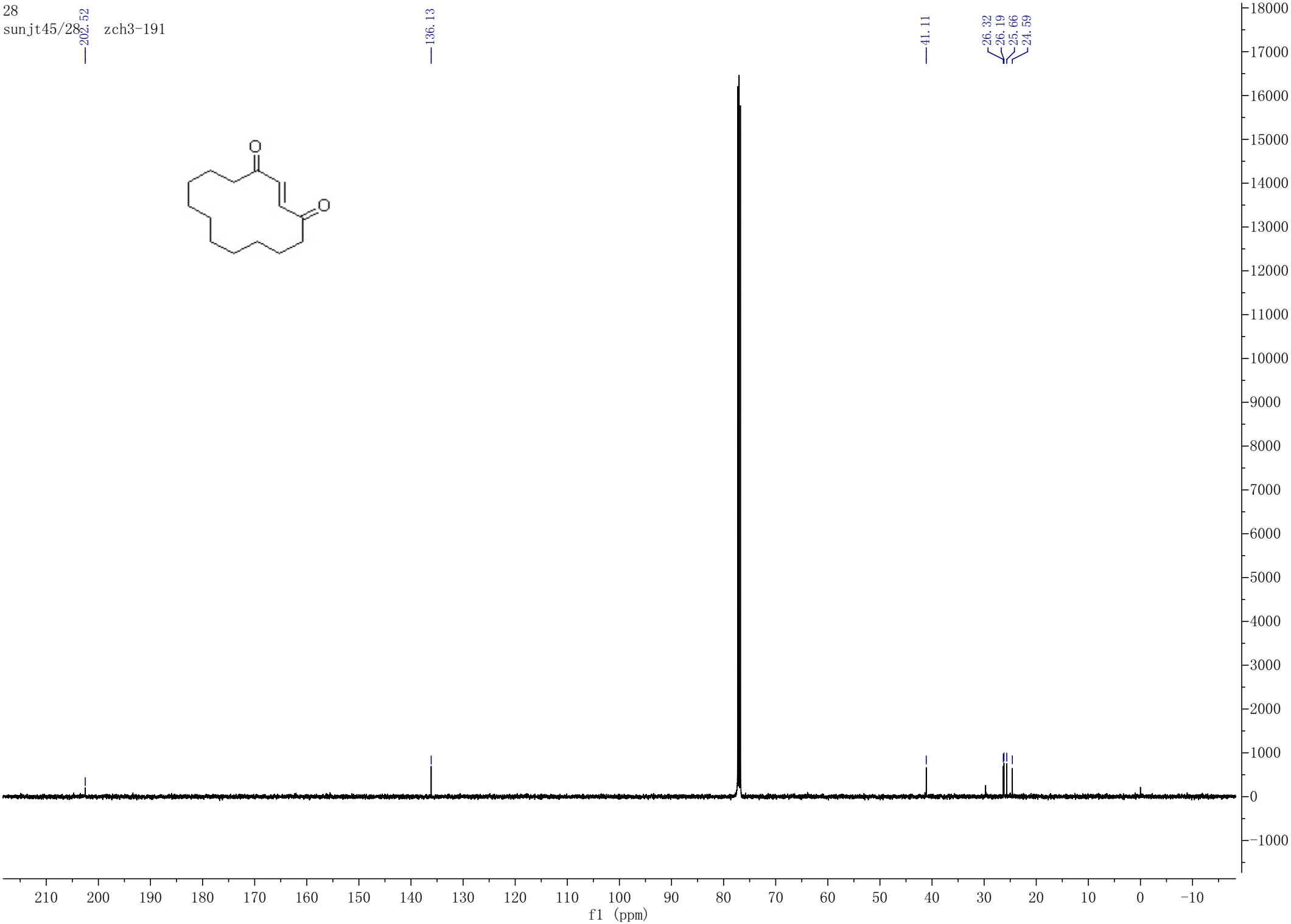
2015-4-30
4sjt56/33 sch3-100-1



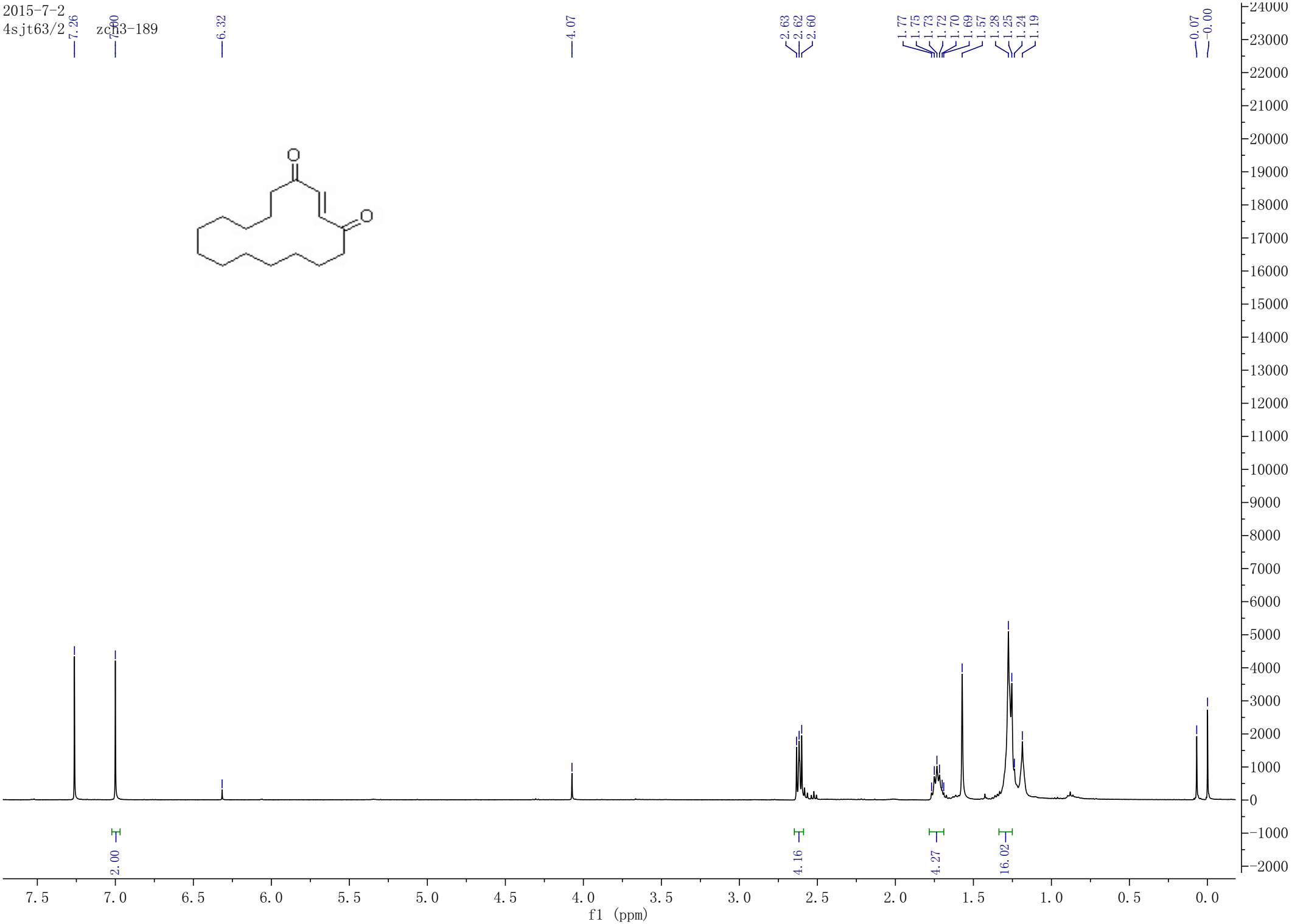
ZCH
4sjt62/63 zch3-191



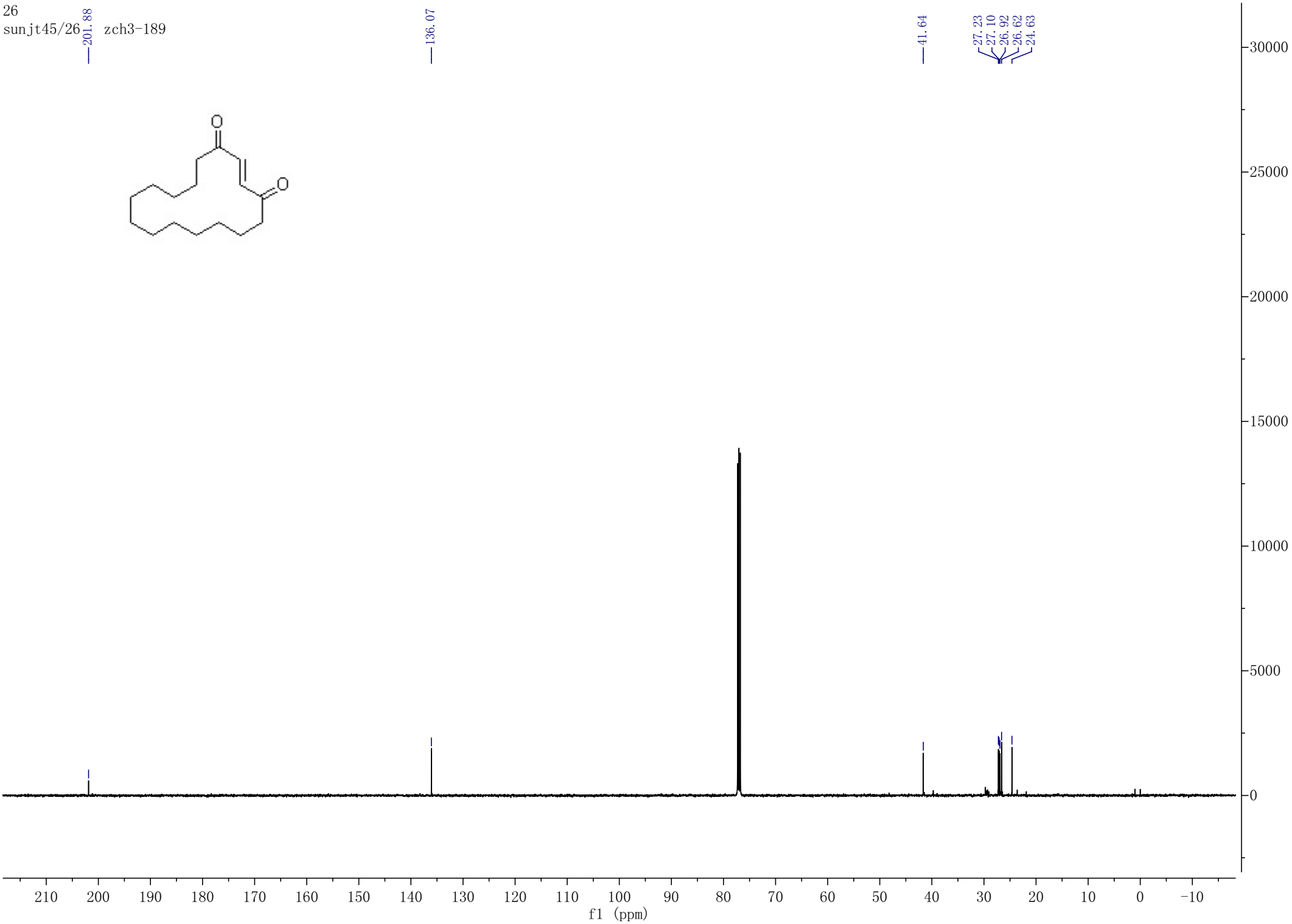
28
sunjt45/28 zch3-191



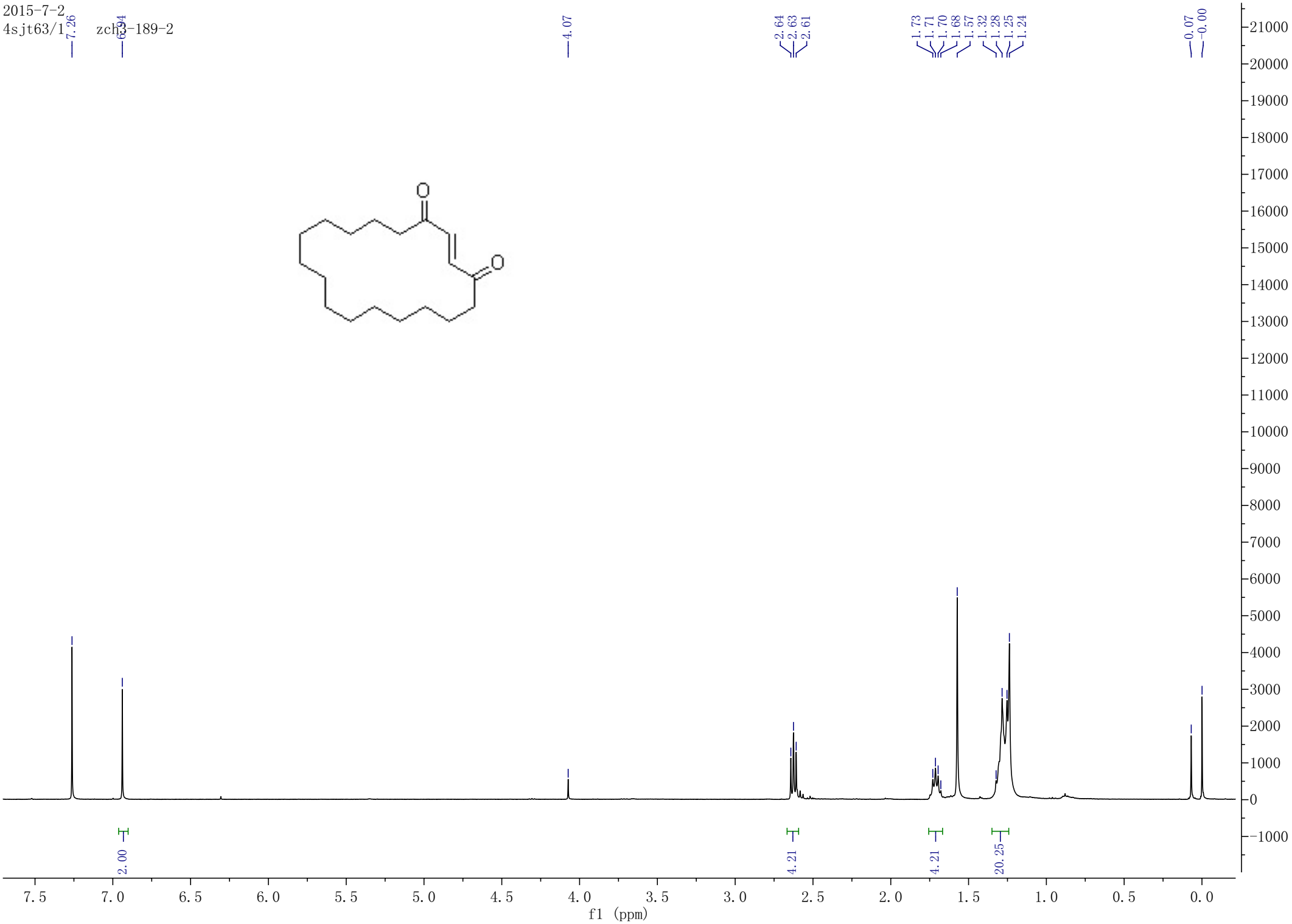
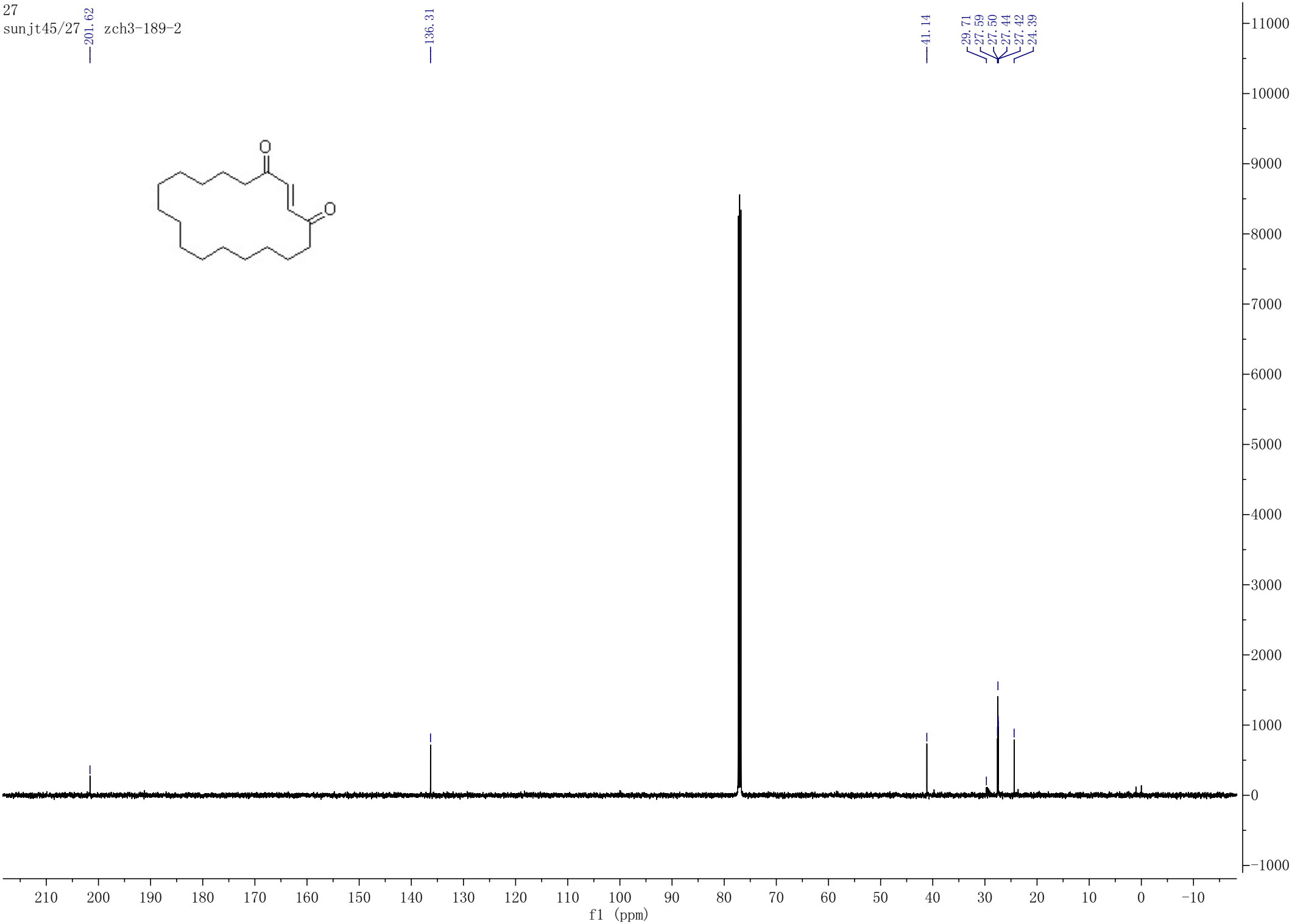
2015-7-2
4sjt63/2 zc33-189



26
sunjt45/26 zch3-189



2015-7-2
4sjt63/1 zch3-189-2



27
sunjt45/27 zch3-189-2